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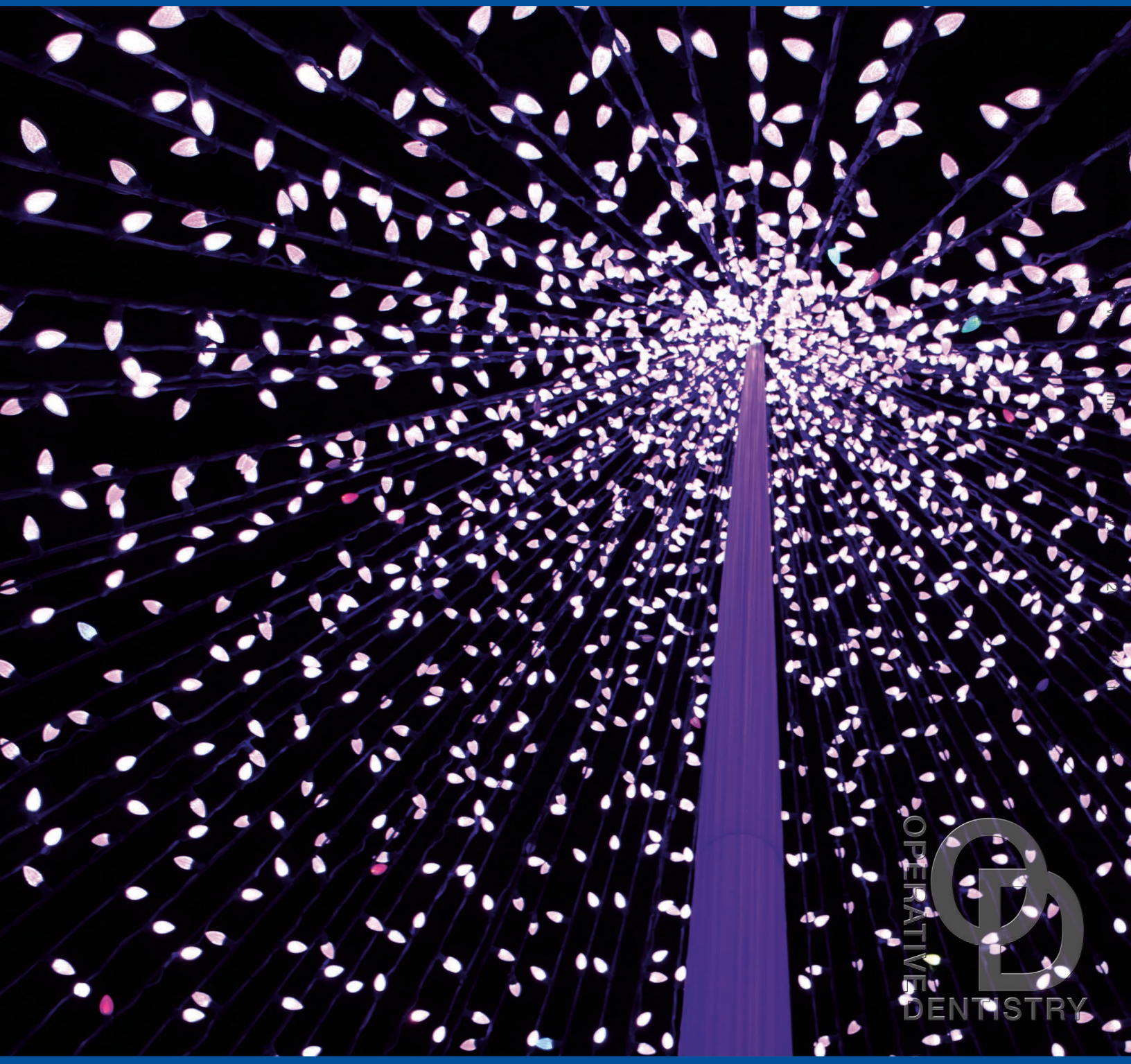

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OPERATIVE DENTISTRY

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

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

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Clinical Research

Effects of At-home Bleaching in Smokers: 30-month Follow-up

JL de Geus • E Fernández • S Kossatz • AD Loguercio • A Reis

Clinical Relevance

The results of this study indicate that bleaching is effective in smokers and nonsmokers but a slight color rebound was observed for both groups of patients after 30 months.

SUMMARY

Objective: This clinical study evaluated the color longevity after 30 months of at-home bleaching with 10% carbamide peroxide (CP) in smokers and nonsmokers.

Methods: Sixty patients, 30 smokers and 30 nonsmokers, were subjected to bleaching with 10% CP (Whiteness Perfect–FGM) for three hours daily for three weeks. The color was measured at baseline and at one month and 30 months after the completion of dental bleaching using the shade guide Vita classical organized by value (ΔSGU) and the shade guide Vita Bleachedguide 3D-MASTER. At the 30-month recall, the color was assessed before and after dental prophylaxis. Data from color evaluation were analyzed by two-way repeated-measures analysis of variance and Tukey test for the contrast of means (α=0.05).

Results: Twenty-one smokers and 22 nonsmokers attended the 30 month recall. For both shade guides, only the main factor of assessment time was statistically significant ($p<0.001$). Effective whitening was observed in both groups at the baseline, which was stable at one month. However, color rebound was observed after 30 months for both groups of participants when color was measured before and after dental prophylaxis.

Conclusion: Thirty months after at-home bleaching with 10% CP gel, dental darkening was detected in both groups, which cannot be solely attributed to stains caused by extrinsic staining from daily food, drinks, and smoke (in smokers).

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INTRODUCTION

Currently dental bleaching is one of the most requested treatments by patients as a result of the fact that white and well-aligned teeth are considered important factors in the concept of a beautiful smile.[1] At-home dental bleaching using 10% carbamide peroxide (CP) gel with custom trays is the most widely used bleaching technique for tooth discoloration treatments.[2]

Although many studies of at-home bleaching have been conducted, most of them exclude smokers from their clinical trials,[3-7] which prevents us from assessing the effect of this cosmetic procedure on such patients. Contrary to this widespread concept, an earlier publication of de Geus and others[8] demonstrated that effective whitening is achievable regardless of whether or not the patient is a smoker.

However, smokers' teeth tend to develop tobacco stains over time.[9,10] Considering that these stains may vary from yellow to black and given that the severity is highly dependent on the length and frequency of the smoking habit, concerns about the longevity of such treatment were raised. The whitening outcome in smokers was shown to remain stable one year after the bleaching treatment so long as teeth were submitted to dental prophylaxis before color evaluation.[11] These results suggest that color rebound after bleaching results from the deposition of pigments or cigarette smoke on the dental surface in such a short, one-year follow-up period.

Although there are numerous studies[12-16] that evaluated the longevity of at-home bleaching, even for periods as long as 12 years, none of them have attempted to appraise bleaching longevity after dental prophylaxis in patients who were smokers. Therefore, the aim of this controlled clinical trial was to compare the 30-month color change associated with at-home bleaching with 10% CP in smokers and nonsmokers before and after dental prophylaxis. The null hypotheses tested were that 1) no significant difference will be detected between smokers and nonsmokers after 30 months and 2) no color rebound will be detected in both groups of participants before and after dental prophylaxis.

METHODS AND MATERIALS

This study is the 30-month follow-up of an earlier study[8] registered at clinicaltrials.gov under the identification number NCT02017873. This earlier study was conducted in Chilean and Brazilian centers,[8] but the follow-up was only performed on the Brazilian participants.

Inclusion and Exclusion Criteria

We evaluated participants in a dental chair, after dental prophylaxis with pumice and water, to check whether they met the study's eligibility criteria. Participants included in this clinical trial were between 18 and 54 years of age and had good general and oral health. Each participant had at least one central incisor of shade A2 or darker, as assessed by means of comparison with a value-oriented shade guide (VITA classical, VITA Zahnfabrik, Bad Säckingen, Germany). Color A2 is the fifth color in the light-to-dark value VITA classical shade guide scale, so there are still five shades to allow measurement of color changes with this scale. This minimal color shade was already employed in many other clinical trials.[4-7]

We did not include participants who had undergone previous dental bleaching procedures during orthodontic treatment or those who were pregnant, lactating, or who exhibited bruxism habits. In addition, we excluded participants with noncarious cervical lesions and buccal restorations in anterior teeth as well as those having veneers or full crowns, dental fluorosis, gingival recession, spontaneous tooth pain, internal tooth discoloration, and endodontically treated anterior teeth. Patients with bruxism habits were excluded as they usually have a high prevalence of noncarious cervical lesions, which are frequently associated with dentin sensitivity.[17]

Study Groups

We asked the participants who met the inclusion criteria about their daily smoking habits. Those who did not smoke were part of the nonsmoker group, and those who smoked at least 10 cigarettes per day belonged to the group of smokers. We included 30 participants in each group.

Bleaching Procedure

We made alginate impressions of each participant's maxillary and mandibular arch, pouring the impressions with dental stone. We did not apply block-out material to the labial surfaces of the teeth.[18] We used a 1-mm–thick soft vinyl material provided by the manufacturer (Whiteness, FGM, Joinville, SC, Brazil) to fabricate the custom-fitted tray to hold the bleaching gel. We trimmed the bleaching tray 1 mm beyond the marginal gingiva and delivered the tray and the 10% CP gel (Whiteness Perfect, FGM) to each participant with oral instructions for use. We instructed all participants to wear the tray with the bleaching agent for three hours daily for three weeks.

We instructed the participants to remove the tray after the daily bleaching period, wash it with water, and brush their teeth as usual. We also provided verbal instructions about oral hygiene, encouraging participants to brush their teeth regularly with fluoridated toothpastes without whitening components.

Color Evaluation

We checked the color in the middle one-third area of the labial surface in the anterior central incisor, according to the American Dental Association guidelines.[19] We used the Vita Bleachedguide 3D-MASTER (VITA Zahnfabrik), which is originally oriented from lightest to darkest color, and the VITA classical shade guide (VITA Zahnfabrik). The 16 classical shade guide tabs (VITA classical, VITA Zahnfabrik) were arranged from lightest to darkest as follows: B1, A1, B2, D2, A2, C1, C2, D4, A3, D3, B3, A3.5, B4, C3, A4, and C4. Although this scale is not linear, we treated the changes as continuous, with linear ranking used in several clinical trials on dental bleaching.[4,6]

We calculated the color changes from the beginning of the active phase through the individual recall times by the change in shade guide units (ΔSGU) that occurred toward the lighter end of the value-oriented list of shade tabs. In cases in which operators disagreed about color matching, we reached a consensus before dismissing the patient.

Two calibrated evaluators, with a previous agreement of at least 85% as determined by means of weighted *k* statistics, recorded the shade of the maxillary right central incisor at the baseline and at one week, one month, 12 months, and 30 months after finishing the bleaching protocol. At 12 and 30 months, the evaluation was performed before and after dental prophylaxis with a rotating brush and prophylaxis paste (Herjos, Vigodent Coltene SA Indústria e Comércio, Rio de Janeiro, Brazil). After dental prophylaxis, teeth were rehydrated in the patient's mouth for 15 minutes before color assessment. This care was taken because the teeth became lighter as they were dehydrated,[20] which could have affected the reliability of the data collected.

Satisfaction Assessment

In the 30-month recall, the participants were asked to answer some closed-ended questions about their satisfaction level concerning the bleaching outcome, their perception of color change, and their perception of color rebound after 30 months.

Statistical Analysis

We performed all of the analyses using the statistical software Statistica for Windows (StatSoft Inc, Tulsa, OK, USA), with a 5% significance level. Two statistical analyses were performed using the per-protocol (only for the available data) and the intention-to-treat approaches. In the latter, the last observation was carried forward for the missing data. The color change in ΔSGU from both shade guide units was submitted to two-way repeated-measures analysis of variance (ANOVA) (group vs assessment period) and Tukey test for pairwise comparisons. As a result of the exploratory nature of the satisfaction assessment data, we did not submit these data to statistical analysis; only descriptive analyses were performed.

Table 1: *Demographic Characteristics of the Participants Included in This Randomized Clinical Trial*

Characteristics	Groups	
	Smokers	Nonsmokers
Baseline color, shade guide VITA classical (mean±SD)	6.8 ± 2.3	7.3 ± 2.5
Baseline color, shade guide Bleachedguide 3D-MASTER (mean±SD)	7.8 ± 1.1	8.2 ± 1.3
Age, y (mean±SD)	26.3 ± 6.5	24.1 ± 6.8
Sex, % male	63.3	53.3
Cigarettes/d (mean±SD)	13.2 ± 4.0	—
Cigarettes/d at 30 mo (mean±SD)	11.8 ± 5.1	—
Average smoking years (mean±SD)	8.0 ± 5.9	—

Abbreviations: SD, standard deviation; SGU, shade guide unit.

RESULTS

At the baseline, we screened 305 patients to obtain 60 participants from the Brazilian center who met the eligibility criteria (Figure 1). The average age and baseline color of the participants were similar between the groups. Most of the participants were men (Table 1). The 12-month data were published earlier.[11] At the 30-month recall, smoking habits did not change among the majority of participants from the smoking group. Only five of the participants stopped smoking, while four reduced the number of cigarettes smoked per day (to less than 10 cigarettes a day).

All participants included in this controlled clinical trial finished the bleaching protocol and attended the one-week and one-month recall visits (Figure 1); however, 17 patients did not attend the 30-month recall. The reasons for not attending the recall included the change of housing location and a single participant lacking time availability to return to the university for a new color assessment.

Per-protocol vs Intention-to-treat Analysis

All statistical analyses were performed with data imputation for missing outcomes (intention-to-treat)

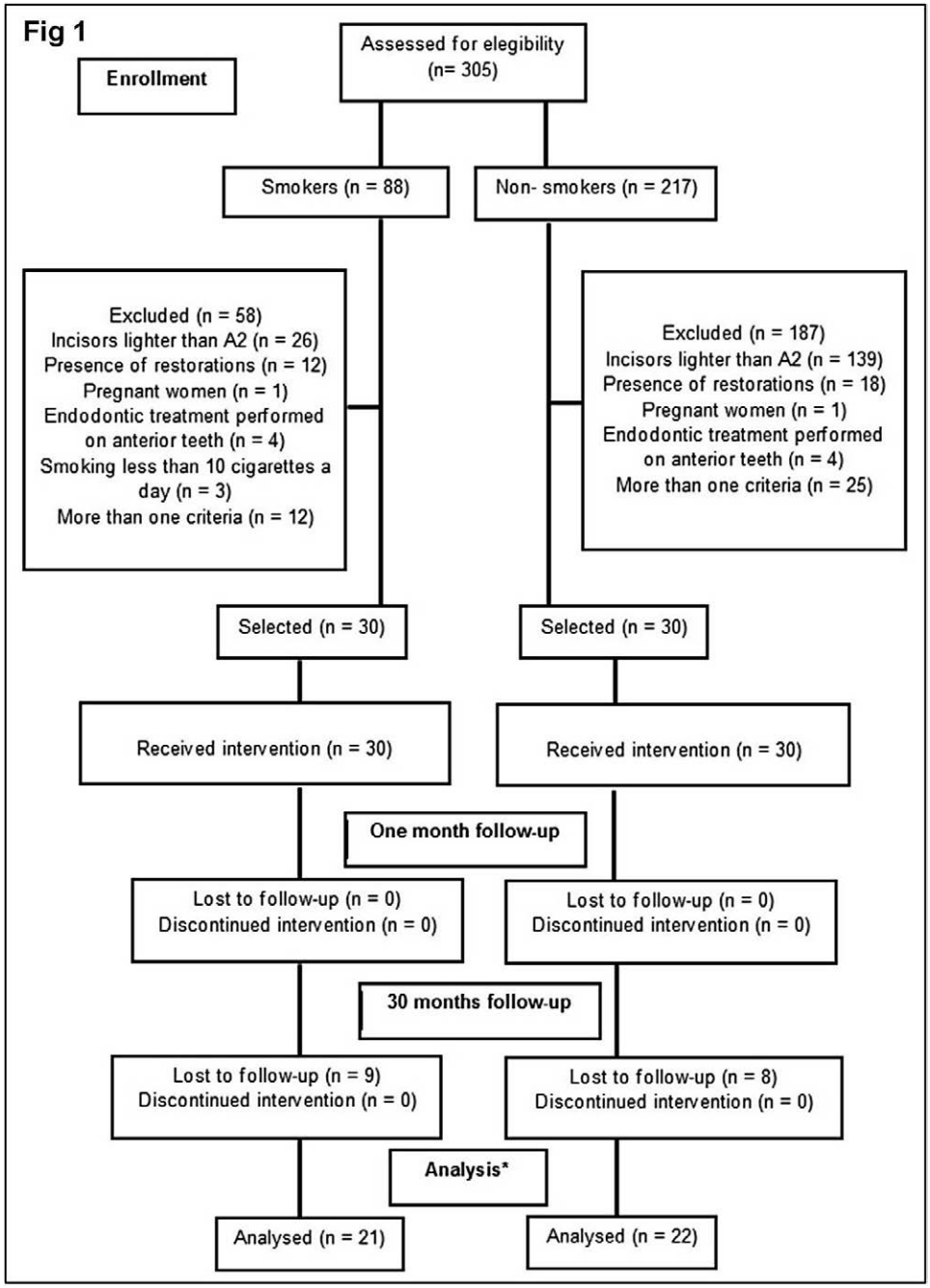

Figure 1. *Flow diagram of the clinical trial, including detailed information regarding the excluded participants. An intention-to-treat analysis, in which unit imputation was used for missing information, was also performed, and the overall conclusions were the same as those of the per-protocol analysis.*

and without data imputation (per-protocol). In all analyses, the same overall conclusions were reached (data not shown). To avoid data repetition, we opted to describe only the results and statistics obtained in the per-protocol analysis because of the fact that a high percentage of patients (17 out of 60 [28%]) could not be evaluated in the 30-month recall. The distribution of missing data was homogeneous among groups (n=9 in the smokers group and n=8 in the nonsmokers group).

Shade Evaluation

For both Vita classical shade guide and Vita Bleachedguide 3D-MASTER, the two-way repeated-measures ANOVA revealed that the cross-product interaction group vs assessment time (p=0.079 and p=0.378, respectively) and the main factor group (p=0.517 and p=0.051, respectively) were not significant. Only the main factor assessment time was statistically significant (Tables 2 and 3; $p<0.001$). The lack of difference between the groups (smokers

Table 2: *Color Change in Shade Guide Units (ΔSGU) Obtained with the Value-oriented Shade Guide Vita Classical at the Different Assessment Points Along with the Effect Size (Mean Difference) and the 95% Confidence Interval (CI)*

Assessment Time	Medians (Interquartile Range)		Mean ± Standard Deviation			
	Smokers	Nonsmokers	Smokers	Nonsmokers	Main Factor Time[a]	Mean Difference (95% CI)
Baseline vs 1 mo	4 (3.75-6.25)	5.5 (4-8)	5.2 ± 2.1	5.7 ± 2.3	5.5 ± 2.2 A	−0.5 (−1.9 to −0.9)
Baseline vs 30 mo before prophy	4 (3-6)	4.5 (3-7)	4.7 ± 2.2	5.1 ± 2.2	5.0 ± 2.2 C	−0.4 (1.8 to 1.0)
Baseline vs 30 mo after prophy	4 (3.5-6.25)	4.5 (3-7)	5.0 ± 2.2	5.2 ± 2.3	5.2 ± 2.3 B	−0.2 (−1.6 to 1.2)

Abbreviation: CI, confidence interval.
[a] *Groups identified with the same letter are statistically similar.*

vs nonsmokers) can also be seen with the 95% confidence interval (CI) of the effect size (mean difference) (Tables 2 and 3) that does include zero.

A significant average color change (ΔSGU) of approximately five shade guide units in the Vita classical guide (Table 2) and four shade guide units in the Vita Bleachedguide 3D-MASTER (Table 3) was observed one month after bleaching for both groups. At 30 months, a slight but significant color rebound could be detected regardless of whether color was measured before or after dental prophylaxis (Tables 2 and 3; $p<0.001$).

Satisfaction Assessment

Participants who attended the shade evaluation at 30 months after bleaching treatment were questioned about their satisfaction level (Table 4). The majority of the smokers reported that they still observed moderate bleaching, while participants in the nonsmokers group reported there was still significant bleaching. Most participants from both groups felt happy with the bleaching result and would repeat the procedure. After 30 months of the bleaching procedure, 70% of participants reported that their teeth darkened slightly.

DISCUSSION

As a part of daily life many people smoke; eat dark-colored food; and drink coffee, tea, red wine, and other colored drinks. Some investigators have reported that colored beverages and foods can induce tooth discoloration.[21,22] This fact, along with the slight demineralization that acidic bleaching gels produce on dental surfaces,[23] led dentists and product manufacturers to request their patients to avoid smoking, drinking, and eating colored beverages and foods during the active bleaching treatment phase.

However, these dentists' recommendations seems to be endorsed by bleaching myths, in relation to efficacy and safety, rather than evidence-based findings.[24] It was reported in a recent publication[25] that at-home bleaching did not induce DNA damage to gingival tissue during the bleaching period in smokers and nonsmokers. The genotoxicity potential of smoking, as reflected by the mean number of micronuclei in exfoliated cells, was not increased by the at-home bleaching procedure.[25]

With regard to the effectiveness, the results of the present study highlight that effective whitening is achievable in smokers even without requesting them to stop smoking during the active phase of the bleaching treatment. In a similar trend, an earlier study[4] reported that exposure to coffee four times a day also did not jeopardize the bleaching efficacy when compared to results in patients that followed a "white diet." "White diet" was a term introduced by Professor Matis in a recent publication[24] that refers to a diet that is free of colored drinks and foods. This was also confirmed in a recent published study.[24]

Table 3: *Color Change in Shade Guide Units (ΔSGU) Using the Bleachedguide 3D-MASTER Shade Guide at the Different Assessment Points Along with the Effect Size (Mean Difference) and the 95% Confidence Interval (CI)*

Assessment Time	Medians (Interquartile Range)		Mean ± Standard Deviation			
	Smokers	Nonsmokers	Smokers	Nonsmokers	Main Factor Time[a]	Mean Difference (95% CI)
Baseline vs 1 mo	4 (3.00-4.25)	4.5 (4-5)	4.1 ± 1.1	4.7 ± 1.4	4.4 ± 1.3 A	−0.6 (−1.4 to 0.2)
Baseline vs 30 mo before prophy	3 (2.00-3.25)	3.5 (3-4)	2.6 ± 1.1	3.3 ± 1.2	3.0 ± 1.2 B	−0.7 (−1.4 to 0.0)
Baseline vs 30 mo after prophy	3 (2.75-4.00)	4 (3-4)	3.0 ± 1.1	3.4 ± 1.2	3.2 ± 1.2 B	−0.4 (−1.1 to 0.3)

[a] *Groups identified with the same letter are statistically similar.*

Table 4: *Degree of Participants' Satisfaction 30 Months After Bleaching*

Question	Smokers	Nonsmokers	Statistical Significance*
1. After the bleaching treatment, you observed that			
a) there was no color change in teeth	0	0	n.s.
b) there was mild whitening, not noticed by others	1	0	n.s.
c) there was mild bleaching, noticed by others	2	5	n.s.
d) there was moderate whitening	10	5	n.s.
e) there was a significant whitening	8	12	n.s.
2. What is your level of satisfaction with the performed bleaching treatment?			
a) Very happy	9	6	n.s.
b) Happy	9	8	n.s.
c) Satisfied	3	8	n.s.
d) Indifferent	0	0	n.s.
e) Dissatisfied	0	0	n.s.
3. Would you repeat the bleaching treatment in case your teeth get darker?			
a) Yes, because I liked the result	16	16	n.s.
b) Yes, because I would like my teeth to become lighter than they are	5	6	n.s.
c) No, I'm satisfied	0	0	n.s.
d) No, because I experienced pain	0	0	n.s.
4. Do your teeth look darker now (30 months after bleaching)?			
a) No	0	1	n.s.
b) A little	14	15	n.s.
c) A reasonable amount	6	5	n.s.
d) Too much	0	1	n.s.
e) I don't know	1	0	n.s.

Abbreviation: n.s., not significant.
** Chi-square test, $\alpha = 0.05$.*

Altogether, these three studies provide contrary evidence to this widespread myth that patients should keep a white diet and/or quit smoking while having their teeth bleached. The self-assessment of the participants is also in agreement with the results of the shade guide units; most of the participants from both groups reported themselves to be happy with the whitening degree obtained and would repeat dental bleaching if necessary.

On the other hand, we cannot deny the fact that over time smokers are theoretically more prone to having stain deposition on their dental surfaces than are nonsmokers. Consequently, concerns about durability and longevity of the bleaching protocol in such groups of patients are critical. Tobacco contains a lot of nicotine,[26] and though it is an inherently colorless substance, it turns yellow when it comes in contact with oxygen. Nicotine penetrates the nooks and crannies[27] of teeth, leading to tooth stains. Apart from nicotine, tobacco smoke contains carbon monoxide, thiocyanate, herbicide, fungicide and pesticide residues, tars, sugar, and cocoa,[28] which cause dental discoloration due to their dark hue and ability to adhere to dental surfaces.[9]

However, our results demonstrated that color rebound was equal in both groups of participants. We expected that the teeth of smokers would be darker than those of nonsmokers, a hypothesis that was not proven by the findings of the present investigation. Perhaps 30 months is still too short term a follow-up for nicotine and tar to penetrate the tooth and change its color intrinsically. Another factor to be considered is that dental prophylaxis was performed in the 12-month assessment[18] so the extrinsic pigments observed in the 30-month recall were the result of an 18-month accumulation. Furthermore, some participants stopped or decreased smoking during this 30-month follow-up. Perhaps the evaluation of such a sample through longer-term follow-ups might allow us to detect if indeed differences in the longevity of the bleaching outcomes in smokers compared to nonsmokers may become evident in longer follow-ups.

Professional mechanical cleaning, such as dental prophylaxis and enamel polishing, are effective means by which to produce partial or complete stain removal.[29] Indeed, this was observed in the present investigation and in the one-year follow-up of this study. By removing the extrinsic stains presented on the dental surface of the smoker group (produced by diet + cigarette smoke) and in the nonsmokers group (produced by diet), teeth became significantly whiter. This was likely one of the reasons that reduced the patient's overall perception of whiter teeth after 30 months of follow-up.

On the other hand, the present study demonstrated that stain deposition is not the single factor responsible for the color rebound observed in the present investigation. Contrary to what was observed in the one-year follow-up,[11] the ΔSGU baseline vs 30-month after prophylaxis result was not statistically similar to the whitening degree obtained one month postbleaching, meaning that other factors, apart from superficial dental staining, might be associated with such slight but significant color rebound.

As teeth get older, there is continuous enamel wear and deposition of secondary dentin by the pulp.[30] As the dentin thickness increases and enamel thickness decreases,[31] teeth become increasingly yellow regardless of the individual's dietary conditions or smoking habits. Interestingly, most of the patients reported that they felt their teeth were darker at the 30-month recall than immediately after bleaching. In the 30-month recall most of the participants in both groups reported that their teeth had darkened slightly.

The participant's perception is important for clinicians, since a positive correlation was found between participants' self-assessment of their tooth shade and that of the clinician.[32] One of the most important factors in determining satisfaction with self-appearance is the tooth color.[33] In the present study most participants, both smokers and nonsmokers, felt happy with the bleaching treatment result, which was previously shown in a study designed to assess patient satisfaction with the whitening treatment performed.[34] Some participants reported that the bleaching treatment provided a slight color change, which may or may not have been noticed by other people. It has been shown[35] that patient expectations regarding the outcome of the bleaching treatment are higher than those of dentists, which could lead to a divergence between them, meaning that clinical trials should include more patient-centered outcomes rather than evaluator-centered outcomes, since the patient's satisfaction or treatment success perception is more important than the care provider's perception. It is worth pointing out, however, that the data provided by the questionnaire included in this trial are simply exploratory, as we have not used a validated instrument.

The literature findings report controversial findings regarding the longevity of at-home bleaching. Some authors[12,13,16] reported stable color in periods ranging from one to two years. Other authors reported color rebound after one year[36,37] and two years[13,38] and also after longer follow-up recalls,[39,40] as demonstrated in this study. Indeed, the longevity of such bleaching procedures is yet to be determined. Despite this, 70% of patients reported that they observed a slight color change in their teeth.

Additionally, the majority of clinical trials that evaluated the longevity of at-home bleaching did not report the patients' dietary habits during and after dental bleaching treatment. Only a few studies[12,37,38] have attempted to associate the effect of dietary habits with the longevity of at-home bleaching, although they did not reach conclusive findings, which emphasizes the need for future studies.

CONCLUSION

After a follow-up of 30 months, we detected a significant color rebound in smokers and nonsmokers with use of 10% CP, which cannot be attributed to extrinsic stains only, as even after dental prophylaxis, teeth appeared slightly darker than the immediate whitening result.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the State University of Ponta Grossa. The approval code for this study is 16211/2014.

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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A Near Visual Acuity Test for Dentists

P Perrin • M Eichenberger • KW Neuhaus • A Lussi

Clinical Relevance

Use of a US $5 bill offers a simple and easily available triage test to assess individuals' near vision relative to that of other dentists and to monitor the progression of presbyopia with increasing age. The interindividual variability of near vision was found to be large.

SUMMARY

Unimpaired near vision is crucial in dentistry, but appropriate visual tests at dental working distance are not publicly available. The aim of this study was to validate a novel visual triage test for dentists that is easy to use and freely available. The near visual acuity at 300 mm of 106 dental professionals (aged 21–65 years) was assessed with 1) a validated near visual test for scientific purposes miniaturized on a microfilm; 2) an experimental test using a US $5 bill, in which the first five words of each line in the Lincoln Memorial frieze had to be read under a dental operating light. The Spearman rank correlation coefficient of 0.784 revealed a strong correlation between the two tests ($p<0.0001$). The ability to read six or more words in the memorial frieze meant there was a 94% chance of having a validated near visual acuity greater than or equal to the median score of the dentists tested. If none of the words could be read, the chance of having a near visual acuity below the median of the peer group was 89%. The influence of the dentists' age and experience on their visual performance reported in former studies was corroborated with this new test. The US $5 bill offers a simple and easily available near visual test to rank individuals' near vision relative to that of other dentists and to recognize the progression of presbyopia with increasing age.

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INTRODUCTION

The visual control of small structures is a vital part of dental diagnostics and therapy; therefore, regular visual tests at working distance should be mandatory for dental professionals. Traditional near visual tests, like the reading type test of the British Faculty of Ophthalmologists,[1,2] are printed with conventional typography and underlie the dimensional limitations of this technique. They are not sensitive enough to discriminate between good and bad eyesight for dental purposes. Consequently, studies using these tests show unproblematic and good near vision for almost all test subjects.[3-5] In contrast, recent studies with visual tests based on microfilms have shown that

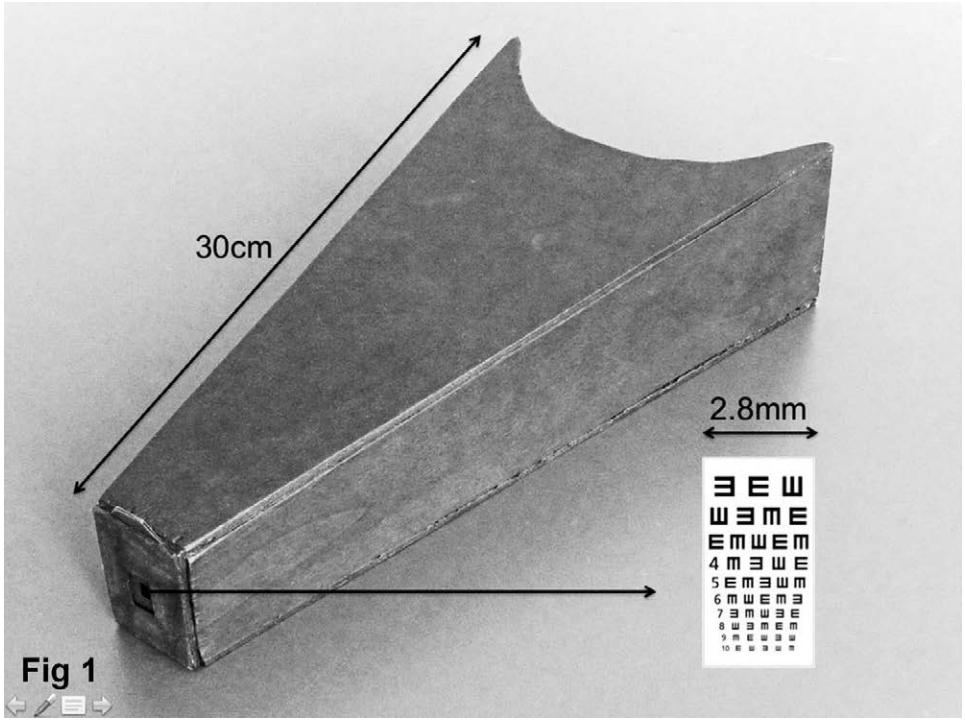

Figure 1. *Near visual test with E-optotypes on a microfilm for the use on a negatoscope. The distance between the bars of the E-optotypes is the relevant dimension and ranged between 0.01 mm and 0.1 mm.*

the near visual acuity of dentists varies widely between individuals if evaluated with tests using suitable dimensions.[6-9] Significant differences in the dimensions of the structures recognized were found in all test groups and age ranges included in these studies. This was also true for hand surgeons when tested using a similar study design.[10] Of particular interest is the presbyopic decrease of visual performance with increasing age. This inevitable and progressive limitation of the near vision starts at an age of around 40 years.[7,11,12] In most cases it remains undetected for years until daily activities like the reading of small letters are affected.[8,9] Magnification devices such as loupes can reliably compensate for presbyopic deficiencies: under simulated clinical conditions, dentists ≥40 years using Galilean loupes (2.5×) had a similar visual performance to younger test subjects with unaided vision. Moreover, it was found that Keplerian loupes and operating microscopes enhance the visual performance of dentists independent of their age.[6-9]

Dentists' self-assessment of their visual performance seems unreliable. Responses to the questionnaires used in the aforementioned studies showed a poor correlation with the objective findings of the visual tests. This was particularly true for dentists ≥40 years.[6-9] Thus, very often, dentists will not be aware of their visual deficiencies, which could easily be compensated for with optical aids.

These findings justify the need for an adequate visual test for dentists. The aforementioned miniaturized visual tests cannot be produced commercially and are limited to use in research. As pointed out earlier, traditional visual tests are not sensitive enough for dental purposes. Therefore, the present study aimed to find and validate an easily accessible and reliable near visual test for dentists.

METHODS AND MATERIALS

One hundred and six dentists and dental students (n=60, <40 years; n=46, ≥40 years; range, 21-65 years) took part in this study. All participants in a continuing education course formed the group of qualified dentists (n=67), and all students in the third year of their course toward graduation (n=39) formed the students' group. No exclusion criteria were applied. The study was approved in accordance with the ethical guidelines of the Review Board of the University of Bern. Visual acuity was tested at a dental working distance of 300 mm with two different visual tests. Correcting eyeglasses or contact lenses had to be worn if necessary. If the focal distance was different from 300 mm, the test was performed at that focal distance.

For the validated near visual test,[7] eye charts with miniaturized E-optotypes (Figure 1) on transparent microfilm were fixed behind fenestrated black cardboard and mounted on a negatoscope (Imatec,

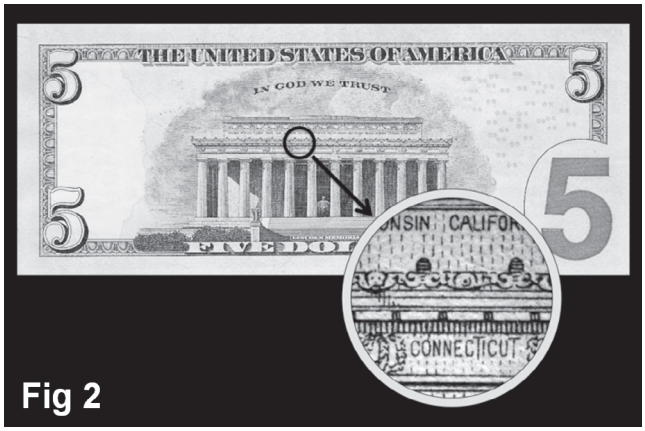

Figure 2. *The US $5 bill shows the Lincoln Memorial in Washington, DC, with the names of all US states aligned in two rows in the frieze. The first five words of both lines had to be read at a distance of 300 mm.*

Basel, Switzerland, 2×8W). The dimensions of the Es ranged between 0.05 mm and 0.5 mm. The distance between the three bars of the E-optotype was the dimension to be detected by the test subject.[13] This distance ranged between 0.01 mm and 0.10 mm. The smallest line that could be read at a distance of 300 mm or at focal distance was recorded. The metric dimension of the bar spacing (eg 0.04 mm) was converted into the reciprocal value (eg 25 mm^{-1}) to obtain a positive association between the value and the detail recognition.[6]

For the experimental near visual test, an unused US $5 bill was fixed on a black A4 sheet and illuminated as rectangularly as possible with a dental operating light (LoLé2, DegréK, Paris, France) from a distance of 700 mm. In the frieze of the Lincoln Memorial, the names of the US states are aligned in two rows (Figure 2). The letters have a width of 0.25 mm. The test subjects had to read the first five words of each row at a distance of 300 mm or at focal distance. The distance of 300 mm was kept constant by means of a 300 mm bar between the test subject's forehead and the bill. The number of words that could be read was recorded.

In the first part of the study, the relationship between the scores of the two tests was evaluated by Spearman rank correlation. In addition, the results were split into four groups for the $5 test (0, 1-4, 6-9, and 10 words read) and into two groups for the E-optotype test ($</\geq$ median mm^{-1}) to describe the suitability as an easy triage test.

For the second part of the analysis, the results were split into three groups: students (n=39), young dentists <40 years (n=21), and older dentists ≥40 years (n=46). By means of a Wilcoxon rank sum test, the influence of age and professional experience on the result was evaluated: students (n=39) vs dentists (n=67), students (n=39) vs young dentists (n=21), and students and young dentists (n=60) vs older dentists (n=46).

In the third part of the study, a self-assessment in the form of a questionnaire answered by 94 test subjects was correlated with the results of the $5 test. Respondents answered the question "How do you estimate your visual performance as a dentist wearing your correction glasses if necessary?" on a visual analog scale with scores ranging from 1 to 10 (1 = very poor, 10 = very good). Of the 67 dentists in the continuing education course, 12 did not answer the questionnaire. The test subjects were split into two groups <40 years (n=49) and ≥40 years (n=45). The relationship between test and self-assessment was evaluated by Spearman rank correlation for both groups.

Statistical analysis was performed with the free statistical software R: version 2.14.1 for the first part of the study and version 3.2.1 for the second and third parts (www.r-project.org). As the analysis was exploratory, no corrections for multiple testing were applied. Correlation coefficients were assessed according to the rules of Hinkle and others.[14] The level of significance was set at α=0.05.

RESULTS

The Spearman rank correlation coefficient between the two tests was 0.784, which indicated a strong correlation.

Data grouped according to the number of words the subjects could read is presented in Figure 3. Of the 106 test subjects, 26 (24.5%) could read all of the requested 10 words of the Lincoln Memorial frieze, whereas 27 of them (25.5%) could read no words at all. Test subjects who could read the majority of the words in the memorial frieze (≥6 words) had a 94% chance of a validated near visual acuity greater than or equal to the median score of the peer group of dentists tested. If none of the words could be read, the chance was 89% of having a near visual acuity below the median of the peer group.

Results of the Wilcoxon rank sum test performed after splitting the test subjects into groups according to age and experience is shown in Table 1. Compared with all the dentists who participated in the study, the students showed significantly better results, while the comparison of students with young dentists <40 years showed significantly better

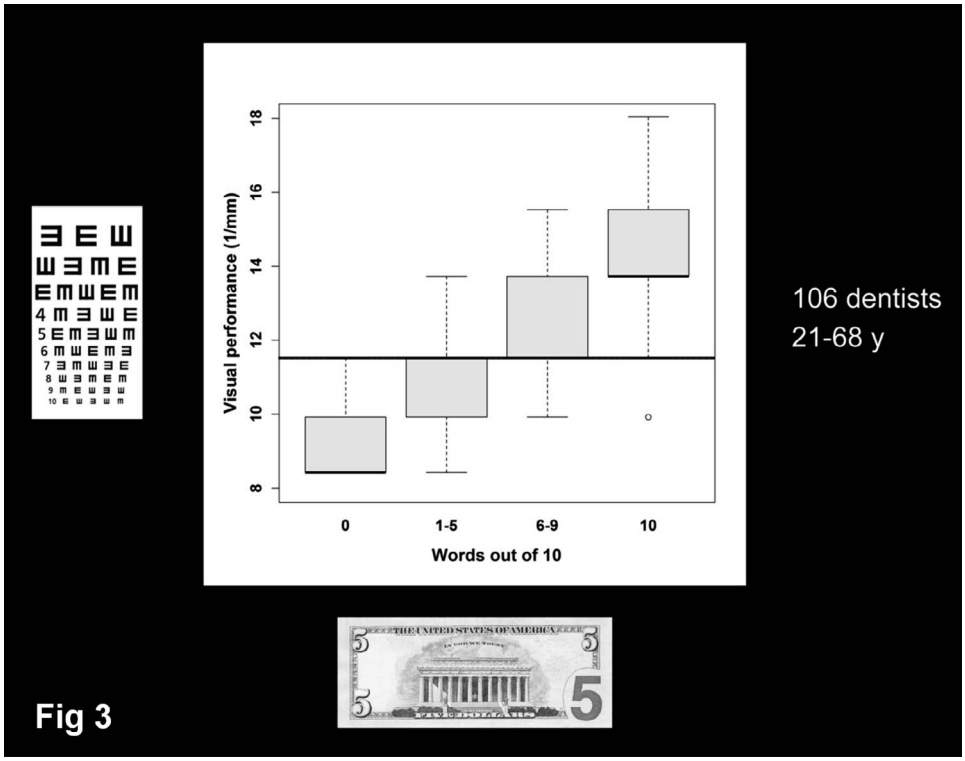

Figure 3. *The results of the $5 test in relation to the median of the E-optotype test (11.7 mm^{-1}). The $5 test allows an easy triage of dentists' visual performance in relation to that of the peer group.*

results for the dentists. The difference between the performance of test subjects <40 years and those aged ≥40 years was highly significant.

Overall, a low correlation was found between the self-assessment and the objective test values (0.41). A negligible correlation was noted for the group of younger participants (0.29) and the group of older participants (0.09). The somewhat higher correlation in the overall data is mainly explained by the discrimination caused by the age groups.

DISCUSSION

Studies with traditional near visual tests suggest that they are not sensitive enough to discriminate between dentists with good and bad eyesight.[3-5] The symbols used in this kind of test are too large due to the dimensional limitations of conventional printing techniques. Thus, the aim of the present study was to find and validate a suitable, simple, and easily available near visual test for dental purposes. The prospective test should reveal visual deficiencies, such as the undetected beginning of presbyopia at an age ≥40 years, which could be easily compensated for by means of medical loupes. Another field of application is in scientific studies, where the visual performance of the examiners is part of the methodology. A suitable visual test could help to exclude test subjects with visual deficiencies before starting the study.

Most banknotes incorporate miniaturized structures or letters printed with a sophisticated technique to prevent forgeries. These letters might serve as potential and easily available near visual tests. Pre-studies with different bills from the United States, the European Union, and Switzerland showed that the US $5 bill, in particular, has the potential to provide a suitable near visual test at a dental working distance. The letters naming all US states in the frieze of the

Table 1: *Comparison of Near Visual Acuity of Different Subgroups*

Comparison	*p*-Value	Assessment
Students vs all dentists	p=0.02	Students performed significantly better
Students vs dentists <40 years	p=0.02	Dentists performed significantly better
Subjects <40 years vs ≥40 years	p< 0.0001	Group of young participants performed significantly better

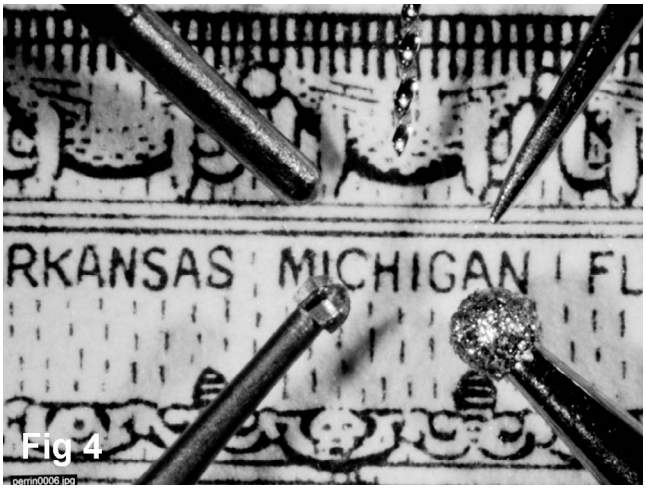

Figure 4. *Common small dental instruments in comparison to the letter size in the frieze (perioprobe, K-file No. 15, explorer No. 12, diamond bur No. 009, and rose bur No. 006).*

Lincoln Memorial have a spacing of 0.15 mm and a width of 0.25 mm. This is in perfect accordance with the size of the E-optotypes used in previous studies[6-9] and with the dimensions of common small dental instruments (Figure 4).

The experimental near visual test includes letters of only one size, unlike traditional visual tests that use progressively smaller lines. This uniform dimension is compensated for by the differing legibility of the letters (eg TEXAS vs DELAWARE). The potential bias that could be introduced by guessing the names of the US states can be counterbalanced by the strong correlation to the standardized E-optotype test.

The hypothesis that the novel test covers the range of a dentist's near visual acuity and offers a valuable opportunity to evaluate dentists' near vision is corroborated by the results of the present study.

Splitting the test subjects into groups—students, dentists $<$40 years, and dentists $\geq$40 years—allowed for the evaluation of differences due to age and due to a lack of professional experience. The results corroborate the hypothesis that the beginning of presbyopia at an age of about 40 years is the main factor limiting near visual acuity. The question why young dentists had a significantly higher near visual acuity than third-year students remains unanswered. A visual training effect is possible, as is the hypothesis that practicing dentists are more conscious of the need for appropriate optical correction.

Both of the tests presented allowed the comparison of one individual's near vision with that of a peer group of dentists. A weak result should prompt further optical examination by an optometrist and the choice of an adequate magnification device to compensate for the visual deficiency.[6-9,12]

It should be noted, however, that there is no established or evidence-based threshold for the visual acuity of dentists. A number of recent studies have shown the advantages of magnification devices for clinical procedures,[15-20] but most of them are case reports or expert opinions with little scientific evidence to support them.[21] Conclusions about the association between near visual acuity and the quality of patient care was beyond the scope of the present study. This question remains open and should be the subject of further research.

CONCLUSION

A US $5 bill can be used as a simple and easily accessible test to qualify the individual near vision of dentists.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Kantonale Ethikkommission Bern. The approval code for this study is Req-2016-00113.

Conflict of Interest

The authors declare that they have no conflict of interest.

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Effect of Refurbishing Amalgam and Resin Composite Restorations After 12 Years: Controlled Clinical Trial

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Clinical Relevance

Some amalgam restorations or resin composite with surface defects, although they can be refurbished, could be monitored over time in patients with low and medium risk of caries.

SUMMARY

Objectives: The aim of this study was to clinically evaluate posterior amalgam and resin composite restorations refurbished over a period of 12 years by investigating the influence of refurbishing on the survival of restorations and comparing their behaviors with respect to controls.

Methods and Materials: Thirty-four patients were enrolled, ages 18 to 80 years, with 174 restorations, 48 restorations of resin composite (RC), and 126 restorations of amalgam (AM). Restorations with localized defects in anatomy, roughness, luster, or marginal staining that were clinically judged as suitable for refurbishing according to US Public Health Service (USPHS) Ryge criteria were assigned to group A—refurbishing (n=85; 67 AM, 18 RC)—or group B—control (n=89; 59 AM, 30 RC); the quality of the restorations was evaluated blindly according to the modified USPHS criteria. Two observers conducted evaluations at the initial state (k=0.74) and after one to five, 10, and 12 years (k=0.88). Wilcoxon, Friedman, and Mantel-Cox tests were performed to compare the groups, respectively.

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Results: After 12 years, both groups experienced a similar decline, except for an evidently better performance in marginal adaptation in RC control (p=0.043) and in anatomy in AM refurbished (p=0.032).

Conclusions: After 12 years, no difference was found in the clinical condition and longevity of the refurbished restorations compared to the control group.

INTRODUCTION

After a restoration is placed, it begins to interact with the oral environment. Erosive forces result from the mechanical mastication process and biochemical interactions of metabolites from the biofilm attached to the surface, which may induce changes in surface roughness within only 30 days of exposure.[1] Also, the roughness of the surface of restorations apparently correlates with its hardness. Munchow and others[2] reported in an *in vitro* study that composite resin surfaces with greater roughness showed lower hardness.

Refurbishing restorations using carbide burs and polishing systems produces immediate clinical improvement in parameters such as surface roughness, luster, and anatomy according to the US Public Health Service (USPHS) Ryge criteria.[3] Moreover, this simple procedure can reverse the decision to replace restorations.[4,5] It is important to assess whether it is necessary to improve the clinical condition of defective restorations that do not need repair or replacement and observe whether this procedure is able to reduce the mechanical failures or incidence of long-term secondary caries. Presently, some reports monitoring refurbished restorations have shown no difference in survival of restorations under long-term follow-up.[6,7]

Patients usually have specific dentists who evaluate and treat them, but today there are many massive health services that have modified the form in which dental care is provided. Each professional evaluates patients and makes a diagnosis and treatment plans with generally different results. The refurbishing procedure could provide dentists a less invasive option when there are doubts about the decision to replace a restoration.

This study evaluated the clinical performance of defective resin composite and amalgam restorations that were refurbished compared to control restorations (the negative control group) over a 12-year follow-up period, thus investigating functional and esthetic failures.

The null hypothesis of this study was that there would be no difference in the clinical condition and longevity of restorations that were refurbished compared to the control group.

METHODS AND MATERIALS

Thirty four patients ages 18 to 80 years (mean 26.5 years), comprised of females (58%) and males (42%), who had a total of 48 composite resin and 126 amalgam restorations were recruited at the Operative Dentistry Clinic at the Dental School of the University of Chile. The restorations presented anatomy, roughness, luster, or marginal staining in composite resin and amalgam restorations that deviated from the ideal and were rated as Bravo or Charlie according to the modified USPHS Ryge criteria. The protocol was approved by the Institutional Research Ethics Committee of the Dental School at the University of Chile (Project PRI-ODO-0207/NCT02043873). All patients signed informed consent forms, completed registration forms, and agreed to participate in the blind study (subjects involved in the study did not know which treatment they received). Patients whose restorations failed were removed from the study and treated but were still included in the final analytical statistics according to the "intention to treat" CONSORT protocol[8] (Figure 1); the structure of this clinical trial and the presentation of methods, results, and additional information were in agreement with the recommendations of STROBE.[9]

The selection criteria are summarized next.

Inclusion Criteria

- Patients with anatomy, roughness, luster, or marginal staining defects in resin and amalgam restorations who were clinically judged suitable for refurbishing according to the USPHS criteria (Table 1).
- Patients with more than 20 teeth
- Restorations in functional occlusion with an opposing natural tooth
- An asymptomatic restored tooth
- At least one proximal contact area with a neighboring tooth
- Patients older than 18 years
- Patients who agreed to and signed the consent form before participating in the study
- Region outside the restoration's failure in good condition

Exclusion Criteria

- Patients with contraindications for regular dental treatment based on their medical history

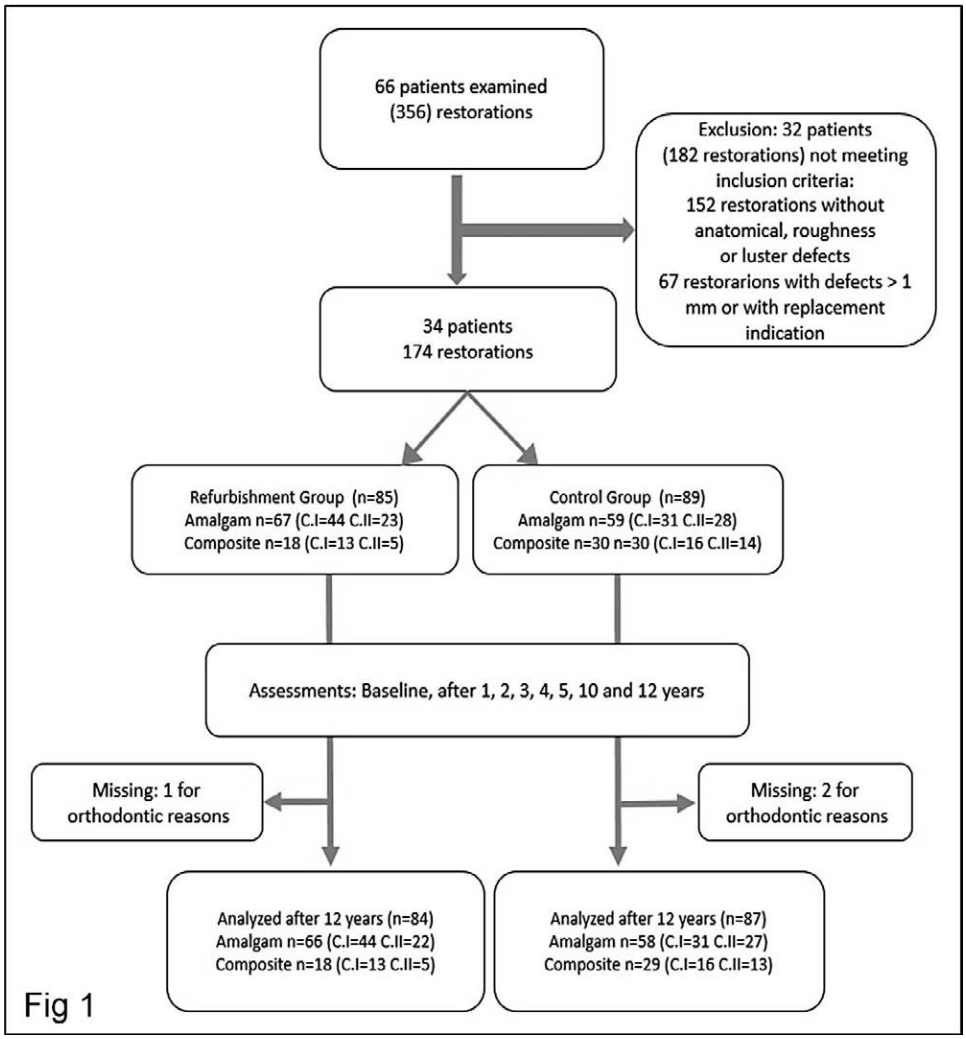

Figure 1. *Flowchart of the clinical design.*

- Patients with special esthetic requirements that would not be solved by refurbishing treatments
- Patients with xerostomia or taking medication that significantly decreased salivary flow
- Patients with a high caries risk
- Patients with psychiatric or physical diseases that interfered with oral hygiene
- Composite and amalgam restorations with localized secondary caries or marginal defects >1 mm and located on the proximal surfaces
- Clinical judgment that refurbishment was not indicated in resin or amalgam restorations

Sample Size Determination and Randomization

Sample size was determined a priori using G*Power version 2.22[10] with an error probability of $\alpha = 0.05$, an effect size of 0.3, and power ($b - 1$ error probability) of 0.80. Only one faculty member provided the refurbishing treatment (PV).

Caries Risk Assessment

Cariogram (a graphical computer program) was used to assess an individual patient's caries risk. The program weighted the interaction between the following 10 caries-related factors: caries experience, related general disease, diet contents, diet frequency, plaque amount by the Silness-Loe Index, semiquantitative detection of *Streptococcus mutans* and *Lactobacilli* in saliva by a caries-risk test bacteria (Ivoclar Vivadent AG, Schaan, Lichtenstein), fluoride program, amount of saliva secretion by a caries-risk test buffer (Ivoclar Vivadent), saliva buffer capacity, and clinical judgment. Patients were classified as high, intermediate and low caries risk. In addition, the results also indicated where targeted

Table 1: *US Public Health Service/Ryge-Modified Clinical Criteria*

Clinical Characteristics	Alpha	Bravo	Charlie
Marginal adaptation	Explorer does not catch when drawn across the restoration/tooth interface	Explorer falls into crevice or has one-way catch when drawn across the restoration/tooth interface	Dentin or base is exposed
Surface roughness	The surface of restoration has no surface defects	The surface of restoration has minimal surface defects	The surface of restoration has severe surface defects
Secondary caries	There is no clinical diagnosis of caries	Not applicable	Clinical diagnosis of caries
Marginal stain	There is no discoloration between the restorations and tooth	There is discoloration on less than half of the circumferential margin	There is discoloration on more than half of the circumferential margin
Teeth sensitivity	No sensitivity when an air syringe is activated for 2 s at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze	Sensitivity is present when an air syringe is activated for 2 s at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze	Sensitivity is present when an air syringe is activated for 2 s at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze; sensitivity does not cease when the stimulus is removed
Anatomic form	The general contour of the restorations follows the contour of the tooth	The general contour of the restoration does not follow the contour of the tooth	The restoration has an overhang
Luster	The restoration surface is shiny and has an enamel-like; translucent surface	The restoration surface is dull and somewhat opaque	The restoration surface is distinctly dull and opaque and is esthetically displeasing

actions to improve the situation would have the best effect.

Restoration Assessment

The quality of the restorations was evaluated using the modified USPHS Ryge criteria (Table 1). Two examiners were calibrated (JM and EF; Cohen's Kappa interexaminer coefficient of 0.74 at baseline and 0.88 at 12 years). The examiners assessed the restorations independently using visual (mouth mirror number 5, Hu Friedy Manufacturing Co Inc, Chicago, IL, USA), tactile (N8 23 explorer, Hu Friedy) and radiographic (Sirona Heliodent Vario, Charlotte, NC, USA; Bite Wing, DF57, Kodak Dental System Healthcare, Rochester, NY, USA) examinations. All restorations were examined at baseline and each year up to 12 years.

The five parameters used in the examination were marginal adaptation, roughness, secondary caries, marginal stain, and tooth sensitivity (Table 1). If any disparity existed between the two examiners and an agreement could not be reached, a third clinician (GM) was invited to assist with the decision process. If the three clinicians did not reach an agreement, the lowest score was recorded.

Treatment Groups

Refurbishment Group—The occlusal, lingual, or facial surfaces of defective resin-based composite (RBC) restorations were refinished with a series of medium aluminum oxide disks (Sof-Lex, 3M ESPE, MN, USA) or carbide burs (12 and 30 blades) and then polished with a series of fine Sof-Lex disks and diamond-impregnated composite polishers (ComposiPro, Diacomp, NH, USA). Defective areas of the amalgam restoration were smoothed using carbide burs (numbers 12 and 30, Brasseler USA, Savannah, GA, USA). On the occlusal and buccal/lingual surfaces, silicone-impregnated points (Brownie/Greenie/Supergreenie, Shofu Dental Corporation, Menlo Park, CA, USA) were used for polishing.

Control Group—The defective restorations did not receive any treatment.

Patients were recalled each year and five, 10, and 12 years after for clinical evaluation by the same examiners, using the same baseline criteria.

Failed restorations were removed and treated according to their diagnosed needs. Digital photographs and bitewing radiographs were taken for all the restorations before and after treatment and every year prior to the examination.

Statistical Analysis

Mann-Whitney and Wilcoxon tests were performed for comparisons within the groups with a significance level of 0.05. A Friedman test was utilized for multiple comparisons between different years of the

Table 2: *Frequency of Alpha, Bravo, and Charlie Scores for Amalgam and Resin Composite Restorations 12 Years From Intervention*

			MA %	A %	R %	MS %	S %	SC %	L %
Amalgam restorations	Refurbishment	Alpha	6.00	20.90	20.90	28.40	94.00	91.00	13.40
		Bravo	88.10	77.60	76.10	61.20	4.50	9.00	80.60
		Charlie	6.00	1.50	3.00	10.40	1.50	0.00	6.00
	Control	Alpha	15.30	16.90	25.40	37.30	98.30	94.90	11.90
		Bravo	83.10	83.10	74.60	57.60	1.70	5.10	81.40
		Charlie	1.70	0.00	0.00	5.10	0.00	0.00	6.80
Resin composite restorations	Refurbishment	Alpha	27.80	50.00	38.90	50.00	88.90	94.40	27.80
		Bravo	66.70	50.00	61.10	50.00	11.10	5.60	61.10
		Charlie	5.60	0.00	0.00	0.00	0.00	0.00	11.10
	Control	Alpha	16.70	20.00	50.00	50.00	100	100.00	20.00
		Bravo	80.00	80.00	50.00	43.30	0.00	0.00	80.00
		Charlie	3.30	0.00	0.00	6.70	0.00	0.00	0.00

Abbreviations: MA, marginal adaptation; A, anatomy; R, roughness; MS, marginal staining; S, sensitivity; SC, secondary caries; L, luster.

same group with a significance level of 0.05. The statistical analysis was performed using SPSS version 21.0 (IBM, New York, NY, USA) and GraphPad Prism version 6.00 for Windows (GraphPad Software, La Jolla, CA, USA) statistical software. The "intention to treat" CONSORT protocol was used to analyze the data on restorations evaluated in year 12 but lacking data from a previous evaluation. Patients who could not be assessed in year 12 were considered absent and were excluded from the analysis.

RESULTS

The recall of this cohort of patients at 12 years was 100%. One restoration (1.17%) was lost in the refurbishment group and two restorations (2.24%) in the control group for orthodontic reasons. The distribution according to patients' caries risk was medium caries risk 80% (n=27) and low caries risk 20% (n=7). Due to local ethics committee requirements at the time of initiation of the clinical trial, high-caries-risk patients were not included since refurbishment was classified as an experimental treatment.

The clinical condition of restorations evaluated at the 12th year is presented in Table 2.

Comparison Between Groups: Mann-Whitney Test

Amalgam Restorations—When comparing 12th year values of all Ryge parameters of refurbishment with the control group, a statistically significant difference was found in the marginal adaptation due to an increased number of alpha values in the control group ($p=0.043$).

Composite Resin Restorations—When comparing 12th year values of all Ryge parameters of refurbishment with the control group, a statistically significant difference was found in the anatomy due to an increased number of alpha values in the refurbishment group ($p=0.032$).

Change in Parameter Scores Over Time (Friedman Test)

Multiple comparisons of scores at different evaluation years showed statistically significant differences in scores ($p\leq0.001$) for all clinical characteristics in the refurbishment and control groups.

Within-Group Comparisons by Wilcoxon Test

Amalgam Restorations—When comparing the baseline with the 12th year for the refurbishment group, all parameters showed a statistically significant difference ($p<0.046$) due to higher Bravo values except for sensitivity ($p=0.059$) and anatomy ($p=0.060$).

Within the control group, all clinical parameters presented a statistically significant difference ($p=0.00$) due to higher Bravo values except for sensitivity ($p=0.317$) and secondary caries ($p=0.083$).

Resin Composite Restorations—When comparing the baseline with the 12th year for refurbishment, all clinical parameters showed a statistically significant difference ($p\leq0.001$) due to higher Bravo values except for marginal adaptation ($p=0.0593$),

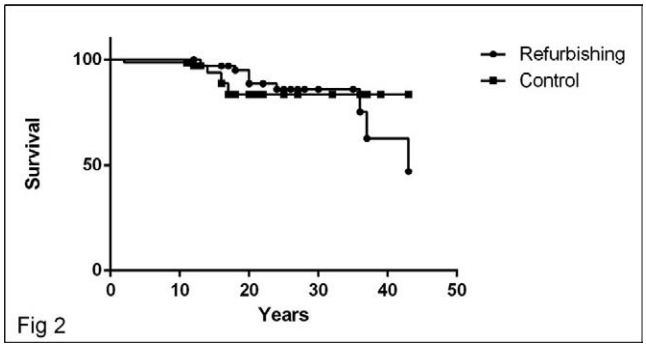

Figure 2. *Kaplan-Meier survival curves for refurbished and control groups of amalgam restorations.*

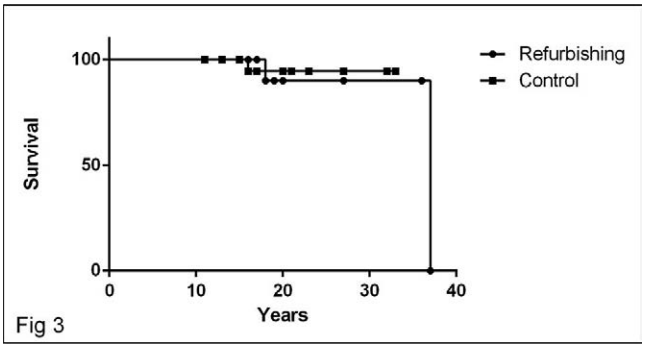

Figure 3. *Kaplan-Meier survival curves for refurbished and control groups of composite restorations.*

marginal staining (p=0.196), and secondary caries (p=0.157).

Within the control group, all clinical parameters showed a statistically significant difference ($p \leq 0.001$) due to higher Bravo values except for sensitivity and secondary caries (p=1.0).

Survival Analysis

Amalgam Restorations—In the case of survival analysis, 10 failures were found in the refurbished group and four failures in the control group. No statistically significant difference (–log rank test, p=0.8835) was found between the groups in the observed dropout dates of each restoration in the Kaplan-Meier analysis. Using the dates when the restorations were originally made, not enough data were available to calculate restoration half-life (Figure 2).

Resin Composite Restorations—In the case of the survival analysis, two failures were found in the refurbished group and one failure in the control group. No statistically significant difference (–log rank test, p=0.8832) was found between the groups in the observed dropout dates of each restoration in the Kaplan-Meier analysis. Not enough data were available to calculate restoration half-life (Figure 3).

Few failures were observed in this study; the reasons for failure and characteristics are presented in Table 4.

Table 3: *Percentage of Alpha Values of the Initial Assessment, First- and 12th-Year Follow-Up (*p*-Values of Wilcoxon Test Between Initial Assessment and 12-Year Recall)*

	Refurbishing			*p*-Value IA vs 12	Control			*p*-Value IA vs 12
Amalgam restorations	IA	1	12		IA	1	12	
MA	40	75	6	0.000	83	75	15	0.000
A	42	77	21	0.060	78	75	17	0.000
R	45	84	21	0.001	92	86	25	0.000
MS	84	90	28	0.000	93	93	37	0.000
S	100	99	94	0.059	100	100	98	0.317
SC	100	97	91	0.014	100	98	95	0.083
L	33	73	13	0.001	68	68	12	0.000
Resin composite restorations								
MA	17	67	28	0.593	73	87	17	0.000
A	78	94	50	0.018	77	73	20	0.000
R	72	94	39	0.046	90	97	50	0.000
MS	50	83	50	0.196	90	87	50	0.000
S	100	100	89	0.046	100	100	100	1.000
SC	100	100	94	0.157	100	100	100	1.000
L	78	89	28	0.000	93	77	20	0.000

Abbreviations: IA, initial assessment (baseline); MA, marginal adaptation; A, anatomy; R, roughness; MS, marginal staining; S, sensitivity; SC, secondary caries; L, luster.

Table 4: *Characteristics of Failed Restorations*

Group	Material	Class	Reason for Failure	Age of Restoration at Time of Failure (y)	Age of Treatment at Time of Failure (y)
Refurbishing	Amalgam	II	Marginal adaptation	18	2
	Amalgam	I	Marginal adaptation	20	2
	Amalgam	I	Marginal adaptation	20	2
	Amalgam	II	Marginal adaptation	13	3
	Resin composite	I	Marginal adaptation	37	4
	Amalgam	I	Secondary caries	24	3
	Amalgam	I	Secondary caries	36	5
	Amalgam	I	Secondary caries	43	10
	Amalgam	I	Secondary caries	20	1
	Amalgam	I	Secondary caries	20	1
	Amalgam	II	Secondary caries	13	3
	Resin composite	II	Secondary caries	18	10
Control	Amalgam	I	Marginal adaptation	13	—
	Resin composite	I	Marginal adaptation	16	—
	Amalgam	I	Secondary caries	12	—
	Amalgam	I	Secondary caries	12	—
	Amalgam	II	Secondary caries	23	—

DISCUSSION

This clinical, prospective study assessed patients who received refurbishment of defects of amalgam and composite restorations for a 12-year observation period compared to control restorations without treatment. In order to evaluate the effect of refurbishment on the clinical characteristics and longevity of restorations, a year-after-year comparison with the initial state of the restorations at the beginning of this study was necessary. The study null hypothesis was not rejected, as a similar clinical condition was observed in the two groups; that is, no difference in longevity of restorations was shown between the refurbishing and the control groups. Over time, continuing deterioration in all parameters was observed for both groups with a similar trend. Interestingly, the refurbished amalgam restorations after 12 years showed a worse status than the control group in marginal adaptation ($p=0.043$); a possible explanation could be that untreated amalgams suffer more corrosive processes on their surface, and with time this could enhance marginal sealing.[11,12] It could also be that refurbishment of the margins causes "microcracks/fissures" at the margin of the restoration that allow further deterioration of the restoration.

The refurbished composite resin restorations after 12 years showed a better condition in anatomy ($p=0.032$); 50% of those restorations remained in the Alpha score vs 20% in the control group. This behavior is consistent over time, according to the Friedman and Wilcoxon tests ($p<0.014$); apparently, modifications to the anatomy of resin composite restorations are more stable over time. On the other hand, Fernández and others[6] showed that refurbished amalgam restorations maintained more than 50% of their Alpha scores for less than five years.

According to the data observed every year, most of the restorations observed in both the treatment group and the control group were—and remained—in the Bravo category (ie, restorations that were not perfect). This condition apparently does not have a negative connotation because during these 12 years of monitoring, few failures occurred. Apparently, this clinical condition, associated with a low to moderate cariogenic risk, does not increase the incidence of secondary caries concordantly, though one study suggested that other sociocultural characteristics or high risk of caries would be more important.[13]

The mode of current dental care, in which the patient is not always being treated by a dentist who knows the complete history or the specific conditions of a restoration, makes it important to keep the restorations of patients in the best possible condition in order to avoid errors in treatment. As mentioned by Cardoso and others,[4] the appearance of a restoration is important in determining the treatment decision (such as replacing a restoration).

Based on the evidence presented in this study, it could be said that the anatomy parameter is a little more stable over time compared with roughness and luster, especially in resin composite restorations. However, after 12 years, at least 50% of them were scored Bravo; for amalgam restorations, this finding occurred at the five-year follow-up. Other parameters, such as roughness and luster, suffer a more rapid deterioration, so if the goal is to improve the appearance of a restoration, these parameters require more frequent monitoring and interventions undertaken when necessary.

Some limitations of this study may be that our focus was on the restorations and not on the patient. Randomization was not performed in the most appropriate way, thus generating different numbers of restorations in both groups. Another important point that was not considered in the initial design of this study was the occlusal contact location and intensity. Obviously, these elements affect the wear of restorations.[2,14] In this regard, as reported by Munchow and others,[2] a restoration with a polished surface increases its surface hardness; this could be important in the case of patients with parafunction, such as bruxism, or the presence of occlusal premature contacts. Due to the study design, we do not have access to that information. It would be interesting to explore the longevity of refurbished restorations and surface defects in patients with increased occlusal forces.

This study allowed investigation of a cohort of patients for a long time. Many of the patients were dental students; this factor allowed patients to be maintained at low and medium cariogenic risk levels due to proper hygiene habits and knowledge about oral health.

The behavior of restorations observed in the Wilcoxon analysis showed that after 12 years, the refurbishment had no effect on clinical condition. In this cohort of patients with low and medium cariogenic risk, few failures due to secondary caries were observed. Also, this group of patients had no conditions, such as bruxism, that resulted in increased masticatory forces. The size of restorations was considered within normal parameters; this situation could explain the reason for no records of fractures in restorations or teeth or irreversible symptoms of tooth sensitivity during the investigation. This finding suggests that restorations with defects in anatomy, roughness, luster, or marginal staining are not important in predicting longevity.

CONCLUSIONS

After 12 years, in general, no difference was found in the clinical condition and longevity of restorations that were refurbished compared to the untreated control group.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Institutional Research Ethics Committee of the Dental School of the University of Chile. The approval code for this study is Project 69 PRI-ODO-0207/ NCT02043873.

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 presented in this article.

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Effectiveness and Impact of the Walking Bleach Technique on Esthetic Self-perception and Psychosocial Factors: A Randomized Double-blind Clinical Trial

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Clinical Relevance

Nonvital bleaching produces positive and immediate impact on esthetic perception and psychosocial factors.

SUMMARY

Objective: This trial evaluates the impact of psychosocial and esthetic self-perceptions of patients undergoing nonvital tooth bleaching using the walking bleach technique. We also assessed the clinical effectiveness of bleaching tooth discoloration.

Methods: Fifty volunteers with nonvital tooth discoloration were enrolled. Teeth were randomized into two groups: 35% hydrogen peroxide (n=25) and 37% carbamide peroxide (n=25). Intracoronal bleaching was performed over four sessions using the walking bleach technique. Tooth color was evaluated at each session to measure total color variation. The shade guide was arranged from highest (B1) to lowest (C4) values to assess the color and calculate the color change in the number of shade guide units. Subjective and objective assessments were compared with the tooth counterpart.

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Esthetic self-perception and psychosocial factors were assessed before and after treatment.

Results: Color change was 15.48<5.17 for hydrogen peroxide and 14.02<4.85 for carbamide peroxide. There was no significant difference at any time point ($p>0.05$) except at sessions 3 and 4 ($p<0.05$). Overall, whitened teeth values were similar to those of counterpart teeth ($p>0.05$). There was a decrease in Oral Health Impact Profile and Psychosocial Impact of Dental Esthetics questionnaire scores after treatment compared with baseline ($p<0.05$).

Conclusion: The walking bleach technique was highly effective on nonvital teeth and had a positive effect on self-esthetic perception and psychological impact for the patients.

INTRODUCTION

Tooth color is one of the most important factors in achieving an esthetically pleasing smile in some cultures of the world.[1] It also substantially influences esthetic self-perception and has a psychosocial impact on people.[2] Thus, when a single tooth is darkened, the negative effect may be more pronounced because the color does not match the rest of the teeth.[3] However, there is no information on the impact of intracoronal bleaching on patient self-perception or on psychosocial impact.[4] Some authors have shown that alterations in cosmetic dentistry can cause psychosocial consequences that could have more of an impact on the person than the biological problems caused by caries lesions do.[5]

Intracoronal bleaching is a minimally invasive method of whitening discolored endodontically treated teeth. In most patients, intracoronal whitening requires more than one appointment to achieve a white smile and to repair or match the dark color of a single tooth to the rest of their teeth.[6] Therefore, it should be considered a color rehabilitation treatment.[7] There are only a small number of clinical studies on the intracoronal whitening technique, and most are difficult to compare with each other, as their conclusions are based on subjective records.[8-10] They are also considered to have low precision.

The most commonly used chemical agents include hydrogen peroxide (>35%) and carbamide peroxide (>37%).[6] Even though these agents are popular, no clinical studies have used objective methodologies that are highly reproducible or that explain the effectiveness of these agents in the whitening treatment of nonvital teeth.

The null hypothesis of this study was that there will be no difference in treatment effectiveness or in the esthetic perception and psychosocial impact of patients treated with 35% hydrogen peroxide or 37% carbamide peroxide. The main objective of this study was to evaluate the psychosocial impact and esthetic self-perceptions of patients undergoing nonvital tooth bleaching with 35% hydrogen peroxide and 37% carbamide peroxide gels using the walking bleach technique and to assess the clinical effectiveness of bleaching for discoloration.

METHODS AND MATERIALS

This randomized clinical study was approved by the Ethics Committee of the Faculty of Dentistry of the University of Chile (2016/04) and was performed according to the Consolidated Standards of Reporting Trials Statement[11] and the Declaration of Helsinki[12] (1975; revised 2000).

Study Design

This trial was a randomized double-blind (patient and evaluator) study. The study groups were randomized using Excel 2013 software (Microsoft, Seattle, WA, USA). Patients were recruited via flyers within the local Faculty of Dentistry and through social networks such as Facebook and Twitter.

Sample Size

The sample size was determined using GPower 3.1[13] software, with a 5% level of significance, 90% statistical power, and a dropout of 25%, based in a previous studies. This study corresponds to a therapeutic equivalence type in which a color variation of ΔE tones in the range of 7-10 or more, based on the original color, was considered significant. This indicated a sample size of 20. To compensate for the dropout rate reported in previous studies, we used a sample size of 25 per group.

Entry Criteria

A total of 74 patients were examined to assess whether they met the entry criteria for this study (Figure 1). Inclusion criteria were as follows: patients over the age of 18 years, with one or more nonvital discolored teeth, whose restoration did not include the vestibular surface, with the root canal in good condition, without apical lesions, with no previous tooth whitening experiences, and with a tooth shade of A2 or higher, according to the Vita Classical scale. Exclusion criteria were as follows: pregnant or breast-feeding, patients with enamel

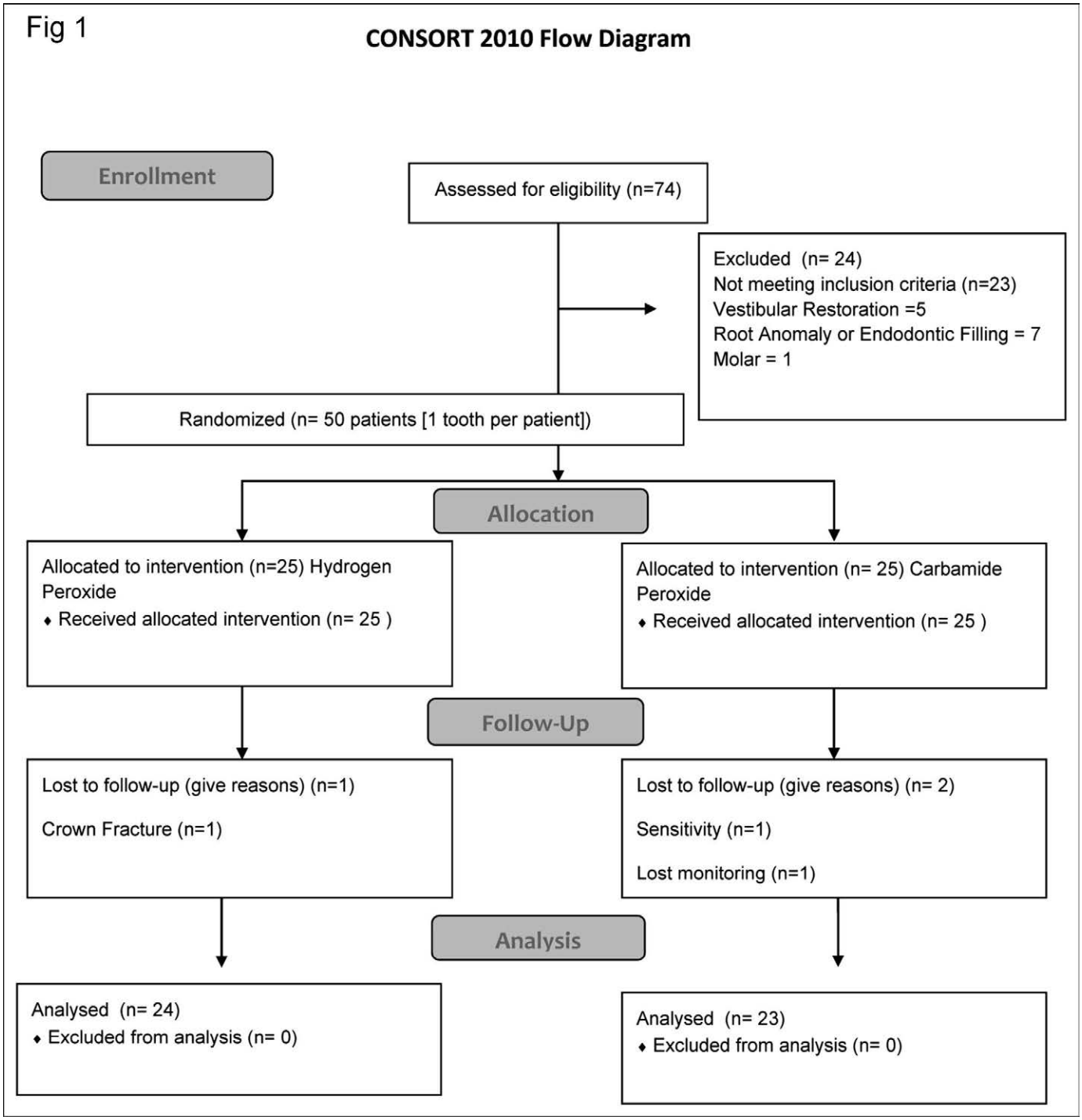

Figure 1. *CONSORT flow diagram.*

hypoplasia, teeth stained by tetracycline or fluorosis, patients receiving orthodontic treatment with fixed devices, patients with cancer, and patients with periodontal pathologies. Volunteers with clinically or radiographically identified caries, periapical lesions, external or internal tooth resorption, and/or periodontal disease were excluded, and these patients were referred to specialty clinics for treatment.

Patients agreed to use the bleaching agents and were randomized into two groups, each with 25 patients, as follows: In group 1, teeth were bleached with 35% hydrogen peroxide (Opalescence Endo,

Ultradent, South Jordan,Utah, USA); and in group 2, teeth were bleached with 37% carbamide peroxide (Whiteness Superendo, FGM, Joinville, Brazil). The bleaching agents were applied according to the manufacturer's instructions over four sessions, using an ambulatory (walking bleach) technique. There was one week between each session.

Preparation Session

The root canal was prepared with absolute isolation and an endodontic seal clearance of 3 mm from the enamel-cement limit. A 2-mm mechanical seal was placed using a glass ionomer reinforced with composite resin (Riva light cure, SDI, Bayswater. Victoria, Australia), and it was cured for 60 seconds at a distance of 1 cm with a 1200 mW intensity lamp (Raddi Cal, SDI). A radiographic control seal of the root canal was then performed. Once the proper seal was confirmed, clinical and radiographic intracoronal bleaching was performed.

Four Whitening Sessions

Application of the whitening agent was performed according to the manufacturer's instructions. The correct amount of bleaching gel for each group was placed into the pulp cavity with the presence of mild moisture (using the walking bleach technique), which allowed a close and optimum cavity seal. Cavity closure was performed using a temporary cement (Fermin, Detax, Ettlingen, Germany) until the next session in seven days. This procedure was repeated at each of the next four sessions. The same amount of gel was used and changed for both products and the same number of times.

Final Session

After the cavity access well was washed with water, a temporary restoration was placed for seven days until the final restoration with composite resin was made. Patients were cautioned not to eat or drink foods that could dye their teeth, such as coffee, tea, or red wine, during the study period. They were given these directions in writing and provided with contact information if they had any questions or experienced adverse events.

Color Evaluation

Objective Evaluation—Two calibrated evaluators (Kappa=0.85) were used to measure tooth color for the baseline immediately after each of the four sessions and again at one week and at one month after the last session. Color evaluation was obtained from a 6-mm area located in the middle third of the labial surface of the left and right central incisors. To standardize this evaluation, an impression of the maxillary arch was taken to make a guide using high-putty silicone (Zetaplus, Zhermack, Badia Polesine, Rovigo, Italy). A window was created on the labial surface in the middle third of the central incisor using a device with well-formed borders and a 3-mm radius corresponding to the reflectance of the spectrophotometer (Vita EasyShade Compact, VITA Zahnfabrik, Bad Säckingen, Germany), which has good reliability.[14] The shade was determined using the obtained parameters L*, a*, and b*. Color alteration after each session was indicated by the differences between the values obtained at the session and the baseline (ΔE). The ΔE was calculated using the following formula: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. The evaluators considered the color of the tooth counterpart (treated tooth: upper right central incisor; tooth counterpart: left central incisor), and the same area (middle) of each tooth was evaluated to compare color matching.

Subjective Evaluation—For the subjective evaluation, the 16 tabs of the shade guide (Vita Classic, Vita Zahnfabrik) were arranged from the highest (B1) to the lowest (C4) value to assess the color. Two calibrated evaluators (Kappa=0.85) recorded the shade of the upper central left and right incisors at baseline and at the same time points as for the objective evaluation. The investigators checked the color in the middle third area of the labial surface on the anterior central incisor, according to the American Dental Association guidelines.[15] The color changes from the beginning of the active phase through the individual recall times were calculated using the change in the number of shade guide units (ΔSGU). The color of the counterpart tooth was also recorded subjectively and compared to that of the treated tooth.

Oral Health Impact Profile Questionnaire

Satisfaction was measured using the Oral Health Impact Profile (OHIP-Esthetics) questionnaire validated in Chilean Spanish.[16] The questionnaire was administered by a research operator at baseline, one week, and one month after bleaching.

Psychosocial Impact of Dental Esthetics Questionnaire

The Psychosocial Impact of Dental Esthetics questionnaire (PIDAQ) consisted of 23 items that were grouped into four components using factor analysis:

Table 1: *Participant Baseline Characteristics*

Baseline Features	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide
Age (years; mean±SD)	30.88±11.58	30.83±11.25
Minimum age (y)	19	20
Maximum age (y)	65	65
Male (%)	50	39.13
Trauma (%)	58.33	39.13
Caries (%)	41.67	60.87
SGU baseline median (min;max) Vita Classic	15 (5; 16)	12 (7; 16)
L* (mean±SD)	73.55±8.54	75.91±6.81
a* (mean±SD)	4.48±3.45	4.85±3.39
b* (mean±SD)	29.14±3.87	31.79±6. 60

Abbreviation: max, maximum; min, minimum; SD, standard deviation.

1) dental self-confidence, 2) social impact, 3) psychosocial impact, and 4) esthetic concern.[17]

Statistical Analysis

After verifying the normality of data distribution and the homogeneity of the variance-covariance matrix, the treatment efficacy was evaluated with respect to color alteration (ΔE and ΔSGU) and analyzed using the Wilcoxon test for within-group comparisons and the Mann-Whitney test for between-group comparisons. The statistical analyses were performed using SPSS 23.0 (SPSS Inc, Chicago, IL, USA) with $\alpha=0.05$. For comparison of OHIP-Esthetics and PIDAQ questionnaire scores, the Wilcoxon test was used.[18]

RESULTS

Participant characteristics are shown in Table 1. There were no statistically significant differences between the participants' characteristics in the different groups (Mann Whitney test $p>0.05$). Results for ΔSGU differed over time, as shown in Table 2, with more effectiveness in group 1 ($p<0.05$). However, at the final measurement one month after bleaching, values were not significantly different ($p=0.59$). The color change determined using ΔL*, Δa*, and Δb* is shown in Table 3. The ΔL* was different at all measurement times, with values higher for group 1 ($p<0.05$). The values of Δa* and Δb* were similar for both groups ($p>0.05$). The ΔE color difference is shown in Table 4. The effectiveness was similar to baseline ($p>0.05$) at all-time points except that a significant difference from baseline ($p<0.05$) was observed for both groups at sessions 3 and 4. These two groups showed high effectiveness, with an average change of 14 color units.

Table 5 shows the subjective comparison of the treated teeth and their counterparts using Vita Classical SGU. There were no statistically significant differences ($p>0.05$) between treated teeth and controls in the first week. However, in the monthly monitoring of group 2 there were differences in color between treated teeth and counterpart teeth ($p<0.05$). Table 6 shows the objective comparison of the treated teeth and their counterparts from the L*, a*, and b* measured by the spectrophotometer. The L* values from one week after bleaching were not statistically significant ($p>0.05$), and the values

Table 2: *Comparison of ΔSGU Values at Different Times Using the Vita Classic Scale*[a]

Assessment Points	Color Change by ΔSGU		Mann-Whitney Test
	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide	
Baseline vs 1-wk bleaching	3.5 (0, 10)	3 (0, 9)	**0.04**
Baseline vs 2-wk bleaching	8 (0, 14)	4 (0, 11)	**0.04**
Baseline vs 3-wk bleaching	9.5 (0, 14)	7 (0, 13)	**0.02**
Baseline vs 4-wk bleaching	11 (1, 15)	7 (1, 14)	**0.02**
Baseline vs 1-wk after bleaching (before restoration)	10 (−1, 14)	7 (1, 13)	**0.04**
Baseline vs 1-wk after bleaching (after restoration)	9.5 (1, 14)	7 (2, 13)	**0.04**
Baseline vs 1-mo after bleaching	8.5 (1, 13)	7 (1, 13)	**0.59**

[a] *Bold indicates statistical difference between groups at indicated time point. Expressed in median (min, max).*

Table 3: *Color Change by ΔL, Δa, and Δb (Means and Standard Deviations) at All Time Points*[a]

Assessment Times	Color Change by ΔL			Color Change by Δa			Color Change by Δb		
	Hydrogen Peroxide	Carbamide Peroxide	Mann-Whitney Test	Hydrogen Peroxide	Carbamide Peroxide	Mann-Whitney Test	Hydrogen Peroxide	Carbamide Peroxide	Mann-Whitney Test
Baseline vs 1-wk bleaching	8.13±4.73	4.33±4.31	**0.006**	–2.88±2.14	–2.63±2.32	.709	–1.22±3.82	–3.18±4.18	.100
Baseline vs 2-wk bleaching	10.26±7.06	5.98±4.70	**0.019**	–4.20±2.82	–3.96±2.97	.782	–3.11±4.70	–5.70±5.97	.105
Baseline vs 3-wk bleaching	11.78±6.55	7.98±4.26	**0.023**	–5.03±2.72	–4.51±2.69	.516	–5.68±5.95	–5.38±5.96	.865
Baseline vs 4-wk bleaching	13.07±7.43	8.64±3.53	**0.013**	–5.70±2.99	–5.20±2.85	.561	–6.18±5.93	–6.63±5.85	.796
Baseline vs 1 w k after bleaching (before restoration)	11.54±8.23	7.64±3.93	**0.045**	–5.52±3.58	–5.45±2.88	.939	–6.60±5.95	–8.00±7.43	.480
Baseline vs 1 w k after bleaching (after restoration)	10.85±7.20	7.25±4.53	**0.047**	–6.32±3.04	–5.67±2.75	.452	–7.24±5.99	–7.92±7.75	.737
Baseline vs 1 mo after bleaching	7.75±6.03	4.41±4.63	**0.039**	–6.53±2.92	–6.07±2.50	.569	–9.52±5.49	–9.97±6.21	.796

[a] *Bold indicates statistical difference between groups at indicated time point.*

from one month after bleaching were statistically significantly different ($p<0.05$) from the L* values of the homologous teeth.

Regarding the a* values in group 1, the comparison of one week after restoration and one month after restoration was statistically significant ($p<0.05$). In group 2 there were no statistically significant differences ($p>0.05$).

Regarding the b* values, there was a statistically significant difference in group 2 at the one week recall, and there was no statistically significant difference after one month ($p>0.05$).

Oral Health Impact Profile

There was a statistically significant difference in the OHIP-Esthetics score for the baseline compared with the sessions and one month after treatment ($p<0.05$; Wilcoxon test, Table 7). The factors of functional limitation and psychological disability were statistically significant compared to the baseline values ($p<0.05$). In group 1, the psychological discomfort and handicap factors were statistically significant at one week and one month compared with baseline ($p<0.05$), and the handicap factor at the one-month assessment was statistically significant in group 2 ($p<0.05$).

Psychosocial Impact of Dental Esthetics Questionnaire

The PIDAQ score was significantly different at baseline compared with one week and one month after treatment ($p<0.05$; Wilcoxon test, Table 8) except for the psychological assessment and total PIDAQ sum for group 2 at one month after treatment. When the one-week and one-month

Table 4: *Color Change Expressed in Units (ΔE; Means and Standard Deviations) at All Time Points*[a]

Assessment Times	Color Change by ΔE		Mann-Whitney Test
	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide	
Baseline vs 1-wk bleaching	9.67±4.79	7.40±4.64	.11
Baseline vs 2-wk bleaching	13.24±5.94	11.04±5.19	.18
Baseline vs 3-wk bleaching	15.69±5.79	12.17±4.93	**.03**
Baseline vs 4-wk bleaching	17.19±6.55	13.31±4.67	**.02**
Baseline vs 1 w k after bleaching (before restoration)	16.44±7.10	14.36±4.74	.25
Baseline vs 1 w k after bleaching (after restoration)	16.22±6.46	14.45±4.88	.30
Baseline vs 1 mo after bleaching	15.48±5.17	14.02±4.85	.32

[a] *Bold indicates statistical difference between groups at indicated time point.*

Table 5: *Comparison of SGU Values at Different Times Using the Vita Classical Scale, Median (Minimum:Maximum)*

Assessment Times	Color Value	
	G1	G2
Homologous teeth	5 (1:9)	3 (2:10)
Baseline	15 (5:16)[a]	12 (7:16)[a]
1-wk bleaching	10.5 (2:16)[a]	9 (5:16)[a]
2-wk bleaching	5 (1:15)	7 (1:15)[a]
3-wk bleaching	4 (1:15)	5 (2:15)[a]
4-wk bleaching	2 (1:12)[a]	5 (1:15)
1 wk after bleaching (before restoration)	4.5 (1:13)	5 (1:15)
1 wk after bleaching (after restoration)	3.5 (1:12)	4 (1:14)
1 mo after bleaching	5 (1:13)	6 (1:14)[a]

[a] *Statistically significant difference intragroup (Wilcoxon test,* $p<0.05$*) versus Homologous teeth (counterpart).*

posttreatment time points are compared, the only statistically significant differences were found in the field of self-confidence ($p<0.05$) for group 1 and the esthetic concern factor for both groups ($p<0.05$).

DISCUSSION

This randomized clinical study showed that the effectiveness of two bleaching agents (35% hydrogen peroxide and 37% carbamide peroxide) can be measured objectively and subjectively, and both agents can be applied using the walking bleach technique for bleaching nonvital intracoronal teeth. Both products showed high effectiveness, and the results were similar and highly reproducible one month after whitening. Our results showed that the treatment had a positive influence on esthetic self-perception and psychosocial impact at one month after treatment, after color improvement in only one tooth in most volunteers in this trial. Therefore, the null hypothesis is accepted, as the two gels were widely effective according to objective and subjective measurements, and they had similar positive effects on the esthetic perception and psychosocial impact of patients in this clinical trial.

Several studies showed that bleaching can be considered effective when there is a change of at least 5 ΔE units.[19] Our results showed that in four sessions using the walking bleach technique and up to one month after treatment, there was a change of 15.48 ± 5.17 of ΔE for group 1 and 14.02 ± 4.85 of ΔE for group 2, which was highly effective. The first session achieved a considerable change of approximately 50% of the final result. There was no difference between the agents in the final results ($p>0.05$), although there was a trend for color to recur at one month after treatment. Statistical significance in ΔE was found only at the second and third sessions during treatment, even though ΔL* was different at all times, with higher values for hydrogen peroxide. However, ΔE was similar in both groups, which suggests that there is a different chemical mechanism for both gels in the respective groups; hydrogen peroxide gel has a lower molecular weight and a quicker and more direct action, so it probably spreads faster than carbamide peroxide.[20] Carbamide peroxide degrades at a lower concentration of hydrogen peroxide,[21] which results in a slower process and no difference in effectiveness one month after bleaching. Subjective evaluation of the change in coloration also indicates a highly effective treatment. When comparing both products using subjective measurement, a statistically significant difference was found at all time points until one week after treatment, while differences in the objective measurement were only found at the second and third sessions of whitening. Luminosity

Table 6: *Color by L*, a*, and b* (Means and Standard Deviations) at All Time Points and Color of the Homologous Teeth*

Assessment Times	L* Values		a* Values		b* Values	
	Group 1	Group 2	Group 1	Group 2	Group 1	Group 2
Homologous teeth	84.39±2.62	84.77±4.91	–0.91±0.55	–1.16±0.82	20.10±3.47	19.43±3.21
Baseline	73.55±8.54[a]	75.91±6.81[a]	4.48±3.45[a]	4.85±3.39[a]	29.14±3.87[a]	31.79±6.60[a]
1-wk bleaching	81.68±7.20	80.24±7.51[a]	1.60±3.57[a]	2.22±4.02[a]	27.93±4.54[a]	28.61±5.51[a]
2-wk bleaching	83.81±6.47	81.89±6.95	0.28±3.38	0.89±4.35	26.03±4.31[a]	26.10±5.49[a]
3-wk bleaching	85.34±5.64	83.89±6.55	–0.55±3.24	0.34±3.98	23.46±5.10[a]	26.41±5.80[a]
4-wk bleaching	86.63±5.80	84.56±6.30	–1.23±3.01	–0.36±3.73	22.96±5.08[a]	25.17±5.30[a]
1 w k after bleaching (before restoration)	85.10±7.08	83.55±5.96	–1.04±3.42	–0.60±3.92	22.54±4.83	23.79±5.99[a]
1 w k after bleaching (after restoration)	84.41±5.64	83.16±7.32	–1.84±2.51[a]	–0.83±3.77	21.90±4.83	23.87±6.08[a]
1 mo after bleaching	81.31±5.73[a]	80.33±5.38[a]	–2.05±2.33[a]	–1.22±3.03	19.62±4.84	21.83±5.29

[a] *Statistically significant difference intragroup (Wilcoxon test,* $p<0.05$*) vs homologous teeth (counterpart).*

Table 7: *Effect of Intracoronal Bleaching on Esthetic Self-Perception Evaluated With the Oral Health Impact Profile Questionnaire*

Dimension	Baseline			1 Wk After Bleaching			1 Mo After Bleaching		
	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide	Mann-Whitney Test	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide	Mann-Whitney Test	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide	Mann-Whitney Test
Functional limitation	5 (2:8)	5 (2:8)	0.754	3 (0:6)[a]	4 (1:6)[a]	0.357	2.5 (0:7)[a]	4 (0:8)[a]	0.435
Physical pain	3.5 (0:6)	3 (0:5)	0.366	3 (1:6)	3 (1:6)	0.575	2.5 (0:6)	2 (0:8)	0.829
Psychological discomfort	5 (2:6)	5 (0:7)	0.670	4 (0:6)[a]	3 (2:7)	0.974	4 (0:6)[a]	4 (0:8)	0.754
Physical disability	1.5 (0:6)	2 (0:5)	0.710	1.5 (0.4)	1 (0:6)	0.893	1 (0:5)	0 (0:6)	0.672
Psychological disability	2.5 (0:6)	3 (0:8)	0.257	2 (0:6)[a]	2 (0.7)[a]	0.905	1.5 (0:5)[a]	2 (0:6)[a]	0.533
Social disability	0 (0:5)	1 (0:6)	0.854	0 (0:4)[a]	0 (0:6)	0.921	0 (0:6)	0 (0:4)	0.950
Handicap	1 (0:6)	1 (0:6)	0.718	0 (0:5)[a]	0 (0:6)	0.690	0 (0:6)[a]	0 (0:4)[a]	0.933
Sum	18 (5:38)	19 (5:42)	0.873	13 (3:33)[a]	15 (4:41)[a]	0.529	14 (2:31)[a]	13 (5:41)[a]	0.983

[a] *Statistically significant difference (Wilcoxon test, $p<0.05$) versus baseline.*

can explain the differences (Table 3) because it is a parameter with more power to determine color than the human eye.[19] At the one month recall, there was a slight rebound of color, which can be explained by the rehydration of the tooth subjected to high concentrations of peroxide for several sessions. This may suggest that clinicians should aim to overbleach by at least a tone to compensate for this rebound.

Few clinical studies have evaluated the effectiveness of nonvital bleaching, and the most commonly used bleaching agent is sodium perborate.[7] An *in vitro* study conducted by Lim and others[22] concluded that 35% carbamide peroxide and 35% hydrogen peroxide are more effective than sodium perborate. In this study, the color of each tooth was evaluated using the Vita Lumin shade guide; after seven days, there was change of eight SGU, and the color changed by two additional units after 14 days for carbamide and hydrogen peroxide.[22]

Regarding comparing subjective and objective evaluations of tooth color with an untreated counterpart, we can say that the color of the treated tooth resembled that of the counterpart according to the subjective evaluation; however, it is not possible to perfectly match the color of all teeth. This represents a difficulty of the technique, and perhaps the clinician should apply a custom technique for each patient based on the tones that should be whitened.

The same situation occurs in the objective assessment of the L* values, which are similar after one week but are different at one month because of the rebound in luminosity. In group 1, there was apparently an influence of color restoration on values a*, which remained for a month, whereas in group 2 there was no difference between the treated tooth

Table 8: Psychosocial Impact of Dental Esthetics *Results at Different Time Points*

Dimension	Baseline			1 Wk After Bleaching			1 Mo After Bleaching		
	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide	Mann-Whitney Test	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide	Mann-Whitney Test	Group 1 = Hydrogen Peroxide	Group 2 = Carbamide Peroxide	Mann-Whitney Test
Dental self-confidence	16.5 (6:26)	14 (10:26)	0.386	21 (11:30)[a]	23 (12:30)[a]	0.653	19 (10:30)[ab]	22 (6:29)[a]	0.708
Social impact	24 (8:40)	23 (8:34)	0.693	17.5 (8:31)[a]	16 (8:26)[a]	0.315	15.5 (8:31)[a]	17 (8:33)[a]	0.764
Psychological impact	19.5 (8:26)	17 (6:24)	0.347	13.5 (6:24)[a]	15 (6:21)[a]	0.949	14 (6:24)[a]	14 (6:25)	0.991
Esthetic concern	9.5 (3:14)	9 (3:14)	0.604	8 (3:12)[a]	5 (3:12)[a]	0.094	5.5 (3:12)[ab]	6 (3:12)[ab]	0.320
Sum	71 (38:98)	66 (39:81)	0.365	63.5 (40:79)[a]	57 (39:74)[a]	0.287	54 (39:79)[a]	58 (40:94)	0.733

[a] *Statistically significant difference (Wilcoxon test, $p<0.05$) vs baseline.*
[b] *Statistically significant difference (Wilcoxon test, $p<0.05$) vs 1 wk after bleaching.*

and the tooth counterpart after one month. As for values b*, there was no difference between the treated tooth hand the counterpart tooth after one month.

It is likely that stronger action of agents generates color change in a darker tooth.[23] The research team was not surprised at the high degree of patient satisfaction achieved with the bleaching procedure in this trial,[24] even though, for various reasons, we were unable to completely match tooth color to the neighboring teeth using our nonvital tooth-whitening method. Findings on the perception of esthetics and psychosocial impact were positive and significant up to one month after whitening. Maintaining this positive effect over time should be confirmed in patients undergoing bleaching of vital teeth[25] and correlated with the maintenance of the effectiveness by a bleaching procedure.

The OHIP-Esthetics survey also showed a positive effect on patients' esthetic self-perception. This result is supported by other studies[2,25] that evaluated the influence on esthetic self-perception of patients undergoing extracoronal vital whitening. Our data confirm that quality of life is complex, important, and poorly reported, and multiple factors influence it. However, dental esthetics is important, even if the influence of patients' perception and the impact of psychological and social factors is unknown. The impact of perception on psychological and social factors is unknown. There were no differences in OHIP-esthetics scores between groups, but there were differences in the changes over time in each group. This resulted in a more pronounced and positive effect for group 1, which may be explained by the more rapid effect on the modification of color brightness, as shown in Table 3. This should be further investigated to assess the correlation between self-perception and esthetic changes in the brightness of the color.

The PIDAQ is not designed for patients who have undergone bleaching as it is usually used for orthodontic patients to determine their esthetic expectations; however, it fails to evaluate which areas are expected to be modified to solve a particular esthetic problem. The PIDAQ has been shown to decrease the negative impact of cosmetic dentistry for a patient and to decrease the values in the field of social impact, psychological impact and esthetic concerns. The psychosocial impact was similar in both groups ($p>0.05$). We found that there was a large impact reported by the PIDAQ data for all relevant factors, but a minimal intervention on a tooth has an important effect on people who undergo intracoronal bleaching.[25]

CONCLUSION

Both 35% hydrogen peroxide and 37% carbamide peroxide are highly effective for the walking bleach technique in nonvital teeth and achieve a high degree of color matching with the counterpart teeth. Each gel resulted in a positive impact on patients' esthetic self-perception and psychosocial self-perception after intracoronal whitening.

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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 CEC FOUCH. The approval code for this study is 2016/04.

Conflict of Interest

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

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Laboratory Research

Effect of Hydrofluoric Acid Concentration and Etching Time on Bond Strength to Lithium Disilicate Glass Ceramic

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Clinical Relevance

The minimum HF concentration and time for proper bonding to lithium disilicate ceramics is 5% and 20 seconds.

SUMMARY

The aim of this study was to evaluate the influence of different concentrations of hydrofluoric acid (HF) associated with varied etching times on the microshear bond strength (μSBS) of a resin cement to a lithium disilicate glass ceramic. Two hundred seventy-five ceramic blocks (IPS e.max Press [EMX], Ivoclar Vivadent), measuring 8 mm × 3 mm thickness, were randomly distributed into five groups according to the HF concentrations (n=50): 1%, 2.5%, 5%, 7.5%, and 10%. Further random distribution into subgroups was performed according to the following etching times (n=10): 20, 40, 60, 120, and 20 + 20 seconds. After etching, all blocks were treated with a silane coupling agent followed by a thin layer of an unfilled resin. Three resin cement cylinders

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(=1 mm) were made on each EMX surface, which was then stored in deionized water at 37°C for 24 hours before testing. The μSBS was in a universal testing machine at a crosshead speed of 1 mm/min until failure. Data were submitted to two-way analysis of variance, and multiple comparisons were performed using the Tukey post hoc test (α=0.05). One representative EMX sample was etched according to the description of each subgroup and evaluated using scanning electron microscopy for surface characterization. The HF concentrations of 5%, 7.5%, and 10% provided significantly higher μSBS values than 1% and 2.5% ($p<0.05$), regardless of the etching times. For 1% and 2.5% HF, the etching times from 40 to 120 seconds increased the μSBS values compared with 20 seconds ($p<0.05$), but etching periods did not differ within the 5%, 7.5%, and 10% HF groups ($p>0.05$). The effect of re-etching was more evident for 1% and 2.5% HF ($p<0.05$). Different HF concentrations/etching times directly influenced the bond strength and surface morphology of EMX.

INTRODUCTION

Glass ceramic materials have been widely used in dentistry to restore lost/fractured/decayed teeth. Their optimum properties, adhesion ability to dental tissues,[1] high esthetics, biocompatibility, and thermal expansion similar to the tooth structure are the key factors for their adoption by dental practioners.[2-4] Glass ceramic reinforced by lithium disilicate crystals has shown excellent clinical outcomes with great optical/mechanical properties[5-7] and high survival rates over time.[8,9]

The clinical success of ceramic restorations depends on several factors, such as ceramic composition and luting procedures.[10] The ideal bonding to lithium disilicate glass ceramic is achieved with the sum of etching with hydrofluoric acid (HF) followed by a silane coupling agent. This protocol has been recognized as the most accepted surface treatment for glass ceramics.[3,4,11,12] HF has the ability to condition the ceramic surface, since it removes the glassy matrix and exposes the lithium disilicate crystals.[4,13-15] As a consequence, an increased surface area for micromechanical entanglement is promoted, improving the interaction between ceramic and resin cement with increased bond strength.[1,3,4,15-17] Also, the use of a thin layer of unfilled resin prior to the resin cement improved bond strength and the interfacial quality between lithium disilicate glass ceramic and resin cement by promoting a better infiltration to the superficial irregularities of the etched ceramic surfaces.[4]

The etching efficiency of HF depends on the concentration, etching time, temperature, and dilution of the acid solution.[4,10,11,15,18] The manufacturer of IPS e.max Press (EMX; Ivoclar Vivadent, Schaan, Liechtenstein), a commercial brand that presents ±70 vol% of lithium disilicate crystals dispersed in an amorphous vitreous phase, recommends that EMX should be etched with a 4.8% HF concentration for 20 seconds. However, several clinical reports and *in vitro* studies, published from 2011 to date, have reported different HF concentrations and etching periods on lithium disilicate ceramic, such as 10% for 15 seconds,[19] 10% for 20 seconds,[6,7,10,20-22] 9.6% for 30 seconds,[23] 9.5% for 60 seconds,[3] 4.8% for 60 seconds,[24,25] 5% for 60 seconds,[26] and 4.8% and 5% for 20 seconds.[27-29] Thus, a consensus regarding the most suitable etching protocol for glass ceramics is not clear,[30] especially for lithium disilicate glass ceramics.

Although the effects of HF on glass ceramics are elucidated in the literature, there have been no reports that specifically associated different HF concentrations with increased etching times on the bond strength of EMX. In addition, given the hazardous nature of HF,[17,18] it is important to discuss how lower HF concentrations and different etching times would influence the bonding characteristics of EMX and, consequently, guide the clinician toward the more adequate etching protocol to be adopted.

Therefore, the purpose of this study was to evaluate the effect of different HF concentrations associated with varied etching times on the microshear bond strength (μSBS) and surface morphology (scanning electron microscopy [SEM] analysis) of EMX luted with a resin cement. The hypotheses tested were 1) HF concentrations affect the μSBS and the etched surface morphology and 2) different etching times affect the μSBS and the etched surface morphology.

METHODS AND MATERIALS

Fabrication of the EMX Specimens

Two hundred seventy-five ceramic blocks of EMX (8 mm × 8 mm × 3 mm thickness), shade LT A2, were fabricated in accordance with the manufacturer's instructions and detailed in a previous study.[4]

After divestment, the EMX blocks were wet polished with silicon-carbide abrasive papers (600-,

1200- and 2000-grit; Norton SA, São Paulo, SP, Brazil) in an automatic polisher (APL4; Arotec, Cotia, SP, Brazil) to obtain a flat and polished surface. Then, all EMX blocks were ultrasonically cleaned (MaxiClean 750; Unique, Indaiatuba, SP, Brasil) in deionized water for 20 minutes and dried with compressed air.

EMX Surface Treatments

The EMX blocks were randomly assigned into five groups (n=50) according to the HF concentrations: 1%, 2.5%, 5%, 7.5%, and 10% (Fórmula & Ação, São Paulo, SP, Brazil). A new random distribution was performed into five subgroups (n=10) according to the etching times: 20, 40, 60, 120, and 20 + 20 seconds. After etching, each EMX surface was rinsed with oil-free compressed air/water spray for 30 seconds. For the re-etched group (20 + 20 seconds), the EMX surface was rinsed as described previously and air dried before the second etching procedure, which focused on simulating the retreatment after clinical contamination of the etched surface.[18] After etching, all EMX blocks were ultrasonically cleaned (MaxiClean 750) in deionized water for 20 minutes and dried for 30 seconds.

One layer of a silane coupling agent (RelyX Ceramic Primer, 3M ESPE, St Paul, MN, USA) was applied to the entire etched EMX surface and left in contact for 60 seconds, followed by air heat drying for 60 seconds to accelerate the water/alcohol evaporation. Then, all EMX surfaces received a thin layer of photo-activated unfilled resin[4] (Scotchbond MultiPurpose, 3M ESPE). The excess was removed with a microbrush and dry air from a dental syringe for five seconds and light cured for 20 seconds using a light-emitting diode (LED) device (Valo Cordless, standard mode; Ultradent Inc, South Jordan, UT, USA) with an irradiance of 1000 mW/cm^2.

μSBS Testing Procedures

The setup design of the μSBS test has been previously described by Naves and others[18] and Sundfeld Neto and others[4] and is represented in Figure 1. Round molds (1-mm thick) containing three cylinder-shaped orifices (1 mm in diameter) were made with elastomer (Oranwash L, Zhermack, Badia Polesine, Italy) and placed onto the EMX surface for the delimitation of the bonding area. The cylindrical orifices were filled with resin cement (Variolink II, Shade Transparent; Ivoclar Vivadent). A mylar strip and a glass slab were placed over the filled mold, followed by a vertical load of 250 g applied for 2 minutes to standardize the height of the resin cement cylinders.[4] Afterward, both vertical load and glass slab were removed and the resin cement cylinders were light-cured for 40 seconds using an LED source with an irradiance of 1000 mW/cm^2 (Valo Cordless). All specimens were stored in deionized water for 24 hours at 37°C. Then, the elastomer mold was sectioned with a No. 11 scalpel blade and carefully removed. Each resin cement cylinder interface was analyzed with optical microscopy (Olympus Corp, Tokyo, Japan) at 40× magnification, and those presenting any flaws or defects were eliminated and replaced. Three cylinders were built on each EMX surface, totaling 30 cylinders for each group.

The μSBS test was performed in a universal mechanical testing machine (model 4411; Instron, Canton, MA, USA) using a thin steel wire (0.2 mm in diameter) at a crosshead speed of 1 mm/min until failure. A mounting jig was used for the parallel alignment of the ceramic-resin cylinder interface to the testing device. The steel wire was looped around each resin cement cylinder and aligned with the bonding interface. The fractured specimens were observed under optical microscopy (Olympus) at 40× magnification, and the mode of failure was classified as follows: adhesive (mode 1), cohesive within ceramic (mode 2), cohesive within resin cement (mode 3), and mixed, involving resin cement and ceramic (mode 4).

SEM Analysis

One representative etched EMX specimen of each group was submitted to SEM analysis to depict the etched surface morphology. The EMX blocks were mounted on aluminum stubs, sputter coated with gold (SCD 050; Balzers, Schaan, Liechtenstein) for 120 seconds at 40 mA, and examined by SEM (JSM 5600 LV; JEOL, Tokyo, Japan) with 2000× magnification and operated at 15 kV by the same operator.

Statistical Analysis

Values of μSBS were calculated and the data provided in megapascals. The average value of the three cylinders was recorded as the bond strength for each specimen. Thus, the mean bond strength values of each group represented the mean of the 10 specimens. Exploratory data analyses were performed prior to applying an analysis of variance (ANOVA). The data of μSBS were submitted to two-way ANOVA (HF concentration × etching time), and multiple comparisons were performed using the Tukey post hoc test (α=0.05).

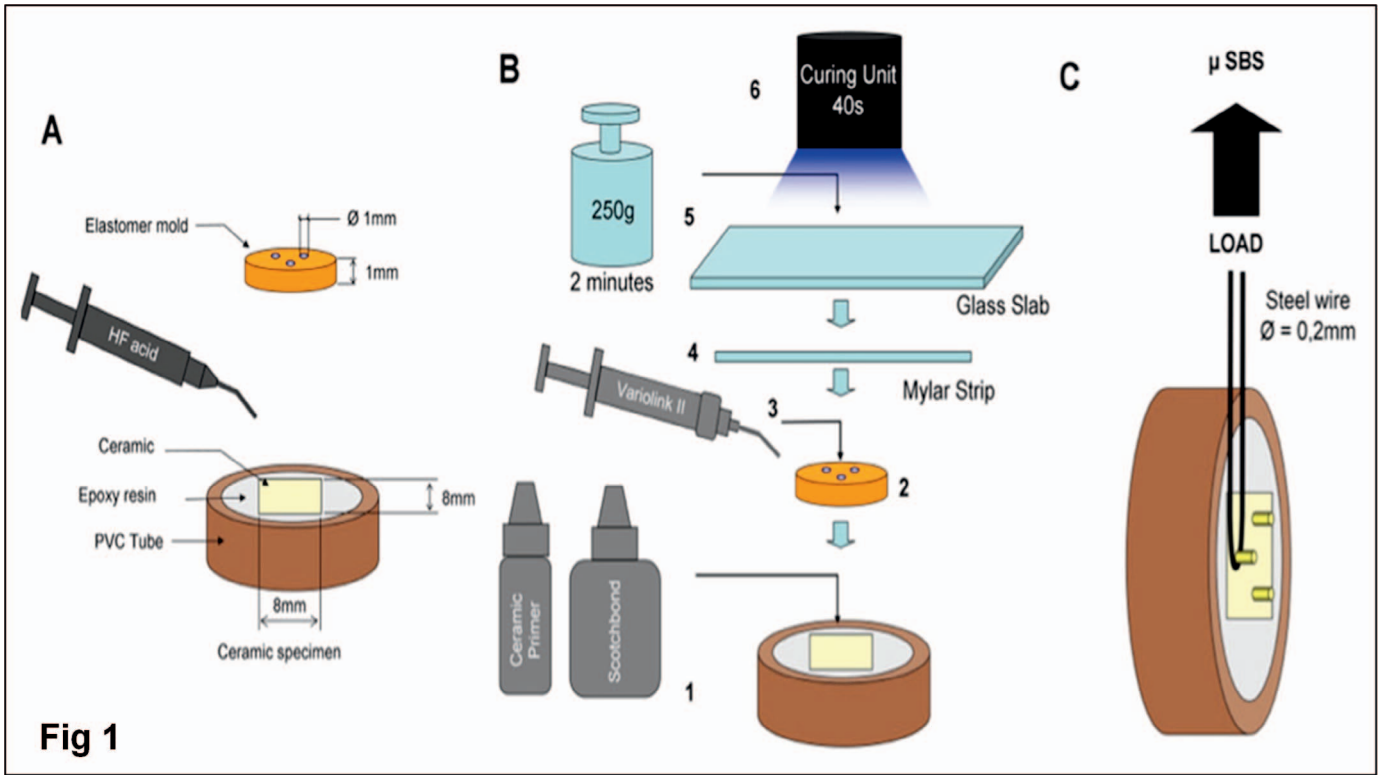

Figure 1. *Experimental design of the study. After etching (A), 1) silane was applied to the ceramic surface, followed by a thin layer of unfilled resin light-cured for 20 seconds; 2) an elastomer mold with cylinder-shaped orifices was positioned onto the surface; 3) orifices were filled with resin cement (Variolink II); 4) a mylar strip and glass slab were placed over the filled mold; 5) cementation load was applied for 2 minutes; 6) light curing of the resin cement was carried out for 40 seconds. (C) Microshear bond strength testing.*

RESULTS

μSBS

The mean μSBS values are shown in Table 1. The interaction between the HF concentrations and etching times ($p<0.01399$) was significant. The HF concentrations ($p<0.0001$) and etching times ($p<0.0001$) directly influenced the μSBS values.

HF concentrations of 5%, 7.5%, and 10% provided significantly higher μSBS values than 1% and 2.5% ($p<0.05$), regardless of the etching times. When the specimens were etched from 40 to 120 seconds, the μSBS for 1% and 2.5% HF concentrations was significantly higher than 20 seconds ($p<0.05$). For 2.5% HF concentrations, no statistical differences were found among the etching times of 40, 60, 120, and 20 + 20 seconds ($p>0.05$). No statistical difference was found among 5%, 7.5%, and 10% HF concentrations ($p>0.05$). When etching times were compared, no statistical differences were found among 5% and 10% HF concentrations ($p>0.05$).

Failure Analysis

A descriptive analysis of the failure modes is shown in Table 2. A predominance of adhesive failures (mode 1) was detected for 1% and 2.5% HF

Table 1: *Means of μSBS (MPa) (±SD) for All Groups*[a]

Hydrofluoric Acid Concentrations (%)	Etching Times (s)				
	20	40	60	120	20 + 20
1	10.2 (±2.1) cC	16.2 (±1.1) cB	14.1 (±1.9) cBC	22.0 (±1.1) bA	16.3 (±1.2) cB
2.5	16.8 (±2.6) bB	21.8 (±2.9) bA	24.2 (±1.2) bA	25.9 (±1.3) bA	22.0 (±1.8) bA
5	27.8 (±1.6) aA	28.4 (±1.2) aA	30.1 (±2.2) aA	30.9 (±1.4) aA	28.9 (±1.7) aA
7.5	28.1 (±1.6) aB	29.9 (±1.3) aAB	32.3 (±1.8) aAB	33.0 (±1.6) aA	30.2 (±2.8) aAB
10	31.1 (±1.7) aA	31.0 (±1.4) aA	32.9 (±2.2) aA	33.8 (±1.5) aA	30.7 (±1.3) aA

[a] *Means followed by different lowercase letters in each column and upper capital letters in each line differ statistically by Tukey's test at 5%.*

Table 2: *Distribution of Failure Modes Among Groups (%)*[a]

HF Concentrations × Etching Times	Failure Modes			
	Mode 1	Mode 2	Mode 3	Mode 4
1%				
20 s	61	0	0	39
40 s	72	0	0	28
60 s	78	0	0	22
120 s	69	0	0	31
20 + 20 s	72	0	0	28
2.5%				
20 s	83	0	0	17
40 s	58	0	0	42
60 s	56	0	0	44
120 s	64	0	0	36
20 + 20 s	75	0	0	25
5%				
20 s	48	0	0	52
40 s	51	0	0	49
60 s	36	0	0	64
120 s	36	0	3	61
20 + 20 s	36	0	0	64
7.5%				
20 s	44	0	0	56
40 s	56	0	0	44
60 s	47	0	0	53
120 s	42	0	0	58
20 + 20 s	50	0	0	50
10%				
20 s	42	0	0	58
40 s	50	0	0	50
60 s	47	0	0	53
120 s	47	0	3	50
20 + 20 s	56	0	0	44

[a] *Failure modes: adhesive (mode 1), cohesive within ceramic (mode 2), cohesive within resin cement (mode 3), and mixed, involving resin cement and ceramic (mode 4).*

concentrations, regardless of the etching times. Conversely, for 5%, 7.5%, and 10% HF concentrations, a slight predominance of mixed failures (mode 4) were verified, regardless of the etching times. No cohesive failures within resin cement or ceramic were recorded.

SEM Evaluation

The SEM images of the etched EMX surfaces are shown in Figures 2, 3, 4, 5, and 6. The HF concentrations and etching times directly influenced the etching morphology of EMX. Different etching patterns were observed for different etching periods for l% HF concentration, with a slight increase of glassy matrix removal with higher etching times (Figures 2A-E). The same effect was verified for 2.5% HF (Figures 3A-E). For 5% HF concentration, the glassy matrix removal and the consequent exposure of lithium disilicate crystals were more marked as the etching times increased (Figures 4A-E). For 7.5% and 10% HF concentrations, different etching times seemed to not have affected the EMX etched morphology to the same extent as lower HF concentrations/etching times (Figs. 5 and 6). Re-etching (20 + 20 seconds) with 2.5% HF concentration showed a slight increase in the glassy matrix removal compared with the other re-etched groups.

DISCUSSION

The present study assessed the influence of several HF concentrations associated with different etching times on the surface morphology/μSBS of lithium disilicate glass ceramic to resin cement. Through SEM and μSBS analysis, a clear effect of HF concentrations and etching times was verified.

The results indicate that the first tested hypothesis was confirmed. The 5%, 7.5%, and 10% HF concentrations showed the highest μSBS values, regardless of the etching times. This was directly associated with a greater dissolution of the vitreous phase and exposure of lithium disilicate crystals (needle-like appearance; Figs. 4-6) than 1% and 2.5% HF concentrations (Figs. 2 and 3). These findings are in accordance with previous studies, which have reported higher μSBS values for HF concentrations ranging from 5% to 10% applied for 20 seconds on EMX at room temperature.[4,15] The etching reaction of HF on EMX is elucidated by the equation $4HF + SiO_2 \rightarrow SiF_4 + 2H_2O$ (1).[31] The selective glassy matrix removal occurs because the affinity of fluoride to silicon is greater than to oxygen, which makes possible the attack of ionized HF to silanol (silicon-oxygen bonds, SiO_2) presented in the glass ceramic.[31,32] Therefore, the higher the concentration of HF, the greater the glassy matrix removal owing to the higher content of ionized HF available to react with SiO_2.

Different etching times were also evaluated. The results confirmed the second hypothesis as well as higher etching times increased the conditioning ability of HF to EMX, especially for 1% and 2.5% HF concentrations. This was evidenced in the SEM images (Figs. 2 and 3; 1% and 2.5% HF, respectively), in which the etching times of 40, 60, 120, and 20 + 20 seconds promoted a gradual increase on the vitreous phase dissolution/bond strength compared with 20 seconds (Table 1). Probably, as HF stays in

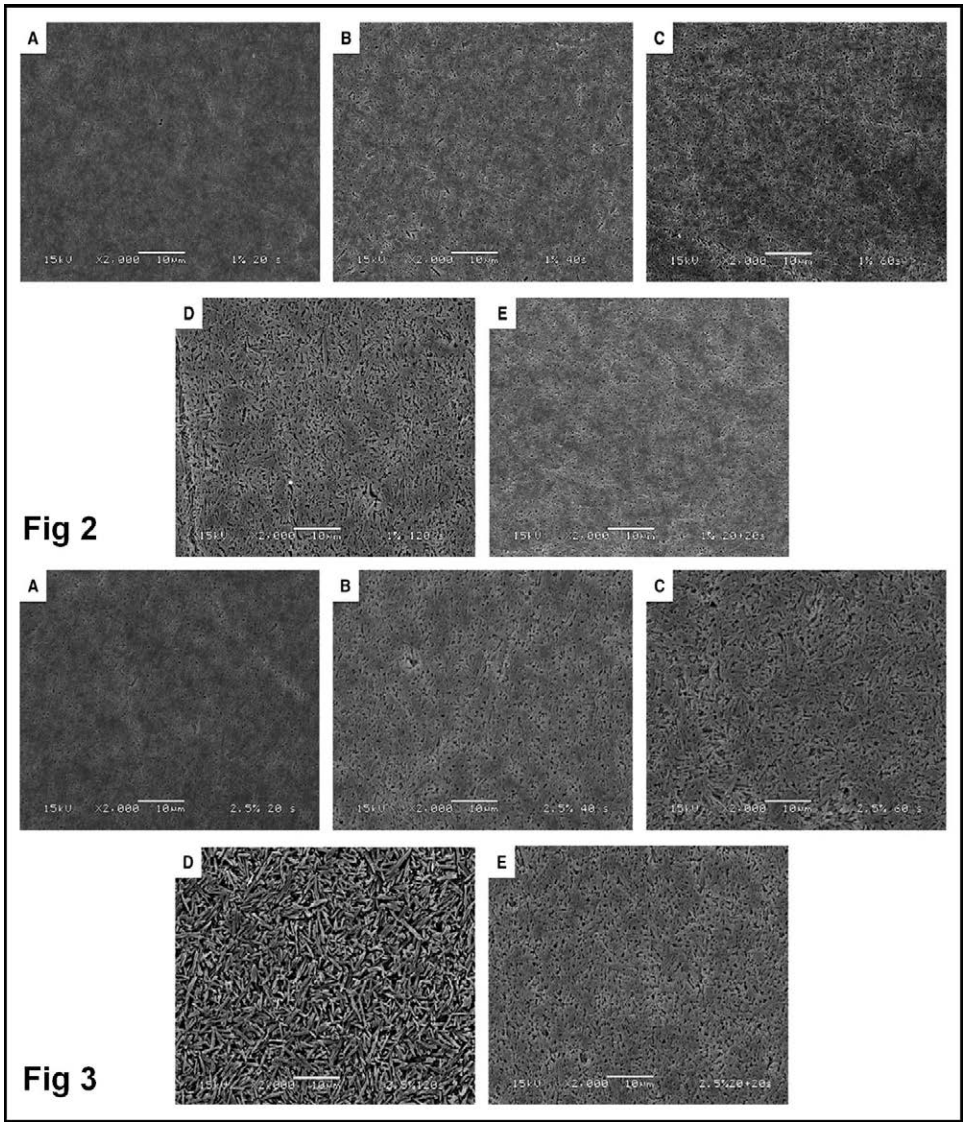

Figure 2. *SEM images (2000× magnification) of etched EMX surfaces made with 1% HF concentration. Different etching patterns were observed for different etching periods. Poor vitreous phase dissolution can be observed in (A) (1%, 20 seconds), (B) (1%, 40 seconds), (C) (1%, 60 seconds), and (E) (1%, 20 + 20 seconds), whereas (D) (1%, 120 seconds) shows slightly greater vitreous phase dissolution than all other etching times.*

Figure 3. *SEM images (2000× magnification) of etched EMX surfaces made with 2.5% HF concentration. Different etching patterns were observed for different etching periods. (A) (2.5%, 20 seconds) and (B) (2.5%, 40 seconds) show poor vitreous phase dissolution, while (C) (2.5%, 60 seconds) shows partial vitreous phase dissolution, exposing lithium disilicate crystals. (D) (2.5%, 120 seconds) shows a greater vitreous phase dissolution/exposure of lithium disilicate crystals than all other etching times. (E) (2.5%, 20 + 20 seconds) shows a higher vitreous phase dissolution compared with 20 seconds only.*

contact for longer periods with the EMX surface, there will be more time available for ionized HF to react with silicon (glassy matrix), thus removing more vitreous phase and, consequently, creating more surface irregularities for the mechanical entanglement with the resin cement by the greater exposure of lithium disilicate crystals. HF etching with 2.5% for 120 seconds was the concentration below 5% that mostly resembled 5% HF applied for 20 seconds. Although the lowest concentration of HF is desired because of the hazardous nature of HF,[31] etching for 120 seconds is time-consuming, which is not efficient for the clinical office.

The results of the present study are not in agreement with those of previous reports, which have found decreased bond strength when a leucite-reinforced glass ceramic was etched with 10% HF concentration for more than 60 seconds[18] or for 90-180 seconds.[33] Such difference reaffirms that the etching effect of HF depends on the composition of the glass ceramic. Since EMX has a lower content of glassy matrix (±30 vol%) than leucite-reinforced glass ceramic (±70 vol%), a lower formation of silicon tetrafluoride (SiF_4) is expected (see reaction 1); SiF_4 is a crystalline residue deposited on the glass ceramic surface[18,30,34] that acts as a physical barrier and may hamper further HF etching.

The increased etching times did not lead to higher μSBS values for 5%, 7.5%, and 10% HF concentrations. It may be claimed that 1) higher HF concentrations do not need extended etching times, as the amount of ionized HF is enough to properly dissolve the glassy matrix of EMX for 20 seconds, and 2) the limited effect of HF concentrations is due

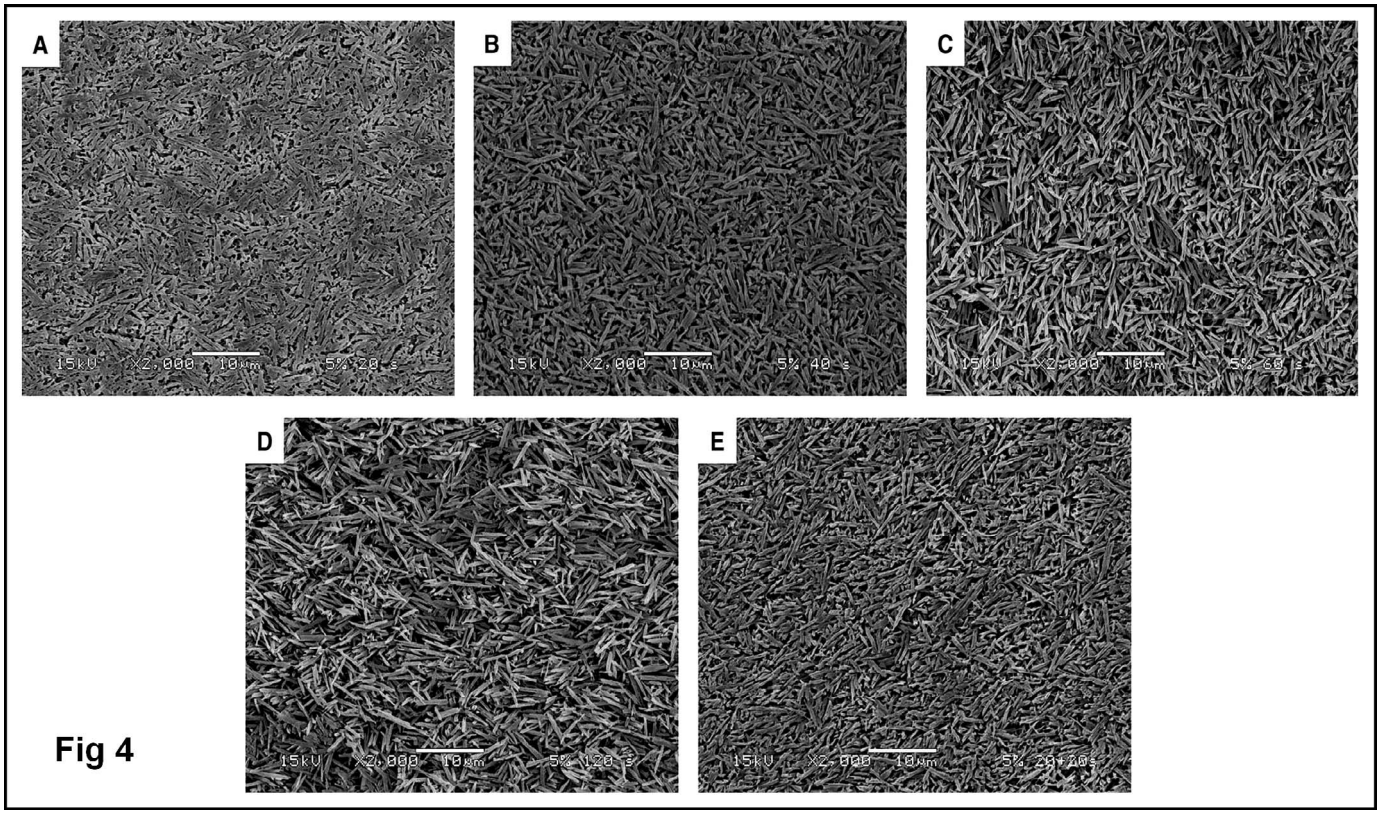

Figure 4. *SEM images (2000× magnification) of etched EMX surfaces made with 5% HF concentration. Different etching patterns can be observed for different etching periods. (B) (5%, 40 seconds), (C) (5%, 60 seconds), and (D) (5%, 120 seconds) show a gradual increase of the vitreous phase dissolution and the exposure of lithium disilicate crystals compared with (A) (5%, 20 seconds), which shows the lowest glassy matrix removal. (E) (5%, 20 + 20) shows a similar etching pattern to (B) (5%).*

to the combination of buffering and dilution of the etchant by the crystalline residue.[18] For 1% and 2.5% HF concentrations, the bond strength was higher when the etching period was extended, which might be explained from the reduced effect of SiF_4 (assumed for the higher content of vitreous phase seen on the EMX surface; Figs. 2 and 3) on the etching capability of HF. Thus, it may be hypothesized that 1% and 2.5% HF concentrations initially form a lower amount of SiF_4 because of the lower content of ionized HF available to react with the silicon. Hence, a lower initial physical barrier is created, which allows HF to continue reacting with the glassy matrix for increased etching times, leading to higher μSBS values.

It was expected that the re-etching would increase the aggressiveness and depth of the glassy matrix dissolution.[18] Furthermore, re-etching was also expected to possibly attenuate the effect of SiF_4, as it is soluble in water[35] and the air/water spray would have removed it from the etched surface before the further new conditioning. However, re-etching provided higher bond strength values for 1% and 2.5% only, which presented statistically similar bond strength values to 40 seconds. Thus, renewing the HF may be done in the case of accidental contaminations of the etched surface, but if the clinician seeks higher bond strength values, re-etching is not necessary.

A slight increase of mixed failures was observed for 5%, 7.5%, and 10% HF concentrations, irrespective of the etching times. The glassy matrix dissolution is higher and the penetration of the resin cement is adequate, although not complete.[4,30] On the other hand, adhesive failures were more frequent for the HF concentrations of 1% and 2.5%, which might point to poor bonding quality for lower HF concentrations, a situation also found by Sundfeld Neto and others.[4]

In summary, the μSBS results and SEM analysis have shown that different etching times and HF concentrations have significant effects on the surface morphology/μSBS of EMX. Adequate etching times and HF concentrations are crucial for optimal bonding properties and clinical performance/longevity. Dental professionals should take care during

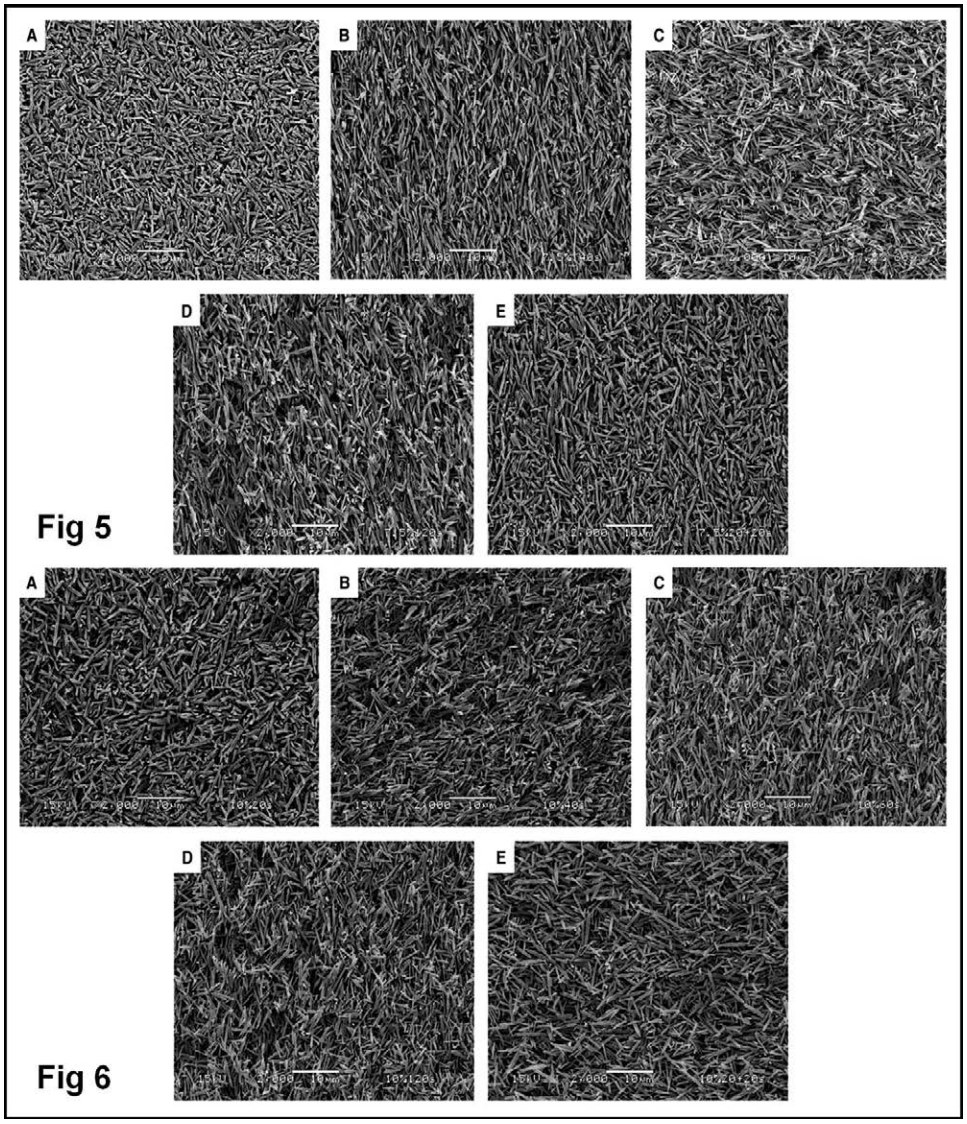

Figure 5. *SEM images (2000× magnification) of etched EMX surfaces made with 7.5% HF concentration. Similar etching patterns (glassy matrix removal and exposure of lithium disilicate crystals) can be observed, regardless of the etching times (A: 20 seconds, B: 40 seconds, C: 60 seconds, D: 120 seconds, and E: 20 + 20 seconds).*

Figure 6. *SEM images (2000× magnification) of etched EMX surfaces made with 10% HF concentration. Similar etching patterns (glassy matrix removal and exposure of lithium disilicate crystals) can be observed, regardless of the etching times (A: 20 seconds, B: 40 seconds, C: 60 seconds, D: 120 seconds, and E: 20 + 20 seconds).*

bonding procedures because HF is a hazardous etchant.[31] Future studies should be carried out to investigate other possible factors, such as viscosities of the resin cement, thermal cycling, and fatigue that may affect the performance of bonded ceramic restorations.

CONCLUSIONS

The effect of HF on the bonding characteristics of lithium disilicate glass ceramic was concentration/time dependent. As the concentration of HF decreased, the etching time had to be increased for greater bond strength. The adequate surface treatment for lithium disilicate glass ceramic was achieved with the HF concentration of 5% applied for 20 seconds, therefore attesting that it is not necessary to use higher concentrations of HF and/or increased etching times.

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Conflict of Interest

The authors 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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Radiopacity and Porosity of Bulk-fill and Conventional Composite Posterior Restorations—Digital X-ray Analysis

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Clinical Relevance

Bulk-fill composites present proper radiodensity when used as posterior restorative materials and allow secondary caries or cervical marginal defects to be easily detected. Furthermore, restorations performed with bulk-fill flowable composites demonstrated a reduced incidence of voids.

SUMMARY

Objectives: To compare radiopacity and porosity as expressed by the presence of voids in restorations carried out using bulk-fill and incremental filling techniques to restore large mesio-occlusal-distal (MOD) cavities.

Methods: Fifty-five molars with MOD preparations were incrementally filled with Filtek Z-350XT (Z350XT) or bulk-fill composite: Filtek Bulk Fill/Z-350XT (FBF/Z350XT), Venus Bulk Fill/Charisma Diamond (VBF/CHA), SDR/Esthet-X HD (SDR/EST-X), Tetric EvoCeram Bulk Fill (TEC). Digital radiographic images (Vistascan scanner) were taken of restored molars and analyzed at the gingival and isthmus floors. Radiodensity measurements were performed using standardized points symmetrically distributed over each region of composite and tooth structure. Three calibrated evaluators visually assessed the presence of voids. Confidence intervals were calculated, and data were analyzed using analysis of variance and χ^2 tests.

Results: TEC and VBF/CHA showed significantly higher radiodensities, while the lowest

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values were observed for FBF/Z350XT and Z350XT. Radiodensity at the cervical regions tended to be greater than that found at the isthmus floor. The lowest incidence of voids was found for VBF/CHA, whereas the incremental insertion technique resulted in the highest rate of voids.

Conclusion: Bulk-fill composite resin demonstrated an adequate level of radiodensity and a reduced presence of voids compared with the incremental filling technique.

INTRODUCTION

Three hundred million direct composite restorations are performed annually in the world, representing one of the most common interventions in the human body.[1] In order to improve the clinical performance of these restorations, different filling techniques have been proposed, and new composites have been developed to reduce polymerization stress and improve the mechanical properties of restorative materials.[2-6] Regarding restorative technique, insertion of a composite using 2-mm oblique increments was advocated for a long time to achieve proper depth of polymerization and reduced polymerization stress.[2-4] However, due to the time-consuming nature of this technique, composites (known as "bulk-fill") that can be light cured through deeper increments while maintaining reduced polymerization stresses were recently introduced to the market.[7] Bulk-fill composites are gaining popularity among clinicians because reducing the time required to restore large cavities has resulted in a simpler restorative procedure.[1,8]

Secondary caries located under the gingival walls of the proximal box is a common cause of failure observed in posterior composite restorations,[9] although this diagnosis is hard to assess clinically. Materials used to restore cavities in posterior teeth must provide enough radiopacity to allow for the diagnosis of secondary caries.[10-12] Moreover, proper radiopacity allows clinicians to diagnose defects in adaptation, the contour of a restoration, contact with adjacent teeth, interfacial gaps, and voids in the restoration.[13,14]

Although most manufacturers state that their materials are radiopaque, there is no clear agreement on the degree of radiodensity for facilitating the detection of caries and defects adjacent to restorations.[14,15] International organizations have recommended procedures for quantifying composite resin radiopacity using an aluminum step as a reference.[16,17] Recent studies using bulk-fill composite resins reported that all composites evaluated demonstrated the minimum requirements demanded by International Organization for Standardization and American National Standards Institute/American Dental Association standards.[17,18] Varying the thicknesses of bulk-fill composite resin also affects the radiopacity,[18] demonstrating that the method used for radiopacity analysis may interfere with the material's performance. Use of a tooth with a standardized cavity preparation is a reasonable alternative for detecting radiolucency around restorations as it allows for the evaluation of the radiodensity of restorative materials in clinically relevant areas, such as the gingival margins of inlay restorations.[14]

The restorative technique is mainly determined by the composite resin used, and when a conventional composite resin is used, the incremental filling technique is recommended.[2,3] The layering methods may affect the adaptability of the composite resin to cavity walls and sequential increments, generating porosities that are expressed by voids.[19] Using a bulk-fill composite resin can eliminate this flaw by improving the homogeneity of material distribution inside the bulk restoration. The increased flowability of most bulk-fill flowable composites may also influence the presence of cavity adaptation and voids. On the other hand, past bulk-fill composites demonstrated no improvement on adaptability.

No prior studies, according to the authors' knowledge, have analyzed the radiopacity and porosity achieved using flowable or regular viscosity bulk-fill composites compared with the incremental filling technique in posterior restorations. Therefore, the aim of this study was to evaluate the radiodensity of conventional composite resin placed incrementally in oblique layers, a high viscosity bulk-fill placed in a single bulk layer, and flowable composites placed in two layers (at the gingival walls and isthmus floor), as well as the detection of voids in the restoration. The null hypotheses were that bulk-fill composite has the same radiopacity performance as the conventional composite resin and that the presence of voids is not affected by the composite and insertion technique.

METHODS AND MATERIALS

Fifty extracted, sound, caries-free, human molars were used in this study. The teeth were selected considering an intercuspal width within a maximum deviation of 10% from the mean calculated among the teeth selected (ranging from 4.8 to 6.0 mm). The

Table 1: *Composites Used in This Study*

Composite	Manufacturer	Lot No.	Composition[a]
Filtek Z-350XT	3M ESPE (St Paul, MN, USA)	N491808	Organic matrix: Bis-GMA, Bis-EMA, UDMA, TEGDMA
			Filler type: Silica and zirconia nanofillers, agglomerated zirconia-silica nanoclusters
			Filler content: 82 wt%/ 60 vol%
Tetric EvoCeram Bulk Fill	Ivoclar Vivadent, (Schaan, Liechtenstein)	R49602	Organic matrix: UDMA, Bis-GMA
			Filler type: Barium glass, ytterbium trifluoride, mixed oxide prepolymer
			Filler content: 79 wt%/ 61 vol%
Venus Bulk Fill	Heraeus-Kuzer (Hanau, Germany)	010103	Organic matrix: UDMA, TEGDMA
			Filler type: Barium glass, ytterbium trifluoride, silicon dioxide
			Filler content: 65 wt%/ 38 vol%
Charisma Diamond	Heraeus-Kuzer (Hanau, Germany)	010049	Organic matrix: TCD-DI-HEA, UDMA
			Filler type: Bariumm, aluminium, fluoride glass
			Filler content: 81 wt%/ 64 vol%
SDR	Dentsply (Konstanz, Germany)	760231E	Organic matrix: Modified UDMA, dimethacrylate and difunctional diluents
			Filler type: Barium and strontium alumino-fluoro-silicate glasses
			Filler content: 68 wt%/ 44 vol%
Esthet-X HD	Dentsply (Konstanz, Germany)	897887F	Organic matrix: Bis-GMA adduct, EBPADMA, TEGDMA.
			Filler type: Ba-F-Al-B-Si-glass, silica.
			Filler content: 76 wt%/ 60 wt%
Filtek Bulk Fill	3M ESPE (St Paul, MN, USA)	N491808	Organic matrix: UDMA, BISGMA, EBPADMA, procrylate resin
			Filler type: Silane-treated ceramic and YbF
			Filler content: 64 wt%/ 42.5%

[a] Composition as given by manufacturers.
Bis-EMA, ethoxylated bisphenol-A dimethacrylate; Bis-GMA, bisphenol A diglycidyl dimethacrylate; EBPADMA: ethoxylated bisphenol-A dimethacrylate; TCD-DI-HEA, 2-propenoic acid, (octahydro-4, 7-methano-1H-indene-5-diyl) bis-methyleneiminocarbonyloxy-2, 1-ethanediyl) ester; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; YbF, ytterbium trifluoride.

roots were embedded in a polystyrene resin (Cristal, Piracicaba, Brazil) up to 2 mm below the cemento-enamel junction. The teeth received a Class II mesio-occlusal-distal (MOD) preparation that was 4/5 of the intercuspal width (buccal-lingual dimension = 5.1±0.8 mm), 4 mm deep in the occlusal box, and 1 mm deep in the proximal box using a diamond bur (#3146 diamond bur, Microdont, São Paulo, Brazil) performed using a high-speed handpiece and copious air-water spray. The handpiece was attached to a cavity preparation machine allowing for the control of the bur movement.[20]

The composites used to restore the cavities are described in Table 1. The cavities were randomly allocated to be restored using one of the techniques/ composites described in Table 2 (n=10). The same self-etching adhesive system (Clear Fill SE Bond, Kuraray, Tokyo, Japan) was used for all groups. The composites were light-activated using a quartz-tungsten-halogen unit (800mW/cm2; Optilux 501, Kerr Manufacturing Co, Orange, CA, USA), according to the manufacturer's instructions: 40 seconds for Filtek Bulk Fill and 20 seconds for all other composites. The Tetric EvoCeram Bulk Fill (TEC) was used for restoring both dentin and enamel in a bulk increment. The flowable bulk-fill composite groups were filled in 2 horizontal layers, the first increment filled the dentin depth (3.5-4.0 mm in depth) using bulk-fill flowable composite; then, the enamel layer (1.0-1.5 mm in depth) was filled using a regular conventional composite from the same manufacturer (Table 2). Cavities in the control group were filled incrementally using six oblique increments with a conventional resin composite. A Teflon matrix was made to standardize each composite resin increment before insertion into the cavity.[2] A device was created to simulate adjacent premolars

Table 2: *Descriptions of Groups*

Group	Technique	Procedure
Z350XT	Incremental	Z350XT was inserted and light cured in increments up to 2mm until the entire cavity was filled.
TEC	Bulk fill 1-increment	Tetric EvoCeram was inserted in a single increment, filling the entire cavity.
VBF/CHA	Bulk fill 2-increment	1. The flowable composite, Venus Bulk Fill, was placed in a single increment to fill the proximal box and the region corresponding to dentin tissue in the occlusal box. 2. The restoration was completed using the composite with regular viscosity, Charisma Diamond.
SDR/EST-X	Bulk fill 2-increment	1. The flowable composite, SDR, was placed in a single increment to fill the proximal box and the region corresponding to dentin tissue in the occlusal box. 2. The restoration was completed using the composite with regular viscosity, Esthet-X HD.
FBF/Z350XT	Bulk fill 2-increment	1. The flowable composite, Filtek Bulk fill flow, was placed in a single increment to fill the proximal box and the region corresponding to dentin tissue in the occlusal box. 2. The restoration was completed using the composite with regular viscosity, Z350XT.

Abbreviations: FBF/Z350XT, Filtek Bulk Fill/Z-350XT; SDR/EST-X, SDR/Esthet-X HD; TEC, Tetric EvoCeram Bulk Fill; VBF/CHA, Venus Bulk Fill/Charisma Diamond; Z350XT, Filtek Z-350XT.

and molars to allow for interproximal contact during restoration.[8]

Radiopacity Measurement m=Method

The samples were positioned over a phosphor plate, and the radiographic exposure was performed using the Timex 70 E (Gnatus, Ribeirão Preto, Brazil), exposing the specimens for 0.25s at 70 kV and 7.0 mA. The focal spot to object distance was set at 50 cm. Three exposures were performed for each sample. The radiographs were transferred from the phosphor plate to a computer using a Vistascan scanner (Vistascan, Durr Dental, Bietigheim-Bissingen, Germany). The radiodensity (in pixels) of the samples was determined using the resident software provided by the manufacturer. Five measurement lines were defined: two at the cervical margins, for both the mesial and distal surfaces, and three points at the isthmus floor.[14] All of the measurements were taken symmetrically on the axial plane of teeth and restorative surfaces (Figure 1). Each digital image had radiodensity measured at each measurement line (Figure 1), immediately after scanning, without any modification to contrast or brightness. The mouse cursor was positioned under this measurement line on the tooth structure or restorative material to obtain the radiodensity values for each sample, and the means of readings were calculated and used in further data analysis.[14]

The digital radiographs were analyzed by three calibrated operators to visually detect the porosity expressed by bubbles and voids. Differences in radiodensity values between the tooth structure and composite restoration were calculated for all measurement lines at each region (cervical and isthmus floor). The absence or presence of voids was recorded by analyzing the entire bulk of restorations. Further, the digital radiographs were transferred to ImageJ software (Java-based image processing and analysis software developed at the National Institutes of Health, Bethesda, MD, USA) to measure the size of each detected void (Figure 2).

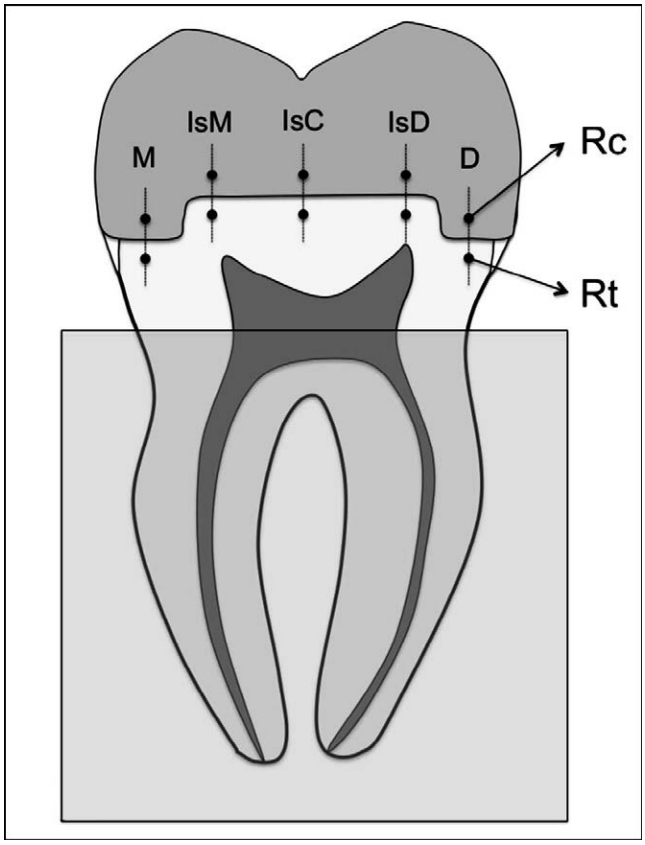

Figure 1. *Location of the cervical (M, D) and isthmus floor measurements (IsM, IsC, and IsD), radiodensity of composite resin restoration (Rc), and radiodensity of tooth (Rt).*

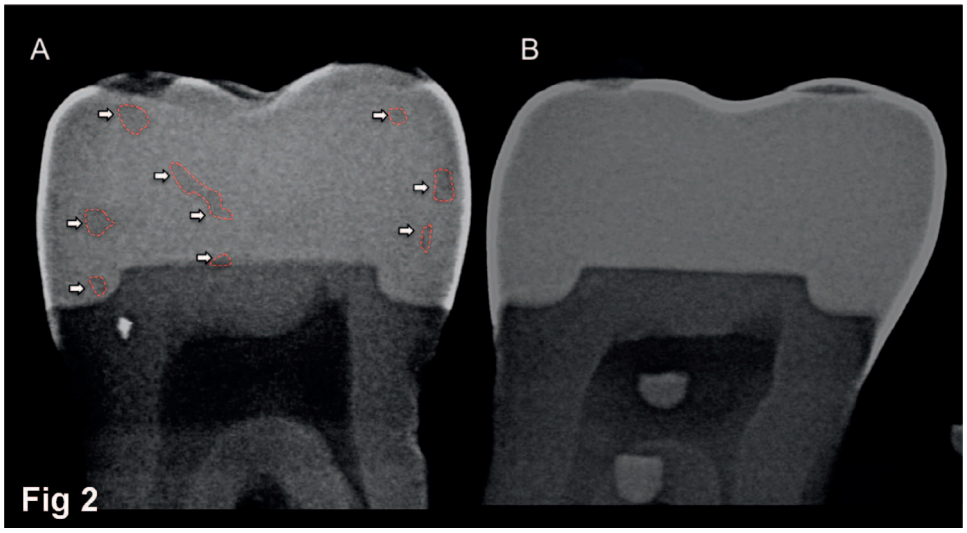

Figure 2. *Amplified radiographs of restorative techniques to detect and measure the voids. (A): tooth with several voids. (B): tooth without voids.*

Statistical Analysis

Data of differences in radiodensity were analyzed for normal distribution and homoscedasticity using the Shapiro-Wilk test and Levene test, respectively. In the initial analysis, the goal was to compare the composites used in this study; therefore, data were analyzed by one-way analysis of variance (ANOVA). In the second analysis, the effect of the region where the measurement was carried out was included in the statistical analysis. Thus, data were analyzed using two-way ANOVA (five composites × five regions). The frequency of voids for each group was calculated, and the effect of the restorative procedure on this frequency was analyzed using the χ^2 test. For all analyses, the 95% confidence interval for experimental conditions was calculated to allow for comparisons.

RESULTS

Figure 3 shows representative radiographs for each group. Significant differences in radiodensity were found among the restorative materials ($p<0.001$). A comparison among the groups is provided in Figure 4. All of the composites tested showed a positive difference in radiodensity compared with sound tooth structure, demonstrating a higher radiopacity than dentin. TEC and Venus Bulk Fill/Charisma Diamond (VBF/CHA) showed significantly higher radiodensities than did the other materials, while the lowest values were observed for FBF/Z350XT and Z350XT.

Significant differences in radiodensity were found among regions ($p<0.001$) and among the composites ($p<0.001$); however, the interaction between these factors was not significant ($p=0.763$). The difference for values of radiodensity among the regions for each restorative procedure are presented in Figure 5. Higher differences for radiodensity were observed in the proximal regions (mesial and distal). No difference was found in the three regions of the isthmus floor, despite the tendency for reduced differences in radiodensity at the center of the isthmus. A representative illustration was plotted relating the range of colors to the averages of differences in radiodensity observed at each point of measurement (Figure 6).

The χ^2 test demonstrated that the restorative procedure affected the presence of voids ($p=0.004$). The number of voids and the average of the size for all voids per group are shown in Table 3.

The void distribution is presented in Figure 7. VBF/CHA demonstrated the lowest incidence of voids, whereas the highest occurrence was observed for Z350XT, followed by TEC.

DISCUSSION

The present study confirmed that bulk-fill composite resins have adequate radiopacity for posterior restorations and that flowable bulk-fill composite reduces the porosity in composite resin restorations. All of the bulk-fill and conventional composite resins showed higher radiopacity values compared with enamel and dentin. The method used for radiopacity analysis can involve the use of the composite resin together with the aluminum step wedge and a tooth specimen positioned over the radiograph sensor.[14,17,18] In this study, tooth selection and cavity preparation were standardized because detection of radiopacity around restorations also depends on the density and thickness of remaining tooth struc-

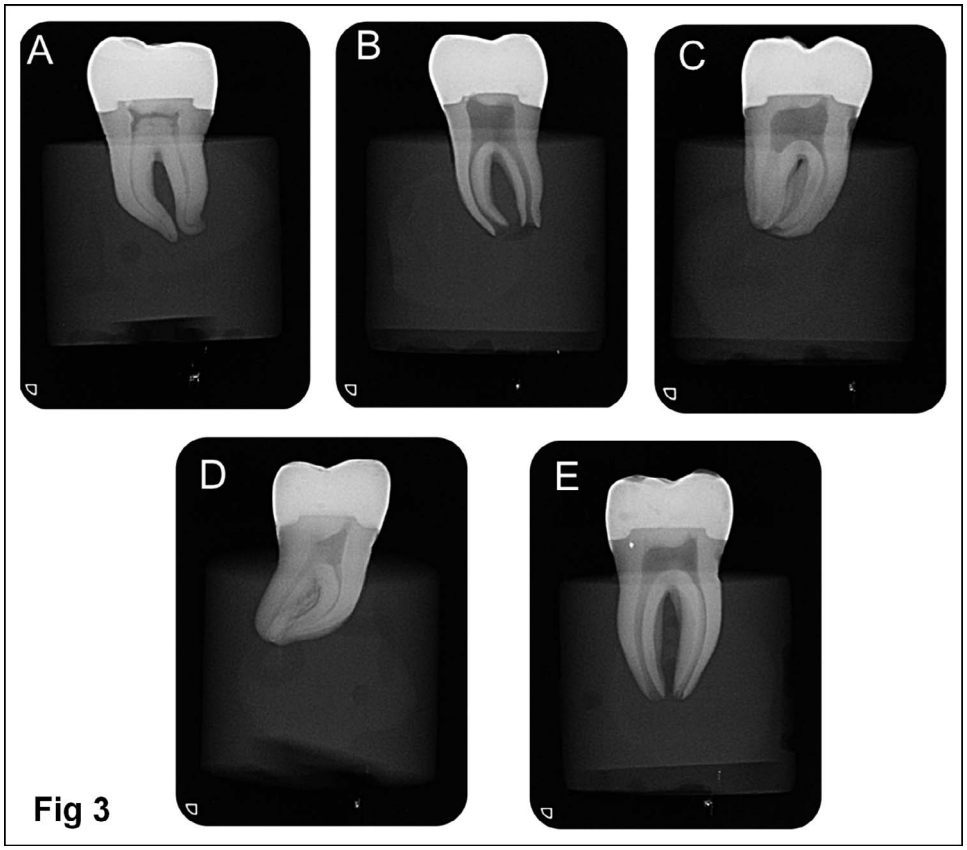

Figure 3. *Radiographs of restorative techniques. (A): SDR/EST-X. (B): TEC. (C): VBF/CHA. (D):, FBF/Z350. (E): Z350.*

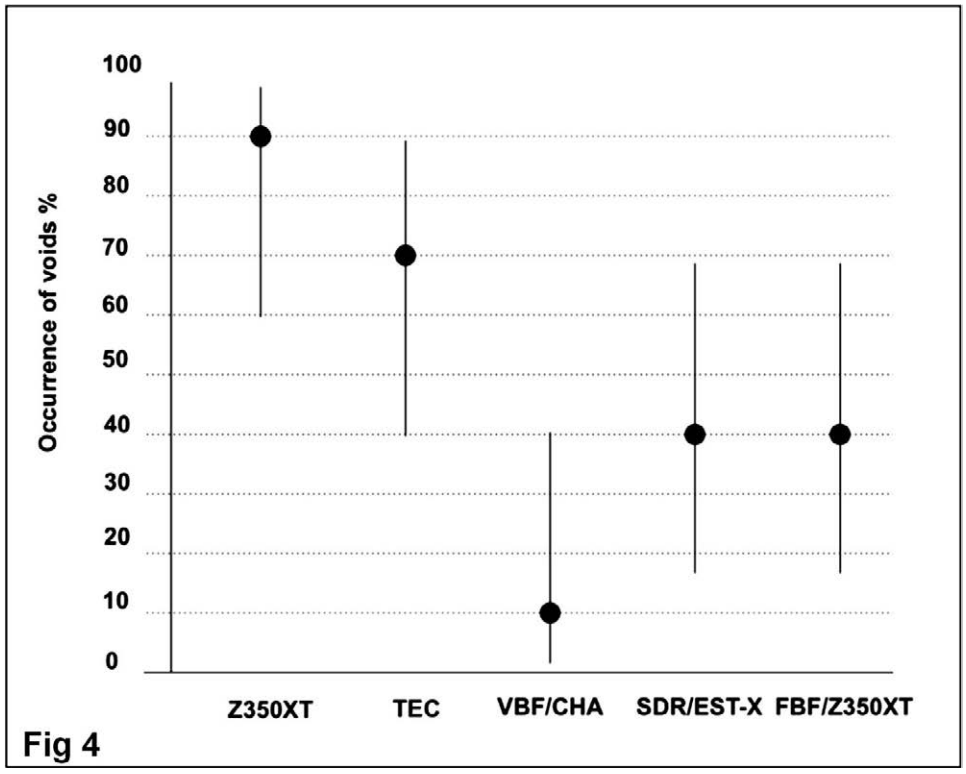

Figure 4. *The 95% confidence interval of difference for radiodensity between the composites and the tooth structure according to the restorative procedures.*

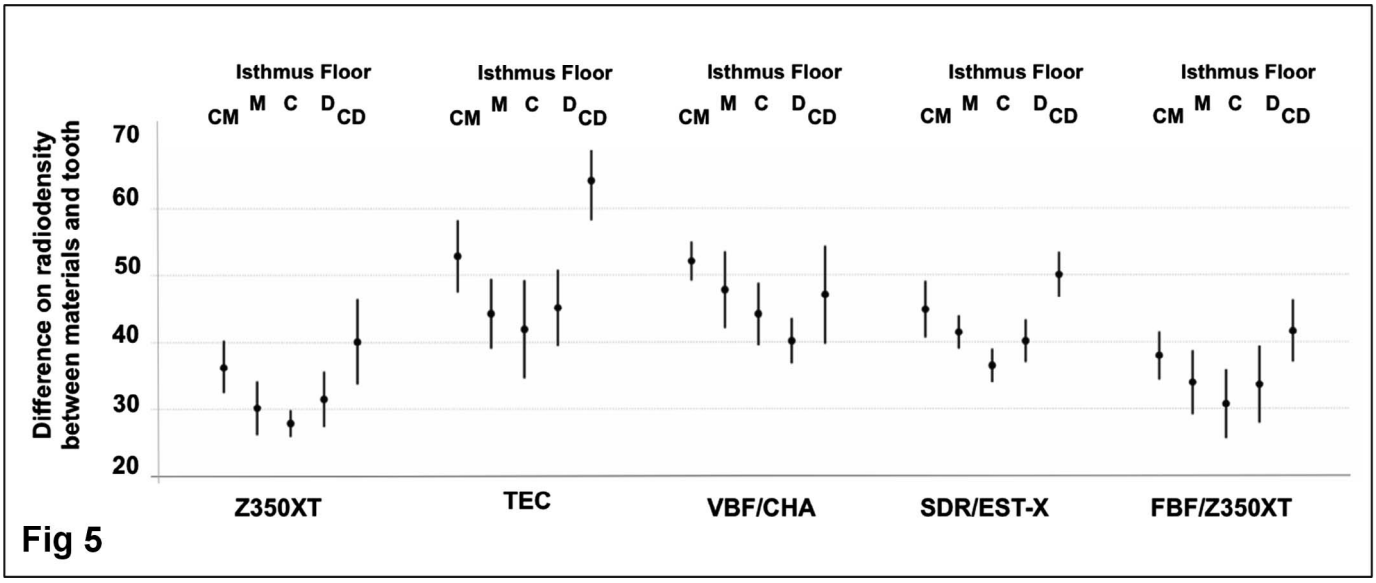

Figure 5. *The 95% confidence interval of difference for radiodensity between the composites and the tooth structure according to restorative procedures and region of measurement.*

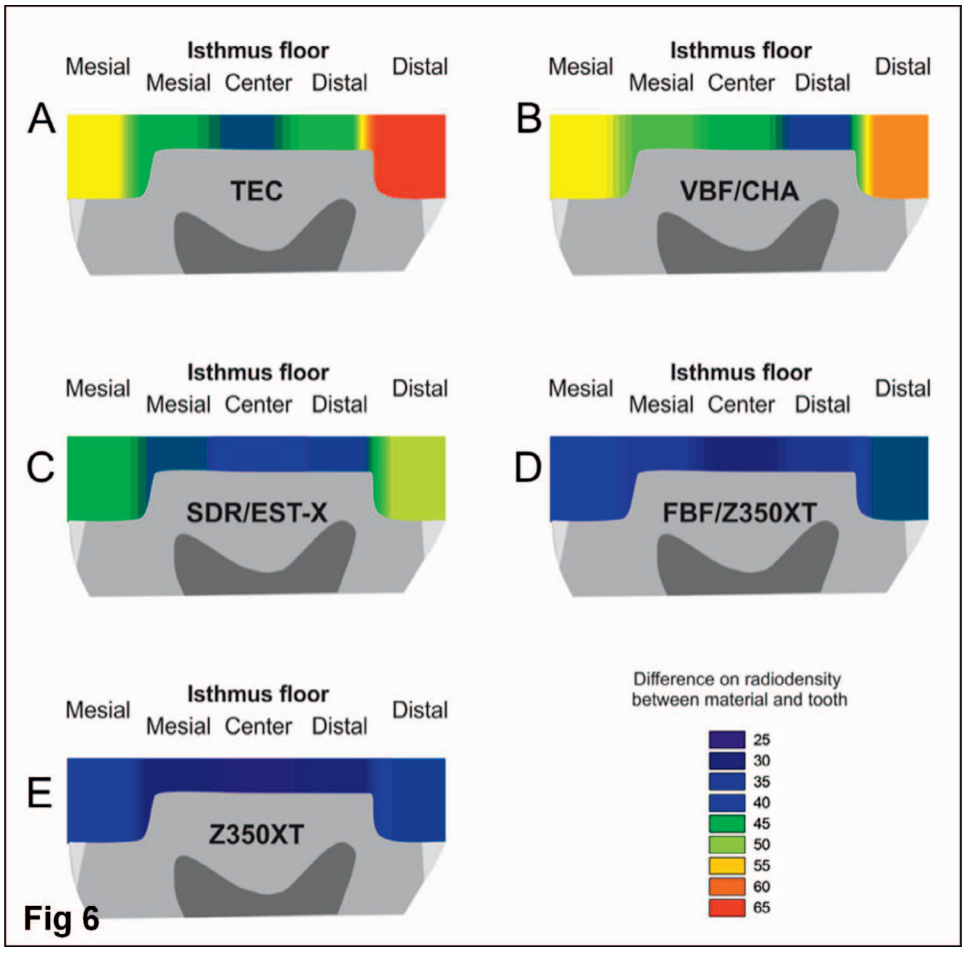

Figure 6. *The representative illustration associating colors with the average of differences on radiodensity. In general, a higher difference was observed in the proximal box.*

Table 3: *Quantitative Analysis of the Void Presence for All Groups*

Group	Total Voids per Group (10 teeth)	Average No. of Voids per Tooth	Average Void size (mm) per Group
Z350XT	44	4.4	0.40
TEC	18	1.8	0.30
VBF/CHA	7	0.7	0.35
SDR/EST-X	12	1.2	0.34
FBF/Z350XT	14	1.4	0.33

Abbreviations: FBF/Z350XT, Filtek Bulk Fill/Z-350XT; SDR/EST-X, SDR/Esthet-X HD; TEC, Tetric EvoCeram Bulk Fill; VBF/CHA, Venus Bulk Fill/Charisma Diamond; Z350XT, Filtek Z-350XT.

ture.[14,21] An important factor in the radiodensity analysis method employed in this study was the systematic evaluation of the restoration placed in the prepared tooth, which allowed clinicians to analyze material radiodensity where that property is clinically critical, at the cervical margin of restorations.[14] The absence of appropriate marginal adaptation is responsible for several problems observed in posterior composite restorations, mainly when the failure occurs in the proximal area.[22] Thus, a composite with a radiopacity lower than that of enamel is not suitable for use as the first increment when an incremental technique is carried out.[17,18] The adequate radiopacity of this increment facilitates the visualization of any defect at the tooth/restoration margin.[15] The results of this study showed that the radiodensity of a material is greater at the cervical region, and that composite resin voids are easier to detect because the remaining tooth region is minimized in this region due to the anatomy of the tooth.[13,20]

The inherent radiopacity of a composite is mainly related to the chemical composition and content of the filler.[17,18,23] Considering that the thickness of composites was similar for all specimens, the difference in the radiodensity detected in this study is strictly related to the inorganic phase of the composites evaluated.[20] This aspect seems to be the most important factor affecting radiopacity.[14,24] The radiopacity of a material tends to increase with a higher filler content and when elements with high atomic numbers are present in the filler particles.[18,25,26] Flowable composites usually contain a reduced filler content since that composite phase is responsible for increasing the viscosity of the material. Therefore, Filtek Bulk Fill and SDR Bulk Fill showed lower radiopacity values compared with the other materials evaluated due to their lower weight and volume percentages of filler.[18] On the other hand, despite the reduced filler content of the flowable composite, Venus Bulk Fill, this composite presented a higher radiopacity compared with other

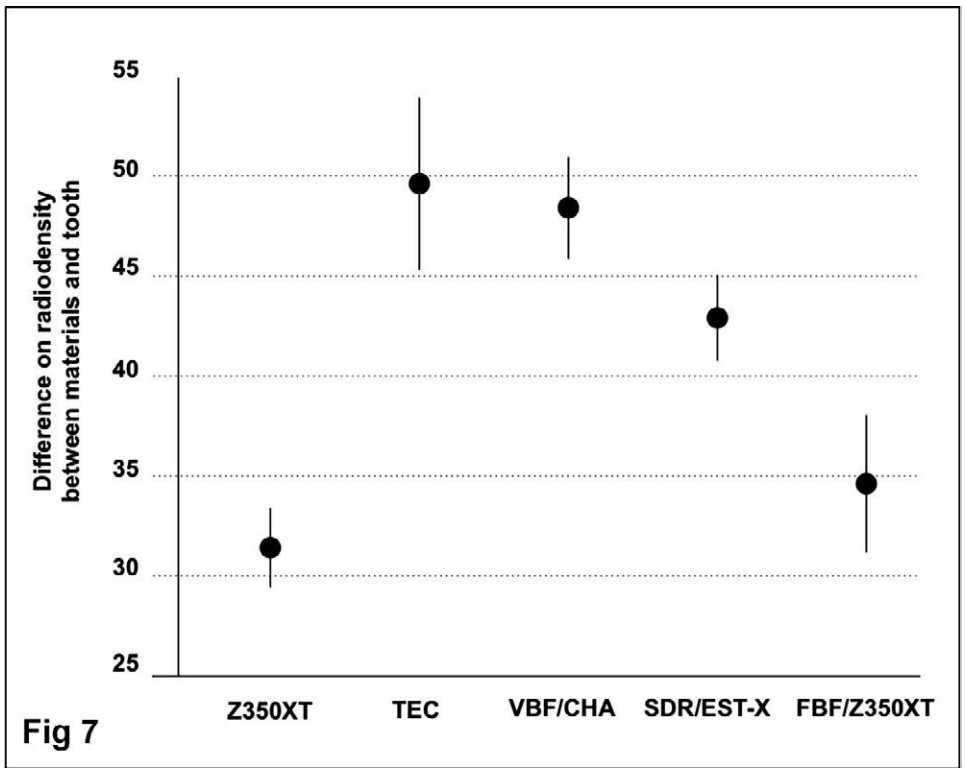

Figure 7. *The 95% confidence interval for occurrence of voids according to restorative procedures.*

tested flowable bulk-fill composites. This finding can be explained by the type of fillers used in this composite, which is similar to those employed in TEC. Those composite materials include radiopaque fillers that are composed of elements with higher atomic numbers (barium and ytterbium trifluoride glasses), resulting in significantly higher radiopacity values compared with the other flowable composites.[17,18]

However, using a bulk-fill flowable composite allows the clinician to fill the entire volume of a cavity corresponding to dentin tissue, while only a single increment of the regular composite is required to complete the restoration. Thus, when considering that the increment contacting the cavity floor and proximal margins is a flowable composite, the radiopacity of this material seems to be more important. Filtek Bulk Fill and Filtek Z-350XT (Filtek Z-350XT) had similar radiodensity despite the reduced filler content of the former. Filtek Bulk Fill compensates for this lower filler content with the presence of elements with higher atomic numbers (ytterbium trifluoride glass). Thus, the first hypothesis of this study was rejected, since the materials evaluated demonstrated differences regarding radiopacity.

Few studies have analyzed the internal adaptation for bulk-fill composites.[11] The porosity generated by the incorporation of bubbles and voids into the composite volume and a composite's failure to adapt to the cavity walls may influence its biomechanical performance. The location of the voids for bulk-fill composites tended to concentrate at the gingival/axial angles; few voids were observed between the flowable composite layer and conventional composite layer used. However, the voids clearly coincided with the region between the increments for Z350XT, thereby rejecting the second hypothesis of study. The insertion of several increments may result in the incorporation of voids due to the difficulty in adequately condensing the increments. A prior study using the same materials demonstrated that the incremental filling technique resulted in lower fracture resistance compared with bulk-fill composite Class II restorations. Interestingly, the fracture lines observed for Filtek Z-350 XT samples coincided with the area of transition between increments, suggesting that the presence of voids can reduce the cohesive strength of the restoration. The necessity of inserting increments with a maximum thickness of 2.0 mm when a regular composite is used, a technique used to allow for adequate polymerization of each increment, indicates that a large number of increments are required to fill large cavities like those used in this study. Voids located at the pulp floor or gingival angles may be caused by the fluid movement that results in postoperative sensitivity. Incorporation of voids inside the bulk volume of the composite was not significant, although this occurrence needs to be carefully analyzed. It may be caused by incorrect insertion of the flowable material or by voids incorporated inside the composite during the manufacturing process.

Although the method used for analyzing the presence of voids is clinically relevant, it cannot detect the volume of the voids. Future microcomputed tomography studies should be performed to detect the void location and volume and to correlate this occurrence with the mechanical performance of the bulk and incremental filling technique.

This study demonstrated that bulk-fill composites have sufficient radiodensity to facilitate detection of secondary caries in marginal defects located at the proximal areas. Additionally, the findings of the present study showed that bulk-fill flowable composites have improved adaptation to the cavity walls, which was demonstrated by a reduced incidence of voids. Thus, bulk-fill composite seems to be a viable option as a direct restorative material to simplify the procedure as it shows proper radiopacity and cavity adaptation.

CONCLUSION

All composite resins demonstrated a positive variation in radiodensity compared with the dental structure. In general, bulk-fill composites demonstrated an adequate level of radiodensity, although the lowest values were observed for Filtek Bulk Fill. The incremental filling technique resulted in a higher incidence of voids, whereas the association of Venus Bulk-fill and Charisma demonstrated significantly fewer voids.

Acknowledgements

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee in Human Research at Federal University of Uberlândia. The approval code for this study is #721985.

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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Microtensile Bond Strength of Resin-Modified Glass Ionomer Cement to Sound and Artificial Caries–Affected Root Dentin With Different Conditioning

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Clinical Relevance

Both self conditioner and cavity conditioner have the potential to improve the bond strength and durability of resin-modified glass ionomer cement (RM-GIC) to sound and carious root dentin; however, ethylenediamine tetraacetic acid conditioning with RM-GIC to root dentin should be avoided.

SUMMARY

In this laboratory study, the microtensile bond strengths (μTBS) of resin-modified glass ionomer cement (RM-GIC) to sound and artificial caries–affected bovine root dentin (ACAD) using three different conditioning agents were evaluated after 24 hours and three months. The fractured interface was examined with a scanning electron microscope (SEM). Specimens were created on bovine root dentin that was embedded in epoxy resin. For the ACAD specimens, artificial carious lesions were created. The RM-GIC (Fuji II LC) was applied either directly (no treatment), after application of self conditioner, cavity conditioner, or 17% ethylenediamine tetraacetic acid (EDTA) applied for 60 seconds, on sound dentin and ACAD, then light cured. They were stored in artificial saliva for 24 hours or three months. Following this, the specimens were cut into sticks for the μTBS test, and the failure mode of the debonded

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specimens was examined by using SEM. Pretest failures were excluded from the statistical analysis of the μTBS values because of their high incidence in some groups. Results showed that the μTBS values were significantly affected by the dentin substrate as well as the conditioning agent. Self conditioner provided the highest and most stable μTBS values, while cavity conditioner showed stable μTBS values on sound dentin. Both self conditioner and cavity conditioner had significantly higher μTBS values than the no treatment groups. EDTA conditioning reduced the μTBS after three months to sound dentin, while it showed 100% pretest failure with ACAD for both storage periods.

INTRODUCTION

Root surface caries is becoming a common clinical phenomenon because of the aging population and increasing number of elderly people maintaining their teeth. This presents new challenges from a restorative perspective, with root surface caries being found to be one of the main risk factors of tooth loss in older adults.[1] Previous epidemiologic studies have shown that the incidence of root caries increases with age.[2,3] In Japan, for example, Imazato and others[4] showed an incidence of root caries of 53.3% in a selected sample of 287 adults older than 60 years.

Resin-modified glass ionomer cement (RM-GIC) was introduced in 1988 to improve the mechanical and esthetic properties of the conventional GICs by the addition of hydrophilic monomers such as 2-hydroxyethyl methacrylate (HEMA) as well as other resins and photoinitiators to the fluoroaluminosilicate glass and polyacrylic acid in the conventional glass ionomer cement.[5,6] Its lower sensitivity to moisture as well as fluoride release made it a successful option for the restoration of root caries lesions, especially in cases adjacent to gingival tissues, which makes complete isolation and access for material placement difficult.

Bonding of the RM-GIC to tooth structure occurs via two mechanisms: chemical bonding between anions of polyalkenoic acid chains and calcium ions in hydroxyapatite[7] and micromechanical adhesion limited to retention provided by the intrinsic surface roughness of dentin and porosity created by the RM-GIC's self-etching characteristics.[8] Several conditioning agents have been evaluated with enamel and coronal dentin.

Caries-affected dentin (CAD) is one of the most relevant substrates in clinical practice. For conservative purposes, CAD should be retained and thus become the bonding substrate. Unlike caries-infected dentin, CAD has much fewer bacteria and can be remineralized[9,10] because of the presence of cross-linked collagen, a key to remineralization.[11] However, CAD has a higher degree of porosity due to mineral loss, greater water content, and typically a thicker smear layer when cut.[12] Previous studies showed lower bond strength values to CAD than to intact dentin with RM-GIC,[13,14] conventional glass ionomer cement,[14] and resin composite materials.[15]

To avoid the great variability in the CAD morphology and also to obtain a flat and uniform surface, an artificial caries–affected model (ACAD) was made in an attempt to simulate natural caries–affected dentin (NCAD). The properties of the ACAD showed a similarity to NCAD in nanohardness and mineral density of the superficial layer (approximately 150 μm). Similar bond strength values were obtained in NCAD and ACAD with a two-step self-etch adhesive.[16]

There is little information about the bond strength of RM-GIC to ACAD; therefore, the aim of this study was to evaluate the microtensile bond strength (μTBS) to sound and artificial caries–affected root dentin using different conditioning materials after storage in artificial saliva for 24 hours and three months. The null hypotheses of this study were that there are no significant differences in μTBS among different conditioning, dentin substrates, and storage periods.

METHODS AND MATERIALS

Forty bovine teeth were collected and stored frozen, pulp tissues were removed, and root surfaces were cleaned with periodontal curettes prior to experimental procedures. A low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) was used to separate the crown and the apical part of the root under water cooling and then discarded. The remaining root portion was cut along the longitudinal axis and transversally to obtain four dentin blocks from the cervical and middle parts of the root, and each block was then embedded in epoxy resin (Epoxicure2, Buehler) using a cylindrical mold. After curing of the resin, the surface was manually wet polished with 600-grit SiC paper to expose flat root-dentin surfaces. The specimens were divided into two main groups: sound dentin and ACAD. For the sound dentin group, the smear layer was standardized by grinding with 600-grit SiC paper for five

Table 1: *Restorative and Conditioning Materials Used in the Study, Manufacturer, Batch Number, Composition, and Method of Application*

Brand Name and Manufacturer	Batch Number	Chemical Composition	Method of Application	Code
Cavity conditioner (GC Corp, Tokyo, Japan)	1508101	77% distilled water, 20% polyacrylic acid, 3% aluminum chloride hydrate pH 1.9	Applied to the surface for 10 seconds Rinsed thoroughly with water and dried without desiccation	CC
Self conditioner (GC Corp, Tokyo, Japan)	1411111	20%-30% HEMA, 5%- 10% 4-META, 30%-35% distilled water, 28%-40% ethanol pH 1.8	Applied to the surface and left undisturbed for 10 seconds Air dried for 5 seconds	SC
EDTA (Dojindo Molecular Technologies, Japan)		0.5 M 2NA(EDTA·2Na) in distilled water pH 7	Applied to the surface for 60 seconds Washed with water for 10 seconds and air dried gently	EDTA
Fuji II LC capsule shade A2 (GC Corp, Tokyo, Japan)	1505151	Fluoro-alumino-silicate glass, polyacrylic acid, HEMA, urethane dimethacrylate, camphorquinone, water	Capsule mixed for 10 seconds and applied on dentin surface, then light cured for 20 seconds	

Abbreviations: EDTA, ethylenediamine tetraacetic acid; HEMA, 2-hydroxyethylmethacrylate; 4-META, 4-methacryloxyethyltrimellitate anhydride.

seconds. For the ACAD group, each specimen was immersed in 15 mL of demineralizing solution (1.5 mM $CaCl_2$, 0.9 mM KH_2PO_4, 50 mM acetic acid, 0.02% of NaN_3 at pH 4.5)[16] at 37°C for 60 hours. Following this, the specimens were rinsed thoroughly with deionized water.

The demineralized surface was ground with 600-grit SiC paper for five seconds to create a demineralized dentin surface with a smear layer, which was checked by optical coherence tomography (Santec OCT-2000, Santec Co, Komaki, Japan) to ensure a standardized demineralized layer approximately 150-μm deep.

Specimen Preparation

The sound and ACAD root dentin surfaces were conditioned with either cavity conditioner (GC Corp, Tokyo, Japan), self conditioner (GC Corp), or 0.5 M ethylenediamine tetraacetic acid (EDTA). For the cavity and self conditioner subgroups, each was applied to the dentin surface according to the manufacturer's instructions described in Table 1. EDTA was prepared by dissolving 2NA(EDTA·2Na) (Dojindo Molecular Technologies, Kumamoto, Japan) in distilled water to obtain an EDTA solution of concentration 0.5 mol/L at pH 7.0. It was applied as a conditioner for 60 seconds and then washed with an air-water syringe for 10 seconds and gently air dried. The dentin surfaces without conditioning were left as a control group. Following this, the Fuji II LC capsule (RM-GIC) was mixed using a GC Capsule Mixer CM-II (GC Corp) for 10 seconds, applied to the dentin surface in a cylindrical mold 2-mm high, and then light cured for 20 seconds using a halogen light curing unit (Optilux 501, Kerr Corp, Orange, CA, USA) at 600 mW/cm^2 output. The bonded specimens were stored separately in artificial saliva at 37°C for 24 hours and three months. Each week, the artificial saliva was replaced with freshly prepared solution at room temperature during the three-month storage period.

Measurement of μTBS

After each storage period, each bonded specimen was longitudinally sectioned in two directions perpendicular to each other across the bonded interface to obtain sticks of approximately 1×1 mm for the μTBS test. Digital calipers (Mitutoyo Corp, Kawasaki, Japan) were used to check the cross-sectional area of the sticks. A total of 20 sticks for each subgroup were tested. Each stick was fixed to the test jig with a cyanoacrylate adhesive (Zapit, Dental Ventures of American, Anaheim Hills, CA, USA) and stressed in tension using a universal testing machine (EZ-Test, Shimadzu, Kyoto, Japan) at a cross-head speed of 1 mm/min until failure. The procedures of specimen preparation for the μTBS test are shown briefly in Figure 1.

When pretest failures occurred during cutting of the bonded specimens, these specimens were excluded from the calculation of the μTBS values. However, the discarded specimens were counted, and the survival rate was calculated for each group. Normal distribution of μTBS strength data was assumed after Shapiro-Wilk test. Bond strength values were analyzed by three-way analysis of variance (ANOVA) with repeated measures to determine the

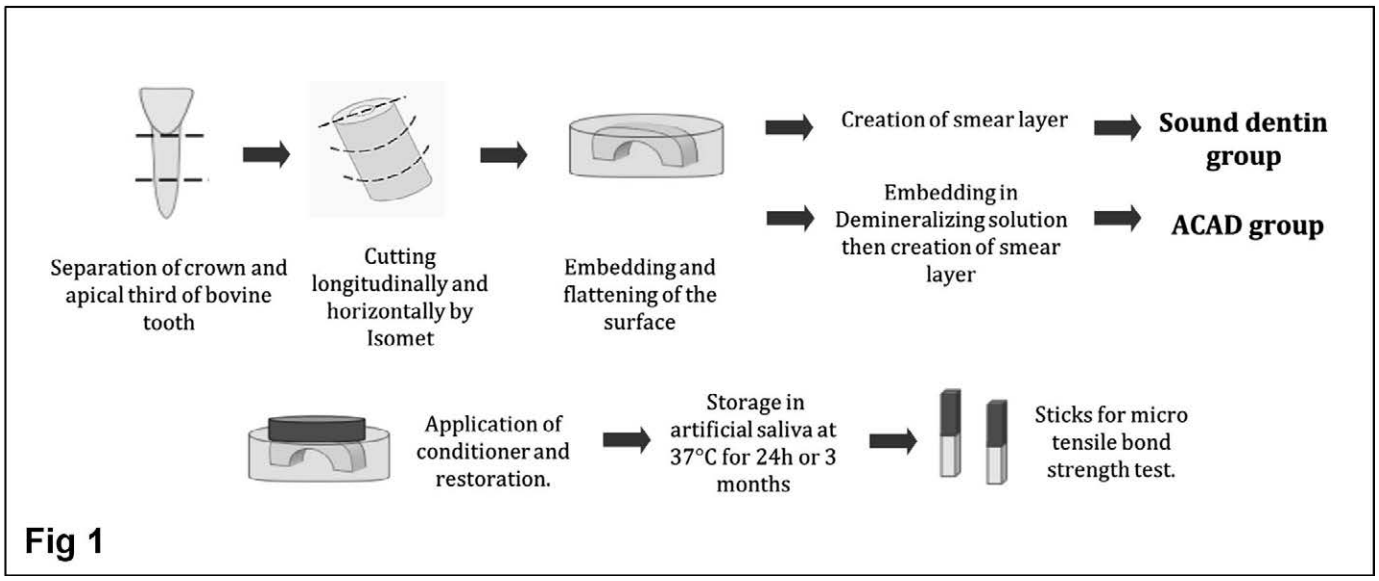

Figure 1. *Schematic diagram showing specimen preparation for the μTBS test.*

effect of dentin substrate, conditioning agent, and storage condition.

Two-way ANOVA was then used to test for significant differences in conditioning agent and storage condition with each dentin substrate, followed by *t*-test with Bonferroni correction for pairwise comparison. The *t*-test was used to analyze the significant differences in μTBS value at 24 hours and three months with each conditioning material under each type of dentin substrate. All statistical calculations were performed using the statistical software package Statistical Package for the Social Sciences for Windows (SPSS, SPSS Inc, Chicago, IL, USA) with $\alpha=0.05$.

For failure mode evaluation after debonding, the debonded specimens were fixed on a specimen holder using double-sided adhesive tape and sputter coated for evaluation under scanning electron microscope (SEM; S-4500, Hitachi, Ibaraki, Japan) at 15 kV accelerating voltage. Failure modes were classified into the following three categories: adhesive failure (failure at the dentin-material interface), cohesive failure (failure within the material or dentin itself), and mixed failure (partially adhesive and cohesive failure).

SEM Observations of Conditioned Dentin Surfaces

To observe the conditioned dentin surfaces, eight sound and ACAD dentin specimens' surfaces were treated in the same manner as described for specimen preparation for the μTBS test to obtain two specimens for each condition. The dentin surfaces were fixed by hexamethyldisilane for 10 minutes,[17] dried in a desiccator for 24 hours, and gold coated for SEM observation.

RESULTS

Table 2 shows the mean μTBS values of the RM-GIC to sound and ACAD after 24 hours and three months of storage in the artificial saliva as well as the survival rate for the different experimental groups. Three-way and two-way ANOVA followed by *t*-test with Bonferroni correction detected that dentin condition, storage period, and conditioning agent have a significant effect of the μTBS to dentin. For each conditioner, the μTBS values in sound dentin were significantly higher than those in ACAD ($p<0.05$), except for three months with no treatment "none" group, due to the significant drop in bond strength with sound dentin, which approaches its corresponding value in the ACAD group.

For the sound dentin, the three conditioners significantly increased the μTBS of RM-GIC to dentin ($p<0.05$) after 24 hours, in which self conditioner provided the highest μTBS, followed by EDTA and cavity conditioner. The lowest μTBS was obtained in the "none" group. After storage for three months, the μTBS of self conditioner remained the highest among the groups. However, a significant reduction in bond strength occurred for the "none" and EDTA groups. The μTBS of the self conditioner and cavity conditioner groups showed no significant change after three months of storage, although

Table 2: *Mean μTBS Values (MPa) and Percentage of Survived Sticks for Resin-Modified Glass Ionomer Cement to Root Dentin*[a]

Conditioning	Sound Dentin		ACAD	
	24 Hours	3 Months	24 Hours	3 Months
None	13.4 ± 2.3 (78)	4.3 ± 1.5^{C} (30)	7.2 ± 1.9 (55)	4.5 ± 1.9^{C} (50)
CC	18.5 ± 3.3^{A} (91)	17.1 ± 5.5^{A} (89)	11.9 ± 2.2 (85)	8.9 ± 2 (85)
SC	26.6 ± 4.4^{B} (100)	25.3 ± 3.9^{B} (100)	14.6 ± 3.3^{a} (100)	14.1 ± 3.5^{a} (100)
EDTA	21.5 ± 3.5^{A} (100)	4.6 ± 1.6^{C} (35)	n.d. (0)	n.d. (0)

Abbreviation: CC, cavity conditioner; SC, self-conditioner, EDTA, ethylenediamine tetraacetic acid; n.d., not detected.
[a] *Mean values ± standard deviation. Groups identified by similar large/small superscripts were not significantly different at* $p<0.05$. *Numbers in parentheses are survival percentage after sticks preparation.*

slight decreases were recorded. The survival rate decreased markedly in the "none" and EDTA groups but was almost stable with cavity conditioner and self conditioner, both of which had the highest survival rates.

For the ACAD groups, self conditioner also showed the highest μTBS values after 24 hours, followed by cavity conditioner and then "none." The difference between them was significant ($p<0.05$). The EDTA-conditioned group resulted in no specimens surviving during trimming procedures at either time period. After storage for three months, there was a significant reduction in bond strength for cavity conditioner and "none," whereas the self conditioner group remained the same.

Regarding failure mode (Table 3), after 24 hours, the mixed mode of failure was predominant in all conditioned sound dentin groups except for EDTA, in which adhesive failure was predominant. For the ACAD group, the predominant mode of failure was adhesive for the "none" group, while both adhesive and mixed mode of failures were equally predominant for the self conditioner and cavity conditioner groups. For the ACAD with EDTA conditioning, no sticks could be obtained for mode of failure investigation after the μTBS test because of debonding during preparation. After storage for three months, adhesive failure decreased for both the cavity conditioner and self conditioner groups but increased for the "none" and EDTA groups with sound dentin. For ACAD, the adhesive failure rate reduced with all groups, and no bonded sticks survived during preparation from the EDTA group.

Representative SEM images of the ACAD and sound dentin surfaces after each conditioner application showed a difference in the action of each conditioner with the smear layer and dentinal tubules openings of each dentin substrate, as shown in Figure 2. Cavity conditioner showed removal of the smear layer to a greater extent and partial opening of dentinal tubules, whereas EDTA totally unplugged the dentinal tubules and completely removed the smear layer. For the ACAD surfaces, a denser smear layer was observed; however, cavity conditioner managed to expose more open dentin tubules and a less compacted smear layer than the nonconditioned surface, while the EDTA-conditioned surface showed a smooth surface with occluded dentin tubules.

DISCUSSION

Previous studies reported that risk for root caries among individuals increased with the presence of exposed root surfaces, especially in elderly patients, patients with gingival attachment loss, or patients with deep pocket probing depths[18]; however, such findings are still disputed. Root dentin was used as the substrate with RM-GIC and conditioners in this study. Although RM-GIC is an excellent restorative choice for root caries treatment, coronal dentin has been routinely used as a bonding substrate in most laboratory studies because of its ease of preparation and use. Root dentin has not generally been used as bonding substrate because of the small region available for bonding, and its anatomical structure varies from coronal dentin.[19] Therefore, no previous published work was noted by the authors on prominent evaluation of the adhesive properties of RM-GIC to human root dentin. Root dentin typically exhibits fewer dentinal tubules and lower perme-

Table 3: *Mode of Failure of Specimens (Percentage)*[a]

Conditioning	Sound Dentin		ACAD	
	24 Hours	3 Months	24 Hours	3 Months
None	35/48/17	55/45/0	70/30/0	32/52/16
CC	30/56/14	25/52/24	45/47/8	17/53/30
SC	17/50/33	9/29/62	45/39/16	19/37/44
EDTA	54/13/33	70/30/0	n.d.	n.d.

[a] *Values are presented as adhesive failure mode/mixed failure mode/cohesive failure mode.*

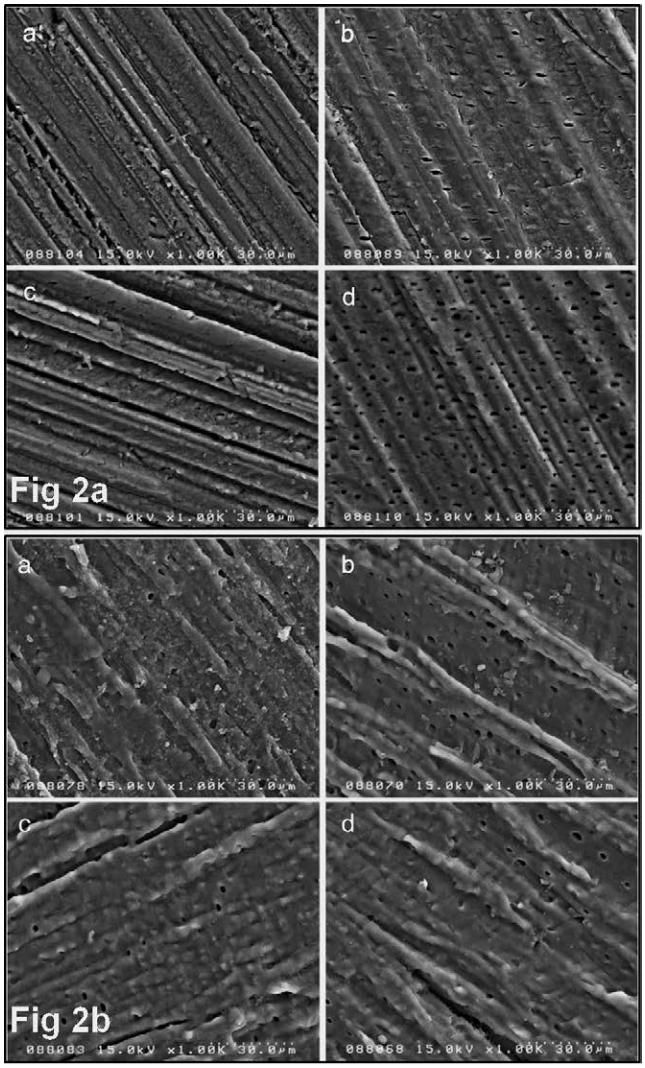

Figure 2. *(A): Representative SEM images of sound dentin surface after conditioning. (a): Sound dentin surface without conditioning with smear layer and surface scratches of SiC No. 600 preparation. (b): Sound dentin after cavity conditioner application showing smear layer removal and partial opening of dentinal tubules. (c): Sound dentin after self conditioner application showing partial dissolution of the smear layer. The hybrid layer between the self conditioner and dentin could not be identified. (d): Sound dentin after EDTA application showing total dissolution of the smear layer and patent dentinal tubules. (B): Representative SEM images of ACAD dentin surface after conditioning. (a): ACAD dentin surface with apparently dense smear layer. (b): ACAD surface after cavity conditioner application showing partial removal of the smear layer and more exposed dentinal tubules. (c): ACAD after self conditioner application. Again, this image could not show its mode of action. (d): ACAD after EDTA application showing large smooth areas of collapsed collagen and plugged dentinal tubules not suitable for micromechanical retention of RM-GIC.*

ability compared with coronal dentin. It should be noted that there is also a difference in the microhardness and the microstructure between coronal and root dentin because of a variation in the organization of both collagen fibrils and apatite minerals.[20] Fewer dentinal tubules exist, and they often have a circuitous path with a greater amount of intertubular dentin in the root dentin. Studies done with resin adhesives to compare bonding of root and coronal dentin showed variations in bond strength even for those undertaken on the same tooth.[19,21]

ACAD simulates the NCAD, which is partially demineralized. NCAD has a lower calcium and phosphate content and greater prevalence of exposed unprotected collagen fibrils than sound dentin.[15] The smear layer of CAD is thicker and appears to be enriched with organic components compared with a sound dentin smear layer.[22] This fact leads to a decrease in both micromechanical and chemical bonding between the carboxyl groups of the polyalkenoic acid and calcium ion of hydroxyapatite.[14,23]

ACAD has a mineral density similar to that of NCAD[16,24]; however, ACAD was created in a short period of time under controlled conditions using a demineralizing solution and is devoid of crystal logs occluding some of the dentinal tubules of the NCAD,[25] making the ACAD more standardized among specimens. According to the current results, the μTBS of the RM-GIC to ACAD was significantly lower compared with sound dentin in all groups. The SEM images of the ACAD groups in Figure 2B showed an apparently denser but diffuse smear layer with fewer open dentinal tubules available for resin-modified glass ionomer retention.

Fuji II LC is a light-cured RM-GIC; its bond strength to the root dentin after 24 hours and three months was evaluated with three conditioning materials (cavity conditioner, self conditioner, and EDTA). To improve RM-GIC bond strength to the tooth structure, the manufacturer recommends dentin conditioning with GC cavity conditioner, which contains 20% polyacrylic acid and 3% aluminum chloride or GC self conditioner, containing 4-META and HEMA. In previous studies,[7,26] EDTA was used for dentin conditioning because of its calcium chelation action and ability to remove the smear layer while having a neutral pH value.

It was reported that the RM-GIC used has an initially higher bond strength than the conventional GICs.[27-30] The RM-GIC used contains HEMA, which provides a superior wetting ability of the dentin[29,31] and improves mechanical interlocking with the dentinal tubules. In addition, the polyacrylic acid in the RM-GIC acts as a mild self-conditioner.[7] When the smear layer on the dentin surface was not conditioned, this layer possibly blocked the chemical

bonding of the RM-GIC to the intertubular dentin and also limited any micromechanical interlocking to exposed collagen fibers or patent dentinal tubules. Retention from inherent dentin irregularities[7] was believed to be responsible for short-term bonding strength, but on storage, the bond dropped significantly,[32] leading to high failure rates and low bond strength of both sound and ACAD.

When cavity conditioner was used as the conditioning agent, the μTBSs were significantly higher than those of the no treatment (none) group in both sound dentin and ACAD. The bond strength was maintained after three months of storage in artificial saliva with sound dentin; however, the bond strength to ACAD decreased significantly during storage, but the survival rate remained unchanged.

Cavity conditioner contains 20% polyacrylic acid and 3% aluminum chloride. The SEM images demonstrated that cavity conditioner partially removed the smear layer but did not totally unplug the dentinal tubules. The increase of μTBS may be due to the removal of the smear layer and partial demineralization of the underlying dentin, which increased the surface area and microporosities.[33] The exposed calcium ions within the hydroxyapatite are available for chemical bonding with the carboxyl groups of the polyalkenoyic acid.[7] The aluminum chloride is believed to play role in stabilization of collagen matrix during demineralization.[7] Abdalla[34] reported formation of a 2-μm-thick hybrid-like layer with resin tags of 1 μm in length when cavity conditioner was used for conditioning of sound dentin. According to Yui and others,[35] cavity conditioner increased the dentin permeability, providing an additional source of water for the acid-base setting reaction of the GIC, promoting the maturation of the glass ionomer at the interface. These factors may contribute to the greater resistance against dentin bonding degradation of the RM-GIC over time[36,37] compared with the "none" group. However, this thin hybrid-like layer or acid-base–resistant layer as described by Tanumiharja and others[38] was less resistant to degradation when cavity conditioner was used with ACAD.

Because of the resin component of the RM-GIC, bond strength can be enhanced by incorporating resin bonding mechanisms. Self conditioner contains HEMA as well as 4-META, a functional monomer that is able to chemically interact with hydroxyapatite in dentin[39]; therefore, by conditioning with self conditioner, dentin wettability should have been improved, allowing better monomer penetration into the dentin and therefore improving the quality of the hybrid layer, which may be the major source of bond strength, as suggested by Imbery and others.[7] Self conditioner provided the highest μTBSs for each substrate, and this outcome corresponds with previous studies.[7,40] Self conditioner treatment led to the only group in which the μTBS did not significantly decrease after three months in both sound dentin and ACAD.

EDTA used in this study was adjusted as 0.5 M concentration at neutral pH, which is commonly and effectively[41] used for dentin treatment. EDTA has the ability to chelate calcium ions and dissolve the mineral phase of the dentin without altering the dentin organic phase.[42] The 24-hour bond with sound dentin was the second highest. The SEM images of the sound dentin treated with EDTA showed complete removal of the smear layer and lack of dentin tubule smear plugs, which may have facilitated the micromechanical retention of the RM-GIC. However, after three months of storage, a significant drop in μTBS with high failure rates was observed. It is suggested that a microporous scaffold of collagen fibrils exposed when EDTA removed the mineral component of the dentin enabled good immediate bonding via micromechanical interlocking, but there may have been limited chemical bonding, which is the essential factor for dentin bond durability for GIC.

Using EDTA with ACAD, no bonded sticks survived during the trimming procedure for measuring the μTBS. This might have been due to disadvantages in the chelating effect of EDTA. The low calcium content that remained in the ACAD substrate with an unsupported weak and collapsed collagen fibril network (Figure 2B) was not suitable for the chemical binding of the RM-GIC. It should be noticed that in this study, it was preferred to standardize the number of sticks subjected to a μTBS test among all subgroups. The high failure rate in some subgroups led to a limitation in the number of tested sticks.

From the failure mode, previous literature reported variability in failure modes for Fuji II LC with different conditioners.[7,38,40,43,44,45] Therefore, investigation of the fractured beams was undertaken using SEM at high magnification. It showed the presence of a fine film of RM-GIC close to the bonded surface, which was also reported by Yap and others,[46] who described this as the ion-exchange layer.[46,47] When calculating the mode of failure, this thin resin layer was considered as part of the RM-GIC. SEM micrographs for some debonded speci-

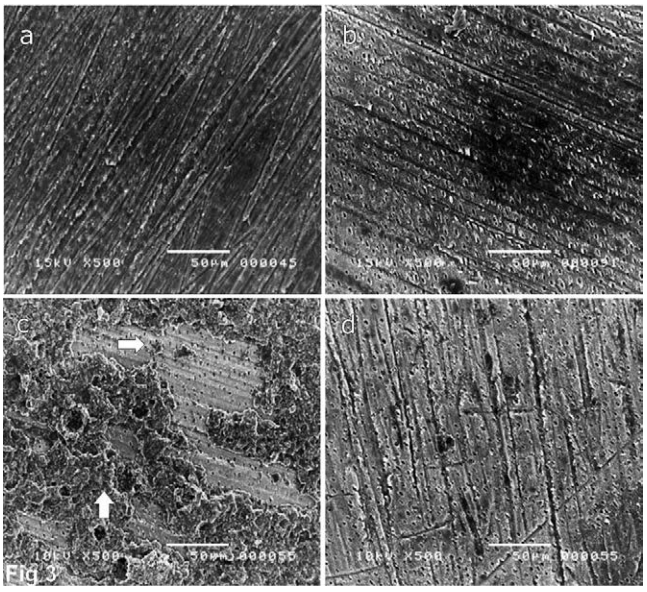

Figure 3. *SEM observations of debonded specimens at various magnifications along the dentin side of sound groups. (a): High magnification of adhesive failure in the "none, 24h" group, showing most of the dentinal tubules occluded by the smear layer, and the remaining scratches from the surface grinding are visible. (b): High magnification of adhesive failure of the cavity conditioner, 24-hour specimen. Dentin scratches are clearly visible with almost no smear layer and partially open dentinal tubules. (c): Self conditioner, 24 hours. The upper arrow points to dentin, while the lower arrow points to a resin area covering the dentin surface. (d): EDTA, 24-hour specimen, showing the dentin surface with no smear layer and open dentinal tubules.*

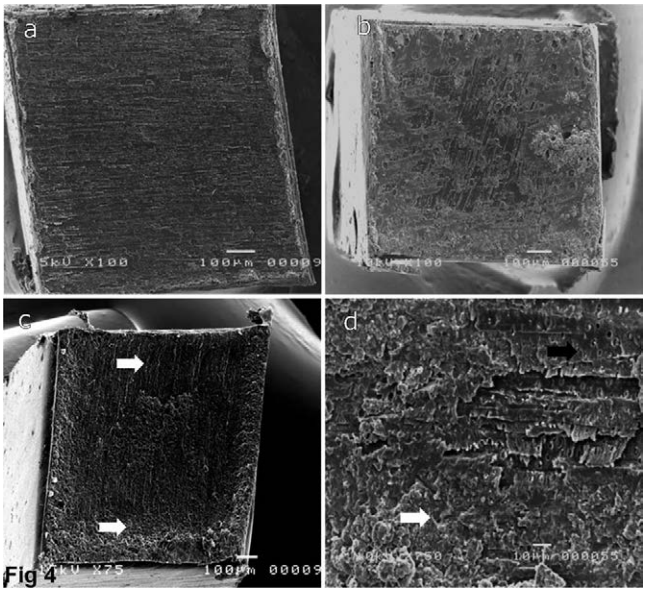

Figure 4. *SEM observations of debonded specimens at various magnifications along the dentin side of ACAD groups. (a): Adhesive failure of none, three months (denser smear layer than sound dentin). (b): Mixed failure specimen of cavity conditioner, three-month group. The ACAD surface appears at the center of the specimen. (c): Cohesive failure of self conditioner, 24 hours, with thick resin-modified glass ionomer at the bottom arrow that becomes thinner at the upper arrow. (d): High magnification of self conditioner specimen to show ACAD marked with a black arrow at the top of the image and thin RM-GIC layer marked with a white arrow at the bottom.*

mens taken during mode of failure investigation are presented in Figures 3 and 4.

For the ACAD groups, the predominant mode of failure was adhesive failure at 24 hours; however, after three months, adhesive failure was reduced and mixed failure was predominant except for the self conditioner group, in which cohesive failure in the RM-GIC predominated. It was noticed that a few beams with mixed failure in the cavity conditioner and self conditioner groups showed partial cohesive fracture in the demineralized dentin layer after three months of storage. It was notable that most of the cohesive failures occurred in the bulk of the RM-GIC after 24 hours. However, most of the cohesive fractures were at the base of the RM-GIC after three months probably because of the maturation of the cement, leaving a thin resin surface covering the dentin and detected by high magnification (marked with white arrow, Figure 4d). This fact suggested maturation of the interface between the RM-GIC and dentin, especially when self conditioner was used. The high incidence of a thin resin layer in the self conditioner specimens indicated penetration of RM-GIC into dentinal tubules and also the formation of a true hybrid layer at the RM-GIC–dentin interface. Therefore, the clinical implication of this study is that use of self conditioner or cavity conditioner has the potential to improve the bond strength and durability of RM-GIC to sound and carious root dentin, while using EDTA as a conditioning agent to root dentin should be avoided.

CONCLUSION

According to the outcomes from the current study, self conditioner provided a superior bond strength with the RM-GIC Fuji II LC compared with the other tested conditioning agents when applied to sound and artificial caries–affected root dentin. However, EDTA is not suitable as a conditioner for root dentin with Fuji II LC.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Tokyo Medical and Dental University. In this study, extracted bovine teeth were used. The study was approved by the Human Research Ethics Committee, Tokyo Medical and Dental University. The approval code for this study is 725.

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 presented in this article.

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Effect of Oxygen Inhibition Layer of Universal Adhesives on Enamel Bond Fatigue Durability and Interfacial Characteristics With Different Etching Modes

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Clinical Relevance

The oxygen inhibition layer of universal adhesives does not impair the enamel bond fatigue durability and interfacial characteristics of the adhesive regardless of etching mode, making it unnecessary for clinicians to consider it a cause for concern.

SUMMARY

Objective: The purpose of this study was to evaluate the effect of the oxygen inhibition layer of universal adhesive on enamel bond fatigue durability and interfacial characteristics with different etching modes.

Methods: The three universal adhesives used were Scotchbond Universal Adhesive (3M ESPE, St Paul, MN, USA), Adhese Universal (Ivoclar Vivadent, Schaan, Lichtenstein), and G-Premio Bond (GC, Tokyo, Japan). The initial shear bond strength and shear fatigue strength to enamel was determined in the presence and absence of the oxygen inhibition layer, with and without phosphoric acid pre-etching. The water contact angle was also measured in all groups using the sessile drop method.

Results: The enamel bonding specimens with an oxygen inhibition layer showed significant-

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ly higher ($p<0.05$) initial shear bond strengths and shear fatigue strengths than those without, regardless of the adhesive type and etching mode. Moreover, the water contact angles on the specimens with an oxygen inhibition layer were significantly lower ($p<0.05$) than on those without, regardless of etching mode.

Conclusion: The results of this study suggest that the oxygen inhibition layer of universal adhesives significantly increases the enamel bond fatigue durability and greatly changes interfacial characteristics, suggesting that the bond fatigue durability and interfacial characteristics of these adhesives strongly rely on its presence.

INTRODUCTION

The polymerization reaction in adhesive systems induced by photo-curing leads to the decomposition of camphorquinone and tertiary amine, resulting in the generation of reactive free radicals, which are able to add to the double bonds of resin monomers.[1] However, oxygen has a greater ability to react with the propagating free radicals than the monomer molecule, oxidizing them into peroxy radicals, which have relatively low reactivity toward the monomer and form peroxides, terminating polymerization if they do react.[2] This leads to retardation or inhibition of the free radical polymerization reaction[3] and consequently to the formation of an oxygen inhibition layer on the superficial surface of photo-cured resin-based materials when these are polymerized in the presence of air.[4]

The evidence concerning the role of the oxygen inhibition layer of adhesives is still controversial, with some studies reporting that the presence of an oxygen inhibition layer is necessary for bonding the adhesive to resin composite[1,5] and others reporting that this layer has no significant effect[6-8] or a negative effect[9-11] on bonding. Therefore, further studies are necessary in order to elucidate its effects on bond durability.

The development of next-generation adhesive systems has aimed to reduce technique sensitivity and the number of clinical steps,[12] leading to time-saving options such as single-step self-etch adhesives.[13] In conjunction with this trend, universal adhesives have been introduced to the profession.[14] Universal adhesives can be used with any of the total-etch, self-etch, or selective-etch techniques, and can bond to various substrates other than tooth substrates.[15] The increasing popularity of these adhesives, due to their versatility, has called into question the role of the oxygen inhibition layer in bonding.

Tsujimoto and others[16] have reported that the interfacial characteristics of the oxygen inhibition layer differ with the type of adhesive system. Its characteristics in photo-cured resin-based materials are influenced by several factors, including monomer chemistry,[4,8] filler morphology and temperature,[17,18] radical concentration,[2] and the rate of oxygen consumption.[19] Therefore, it is possible that the interfacial characteristics of the oxygen inhibition layer of universal adhesives may differ from those of other adhesive systems. These characteristics are often examined using water as a probe liquid and provide information on the suitability of a surface for bonding,[20] which affects the strength of the bond across the interface.[21] Thus, analyzing the interfacial characteristics of universal adhesives in terms of water contact angles may provide insights into the influence of the oxygen inhibition layer on their bond durability.

The purpose of this laboratory study was to investigate the effect of the oxygen inhibition layer of universal adhesives on enamel bond fatigue durability and interfacial characteristics. The null hypothesis tested posited that the enamel bond fatigue durability and interfacial characteristics of universal adhesives would not be influenced by the presence or absence of the oxygen inhibition layer.

METHODS AND MATERIALS

Study Materials

The three universal adhesives used were Scotchbond Universal Adhesive (SU; 3M ESPE, St Paul, MN, USA), Adhese Universal (AU; Ivoclar Vivadent, Schaan, Lichtenstein), and G-Premio Bond (GB; GC, Tokyo, Japan). Ultra-Etch (Ultradent Product, South Jordan, UT, USA) was used as a phosphoric acid pre-etching agent for bonding to enamel, and Z100 Restorative (3M ESPE) was used as the resin composite for the bonding procedures. The lot numbers and compositions of the materials used are listed in Table 1.

Initial Shear Bond Strength Tests

This study used extracted noncarious deidentified human molars. The experimental protocol for using deidentified human molar teeth was reviewed and approved by the Biomedical Institutional Review Board at Creighton University, Omaha, NE, USA (No. 760765-1). The enamel bonding sites were

Table 1: *Materials Used in This Study*

Materials (Lot No.)	Type of Material (Code)	Main Components	Manufacturer
Scotchbond Universal Adhesive (566724)	Universal adhesive (SU)	Bis-GMA, HEMA, MDP, ethyl methacryalate, methyl-reaction products with decanediol and phosphorous oxide, propenoic acid, copolymer of acrylic and itaconic acid, dimethylaminobenzoate, methyl ethyl ketone, ethanol, water, camphorquinone, silane-treated silica	3M ESPE, St Paul, MN, USA
Adhese Universal (164453)	Universal adhesive (AU)	BIS-GMA, HEMA, MDP, MCAP, decanediol dimethacrylate, dimethacrylate, ethanol, water, initiator, stabilizers, silicon dioxide	Ivoclar Vivadent, Schaan, Lichtenstein
G-Premio Bond (541424)	Universal adhesive (GB)	MDP, 4-MET, MEPS, methacrylate monomer, acetone, water, initiator, silica	GC, Tokyo, Japan
Ultra-Etch (G019)	Pre-etching agent	35% phosphoric acid, glycol, cobalt aluminate blue spinel	Ultradent Products, South Jordan, UT, USA
Z100 (1312131)	Resin composite	Bis-GMA, TEGDMA, silane-treated ceramic, benzotriazolyl methylphenol	3M ESPE, St Paul, MN, USA

Abbreviations: bis-GMA, bisphenol A glycidyl methacrylate; 4-MET, 4-methacryloyloxyethyl trimellitate; HEMA, 2-hydroxyethyl methacrylate; MCAP, methacrylated carboxylic acid polymer; MDP, 10-methacryloyloxydecyl di-hydrogen phosphate; MEPS, methacryloyloxyalkyl thiophosphate methylmethacrylate; TEGDMA, triethylene glycol dimethacrylate.

prepared by sectioning the teeth mesiodistally and removing approximately two-thirds of the apical root structure. Sectioned buccal and lingual halves were mounted in 25-mm brass rings with Bosworth Fastray acrylic material (Keystone Industries, Gibbstown, NJ, USA). The enamel surfaces were ground flat with 180-, 320-, 600-, 1200-, and 4000-grit silicon carbide paper (Struers, Cleveland, OH, USA) using a grinder-polisher (Ecomet 4, Buehler, Lake Bluff, IL, USA). These surfaces were then washed and dried using a dental three-way syringe at a distance of 5 cm above the surface at air pressure of 0.3 MPa. Some enamel surfaces were pre-etched with phosphoric acid for 15 seconds prior to application of the adhesive (with pre-etching), whereas others were not (without pre-etching). The specimens were prepared under ambient conditions of 23°C ± 2°C and 50% ± 10% relative humidity.

Fifteen specimens per group were used to determine the initial shear bond strength of the universal adhesive. The adhesives were applied to the enamel surfaces according to the manufacturers' instruc-

Table 2: *Application Protocol for Phosphoric Acid Pre-Etching, Universal Adhesives, and Protocol for Making Specimens of Presence- and Absence-of-Oxygen-Inhibition-Layer Groups*

Component	Protocol
Pre-etching	Pre-etching
With pre-etching	Enamel surface was acid etched with phosphoric acid for 15 s, rinsed with water for 15 s (three-way dental syringe) and air-dried
Without pre-etching	Phosphoric acid pre-etching was not performed
Adhesive	Adhesive application
SU	Adhesive applied to air-dried tooth surface with rubbing action for 20 s and then medium air pressure applied to surface for 5 s. Adhesive photo-cured for 10 s.
AU	Adhesive applied to air-dried tooth surface with rubbing action for 20 s and then medium air pressure applied to surface for 5 s. Adhesive photo-cured for 10 s.
GB	Adhesive applied to air-dried tooth surface for 10 s and then maximum air pressure applied to surface for 5 s. Adhesive photo-cured for 10 s.
Oxygen inhibition	Making specimens
Presence of oxygen inhibition layer	The adhesives were applied to the enamel surfaces according to the manufacturer instructions and photo-cured for 10 s.
Absence of oxygen inhibition layer	The top surface of the specimens of presence group was removed with ethanol-impregnated cotton pads.

Abbreviations: AU, Adhese Universal; GB, G-Premio Bond; SU, Scotchbond Universal Adhesive.

tions (Table 2) and photo-cured for 10 seconds at a standardized distance of 1 mm with a quartz-tungsten halogen unit (Spectrum 800 Curing Unit, Dentsply Caulk, Milford, DE, USA) set at 600 mW/cm^2 (presence-of-oxygen-inhibition-layer group). To produce specimens without an oxygen inhibition layer (absence-of-oxygen-inhibition-layer group), the top surface of the specimens was treated with ethanol-impregnated cotton pads (Medline Sterile Alcohol Prep Pads, Medline Industries, Mundelein, IL, USA).

Metal rings machined from 304 stainless steel and with an inner diameter of 2.4 mm, outer diameter of 4.8 mm, and height of 2.6 mm were used to confine the resin composite on the enamel surfaces for shear bond strength tests. Following the application of adhesive to the bonding sites, a releasing agent (3% solution of paraffin in hexane) was applied to the contact surface of the metal ring, which was positioned over the bonding site and secured in place by clamping in a custom fixture. The resin composite was filled in the ring and photo-cured for 30 seconds at a standardized distance of 2 mm, resulting in a resin composite cylinder measuring 2.4 mm in diameter and 2.5 mm in height inside the ring. The ring was left in place for the tests, and the bonded specimens were stored for 24 hours in distilled water at 37°C before testing.

The specimens were loaded to failure at 1.0 mm per minute using an ElectroPuls E1000 machine (Instron Worldwide Headquarters, Norwood, MA, USA). A metal rod with a chisel-shaped end was used to apply the load to the metal ring immediately adjacent to the flat-ground enamel surface. The initial shear bond strength (MPa) was calculated by dividing the peak load at failure by the bonded surface area. After testing, the bonding site tooth surfaces and resin composite cylinders were observed under an optical microscope (MZ16, Leica Microsystems, Buffalo Grove, IL, USA) at a magnification of 20× to determine the bond failure mode. The proportion of the resin composite surface with adherent enamel and visible residues was estimated to determine the type of failure (adhesive failure, cohesive failure in enamel, cohesive failure in resin composite, or mixed failure [combination of adhesive and cohesive failure]).

Shear Fatigue Strength Tests

The staircase method was used for shear fatigue strength tests,[22,23] and the specimens were prepared in the same way as they were for initial shear bond strength tests. The lower load limit was set near zero (0.4 N) and the setting between 50% and 60% of the initial shear bond strength determined for each of the adhesives tested was used for the initial maximum load. The load was applied at a frequency of 20 Hz using an electronic dynamic test instrument (ElectroPuls E1000, Instron Worldwide Headquarters) with a sine wave for 50,000 cycles or until failure occurred. The specimens were immersed in water at approximately 23°C to minimize the temperature rise, which might influence the mechanical properties. The load was incrementally (approximately 10% of the initial load) adjusted upward or downward (depending on survival or failure, respectively), and 20 specimens were used to determine the shear fatigue strength under each test condition. Using the calculation described by Draughn[22] and Dewji and others,[23] the test stress likely to produce 50% failure was termed *shear fatigue strength*. The mean shear fatigue strength (X) and its standard deviation (S) were calculated by the following formulae.

$$X = Xo + d\left(\frac{A}{N} - \frac{1}{2}\right)$$

$$S = 1.62d\left(\frac{NB - A^2}{N^2} + 0.029\right)$$

$$N = \sum n_i,\ A = \sum i n_i,\ B = \sum i^2 n_i,$$

where Xo is the lowest stress level considered in the analysis, and d is the stress increment used in the sequential tests. The lowest stress level at which a failure occurs is denoted by i = 0, the next i = 1, etc; n_{ii} is the number of failures after shear bond fatigue strength tests in each increment.

After testing, the type of bond failure was examined in the same manner.

Water Contact Angle Measurements

Specimens of the presence and absence groups with and without pre-etching were prepared as described for initial shear bond strength tests. The equilibrium water contact angles were measured in 10 specimens per group by the sessile drop method under ambient conditions of 23°C ± 2°C and 50% ± 10% relative humidity using a contact angle measurement apparatus (DM 500, Kyowa Interface Science, Saitama, Japan) fitted with a charge-coupled device camera to enable automatic measurement. A standardized 3 μL drop of distilled water was placed on the cured adhesive surface, and a profile image was captured after 500 milliseconds using the apparatus. Water

Table 3: *Initial Shear Bond Strengths (MPa) and Standard Deviations (in Parentheses) for Universal Adhesives*

Oxygen Inhibition	With Pre-Etching[a]			Without Pre-Etching		
	SU	AU	GB	SU	AU	GB
Presence of oxygen inhibition layer group	44.3 (4.8) a,A	40.6 (3.9) a,A	42.4 (5.5) a,A	27.2 (2.3) a,B	25.4 (3.5) a,B	26.1 (2.4) a,B
Absence of oxygen inhibition layer group	36.6 (4.2) b,A	34.8 (4.4) b,A	34.1 (3.1) b,A	22.0 (3.0) b,B	20.3 (3.7) b,B	20.4 (3.1) b,B

Abbreviations: AU, Adhese Universal; GB, G-Premio Bond; SU, Scotchbond Universal Adhesive.
[a] *Same small letter in same column indicates no significant difference ($p>0.05$). Same capital letter within individual rows indicates no significant difference ($p>0.05$).*

contact angles were then calculated by the θ/2 method using the built-in interface measurement and analysis system (FAMAS, Kyowa Interface Science).

Scanning Electron Microscopy Observation of Fracture Surface

Representative images of the fracture surfaces after initial shear bond strength and shear fatigue strength tests were taken in three specimens per group using scanning electron microscopy (SEM; TM3000 Tabletop Microscope, Hitachi High Technologies, Tokyo, Japan). The specimens were coated with a thin film of gold-palladium in a vacuum evaporator (Emitech SC7620 Mini Sputter Coater, Quorum Technologies, Ashford, UK), and SEM observations were carried out using an operating voltage of 15 kV.

SEM Observation of Resin-Enamel Interface

Representative images of the resin-enamel interfaces were carried out in three specimens per group using field-emission SEM (ERA 8800FE, Elionix, Tokyo, Japan). Bonded specimens of each group were stored in distilled water at 37°C for 24 hours, embedded in self-curing epoxy resin (Epon 812, Nisshin EM, Tokyo, Japan), and then stored at 37°C for a further 24 hours. They were then sectioned along the diameter of the resin composite post, and the surfaces of the cut halves were polished with 180-, 320-, 600-, 1200-, and 4000-grit silicon carbide paper using a grinder-polisher (Ecomet 4, Buehler). The surface was finally polished with a soft cloth using 1.0-μm-grit diamond paste. SEM specimens of the resin-enamel interface were dehydrated by first immersing them in ascending concentrations of aqueous tert-butanol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for two hours) and then transferring them to a critical-point dryer (Model ID-3, Elionix, Tokyo, Japan) for 30 minutes. To enhance the visibility of the layers, the polished surfaces were etched for 40 seconds using an argon ion beam (Type EIS-200ER, Elionix) directed perpendicularly to the surface at an accelerating voltage of 1.0 kV and an ion current density[24] of 0.4 mA/cm^2. Surfaces were coated with a thin film of gold in a vacuum evaporator (Quick Coater Type SC-701, Sanyu Electron, Tokyo, Japan). SEM observations were carried out using an operating voltage of 10 kV.

Statistical Analysis

All statistical tests were carried out using a commercial statistical software package (SPSS Statistics Base for Windows, IBM, Armonk, NY, USA), except for the modified *t*-test with Bonferroni correction, which was performed using custom software. Because the shear bond strength and water contact angle data exhibited normal distribution (Kolmogorov-Smirnov test), a three-way analysis of variance (ANOVA) and Tukey post hoc test at a significance level of 0.05 were used to analyze differences.

RESULTS

Initial Shear Bond Strength Tests

The initial shear bond strengths of universal adhesives in the presence and absence groups with and without pre-etching are listed in Table 3. The three-way ANOVA revealed that the presence or absence of an oxygen inhibition layer, adhesive type, and etching mode had a significant influence ($p<0.05$) on shear bond strength, but the interactions among these factors were not significant ($p>0.05$).

Regardless of etching mode, the initial shear bond strengths in the presence-of-oxygen-inhibition-layer group were significantly higher ($p<0.05$) than those in the absence group. Moreover, universal adhesives with pre-etching exhibited significantly higher ($p<0.05$) initial shear bond strengths than those without pre-etching, regardless of the presence or absence of oxygen inhibition layer and adhesive type.

Shear Fatigue Strength Tests

Shear fatigue strengths of universal adhesives in the presence and absence groups with and without pre-

Table 4: *Shear Fatigue Strengths (MPa) and Standard Deviations (in Parentheses) for Universal Adhesives*

Group	With Pre-Etching [a]			Without Pre-Etching		
	SU	AU	GB	SU	AU	GB
Presence of oxygen inhibition layer	22.2 (2.6) a,A	20.3 (2.3) a,A	21.1 (2.1) a,A	13.8 (2.7) a,B	12.6 (1.6) a,B	13.8 (2.4) a,B
Absence of oxygen inhibition layer	18.1 (2.2) b,A	17.1 (2.5) b,A	16.0 (2.1) b,A	11.0 (1.0) a,B	9.8 (0.8) b,B	10.3 (1.0) b,B

Abbreviations: AU, Adhese Universal; GB, G-Premio Bond; SU, Scotchbond Universal Adhesive.
[a] Same capital letter within individual rows indicates no significant difference (p>0.05).

etching are listed in Table 4. Regardless of the etching mode, the shear fatigue strengths of the presence-of-oxygen-inhibition-layer group were significantly higher ($p<0.05$) than those of the absence group. Moreover, universal adhesives with pre-etching exhibited significantly higher ($p<0.05$) shear fatigue strengths than those without pre-etching, regardless of the presence or absence of an oxygen inhibition layer and adhesive type.

Fracture Mode Analysis of Debonded Specimens

The failure modes of debonded specimens after initial shear bond strength and shear bond strength tests are shown in Table 5. The predominant mode of failure observed was the adhesive type, and a χ^2 test showed no significant differences in failure mode with presence or absence of an oxygen inhibition layer, adhesive type, or etching mode.

Water Contact Angle Measurement

The water contact angles of universal adhesives in the presence and absence groups with and without pre-etching are listed in Table 6. The three-way ANOVA test revealed that the presence or absence of an oxygen inhibition layer and adhesive type exerted a significant influence ($p<0.05$) on water contact angles, whereas etching mode had no such influence ($p>0.05$). The interaction between the presence or absence of an oxygen inhibition layer and adhesive type was also more significant than other interactions for water contact angles.

The water contact angles of the presence-of-oxygen-inhibition-layer group were significantly lower ($p<0.05$) than those of the absence group, regardless of the adhesive type and etching mode. Moreover, the water contact angles of universal adhesives in the presence and absence groups were also material dependent.

SEM Observation of Fracture Surface

Representative SEM images of the debonded specimens after shear fatigue strength tests are shown in Figure 1 (images after initial shear bond strength tests were similar). The specimens predominantly exhibited adhesive failure at a lower magnification, and the specimens with pre-etching showed more enamel fragments than those without pre-etching at higher magnification, regardless of the presence or absence of an oxygen inhibition layer.

SEM of Resin-Enamel Interface

Representative SEM images of the resin-enamel interfaces are shown in Figure 2. The resin-enamel interfaces of all the adhesives showed excellent adaptation, regardless of which group they belonged to. Moreover, the thickness of the adhesive layer in the presence-of-oxygen-inhibition-layer group ap-

Table 5: *Failure Mode Analysis of Debonded Specimens After Initial Shear Bond Strength and Shear Fatigue Strength Tests*[a]

Bond Strength Test	Group	Pre-Etching	SU	AU	GB
Initial shear bond strength	Presence of oxygen inhibition layer	With pre-etching	[67/0/13/20]	[93/0/7/0]	[86/0/7/7]
		Without pre-etching	[100/0/0/0]	[100/0/0/0]	[100/0/0/0]
	Absence of oxygen inhibition layer	With pre-etching	[86/7/7/0]	[100/0/0/0]	[100/0/0/0]
		Without pre-etching	[100/0/0/0]	[100/0/0/0]	[100/0/0/0]
Shear fatigue strength	Presence of oxygen inhibition layer group	With pre-etching	[60/10/20/10]	[90/0/10/0]	[80/0/10/10]
		Without pre-etching	[100/0/0/0]	[100/0/0/0]	[90/0/10/0]
	Absence of oxygen inhibition layer group	With pre-etching	[80/0/15/5]	[90/0/10/0]	[85/0/5/10]
		Without pre-etching	[100/0/0/0]	[100/0/0/0]	[100/0/0/0]

Abbreviations: AU, Adhese Universal; GB, G-Premio Bond; SU, Scotchbond Universal Adhesive.
[a] Percentage of failure mode [adhesive failure/cohesive failure in resin/cohesive failure in enamel/mixed failure]. There was no significant differences in failure mode with presence or absence of oxygen inhibition layer, type of adhesive, or pre-etching status.

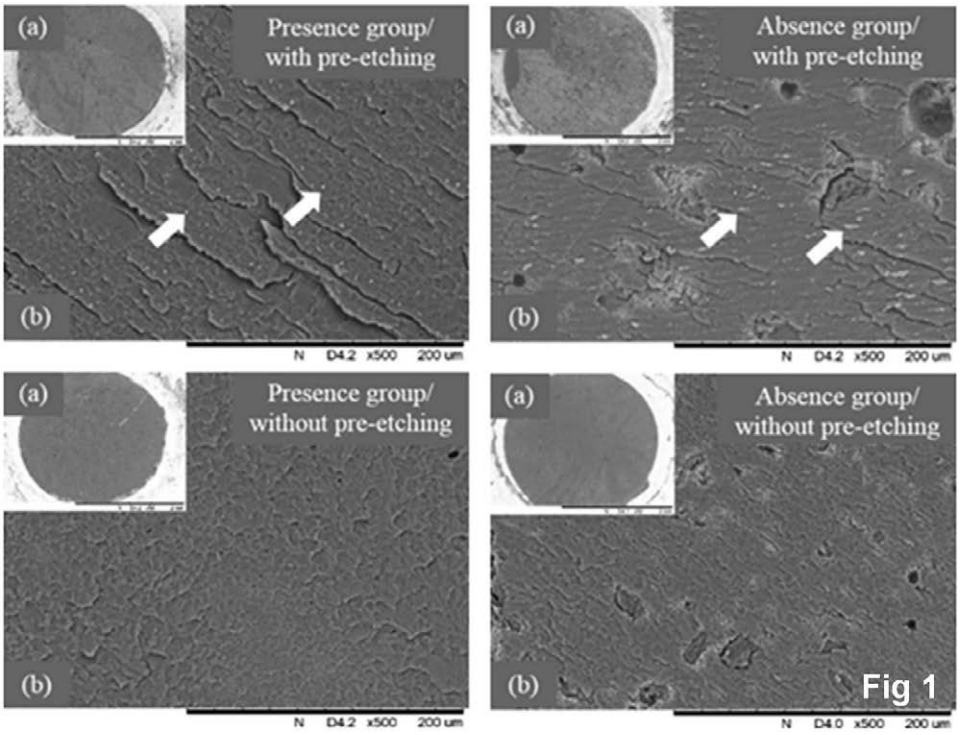

Figure 1. *Representative scanning electron microscopy images of debonded specimens after shear fatigue strength tests at (a): 50× magnification and (b): 500× magnification. The debonded specimens predominantly exhibited adhesive failure at lower magnification, and cracks and cleavages could be clearly seen in the specimens at higher magnification. Some features of enamel fragments such as benchmarks (arrows) can be observed in the specimens with phosphoric acid pre-etching, indicating strong bonding between the enamel and adhesive.*

proximated 9 μm, whereas that of the absence group was approximately 4 μm, irrespective of whether they had undergone pre-etching.

DISCUSSION

The present study indicates that the initial shear bond strengths and shear fatigue strengths of universal adhesives in the presence-of-oxygen-inhibition-layer group were significantly higher than those in the absence group, regardless of adhesive type and etching mode. Bond fatigue strength testing is one way to investigate clinical bond fatigue durability for *in vitro* studies, given that bonded specimens are subjected to repeated subcritical loading challenges, simulating intraoral conditions.[25,26] Therefore, the results of the current study indicate that the oxygen inhibition layer of universal adhesives may not impair enamel bonding in terms of bond fatigue durability. However, there were no significant differences in failure type between the presence and absence groups, and adhesive failure was predominant in both initial shear bond strength and shear fatigue strength tests. In the present study, the shear load was applied to the metal rings enclosing the resin composite bonded to enamel, and adhesive failures were observed most frequently, regardless of the presence or absence of an oxygen inhibition layer. This can be explained by the fact that bonding specimens enclosed within a metal mold exhibit a higher frequency of adhesive failure than unenclosed specimens.[27,28] Scherrer and others[29] argued that an increased production of adhesive failure in bond strength tests was desirable to provide more relevant information about the mean bond strength. Therefore, in this study, the type of fracture observed may provide evidence on the relevance of the initial shear bond strengths and shear fatigue strengths of universal adhesives.

Table 6: *Water Contact Angles (°) of the Universal Adhesives Under All Experimental Conditions*[a]

Group	With Pre-Etching			Without Pre-Etching		
	SU	AU	GB	SU	AU	GB
Presence of oxygen inhibition layer	51.1 (3.8) a,A	49.2 (3.1) a,A,B	46.4 (3.9) a,B	51.4 (3.2) a,A	49.1 (3.5) a,A,B	47.9 (3.0) a,B
Absence of oxygen inhibition layer	58.5 (2.4) b,A	55.1 (2.1) b,A,B	54.3 (2.6) b,B	57.9 (2.5) b,A	55.8 (3.0) b,A,B	53.7 (2.2) b,B

Abbreviations: AU, Adhese Universal; GB, G-Premio Bond; SU, Scotchbond Universal Adhesive.
[a] *Values in parentheses are standard deviations. Same small letter in same column indicates no significant difference ($p > 0.05$). Same capital letter within individual rows indicates no significant difference ($p > 0.05$).*

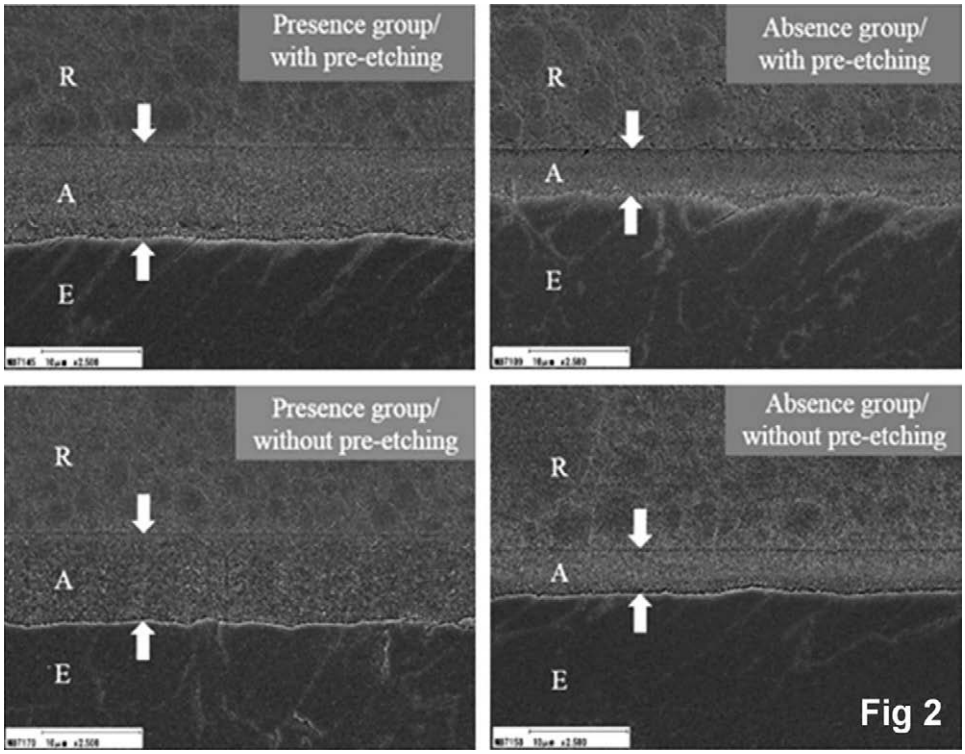

Figure 2. *Representative field-emission electron microscopy images of the resin-enamel interface at 2500× magnification. The resin-enamel interface of the universal adhesives used showed excellent adaptation, regardless of the presence or absence of an oxygen inhibition layer. Arrows indicate the thickness of the adhesive layer. Regardless of the etching mode, the thickness of the adhesive layer of the specimens in the presence-of-oxygen-inhibition-layer-group was approximately 9 μm, whereas that of the absence group was approximately 4 μm. Abbreviations: A, adhesive; E, enamel; R, resin composite.*

Interfacial characteristics in terms of water contact angle were also investigated to understand their relationship with the results of the initial shear bond strength and shear fatigue strength tests. Cured universal adhesives in the presence-of-oxygen-inhibition-layer group exhibited significantly lower water contact angles than those in the absence group, demonstrating their greater polarity and hydrophilicity. During the polymerization reaction, monomers in universal adhesives form highly cross-linked polymeric networks, which diminish their polarity and hydrophilicity.[30] Therefore, if the polymerization reaction of monomers in universal adhesives progresses to completion, the polarity and hydrophilicity should be reduced in the cured adhesive. Conversely, incomplete polymerization may lead to residual functional monomers within the adhesive.[31] Therefore, oxygen impairment of cross-linking within the oxygen inhibition layer of a cured adhesive may have led to lower water contact angles in the presence group.

In addition, the water contact angles of universal adhesives increased when the oxygen inhibition layer was removed, whereas the initial shear bond strengths and shear fatigue strengths decreased. Optimal wettability is important to enable the materials to spread across the entire surface and establish adhesion.[32] Thus, the maximum bond strength is assumed to occur when the wettability of the adherent surface is maximized.[33] This suggests that the interfacial characteristics of the universal adhesives strongly influenced the results of the initial shear bond strength and shear fatigue strength tests. Moreover, the oxygen inhibition layer readily adapts to the overlying material to increase the contact area; it also allows the materials on both sides to cross the interface and blend together to form an interdiffused zone where copolymerization can take place to produce a chemical bond between residual functional monomers in the oxygen-inhibition layer and the overlying resin composite.[7] All these effects tend to strengthen the layer-layer interaction.[34] This may explain why the cured universal adhesive in the presence-of-oxygen-inhibition-layer group with lower water contact angles exhibited higher initial shear bond strengths and shear fatigue strengths. Furthermore, the thickness of the universal adhesive layer of the presence group was approximately 9 μm, whereas that of the absence group was 4 μm, as seen in SEM observation of the resin-enamel interfaces. The relatively thin oxygen inhibition layer may have allowed complete diffusion of the photo-initiator into the overlying composite, resulting in higher initial shear bond strengths and shear fatigue strengths.

On the basis of the results of this study, our null hypothesis (that the enamel bond fatigue durability

and interfacial characteristics of universal adhesives would not be influenced by the presence or absence of the oxygen inhibition layer) was rejected.

CONCLUSION

The results of this study indicate that the oxygen inhibition layer of universal adhesives significantly increases the enamel bond fatigue durability and greatly changes interfacial characteristics, suggesting that the bond fatigue durability and interfacial characteristics of these adhesives strongly rely on its presence.

Acknowledgement

The authors thank Mr Jason M Moody for technical contributions.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Biomedical Institutional Review Board at Creighton University, Omaha, NE, USA. The approval code for this study is 760765-1.

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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Stress Distribution, Tooth Remaining Strain, and Fracture Resistance of Endodontically Treated Molars Restored Without or With One or Two Fiberglass Posts And Direct Composite Resin

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Clinical Relevance

The use of one fiberglass post is necessary and sufficient to achieve better biomechanical behavior of endodontically treated molars with severe tooth structure loss using direct composite resin.

ABSTRACT

Objectives: To evaluate the effects of direct composite resin without a post or with one or two fiberglass posts on the restoration of severely compromised endodontically treated molars.

Methods and Materials: Forty-five molars with 2 mm of "remaining tooth structure" were divided into three groups: Wfgp, restored with Filtek Z350XT without a fiberglass post; 1fgp, restored with Z350XT with one fiberglass post in the distal root canal; and 2fgp, restored with Z350XT with two fiberglass posts, one in the distal root canal and the other in the mesial-buccal root canal. The teeth were load cycled. Tooth remaining strain was measured using strain gauges (n=10) at two moments: TrSt-100 N, during 100 N occlusal loading, and TrSt-Fr, at fracture load. Fracture resistance was calculated, and fracture mode was classified. The elastic modulus and Vickers hardness were

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calculated using dynamic indentation (n=5). Stress distribution was analyzed by three-dimensional finite element analysis.

Results: The use of two fiberglass posts resulted in lower fracture resistance than was noted in the groups with one fiberglass post and without fiberglass posts. The lingual surface of the remaining tooth had higher strain values than the buccal surface, regardless of the restorative technique and moment of evaluation. The absence of a fiberglass post resulted in significantly higher strain values and more irreparable fracture modes than were noted in the other groups. The use of one fiberglass post had a better strain/fracture resistance ratio. Stresses were concentrated in the occlusal portion of the post and in the furcation region. The presence of one fiberglass post resulted in better stress distribution in the entire distal root dentin, reducing stress on the critical areas.

Conclusions: The use of one fiberglass post for restoring molars with direct composite resin resulted in higher fracture resistance than did the use of two fiberglass posts; it also resulted in better tooth remaining strain and stress distribution and more reparable fracture modes than were seen in the group without a fiberglass post.

INTRODUCTION

In emerging countries, dental caries remain one of the most prevalent health problems—mainly in children—that affects quality of life as a result of reduced esthetics and chewing abilities.[1] Dental caries comprise the most common reason for tooth extractions in young patients.[2] A considerable number of children have restricted access to dental services, and parents generally take their children to the dentist only when a problem becomes serious and causes discomfort or pain.[3] When the molar teeth affected by occlusal caries are not extracted, they can require endodontic treatment as a result of deep caries.[4]

Compared to intact teeth,[5] endodontically treated teeth are more susceptible to fracture as a result of extensive tissue loss and the loss of moisture content and flexibility,[6] as well as reduced resistance due to endodontic access preparations.[7] Despite the improvements in restorative materials and techniques, root fractures of endodontically treated teeth are still observed.[8,9] The most efficient way to prevent root fractures in endodontically treated teeth is through tooth structure conservation during restorative and endodontic procedures.[10] Therefore, better restorative methods are needed to effectively restore endodontically treated molar teeth.[10]

Evaluation of the remaining coronal structure is used to identify whether the use of a fiberglass post is necessary to retain the restoration material.[11] Direct composite resin is a treatment option that conserves the remaining tooth structure and results in good patient compliance.[12] Composite resin restorations associated with a fiberglass post have good results in terms of cusp strain and fracture resistance when used as indirect restorations for posterior teeth.[13] Teeth with a major loss of the coronal tooth structure require the placement of a post to ensure satisfactory core retention.[14,15] Of the different post materials, fiberglass posts have advantageous characteristics due to their mechanical properties.[16,17] Fiberglass posts have an elastic modulus similar to that of dentin structure, which has been reported[18] to reduce root fracture and to provide better stress distribution. Similar to the elastic modulus of fiberglass posts, resin cements, composite resin, and dentin are beneficial for improving stress distribution and produce better biomechanical restoration results in endodontically treated teeth.[19,20] The bonding interaction between the composite materials, specifically of the fiberglass post to the dental substrate, may create a structure, known as "monoblock," that dissipates the stresses produced by occlusal loads.[21] Nonetheless, clinicians frequently ask "How many posts are necessary to restore endodontically treated molars with severe tooth loss?" The use of two posts in a molar has, to our knowledge, not been described.

Therefore, the aim of this study was to evaluate the effects of the absence and the presence of one or two fiberglass posts in the restoration of severely damaged endodontically treated molars. The null hypothesis was that the mechanical behavior of endodontically treated molars, expressed as strain, fracture resistance, and stress distribution, is not influenced by the presence or number of fiberglass posts.

METHODS AND MATERIALS

Forty-five extracted, intact, caries-free human molars were used (Ethics Committee in Human Research approval No. 35506614.2.0000.5152). The selected teeth had an intercuspal width within a maximum deviation of 10% from the determined mean.[22] The intercuspal width varied between 4.81 mm and 5.98 mm. The teeth were cleaned using a rubber cup and fine pumice water slurry. The tooth

roots were covered with a 0.3-mm layer of a polyether impression material (Impregum, 3M ESPE, St Paul, MN, USA) to simulate a periodontal ligament and were then embedded in a polystyrene resin (Cristal, Piracicaba, Brazil) up to 2 mm below the cemento-enamel junction to simulate the alveolar bone.[23]

The teeth were randomly divided into three groups (n=15) according to the rehabilitation technique used. All groups had their coronal portions reconstructed directly with composite resin. The "without fiberglass post" (Wfgp) group did not receive a fiberglass post, the "one fiberglass post" (1fgp) group was restored using one fiberglass post in the distal root canal, and the "two fiberglass post" (2fgp) group was restored using two fiberglass posts, one in the distal root canal and the other in the mesial buccal root canal. The teeth (n=10) were submitted to mechanical fatigue cycles. Then the "tooth remaining strain" (TrSt) was measured using the strain gauge method, and the teeth were loaded until fracture. The other five teeth per group were restored and used for Vickers hardness (VH) and elastic modulus (E) measurements using the dynamic indentation method.

Specimen Preparation

A plastic mold of the occlusal surface of each tooth was created using an acetate matrix (Bio-art, São Carlos, SP, Brazil) and a vacuum-forming machine (Plastivac P7; Bio-art, SP, Brazil). The teeth had their coronal portion cut 2 mm above the enamel-cement limit by a precision saw (Isomet 1000, Buehler, Lake Bluff, IL, USA), simulating severe coronal tooth loss. Endodontic access was performed using spherical diamond burs (No. 1016; KG Sorensen, Barueri, SP, Brazil) and tapered carbide burs with noncutting tips (Endo-Z; Dentsply Maillefer, Ballaigues, Switzerland) in a high-speed handpiece with abundant irrigation. The root canals were located and initially explored with a No. 10 K-file (Dentsply Maillefer). The working length was determined by subtracting 1 mm from the length measured when the tip of the file emerged from the apical foramen. The cervical portion was prepared using Gates Glidden drills (Dentsply Maillefer) and was irrigated with 1.0% NaOCl and saline solution. An operator was calibrated to use the rotary nickel-titanium (Ni-Ti) System (Dentsply Maillefer) to perform all of the endodontic treatments and restorative procedures. The root canals were instrumented at the previously determined working length using rotary files (ProTaper Universal; Dentsply Maillefer), following the manufacturer's instructions. Each instrument was passively introduced into the root canals at a 250-rpm rotation rate (X Smart; Dentsply Maillefer). Irrigation was performed using 1% NaOCl after each instrument. The roots were filled with gutta-percha (Dentsply Maillefer) and calcium hydroxide–based endodontic sealer (Sealer 26, Dentsply Maillefer).

Post Space Preparation

Post spaces were prepared maintaining 4 mm of the gutta-percha. Heated instruments (GP heater; Dentsply Maillefer) were used to remove the gutta-percha, and a specific drill system corresponding to the fiberglass conical post (Exacto #2, Angelus, Londrina, PR, Brazil) properly shaped the root to receive the posts. The canal roots were cleaned with ethylenediaminetetraacetic acid for three minutes, rinsed well, and dried with absorbent paper points (Dentsply Maillefer).

Post Cementation and Remaining Tooth Restoration

All posts were cemented with self-adhesive resin cement (RelyX U200; 3M ESPE). The fiberglass posts were cleaned using a single application of 70% alcohol by microbrush (KG Sorensen) for one minute; then posts were dried and immersed in a solution of 24% hydrogen peroxide (H_2O_2; Dinâmica, SP, Brazil) for one minute.[24] Silano (Angelus) was applied to the post surface for one minute. Self-adhesive resin cement (RelyX U200; 3M-ESPE) was prepared according to the manufacturer's instructions and introduced into the canal; the post was seated under digital pressure. Excess cement was removed at one minute and five minutes later,[25] and the resin cement was light-cured at each coronal root surface (buccal, lingual, and occlusal) for 40 seconds using a quartz-tungsten-halogen unit (800 mW/cm^2; Optilux 501, Kerr Mfg Co, Orange, CA, USA).

The coronal reconstruction was incrementally built with nanofilled composite resin (Filtek Supreme, A2 Shade; 3M-ESPE). Each increment was light-cured for 20 seconds. The fiberglass posts were cut off 2 mm below the occlusal surface using a diamond bur. The last increment of the composite resin was applied using the acetate matrix to ensure adequate coronal anatomy reconstruction.

Mechanical Fatigue Cycling

Mechanical fatigue cycling was used to simulate chewing (Biocycle, Biopdi, São Paulo, SP, Brazil).

The samples were immersed in water maintained at approximately 37°C and were cycled 1.2 X 10^6 times at a 0 to 50–N axial compressive load with a 2-Hz frequency and an 8-mm-diameter stainless-steel sphere on the occlusal, simulating five years of aging.[26]

Tooth Remaining Strain During Occlusal Load Simulation (TrSt-100 N) of the Fracture Procedure (TrSt-Fr), Fracture Resistance, and Fracture Mode

To measure the TrSt, two strain gauges (PA-06-038AA-120-L; Excel Sensores, Embú, SP, Brazil) were attached to all specimens. One gauge was placed on the remaining buccal surface parallel to its long axis, and the other was placed on the remaining lingual surface.[27] The strain gauges were bonded using cyanoacrylate adhesive (Super Bonder; Loctite, Itapeví, SP, Brazil) and connected to a data acquisition device (ADS0500IP; Lynx Tecnologia Eletrônica, SP, Brazil).

A control specimen was attached adjacent to the tooth being tested to compensate for temperature variations due to electrical gauge resistance or the local environment.[27] The specimens with strain gauges were subjected to a nondestructive ramp load from 0 to 100 N in a mechanical testing machine (EMIC DL2000; EMIC Ltd, São José dos Pinhais, Paraná, Brazil).[28] The load was applied to the long axis of the tooth with an 8-mm-diameter metal sphere at a crosshead speed of 0.5 mm/min in a universal machine (DL2000, EMIC). The data were recorded on a computer that performed the signal transformation and the data analysis (AqDados 7.02 and AqAnalysis; Lynx, SP, Brazil).

The specimens were loaded until fracture using an axial compressive loading test. The force required (N) to cause fracture was recorded by a 500-kN load cell hardwired to software (TESC; EMIC) that detected any sudden load drop in the load cell during the compression tests. Strains were also recorded at failure load (TrSt-Fr). The fractured specimens were then analyzed in failure mode to determine the fracture types using the following classifications: I) fractures involving less than half of the composite resin restoration without post involvement; II) fractures involving more than half of the composite resin restoration without post involvement; III) fractures involving the restoration and tooth structure without post involvement, which are repairable with periodontal surgery; and IV) severe root and composite/post restoration fractures, which require extraction of the tooth, as shown in Figure 1.

Vickers Hardness and Elastic Modulus

The remaining five specimens from each group were used to analyze the Vickers hardness (VH) and elastic modulus (E) of the enamel and dentin of the remaining tooth structure, in addition to longitudinal and perpendicular analyses of the composite resin and fiberglass post. Each restored tooth was sectioned into two halves using a precision saw (Isomet 1000, Buehler). One section per tooth was randomly selected for the assessment of mechanical properties. The specimens were embedded in polyester resin (Instrumental Instrumentos de Medição Ltda, São Paulo, SP, Brazil). The surfaces were finished using silicon carbide papers (No. 600, 800, 1200, and 2000 grit; Norton, Campinas, Brazil) and polished with metallographic diamond pastes (6-, 3-, 1-, and 1/4-μm; Arotec, São Paulo, Brazil). After polishing, the specimens were cleaned using an ultrasonic cleaner filled with absolute alcohol for 10 minutes. Using a Vickers indenter (CSM Micro-Hardness Tester, CSM Instruments, Peseux, Switzerland), five indentations were made in each area to be analyzed. The indentations were made with controlled force, and the test load was increased or decreased at a constant speed of 0 to 500 mN in 20-second intervals. A maximum force of 500 mN was maintained for 15 seconds. The load and the penetration depth of the indenter were continuously measured during the load-unload hysteresis. The universal hardness was defined as the applied force divided by the apparent area of the indentation at the maximum force. The measurements were expressed in VH units by applying the conversion factor supplied by the manufacturer. The E value was calculated from the slope of the tangent of the indentation depth curve at the maximum force.[29]

Stress Distribution—Three-dimensional Finite Element Analysis (3D FEA)

To calculate stress distribution, a 3D finite element model was generated using a molar tooth with dimensions and geometry representative of the average selected teeth, employing the method based on MicroCT image.[30,31] A molar tooth was scanned by MicroCT (Model 1272, Bruker Skyscan, Kontich, Belgium) (Figure 2A). This MicroCT is an X-ray micro–computer tomographic unit composed of a scanner coupled to a Dell Precision workstation T5600 Intel Xeon (128GB 1600 MHz) and a Dell Precision cluster Intel Core (4 Gb CPU, 2.13 GHz) with NRecon software (Skyscan).

The equipment was adjusted to scan the whole tooth using the following: a beam accelerating

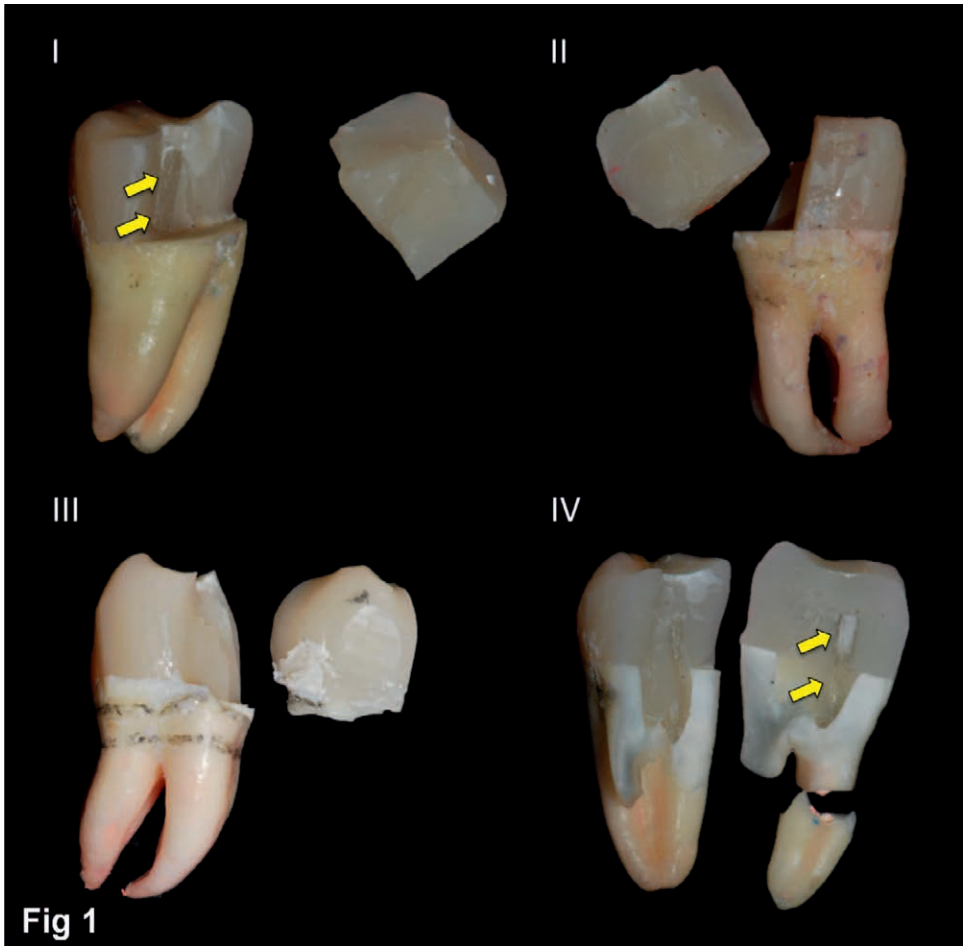

Figure 1. *Representative images of fracture modes: I, fracture involving less than half of the composite resin restoration without post involvement; II, fracture involving more than half of the composite resin restoration without post involvement; III, fracture involving the restoration and tooth structure without post involvement, which can be repaired by periodontal surgery; and IV, severe root and composite/post restoration fracture, which requires tooth extraction. Yellow arrow indicates the fiberglass post presence.*

voltage of 100 kV, an X-ray beam current of 100 μA, a copper (Cu) filter of 0.11 mm, an image pixel size of 13 μm, a resolution of 1632 × 1092, and a rotation step of 0.6°. Three frames per 1850 milliseconds with 20 random movements were obtained, which resulted in 1692 slices. Using NRecon software, 847 slices of tooth structure were selected, and the artefact correction parameters of smoothing 4 and ring 9 were applied to obtain an image with different shades of gray due to the different densities of the structure. The *.bpm files obtained from the MicroCT were viewed using an interactive medical image control system (MIMICS 16.0, Materialise, Leuven, Belgium). The segmentation of dental structures was accomplished based on image density thresholding. The masks of the cylinder, which simulate the bone, periodontal ligament, enamel, dentin, resin cement, gutta-percha, fiberglass post, and composite resin, were converted into a 3D file (via *.STL, bilinear, and interplane interpolation algorithms) using the Mimics *.STL shown in Figure 2B. The aspect ratio and connectivity of the triangles in the *.STLs resulted in an inappropriate model for FEA use. Therefore, the remesh component present in Mimics software was used to reduce the number of triangles and to simultaneously improve the quality of the triangles while maintaining the geometry. In addition, an advanced *.STL file design and meshing software (3-Matic 8.0; Materialise) were used to simulate the forms of treatment used in the specimens. The treatment of each *.STL was performed separately, followed by merging all the parts into a single *.STL file called the "assembly." The final assembly was then remeshed using the 3-matic remesh component and Boolean operations shown in Figure 2C. Self-intersecting curves were maintained, and the tolerance variation from the original data was specified (the quality of triangles does not mean tolerance variation from the original data). The Mimics remesh using the high-quality triangle height/base ratio can be imported to the FEA software package without producing errors.

As a specific approach for better model generation, the *.STL models were imported to MSC.Patran 2010r2 (MSC.Software, Santa Ana, CA, USA) and

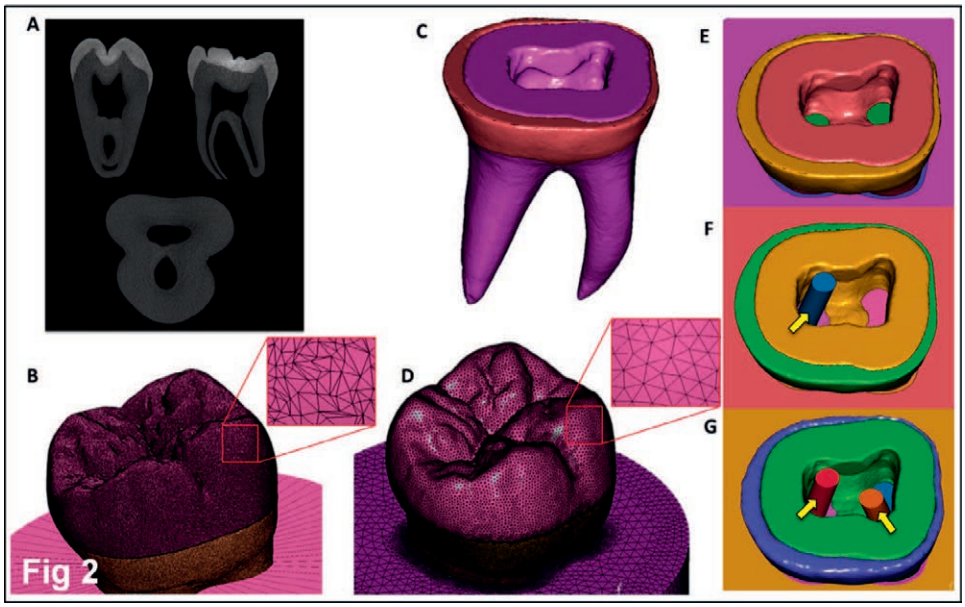

Figure 2. *3D finite element model generation; (A) MicroCT scanning of the molar; (B) the mesh process on Mimics software; (C) Boolean operation for cavity preparation; (D) the remeshing process on Patran software; (E) a model without a post; (F) a model with one fiberglass post; and (G) a model with two fiberglass posts. Yellow arrow indicates the fiberglass post presence.*

meshed (Figure 2D); four nodes of the tetrahedral elements were used to ensure the smooth contact of all interfaces of the models. The volumetric meshes of the model components were created based on the optimized surfaces standard triangulated language *.STL descriptions.[32] Afterwards, the meshes were imported to the FEA software package (MSC.Marc/MSC.Mentat, MSC.Software) for the attribution of material properties to the other model components (ie, cylinder, periodontal ligament, enamel, dentin, resin cement, gutta-percha, fiberglass post, and composite resin). The elastic modulus of the fiberglass post composite resin, enamel, and dentin were experimentally determined in this study and are shown in Table 1. The other material properties used were as follows: enamel with a Poisson ratio of 0.30 and dentin with a Poisson ratio of 0.23;[33] composite resin with a Poisson ratio of 0.24;[34] periodontal ligament elastic modulus of 50 MPa and a Poisson ratio of 0.45; gutta-percha elastic modulus of 0.69 MPa and a Poisson ratio of 0.45; and polyester resin elastic modulus of 13.7 GPa and a Poisson ratio of 0.30.[21] To simulate the interface among the model components, precisely bonded contacts were maintained. The nodes on the base of the bone model structure were rigidly fixed in the x, y, and z directions to simulate the experimental test. The loading conditions were simulated with nodal point load using individual force as experimentally determined (100 N on the occlusal surface of the restored model structure). The load application was in the coronal-apical direction in relation to the tooth longitudinal axis. The evaluation and postprocessing for each model were performed using the modified von Mises analysis with MSC.MARC/Mentat 2010r3 software (MSC.Software). The modified von Mises parameter takes into account the difference between the compressive and tensile strengths of the enamel, dentin, and composite resin.

Statistical Analysis

The TrSt and fracture resistance data were tested for normal distribution (Shapiro-Wilk) and equality of variances (Levene test), followed by parametric statistical tests. One-way analysis of variance (ANOVA) was performed to analyze the TrSt and fracture resistance. One-way ANOVA was also performed to analyze the effect of the depth of the cavities on the VH and E values for composite resin. One-way ANOVA was performed to analyze fracture resistance values. Multiple comparisons were made using the Tukey test. The failure mode data were

Table 1: *Means and (standard deviation) of ElasticModulus (GPa) and Vickers Hardness (N/mm^2) of Restorative Materials and Tooth Structures*

Materials/Tooth Structures	Elastic Modulus, GPa	Vickers Hardness, N/mm^2
Z350XT	14.9 (0.4)	153.4 (4.7)
Enamel	50.2 (0.9)	399.3 (8.6)
Dentin	18.3 (1.1)	180.5 (5.8)
Exacto Fiberglass post		
Parallel	29.8 (1.7)	201.9 (12.1)
Transversally	10.6 (0.8)	108.0 (7.6)

Table 2: *Means and (standard deviation) of Tooth Remaining Strain (μS) Measured by Strain Gauges (n=10)*[a]

Groups	Tooth Remaining Strain at 100-N Loading, μS			Tooth Remaining Strain at Fracture Load, μS		
	Buccal	Lingual	Mean	Buccal	Lingual	Mean
Without post	572.6 (142.5)	785.0 (173.9)	687.8 (189.3) B	3806.8 (341.8)	3419.8 (341.1)	3613.3 (387.1) B
One fiberglass post	563.6 (175.8)	631.8 (173.0)	583.1 (170.9) A	3612.0 (340.9)	3173.2 (449.4)	3392.6 (475.0) A
Two fiberglass posts	551.6 (194.4)	581.8 (194.4)	566.7 (185.2) A	3326.2 (497.0)	2807.2 (422.9)	3066.7 (522.1) A

[a] *Different letters indicate a significant difference between the restorative techniques (p<0.05).*

subjected to a chi-square test. Analysis of variance (two-way ANOVA) was performed to analyze the TrSt (B and L) and rehabilitation techniques. All tests were performed using a significance level of $\alpha = 0.05$, and all analyses were performed using the Sigma Plot version 13.1 statistical package (Systat Software Inc, San Jose, CA, USA). The stress distribution using the finite element method was analyzed descriptively.

RESULTS

VH and E Values

The means and standard deviations of VH and E for all tested materials are shown in Table 1. One-way ANOVA analysis showed no significant differences among the depth of the cavities for both mechanical properties of the composite resin restorations ($p=0.435$).

TrSt Values

The values of TrSt during the simulation at 100-N occlusal loading and maximum fracture loading are shown in Table 2. Factorial ANOVA showed significant differences in the TrSt values for the moment of measurement factor ($p=0.008$) and the restorative technique ($p<0.001$); however, no significant difference was found for the interaction between both factors ($p=0.212$). The lingual surface had higher TrSt values than the buccal surface, regardless of the restorative technique ($p=0.008$). For TrSt measured during the 100-N occlusal loading and until fracture, the restorative technique without posts had significantly higher TrSt values than did the other groups ($p<0.001$). No significant difference was found between the groups with one or two posts ($p=0.783$).

Fracture Resistance and Failure Mode

The means and 95% confidence intervals of fracture resistance and for the three restorative techniques are shown in Table 3. One-way ANOVA showed significant differences among the groups ($p<0.001$). The group with two fiberglass posts had significantly lower fracture resistance than the group with one fiberglass post ($p<0.001$) and the group without a fiberglass post ($p=0.002$). No difference was found between the group with one fiberglass post and the group without a fiberglass post ($p=0.798$).

The group without a fiberglass post had significantly more irreparable fracture modes than the groups with one or two fiberglass posts ($p=0.031$) (Table 3). The ratio between the maximum resistance and strain at the fracture moment is shown in Table 3. The group with one fiberglass post had a better ratio value than the group with two fiberglass posts ($p=0.001$), followed by the group without a fiberglass post, and the worst ratio was for the one fiberglass post group.

FEA Values

Stress distribution (MPa) during occlusal loading with 100 N was evaluated by modified von Mises criterion. The stress concentration values are visualized according to a linear scale of colors: blue indicates low stress values and gray and yellow indicate high stress values. The FEA indicated that

Table 3: *Means (Confidence Interval–95% Confidence Interval) of Fracture Resistance (N), Mode of Fracture, and the Ratio Between Maximum Cusp Deformation/Fracture Resistance Measured by the Axial Compression Test (n=10)*[a]

Groups	Fracture Resistance, N	Fracture Mode				Ratio Between Strain/ Fracture Resistance
		I	II	III	IV	
Without post	2939.4 (2617.3 – 3261.5) A	0	2	1	7	1.23 B
One fiberglass post	3096.1 (2758.6 – 3433.6) A	6	0	0	4	1.10 A
Two fiberglass posts	2023.7 (1670.1 – 2377.4) B	5	0	1	4	1.52 C

[a] *Different letters indicate a significant difference between the restorative techniques (p<0.05).*

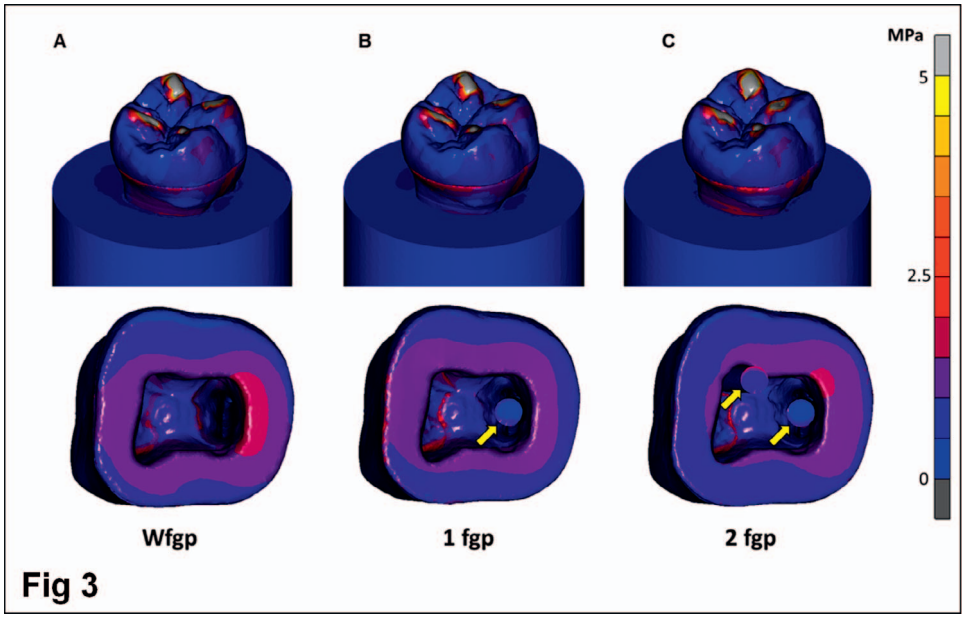

Figure 3. *Stress distribution (modified von Mises stress–MPa); (A) stress on the load application points on the occlusal surface of the composite resin restoration and (B) the pulp chamber and inner root canal surface. Yellow arrow indicates the fiberglass post presence.*

the group without a fiberglass post had higher stress levels in the external occlusal surface and higher stress levels in the cervical region than did other groups (Figure 3). We also observed higher stress levels in the mesial root and in the furcation region (Figure 4A) of the model without a post. The presence of one fiberglass post distributed stress to the entire distal root dentin, thereby reducing the stress on the critical areas (Figure 4B). Two fiberglass posts did not result in significant stress reduction when compared to one fiberglass post (Figure 4C).

DISCUSSION

The mechanical behavior, expressed by the TrSt, fracture resistance, fracture mode, and stress distribution of endodontically treated human molars, was affected by the presence and number of fiberglass posts; therefore, the null hypothesis was rejected.

The mechanical characterization of the restorative materials is very important for understanding the functional biomechanical behavior of rehabilitated endodontically treated teeth. When loads are applied to tooth structures, stress and strain are generated. Stress and strain are not bad; they are important for maintaining the synergism of the structures and biological components. If such stress becomes excessive and exceeds the elastic limit, structural failure may result.[35] The tooth structure is better able to support compressive stress than tensile stress.[36] When a rehabilitated tooth is subjected to occlusal loading, the stress and strain generated can be dissipated depending on the characteristics of each material and its adhesive integrity.[23]

In this study, mechanical fatigue was induced to simulate chewing cycles, as the oral environment experiences functional load that can lead to the degradation and subsequent fatigue failure of weakened regions. Supposing 240,000 to 250,000 occlusal contacts occur per year, 1.2 million cycles is equivalent to five years of masticatory simulation.[37] In this investigation, a force of 100 N was chosen to simulate the effect of chewing on TrSt and for the stress analysis because physiological biting forces during eating were found to be between 20 and 160 N.[37,38] The periodontal ligament simulation using elastomeric material and embedding of the root inside the polystyrene resin cylinder, which has a similar elastic modulus to bone tissue, offered more similarities to the oral environment than to the *in vitro* experiment.[37,38] The combination of using destructive and nondestructive methods on the same specimen, such as the strain gauge method, the fracture resistance, and fracture mode analysis, permits the sequential understanding of failure.[28,39]

Measuring strain before fracture may contribute to a better understanding of the entire fracture process, from initiation to ultimate rupture.[40] However, stress cannot be determined experimentally because it is necessary to use a FEA with simulation parameters in more realistic conditions.[41] During mastication, maximum stresses are applied to the cervical tooth area.[42] As the maximum stress concentration tends to coincide with higher tooth

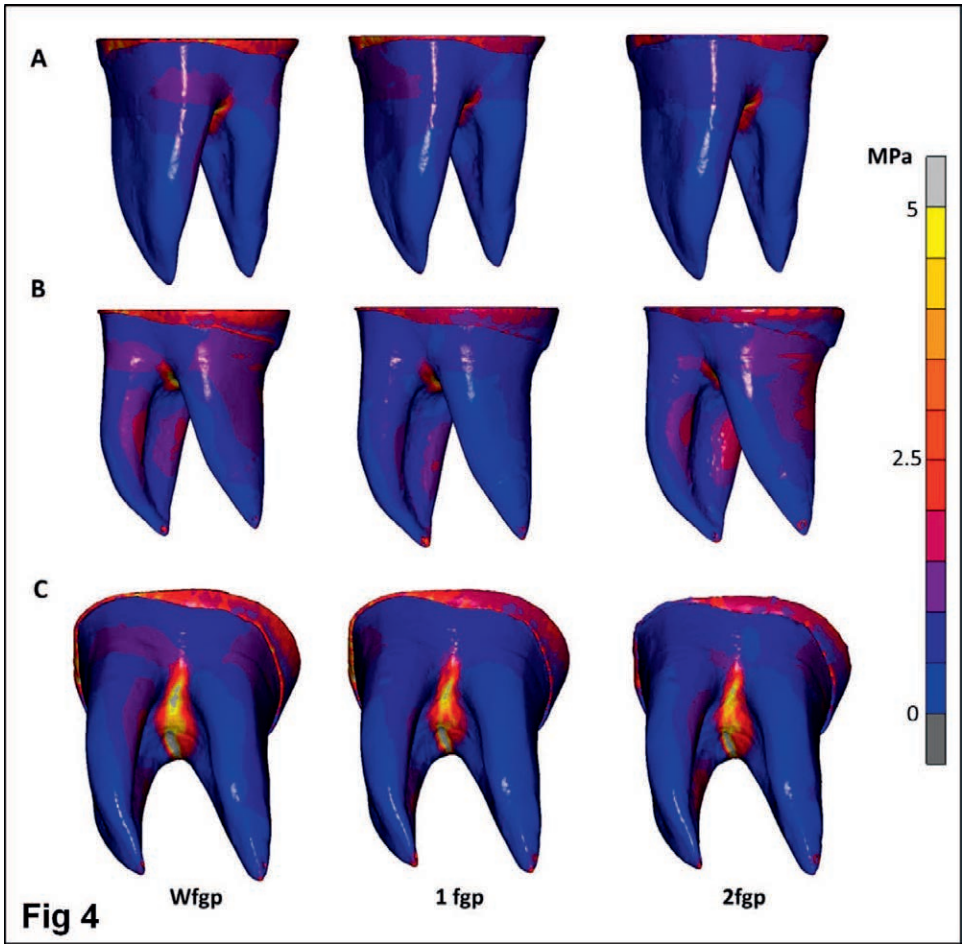

Figure 4. *Stress distribution (modified von Mises stress–MPa); (A) mesial root surface; (B) internal mesial root surface and distal root; and (C) furcation region.*

strain, strain gauges were fixed on the buccal and lingual cervical area; however, we observed higher FEA stress concentrations in the furcation region. For these reasons, future studies should use strain gauges in places with higher stress concentrations, as previously defined by FEA. In this study, the simulation of a 3D representative molar better qualified and quantified stress and strain in the inner dental structure and in the restorative materials.[43] Other very important and usually neglected aspects of FEA studies are the mechanical properties used to input the data from the restorative materials and dental structures.[41] In this study, we calculated the E and compressive strength of the restorative materials and dental structures isolated for the FEA. In this study we used a light cure composite material, Filtek Z350XT, which has an E value similar to that of the dentin. The use of light cure composite resin may be questionable for the direct core of endodontic treatment because of the reduced light intensity that reaches deep areas in the pulp chamber. In this study, we measured the VH in different depths of the restorations, and it was verified that the values are similar, demonstrating that polymerization of the light-curing composite resin was effective in deep areas of the cavities (Table 1).

TrSt values during the 100-N occlusal loading simulation and at the maximum fracture loading were influenced by study factors, tooth location, and the rehabilitation technique. The lingual surface had a greater degree of tooth remaining deformation during the 100-N occlusal loading and at the failure moment compared to the buccal surface, regardless of the rehabilitation technique used. The volume of dentin in the cervical area may explain the higher deformation at this location.[27] During the 100-N occlusal loading and at the failure moment, the tooth remaining of the Wfgp group had significantly higher TrSt values than did the other groups. The absence of a post resulted in higher strain to the composite resin coronal reconstruction. Because the bond integration was adequately created using a self-etching adhesive system and nanofilled composite resin, the stress

is transferred to the root dentin, explaining the higher strain values.

Fiberglass posts have an E value similar to that of dentin, although the orientation of the fibers determines the orthotropic characteristics, resulting in more axial stiffness.[44] The loading application, located close to the occlusal limit of the fiberglass post in the composite resin reconstruction, restricts the composite resin strain and increases the stress and strain concentration factors in this area. This aspect may explain the lower TrSt observed in the groups with one or two posts. We also observed that stress is distributed to the root canal and is dissipated throughout the entire root dentin in the FEA. The number of fiberglass posts did not significantly affect the TrSt.

Destructive mechanical tests, such as compressive occlusal loading, are used to define fracture resistance in situations of concentrated high-intensity load application. Generally, this test produces failure loads that exceed the average masticatory forces.[45,46] This test predicts the failure of restored teeth in complex conditions. The established integration of similar restorative materials, such as composite resin, adhesive system, silane, and self-adhesive resin cement, is essential for improving the fracture strength of endodontically treated teeth.[47,48]

Fracture resistance and the failure mode were influenced by the presence and number of fiberglass posts, which means that the posts had a significant effect on fracture strength. The two fiberglass post group had significantly lower fracture resistance than the other groups. The main purpose of a post is to retain the core material or restoration and not to reinforce the root dentin.[21] The presence of the pulp chamber and the retention capacity of one post are sufficient to retain the composite resin restoration.[27] However, in the presence of two fiberglass posts and when the stress concentration is located close to the loading area, the high stress level is concentrated close to the loading area, and the fracture resistance is reduced as a result of the Saint-Venant principle, as it is present at a stress concentration factor very close to the load application.[48] The presence of two posts determined more coronal fractures, confirming the stress concentration on the composite resin close to the load application area. The absence of a post resulted in lower fracture resistance and more irreparable fractures when compared to one and two fiberglass posts. The higher strain and stress concentrations at the remaining root dentin may explain the more catastrophic failure modes and the lower fracture resistance. The FEA 3D model of an endodontically treated molar is not common. The observation of higher stress concentrations at the furcation region should be taken into account during root canal preparation and during post space preparation. The preservation of dentin in this region is required for improving endodontically treated molar survival.

This study expands our understanding of the nature and development of stress and strain distributions in endodontically treated molars with or without fiberglass posts. The static load used in this study limits direct extrapolation of our findings to clinical conditions. More studies are necessary, especially clinical trials, in order to better maintain severely damaged molar teeth in young patients with debilities. In the present study, we showed that direct composite resin with one fiberglass post is a promising restorative procedure for endodontically treated molars with severely damaged structures, especially in communities in which people cannot afford ceramic restorations.

CONCLUSIONS

The use of one fiberglass post for restoring molars with direct composite resin showed higher fracture resistance than did the use of two fiberglass posts; it also resulted in better TrSt and stress distribution as well as more repairable fracture modes than were observed in the group without fiberglass post. Therefore, clinicians may choose to restore endodontically treated molars with severe tooth structure loss using direct composite resin and the one fiberglass post that is necessary and sufficient to determine better biomechanical behavior.

Acknowledgements

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee in Human Research, Federal University of Uberlândia, Minas Gerais, Brazil. The approval code for this study is 35506614.2.0000.5152.

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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Evaluation of Interfacial Gap Volume of Two Low-shrinkage Composites Using Micro–Computed Tomography

HNA Al Nahedh • NS Sibai

Clinical Relevance

The differences in the cavity configuration factor and volume of composite restorations do not always have a pronounced effect on gap volumes of low-shrinkage composites. Interfacial gaps result from an interplay of different factors related to the composition of the materials used and adhesive layer characteristics.

ABSTRACT

Objectives: To investigate the efficacy of X-ray micro–computed tomography (μCT) in the detection and quantification of interfacial gap formation in standardized Class I and Class II resin composite restorations, to compare the interfacial gaps for two low-shrinkage resin composites with a methacrylate composite material, and to determine any correlation between the cavity configuration factor (C-factor) and the volume of gaps formed.

Methods and Materials: Sixty standardized Class I and Class II cavities were prepared and divided into six groups. Three types of composites, with their recommended self-etching adhesive systems, were used: Filtek Z250 XT; Estelite Sigma Quick; and Filtek P90. Each of the composite materials was placed in 10 Class I and 10 Class II cavities. The specimens were digitized using Skyscan 1172 μCT. They were examined for gap volume measurements, the thickness of the adhesive layer, and location of interfacial gaps.

Results: There was a significant difference in the mean gap volume percentages of the three materials. The gap volume percent of Estelite Sigma quick was significantly lower than that of Filtek P90. No significant difference in the mean gap volume percentages of Class I and Class II restorations was found, except for Estelite Sigma Quick, in which the Class I gap volume percentage was higher than that of the Class II restorations.

Conclusions: μCT is an efficacious tool for the measurement of volumetric gaps formed at the tooth/restoration interface and for the evaluation of the adhesive layer. The differences in the C-factor do not always have a pronounced effect on the gap volumes of low-shrinkage composites.

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INTRODUCTION

Dental resin–based composites are complex materials that set through a free radical polymerization mechanism that involves cross-linking of monomer chains, resulting in a high polymerization shrinkage ranging between 2% and 6% by volume.[1-3] The volume reduction causes bond failure and, subsequently, gap formation at the weakest areas of the composite/tooth interface, leading to microleakage, postoperative sensitivity, and secondary caries.[4]

In recent years, manufacturers have attempted to reduce polymerization shrinkage through modifications in the chemical composition of the composite resin and filler material or through alterations in the size or percentages of filler content.[5-7] Resin-based composites with a higher filler content show reduced polymerization shrinkage as the volume of resin is minimized. Recent innovations in the field of restorative dentistry utilize nanotechnology to further enhance filler loading and esthetic properties of contemporary resin-based composites.[7-9]

Silorane-containing resin composites undergo a ring-opening polymerization reaction, in which monomers are linked together by opening, flattening, and extending toward each other. Reported volumetric shrinkage values are significantly lower than those of methacrylate-based composites.[10-12]

One factor that contributes to the amount of stress developed and, subsequently, the gap formed at the adhesive interface is the cavity configuration factor (C-factor). This term, created by Feilzer and others,[13] is a factor calculated as the ratio of bonded surfaces (restrained) to unbonded surfaces (free) of the resin composite restoration and is used in to describe the relationship between confinement conditions and stress values. de la Macorra and Gomez-Fernandez[14] suggested that it should be based on the calculation of the specific surface area of the cavity walls.[15,16] In addition, the volume of the composite resin increment is proportional to the degree of shrinkage that occurs, and it could be more detrimental than the C-factor itself.[17] In cavities with different volumes, the C-factor shows poor correlation with interfacial gaps; therefore, it may be a relevant parameter only when comparing restorations with similar volumes.[18,19]

Several methods have been used to study and measure the interfacial gap in dental restorative materials. However, most microleakage measurement methods are destructive, two-dimensional, and cannot allow study of the whole restoration interface.[20,21]

A standout among the progressive tools that have been introduced for the study of many focuses in dentistry is X-ray micro–computed tomography (μCT).[22,23] Early μCT-based studies[24-27] that compared internal adaptation of composite resin restoration were conducted without the use of a bonding agent. The use of bonded resin composites was reported in only a few studies.[28-30] In 2015, Hirata

Table 1: *Resin Composite Materials Used in the Study With Their Physical Properties*

Composite Material	Material Type	Composition	Filler Content	Shade	Elastic Modulus (GPA)	Volumetric Shrinkage	Manufacturer/ Lot No.
Filtek Z250 XT	Nanohybrid universal composite	Monomer: Bis-GMA, UDMA, Bis-EMA, PEGDMA, and TEGDMA Filler: ZrO_2/SiO_2	71% volume 82% weight	A2	12.5 (VOCO – Technical Guide)	1.7% (3M ESPE – Technical Report)	3M-ESPE, MN, USA/N261633
Filtek P90	Silorane-based microhybrid composite (low-shrinkage)	Monomer: Silorane (3,4 epoxycyclo-hexylethylcyclopoly-methylsiloxane,bis-3,4 epoxycyclo-hexylethylphenyl-methylsilane) Filler: SiO_2, ytterbium trifluoride	53% volume 73% weight	A2	12.8 (Ille and others[44])	0.9% (3M ESPE – Technical Report)	3M-ESPE, MN, USA/N326600
Estelite Sigma Quick	Supra–nano-spherical filled composite (low-shrinkage)	Monomer: Bis-GMA, TEGDMA Filler: SiO_2-ZrO_2, SiO_2-TiO_2	71% volume 82% weight	AO2	11.38 (Ille and others[44])	1.3% (Tokuyama – Technical Report)	Tokuyama, Tokyo, Japan/ W883

Abbreviations: Bis-GMA, bisphenol A-glycidyl methacrylate; UDMA, urethane dimethacrylate; Bis EMA, Ethoxylated bisphenol A dimethacrylate; PEGDMA, poly(ethylene glycol) dimethacrylate; TEGDMA, triethylene glycol dimethacrylate, SiO_2, silicon dioxide; ZrO_2,, zirconium dioxide; TiO_2, titanium dioxide.

and others[29] attempted to quantify the polymerization volumetric shrinkage of one regular and two low-shrinkage bulk fill composites in Class I cavities with or without an adhesive layer. The low-shrinkage composites showed less volumetric polymerization contraction than did the regular composite, and the use of dental adhesive decreased the total volumetric shrinkage for all evaluated composites. On the other hand, Carrera and others[30] developed a more comprehensive method to quantify interfacial leakage using silver nitrate infiltration and image subtraction.

μCT was also used in several studies[31-33] to evaluate the magnitude and direction of polymerization shrinkage. Recently, Van Ende and others[33] proposed a digital volume correlation enabling measurement and visualization of the regional shrinkage strain vectors within the entire volume of the composite restoration.

The first objective of this study was to investigate the efficacy of μCT in the detection and quantification of interfacial gap formation in standardized Class I and Class II resin composite restorations. The second objective was to compare the interfacial gap volume and distribution for two low-shrinkage resin composite materials with a conventional methacrylate nanohybrid composite material. The third objective of the study was to determine any possible correlation between the C-factor and the volume of gaps formed at the interface of Class I and II restorations for the three materials.

The null hypotheses were as follows:

1) μCT is not an effective tool for the evaluation of interfacial gap formation;
2) There is no difference between the three composite materials used in terms of the degree of interfacial gap volume, regardless of formulation; and
3) Different cavity configurations will not play a prominent role in the quantity or distribution of interfacial gaps for the three materials tested.

METHODS AND MATERIALS

This research was approved by the College of Dentistry Research Center and Deanship of Scientific Research at King Saud University (Research NF 2360). Sixty noncarious human premolars extracted for orthodontic reasons were cleaned with a hand scaling instrument, a rubber cup, and pumice, and were then stored in distilled water containing 0.05% thymol and refrigerated until use. After rinsing with water, all occlusal surfaces were flattened and polished using wet silicon carbide (SiC) papers (Buehler, Lake Bluff, IL, USA) mounted in an Automata machine (Jeanwirtz, Charlottenstrasse, Germany) to create a uniformly flat surface, starting with 240-grit, then 400-grit, and ending with 600-grit SiC papers. The teeth were then grouped into two preparation categories representing two configuration factors: Class I with a depth of 2 mm, width of 2 mm, and length of 3 mm; and Class II with an occlusal portion depth of 2 mm and dimensions of 2 × 2 mm, proximal box 3 mm deep from occlusal to gingival margin, and a 1-mm-wide gingival seat with rounded internal line angles and all enamel margins (Figures 1 and 2). All cavities were prepared using 330L pear-shaped tungsten carbide burs (Komet Brasseler, Savannah, GA, USA) and finished with a needle bullet finishing carbide bur (Komet Brasseler) on a high-speed handpiece (Sirona, Munich, Germany) under copious water irrigation. Burs were changed after preparation of five teeth. Cavities

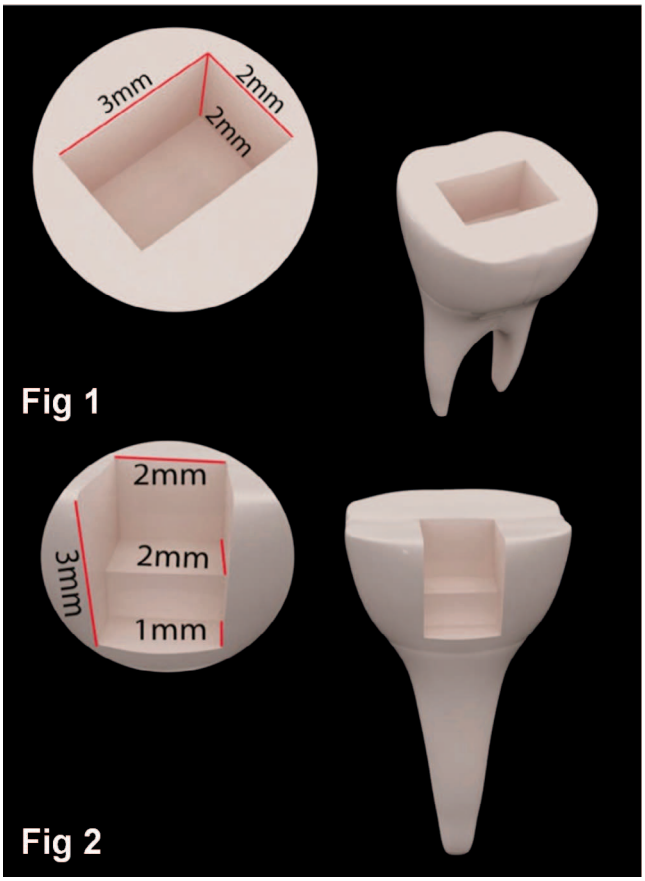

Figure 1. *Three-dimensional representation of Class I cavity preparations.*
Figure 2. *Three-dimensional representation of Class II cavity preparations.*

were precisely prepared by one operator using eye loupes (2.5×) and were standardized using an external verification method (Flexbar Tools, New York, NY, USA) with a depth-measuring digital micrometer and a custom indicator point stem that has a 0.120-inch (3.05-mm)–long measuring tip with a very small diameter that allows it to be easily inserted into the prepared cavity with an accuracy level of up to 0.05 mm. All teeth were restored within 24 hours of preparation, during which they were stored in 0.05% thymol solution.

The sample size was calculated to be at least eight teeth per group, based on an alpha level of significance of 0.05, a power of 0.87, and a standard deviation of mean interfacial gap volume of 0.7 mm^3. Cavities were divided into six groups of 10 teeth each. One group of each cavity type (Class I and Class II) was restored with one of the three types of composite materials used in this study (Table 1): the nanohybrid methacrylate-based composite Filtek Z250 XT (3M ESPE, St Paul, MN, USA); the low-shrinkage silorane-based microhybrid composite Filtek P90 (3M ESPE); or the low-shrinkage Supra–nano- spherical filled composite Estelite Sigma Quick (Tokuyama, Tokyo, Japan). All composites were used with their recommended self-etching adhesive system: Single Bond Universal (3M ESPE), P90 Self-etching Primer & P90 Bond (3M ESPE), and Bond Force (Tokuyama), respectively, according to the manufacturers' instructions (Table 2). Composite restorations were placed in one increment and cured for 40 seconds with Elipar™ S10 LED Curing Light (3M ESPE), with the light-curing tip placed perpendicular to the surface to be cured and 1 mm away from the top surface of the restoration. The light intensity was verified using a Demetron Optilux Radiometer (Kerr Corp, Orange, CA, USA) to be at 1000 mW/cm^2 prior to the placement of composite restorations for each group. All composite restorations were finished and polished using Soflex polishing discs (Kerr Corp) and a No. 12 blade.

Table 2: *Self-etching Adhesive Systems Used in the Study*

Composite Type	Self-etching Adhesive System	Composition	Manufacturer Instructions	Manufacturer/ LOT No.
Filtek Z250 XT	Single Bond Universal (one-bottle self-etch adhesive)	MDP phosphate monomer, dimethacrylate resin, HEMA, vitrebond copolymer, filler, ethanol, water, initiators, silane	Apply the adhesive to the prepared cavity and rub it in for 20 s Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent has evaporated completely Light-cure the adhesive for 10 s	3M-ESPE, St Paul, MN, USA/495603
Filtek P90	P90 Self-Etch Primer and Bond (two bottle self-etch primer & bond)	P90 Self-Etch Primer: Phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, Water, Ethanol, Silane-treated silica filler, Camphorquinone, Stabilizers P90 Bond: TEGDMA, Phosphoric acid methacryloxhexylesters, 1,6-hexanediol dimethacrylate, Bis-GMA, UDMA, Bis-EMA	Apply P90 Self-Etch primer and agitate primer on cavity surface for 15 s Light-cure for 10 s after drying with oil-free air Apply P90 Bond, disperse bond to a homogeneous film with oil-free air Light-cure for 10 s	3M-ESPE, St Paul, MN, USA/N239050, N239051
Estelite Sigma Quick	Bond Force (one bottle self-etching adhesive)	Phosphoric acid monomer, Bisphenol A di(2-hydroxy propoxy), dimethacrylate (Bis-GMA), Triethylene glycol dimethacrylate (TEGDMA),2-Hydroxyethyl methacrylate (HEMA), camphorquinone, dibutyl hydroxyl toluene	Apply the adhesive to the cavity walls and extend it to uncut enamel side Rub the adhesive under light finger pressure for 20 s Apply weak air flow for 5 s until the runny adhesive stays without any motion then finish with strong air flow for another 5 s Light-cure for 10 s	Tokuyama, Tokyo, Japan/175MM

Abbreviations: MDP Phosphate Monomer, 10-methacryloyloxydecyl dihydrogen phosphate monomer; HEMA, 2-hydroxylethyl methacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate, UDMA, urethane dimethacrylate; Bis EMA, ethoxylated bisphenol A dimethacrylate

Table 3: *Mean and Standard Deviation Values and Multiple Comparisons of Gap Volume Percentages Among the Three Materials for Class I and Class II Restorations*

Type of Class	Z250 XT	Filtek P90	Estelite Material	*F*-Value	*p*-Value
Class I	0.4869 (0.21)	0.8016 (0.47)	0.3682 (0.16)*	4.841	0.017
Class II	0.4762 (0.25)	0.7966 (0.50)	0.1640 (0.14)*	7.886	0.003

** Statistically significant $p<0.05$.*

The specimens were then sealed with nail polish and orthodontic self-cure resin at the apices and stored in deionized water at 37°C for seven days. During this time, they were subjected to 5000 cycles of thermal stressing between 5°C and 55°C and with a dwell time of 30 seconds and transfer time of five seconds using a thermocycling apparatus (Thermocycler 1106/1206 SD Mechatronik, Feldkirchen-Westerham, Germany). Afterwards, specimens were sectioned at the cemento-enamel junction using an Isomet 2000 electronic saw (Buehler, Lake Bluff, IL, USA) under water irrigation to facilitate their ease of placement closer to the μCT scanning machine. The pulp chambers were sealed with sticky wax and two coats of Bosworth Copaliner varnish (HJ Bosworth Company, Skokie, IL, USA) and placed in deionized water until the time of their scan.

All specimens were digitized within two weeks of the time of their restoration using Skyscan 1172 μCT. The images were converted to tomograms (cross sections) to be examined for gap volume measurements using the SkyScan CT-analyser TM (CT-An) software (Bruker microCT, Kontich, Belgium). Gap volume was calculated (in mm^3) as air space that existed at the composite/adhesive/tooth interface only and did not include any air space related to voids within the composite mass.

Using Data Viewer TM software (Bruker microCT), the Class I images were qualitatively examined for the general quality of each restoration, location of the gaps (line angles, margins, inner or outer half of walls, pulpal floor), the interfaces at which gaps were formed (tooth/adhesive, composite/adhesive, or within adhesive layer), presence of voids in the composite mass (size and location), and possibility of adhesive pooling (floor, walls, margins, or corners). In Class II restorations, the same parameters were examined, along with the location of gingival margins (enamel or dentin), adaptation of composite at the axiopulpal line angle, and position of gaps in the proximal box. Representative samples of each group were chosen for three-dimensional (3D) reconstruction using the SkyScan CT-Voxel TM (CT-Vox) program (Bruker microCT).

Total volume of the cavities was calculated based on their dimensions, as follows: 12 mm^3 for Class I and 14 mm^3 for Class II. Gap volume was quantitatively measured as a percentage of the total volume of the restoration. The data were statistically analyzed using statistical software (SPSS, Version 16; SPSS Inc, Chicago, IL, USA). Two-way analysis of variance (ANOVA) was used to compare all six groups together and to determine possible interactions between composite material and between classes with the interfacial gap size. The multiple comparison test was used to test if there was a significant difference in gap size among the three materials within the same class for Class I and Class II. Significance level was set at $p < 0.05$. Only 52 specimens were used for the statistical analysis, as eight specimens were discarded as a result of cracks or large composite voids that interfered with gap volume measurements.

RESULTS

The results of the gap volume percentages for the three materials and multiple comparisons are shown in Table 3.

Two-way ANOVA revealed a statistically significant difference in the mean gap volume percentages of the three materials (Filtek P90, Filtek Z250 XT, and Estelite Sigma Quick) (F=12.597; $p<0.0001$), but no statistically significant difference in the mean gap volume percentages of the two classes (Class I and Class II) (F=0.709; p=0.404). The interaction of the class vs the material type was not statistically significant (F=0.593; p=0.557).

A multiple comparison test was used to compare differences between the mean gap volume percentages of the three different materials within each class (Table 3). The results showed a statistically significant difference in the mean values of gap volume percentages among the three materials of Class I and Class II (F=4.841, p=0.017 and F=7.886, p=0.003, respectively). For Class I and Class II, the mean gap volume percentage of the Estelite material was significantly lower than the mean gap volume percentage of the Filtek P90 material, but not significantly different from the mean gap volume

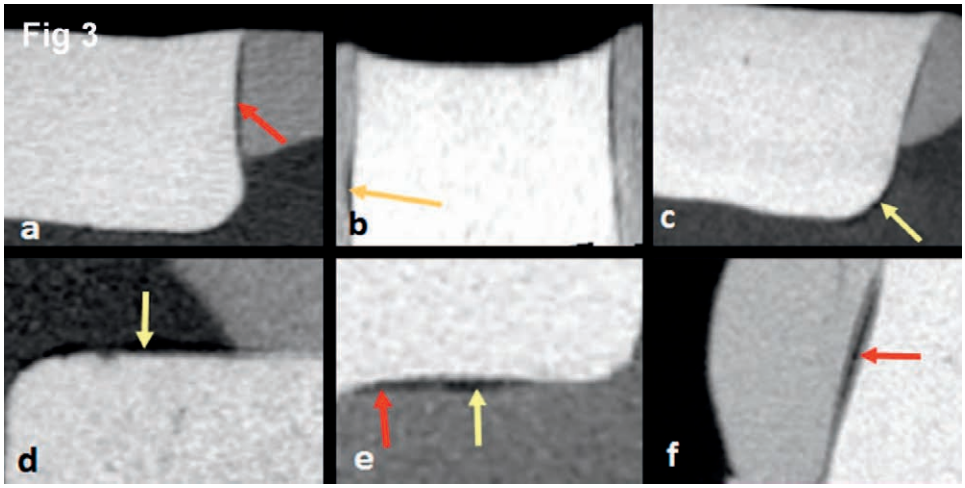

Figure 3. *Representative sections of μCT images taken for Classes I (Figure 3a through 3c) and II (Figure 3d through 3f) Filtek Z250 XT restorations showing locations of interfacial gaps; at the tooth adhesive interface (indicated with yellow arrows) and also showing separation within the adhesive layer (indicated with red arrow).*

percentage of the Filtek Z250 XT material. The data did not provide evidence of a statistically significant difference between the mean gap volume percentage values of the Filtek P90 and Filtek Z250 XT materials for either Class I or Class II.

On the other hand, the results showed a statistically significant difference between the mean values of gap volume percentage of Class I and Class II for Estelite Sigma Quick material, in which the mean gap volume percentage of Class I is significantly higher than that of Class II (p=0.01). However, there was no statistically significant difference in the mean values of gap volume percentages of Class I and Class II of the other two materials.

For the Filtek Z250 XT groups, gaps were seen in the inner and outer half of the walls in Class I restorations, predominantly in enamel margins, and along the axial wall in Class II restorations. The gaps were mainly between the adhesive and tooth structure (Figure 3b through d). Minimal pooling of adhesive was seen (in two specimens). The adhesive layer of the Single Bond Universal was around 70 μm in thickness. Small voids were noted within the composite mass in a few specimens (Figure 3c,d).

Images of the Filtek P90 specimens generally showed gaps on the inner half of the walls of Class I restorations and the inner half of the walls and line angles of Class II restorations. The gaps were mainly within the thick adhesive layer (Figure 4a,b,d) and between the composite and adhesive, predominantly in dentin margins (Figure 4a,e). Small to medium composite voids within the composite mass and pooling of the adhesive in the corners were seen in several specimens (Figure 4c,f). The adhesive layer presented by the Filtek P90 adhesive system was the thickest (around 80 μm) and was most evident on μCT images, in comparison to the other materials tested (Figure 4c,e).

The Estelite Sigma Quick groups showed the best composite adaptation, with minimal voids (Figure

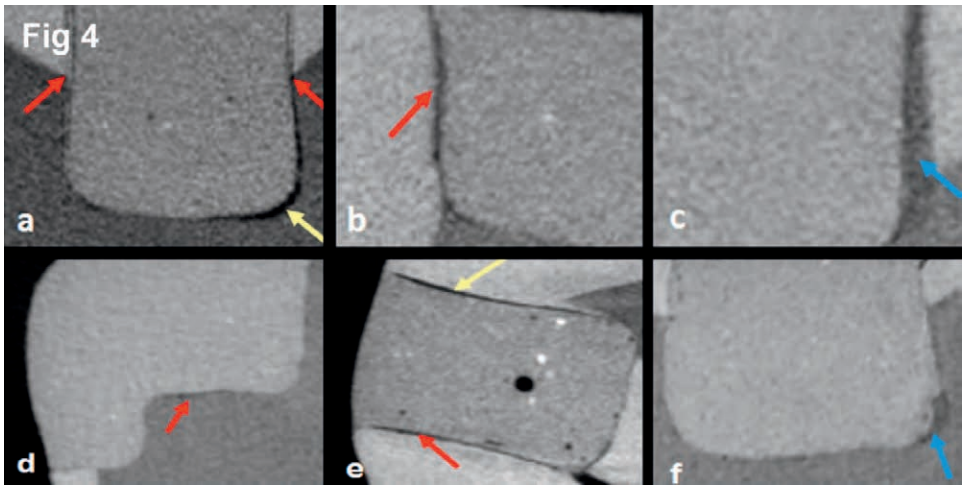

Figure 4. *Representative sections of μCT images taken for Classes I (Figure 4a through 4c) and II (Figure 4d through 4f) Filtek P90 restorations showing the interfacial gap as a separation between composite and Filtek P90 adhesive layer (indicated with yellow arrow), thick Filtek P90 adhesive layer with gaps within the adhesive layer (red arrows), and pooling of Filtek P90 adhesive (blue arrow).*

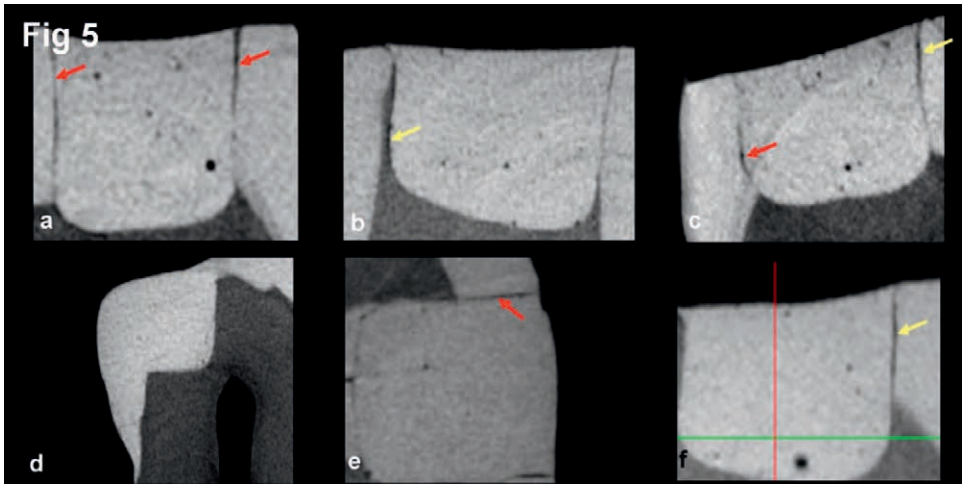

Figure 5. *Representative sections of μCT images taken of Classes I (Figure 5a through 5c) and II (Figure 5d through 5f) Estelite Sigma Quick restorations showing perfect adaptation of the composite, minimal adhesive layer, with interfacial gaps mostly present at the tooth/adhesive interface (yellow arrow) and some interfacial gaps within the adhesive layer (red arrow).*

5a,d), the thinnest adhesive layer (approximately 40 μm), and minimal gap formation. As a result of the minimal thickness of the adhesive layer, it was very difficult to distinguish gap position, but most of the gaps were seen on the walls and line angles between the tooth and adhesive layer (Figure 5b,c,f), with no obvious difference in the incidence of gap occurrence between the enamel and dentin/adhesive interfaces (Figure 5).

Sections of 3D reconstructed images using CT-Vox are displayed in Figures 6 and 7. The colored images show the adhesive layer clearly in yellow, with voids showing as dark brown or black air space.

DISCUSSION

The use of μCT imaging for examination of the internal adaptation of resin-based composite restorations is a new and innovative technology. Compared to traditional techniques that require sectioning of the specimen, μCT represents a precise and nondestructive 3D method that allows both linear and volumetric measurement of the gaps at the composite/tooth interface. In this study, the detection of gap volume (air space) was easy and accurate, and comparisons between different materials were possible. This method allowed 3D visualization of gap distribution, which represents possible pathways of interfacial leakage along the cavity walls; furthermore, it provided concomitant information about the spatial distribution of these gaps as well as the detailed characteristics of the adhesive layer. Therefore, the first null hypothesis can be rejected, as μCT proved to be an effective tool for the evaluation of interfacial gap formation and visualization of adhesive layer.

All of the resin-based composites used in this study demonstrated very low gap volume percentages, with Estelite Sigma Quick having the lowest gap volume in both classes. The Filtek P90 composite had a significantly higher gap volume percentage than did Estelite Sigma Quick. The gaps noted in the Filtek P90 group were mostly due to separations within the adhesive layer and between the adhesive and composite. This is likely due to the nature of the adhesive used, as the Filtek P90 adhesive is a viscous two-component 5%-10% filled adhesive[34] with poor wetting ability that gave rise to a thick adhesive layer, which may have contributed to void incorporation and microbubble formation.[16] This possibility is supported by the presence of voids within the adhesive layer, as opposed to at the cavity/restoration interface. In addition, voids in the adhesive layer act as stress concentration areas that could lead to an increase in the overall stress within the restoration and, ultimately, larger gap formation. The microbubbles seen within the Filtek P90 adhesive were also evident in the confocal microscopy images of D'Alpino and others,[35] who studied the adhesive interface after adding a fluorophore to the adhesive components.

As for the composite/adhesive interfacial separation, our finding is in agreement with previous reports[16,36] that showed adhesive failure of the Filtek P90 bond at the composite/adhesive interface to be the most prominent cause of bond failure with this composite. These authors[16,36] also reported that the higher modulus measured for some low-shrinkage resins like the Filtek P90 composite and its stiffer consistency may lead to air bubble incorporation within the composite mass that is more frequently enclosed near the bottom of the cavity. These pores can provide a free surface for the

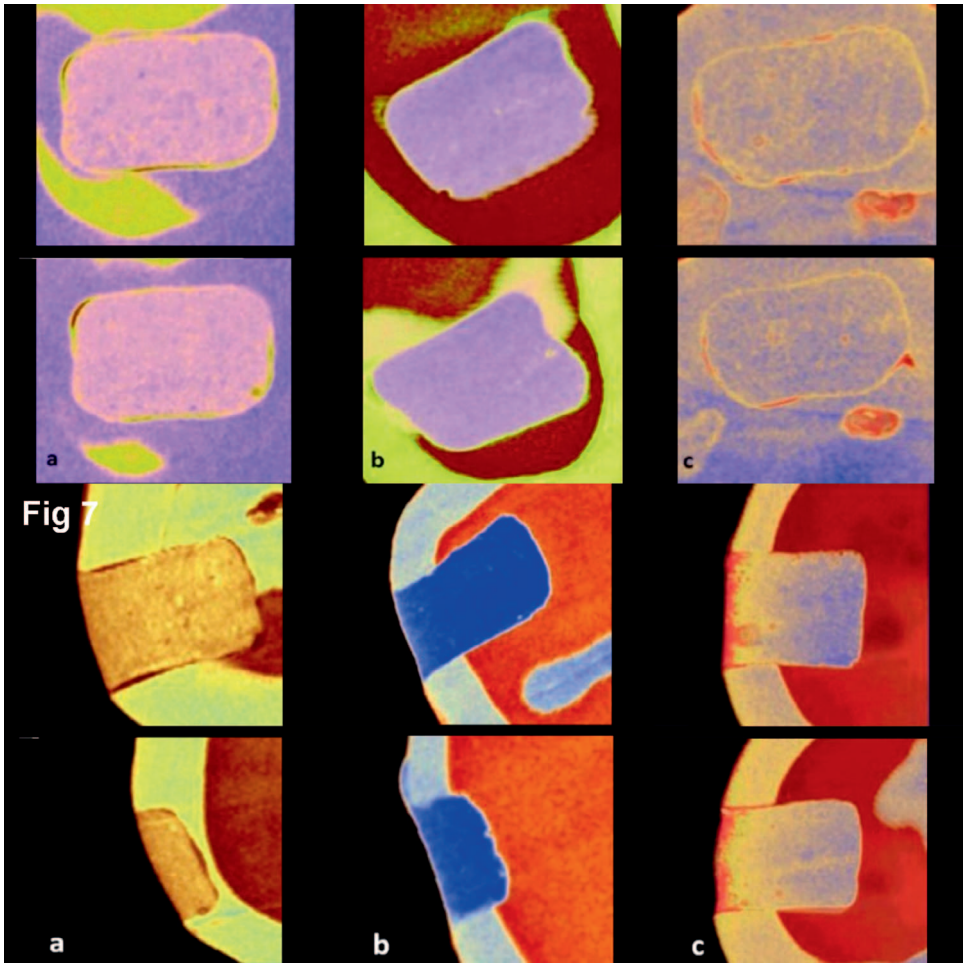

Figure 6. *Sections of Class I restorations using CT-Vox 3D reconstruction program showing clear adhesive layers (yellow), with voids showing as black air space: (a) Filtek Z250 XT; (b) Filtek P90; (c) Estelite Sigma Quick.*

Figure 7. *Sections of Class II restorations using CT-Vox 3D reconstruction program showing clear adhesive layers (yellow), with voids showing as black air space: (a) Filtek Z250 XT; (b) Filtek P90; (c) Estelite Sigma Quick.*

composite to flow and relieve shrinkage stresses,[37] but they can also affect bond strength, as they act as structural discontinuities within the restoration mass.[38]

In contrast, Bond Force self-etching adhesive is a single-component adhesive and has low viscosity, which facilitates wetting of the tooth surface and results in a thinly spread layer with minimal separation between tooth and adhesive. Similarly, Single Bond Universal self-etching adhesive showed minimal gaps within the adhesive layer and between the tooth and adhesive. As the results of this research showed that there was a difference in gap volume percentages among the different kinds of composites used, the second null hypothesis can be rejected.

The ease of manipulation of Estelite Sigma Quick and the low viscosity of the Bond Force adhesive may have contributed to the very small gap size measured with these materials. Although Bond Force is a one-bottle self-etch adhesive, it has shown very promising clinical results even when used on enamel without prior etching and after two years of clinical service.[39] In addition, Estelite Sigma Quick had a thin adhesive layer and the best cavity adaptation that could possibly be due to the low viscosity of this unfilled adhesive and the supra–nano-spherical fillers, which allowed a better adaptation and more efficient sealing of the cavity walls than were permitted by other types of fillers.[34]

Previous reports have looked at the effects of filler load and elastic modulus on polymerization shrinkage and shrinkage stresses. Researchers have reported a positive correlation between the elastic modulus and contraction stress. It has also been established that increasing the filler load results in reduced polymerization shrinkage, higher elastic modulus, and higher shrinkage stresses.[3,40] In this study, the Filtek P90 and Filtek Z250 XT gap volume percentage values were not significantly different from each other. This could be explained by the fact that although Filtek P90 has reportedly low volumetric shrinkage levels, it might have higher stress levels at the interface due to voids in

the adhesive layer and possible void incorporation within the composite mass. Filtek Z250 XT is not a low-shrinkage composite resin, but it is nano-filled with a higher filler content, which may have contributed to its small gap percentage values. As for Estelite Sigma Quick, which had the lowest gap percentage values, it is a low-shrinkage composite with a higher filler content than that of Filtek P90 and a lower elastic modulus (Tables 1 and 2). This combination of properties may have led to reduced contraction stress levels, which could have contributed to the minimal interfacial gap formation with this material.

The volumetric gap measurement is a very promising method with which to examine different composites and monitor their shrinkage behavior. However, gap volume percentage does not necessarily predict future microleakage or possible clinical performance. In this study, the interfacial gaps were detected only on some sections of the restorations and not along the entire walls of the preparation. However, while gaps present areas for potential microleakage, they cannot be used as a precise indicator for it. Sun and others[27] reported that microleakage results predicted by μCT analysis combined with 3D image analysis agreed well with those obtained by dye penetration, but they were not identical. Another study by Frankenberger and others[41] suggested that although *in vitro* testing of dental materials is routine for the preclinical investigation of restoratives because clinical performance cannot be predicted from lab results, clinical trials remain the ultimate instrument. In addition, the authors stated that marginal integrity is reliably predictable in laboratory *in vitro* studies by simulating clinical circumstances. However, marginal analysis of direct restorations *in vitro* is still limited in its ability to determine a lower borderline, and, actually, materials with worse *in vitro* results may still result in acceptable restorations *in vivo*.[41]

The change in cavity configuration between Class I and Class II in the present study changes the total volume from 12 mm^3 to 14 mm^3 and the C-factor from 5 to 4 if the number of walls of the cavity is to be calculated, and it changes it from 4.33 for Class I to 2.17 for Class II if the area of the walls is to be used for the C-factor calculation.[14] The results showed that the Class II restorations (with a smaller C-factor) had lower gap volume percentage measurements in comparison to the Class I restoration, but the difference was not statically significant, except in the Estelite Sigma Quick material. The results prove that the effect of cavity configuration on the gap volume percentages is neither predictable nor consistent. These results are in agreement with those of previous studies that reported no significant effect of the C-factor on gap volume. In 2011, Ghulman[42] suggested that although the microleakage score with silorane tended to increase as the C-factor increased, the effect of the C-factor on the low-shrinkage composite is much less evident than in methacrylate-based composites, as they generally shrink less and induce lower contraction stresses. Another study[43] comparing the microtensile bond strength of different composite materials showed that the increased C-factor had no effect on low-shrinkage composites, including Filtek Silorane, but did cause a significant drop in microtensile bond strength values for conventional methacrylate-based composite.

In the present study, only Estelite Sigma Quick was affected by the difference in the C-factor; therefore, the third null hypothesis is partially rejected. Estelite Sigma Quick has a rapid curing brought about by its unique initiator system, "Radical Amplified Photopolymerization Technology" (RAP), that results in faster stiffness attenuation and possibly high stress levels in restorations with a higher C-factor.[44] Furthermore, Estelite Sigma Quick displayed the thinnest adhesive layer of the materials tested, and it is possible that this contributed to the development of stress through lack of adhesive compensation through flow. Additionally, Estelite has a higher filler content and polymerization contraction than Filtek P90, and this may have led to a higher stress development and reduction of the Estelite's ability to deform elastically.[45]

The results of this study indicate that factors other than polymerization shrinkage and configuration influence the formation of gaps at the tooth/composite interface. Factors that may influence the marginal adaptation are related to the viscosity of the adhesive and the thickness of the adhesive layer. Therefore, reduction of polymerization shrinkage does not inevitably lead to a reduction in overall stress and better adaptation. Gap formation at the margins of a restoration is determined by a complexity of factors related to the material as well as the conditions under which it is placed.

CONCLUSIONS

Based on the results of this study, it can be concluded that

- μCT is a promising tool for evaluation of interfacial gaps of composite restoration as well as the adhesive layer.

- The differences in the C-factor and volume of composite restorations do not always have a pronounced effect on the gap volumes of low-shrinkage composites.
- Estelite Sigma Quick showed the best cavity adaptation and smallest gap volume compared to the two other materials.
- Interfacial gaps formed around composite restorations and within adhesive layers resulted from an interplay of different factors related to the composition of the composite material and adhesive, adhesive layer thickness, and physical properties of the composite resin used.

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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 CDRC. The approval code for this study is NF 2360.

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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Grinding With Diamond Burs and Hydrothermal Aging of a Y-TZP Material: Effect on the Material Surface Characteristics and Bacterial Adhesion

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Clinical Relevance

Finishing of Y-TZP restorations with diamond burs altered the material surface characteristics, but neither the grinding nor an aging condition affected biofilm formation.

SUMMARY

The aim of this study was to evaluate the effect of grinding with diamond burs and low-temperature aging on the material surface characteristics and bacteria adhesion on a yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) surface. Y-TZP specimens were made from presintered blocks, sintered as recommended by the manufacturer, and assigned into six groups according to two factors—grinding (three levels: as sintered, grinding with extra-fine diamond bur [25-μm grit], and grinding with coarse diamond bur [181-μm grit]) and hydrothermal aging—to promote low-temperature degradation (two levels: presence/absence). Phase transformation (X-ray diffractometer), surface roughness, micromorphological patterns (atomic force microscopy),

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and contact angle (goniometer) were analyzed. Bacterial adhesion (colony-forming units [CFU]/biofilm) was quantified using an *in vitro* polymicrobial biofilm model. Both the surface treatment and hydrothermal aging promoted an increase in *m*-phase content. Roughness values increased as a function of increasing bur grit sizes. Grinding with a coarse diamond bur resulted in significantly lower values of contact angle ($p<0.05$) when compared with the extra-fine and control groups, while there were no differences ($p<0.05$) after hydrothermal aging simulation. The CFU/biofilm results showed that neither the surface treatment nor hydrothermal aging simulation significantly affected the bacteria adherence ($p>0.05$). Grinding with diamond burs and hydrothermal aging modified the Y-TZP surface properties; however, these properties had no effect on the amount of bacteria adhesion on the material surface.

INTRODUCTION

Zirconia-based ceramics are a contemporary option for fixed dental prostheses, dental implants and abutments[1] because of their esthetic and superior mechanical strength.[2,3] Among the different types of zirconia-based ceramics, yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) has been highlighted.[4,5] Y-TZP ceramic shows high biocompatibility, chemical stability, and a fracture strength/toughness higher than other ceramic systems.[6] More recently, it has been used to produce monolithic zirconia crowns in posterior teeth.[7]

Zirconia is a polymorphic material that has three crystalline forms that are stable at different temperatures: monoclinic (*m*: up to 1170°C), tetragonal *(t*: above 1170°C and up to 2370°C), and cubic (*c*: above 2370°C).[8] Phase transformation from monoclinic to tetragonal zirconia ($m \rightarrow t$) occurs during the sintering process and is associated with a volume decrease of approximately 4%. After sintering, stabilizing oxides (ie, Y_2O_3) are added to pure zirconia, keeping the tetragonal form stable at room temperature and avoiding the deleterious effects of volume expansion during the cooling process due to $t \rightarrow m$ transformation.[6]

Some factors associated with the clinical use of Y-TZP may also induce a $t \rightarrow m$ transformation of this material, such as intermittent mechanical loading (stress) and corrosion in the presence of humidity (low-temperature degradation [LTD]).[9,10] Additionally, stress concentration with subsequent phase transformation will occur to Y-TZP after adjustment of the Y-TZP surface (outer or intaglio) by grinding and/or polishing.[11] These procedures introduce different types of damage to the Y-TZP surface, such as scratches and cracks of various depths, from the surface toward the subsurface of the material.[12,13]

These damages to the Y-TZP surface (scratches and cracks) may be limited by a phenomenon known as transformation toughening. The $t \rightarrow m$ transformation associated with localized volumetric expansion results in compressive stresses at an existing crack that counteract tensile stresses in this region and limit crack propagation. However, the increase in the transformation area may also result in material loss (grain pullout), a rougher surface, and a higher incidence of cracks, all of which decrease the material strength.[14]

Moreover, grinding with diamond burs produces a modification of the surface characteristics of the Y-TZP material, and this might increase bacterial adhesion[15-19] and favor the incidence of secondary caries and periodontal inflammation,[20] relevant aspects of the longevity of restorations. The restoration surface properties, such as roughness and the surface free energy, seem to play a key role in this process.[21] The surface free energy influences the acquired film formed over the restorative surface.[22,23] The increase in free energy of the substratum surface can result in a higher plaque growth rate and plaque retention capacity of the surface and the selection of specific organisms.[21] Regarding the surface roughness, previous studies suggest that the biofilm is formed in larger amounts and more rapidly on rough surfaces when compared to smooth surfaces.[23] *In situ* studies using scanning electron microscopy revealed that the initial adhesion of microorganisms starts on irregularities and sequentially expands to the rest of the surface.[19] Additionally, previous studies have demonstrated a positive association between the amount of biofilm and the surface roughness in different dental materials, such as ceramics, composite resin, acrylic resin, and titanium.[18,24,25]

Although there is evidence regarding the influence of surface characteristics on bacteria adhesion to restorative materials and the importance of these factors on the longevity of prosthetic restorations, there are no studies that have investigated the effects of grinding with diamond burs and hydrothermal aging (Y-TZP under LTD) on bacterial adhesion (biofilm formation) on a Y-TZP surface. These conditions can be clinically relevant when utilizing a Y-TZP ceramic for implant abutments,

Table 1: *Study Groups*

Groups	Surface Treatment	Low-Temperature Aging
Control	As sintered	Without
Control aging	(untreated)	With
Coarse	Coarse diamond bur #3101G	Without
Coarse aging	(average grit size 181 μm)	With
Xfine	Xfine diamond bur #3101FF	Without
Xfine aging	(average grit size 25 μm)	With

which are placed subgingivally and at areas close to gingival tissues (marginal and connector zones of fixed prostheses). Thus, the present study aimed to evaluate the effect of grinding with diamond burs and hydrothermal aging on the material surface characteristics (*m*-phase transformation, surface roughness, superficial topography, and surface free energy) and bacteria adhesion on a Y-TZP ceramic surface. The null hypothesis (H_0) was that grinding with diamond burs of different grit sizes and hydrothermal aging conditions would yield equivalent bacteria adhesion on the Y-TZP surface.

METHODS AND MATERIALS

Specimen Preparation

Y-TZP specimens (In-Ceram YZ, Vita Zahnfabrik, Bad Sackingen, Germany) were prepared from prefabricated blocks. For the complementary analysis of surface characterization, specimens were manufactured with a final size of 14 × 14 × 2 mm, while for the microbiological evaluation with an *in vitro* biofilm formation model, specimens were used with a final size of 7 × 6 × 2 mm.

To remove the cutting irregularities, the presintered specimens were polished with 1200-grit SiC paper and cleaned in an ultrasonic bath (1440 D, Odontrobras, Ribeirão Preto, Brazil) using 78% isopropyl alcohol for 10 minutes. Then the specimens were sintered as recommended by the manufacturer (Zyrcomat T, Vita Zahnfabrik).

Experimental Groups

After sintering, the Y-TZP specimens were allocated into six groups according to two factors: grinding with diamond burs and low-temperature aging to simulate LTD, as shown in Table 1.

Surface Treatment

Specimens from the control groups (control and control aging) remained untreated after the sintering process. For the other groups, a single trained operator performed the grinding procedures using diamond burs (Xfine #3101FF, 25-μm grit size, and coarse #3101G, 181-μm grit size, KG Sorensen, Cotia, Brazil) coupled with a low-speed motor (Kavo Dental, Biberach, Germany) associated with a contra-angle hand piece (T2 REVO R 170 contra-angle hand piece up to 170,000 rpm, Sirona, Bensheim, Germany) under constant water cooling (≅30 mL/min). The diamond bur was replaced after each specimen.

A marking with permanent marking pen (Pilot, São Paulo, Brazil) was made over the entire surface of each specimen prior to the grinding procedures. Afterward, the specimens were fixed to a device that ensured parallelism between the specimen and diamond bur. Grinding was carried out by similar horizontal movements until the pen mark was eliminated. This protocol standardized the grinding thickness while ensuring that the entire specimen surface was subjected to bur grinding.[26]

Low-Temperature Aging

The hydrothermal aging was simulated in an autoclave (Sercon HS1-0300, no. 1560389/1) at 134°C under 2 bars for 20 hours.[27]

Phase Analysis by X-Ray Diffraction

Quantitative analysis of phase transformation was conducted (one specimen per group) to determine the relative amount of *m*-phase and depth of the transformed layer under each condition. This analysis was performed using an X-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany). Spectra were collected into the 2θ, with a range of 25-35 degrees, at a step interval of 1 second and step size of 0.03 degrees. The amount of *m*-phase was calculated using the method introduced by Garvie and Nicholson:[28]

$$X_M = \frac{(-111)_M + (111)_M}{(-111)_M + (111)_M + (111)_T} \quad (1)$$

where $(-111)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ = 28 degrees and 2θ = 31.2 degrees, respectively) and $(101)_T$ indicates the intensity of the respective tetragonal peak (2θ = 30 degrees). The volumetric fraction of the *m*-phase was calculated according to Toraya and others:[29]

$$F_M = \frac{1.311 \cdot X_M}{1 + 0.311 \cdot X_M} \quad (2)$$

The depth of the transformed layer was calculated based on the amount of the *m*-phase, considering

that a constant fraction of grains had symmetrically transformed to the *m*-phase along the surface, as described by Kosmac and others:[30]

$$PZT = \left(\frac{sen\theta}{2\mu}\right)\left[ln\left(\frac{1}{1-FM}\right)\right] \quad (3)$$

where $\theta = 15$ degrees (the angle of reflection), $\mu = 0.0642$ is the absorption coefficient, and F_M is the amount of *m*-phase obtained using equations 1 and 2.

Surface Roughness and Micromorphological Analysis

Y-TZP specimens were evaluated for quantitative (10 specimens per group) and qualitative (two specimens per group) analysis of the micromorphological pattern generated by the grinding procedure. Specimens were analyzed using a surface roughness tester (Mitutoyo SJ-410, Tokyo, Japan) and atomic force microscopy (AFM, Agilent Technologies 5500 equipment, Chandler, AZ, USA), respectively.

For the roughness analysis, four measurements were made for each specimen (two following the grinding direction and two in the opposite direction) according to the ISO 1997 parameters (Ra, arithmetical mean of the absolute values of peaks and valleys measured from a medium plane [μm], and Rz, average distance between the five highest peaks and five major valleys [μm])[31] with a cutoff (n=5) of λC 0.8 mm and λS 2.5 μm. After that, the arithmetic mean of all measurements from each specimen was obtained.

Afterward, two specimens of each group were randomly selected for qualitative analysis of superficial topography using AFM. First, all selected specimens were submitted to the cleaning protocol in an ultrasonic bath as previously described. The AFM images were obtained by noncontact methodology and specific probes from an area of 20×20 μm (PPP-NCL probes, Nanosensors, force constant = 48 N/m) and evaluation using specific computer software (Gwyddion version 2.33, GNU, Free Software Foundation, Boston, MA, USA).

Contact Angle

The contact angle was measured (10 specimens per group) using the sessile drop technique and a goniometer (DSA30S, Drop Shape Analyzer, KRÜSS, Hamburg, Germany) associated with a computer device using specific software (Advanced Drop Shape Analysis, KRÜSS). For the contact angle measurement, a syringe was used to place a drop (10 μL) of preselected liquid (deionized water) on the treated surface of the specimen, and the contact angle (angle between the drop and the surface plane) was measured after 5 seconds.[32] The software carried out five measurements, and the average value from each specimen was calculated.

Biofilm Model

In vitro biofilms were grown using the Amsterdam Active Attachment (AAA) model.[33] This model consisted of a custom-made stainless-steel lid with 24 clamps in which the substratum was fixed.

Saliva Collection

Stimulated saliva was previously collected from a single donor (DAMD) who refrained from dental hygiene for 24 hours before the collection procedure. The saliva was diluted twofold with 60% sterile glycerol to protect the bacterial cells from cryodamage and stored at −80°C.

Initial Bacterial Attachment

The inoculation medium for the polymicrobial biofilms was 50-fold diluted saliva in a semidefined medium[34] with 0.2% sucrose and 50 mmol/L PIPES at pH 7.0.

Y-TZP specimens (six specimens per group) were fixed in the lid clamps and placed onto standard polystyrene 24-well plates (multiwell plates, Greiner Bio One, Alphen aan den Rijn, Netherlands). Biofilms were produced by adding 1.7 mL of the inoculation medium to each well, and the model was subsequently incubated anaerobically (10% CO_2, 10% H_2, 80% N_2) at 37°C for 6 hours.

Determination of Colony-Forming Units

After allowing for biofilm growth, the specimens with the biofilms were removed from the lid and transferred into 2-mL cysteine peptone water. The biofilms were dispersed by sonication for 2-minutes, 1-second pulsations at an amplitude of 40 W (Vibra Cell, Sonics & Materials Inc, Newtown, CT, USA) and vortex mixing for 30 seconds, and then a series of dilutions were made.

The polymicrobial biofilm suspensions were plated on tryptic soy agar blood plates for total counts. Plates were incubated for 96 hours at 37°C under anaerobic conditions (10% CO_2, 10% H_2, 80% N_2).

Data Analysis

Statistical analysis was executed using SPSS 18. Roughness (Ra and Rz) and contact angle data were

Table 2: *X-Ray Diffractometry Analysis (F_m, % of Monoclinic Phase; PTZ, Depth of Transformed Layer), Roughness (Ra and Rz), and Contact Angle Results for Grinding and Aging Factors*

Groups	Diffractometry Analysis[a]		Ra (μm)[b]	Rz (μm)[b]	Contact Angle[b]
	F_m (%)	PTZ (μm)	Mean (SD)		
Control	0.00	0.00	0.13 (0.02) A	1.17 (0.19) A	81.02 (9.83) A
Control aging	54.38	3.97	0.14 (0.02) A	1.32 (0.27) A	59.55 (8.30) CD
Xfine	8.93	0.47	0.70 (0.21) B	4.56 (0.94) B	75.88 (11.90) AB
Xfine aging	12.72	0.68	0.53 (0.11) C	3.47 (0.65) C	67.71 (9.01) BC
Coarse	10.66	0.57	1.16 (0.14) D	6.87 (0.71) D	53.75 (7.27) D
Coarse aging	19.95	1.12	0.99 (0.08) E	6.11 (0.54) D	60.01 (14.12) CD

[a] *Diffractometry analysis: F_m, % of monoclinic phase; PTZ, depth of transformed layer.*
[b] *Two-way ANOVA and Tukey test. Same letters show no statistical difference between the groups ($p>0.05$). Different letters represent differences between groups ($p<0.05$).*

analyzed using two-way analysis of variance (ANOVA) considering two factors (grinding and aging) and the interaction of both factors. Colony-forming units (CFU)/biofilm counts were compared using one-way ANOVA and the Tukey test. All statistical tests were performed considering a 5% significance level.

RESULTS

Phase Analysis

Surface treatment alone promoted an increase in the *m*-phase content and transformation depth, showing higher values for both the bur grit sizes (Xfine and coarse) when compared to the control (Table 2). Furthermore, all groups showed a higher amount of *m*-phase content and transformation depth after hydrothermal aging, and these differences were more pronounced for the as-sintered control group (*m*-phase from 0% to 54% and depth from 0 to 3.97 μm, respectively).

Surface Roughness and Micromorphological Analysis

The bur grit size directly affected the Ra and Rz parameters on the material surface (Table 2). These results showed an increase ($p<0.05$) in the roughness parameters as a function of increasing bur grit size. Additionally, there was an effect of hydrothermal aging on the roughness parameters for the treated groups (Xfine and coarse groups), with a decrease ($p<0.05$) of the Ra parameter after aging for the Xfine (0.70 to 0.53 μm) and coarse (1.16 to 0.99 μm) groups. There was no difference between control groups, either with or without aging ($p>0.05$).

Micromorphological analysis showed that grinding with a diamond bur (Xfine and coarse) resulted in similar surface patterns, with scratches parallel to the direction of the grinding tool motion and a depth proportional to the grit size of the diamond bur used. The untreated surface showed a distinct micromorphological pattern, with a smoother surface where superficial Y-TZP grains can be seen.

Contact Angle Measurements

The data from contact angle measurements indicated that the surface treatment alone also modified the surface free energy (Table 2). This result indicates that the specimens ground with the coarse diamond bur had significantly lower values of contact angle measurement ($p<0.05$) when compared with the Xfine and control groups. Moreover, hydrothermal aging significantly affected ($p<0.05$) the contact angles values between the control groups (81 to 59 degrees), but no difference ($p>0.05$) was observed between the Xfine and coarse groups. When only the aged groups were compared, the contact angle values showed no significant differences ($p>0.05$) between the groups.

Bacteria Adherence

The bacteria adherence was evaluated using an *in vitro* model of biofilm formation. The CFU/biofilm results showed that neither the surface treatment nor hydrothermal aging simulation significantly affected ($p>0.05$) bacteria adherence on the material surface (Figure 2).

DISCUSSION

In the present study, grinding with diamond burs (Xfine and coarse) promoted higher *m*-phase content when compared to the as-sintered condition (control). Additionally, the grinding procedures altered the superficial topography, roughness, and surface free energy of the Y-TZP ceramic. Regarding aging,

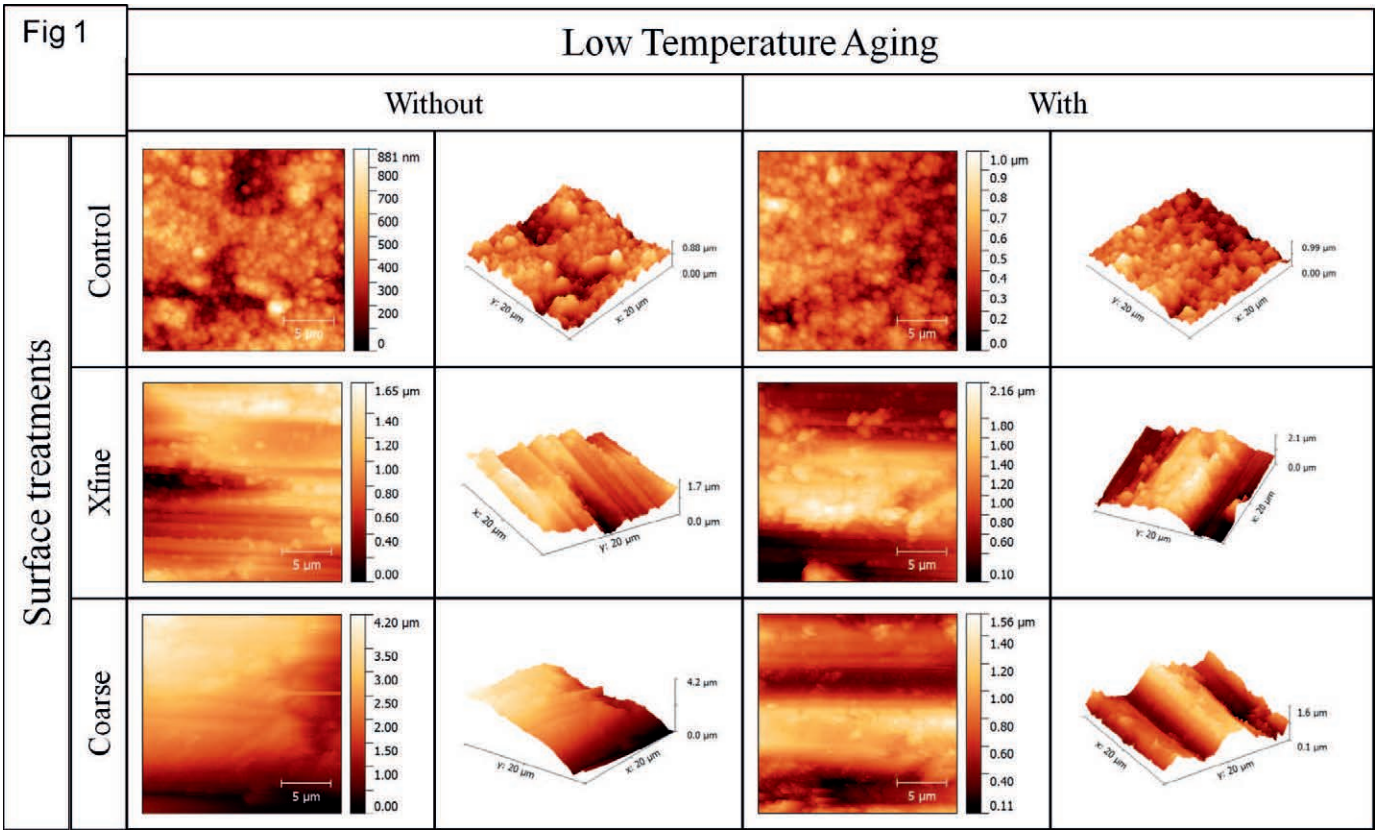

Figure 1. *Atomic force micrographs from zirconia samples of different groups considering the two factors (grinding and aging). It can be noticed that the grinding procedures promoted surface alterations compared to the as-sintered group and that the low-temperature aging did not change the micromorphological pattern.*

distinct effects were observed depending on the presence/absence of grinding. However, despite these differences observed regarding the surface treatment, no significant effect was observed on the bacterial adhesion to Y-TZP surface using an *in vitro* model of biofilm formation.

As aforementioned, some conditions associated with the clinical use of Y-TZP may induce phase transformation ($t \rightarrow m$), such as intermittent loading, humidity, and adjustment by grinding of the Y-TZP surface.[11] In this study, the clinical adjustment was simulated by grinding using diamond burs with different grit sizes (Xfine and coarse), and LTD was artificially induced by hydrothermal aging. In agreement with the literature,[26,35-36] the current data indicate that grinding increased the *m*-phase content (control: 0%; Xfine: 8.9%; coarse: 10.6%), and it decreased the susceptibility of Y-TZP to phase transformation during aging (control aging: 54.3%; Xfine aging: 12.7%; coarse aging: 19.9%). Muñoz-Tabares and Anglada[37] stated that grinding induces a recrystallization of a very thin surface layer of tetragonal nanograins from the highly deformed surface, whose size is smaller than the critical size for phase transformation in a humid environment, such that this process may decrease Y-TZP susceptibility to $t \rightarrow m$ transformation.

Surface topography (AFM images) and roughness examinations (Ra and Rz parameters) were conducted to evaluate the direct effect of grinding on the Y-TZP surface. Roughness results from nonaged groups showed that Ra and Rz values increased with increasing bur grit sizes, and these differences among groups can also be observed in the surface topography images obtained using AFM (Figure 1). The as-sintered condition (control) presented a smoother topographical pattern (zirconia grains at the surface can be seen), and that grinding, regardless of grit size, changed this pattern by introducing scratches and promoting deformations in the direction of the bur movement.

Previous studies have suggested that the increase in the transformation area ($t \rightarrow m$) would result in material loss (by grain pullout) and increasing surface roughness.[10,14,37,38] However, even with the

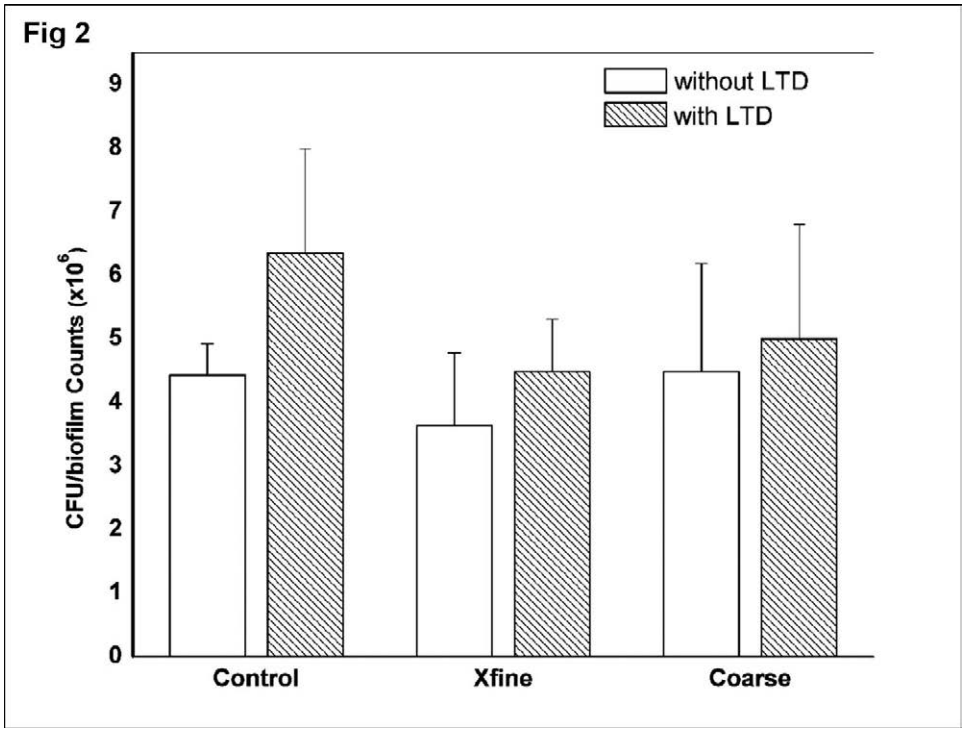

Figure 2. *CFU/biofilm counts of bacteria grown* in vitro *on zirconia surfaces. Two-way ANOVA was performed considering the two factors (grinding and aging) and showed no significant differences between the experimental groups (*p*>0.05). Error bars show the standard deviation from the average value.*

higher *m*-phase content presented after aging for all groups of this study, higher roughness values did not present as a result. Both the Xfine and the coarse groups had lower Ra (Xfine: 70 to 53 μm; coarse: 1.16 to 0.99 μm) and Rz (Xfine 4.56 to 3.47 μm; coarse: 6.87 to 6.11 μm) values after hydrothermal aging, and the difference between the control groups was not significant ($p>0.05$), even with an extensive increase in *m*-phase (0% to 54%). On the other hand, Deville and others[39] stated that $t \rightarrow m$ transformation is triggered preferentially on surrounding areas of superficial defects and residual stress concentration. Thus, it is possible to hypothesize that effects of aging (ie, grain pullout) on ground surfaces occur initially around the highest topographical grains (superficial layer), which are also more susceptible to water contact, resulting in a less rough surface when compared to nonaged ground surfaces. This fact could also be indicative that aging by autoclave for 20 hours at 134°C with 2 bars of pressure was not significant enough to promote the deleterious effects described by Lughi and Sergo[14] on the Y-TZP ceramic used here.

Moreover, the effect of surface roughening on the material surface wettability has been previously reported.[40] In this study, the grinding effect on the contact angle analysis of the Y-TZP surface was observed only for the coarse group, which presented higher surface free energy than the Xfine and control groups. Additionally, it is important to notice that, after aging, there was no difference between the groups regardless of the presence or absence of surface treatment.

The relationship between material surface characteristics and bacteria adhesion has been studied extensively;[40,41] however, few studies have been performed on ground Y-TZP. The understanding of bacteria–surface interactions and how grinding using diamond burs and aging affect biofilm accumulation becomes an important tool for biofilm control and a relevant aspect to preview the longevity of Y-TZP restorations and implant abutments. Regarding the surface characteristics, previous studies have reported that roughness and surface free energy seem to play an important role in the process of bacteria adhesion on restorative surfaces.[40-42] Quirynen and Bollen[23] found that increased surface free energy attracts more bacteria when compared to more hydrophobic surfaces. Likewise, Al-Radha and others[43] concluded that the influence of surface free energy on initial bacterial adhesion to smooth implant materials *in vitro* appears to be the most important factor, in addition to the material type. However, these studies have compared materials with similar patterns of surface roughness. When both the roughness and the surface free energy were evaluated together, the influence of surface roughness on the accumulation and compo-

sition of biofilm is more important than the influence of surface free energy.[44]

In general, an increase in surface roughness promotes an increase in bacterial attachment due to the initial adhesion of bacteria at locations where they are sheltered against shear forces[40] and also because roughening of the surface increases the contact area between the material surface and bacterial cells available for adhesion.[45] It is accepted that an increase in surface roughness above a threshold of 0.2 μm facilitates biofilm formation on restorative materials, while bacterial adhesion to surfaces below the threshold of 0.2 μm cannot be reduced.[46] On the other hand, while both the Xfine and the coarse groups presented Ra values higher than the threshold of 0.2 μm (0.70 and 1.16 μm, respectively), they did not present an increase in bacterial adhesion when compared with the control group (0.13 μm). Hence, it is possible to hypothesize that the range of surface roughness observed in our results is not the main factor for promoting bacterial adhesion on the Y-TZP ceramic *in vitro* and that this low susceptibility to bacterial adhesion can be considered an advantage of this material. This result is in agreement with other studies that indicated that bacteria adhesion cannot be fully explained by small differences in the surface roughness and surface free energy.[47,48]

This inconsistency regarding the effect of surface characteristics on bacteria adhesion on material surface may be explained mainly by 1) characteristics derived from the distinctive materials, such as material chemical composition; 2) the range of roughness promoted on the material surface; and 3) culture conditions used in the tests. In relation to culture conditions, this study evaluated a complex *in vitro* polymicrobial biofilm consisting of diluted-saliva inoculation medium, which differs from other studies with similar purposes that used a single-specimen biofilm, with less varied modes of attachment and without a significant degree of interspecies interactions.[49] The protocol of 6 hours of biofilm growth was chosen in order to evaluate early bacteria adhesion. Additionally, the current study evaluated bacteria adhesion on a Y-TZP surface using the AAA model,[33] a validated and extensively studied polymicrobial model of biofilm formation *in vitro*. However, the use of the AAA model can be considered a limitation of this study, as this *in vitro* model does not simulate some factors from a typical oral environment, such as low shear forces, which can limit the roughness effect on bacteria adherence capacity.

The findings of the current study indicate that grinding with diamond burs and hydrothermal aging modify the surface properties (ie, *m*-phase content, surface roughness, and surface free energy) of the assessed Y-TZP material; however, those properties/characteristics did not significantly affect bacterial adhesion when using the AAA model of *in vitro* biofilm formation. These results suggest that the Y-TZP ceramic may have low susceptibility to bacterial adhesion regardless of the surface condition. However, even if our results have shown no differences between the control and other groups with regard to bacterial adhesion, the surface roughness may affect other properties of the material, such as its mechanical behavior and wear of antagonist teeth, so a smoother surface is clinically preferable. Thus, when clinical grinding is necessary, it should be made using extra-fine diamond burs followed by polishing.[50] Further studies should be performed to provide additional information regarding the behavior of this material using biofilm models that simulate clinical conditions and/or clinical studies to better understand the influence of these factors on the longevity of the prosthetic restorations.

CONCLUSION

- Grinding with diamond burs and hydrothermal aging promoted *m*-phase content, surface roughness, and surface free energy alterations of the assessed Y-TZP material.
- Bacterial adhesion was not affected by grinding with different diamond burs.

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Regulatory Statement

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

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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Adhesive Durability Inside the Root Canal Using Self-adhesive Resin Cements for Luting Fiber Posts

K Bitter • A Maletic • K Neumann • L Breschi • G Sterzenbach • M Taschner

Clinical Relevance: Adhesive luting of fiber posts using self-adhesive resin cements resulted in reliable bond strength values even after aging, although product-specific differences could be observed and qualitative nanoleakage analyses revealed initial degradation effects.

doi: http://dx/doi.org/10.2341/17-017-L

Polymerization Behavior and Mechanical Properties of High-Viscosity Bulk Fill and Low Shrinkage Resin Composites

S Shibasaki • T Takamizawa • K Nojiri • A Imai • A Tsujimoto • H Endo • S Suzuki • S Suda • WW Barkmeier • MA Latta • M Miyazaki

Clinical Relevance: Bulk-fill resin composites may be less suitable than low-shrinkage resin composites for the restoration of large cavities due to their high volumetric shrinkage.

doi: http://dx/doi.org/10.2341/16-385-L

Role of Proteolytic Enzyme Inhibitors on Carious and Eroded Dentin Associated With a Universal Bonding System

MC Giacomini • PMC Scaffa • LP Chaves • CMP Vidal • TN Machado • HM Honório • L Tjäderhane • L Wang

Clinical Relevance: Carious dentin is a challenging substrate with which to establish an effective bond. Using an etch-and-rinse mode with universal adhesive system, stable resin-dentin bond strength over time can be achieved for those substrates. However, the use of E-64 associated with it presented better performance compared with CHX. Thus, it appears to be an appropriate bonding strategy for the maintenance of bond strength to eroded and carious dentin over time.

doi: http://dx/doi.org/10.2341/16-178-L

Effect of Preparation Designs on the Prognosis of Porcelain Laminate Veneers: A Systematic Review and Meta-Analysis

N Hong • H Yang • J Li • S Wu • Y Li

Clinical Relevance: Window-type preparations are recommended for porcelain laminate veneers with a butt-joint considered when incisal coverage is needed.

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Adhesive Durability Inside the Root Canal Using Self-adhesive Resin Cements for Luting Fiber Posts

K Bitter • A Maletic • K Neumann • L Breschi • G Sterzenbach • M Taschner

Clinical Relevance

Adhesive luting of fiber posts using self-adhesive resin cements resulted in reliable bond strength values even after aging, although product-specific differences could be observed and qualitative nanoleakage analyses revealed initial degradation effects.

SUMMARY

Objectives: The aim of the study was to investigate the effects of various self-adhesive resin cements on the push-out bond strengths and nanoleakage expression at the luting interfaces of fiber posts immediately and after one year of aging.

Methods and Materials: One hundred forty-four extracted human anterior teeth were endodontically treated. After post space preparation, fiber posts were luted using five commercially available self-adhesive resin (SAR) cements and a core build-up material applied with a self-etch adhesive (BF: Bifix SE/Rebilda Post, VOCO; CSA: Clearfil SA Cement/Rely X Fiber Post, 3M ESPE; RX: RelyX Unicem 2/Rely X Fiber Post, 3M ESPE; SPC: Speed Cem/FRC Postec, Ivoclar Vivadent; SMC: Smart Cem/X Post, Dentsply; RB: Rebilda DC-Futurabond/Rebilda Post; n=22). For each group, half of the specimens were subjected to thermocycling (TC) (5°C-55°C, 10,000 cycles) and stored humid for one year at 37°C. Push-out bond strength data of six slices (thickness 1 mm) per root and nanoleakage expression of representative specimens were evaluated after 24 hours (baseline) and after TC and storage for one year (aging), respectively.

Results: Bond strength differed significantly among resin cements ($p<0.0005$) and the location inside the root canal ($p<0.0005$), but not by aging ($p=0.390$; repeated-measures analysis of variance). SMC (14.6±5.8 MPa) and RX (14.1±6.8 MPa) revealed significantly higher bond strength compared to BF (10.6±5.4 MPa) and RB (10.0±4.6 MPa) but differed not significantly from SPC (12.8±4.8) MPa; CSA (6.1±4.6

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MPa) revealed significantly lower bond strength compared to all other investigated materials ($p<0.05$; Tukey Honestly Significantly Different). Qualitative nanoleakage analysis revealed more silver deposits at the interface in all groups after aging. For CSA, a large amount of silver deposits inside the cement was also observed at baseline and after aging.

Conclusions: Fiber post luting using SAR cements demonstrated reliable bond strengths. Product-specific differences and initial degradation effects could be demonstrated.

INTRODUCTION

Fiber-reinforced posts are often used for increasing the retention of the coronal restoration of severely destroyed endodontically treated teeth.[1,2] They have been recommended because of their elastic modulus, which is similar to that of dentin,[3] and it has been speculated that stress should be evenly distributed along post, luting cement, and dentin compared to more rigid post materials.[4,5] Conversely, clinical evidence that fiber posts reduce the occurrence of root fractures is missing.[6] Moreover, adhesively luted titanium as well as fiber posts revealed a comparable success rate after seven years in a controlled randomized trial.[7]

Adhesive luting inside the root canal is still challenging because of anatomic variability of the root canal dentin,[8] limited visibility, difficult moisture and application control, and the high C-factor, which might affect durable and stable bonding.[9,10] Consequently, adhesive luting of posts inside the root canal should be as simple as possible.

The most simplified adhesive strategy involves the use of self-adhesive resin cements where no previous application of bonding agents is required. These cements have acid-functionalized monomers, such as 4-methacryloxyethyl trimellitic anhydride and pyromellitic glycerol dimethacrylate, or phosphoric acid groups, such as 2-methacryloxyethyl phenyl hydrogen phosphate, 10-methacryloyloxydecyl dihydrogen phosphate, bis(2-methacryloxyethyl) acid phosphate, and dipentaerythritol pentaacrylate monophosphate, in their composition to achieve bonding to the tooth substrate.[11] As a consequence of the mixing process of self-adhesive resin cements, a pH ranging between 1.5 and 3 is created by the acidic monomers in order to demineralize the dentin.[11] These acidic groups then bind with calcium in the hydroxyapatite to form an ionic attachment between the methacrylate network and dentin. Ions released from the acid-soluble filler neutralize the remaining acidic groups to create a chelate-reinforced three-dimensional methacrylate network. Therefore, these materials become more hydrophobic during the polymerization process,[11] and it has been speculated that these products would be less prone to hydrolytic degradation than are simplified etch-and-rinse systems and self-etch adhesive systems. A recent meta-analysis[12] revealed positive effects of the use of self-adhesive resin cements for luting fiber posts inside the root canal compared to other adhesive strategies. Especially for less experienced operators, the use of this simplified self-adhesive technique seems to be advantageous.[13] However, there is still controversy regarding whether or not this simplified technique for bonding fiber posts is affected by degradation processes inside the root canal.

Previous studies revealed that lower bond strength after artificial aging of self-adhesive resin cements was also dependent on the type of cement, and they attributed this decrease in bond strength to the hydrolytic degradation of the polymer matrix. It was speculated that self-adhesive resin cements may not all adequately shift to neutrality and hydrophobicity, depending on the product used.[14-16] In contrast to that finding, two recent studies[17,18] could not detect any significant reduction of bond strength after artificial aging of self-adhesive resin cements.

Consequently, the aim of this study was to analyze the bond strength of five different self-adhesive resin cements for luting fiber posts—initially and after long-term storage and thermocycling—in comparison to a self-etch adhesive and dual-curing core build-up material. In addition, representative specimens of each group were analyzed using qualitative interfacial nanoleakage analysis. The null hypotheses tested were the following: 1) No differences in bond strength between the different resin cements exist; 2) Long-term water storage for one year and thermocycling do not affect the bond strength of the investigated materials; and 3) Location inside the root canal does not affect the bond strength of the tested materials.

METHODS AND MATERIALS

One hundred and forty-four sound human maxillary central incisors were obtained with written informed consent under an ethics-approved protocol (EA4/102/14) by the Ethical Review Committee of the Charité–Universitätsmedizin Berlin (Germany) and stored in 0.5% chloramine T solution until use. Crowns were removed and root canal preparations were performed at a working length of 1 mm from the apical

foramen using a single length technique with MTwo rotary instruments (VDW, Munich, Germany) by one trained operator. Apical enlargement was performed to a size of 60/.02 using Flex Master rotary files (VDW) combined with irrigation (Endoneedle; Vedefar, Dilbeek, Belgium) using 1 mL of 1% NaOCl solution after every change of file size. The teeth were filled with warm, vertically condensed gutta-percha (Calamus Dual; Dentsply Maillefer, Ballaigues, Switzerland) and AH Plus sealer (Dentsply DeTrey, Konstanz, Germany) and stored in distilled water for 24 hours.

All root canals were enlarged with a slow-speed drill provided by the respective manufacturer of the fiber post systems that are presented in Table 1.

The depth of the post space preparation was 8 mm, leaving at least 4 mm of gutta-percha inside the canal to guarantee an apical seal. The post space was checked for cleanliness using an operating microscope (magnification 23×, OPMI pico; Zeiss, Jena, Germany). The following self-adhesive resin cements were used for luting fiber posts according to the manufacturers' recommendations (Table 1): Bifix SE/Rebilda Post (BF; VOCO, Cuxhaven, Germany), Clearfil SA Cement (Kuraray Dental, Osaka, Japan)/ RelyX Fiber Post (3M ESPE, Seefeld, Germany) (CSA), RelyX Unicem 2/RelyX Fiber Post (RX; 3M ESPE), Speed Cem/FRC Postec Post (SpC; Ivoclar Vivadent, Schaan, Liechtenstein), and Smart Cem/X Post (SmC; Dentsply). The core build-up material Rebilda DC in combination with the self-etch adhesive Futurabond DC and the Rebilda Post (RB; all supplied by VOCO) served as the comparison group. Excess luting material was removed, and light-curing was performed using an LED curing unit (1200 mW/cm^2; Elipar Freelight 2, 3M ESPE) according to the manufacturers' recommendations. A core build-up (height 4 mm) was performed freehand using Rebilda DC (VOCO) in the BF and RB groups, Clearfil Core/New Bond (Kuraray Dental) in CSA and RX groups, Multicore Flow/Adhese DC (Ivoclar Vivadent) in the SpC group, and XP Bond/CoreX Flow (all Dentsply) for the SmC group, according to the manufacturers' recommendation, in order to seal the adhesively luted fiber post and to mimic the clinical situation during aging. Half of the samples from each group were subjected to push-out testing after 24-hour storage in 100% humidity (baseline) and the other half after thermocycling (10,000 cycles, 5°C-55°C) and storage for 12 months at 37°C in 0.9% NaCl solution (aging), which was changed weekly as previously described.[17]

After storage, all roots were cut into six slices of 1-mm thickness perpendicular to the long axis of the tooth using a band saw (Exakt Apparatebau, Noderstedt, Germany). Thickness of the slices was measured using a micrometer screw (Mitutoyo Messgeräte, Neuss, Germany), and the coronal (R1) and apical arc radius (R2) of the post segment were evaluated using a stereomicroscope (DV 4; Zeiss). Push-out testing was performed (Universal Testing Machine Zwick; Roell, Ulm, Germany) at a crosshead speed of 0.5 mm/min. The maximum stress was calculated from the recorded peak load divided by the computed surface. In order to calculate the exact bonding surface, the tapered design of the posts was considered and the formula of a conical frustum was applied, thus: $\pi(R_1 + R_2)\sqrt{(R_1 - R_2)^2 + h^2}$. A conical frustum is a frustum created by slicing the top of a core parallel to the base.

After the push-out test each specimen was observed using a stereomicroscope (DV 4) at 40× magnification to determine the failure mode. A scoring system was applied according to the failure modes: 1) adhesive failures between dentin and luting agent; 2) adhesive failures between post and luting agent, 3) mixed failures, and 4) cohesive failures inside the post.

For qualitative interfacial nanoleakage analysis two further specimens from each group were analyzed (n=2 from each aging interval). The roots were sectioned into six slices (thickness 1 mm) as described above after removing the core build-up. The obtained slices were covered with nail varnish, leaving 1 mm free at the interface, and immersed into 50 wt% ammoniacal silver nitrate ($AgNO_3$) solution for 24 hours; this step was followed by application of a photo development solution for eight hours.[19] Afterward the specimens were dried for one hour, glued with cyanoacrylate onto glass slides (Menzel, Niedersachsen, Germany), and ground under running water using 180-, 600-, 1200-, 2400-, and 4000-grit silicon carbide paper (LS2, Remet; Casalecchio di Reno, BO, Italy).[20] All interfaces were analyzed by light microscopy (E800; Nikon, Tokyo, Japan) at 600× magnification.

Data from the push-out tests were aggregated using the break variables "tooth" and "location." The alpha (Type I) error level was set to 0.05. A repeated-measures analysis of variance (ANOVA) with the interindividual factors "resin" and "thermocycling" (TC), and storage was applied at six (BF, CSA, RX, SpC, SmC, and RB) and two levels (initial bond strength and bond strength after storage) and

Table 1: *Composition of Materials Used, According to Manufacturers*

Adhesive Composition (Lot No.)	Luting Cement (Lot No.)	Composition of Luting Cement	Post Composition and Size (Lot No.)	Core Build-up Material Composition (Lot No.)	Adhesive Approach
Futurabond DC (VOCO): organic acids, Bis-GMA, HEMA, TMPTMA, BHT, ethanol, fluorides, CQ, amine, catalysts (0946262, 0946263)	Rebilda DC (VOCO) (09511232)	Bis-GMA, UDMA, DDDMA, BHT, dibenzoyl peroxide, CQ, silica, barium borosilicate glass ceramic, accelerators	Rebilda Post (VOCO): Solid composite of glass fibers, inorganic fillers, PDMA (0926167) Cor. Diam.: 2 mm Ap. Diam.: 1.02 mm Taper 5.3°	Rebilda DC (VOCO): Bis-GMA, UDMA, DDDMA, BHT, dibenzoyl peroxide, CQ, silica, barium borosilicate glass ceramic, accelerators (09511232)	Self-etch
	BiFix SE (VOCO) 0951036 1003252)	UDMA, GDMA, catalyst, initiator	Rebilda Post (VOCO): Solid composite of glass fibers, inorganic fillers, PDMA (0904227) Cor. Diam.: 2 mm Ap. Diam.: 1.02 mm Taper 5.3°	Rebilda DC (VOCO): Bis-GMA, UDMA, DDDMA, BHT, dibenzoyl peroxide, CQ, silica, barium borosilicate glass ceramic, accelerators (09511232)	Self-adhesive
	Speed Cem (Ivoclar Vivadent) (M31940)	DMA, acidic monomers, barium glass, ytterbium trifluoride, co-polymer, silicon dioxide, initiators, stabilizers, color pigments	FRC Postec (Ivoclar Vivadent): Glass fibers, aromatic and aliphatic DMA, ytterbium trifluoride Cor. Diam.: 2 mm Ap. Diam.: 1 mm Taper 5.18°	Multicore flow DMA, barium glass, ytterbium trifluoride, barium-aluminum-fluorosilicate glass, silicon dioxide, catalysts, stabilizers, pigments (N55968)	Self-adhesive
	RelyX Unicem 2 (3M ESPE) (L427307)	Alkaline and silanated fillers, initiator components, pigments, methacrylate monomers containing phosphoric acid groups, methacrylate monomers, stabilizers	RelyX Post (3M ESPE): Glass fibers (zirconia base), epoxy resin (117680909 127931002) Cor. Diam.: 1.6 mm Ap. Diam.: 0.8 mm Taper 4.58°	Clearfil Core (Kuraray Dental): TEGDMA, Bis-GMA, silanated glass filler, colloidal silica, catalysts, accelerators (41396)	Self-adhesive
	Smart Cem2 (Dentsply) (090330)	UDMA, DMA, TMA, phosphoric acid–modified acrylate resin, PENTA, proprietary photoinitiating system, proprietary self-cure initiating system	X Post (Dentsply): Quartz fibers, epoxy resin (648688B) Cor. Diam.: 1.67 mm Ap. Diam.: 0.8 mm Taper 2.65°	Core X Flow (Dentsply): UDMA, DMA, TMA, barium boron fluoroaluminosilicate glass, CQ, silicon dioxide, benzoyl peroxide (091021)	Self-adhesive
	Clearfil SA Cement (Kuraray Dental) (13BBA, 0013BB)	Bis-GMA, TEGDMA, MDP, hydrophobic aromatic and aliphatic DMA, silanated barium glass filler, DL-camphorquinone, benzoyl peroxide, initiator, pigments	Rely X Post (3M ESPE): Glass fibers (zirconia base), epoxy resin (117680909, 127931002) Cor. Diam.: 1.6 mm Ap. Diam.: 0.8 mm Taper 4.58°	Clearfil Core (Kuraray Dental): TEGDMA, Bis-GMA, silanated glass filler, colloidal silica, catalysts, accelerators (41396)	Self-adhesive

Abbreviations: Ap. Diam., apical diameter; BHT, butylhydroxytoluene; Bis-GMA, bisphenol A diglycidyl methacrylate; Cor. Diam., coronal diameter; CQ, camphorquinone; DMA dimethyl ammonium; DDDMA, dodecandiol-dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; GDMA; glycerin dimethacrylate, MDP, 10-methacryloyloxydecyl dihydrogen phosphate; PENTA; dipentaerythritol pentaacrylate monophosphate; PDMA; polydecyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; TMA; trimethyl aluminum; TMPTMA, trimethylolpropane; UDMA, urethane dimethacrylate

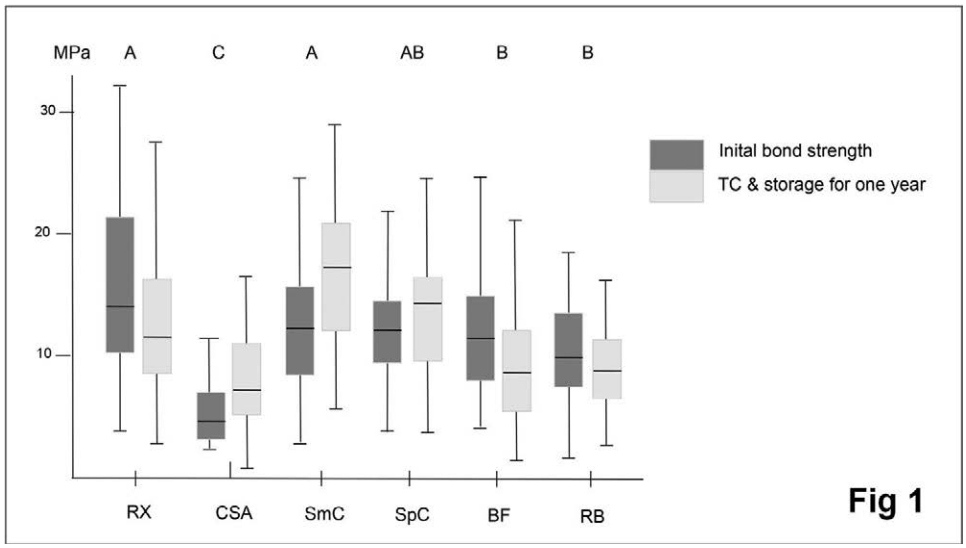

Figure 1. *Box plot demonstrating initial bond strength values as well as bond strength after TC and storage for all investigated materials. Uppercase letters indicate significant differences between materials for pooled data for baseline and aging (*$p<0.05$*; Tukey Honestly Significantly Different).*

location as intraindividual factor (coronal, middle, apical) was applied (SPSS, version 21.0, Chicago, IL, USA). Analysis of the failure modes was conducted using crosstabs and a Chi-square test.

RESULTS

Bond strength was significantly affected by the resin cement ($p<0.0005$) and the location inside the root canal ($p<0.0005$), but not by aging ($p=0.390$; repeated-measures ANOVA). A significant interaction was observed between the factors of resin cement and aging ($p<0.0005$; ANOVA).

Overall, SmC (14.6±5.8 MPa) and RX (14.1±6.8 MPa) revealed significantly higher bond strength compared to BF (10.6±5.4 MPa) and RB (10.0±4.6 MPa) but did not differ significantly from SpC (12.8±4.8 MPa); CSA (6.1±4.6 MPa) revealed significantly lower bond strength compared to all other investigated materials ($p<0.05$; Tukey Honestly Significantly Different) (Figure 1).

With respect to the significant interaction between the factors resin cement and aging, Figure 1 illustrates that bond strength was slightly decreased in groups RX, BF, and RB, whereas it was slightly increased in groups CSA, SmC, and SpC.

Bond strength was significantly affected by the location inside the root canal ($p<0.0005$; ANOVA) and demonstrated significantly higher bond strength in the coronal part of the root canal (12.4±6.8 MPa) compared to the middle (10.7±5.8 MPa) and apical (11±5.5 MPa) parts. No significant interaction for the factors of location and material has been observed ($p=0.104$; repeated-measures ANOVA).

The failure mode was significantly affected by the resin cement initially ($p<0.0005$) and after aging ($p=0.002$; Chi-square test) (Table 2). Most failures occurred between resin cement and dentin, with an increase of this failure mode after aging for four out of six groups.

Qualitative interfacial nanoleakage analyses (Figure 2a-l) showed an increasing amount of nanoleakage after one year, mainly on the dentin/cement interface for all groups. BF and RB exhibited slightly more silver deposits after aging than did RX, SmC, and SpC. For CSA, interfacial nanoleakage occurred more than in other groups, even inside the luting material at baseline and after aging (Figure 2c,d).

DISCUSSION

The first null hypothesis of the present study was rejected because the type of resin cement significantly affected bond strengths of fiber posts inside the root canal. Product-specific differences in bond strengths of self-adhesive resin (SAR) cements inside the root canal have also been reported previously.[14]

Three SAR cements revealed significantly higher bond strength values in the present study compared to a dual-cure core build-up material applied in combination with a self-etch adhesive. These data are in agreement with those of a previous meta-analysis[12] that also favored the use of SAR cements for luting fiber posts. Less technique sensitivity as well as moisture tolerance of SAR cements were supposed to contribute to the good performance of SAR cements inside the root canal. However, product-specific effects on bond strengths have been observed, and the investigated SAR cement CSA

Table 2: *Failure Modes of the Investigated Materials with Respect to Artificial Aging*

Resin Cement	Aging	Failure Modes, %				
		Adhesive Cement/Post	Adhesive Cement/Dentin	Mixed	Cohesive Post	Not Detectable
RX	Baseline	1.7	60.0	11.7	26.7	0
	Aging	3.3	73.3	20.0	1.7	1.7
CSA	Baseline	16.7	30.0	51.7	1.7	0
	Aging	8.3	63.3	26.7	1.7	0
SmC	Baseline	1.7	46.7	43.3	8.3	0
	Aging	3.3	70.0	15.0	6.7	5.0
SpC	Baseline	1.7	63.3	6.7	28.3	0
	Aging	10.0	60.0	23.3	6.7	0
BF	Baseline	0	75.0	21.7	3.3	0
	Aging	3.3	60.0	35.0	1.7	0
RB	Baseline	6.7	28.3	60.0	5.0	0
	Aging	6.7	45.0	48.3	0	0

Abbreviations: BF, Bifix SE; CSA, Clearfil SA Cement; RB, Rebilda DC/Futurabond DC; RX, RelyX Unicem 2; SmC, Smart Cem; SpC, Speed Cem.

seemed to benefit from additional adhesive application.[21]

Matrix composition of resin-based materials influences the degree of conversion and cross-link density and therefore may affect stress development during polymerization.[22] A previous study[23] that evaluated contraction stress, microhardness, and degree of conversion of three SAR cements revealed for Clearfil SA Cement significantly lower contraction stress compared to that associated with RelyX Unicem, but also significantly lower microhardness and degree of conversion. This may clarify the occurrence of low bond strength values of this cement obtained in the present study, in which the predominantly occurring failure mode for the initial samples was mixed (possibly indicating low mechanical properties of this cement). Moreover, interfacial nanoleakage analysis revealed a remarkably higher degree of interfacial silver deposits for the CSA, which is well correlated to the significantly lower bond strength values of this material. The polymer matrix of SAR cements becomes more hydrophobic over the setting time as a result of interactions with calcium ions from the tooth surface and alkaline filler particles.[11,24] The pH neutralization behavior shows a wide variability among SAR cements[25] and influenced significantly flexural strength and the degree of water sorption in a reverse manner.

Another study[26] investigated micromechanical properties of various SAR cements and revealed significantly higher Vickers hardness values for RelyX Unicem compared to Smart Cem 2, which again showed higher values compared to Clearfil SA Cement. The modulus of elasticity of Clearfil SA cement was also significantly lower compared to that of RelyX Unicem.[26] These results demonstrate high variation in micromechanical properties of SAR cements that might have also affected bond strength values in the present study.

In the present study, four different fiber posts from different manufacturers have been used (Table 1). This might have affected the results of the present study. However, when testing a post-and-luting system of one manufacturer, possible incompatibilities between post and luting material should be excluded, and the full potential of each system under laboratory conditions can be assessed.[27] Nonetheless, chemical interaction of co-polymerizing between methacrylate-based resins of the luting agents and highly cross-linked epoxy resin matrices of the posts is less likely.[28] Recently published data indicate that the degree to which both micromechanical interlocking and chemical bonding contribute to the bond strength between fiber posts and resin cements is currently not known.[29] However, it is assumed that micromechanical interlocking that is basically depending on the post-surface topography might be the most contributing factor.[29] Moreover, data from the literature[15,27,30,31] indicated that failure occurs frequently at the interface between root canal dentin and luting agent. The most frequent failure modes observed in the present study were also adhesive failures at the dentin/luting agent interface and mixed failures, and, thus, minimal effects of the various post types used on bond strength can be assumed.

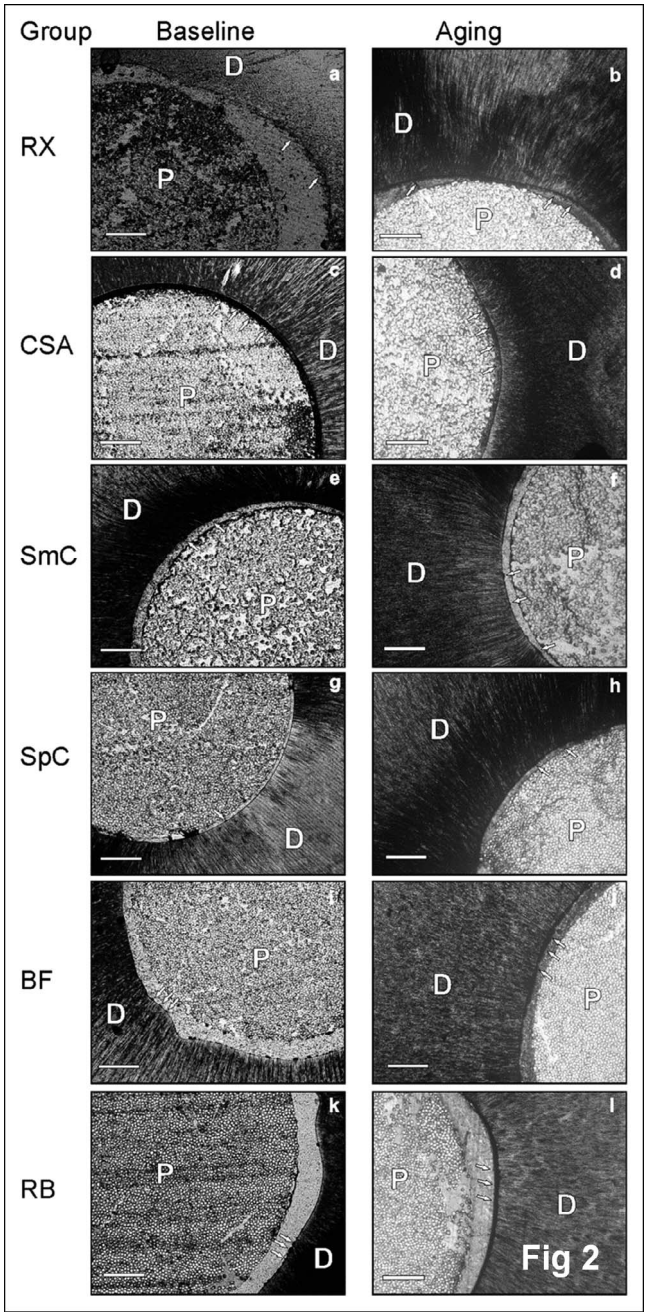

Figure 2. *Light microscopy images (original magnification, 200×) showing representative areas of the post/cement/dentin interface. At baseline a slight amount of silver deposits could be detected, mainly at the dentin/cement interface, for most of the groups (a,e,g,i,k); CSA demonstrated a large amount of silver staining at the interfaces and inside the luting cement at baseline and after aging (c,d). After aging, a slight increase in nanoleakage could be observed in all other groups (b,f,h,j,l). P, fiber post; D, dentin; white arrows indicate presence of silver nitrate at the luting interfaces; white bar = 100 μm.*

The second null hypothesis of this investigation can be accepted, because aging did not affect the bond strength of the investigated materials in the present study, although a significant interaction between these two factors has been observed. The present study subjected whole roots with a core build-up to aging. Previous studies[14,15] subjected slices of samples directly to TC and storage and revealed a significant decrease of bond strength for SAR cements. In general, hydrolytic degradation of the adhesive interface due to fluid movement through dentinal tubules together with residual water from the respective adhesive technique are considered the main factors responsible for the decrease in bond strength over time.[32,33] However, direct intraoral exposure of the post-composite-dentin interface is usually avoided by immediately restoring the tooth, which means that water is not in direct contact with the bonded post.[34,35] Therefore, for the present study design, we decided to subject whole roots with an adhesive core build-up to aging in order to mimic the clinical situation. Nonetheless, thermomechanical loading would have come closer to the clinical situation, but sample preparation in this case would have required crown restorations,[36] which is supposed to hamper specimen preparation for the push-out testing.[37] The use of whole roots might partially explain the results of the present study, in which no overall effects on bond strength of aging could be observed. Nonetheless, a significant interaction between the factors of aging and luting cement could be detected, again indicating product-specific differences between the investigated SAR cements. This effect has been demonstrated previously[38] with respect to the sorption characteristics of various SAR cements in a study in which Smart Cem 2 revealed significantly higher sorption after 168 hours of storage in distilled water when compared to RelyX Unicem 2. Water sorption and hygroscopic expansion are positively correlated for resin-based filling materials.[39] Notably, hygroscopic expansion due to water sorption after long-term incubation using SAR cements as core build-up material was considered to be reasonable as a means by which to provoke crack propagation in lithium disilicate crowns.[40] Thus, a higher hygroscopic expansion could have possibly contributed to the increase of bond strength of Smart Cem 2 after TC and storage in the present study. However, an increased amount of silver deposits has been observed in exemplary samples after aging, indicating that degradation of the interface occurred but did not result in bond strength decrease at the investigated stage.

The third null hypothesis must be rejected since bond strength was significantly affected by the location inside the root canal. The coronal part of the root canal revealed significantly higher bond

strength compared to the other regions inside the root canal, which is in accordance with the findings of previous studies,[17,41] and a previous review[42] summarized that application of adhesive techniques in the apical part of the root canal is less predictable.

The thin push-out test is considered a valid method with which to analyze bond strengths of fiber posts to root canal dentin because the test demonstrated a more homogeneous stress distribution by finite element analysis, when compared to the microtensile bond strength, and less variability in mechanical testing.[43] Nevertheless, the contribution of friction to the detected bond strength values cannot be excluded.[44] If higher hygroscopic expansion leads to increased push-out values, the influence of friction will become more relevant, and correct positioning of the punch pin as well as the use of a pin that is only slightly smaller than the post is of importance. Therefore, we used three different pins according to the post diameter of the respective slice.

Qualitative interfacial nanoleakage analysis demonstrated a large amount of silver deposits even inside the luting material for CSA in combination with the lowest bond strength data, which might be explained with a lower degree in conversion (DC).[23,45] A lower DC is mainly combined with a weaker cross-linked polymer network, resulting in a material being more prone to hydrolytic degradation. Insufficient curing and residual uncured monomers of the material CSA might have resulted in silver staining inside this material at baseline. This phenomenon has been observed previously for CSA for the chemical-cure mode[46] and related to residual water on the dentin surface that affected the integrity of the SAR cement and created defects within the matrix. This might also be an explanation for the low bond strength data in the present study. A higher amount of silver uptake inside insufficiently cured materials has been also demonstrated previously.[47]

CONCLUSIONS

Within the limitations of this *in vitro* study it can be concluded that the push-out bond strength of the tested SAR cements and the control group of a core build-up material were durable after aging for one year. However, bond strength depended on the specific material compositions and characteristics that might have an impact on push-out bond strength values.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Charité–Universitätsmedizin Berlin, in Berlin, Germany.

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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Polymerization Behavior and Mechanical Properties of High-Viscosity Bulk Fill and Low Shrinkage Resin Composites

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Clinical Relevance

Bulk fill resin composites may be less suitable than low-shrinkage resin composites for the restoration of large cavities due to their high volumetric shrinkage.

SUMMARY

The present study determined the mechanical properties and volumetric polymerization shrinkage of different categories of resin composite. Three high viscosity bulk fill resin composites were tested: Tetric EvoCeram Bulk Fill (TB, Ivoclar Vivadent), Filtek Bulk Fill posterior restorative (FB, 3M ESPE), and Sonic Fill (SF, Kerr Corp). Two low-shrinkage resin composites, Kalore (KL, GC Corp) and Filtek LS Posterior (LS, 3M ESPE), were used. Three conventional resin composites, Herculite Ultra (HU, Kerr Corp), Estelite ∑ Quick (EQ, Tokuyama Dental), and Filtek Supreme Ultra (SU, 3M ESPE), were used as comparison materials. Following ISO Specification 4049, six specimens for each resin composite were used to determine flexural strength, elastic modulus, and resilience. Volumetric polymerization shrinkage was determined using a water-filled dilatometer. Data were evaluated using analysis of variance followed by Tukey's

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honestly significant difference test (α=0.05). The flexural strength of the resin composites ranged from 115.4 to 148.1 MPa, the elastic modulus ranged from 5.6 to 13.4 GPa, and the resilience ranged from 0.70 to 1.0 MJ/m^3. There were significant differences in flexural properties between the materials but no clear outliers. Volumetric changes as a function of time over a duration of 180 seconds depended on the type of resin composite. However, for all the resin composites, apart from LS, volumetric shrinkage began soon after the start of light irradiation, and a rapid decrease in volume during light irradiation followed by a slower decrease was observed. The low shrinkage resin composites KL and LS showed significantly lower volumetric shrinkage than the other tested materials at the measuring point of 180 seconds. In contrast, the three bulk fill resin composites showed higher volumetric change than the other resin composites. The findings from this study provide clinicians with valuable information regarding the mechanical properties and polymerization kinetics of these categories of current resin composite.

INTRODUCTION

The mechanical properties of resin composites have improved following the development of superior filler components and resin monomers.[1] However, one of the major shortcomings of resin composites is the shrinkage that accompanies polymerization.[2,3] In addition, the viscosity of the resin composites increases during the polymerization process, leading to increased stiffness of the material.[4,5] As a result, contraction stress occurs not only within the material but also at the bonded interface.[3] It is thought that this morphologic alteration leads to negative sequelae, such as postoperative sensitivity, contraction gaps, microcracks in the tooth structure, cuspal deformation, and recurrent caries.[6-8] To reduce the effects of polymerization shrinkage and contraction stress, various clinical approaches have been attempted. For instance, a sandwich technique with glass-ionomer cements,[9] incremental filling techniques,[10,11] and the use of low-viscosity flowable resin composites[12,13] have been advocated. However, these techniques increase the number of clinical steps and may increase technique sensitivity.

Polymerization of light-cured resin composites is influenced by a wide range of factors, including shade, light absorption and dispersion within the resin composite, types of fillers and monomer, and light irradiance conditions.[14] In particular, resin monomers have a decisive influence on the characteristics of polymerization.[15,16] In general, during the course of polymerization, the intermolecular separations between the dimethacrylate monomers are reduced due to the conversion of carbon–carbon double (C=C) bonds, resulting in volumetric shrinkage.[2,17] Hence, over recent decades, research and development have focused on creating and adopting new resin monomers to reduce polymerization shrinkage and contraction stress. Recently, new resin monomers with increased molecular weight and a small number of C=C bonds[18,19] or cationic polymerization[20] have been introduced in several low-shrinkage resin composite products.[21]

It should be noted that other trends in resin composite development are also of interest. To reduce contraction stress and the number of clinical steps, bulk fill resin composites have recently been marketed. Bulk fill resin composites are intended to allow placement in one layer of up to 4 mm thickness with adequate polymerization.[22-24] In addition, these composites have a short activation time due to the presence of modified initiation systems[25] and increased translucency owing to decreased filler load and increased filler size.[26] Although there is no consensus on the classification, bulk fill resin composites are described either as low-viscosity that are similar to flowable resin composites or as high-viscosity that resemble conventional resin composites.[27] However, *in vitro*-simulated wear studies showed that bulk fill resin composites have lower wear resistance compared with conventional resin composites.[28] Therefore, independent evaluations of the materials are important if dental professionals are to make an informed choice of the optimal product.

There have been many studies investigating the mechanical properties and polymerization kinetics of low-shrinkage[18,19,21,29,30] and bulk fill resin composites.[22-27,31,32] However, few studies have compared the mechanical properties and polymerization behaviors of different types of resin composite under the same conditions. The purpose of this study was to assess the mechanical properties and volumetric polymerization shrinkage of currently available resin composites, including bulk fill and low-shrinkage resin composites, and to compare their characteristics with those of the conventional resin composites. The null hypothesis to be tested was that there are no significant differences in mechan-

Table 1: *Materials used in this study*

Code	Resin composite (shade: lot no.)	Main components	Manufacturer
TB	Tetric EvoCream Bulk Fill (Shade; A2: T21387)	bis-GMA, UDMA, ytterbium trifluoride, Ba glass filler, mixed oxide, prepolymer filler, EBPADMA, additives, catalysts, stabilizers, pigments	Ivoclar vivadent
FB	Filtek Bulk Fill posterior restorative (Shade; A2: N701975)	silane treated creamic, aromatic UDMA, ytterbium fluoride, UDMA, silane treated silica, DDDMA, silane treated zirconia, water, modified methacrylate monomer, EDMAB benzotriazol	3M ESPE
SF	SonicFill (Shade; A2:5037026)	poly(oxy-1,2-ethanediyl), α, α'- [(1-methylethylidene)ßdi-4, 1-phenylene]bis [ω-[(2-methyl-1-oxo-2-propen-1-yl)oxy]-2,2′ -ethylenedioxydiethyl dimethacrylate, bis-GMA, TEGDMA, bis-EMA, barium glass, silica, rheological modifier	Kerr Corp
KL	Kalore (Shade; A2: 9EP)	ytterbium trifluoride, UDMA, bis-EMA, DX-511, dimethacrylate, BHT, silane	GC Corp
LS	Filtek LS (Shade; A2: 9EP)	silane treated quartz, ytterbium trifluoride 3,4-epoxycyclohexylcyclopolymethylsioxane, Bis-3,4-epoxycyclhexylethyl-phenyl-methylsilane, Borate (1-), thtrakis (pentafluorophenyl) -[4-(methylethyl) phenyl] (4-methylphenyl) iodonium	3M ESPE
HU	Herculite Ultra (Shade; EA2: 4495606)	bis-GMA, TEGDMA, bis-EMA barium glass, silica, prepolymerized filler	Kerr Corp
EQ	Estelite ∑ Quick (Shade; A2:154095P)	bis-GMA, TEGDMA, silica-zirconia filler, dibutyl hydroxy toluene	Tokuyama Dental
SU	Filtek Supreme Ultra (Shade; BA2: N339152)	bis-GMA, bis-EMA, UDMA, TEGDMA, PEGDMA, silica nanofiller, zirconia/silica Aggregated zirconia/silica clusters	3M ESPE

Abbreviations: BHT, butylated hydroxytoluene; bis-EMA, bisphenol A polyethethylene glycol diether dimethacrylate; bis-GMA, 2, 2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]propane, DDDMA, 1,12-dodecane dimethacrylate; EBPADMA, ethyoxylated bisphenol A dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate; PEGDMA, poly ethethylene glycol diether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate.
A Sonic Fill Handpiece 2010 (lot no. 1007885) was used for insertion of the SonicFill material.

ical properties and polymerization behaviors among the three different types of resin composites.

METHODS AND MATERIALS

Three high-viscosity bulk fill resin composites were tested: Tetric EvoCeram Bulk Fill (TB, Ivoclar Vivadent, Schaan, Liechtenstein), Filtek Bulk Fill posterior restorative (FB, 3M ESPE, St Paul, MN, USA), and SonicFill (SF, Kerr Corp, Orange, CA, USA). Two low-shrinkage resin composites, Kalore (KL, GC Corp, Tokyo, Japan) and Filtek LS Posterior (LS, 3M ESPE), were used. Three conventional resin composites, Herculite Ultra (HU, Kerr Corp), Estelite ∑ Quick (EQ, Tokuyama Dental, Tokyo, Japan), and Filtek Supreme Ultra (SU, 3M ESPE), were used as comparison materials. The test materials and their components are listed in Table 1. To avoid any influence from the reported nonuniformity of LED curing units,[33,34] a quartz-tungsten-halogen curing unit was used (Optilux 501, sds Kerr, Danbry, CT, USA) with a curved light guide (type 20812; diameter, 13 mm; sds Kerr), and the light irradiance (average 600 mW/cm^2) of the curing unit was checked using a dental radiometer (Model 100, Kerr).

Inorganic Filler Content

The inorganic filler content of the materials was measured using thermogravimetry and differential thermal analysis (TG/DTA6300, Seiko Instruments, Tokyo, Japan). For each resin composite tested, resin paste (50 mg) was placed in a cylindroid crucible of pure platinum (7 mm in diameter, 10 mm in depth) and heated in the thermogravimeter from 25°C to 800°C at a heating rate of 10°C/min under atmospheric air until the organic components were completely incinerated. The weight of the residual resin paste was automatically measured by the built-in horizontal differential balance, and the inorganic filler content (wt%) was calculated using the compensated blank curve. Five measurements per test material were conducted to obtain an average inorganic filler content (wt%).

Coefficient of Linear Thermal Expansion

The coefficients of linear thermal expansion of the test materials were measured using a thermomechanical analyzer (TMA/SS 6300, Seiko Instruments). For each resin composite tested, the resin paste was condensed into a cylindrical Teflon (Sanplatec Corp, Osaka, Japan) mold (8.0 mm in

high, 3.0 mm in diameter). Light irradiance was performed at both the top and bottom of the mold for 40 seconds; the mold was then cut with a sharp scalpel and removed from the cured resin composite. To ensure full polymerization, the center of the specimen was irradiated perpendicular to the axis for 40 seconds; this was then repeated on the opposite side of the specimen. The specimens were stored under dark conditions at 25°C for 24 hours before the measurements were conducted. Each specimen was placed on the stage of the chamber, and the thermomechanical analyser probe contacted the top surface of the specimen at 50-mN load stress. Five specimens were prepared for each material and separately tested at a heating rate of 2°C/min from 25°C to 130°C under atmospheric air. Measurements were performed four times per specimen, but the first measurement outcome was discarded as unreliable. The average coefficient of linear thermal expansion ($\times 10^{-6}$/°C) was measured over a temperature range of 50°C (from 30°C to 80°C), the same range width as that used in standard thermal cycling experiments, starting at a low temperature of 30°C to avoid any influence of fluctuations in ambient temperature.

Flexural Properties

Flexural properties of the resin composites were tested according to the International Standards Organization Standard 4049.[35] Each resin composite was compacted into a stainless-steel split mold with dimensions $25 \times 2 \times 2$ mm and positioned on a glass slide. The middle third of the specimen was first irradiated for 30 seconds, after which the remaining thirds were irradiated for 30 seconds each. The opposite side of the specimen was irradiated in the same manner. After the removal of the hardened specimen from the mold, all six sides were wet polished with #1200 silicon carbide (SiC) paper (Fuji Star Type DDC, Sankyo Rikagaku Co, Ltd, Saitama, Japan). The specimens were then stored for 24 hours in distilled water at 37°C before conducting the mechanical tests. Six specimens per test group were subjected to a three-point bending flexural strength test using a universal testing instrument (Type 5500R, Instron Corp, Canton, MA, USA) at a cross-head speed of 1.0 mm/min until the specimen fractured. The specimens were positioned on a three-point bending apparatus with a span length of 20.0 mm. Flexural strength (S) and modulus of elasticity (E) were determined from the stress–strain curve obtained using the Bluehill Ver 2.5 computer software (Instron Corp) linked to the testing instrument. The modulus of resilience (R) was calculated using the following equation by Peutzfeldt & Asmussen[36]: $R = S^2/2E$.

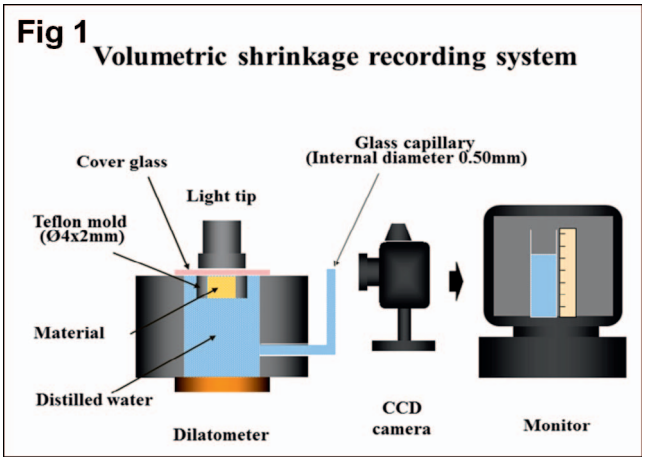

Figure 1. *Diagram of the volumetric polymerization shrinkage recording system.*

Volumetric Shrinkage Measurement

For volumetric polymerization shrinkage measurements, the test apparatus was composed of a water-filled dilatometer and a capillary tube (uniform diameter, 0.5 mm; length, approximately 130 mm). It was attached to a 25 cm^3, brass-bottomed density bottle using a ground glass joint. A diagram of the volumetric shrinkage recording system is shown in Figure 1. The density bottle was filled with distilled water. During the course of testing, the temperature of the distilled water was maintained by placing the density bottle on a thermostatically controlled plate to avoid the influence of temperature changes in the water.

Resin composite paste was placed into a Teflon mold (4.0 mm diameter $\times$ 2.0 mm height) and covered by a 0.5-mm-thick glass plate. The curing light tip end was placed in a hole on the glass lid, and the specimen was light irradiated for 30 seconds. The change in the height of the water meniscus was recorded using a CCD camera (DS-505, Nikon Corp, Tokyo, Japan) every five seconds from light irradiation to 180 seconds and projected onto a VCR (CT-1450, Hitachi Corp, Tokyo, Japan) from the start of light irradiation. A period of 180 seconds was chosen because polymerization shrinkage tends to plateau after about 120 seconds, and water absorption affects volume over longer periods. Measurement accuracy of the water meniscus was 0.2 mm.

Volumetric shrinkage (ΔV) of the specimens was calculated from the change in the height of water

Table 2: *Inorganic filler contents and thermal expansion*

Code	Inorganic filler content (wt%)	Tukey group	Coefficient of linear thermal expansion	Tukey group
TB	77.1 (0.4)	b	42.2 (0.8)	c
FB	75.1 (0.5)	c	48.9 (0.7)	a
SF	81.3 (0.5)	a	46.1 (0.4)	b
KL	69.0 (0.4)	e	36.6 (0.6)	e
LS	77.0 (0.4)	b	33.5 (0.9)	f
HU	67.2 (0.3)	f	39.0 (1.1)	d
EQ	67.6 (0.4)	f	41.8 (0.6)	c
SU	73.7 (0.5)	d	38.0 (0.9)	d

Values are mean (SD). Same lowercase letter in vertical columns indicates no difference at 5% significance level.

meniscus (Δh) using the equation $\Delta V = 0.25\pi \times \Delta h \times d^2$, where d is the diameter of the capillary tube. The percentage volumetric change was then calculated. Five specimens per material were measured, and the average volumetric shrinkage was obtained.

Scanning Electron Microscopy Observations

The surfaces of the cured resin composite specimens were polished to a high gloss with abrasive discs (Fuji Star Type DDC, Sankyo Rikagaku Co) and a series of diamond pastes down to 0.25-μm particle size (DP-Paste, Struers, Ballerup, Denmark). The polished surfaces were then subjected to unfiltered argon ion beam etching (IIS-200ER, Elionix, Tokyo, Japan) for 45 seconds in the direction perpendicular to the polished surface at an accelerating voltage of 1.0 kV and ion current density of 0.4 mA/cm^2. This is known to improve the visibility of the layers in the sample.[37] The surfaces were coated in a vacuum evaporator (Quick Coater Type SC-701, Sanyu Denshi Inc, Tokyo, Japan) with a thin film of gold (thickness count, 300). Examinations of the surfaces were carried out by scanning electron microscopy (SEM; FE-8000, Elionix) with an operating voltage of 10 kV and at magnifications of 5000× and 20,000×.

Statistical Analysis

Because of their homogeneity of variance (Bartlett's test) and normal distribution (Kolmogorov-Smirnov test), the data for each material were subjected to analysis of variance followed by Tukey's honestly significant difference test at a significance level of 0.05. Bartlett's test was performed with a custom program implemented in a spreadsheet (Excel, Microsoft Inc, Redmond, WA, USA), and the other statistical analysis was conducted using a statistical software system (Sigma Plot ver. 11.0, SPSS Inc, Chicago, IL, USA).

RESULTS

Inorganic Filler Content and Coefficient of Thermal Expansion

The inorganic filler contents and coefficients of linear thermal expansion are shown in Table 2. The average inorganic filler contents of the resin composites ranged from 67.2 to 81.3 wt%. The resin composites used in this study had significantly different inorganic filler contents, and the bulk-fill resin composite SF had the highest value. The bulk fill resin composites tended to have higher inorganic filler contents than the other resin composites.

The average coefficients of thermal expansion of the resin composites ranged from 33.5 to 48.9 ($\times 10^{-6}$/°C). Although the bulk fill resin composites exhibited higher thermal expansion, the low-shrinkage resin composites had lower thermal expansion than the conventional resin composites.

Flexural Properties

The results of flexural properties testing are shown in Table 3. The flexural strength of the resin composites ranged from 116.9 to 148.1 MPa, the elastic modulus ranged from 5.6 to 13.4 GPa, and average resilience ranged from 0.70 to 1.0 MJ/mm^3. There were significant differences between the

Table 3: *Flexural strength, elastic modulus, and resilience*[a]

Code	Flexural strength (MPa)	Tukey group	Elastic modulus (GPa)	Tukey group	Resilience (MJ/mm^3)	Tukey group
TB	115.4 (5.0)	d	9.0 (0.8)	d	0.74 (0.15)	a,b
FB	138.1 (7.5)	a,b	10.5 (0.9)	c,d	0.92 (0.11)	a,b
SF	120.0 (6.5)	c,d	5.6 (0.5)	e	0.78 (0.15)	a,b
KL	116.9 (7.3)	c,d	8.9 (0.4)	d	0.77 (0.20)	a,b
LS	129.7 (7.9)	b,c	12.1 (0.5)	a,b	0.70 (0.15)	b
HU	148.1 (7.0)	a	11.0 (1.3)	b,c	1.0 (0.15)	a
EQ	120.5 (8.5)	c,d	9.6 (0.6)	d	0.75 (0.14)	a,b
SU	138.2 (7.2)	a,b	13.4 (0.5)	a	0.71 (0.13)	b

[a] *Values are mean (SD). Same lowercase letter in vertical columns indicates no difference at 5% significance level.*

Table 4: *Volumetric change of resin composites as a function of time*[a]

Code	10 seconds	30 seconds	60 seconds	120 seconds	180 seconds
TB	−0.68 (0.05)a,A	−1.75 (0.05)a,B	−1.94 (0.05)b,C	−2.19 (0.10)b,D	−2.19 (0.09)b,D
FB	−0.49 (0.06)b,A	−1.37 (0.06)b,B	−1.62 (0.08)c,C	−1.93 (0.06)c,D	−1.95 (0.08)c,D
SF	−0.59 (0.06)ab,A	−1.37 (0.07)b,B	−2.15 (0.09)a,C	−2.91 (0.12)a,D	−2.93 (0.13)a,D
KL	−0.48 (0.05)b,A	−0.66 (0.06)d,B	−0.88 (0.06)g,C	−1.05 (0.08)f,D	−1.11(0.08)g,D
LS	0.21 (0.05)d,A	−0.18 (0.06)e,B	−0.39 (0.05)h,C	−0.83 (0.06)g,D	−1.02 (0.06)g,E
HU	−0.35 (0.06)c,A	−0.96 (0.08)c,B	−1.39 (0.07)d,C	−1.56 (0.10)d,D	−1.56 (0.06)e,D
EQ	−0.57 (0.07)b,A	−0.78 (0.09)d,B	−1.05 (0.06)f,C	−1.37 (0.05)e,D	−1.37 (0.10)f,D
SU	−0.49(0.08)b,A	−0.76(0.06)d,B	−1.2 (0.09)e,C	−1.58 (0.06)d,D	−1.67 (0.09)d,D

[a] *Values are mean (SD). Same lowercase letter in vertical columns indicates no difference at 5% significance level. Same uppercase letter in horizontal columns indicates no difference at 5% significance level.*

flexural strengths but no clear outliers. Regarding the elastic modulus, the bulk fill SF showed a significantly lower value than the other resin composites. HU showed the highest resilience, but it was significantly different only from LS and SU.

Polymerization Shrinkage

The results for volumetric changes are shown in Table 4 and Figure 2. Volumetric changes as a function of time over a duration of 180 seconds depended on the type of resin composite. However, for all the resin composites, apart from LS, volumetric shrinkage began soon after the start of light irradiation, and a rapid decrease in volume during light irradiation followed by a gradual decrease was observed (Figure 2). For the measuring point at 10 seconds, volumetric changes ranged from +0.21% to –0.68%. Only LS showed a volumetric increase, and the difference between LS and the other resin composites was statistically significant. For the measuring point at 180 seconds, volumetric changes ranged from –1.02% to –2.93%. The highest volumetric shrinkage was seen with SF, whereas LS showed the lowest final shrinkage among the tested materials. No significant differences in final shrinkage were observed in the low-shrinkage resin composites KL and LS. On the other hand, the bulk fill resin composites TB, FB, and SF showed higher volumetric changes compared with the other resin composites. Based on the volumetric changes, the resins were segregated into three groups: KL and LS belonged to the lower shrinkage group; HU, EQ, and SU formed the middle shrinkage group; and TB, FB, and SF were in the higher shrinkage group. All the tested materials continued to exhibit slower volumetric changes even after cessation of the light irradiation. Although there was a significant difference between the measuring point at 60 seconds and

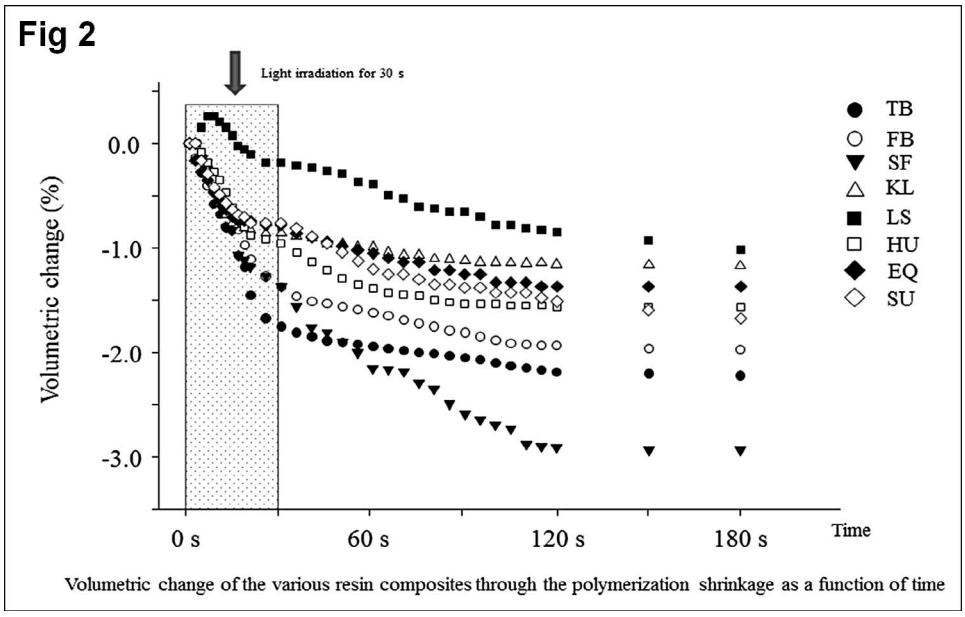

Figure 2. *Volumetric changes in the various resin composites due to polymerization shrinkage as a function of time.*

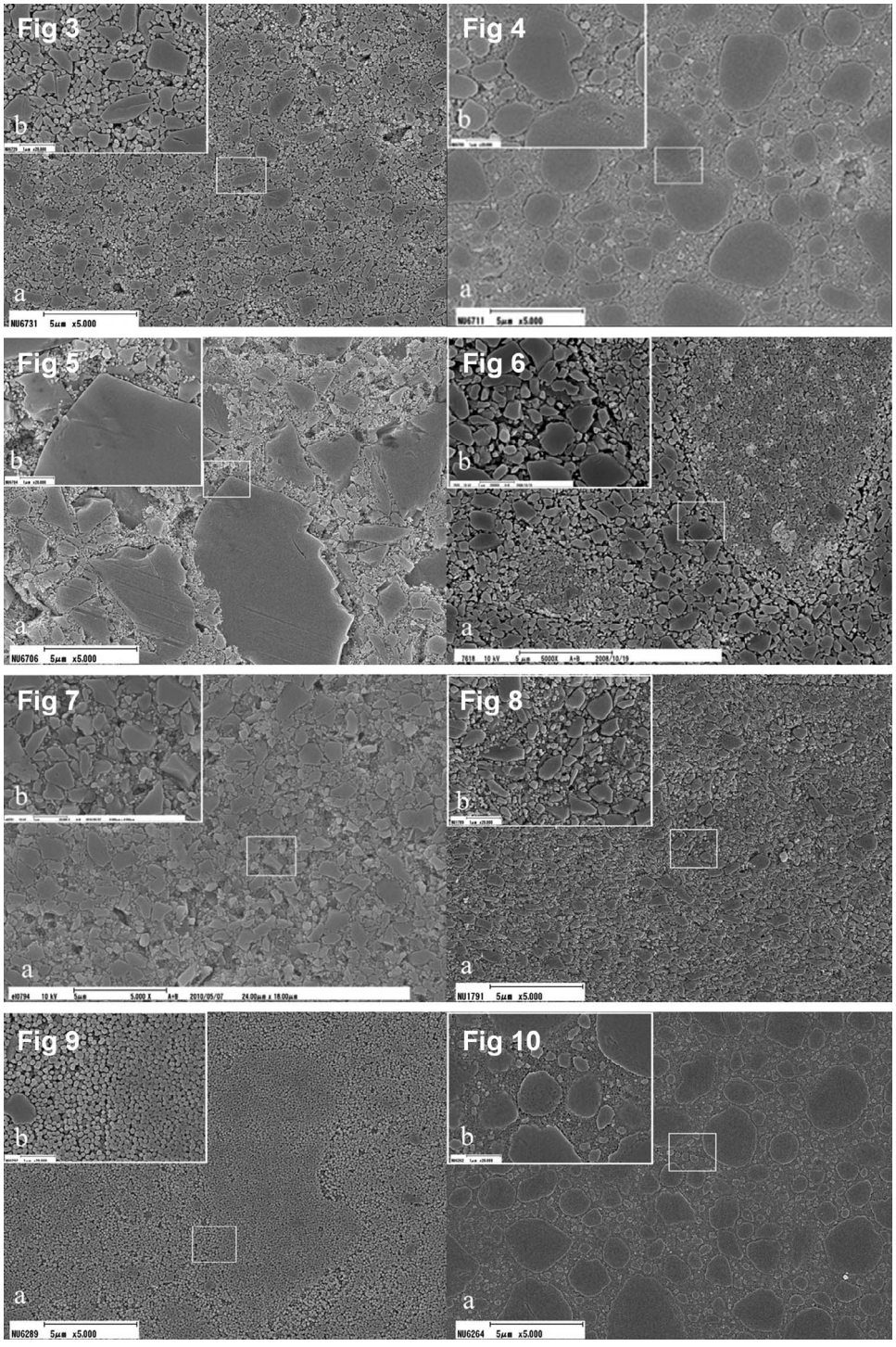

Figure 3. *Tetric EvoCeram Bulk Fill–argon ion–etched surface (a) 5000× and (b) 20,000×.*
Figure 4. *Filtek Bulk Fill posterior restorative–argon ion–etched surface (a) 5000× and (b) 20,000×.*
Figure 5. *Sonic Fill–argon ion–etched surface (a) 5000× and (b) 20,000×.*
Figure 6. *Kalore–argon ion–etched surface (a) 5000× and (b) 20,000×.*
Figure 7. *Filtek LS–argon ion–etched surface (a) 5000× and (b) 20,000×.*
Figure 8. *Herculite Ultra–argon ion–etched surface (a) 5000× and (b) 20,000×.*
Figure 9. *Estelite ∑ Quick–argon ion–etched surface (a) 5000× and (b) 20,000×.*
Figure 10. *Filtek Supreme Ultra–argon ion–etched surface (a) 5000× and (b) 20,000×.*

that at 120 seconds, no significant differences were noted at 120 and 180 seconds for all the materials, apart from LS.

SEM Observations

SEM images of the eight resin composites after argon ion etching are presented in Figures 3–10. These figures illustrate the differences in filler shape, particle size and distribution. TB, LS, and HU had similar appearances with 0.2- to 2-μm milled irregularly shaped glass filler particles, as shown in Figures 3, 7, and 8, respectively. FB and SU had rounded nano-sized zirconia/silica filler particles and 0.5- to 5-μm spheroidal aggregates of nano-sized

fillers (Figures 4 and 10). EQ also had spherical nano-sized filler particles, but 5- to 10-μm irregular aggregated fillers were observed (Figure 9). The glass filler particles of SF (Figure 5) were larger than the fillers in the other resin composite systems containing ground glass filler particles (TB, LS, and HU). KL had 0.2- to 1-μm irregularly shaped glass filler particles and 5- to 20-μm irregular aggregates of glass filler (Figure 6).

DISCUSSION

The aim of this study was to aid the clinical selection of resin composites by measuring the mechanical properties and polymerization behavior of resin composites belonging to various categories. Clear differences between the tested high-viscosity bulk fill resin composites and the other types of resin composite were shown.

For bulk fill resin composites, several developments and technologies have contributed to a deeper cure. To enhance the matrix resin degree of conversion, new initiator systems with a higher photocuring activity than camphorquinone are used,[25] or the translucency of the resin is adjusted to increase the transmission of light.[2,38] In addition, larger filler particles are used to reduce light scattering. Although initial low-viscosity bulk fill resin composites seemed to have good cavity wall adaptation due to flowability, they did not have adequate surface and bulk properties for endurance in high-stress bearing areas.[39] The tested high-viscosity bulk fill resin composites showed slightly higher inorganic filler content than the other resin composites. Improved handling, surface, and bulk mechanical properties, compared with the low-viscosity bulk fill resin composites, have been attributed to the higher filler content.

A large difference in thermal expansion induces gaps in the vicinity of the resin/tooth interface, resulting in the penetration of oral fluids between the restoration and the tooth surface.[40] This could lead to irritation of the dental pulp, marginal degradation, and secondary decay. A higher inorganic filler content is expected to reduce thermal expansion because of the reduction in the amount of resin matrix, which is more sensitive to thermal changes. However, despite the higher inorganic filler content in the bulk fill resin composites, they exhibited significantly higher thermal expansion compared with the other resin composites in this study. It has been reported that no correlation exists between the inorganic filler content and the coefficient of thermal expansion of the resin cements and dual-cured provisional resins.[41,42] Therefore, regardless of the type of resin composite, other factors such as the type of matrix resin, molecular mobility, surface treatment of fillers, and size of filler particles might have a significant impact on the thermal properties.

Despite having the highest inorganic filler content among the bulk-fill resin composites, SF had a relatively low flexural strength and a significantly lower elastic modulus compared with the other resin composites. Previous research has shown that SF showed significantly lower wear resistance compared with other bulk fill and conventional resin composites, and SEM observations revealed that plucking of filler particles was obvious after wear testing.[28] This finding is consistent with our results that SF had a lower flexural strength and elastic modulus compared with the other resin composites because flexural properties and wear resistance are related.[1] In particular, it is possible that the large glass filler particles in SF (Figure 5) create easy routes for crack propagation between the glass fillers and resin matrix.

All the tested bulk fill resin composites showed significantly higher volumetric shrinkage than the other resin composite types. In particular, SF showed approximately twice the volumetric shrinkage as the conventional composites HU and EQ. To obtain good adaptation to the cavity wall, SF uses a diluted triethylene glycol dimethacrylate monomer and a rheological modifier to support the enhancement of the flowability of the resin paste with ultrasound.[43] It can be speculated that, although these components of the resin matrix may contribute to increased volumetric shrinkage, they, along with lower elastic modulus, may also cause a reduction in polymerization shrinkage stress. The viscoelastic behavior of a resin-based material, as determined by their flow during the early stages of polymerization and the increase in elastic modulus, plays an important role in polymerization contraction stress.[16] It has been reported that although low-viscosity bulk fill resin composites showed higher volumetric polymerization shrinkage than their high-viscosity counterparts, the contraction stresses of low-viscosity bulk fill resin composites were lower than those of the high-viscosity bulk fill resin composites.[23] Viscoelasticity and volumetric shrinkage are both determined by similar factors that make it difficult to separate their influence on the development of contraction stress.[12,44] The complexity of contraction stress means that it is important to have a broad understanding of the properties of resin

composites when making clinical recommendations or decisions.[16]

For all the resin composites used in this study apart from LS, the volumetric shrinkage began soon after the start of light irradiation and polymerization shrinkage continued after the end of light irradiation. The shrinkage noted after removal of the light source might be attributed to the postirradiation reaction of residual monomers.[45,46] The low-shrinkage resin composites, KL and LS, showed significantly lower volumetric shrinkage compared with the other resin composites at measuring point 180 seconds. KL has prepolymerized filler particles, which include a lot of closely aggregated small splintered particles (Figure 6); it uses a new high-molecular-weight monomer rather than 2, 2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]propane or urethane dimethacrylate.[2,17] The degree of shrinkage is dictated by the number of covalent bonds formed, which is the extent of the polymerization reaction.[2,17] The monomer in KL (DX-511) has a high molecular weight of 895 g/mol and a low number of carbon–carbon double bonds, which help to reduce polymerization shrinkage.[18] In contrast, LS adopts ring-opening chemistry in siloranes to replace methacrylates in the resin matrix.[18,20,21] Uniquely, LS showed volumetric increase during the first 10 seconds of light irradiation. This phenomenon might be attributed to the molecular structure of the silorane monomer and the polymerization process. During the initial phase of polymerization, intermolecular space may be expanded due to cationic ring opening polymerization of the cycloaliphatic oxirane moieties. Furthermore, the low-shrinkage resin composites KL and LS showed significantly lower thermal expansion than the other resin composites, and it could be speculated that even after the polymerization process is complete, the molecular mobility of the resin matrix in the low-shrinkage resin composites may be less than that of the other types of resin composite.

FB and SU, which were obtained from the same manufacturer, showed significantly higher flexural strength than most of the other tested materials. Although the two products have different intended uses and utilize different resin matrix monomers, the appearance and distribution of the fillers were found to be similar under SEM observation (Figures 4 and 10). Fillers can distribute the load stress and inhibit crack propagation due to pinning effects.[47] Therefore, for FB and SU, the balance of filler size, distribution of fillers, and the interaction between fillers and the resin matrix through silane coupling may contribute effectively to dispersion of load stress. However, the elastic modulus and resilience of FB were different from those of SU, which may be attributed to a difference in resin monomers. For instance, only the high-viscosity bulk fill resin composite FB contains a 1,12-dodecane dimethacrylate (DDDMA) which has a hydrophobic backbone and increased molecular mobility. In addition, DDDMA provides flexibility, fast cure, and improved surface characteristics to the polymer matrix, which are suitable properties for bulk fill resin composites.[48,49] Furthermore, different resin monomers may influence the polymerization kinetics and thermal expansion of FB and SU, which were found to be different between the two resin types.

Lower elastic modulus may allow increased deformation and dimensional changes on the occlusal surfaces under high stress that could result in defect formation, marginal breakdown, or reduction of wear resistance.[1] In general, materials with high flexural strength also have a high elastic modulus, and these materials broadly follow that trend. However, LS has a high elastic modulus relative to its flexural strength. The elastic modulus of LS showed significantly higher values than the other resin composites, apart from SU and HU, whereas its flexural strength was only significantly higher than that of TB. LS uses an epoxy-based silorane system that involves the opening of an oxirane ring.[20] This epoxy-based silorane system contributes not only to the reduction of polymerization shrinkage but also to the evasion of the creation of an oxygen inhibited layer.[18,19] Thus, the resin monomer and resin have completely different properties from those of the other materials. Resilience is defined as the ability of a material to absorb energy when it is deformed elastically from external stress before failure.[41] Therefore, a high resilience is preferred for the long-term stability of restorations.[50] HU and FB showed somewhat higher resilience than the other resin composites in this study; however, the differences are not striking and their clinical effect is unclear.

The null hypothesis that there would be no significant differences in mechanical properties and polymerization behaviors among the three different categories of resin composite was rejected. In addition, even within the same category of resin composites, properties and state of fillers were material dependent. Therefore, it is important to understand the characteristics of each resin composite before clinical use and select the optimal one or combination in light of cavity size, depth, C-factors, and other factors. Furthermore, although mechanical properties

and polymerization behavior are not the only parameters for consideration in the selection of the optimal resin composite, these characteristics can provide valuable information and support the research and development of new resin composites.

CONCLUSION

The results of this study provide useful additional information on the mechanical properties and polymerization behavior of different categories of resin composite. The tested bulk fill resin composites tended to have higher inorganic filler content and linear thermal expansion than the other categories. In contrast, low-shrinkage resin composites showed significantly lower thermal expansion than the other resin composites. The flexural properties of the tested resin composites were material dependent with no clear outliers. The low-shrinkage resin composites showed significantly lower volumetric shrinkage while the bulk fill resin composites had significantly higher volumetric shrinkage than the conventional resin composites. It is concluded that each category of resin composite has different mechanical properties and polymerization behavior.

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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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Role of Proteolytic Enzyme Inhibitors on Carious and Eroded Dentin Associated With a Universal Bonding System

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Clinical Relevance

Carious dentin is a challenging substrate with which to establish an effective bond. Using an etch-and-rinse mode with universal adhesive system, stable resin-dentin bond strength over time can be achieved for those substrates. However, the use of E-64 associated with it presented better performance compared with CHX. Thus, it appears to be an appropriate bonding strategy for the maintenance of bond strength to eroded and carious dentin over time.

SUMMARY

Objectives: The aim of this study was to evaluate the effect of proteolytic inhibitors on the bond strength of a universal adhesive system (etch-and-rinse mode) applied to artificial carious and eroded dentin. Methods: Ninety molars were prepared and randomly divided into three groups according to the substrate: N, no challenges; ACD, artificial carious dentin simulation and ERO, artificial erosion simulation

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with orange juice. All groups were redivided into three subgroups according to the dentin pretreatment: W, water; CHX, 2% digluconate chlorhexidine; and E-64 (trans-epoxysuccinyl-L-leucylamido-[4-guanidino] butane), 5 μM E-64 inhibitor. They constituted a total of nine groups (n=10): N-W, N-CHX, N-E64, ACD-W, ACD-CHX, ACD-E64, ERO-W, ERO-CHX, and ERO-E64. All specimens were restored with Adper Single Bond Universal/Filtek Z250. Beams (0.64 mm^2) were obtained and subjected to the microtensile test (μTBS) in a universal testing machine at 0.5 mm/min. The failure mode of the interfaces was determined by optical microscopy (40× magnification). Data were statistically analyzed by three-way analysis of variance and Tukey tests ($p<0.05$). Results: All individual factors ($p<0.0001$) and the interaction between substrate and treatment ($p=0.0011$) and between substrate and time ($p=0.0003$) were statistically significant. The caries substrate contributed negatively to bond strength. Chlorhexidine reduced bond strength for normal and eroded conditions. Only the normal substrate was negatively affected by time despite the pretreatment. Conclusions: The universal bonding system appears to be a promising bonding strategy for the maintenance of bond strength to affected dentin. E-64 did not affect bonding to the dentin in contrast to the use of chlorhexidine, which, when associated with the universal system, did affect the microtensile bond strength for artificial carious dentin.

INTRODUCTION

Dental caries and erosion are the most clinically significant demineralizing events, provoking changes in dental hard tissues, depending on the etiologic agent and its intensity and frequency. In dentin, dental caries result in a mineral loss of the organic matrix and disorganization of the collagen fibrils.[1] Dental erosion is an increasingly common multifactorial event[1,2] associated with dietary habits or gastric disorders that favor the presence of acid components in the oral cavity.[2] However, different from caries, microorganisms are not involved in the erosion, which determines distinct clinical consequences.

Based on an improved understanding of the dental remineralizing mechanism and the use of advanced materials and technique, affected dentin can be maintained.[3,4] During the bonding process, a hybrid layer involves the interlocking of the bonding agent in a demineralized organic matrix based on collagen (90%) and noncollagen proteins (10%).[5] Therefore, in both carious and eroded clinical conditions, this layer might be more susceptible to degradation by intrinsic collagenolytic enzymes, such as matrix metalloproteinases (MMPs) and cysteine cathepsins (CCs), which are present in saliva and in dental hard tissues.[1,5-8] It has been shown that MMPs and CCs, exposed by the loss of minerals, can be activated in acidic conditions and act synergistically in the degradation of the adhesive interface through the hydrolytic degradation of collagen fibrils.[5,6] Therefore, strategies to paralyze or minimize their activity might be beneficial to dentin protection.

Several studies have demonstrated the inhibitory potential of several synthetic and natural inhibitors of MMP and CC activity.[1,5,7] The role of chlorhexidine in this process has been investigated the most.[1,5,7,9] Chlorhexidine is a widely employed proteolytic inhibitor, mainly due to its nonspecific action, practical use, substantivity, and low cost.[10,11]

E-64 (trans-epoxysuccinyl-L-leucylamido-[4-guanidino] butane) is a well-known specific inhibitor of CCs.[12] It is synthetically prepared, and its inhibition role is irreversible (covalent type) since there is a connection to the active site between the inhibitor and CCs. In 2010, Tersariol and others[13,14] introduced E-64 into dentistry at a concentration of 5 μM. In 2011, Nascimento and others[6] evaluated its proteolytic activity on CCs with satisfactory results. As it is soluble in water, it can be indicated for clinical use as a solution, following a similar protocol as digluconate chlorhexidine (CHX). Until now, its potential use in dental tissues has been limited to its effect on the progression of erosion,[15] and its effect on dentin bond strength has never been tested.

Bonding to tooth substrates is researched primarily using *in vitro* models with healthy dentin as a substrate to evaluate the impact of the mechanisms of interaction of different restorative materials under various adhesive protocols.[11,15] However, clinically, it is commonly tested on caries- or erosion-modified dentin that render the tissue biochemically, structurally, and mechanically different from that used in major laboratory studies. Thus, studies using naturally altered substrates or simulating demineralization conditions aim to reproduce situations in the laboratory in a more clinically relevant way.[16-21]

Simultaneously, universal restorative systems have been proposed to allow versatility in clinical

work, as they can be used in etch-and-rise and self-etching modes and in dry and wet conditions.[22-26] However, previous studies have shown controversial results. While one study demonstrated promising longevity results,[26] more susceptibility to degradation was observed when universal adhesives were applied on acid-etched dentin,[22,23] or no differences were found between etch-and-rinse and self-etch modes.[24,25] In this study, a universal adhesive system was used in etch-and-rise mode since an acidic condition activates enzymatic action, allowing for the better evaluation of the role of the inhibitors. As such, more studies are needed to better understand the degradation of the dentin organic matrix by host-derived enzymes when this new adhesive approach is used in distinct substrate conditions.

This study aimed to investigate the use of enzyme inhibitors associated with a new adhesive technology in clinically relevant conditions. The objective was to evaluate the performance of a universal adhesive system (etch-and-rinse mode) on bond strength to dentin in different conditions (artificial carious dentin or eroded dentin) after pretreatment with proteolytic inhibitors over time. The null hypotheses tested were as follows: 1) there is no difference in dentin bond strength to normal, carious, and eroded dentin; 2) there is no difference in dentin bond strength after treating the affected dentin with CHX or E-64; and 3) there is no difference on bond strength over time (six months) regardless of the substrate and pretreatment.

METHODS AND MATERIALS

Ninety extracted caries-free third molars were collected and stored for no more than one month in 0.1% sodium azide at room temperature. The roots were sectioned 3 mm below the cement–enamel junction. The occlusal enamel was removed horizontally (perpendicular to the long axis of the tooth) using a water-cooled diamond disc (Extec Corp, Enfield, CT, USA) to expose a flat dentin surface. The dentin surface was ground flat, and a smear layer was standardized using 600-grit SiC paper under running water for 30 seconds (Politriz APL-4 AROTEC, Cotia, São Paulo, Brazil). The specimens were divided according to the pretreatment of the dentin before the bonding procedures as follows: normal dentin (control [N]), artificial carious dentin (ACD) or erosion (ERO). The specimens in the control group were maintained in artificial saliva (1.5 mM $Ca[NO_3]_2{\cdot}4H_2O$, 0.9 mM $NaH_2PO_4{\cdot}2H_2O$, 150 mM KCl, 0.1 mol/L Tris, 0.03 ppmF, pH 7.0) at 37°C for seven days. Artificial carious dentin lesions were created by cycles of six hours of demineralization (2 mM $Ca[NO_3]_2{\cdot}4H_2O$, 2 mM $NaH_2PO_4{\cdot}2H_2O$, 0.075 mM acetate buffer, 0.02 ppm F, pH 4.0), followed by 18 hours of incubation in artificial saliva with remineralizing action. Daily renewal cycles were performed for five days followed by 48 hours of incubation in remineralizing solution.[27]

The erosive challenge consisted of immersion in industrialized orange juice with a pH of 3.2 (Suco Del Valle do Brasil, Coca-Cola, Americana, São Paulo, Brazil), composed of water, sugar, orange juice concentrate, natural flavor, citric acid, and antioxidant ascorbic acid, for five minutes, three times per day, for five days. As the consumption of this beverage is increasing, laboratory protocols have investigated its impact.[28] Between the erosive challenges and after the completion of the erosion, the specimens were stored in daily renewed artificial saliva.

Both substrates were assessed by transverse microradiography, after all cycles were completed, to validate the creation of artificial carious dentin and eroded dentin. For the artificial carious dentin substrate, we observed a demineralized subsurface layer with the preservation of the outer enamel surface, an aspect of carious lesions. For the artificially eroded substrate, a thin, worn superficial layer was evident.

All specimens were etched with 37% phosphoric acid (Dentsply, Catanduva, São Paulo, Brazil) for 15 seconds. Specimens from each dentin substrate were subdivided into three pretreatment groups (n=10), including application of distilled water (W), 2% chlorhexidine digluconate aqueous solution pH 5.8 (CHX, Pharmácia Específica, Bauru, São Paulo, Brazil), or 5 μM E-64 aqueous solution (E-64, Sigma-Aldrich, St Louis, MO, USA) pH 5.5 (E-64). After passive application for 60 seconds, excess was removed with absorbent paper. Then the adhesive system (Adper Single Bond Universal, 3M ESPE, St Paul, MN, USA) was applied according to the manufacturer's instructions and light cured using a 1000-mW/cm^2 LED unit (Radical, SDI, Bayswater, VIC, Australia). Two increments of 2-mm layers of the resin-based composite (Filtek Z350 Universal Restorative, 3M ESPE) were layered and light cured for 20 seconds each. The specimens were immersed in artificial saliva for 24 hours at 37°C and then longitudinally sectioned, perpendicularly to the bonding interface, using an Isomet 1000 digital saw (Buehler, Lake Bluff, IL, USA) to obtain beams of ≅ 0.64-mm^2 area (0.8×0.8 mm) beams. Each beam was measured at the dentin–adhesive interface using a

Table 1: *Mean Values (MPa) and Standard Deviations of the Test Conditions*[a]

	W		CHX		E-64	
	Immediate	**6 mo**	**Immediate**	**6 mo**	**Immediate**	**6 mo**
N	35.32 (5.30) AaΔ	27.45 (5.33) Aa†	28.36 (5.88) AbΔ	16.50 (3.89) Ab†	28.33 (5.42) ABbcΔ	20.80 (3.71) ABbc†
ERO	29.85 (4.77) AaΔ	26.07 (4.96) AaΔ	22.53 (4.76) ABbΔ	20.13 (4.62) ABbΔ	30.23 (6.51) AaΔ	27.70 (5.32) AaΔ
ACD	23.42 (4.95) BabΔ	20.28 (3.55) BabΔ	18.31 (3.50) BbΔ	16.50 (3.90) BbΔ	24.51 (4.41) BaΔ	20.80 (3.71) BaΔ

Abbreviations: ACD artificial carious dentin; CHX, digluconate chlorhexidine; ERO, artificial erosion simulation;E-64, trans-epoxysuccinyl-L-leucylamido-(4-guanidino) butane; N, normal dentin; W, water.

[a] *Different uppercase letters indicate differences between substrates in each treatment and time (columns) ($p<0.05$). Different lowercase letters indicate differences between treatments in each substrate and time (rows) ($p<0.05$). Different symbols (Δ,†)indicate differences between times (immediate vs 6 mo) in each substrate and treatment ($p<0.05$).*

digital caliper (Mitutoyo America, Aurora, IL, USA) to obtain the exact surface area of the interface, which was fixed to the Bencor Multi-T testing apparatus (Danville Engineering Co, Danville, CA, USA) with cyanoacrylate resin (Super Bonder Flex Gel-Loctite, Henckel Ltda, Itapevi, São Paulo, Brazil) and tested in tension in a universal testing machine (Instron 3342, Instron Co., Canton, MA, USA) at a 0.5-mm/min crosshead speed and with a 500 N load cell. Each tooth was considered the experimental unit. From each specimen (tooth), an average of eight to 10 beams was obtained for each time (after 24 hours and six months). During the six-month aging period, all beams were stored in a weekly renewed artificial saliva at 37°C.

The microtensile bond strength (μTBS) was expressed in MPa by dividing the maximum load (kgf) by the specimen cross-sectional area (mm^2). Each fractured surface was analyzed with a handheld digital microscope (DINO-LITE^{plus} digital microscope, AnMo Electronics Corp, Hsinchu, China) at 40× magnification, and failure was classified in the adhesive (failure in the adhesive layer), cohesive in dentin, cohesive in composite resin, or mixed. For the statistical analysis, as the tooth was the experimental unit, an average of all beams per tooth was performed to determine the tooth μTBS. Data were calculated and statistically analyzed with Statistica software (Statsoft, Tulsa, OK, USA). Assumptions of a normal distribution and equality of variance were tested for all the variables using the Kolmogorov-Smirnov and the Levene test, respectively. As the assumptions were satisfied, the data were subjected to three-way analysis of variance ($p\leq0.05$), followed by the Tukey test ($p<0.05$) for individual comparisons.

RESULTS

All tested factors (substrate, pretreatment, and time) were statistically significant ($p<0.0001$). Significant interactions between substrate and treatment ($p=0.0011$) and between substrate and time ($p=0.0003$) were also detected. Table 1 shows the mean and standard deviation values (MPa) of dentin bond strength and comparisons of all substrates and pretreatment methods.

Overall, the results indicate that bond strength was compromised related to three conditions: artificial carious dentin condition, chlorhexidine as a pretreatment, and six months of aging. Bond strength trended lower in the artificial carious dentin condition, even with chlorhexidine as a pretreatment, at six months, and an analysis of interactions was performed.

Regarding the substrate, artificial carious dentin consistently demonstrated the lowest bond strength values regardless of pretreatment and time. Overall eroded dentin also had lower bond strength immediately and after six months of aging compared to normal dentin, except when E-64 was used. However, no statistical significance was observed. Bond strengths to dentin decreased during aging. For all groups based on normal dentin, a significant difference between the immediate and the six-month groups was observed, regardless of pretreatment. In the ACD and eroded dentin groups, a reduction of dentin bond strength after aging was noted, although it was not statistically significant in all pretreatment groups.

The CHX groups had the lowest bond strength values regardless of the substrate and time. E-64 significantly reduced bond strength of normal dentin, both immediately and after six months of aging. However, the same trend was not observed for artificial carious and eroded dentin. In ACD, CHX bond strengths were significantly lower than in E-64-treated samples immediately and after six months. Interestingly, the use of E-64 preserved the bond strength overtime (after 6 months) for both ACD and ERO, and a significant reduction after 6 months was observed only for normal dentin.

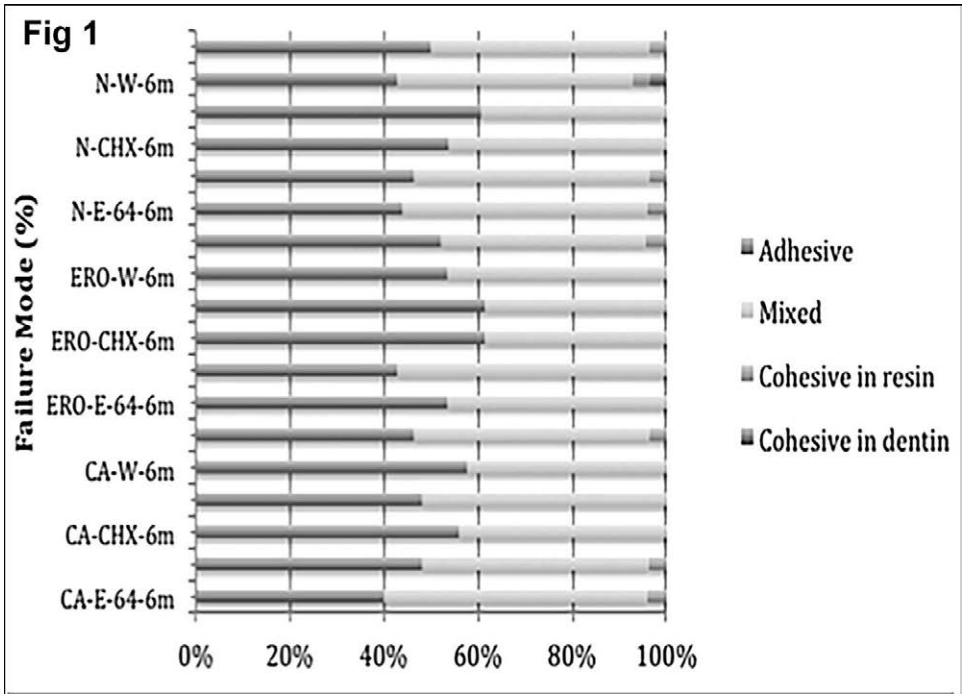

Figure 1. *Failure mode distribution (%) for all substrates and pretreatment with inhibitors immediately and at six months.*

The distribution of failure mode analysis is presented in Figure 1, revealing that adhesive and mixed failures were predominant in all groups, independent of the substrate condition, treatment, and aging.

DISCUSSION

Most studies use healthy substrates to understand the mechanism involved in the hydrolytic degradation of the hybrid layer. In this study, artificial simulation of carious and eroded dentin was performed to obtain the most common substrates seen in clinical practice, beyond normal dentin. These different substrates were associated with the use of enzyme inhibitors in bonding protocols of a universal dentin bonding system.

The results demonstrated significant differences in adhesion according to the dentin substrate condition. The normal substrate showed the highest value, and the carious dentin showed the lowest bond strength value, while the eroded substrate did not show a significant difference compared to normal dentin. For this reason, the first null hypothesis was rejected. This outcome was supported by previous studies showing that, in general, hybrid layers that are poorly infiltrated by adhesives,[17] such as carious and eroded substrates, have changed adhesion.[29,30] It suggests clinical relevance since most techniques for carious dentin removal leave caries-affected and even caries-infected dentin to serve as the bonding substrate,[30,31] and the removal method may also affect immediate dentin bond strength.[31]

For the eroded substrate, previous studies also reported the impact of erosion on reducing bond strength to dentin.[19,20] It is likely that the presence of the collapsed demineralized fibrils and the high water content may prevent the proper polymerization of adhesives. In the eroded dentin, the thicker layer of collagen exposed by erosion cannot be adequately infiltrated by resin monomers.

Orange juice was used in this study to promote eroded substrate, as it is a relevant source of vitamin C. This vitamin has been thought to play a potent antioxidant role, and thus its use has been investigated as an adjunctive strategy to improve resistance against hybrid layer degradation.[32,33] According to Gotti and others,[33] vitamin C acts by reducing the potential degradation effects of free radicals and hydrolysis. They found that antioxidant-doped adhesives appear to have a positive effect on the durability of the adhesive interface since their bond strength after 24 hours of water storage was maintained or increased over time. Therefore, this mechanism can explain the similar performance of normal and eroded substrates.

In regard to pretreatment with enzyme inhibitors, CHX and E-64 reduced bond strength for normal but not for caries- or erosion-affected dentin immediately and after six months of aging, resulting in the rejection of the second null hypothesis.

Previous studies have reported that MMPs and CCs typically coexist in normal dentin.[1,9,13,34] In carious lesions, they contribute to the degradation of the dentin organic matrix.[1] In the dental bonding process, these enzymes participate in the degradation of the hybrid layer.[8,35] Evidence also suggests their role in eroding dentin.[20] Strong evidence also indicates that there is an increase of CC in carious dentin with increasing depth toward the pulp, suggesting that MMPs and CCs have synergistic and dependent activities.[7] Based on all this evidence, the pretreatment of dentin substrates before bonding could improve the biological conditions for adequate bonding over time.

The significant reduction in the immediate dentin bond strength with both inhibitors was an unexpected finding. Chlorhexidine is the most commonly used nonspecific proteolysis inhibitor in bond strength durability studies and the only one used clinically.[15] In general, it has demonstrated satisfactory performance, as it did not affect the immediate dentin bonding and postpones the reduction in bond strength.[8,18-20,36,37]

A reasonable speculation about this performance might be the possibility of an interaction between CHX and 10-methacryloyloxydecyl-dihydrogen phosphate (MDP), present in the adhesive system. Adper Single Bond Universal presents MDP as a relevant ingredient that is a phosphate and bifunctional monomer that is able to bind chemically to dental substrate.[22-26,38] CHX binds to both the mineral and the organic components of dentin,[39] so it can be speculated that it may have affected the competitive MDP bonding to calcium, thus contributing to the reduction in bond strength.

On the other hand, the lower dentin bond strength in normal dentin may be related to the pH of the applied inhibitors. Even though only mildly acidic (pH 5.8 and 5.5 for CHX and E-64, respectively), 60-second applications may have increased demineralization. The mild acid pH CHX solutions may have provoked an erosive effect. This, in turn, would have resulted in demineralized collagen that is too thick, leading to poor penetration of adhesives and thus a reduction of the immediate bond strength.[40]

Nevertheless, a recent study demonstrated that 2% CHX did not have a significant effect on the immediate or 12-month bond strength of Adper Single Bond Universal Adhesive.[41] However, similar to most CHX bonding studies, the pH of the solution was not reported. The effect of pH on the CHX effect on the dentin bond remains to be evaluated.

While bond strength to normal dentin was reduced significantly during aging, nonsignificant changes were seen in the affected substrates regardless of the pretreatment. In eroded dentin, CHX again showed lower bond strength compared to the control in contrast to E-64, and in artificial carious dentin, bond strength in the CHX-treated group was significantly lower than that of the E-64 group at both time points. This result suggests that, in caries- and erosion-modified substrates, factors other than pH may exert an effect on bonding.

The third hypothesis was also rejected. It is interesting to note that while bond strength showed a significant time-related reduction in normal dentin, the loss of bond strength was markedly lower in eroded or artificial carious dentin regardless of the treatment.

Both enzyme-inhibiting solutions, CHX and E-64, promoted significantly lower bond strengths after six months when compared to the immediate values in normal dentin, especially with CHX, which is supposed to inhibit both MMPs and CCs. As it also occurred even though the immediate bond strength was already significantly reduced, a hypothesis of an interaction between MDP and CHX may be considered.

For the artificial carious and eroded dentin, all groups had reduced bond strength. Mechanisms that may explain this result include the mode of inhibitor application or drying, the effect of the acidity of CHX and E-64 on the matrix collagen, or the fact that SB Universal is not susceptible to inhibitors.

This is in accordance with previous findings[18] and has been proposed to be caused by the inferior quality of the hybrid layer in caries-affected dentin.[30] Logically, this should lead to an increased rate of hydrolytic degradation of the hybrid layer components because, in caries-affected layers, the integrity of collagen is seriously affected[1,42] and the levels of MMPs and CCs are increased.[7,9] It would be expected that bond strength would be lost faster relative to intact dentin.

However, in the poorly structured hybrid layer of artificial carious and eroded dentin, the bond based on the micromechanical interlocking of collagen is not as imperative as in the "normal" hybrid layer; thus, the loss of collagen may have a lesser effect.[1] The remaining low bond strength may be due to the weaker physical forces (eg, the van der Waals forces), which may be less susceptible to structural changes in the adhesive–dentin interface. As stated by Marshall and others,[43] van der Waals forces occur

at every interface; however, they are often supplemented by significant contributions from strong bonds that may be present. Whatever the explanation, the difference should perhaps be considered in future research. First, dentin-bonding studies using only normal intact dentin give overly optimistic estimations of dentin bond strength and should perhaps be replaced or complemented by studies using caries-affected dentin. Second, since the lower bond strength of caries-affected dentin is widely acknowledged,[29,30] perhaps more emphasis should be placed on attempts to improve immediate bond strength in carious dentin rather than the preservation of bond strength in intact dentin, as is currently being done. If the immediate bond strength of carious dentin can be increased or reach levels similar to intact dentin, then it will be interesting to see if bond strength reduction remains nonsignificant. That kind of adhesive would be of an actual clinical benefit, as dentin bonding is almost always performed on caries- or erosion-modified substrates.

Moreover, it is important to note the relationship between proteolytic enzyme inhibitors and the universal adhesive system, especially CHX, which seems to present an interaction with MDP. On the other hand, E-64 resulted in a lower decline of bond strength over time, mainly in erosive and carious substrates. For aging, the use of CHX and E-64 demonstrated no difference at baseline or at six months, and thus it is possible that proteolytic enzyme inhibitors have an interaction with altered substrates.

CONCLUSIONS

In conclusion, the present study confirms the previous findings of significantly lowered bond strength in artificial carious dentin compared to normal intact dentin. Simulated erosion also impaired bond strength to dentin. Proteolytic enzyme inhibition did not improve the durability of bond strength associated with the universal bonding system. However, E-64 preserved bond strength overtime in carious and eroded dentin so this treatment may be a promising strategy for hybrid layer longevity. Research should focus more on identifying methods to improve the initial bond strength in eroded and caries-affected dentin rather than trying to eliminate the degradation of the hybrid layer components created on intact dentin.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethic Committee for Human Studies of Bauru School of Dentistry, University of São Paulo, Brazil. The approval code for this study is 16558913.2.0000.5417.

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 Preparation Designs on the Prognosis of Porcelain Laminate Veneers: A Systematic Review and Meta-Analysis

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Clinical Relevance

Window-type preparations are recommended for porcelain laminate veneers with a butt-joint considered when incisal coverage is needed.

SUMMARY

Objective: To investigate the association between preparation designs and prognosis of porcelain laminate veneers (PLVs).

Methods: Electronic and manual literature searches were performed in Medline, Embase, CENTRAL, and Scopus databases for randomized controlled trials and retrospective and prospective cohort studies comparing any two of three preparation designs. The quality of the included studies was assessed using the Newcastle-Ottawa scale. Pooled hazard ratios and risk ratios were used to evaluate the difference between two preparation designs. Subgroup analyses, sensitivity analysis, and evaluation of publication bias were performed if possible.

Results: Of 415 screened articles, 10 studies with moderate to high quality were included in the meta-analysis. Comparison of preparations with incisal coverage to preparations without coverage revealed a significant result based on time-to-event data (hazard ratio=1.81, 95% confidence interval [CI]=1.18-2.78, I^2=12.5%), but the result was insignificant based on dichotomous data (risk ratio=1.04, 95% CI=0.59-1.83, I^2=42.3%). The other comparisons

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between any two of overlap, butt-joint, and window types revealed no statistically significant difference. Subgroup analyses regarding the porcelain materials, location of prosthesis, and tooth vitality could account for only part of the heterogeneity. No evidence of publication bias was observed.

Conclusions: Within the limitation of the present study, it can be concluded that preparation design with incisal coverage for PLVs exhibits an increased failure risk compared to those without incisal coverage. The failure risk of the overlap type may be higher than the butt-joint type but must be validated in further studies.

INTRODUCTION

Porcelain laminate veneers (PLVs) have been introduced as a conservative solution to esthetic prosthodontics for anterior teeth since the 1980s[1,2] and are widely indicated for those with discoloration, malformation, misalignment, or any other dental defect. With the progress of materials and bonding systems, the long-term success of PLVs has greatly increased. However, no clinical consensus is available regarding the type of design preferred for PLVs.

There are various classification systems to distinguish the different preparation designs for PLVs, of which the traditional three-type classification is frequently used, namely, window, butt-joint, and overlap type.[3] The window type refers to those preparations that do not reduce the incisal edge, which is indicated for teeth with satisfactory incisal length. The latter two preparations are indicated for those who need modification of incisal length or translucency. As to whether a palatal chamfer is prepared, the types are further grouped into butt-joint type and overlap type.

In vitro studies have investigated the stress distribution and fracture strength of PLVs with different preparation designs. Two- and three-dimensional finite element analyses revealed that the butt-joint type tolerates stress better, whereas the overlap type distributes stress more uniformly. In contrast, the window type concentrates stress in the incisal area.[4-7] However, controversy exists regarding dynamic loading tests.[7-12] A meta-analysis focusing on these *in vitro* studies yielded synthetic outcomes suggesting that the fracture strength of the butt-joint type is similar to nonprepared teeth and that overlap type is more prone to fracture compared to the window type.[3]

To date, limited studies have focused on the effect of preparation designs on the prognosis of PLVs. Concerning preparations with or without incisal coverage, the survival rates showed no significant difference in a two-and-a-half-year follow-up study,[13] whereas another study reported that the four-year survival rate of porcelain veneers with incisal coverage was significantly increased compared to that without coverage.[14] Still other studies reported opposite results.[15-17] The survival or success rates of veneers with window, butt-joint, and overlap types are also under discussion. In general, there seemed to be no difference among these three preparation designs;[13,18] nevertheless, some observational studies noted that the survival of the overlap type was superior to the butt-joint type.[19-21] Therefore, the relationship of preparation designs to PLV survival remains unclear.

A recent systematic review investigating the survival rates with or without incisal coverage showed that either type was successful and that incisal coverage tended to be associated with an increased but statistically insignificant failure risk.[22] The single survival rates from original studies were extracted to synthesize an overall value with or without incisal coverage separately by the authors, whereas only three directly comparative studies were identified for the overall odds ratio estimate. However, the failure risk among window, butt-joint, and overlap types remains unknown. The purpose of this article is to comprehensively determine if PLVs with different preparation designs differ in their prognostic survival/success. A secondary purpose is to disclose potential confounding factors influencing the result of meta-analysis for future clinical trials.

METHODS

Protocol and Registration

This review was registered at the PROSPERO (CRD42016040166) and conformed to the proposed MOOSE (Meta-Analysis of Observational Studies in Epidemiology) guidelines.[23]

Search Strategies

The MeSH terms, free key words in the search strategy, were defined based on the PICOS question:

1) Population (P): patients who received PLV restorations.
2) Intervention/comparison (I/C): any two kinds of overlap, butt-joint, and window types.

Table 1: *Search Strategy Used in Electronic Databases*

Database	Search Strategy/Terms
Medline via PubMed	#1 "dental veneers"[mesh]
	#2 porcelain laminate veneer*[tw] OR porcelain veneer*[tw] OR ceramic laminate veneer*[tw] OR ceramic veneer*[tw]
	#3 "Tooth Preparation, Prosthodontic"[mesh]
	#4 tooth preparation[tw] OR dental preparation[tw] OR preparation design*[tw] OR preparation type*[tw] OR incisal preparation*[tw] OR incisal edge preparation*[tw] OR incisal edge reduction[tw] OR incisal porcelain coverage[tw] OR window preparation[tw] OR feather preparation[tw] OR feathered incisal edge[tw] OR butt joint[tw] OR bevel preparation[tw] OR modified overlap[tw] OR full veneer*[tw] OR palatal chamfer[tw] OR palatal extension[tw] OR incisal overlap[tw] OR without preparation[tw] OR nonprepared[tw] OR non-prepared[tw]
	#5 survival[tw] OR success[tw] OR failure[tw] OR longevity[tw]
	#6 #1 OR #2
	#7 #3 OR #4
	#8 #5 AND #6 AND #7
Embase via embase.com	#1 "dental veneer"/exp
	#2 (porcelain OR ceramic) NEXT/2 veneer*
	#3 "tooth preparation" OR "dental preparation" OR "preparation design*" OR "preparation type*" OR (incisal NEXT/2 (preparation* OR coverage*)) OR "incisal edge reduction" OR "window preparation" OR "feather preparation" OR "feathered incisal edge" OR "butt joint" OR "bevel preparation" OR "modified overlap" OR "full veneer*" OR "palatal chamfer" OR "palatal extension" OR "incisal overlap" OR "without preparation" OR "nonprepared" OR "non-prepared"
	#4 "survival" OR "success" OR "failure" OR "longevity"
	#5 #1 OR #2
	#6 #3 AND #4 AND #5
CENTRAL via Cochrane Library	#1 MeSH descriptor: [Dental Veneers] explode all trees
	#2 (porcelain laminate veneer*) OR (ceramic laminate veneer*) OR (porcelain veneer*) OR (ceramic veneer*) OR (dental veneer*) OR (dental laminate*) OR (Veneer*, Dental) OR (Laminate*, Dental)
	#3 #1 OR #2
	#4 MeSH descriptor: [Tooth Preparation, Prosthodontic] explode all trees
	#5 (tooth preparation) OR (dental preparation) OR (preparation design*) OR (preparation type*) OR (incisal preparation*) OR (incisal edge preparation*) OR (incisal edge reduction) OR (incisal porcelain coverage) OR (window preparation) OR (feather preparation) OR (feathered incisal edge) OR (butt joint) OR (bevel preparation) OR (modified overlap) OR (full veneer*) OR (palatal chamfer) OR (palatal extension) OR (incisal overlap) OR (without preparation) OR (nonprepared) OR (non-prepared)
	#6 #4 OR #5
	#7 (survival) OR (success) OR (failure) OR (longevity)
	#8 #3 AND #6 AND #7
SCOPUS	(TITLE-ABS-KEY(porcelain laminate veneers) OR TITLE-ABS-KEY(ceramic laminate veneers) OR TITLE-ABS-KEY(porcelain veneers) OR TITLE-ABS-KEY(ceramic veneers)) AND (ALL("preparation designs") OR ALL("preparation types") OR ALL("incisal coverage") OR ALL("incisal overlap")) AND (ALL("survival") OR ALL("success") OR ALL("failure"))

3) Outcome (O): mechanical failure of a PLV, including fracture and debonding.
4) Study design (S): clinical follow-up studies, including controlled clinical trials and cohort studies.

Literature searches were conducted up to June 2016 using the following databases: PubMed/Medline, Embase, and the Cochrane Library (Table 1). There were no restrictions on language or year of publication. References cited in the excluded reviews and included articles were also accessed to identify other potentially relevant studies.

Selection Criteria

After the identification of articles in the databases, the articles were imported into Endnote X7 software (Thompson Reuters, Philadelphia, PA, USA) to remove duplicates. Two independent reviewers initially screened the titles and abstracts of all documents, after which full copies of all potentially relevant studies were obtained for further identifi-

cation. Any disagreement regarding the eligibility of included studies was resolved through discussion and consensus or by a third reviewer.

Studies were considered eligible and were included according to the following criteria: 1) PLVs were restored for anteriors or premolars, regardless of the porcelain materials; 2) at least two types of preparation designs were mentioned or compared; 3) the respective number of survival/failure could be acquired or inferred from articles, or hazard ratios (HRs)/risk ratios (RRs) with 95% confidence intervals (CIs) were provided; and 4) both clinical trials and prospective or retrospective cohort studies were allowed, excluding *in vitro* studies.

Data Extraction

Data were extracted by two reviewers independently and tabulated into separate databases using a standard collection form. Completed forms were then compared and discussed to achieve a consensus. The extracted information included the name of first author, year of publication, country, study design, duration of recruitment and follow-up, number of patients and veneers, composition of gender and age, the porcelain materials and adhesives, and investigated preparation designs and their corresponding number/proportion of events. Moreover, potential confounding factors influencing the survival of PLVs were particularly identified, such as the location of prostheses, tooth vitality, restorative cause, and consistency of operators. Once any missing information was noted, the authors of the included articles were contacted via e-mail to retrieve details.

Quality Assessment

The bias risk and quality of included studies were assessed using the Newcastle-Ottawa Scale (NOS)[24] because the pilot experiment indicated that retrospective or prospective cohort studies were most common. The NOS contains eight scoring items categorized into three fields: Selection, Comparability, and Outcome. Each field could be scored a maximum of one star with the exception of the item of comparability, which could be given a maximum of two stars. Therefore, studies were graded with zero to nine stars based on their matching with the eight items in NOS, where zero to four stars represented low quality, five to six moderate quality, and seven to nine high quality.

Items in need of specific definitions were defined prior to the formal scoring procedure. PLVs restored in worn-out teeth or patients with bruxism were considered with compromised representativeness in the field of Selection. Regarding the field of Comparability, two stars can be given once the PLV restorations were made both in the same porcelain and by the same operator. If only either one was matched but the *post hoc* analysis had considered an adjustment (eg, adjusted HR estimate), the study would still receive two stars. When the duration of follow-up was less than two years or the dropout rate was greater than 20%, the corresponding item regarding follow-up failed to receive a star.

Statistical Analysis

To illustrate the strength of association between preparation designs and survival of PLVs, time-to-event data are considered best in prognostic studies, which often present these data as HRs, Kaplan-Meier curves, or lifetime tables.[25] In this review, both HR and RR were used as measures of the association between preparation designs and failure given the possibility that limited studies may focus on time-to-event data reports. Compared with RR calculated from the event number in different groups, HR appears to be more difficult to acquire. When HR and 95% CI were not specified in the articles, Kaplan-Meier curves were read by the digitizer tool in OriginPro 2016 (OriginLab Corp, Northampton, MA, USA), and then data were utilized to summarize the HR and standard error of ln(HR) by performing survival analyses.[26] Authors were contacted via e-mail for original data if HR or survival curves were not reported. Then HR was calculated using SPSS 22.0 (IBM Corp, Chicago, IL, USA). In the worst case, RR served as an alternative when time-to-event data were not available using all the above methods.

HRs and RRs with their variance of natural logarithm were pooled by a random-effects model using STATA 12.0 (Stata Corp, College Station, TX, USA). An HR or RR >1 indicated a worse prognosis in PLV survival. Statistical heterogeneity was tested using the I^2 statistic (significance level at $I^2 \geq 50\%$). If determinant factors such as porcelain materials, location of prosthesis, and tooth vitality were identified, subgroup analyses was performed using a random-effects model. Given that confounding factors were not consistent among studies, a sensitivity analysis, eliminating one study in each calculation, was performed to explore possible explanations for heterogeneity. Potential publication bias was assessed by visual inspection of the Begg's funnel plots and Begg's test. A two-sided $p \leq 0.05$ was considered statistically significant except where otherwise specified.

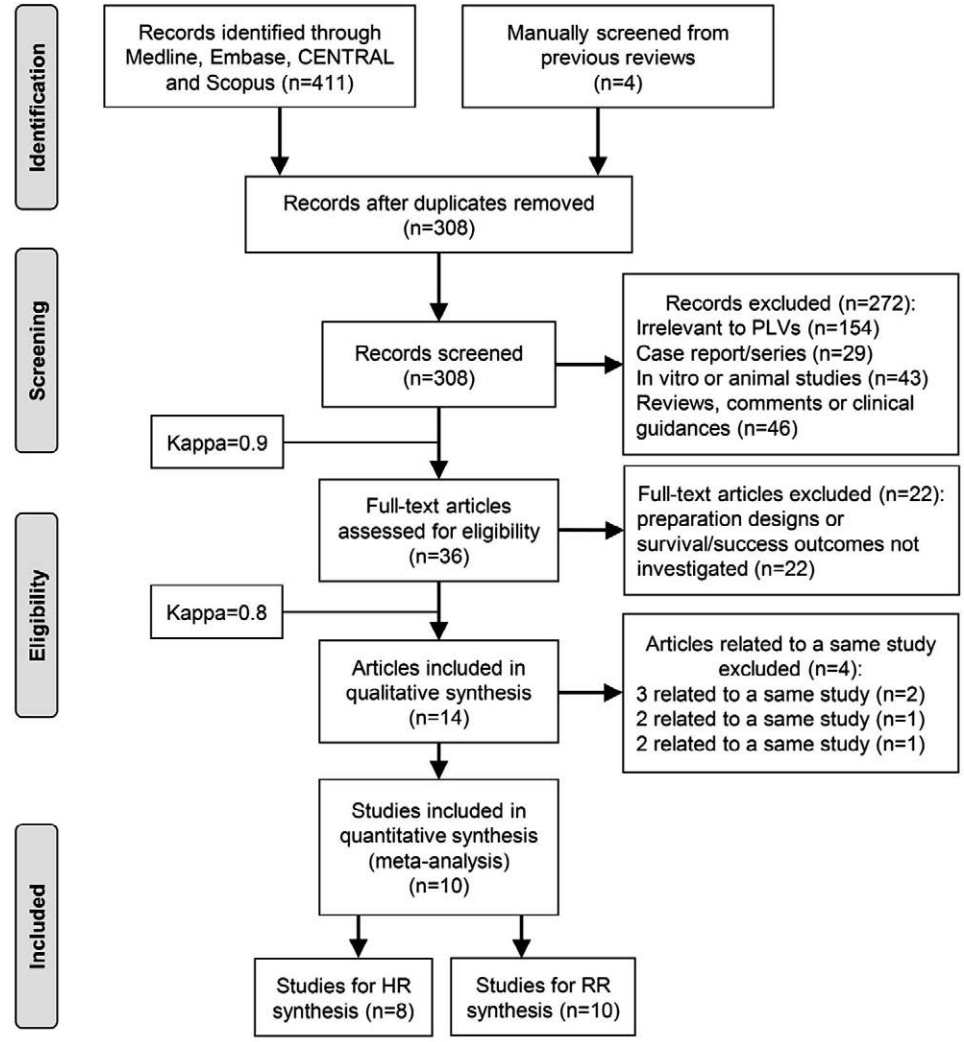

Figure 1. *Flowchart summarizing the study selection.*

Fig 1

RESULTS

Study Selection and Characteristics

With the defined criteria, electronic database searches via Medline, Embase, CENTRAL, and Scopus retrieved 199, 50, 21, and 141 articles, respectively. A total of 411 articles were identified. In addition, four articles were identified after manually screening the references of previous reviews. After the process of duplicate removal, initial screening, and full-text review, 10 studies with 14 articles were included in the meta-analysis (Figure 1). Of the 10 studies, only one reported HR and its 95% CI,[15,17] one provided a simplified lifetime table,[16] five provided Kaplan-Meier curves,[14,19-21,27,28] and one from China provided original data to calculate the HR value on author contact.[29] We did not receive replies from the authors of the remaining two studies.[18,30,31] Thus, eight studies were eligible for the overall HR estimate, and 10 were available for overall RR estimates.

The characteristics of the 10 studies are presented as follows (Table 2). These studies were published between 1998 and 2014, including one from the Netherlands,[18] two from Austria,[16,30,31] one from Australia,[14] three from Turkey,[15,17,21,28] one from China,[29] one from Spain,[27] and one from Germany.[19,20] More than 463 patients with a total of 2429 porcelain veneers were involved in the 10 studies. The designs of most studies were retrospective or prospective cohort studies, except one interim analysis of a randomized controlled trial.[18] However, the trial was incomplete, and its report resembled a cohort study.

Table 2: *Characteristics of the 10 Included Studies*

First Author	Country, Recruitment Period	Study Design; Duration (yrs)	No. of Patients and Veneers	Investigated Comparisons	Corresponding Number of Events/Total	HR Estimate with sSe[ln(HR)]
Meijering (1998)	Netherlands, 1994	Interim of RCT; 2.5	NA; 56	Butt-joint vs window	2/24; 1/32	NA
Dumfahrt (1999, 2000)	Austria, 1987–1996	RCS; 1–10 (mean: 4.5)	72; 191	With vs without incisal coverage	3/137; 4/54[a]	NA
Smales (2004)	Australia, 1989–1993	RCS; 1–7	50; 110	With vs without incisal coverage	1/46; 7/64	0.01 (408.25)[a]
Cortert (2009)	Turkey, 1999–2005	PCS; NA (median: 1.5)	40; 400	Overlap vs butt-joint	8/376; 4/24[a]	0.12 (0.72)[a]
Du (2009)	China, 1999–2007	RCS; 1–8	49; 308	With vs without incisal coverage	22/253; 4/55[c]	0.897 (0.546)[c]
Granell-Ruiz (2010)	Spain, 1995–2003	RCS; 3–11	70; 323	Overlap vs window	25/199; 17/124	1.39 (0.33)[a]
Beier (2012)	Austria, 1987–2009	RCS; 1–21 (mean: 10)	74; 292	Overlap vs window	20/245; 0/47	3.65 (0.54)[b]
Gurel (2012, 2013)	Turkey, 1997–2009	RCS; NA-12	66; 580	With vs without incisal coverage	24/261; 18/319	2.31 (0.3184)
Guess (2008, 2014)	Germany, 2000–2003	PCS; 1–7	14; 44	Overlap vs butt-joint	2/12; 10/32[a]	1.95 (1.61)[a]
Ozturk (2014)	Turkey, 2008–2011	PCS; 0.5–2	28; 125	Overlap vs butt-joint	6/42; 5/83[a]	3.95 (0.68)[a]

Abbreviations: F, female; HR, hazard ratio; M, male; NA, not available; PCS, prospective cohort study; RCS, retrospective cohort study; RCT, randomized controlled trial; RR, risk ratio; USPHS, US Public Health Service.
[a] *Not directly provided in papers but calculated from percentages or estimated from Kaplan-Meier curves.*
[b] *Estimated from Kaplan-Meier curves and lifetime table.*
[c] *calculated from the original data provided by the author.*

Of the 10 studies, descriptions of preparation designs varied, although they were referred to as the same type. The window type was sometimes described as the nonoverlap design or the design with uncovered incisal edge. The butt-joint type was also called the incisal bevel type or modified overlap design, while the overlap type was also called the functional type, full-veneer type, or even overlap type with palatal chamfer. The heterogeneity of names was then unitized and recoded into the same definition according to the respective reporting preparation methods. Regarding the comparisons, four reported with vs without incisal coverage,[14,15,17,29-31] three reported overlap type vs butt-joint type,[19-21,28] one reported butt-joint type vs window type,[18] and two reported overlap type vs window type.[16,27] Of these comparisons, the latter three could be categorized into "with vs without incisal coverage" simultaneously. As a result, four different meta-analyses for HR or RR estimates were performed based on the study comparisons.

Study Quality

The quality of included studies was evaluated with a global score consisting of three fields: Selection, Comparison, and Outcome (Table 3). In total, nine (90%) studies were of high quality, and only one (10%) was of moderate quality. Two studies included patients with worn-out teeth or bruxism, so they could not be scored for the item of representativeness. Similarly, an additional three studies did not meet the requirement of comparability, duration of follow-up, and adequacy of follow-up, respectively.

Failure Risk of Preparation With vs Without Incisal Coverage

Seven studies involving 1860 PLVs were included in this comparison, two of which were excluded because limited information was provided to estimate the HR value. The pooled HR using a random-effects model revealed that PLVs with incisal coverage had a worse prognosis compared to those

Table 2: *Characteristics of the 10 Included Studies (ext.)*

First Author	Potential Confounding Factors					
	Porcelain Materials	Location of Prostheses	Tooth Vitality	Adhesive Systems	Bruxism Excluded	Same Operator
Meijering (1998)	Feldspathic: Flexo-ceram	Maxillary anteriors	Vital+non-vital	Flexo-ceram	NA	No
Dumfahrt (1999, 2000)	Feldspathic: Optec	Maxillary and mandibular anteriors and premolars	Vital+non-vital	Multiple: Variolink; Optec; etc.	No	No
Smales (2004)	Feldspathic: Mirage	Maxillary and mandibular anteriors	NA	Multiple: Mirage; Ultra-Bond	Yes	No
Cortert (2009)	Nonfeldspathic: IPS Empress	Maxillary and mandibular anteriors	Vital+non-vital	Variolink II	Yes	Yes
Du (2009)	Nonfeldspathic: Vintag; Ceramco	Maxillary and mandibular anteriors and premolars	Vital only	3M ESPE	No	Yes
Granell-Ruiz (2010)	Nonfeldspathic: IPS Empress	Maxillary and mandibular anteriors and premolars	Vital only	Syntace	NA	No
Beier (2012)	Feldspathic and non-feldspathic (NA)	Maxillary anteriors	Vital only	Multiple: Optibond FL; Syntac Classic; etc.	NA	NA
Gurel (2012, 2013)	Feldspathic and nonfeldspathic: IPS Empress I/II/ Esthetic; Creation	Maxillary and mandibular anteriors and premolars	Vital+non-vital	Multiple: Variolink II; 3M ESPE; Variolink Veneer; etc.	No	Yes
Guess (2008, 2014)	Nonfeldspathic: IPS Empress	Maxillary and mandibular anteriors	Vital only	Variolink II	NA	No
Ozturk (2014)	Nonfeldspathic: IPS Emax	Maxillary anteriors	Vital+non-vital	Variolink Veneer	Yes	Yes

Table 3: *Study Quality Assessment According to the Newcastle-Ottawa Scale*

Study	Selection				Comparability	Outcome			Global Score
	Representativeness of Exposed Cohort	Selection of Nonexposed cohort	Ascertainment of Exposure	Outcome Not Present at Start		Assessment of Outcome	Follow-Up Long Enough	Adequacy of Follow-Up	
Meijering (1998)	*	*	*	*	*	*	*		7
Dumfahrt (1999, 2000)		*	*	*			*	*	5
Smales (2004)	*	*	*	*	*	*	*	*	8
Cortert (2009)	*	*	*	*	**	*		*	8
Du (2009)		*	*	*	**	*	*	*	8
Granell-Ruiz (2010)	*	*	*	*	*	*	*	*	8
Beier (2012)	*	*	*	*	*	*	*	*	8
Gurel (2012, 2013)		*	*	*	**	*	*	*	8
Guess (2008,2014)	*	*	*	*	*	*	*	*	8
Ozturk (2014)	*	*	*	*	**	*		*	8

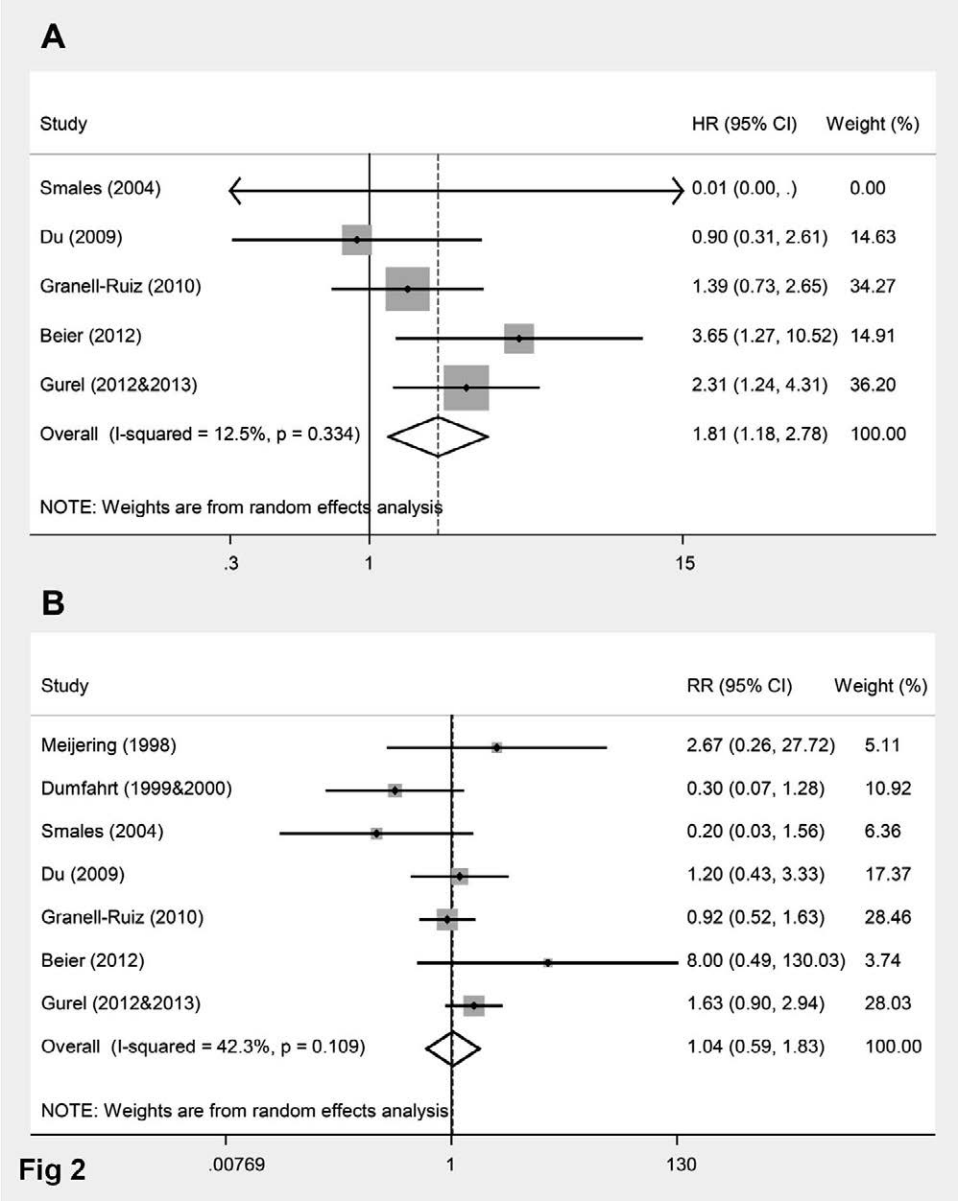

Figure 2. *Meta-analysis of pooled hazard ratios (HRs) (A) and risk ratios (RRs) (B) comparing preparations with and without incisal coverage.*

without incisal coverage (HR=1.81, 95% CI=1.18-2.78) (Figure 2A). Test of inconsistency (I^2=12.5%, p=0.334) excluded significant heterogeneity. However, subgroup analyses were still performed to investigate differences in the results with respect to the porcelain materials, location of prosthesis, and tooth vitality. In the subgroup analysis concerning the porcelain materials, we identified two studies involving both feldspathic and nonfeldspathic PLVs indicating a statistically significant association between failure risk and preparation type with incisal coverage (HR=2.60, 95% CI=1.52-2.65) (Figure 3A). Similarly, when divided based on location of prosthesis, the study involving maxillary anteriors revealed that a significantly increased failure risk was related to PLVs with incisal coverage compared to those without coverage (HR=3.65, 95% CI=1.27-10.52) (Figure 3C). Moreover, studies with PLVs restored on both vital and nonvital teeth indicated that an increased risk was linked to preparations with incisal coverage (HR=2.31, 95% CI=1.24-4.31) (Figure 3E).

Nevertheless, the pooled RR of all studies showed that the failure risk was not related to the preparation type with incisal coverage (RR=1.04, 95% CI=0.59-1.83, I^2=42.3%, p=0.109) (Figure 2B). Then we also divided studies based on the preset perspectives. However, neither of the subgroup analyses regarding the porcelain materials, location of pros-

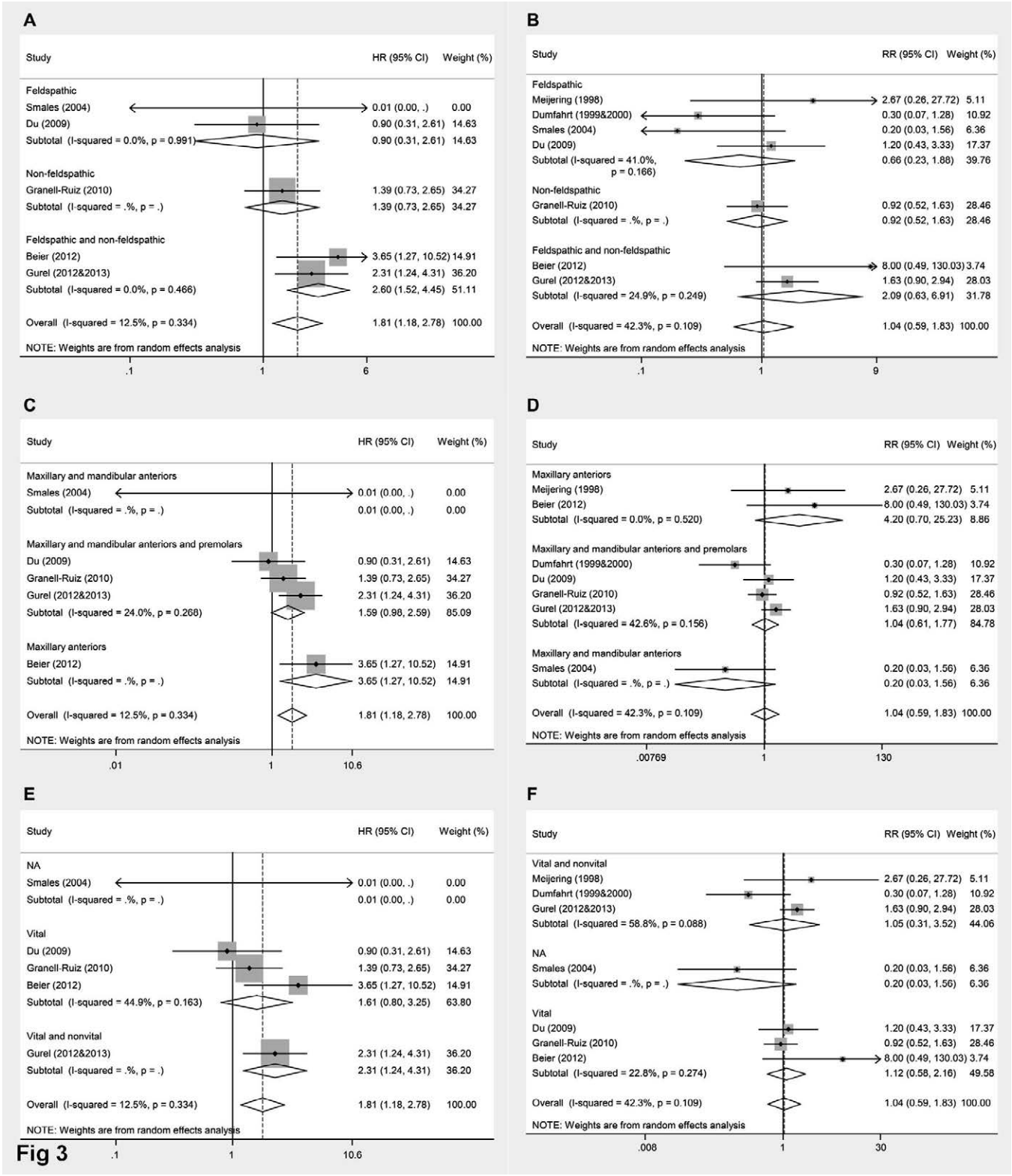

Figure 3. *Subgroup analyses of pooled hazard ratios (HRs) (A,C,E) and risk ratios (RRs) (B,D,F) comparing preparations with and without incisal coverage.*

Table 4: *Sensitivity Analysis for the Pooled Hazard Ratios (HR)/Risk Ratios (RRs) by Omitting a Single Study Sequentially*

Study Omitted	HR (95% CI)	RR (95% CI)
With vs without incisal coverage		
Meijering (1998)	—	0.98 (0.53, 1.79)
Dumfahrt (1999, 2000)	—	1.21 (0.72, 2.01)
Smales (2004)	1.81 (1.10, 2.97)*	1.16 (0.69, 1.95)
Du (2009)	2.01 (1.33, 3.04)*	0.99 (0.49, 2.00)
Granell-Ruiz (2010)	2.06 (1.17, 3.65)*	1.05 (0.47, 2.33)
Beier (2012)	1.62 (1.08, 2.46)*	0.97 (0.56, 1.67)
Gurel (2012, 2013)	1.59 (0.88, 2.85)	0.87 (0.43, 1.74)
Overlap type vs butt-joint type		
Cortert (2009)	3.55 (1.04, 12.12)*	1.18 (0.28, 5.09)
Guess (2008, 2014)	0.69 (0.02, 21.30)	0.55 (0.03, 9.64)
Ozturk (2014)	0.33 (0.02, 4.64)	0.25 (0.06, 1.00)

*$p < 0.05$ in significance test of the pooled effect statistic.

thesis, and tooth vitality yielded statistically significant outcomes (Figure 3B,D,F).

To evaluate the stability of our results, we performed a sensitivity analysis by omitting a single study sequentially and recalculating the summarized HR or RR for the remaining studies (Table 4). The result changed only when the study by Gurel and others[15,17] was removed, indicating that our findings were quite robust and reliable. In addition, visual inspection of Begg's funnel plot and Begg's test to evaluate publication bias revealed no evidence of publication bias among studies investigating preparations with or without incisal coverage (HR: p=0.734; RR: p=0.764) (Figure 4).

Failure Risk of Overlap Type vs Butt-Joint Type

A total of three studies were eligible for the comparison of the overlap type and butt-joint type. The global analysis of either the pooled HR or the RR estimates revealed insignificant results (HR=0.91, 95% CI=0.07-12.02; RR=0.54, 95% CI=0.09-3.32) (Figure 5), indicating no distinction between the failure risk of the overlap type compared to the butt-joint type. The test of inconsistency indicated that distinct heterogeneity existed among these three studies (HR: I^2=84.3%; RR: I^2=85.3%). Then the subgroup analyses based on location of prosthesis and tooth vitality (because all were composed of nonfeldspathic PLVs) were performed to work out the cause of heterogeneity (Figure 6). The heterogeneity was reduced only when the studies were divided according to the location of prosthesis. The study by Ozturk and others[28] revealed an increased failure risk along with the preparation of overlap type compared to butt-joint type (HR=3.95, 95% CI=1.04-14.98). This finding favors the butt-joint type when determining the reduction of the incisal edge for preparation of a porcelain veneer. Sensitivity analysis, when excluding the study by Cortert and others,[21] made the pooled HR >1 significant (Table 4). However, given the limited number of studies and incomprehensible heterogeneity, the failure risk of the overlap type compared to the butt-joint type remained unclear.

Failure Risk of Overlap Type vs Window Type

Only two studies were eligible for the comparison of the overlap type and window type, and the pooled HR and RR indicated no differences between their failure risks (HR=2.05, 95% CI=0.81-5.18; RR=1.84, 95% CI=0.22-15.64) (Figure 7). The test of inconsistency revealed obvious heterogeneity between the studies (HR: I^2=57.0%; RR: I^2=61.3%). Although the subgroup analyses were limited due to the limited number of included studies, the study by Beier and others[16] involving "feldspathic and nonfeldspathic" PLVs and location in "maxillary anteriors" yielded a positive outcome (HR=3.65, 95% CI=1.27-10.52), which is similar to the results of subgroup analyses performed in the comparison of with vs without incisal coverage.

Failure Risk of Butt-Joint Type vs Window Type

Only one study, by Meijering and others,[18] was eligible for the comparison of butt-joint type and window type. This study could provide only the failure frequency in each cohort so as to calculate the RR estimate of the butt-joint type vs window type. It investigated feldspathic veneers restored on maxillary anterior vital or nonvital teeth. No difference

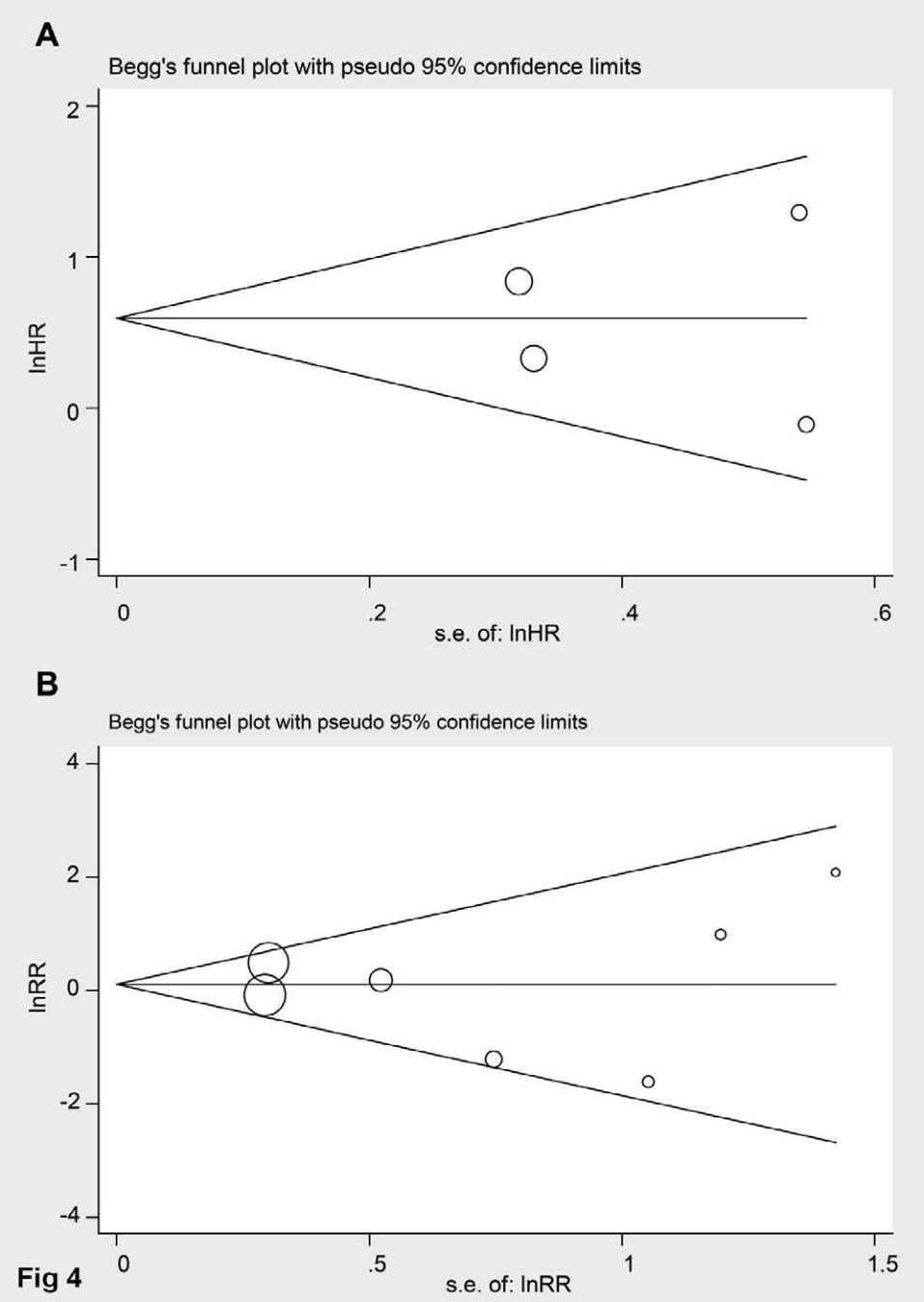

Figure 4. *Begg's funnel plots for included studies for pooled hazard ratios/risk ratios (HRs/RRs) comparing preparations with and without incisal coverage.*

was noted between the failure risks of the butt-joint type compared to the window type (RR=2.67, 95% CI=0.26-27.72).

DISCUSSION

The interest in the association between preparation designs and risk of failure of PLVs is rapidly growing. Various studies have discussed the effect of preparation designs on PLV survival or success since 1998 from the perspective of comparing with and without incisal coverage or comparing any two of the overlap, butt-joint, and window types. Some studies reported no statistically significant survival rates between veneers with and without incisal coverage,[13,14] whereas some studies revealed significantly more failures in veneers with coverage compared to those without coverage.[15-17] Thus, controversy exists in studies investigating any two concrete designs.[13,16,18-21,27-29] To better illustrate the role of preparation types in the prognosis of

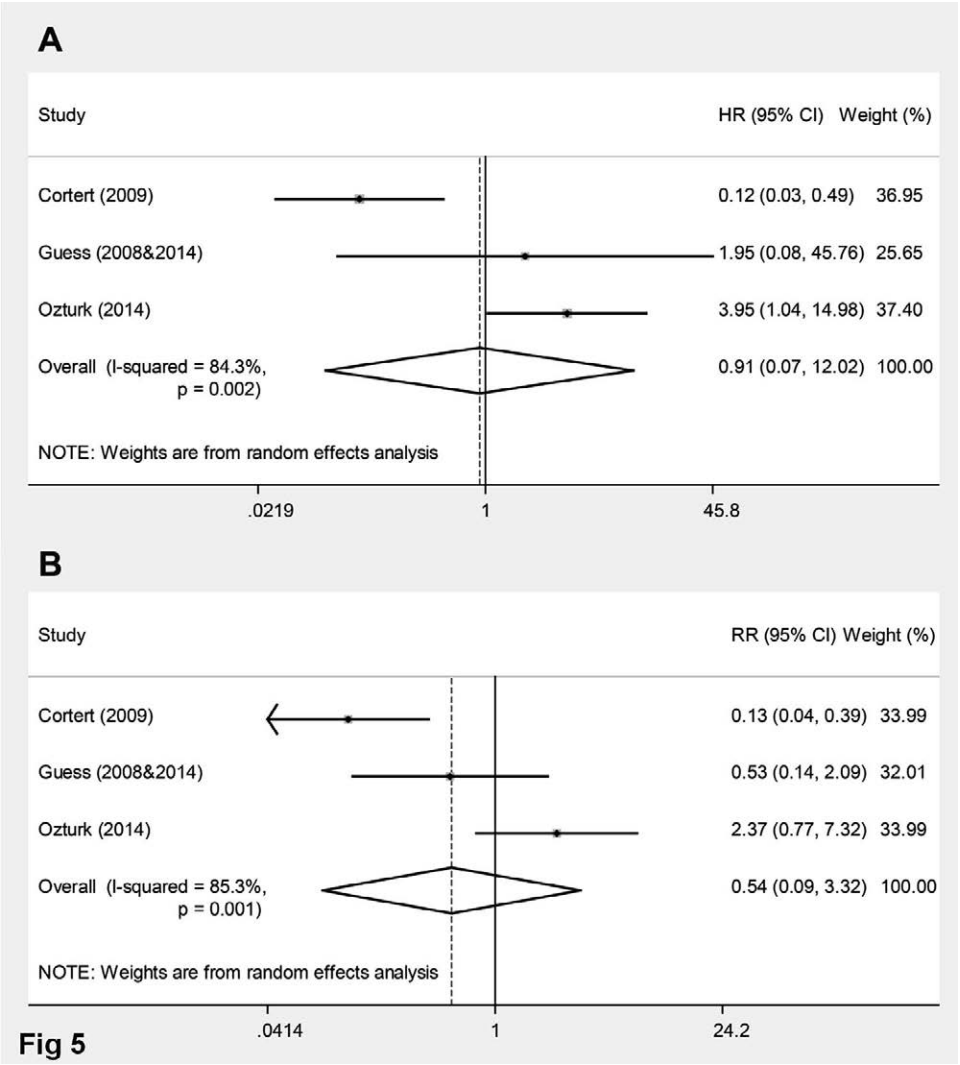

Figure 5. *Meta-analysis of pooled hazard ratios (HRs) (A) and risk ratios (RRs) (B) comparing overlap and butt-joint types.*

PLVs, systematic review and meta-analysis are advantageous to augmenting sample size and statistical power from individual studies. An earlier systematic review focusing on incisal coverage stated that an overall OR of 1.25 pooled from three studies was associated with incisal-edged-covered veneers, but the results were statistically insignificant (95% CI=0.33-4.73).[22] Furthermore, a recommended preparation design among overlap, butt-joint, and window types remains unknown, although a fairly large amount of clinical research has arisen.

The results in the present review indicated that an increased failure was related to incisal coverage. However, as the design of incisal coverage includes the concept of overlap type and butt-joint type, a thorough analysis becomes necessary to clarify which one declines the survival rate of incisal coverage and whether differences exist between overlap and butt-joint types. The multiple comparisons yielded three, two, and one studies, respectively, as well as substantial between-study heterogeneity. Therefore, the evidence was still insufficient to answer the above questions.

The most recent study by Ozturk and others,[28] who investigated nonfeldspathic PLVs (IPS E.max) restored on maxillary anteriors (vital or nonvital), reported an HR of 3.95 comparing the overlap type to the butt-joint type. This finding is consistent with the sensitivity analysis concerning overlap type vs butt-joint type when omitting the study of Cortert and others.[21] Thus, we speculate that an increased risk was associated with the overlap type compared to the butt-joint type. Moreover, a tentative inference was built that the overlap type may predict a poorer prognosis in the comparison of overlap and window types (Figure 7).

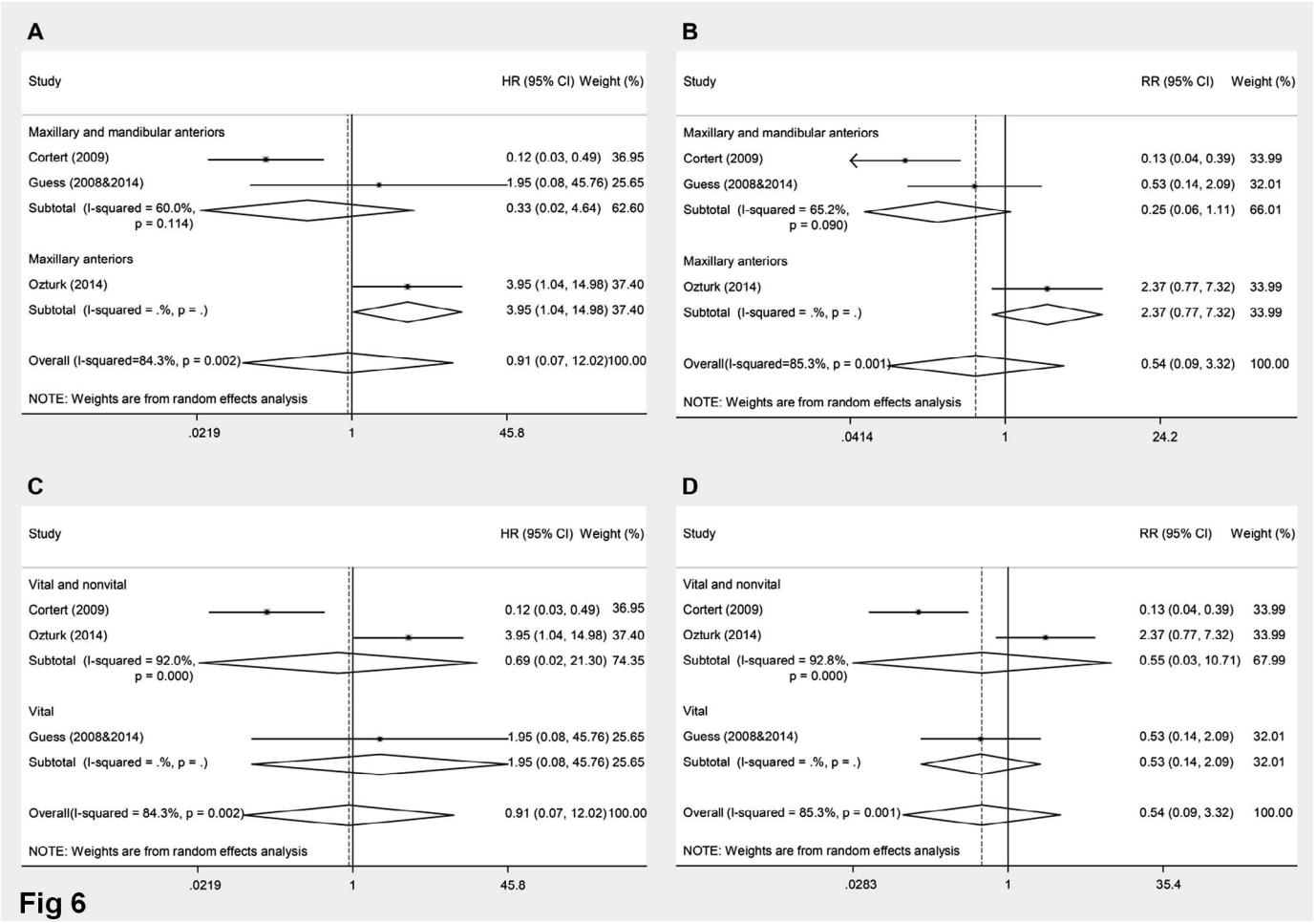

Figure 6. *Subgroup analyses of pooled hazard ratios (HRs) (A,C) and risk ratios (RRs) (B,D) comparing overlap and butt-joint types.*

Finite element analysis (FEA), as corroborative evidence, can help us understand this issue since it is an effective and powerful solution to describe stress distribution with different preparation designs theoretically. The two-dimensional FEA revealed that a butt-joint design suffers less tensile stress but more compressive stress than an overlap design.[5] This is due to the chamfer margin extending to the palatal concavity and consequently an increased stress concentrating at the restoration interface. The characteristic of overlap type was also seen when compared to the window type;[32] however, the overlap type shows a higher principal maximum stress to withstand than the window type.[7] The three-dimensional FEA presented a more uniform stress distribution in the adhesive layer in the overlap design than other designs;[4,6] thus, the overlap type was recommended for PLVs. Nevertheless, Bergoli and others[7] supposed it possible that results of FEA change if fragile materials, such as feldspathic porcelain, are used, which is consistent with the conclusion in the above FEA analysis.[5] In conclusion, we insist that the butt-joint type is recommended when incisal coverage is needed, especially for feldspathic porcelain.

Indeed, the three-type classification for PLVs is not a sufficiently comprehensive method to describe the preparation. Clinicians were often required to deal with the proximal preparation with the consideration of diastema, interproximal caries, and severely discolored teeth. Of the included studies in this systematic review, Cortert and others[21] divided the proximal preparation design into a proximal chamfer and proximal slice, the survival rate of which differed significantly. The reason why the study by Du and others[29] was only classified into preparation with or without incisal coverage was because a Chinese "ILU" classification system was used. The concept of "ILU" arose from the three-dimensional shape of PLVs, with the window type named "I" and the butt-joint or overlap type not

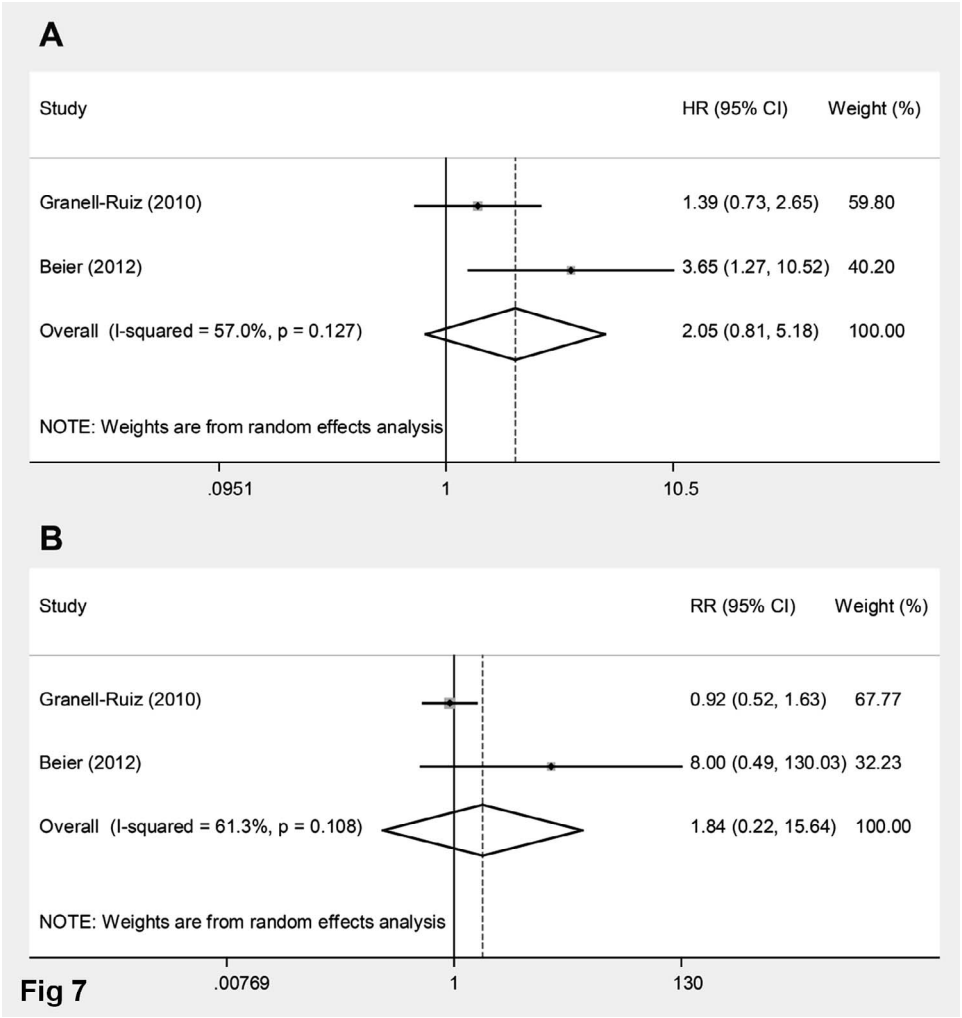

Figure 7. *Meta-analysis of pooled hazard ratios (HRs) (A) and risk ratios (RRs) (B) comparing overlap and window types.*

involving proximal preparation named "L." Thus, the butt-joint or overlap type with proximal preparation was named "U." Given that there was no evidence for the effect of proximal preparation on the survival of PLVs, the "ILU" classification was worth using.

It is worth mentioning that the HR was adopted as the effect statistic in the present meta-analysis. The failure of a veneer is a time-related variable or, rather, time-to-event data instead of a merely categorical variable. Time-to-event data can be treated as dichotomous data only when the observation duration of all investigated individuals is consistent.[33] Therefore, the HR was introduced to describe how many more times a PLV with a specific preparation design suffered failure at a particular time point than did a PLV with another design. As recommended by Parmar,[25] the log HR and its variance were the most appropriate for time-to-event data to improve the efficiency and reliability of meta-analysis. The HR can identify the intrinsic difference more effectively in dealing with time-to-event data than the RR, which is often used in cohort studies measuring dichotomous data. With this in mind, we preferentially extracted HR from Kaplan-Meier curves, lifetime tables, or original data from authors. The routinely used RR was also involved to determine whether results were similar.

Our study has some limitations. First, the number of included studies was limited, especially for detailed comparisons of different preparation designs. Although a variety of well-reported clinical trials or long-term observational studies were included, few involved more than one preparation design.[34-39] Second, in order to work out and reduce heterogeneity among included studies, subgroup analyses were performed in our systematic review. We performed the subgroup analyses stratified by tooth vitality because Coelho-de-Souza and others[40] had pointed out that veneers in nonvital teeth have two times higher risk of failure than those in vital

teeth. However, the heterogeneity was supposed to be reduced theoretically with reasonable grouping factors, some of which remained unaccountable even in subgroup analysis stratified by porcelain materials, location of prosthesis, and tooth vitality. This limitation was likely due to the different evaluation criteria for failure, follow-up duration, and whether one or more operators were involved among the analyzed studies. In addition, the different events of mechanical failure should be categorized into debonding and fracture, which could help categorize relative and absolute failures and determine whether a new porcelain veneer can be made.

Some extra findings were discovered, although not all studies have addressed as many confounding factors as possible. Preparation involving dentin exposure or, rather, the declined percentage of enamel vs dentin bonding surface[17,21,28,41,42] would reduce the long-term survival of PLVs. So did the preparation based on existing composite restorations.[36,43,44] Furthermore, no study has thus far investigated the impact of occlusion types and adhesive systems. Finally, but interestingly, we identified that three studies[15-17,28] published between 2012 and 2014 tended to generate a positive outcome. All of them had quite a large sample size and investigated nonfeldspathic PLVs. It was hypothesized that the failure risks among different preparation designs could be interpreted in ceramics with high strength since feldspathic porcelain is too fragile to survive any other risk factors.

In summary, further studies are needed to clarify the prognosis among overlap, butt-joint, and window types for PLV restoration. We recommend that porcelain materials, adhesive systems, location of prosthesis, tooth vitality, operator, different events of failure, and occlusion types be taken into consideration when designing a clinical trial or analyzing a follow-up study.

CONCLUSIONS

Current evidence indicates that preparations with incisal coverage for PLVs exhibit an increased failure risk compared to those without incisal coverage, whereas there is no difference between overlap and butt-joint types. With the limitation of the present study, well-designed clinical trials or observational studies are needed to confirm our results.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Department of Prosthodontics, Guanghua School of Stomatology, Hospital of Stomatology, Sun Yat-sen University, and Guangdong Provincial Key Laboratory of Stomatology, Guangzhou, China. This study protocol was registered at the PROSPERO (CRD42016040166) and conformed to the proposed MOOSE (Meta-Analysis of Observational Studies in Epidemiology) guidelines.

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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