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Editorial

Lest We Lose Sight

A forgotten question: "What is in the best interest of the patient?"

A late evening phone call from a woman who spent some of her high school days working in our dental office was most welcomed. I had not heard from her in some time. She was having dental care done at a local Midwest dental school and was troubled by the fact that her student dentist had recommended and was planning on restoring tooth #14 with a two-surface composite. She recalled that this was not a material we had used in that location and wanted to know what she should do.

Currently, she is unemployed and on disability and admitted that she knew a gold casting would be preferred. "Would amalgam be better than the composite?" she asked. I replied in the affirmative given her financial situation.

Routine conversation so far, right?

We finished the conversation with my encouraging her to act on what she knew to be best for her and reassured her that she was *ALWAYS* in charge of her own body.

Now comes the interesting part.

Enter the senior dental student, who has the following conversation with the patient.

Patient: I want you to restore my tooth with amalgam.

Dental student: Oh, no, composite is new, tooth colored, and better.

Patient: I don't agree.

Dental student: Trust me. I think you are wrong.

Patient: But it *is* my mouth.

Dental student: Just a minute. [Goes off to find an instructor.]

Then the dental student has the following conversation with the instructor.

Dental student: Tooth #14 needs an MO composite restoration, and the patient is telling me she wants amalgam.

Instructor: The patient is right.

Dental student: But I don't know how to do an amalgam.

Instructor: I'll work with you, don't worry.

Whew! Fortunately, a knowledgeable instructor saved the day for our patient.

However, this raises a question: How much longer will we have educators who are trained in the use of all materials so that informed patients can receive the care they know to be appropriate for them?

Of course, this exchange points to several issues facing dental education today.

1. The environment (real and less real) that surrounds the use of silver amalgam.
2. The pressure from the cosmetic industry, which convinces consumers that having a white restoration is the only way to look good (regardless of length-of-service issues).
3. The struggles facing dental schools to have a well-trained, well-rounded, and well-paid staff so that there is more than the altruistic drive to work in a dental school.
4. The push toward cosmetic dentistry, forgetting that *all dentistry* can be cosmetic when done appropriately.
5. The high costs involved with establishing a practice, which often influences the practitioner to make decisions that are more in the financial best interest and well being of the office than of the patient.

So we come back to the original question: "Taking all factors into consideration, what is in the *best* interest of the patient?"

I worry deeply that if the dental educational system refuses to provide a complete dental education (ie, skills in all dental materials) for their

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students, we are all—both dentists and patients—in deep trouble.

Two things could result (and some might say that they already have):

1. Dental students spend too much money and get an inadequate education.
2. Dental patients are given limited options when it comes to choice in their dental care.

Our profession can do better. After all, look at the information available to us that G.V. Black did not have, and yet he still focused on some basic skill-driven talents, cared about the patients' needs dentally, and was successful.

Each of us needs to be willing to spend time to listen to our informed patients. If they lack information, then we are obliged to give it to them as scientifically and clearly as possible. We then need to look into our own skills tool kit, and where skills are missing, obtain them. We need to practice and practice until we can become truly capable and caring *and listening* servants to the public.

Preachy? Well, maybe, but we still need to always ask, "What's in the best interest of our patient?"

Robert C. Keene, DMD

Staff Letter

Open Letter to All Submitting Authors of Operative Dentistry

Dear Authors,

We are so grateful to each of you for continuing to provide Operative Dentistry with such outstanding manuscripts to consider. We have seen a steady increase each year in the number of manuscripts that are sent to us for publication consideration. For example 20 years ago, during the 1992 volume year, we received 68 manuscripts for consideration, and printed 33 an acceptance rate of 48%; 10 years ago, during the 2002 volume year, we received 212 manuscripts—a 311% increase, 93 were printed putting our acceptance rate at 43%; last year we received 505—a 238% increase over 2002 and a 742% increase over 1992, 80 were printed (more pages than in 2002) for a 15% acceptance rate. As you can see, we are having to become more and more selective in the articles we publish, not only that, but because of the costs of maintaining a manuscript submission system that can deal with this kind of traffic, our costs are exponentially increasing as well. Unfortunately, it has come time that we need to pass a small portion of these costs on to our submitting authors. Operative Dentistry is charged by our submission vendor 25.00USD per manuscript that goes through our system, whether it is accepted for publication or not. Beginning with the first submission of 2013, that cost will now be charged to our authors to submit a manuscript into our system. This 25.00USD fee will be required for a manuscript to be considered in any way. Please understand that this fee is charged to us by our vendor, and will be non-refundable. Paying the submission fee will have no bearing on whether or not your manuscript will be accepted either for review, or for publication. We thank you for understanding the necessity of this step. Should you have any questions about this new policy, please contact our offices at editor@jopdent.org.

Sincerely,

Operative Dentistry Office Staff

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Clinical Research

Clinical Evaluation of Ceramic Inlays and Onlays Fabricated With Two Systems: Five-Year Follow-Up

MJ Santos • RFL Mondelli • MF Navarro
CE Francischone • JH Rubo • GC Santos Jr

Clinical Relevance

The adhesively bonded ceramic restorations presented satisfactory results after five years of clinical service. There was no significant difference between ceramic systems regarding survival.

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SUMMARY

This study evaluated the five-year clinical performance of ceramic inlays and onlays made with two systems: sintered Duceram (Dentsply-Degussa) and pressable IPS Empress (Ivoclar Vivadent). Eighty-six restorations were placed by a single operator in 35 patients with a median age of 33 years. The restorations were cemented with dual-cured resin cement (Variolink II, Ivoclar Vivadent) and Syntac Classic adhesive under rubber dam. The evaluations were conducted by two independent investigators at baseline, and at one, two, three, and five years using the modified United States Public Health Service (USPHS) criteria. At the five-year recall, 26 patients were evaluated (74.28%), totalling 62 (72.09%) restorations. Four IPS restorations were fractured, two restorations presented secondary caries (one from IPS and one from Duceram), and two restorations showed unacceptable defects at the restoration margin and needed replacement (one restoration from each ceramic system). A general success rate

of 87% was recorded. The Fisher exact test revealed no significant difference between Duceram and IPS Empress ceramic systems for all aspects evaluated at different recall appointments ($p>0.05$). The McNemar chi-square test showed significant differences in relation to marginal discoloration, marginal integrity, and surface texture between the baseline and five-year recall for both systems ($p<0.001$), with an increased percentage of Bravo scores. However, few Charlie or Delta scores were attributed to these restorations. In conclusion, these two types of ceramic materials demonstrated acceptable clinical performance after five years.

INTRODUCTION

Ceramic restorations are considered an excellent option to restore posterior teeth when esthetics is required and the size of the cavity preparation has exceeded the conventional indication for direct resin composites. Ceramic systems can combine esthetics with wear resistance, being considered a reliable treatment choice. Peutzfeldt[1] investigated the failure rate of different restorative materials used in posterior teeth and reported that gold inlays did not present a much lower failure rate than ceramic or composite inlays. Consequently, the author suggested that other aspects besides longevity, such as esthetics, price, and number and duration of dental appointments should also be considered when comparing treatment options. Different ceramic systems for fabricating inlay and onlay restorations are available on the market. Some of these ceramic restorations are made using feldspathic ceramic (from the conventional application of a slurry powder onto a refractory die); the CEREC computer-aided design/computer-aided manufacturing, CAD/CAM system, (Sirona Dental Systems GmbH, Bensheim, Germany); or the hot-pressed leucite-reinforced ceramic fabricated by the conventional lost-wax technique (IPS Empress, Ivoclar Vivadent, Schaan, Liechtenstein). Other systems that employ alumina or zirconia are available, but they are generally indicated for single- and multiple-unit crowns.[2,3]

Clinical studies have shown that higher success rates can be achieved when ceramic systems are used in conjunction with an adhesive cementation technique.[4,5] In order to accomplish the bonding requirements, the ceramic should permit selective dissolution via etching to create micromechanical adherence to the resin-based cements.[6] When etchable ceramics are treated with a hydrofluoric acid (HF), a volatile silicon tetrafluoride complex is first formed, and then a second reaction takes place to form a soluble complex ion, hexafluorosilicate, which will further react with the protons to form tetrafluorosilicic acid, which can be rinsed off with water.[2] This reaction enables the etched ceramic surface to be bonded to resin-based cements through the silane. The silane coupling agent is used to promote the chemical adhesion, functioning as a mediator between inorganic and organic substrates through dual reactivity to achieve adhesion.

Although short- and long-term clinical studies have shown low failure rates of adhesively bonded ceramic inlay and onlay restorations, some drawbacks have been reported, bulk fracture and marginal discoloration being the most commonly cited problems.[7–13] Deterioration of marginal quality has been addressed with regard to cement wear, which may be accelerated due to high differences in modulus of elasticity between ceramic and resin cement materials,[8,14] while bulk fracture has been associated with crack propagation through the ceramic, due to the brittle characteristic of the ceramic material.[1] Also, some other factors have been reported as coadjutants on the ceramic crack propagation, such as the microstructure of the ceramic material, the fabrication technique, the surface finishing, and the luting protocol.[15] It has been suggested that an observation period of at least five years should be employed to evaluate the performance of all-ceramic restorations in the posterior region.[16,17]

In that context, the aim of the present prospective study was to evaluate the clinical performance of adhesively bonded all-ceramic inlay and onlay restorations made with two different systems (IPS Empress and Duceram), according to United States Public Health Service (USPHS) criteria over five years. The null hypothesis tested was that there would be no difference in clinical performance between the two systems: IPS Empress and Duceram.

MATERIALS AND METHODS

This study involved 86 Class II inlay and onlay restorations fabricated with two different ceramic systems: 42 sintered ceramics (Duceram Plus and Duceram LFC, Dentsply Degussa Dental, Hanau, Germany) and 44 pressable ceramics (IPS Empress, Ivoclar Vivadent). A total of 33 onlays and 53 inlays were made in 27 premolars and 59 molars by one operator to create a standardized cavity preparation. In patients who had more than one restoration

placed, the two systems were used in an attempt to achieve the same number of each ceramic system in all patients.

Thirty-five patients, including 17 women and 18 men, with a median age of 33 years (ranging from 25 to 44 years) who required inlay and onlay restorations were selected for this study. The involved teeth were in occlusal contact. The volunteers underwent a careful case history review, and bitewing and periapical radiographs were taken. Vitality of the teeth was tested with carbon dioxide snow of −26.2°C.

The following items were considered as exclusion criteria: high caries risk (presence of incipient lesions, plaque, and xerostomia), periodontal disease, the presence of a removable or fixed orthodontic appliance, signs of bruxism or clenching, the absence of more than one unit in the posterior region, and poor oral hygiene or pregnancy. All patients were treated at the Bauru Dental School, University of São Paulo, SP, Brazil. They were informed about the research methodology, risks and benefits, and their right to withdraw participation in this research at any time. A written informed consent was signed. The study was carried out according to research norms and guidelines for human beings deriving from Resolution 196 approved in October 1996 by the National Health Council and Ethics Research Committee from the Bauru Dental School, University of São Paulo, SP, Brazil.

Tooth Preparation

All cavities were prepared according to the general principles for adhesive inlays and onlays.[18–19] The isthmus width was established between 1.5 and 2.0 mm, the pulpal floor depth was between 1.5 and 2.0 mm, the axial wall depth was 1.5 mm, the internal line angles were rounded, and the divergence angle of the cavity was approximately 10° to 15°, with no bevel. For onlays, the cusp reduction was established at 2.0 mm for centric cusps, and 1.5 mm for noncentric cusps. The undercuts were covered with resin-modified glass ionomer (Vitremer, 3M ESPE Dental Products Div, St Paul, MN, USA) to achieve the cavity form by removing the build-up material in order to preserve sound tooth structure. The tooth was prepared by means of a tapered, rounded diamond tips #4137 (ISO #025) and #4138 (ISO #018) (KG Sorensen Ind Com Ltda, São Paulo, SP, Brazil) in a high speed handpiece with water spray. The enamel margins were subsequently finished using hand instruments (Zerfing chisel, Duflex, S.S. White, Rio de Janeiro, RJ, Brazil).

Impression and Provisional Restoration Procedures

Full-arch impressions were made with a polyvinylsiloxane material (Express, 3M ESPE) for the prepared arches and with irreversible hydrocolloid (Jeltrate, Dentsply International Inc, York, PA, USA) for the antagonist arches. Both casts were poured with dental stone type IV (Durone, Dentsply). The bite-registration records were made by a polyvinylsiloxane material (Bite Registration, 3M Dental). Two dental ceramists were selected to produce the inlays and onlays, whose shades were selected from the Classical Vita shade guide (VITA Zahnfabrik, Bad Säckingen, Germany).

Provisional restorations were directly fabricated with the use of self-curing acrylic resin (DuraLay, Reliance Dental Mfg Co, Worth, IL, USA) and fixed with eugenol-free cement (TempBond NE, Kerr, Orange, CA, USA).

Luting Procedures

The intraoral fit was evaluated under rubber dam, and the internal adjustments were performed using diamond burs (KG Sorensen) with low speed. When the fit was not considered satisfactory, the restoration was rejected. Only two restorations were repeated.

Following adjustments, the internal surfaces were sandblasted with 50-μm aluminum oxide particles at a pressure of 87 psi (Opiblast, Buffalo Dental Mfg Inc, New York, NY, USA). These surfaces were then etched with 10% hydrofluoric acid (Dentsply) for 60 seconds and washed, and the silane agent (Monobond-S, Ivoclar Vivadent) was applied for 60 seconds and dried. The cavity was cleaned with pumice slurry and etched with 35% phosphoric acid gel for 15 seconds, rinsed with water, and gently air dried, taking care to avoid desiccation of the tooth substrate. The dentinal surface was treated with a dentin-bonding agent (Syntac primer and adhesive, Ivoclar Vivadent). Subsequently, the cavity preparation and intaglio surface of the ceramic inlays were covered with a layer of bonding agent (Heliobond, Ivoclar Vivadent) that was air-thinned but not light-cured. The dual-cured resin cement Variolink II (Ivoclar Vivadent) was used for the cementation of all inlays and onlays according to the manufacturer's instructions. The same color luting cement was used for all restorations. Polymerization of the luting

agent was performed by light curing the restoration from different positions—occlusal, buccal, lingual, and proximal surfaces for 60 seconds in each direction (XL2500, 570 mW/cm^2; 3M Dental).

Finishing Procedures

Excess luting composite was removed and the occlusal contacts adjusted with diamond finishing burs #1190 FF (ISO #010) and #3203 FF (ISO #012) (KG Sorensen) under water cooling. The surfaces were carefully polished with rubber tips (Cerapol Plus, Edenta AG Dental Rotary Instruments, Au, Switzerland) and the final polishing was conducted using felt discs with diamond polishing gel (KG Sorensen).

Evaluation Procedures

One week following placement, the restorations were assessed according to the modified USPHS criteria[20] (Table 1) by two independent investigators calibrated in the use of the system using only mirrors and probes. The investigators did not participate in the clinical procedures and did not know which system was used on the teeth they were evaluating. The same procedures performed at the baseline were performed at one, two, three, and five years. Statistical analyses were carried out with Fisher and McNemar tests at a 0.05 level of significance.

RESULTS

Table 2 summarizes the results of Alpha ratings obtained for both ceramic materials at baseline and at one-, two-, three-, and five-year recalls, according to the USPHS criteria.

Recall Rate

At five-year recall, 26 patients (including 62 restorations) were evaluated. Thirty-two IPS Empress restorations (72.72%) and 30 Duceram restorations (71.42%) were assessed by two independent evaluators. The recall rate at five-year examination was 74.28%.

Marginal Discoloration/Marginal Integrity

Marginal discoloration increased drastically after the five-year recall. A small number of restorations from both groups were rated as Alpha (Duceram=23.3%; IPS=21.9%). Nevertheless, marginal discoloration was considered clinically acceptable (Bravo), and no Charlie score was rated for any of the ceramic restorative materials. Concerning marginal integrity, only 33.3% of Duceram ceramic restorations and 31.3% of IPS Empress ceramic restorations exhibited perfect marginal adaptation and were rated as Alpha. However, for most of the ceramic restorations from the Duceram and IPS Empress systems, the decrease in marginal integrity was rated as clinically acceptable (Bravo). Just one restoration from each group was rated as Charlie and needed replacement.

Surface Texture

Upon assessment of the surface texture, there was no significant difference between the two ceramic systems ($p>0.05$). The number of restorations presenting ideal surface texture decreased noticeably after five years (Duceram=10%; IPS=25%).

Postoperative Sensitivity/Secondary Caries

After the five-year follow-up, none of the teeth restored with Duceram presented postoperative sensitivity (100%), and just two patients reported sensitivity for the IPS system (93.8%). In relation to secondary caries, one onlay ceramic restoration from the Duceram system and one onlay ceramic restoration from the IPS Empress presented recurrent caries and were classified as failures.

Fracture

Four restorations from the IPS Empress ceramic system exhibited fractures, lowering the Alpha rate to 87.5%. The fractured restorations consisted of two inlays and two onlays located on the molar region. No fractures were recorded for the restorations fabricated with the Duceram ceramic system (100%).

Color Match

In assessing the color match between the restoration and the tooth, 56.7% of the Duceram ceramic restorations were rated Alpha, while 37.5% of IPS Empress restorations were categorized as Alpha. One restoration of the Duceram system was rated Charlie, while four restorations from the IPS Empress system were classified as Charlie. Although the Duceram system showed slightly better results, this difference was not statistically significant ($p>0.05$).

Clinical Success Rate

At the five-year recall, four IPS Empress ceramic restorations were fractured, two restorations presented secondary caries (one from the IPS Empress system and one from Duceram system) and two

Table 1: *Modified United States Public Health Service Criteria for the Clinical Evaluation of Ceramic Inlays and Onlays Used in This Study*

Characteristic	Rating	Criteria
Postoperative sensitivity	Alpha	No postoperative sensitivity
	Bravo	Postoperative sensitivity
Secondary caries	Alpha	No evidence of caries contiguous with the margin of the restoration
	Bravo	Caries evident contiguous with the margin of the restoration
Marginal discoloration	Alpha	No discoloration on the margin between the restoration and the tooth structure
	Bravo	Discoloration on the margin between the restoration and the tooth structure
	Charlie	Discoloration has penetrated along the margin of the restorative material in a pulpal direction
Surface texture	Alpha	Smooth surface
	Bravo	Slightly rough or pitted, can be refinished
	Charlie	Rough, cannot be refinished
Marginal integrity	Alpha	No visible evidence of ditching along the margin
	Bravo	Visible evidence of ditching along the margin not extending to the dentinoenamel junction
	Charlie	Dentin or base is exposed along the margin
	Delta	Restoration is mobile, fractured, or missing
Color match	Alpha	No mismatch in color, shade, and translucency between restoration and adjacent tooth structure
	Bravo	Mismatch between restoration and tooth structure within the normal range of color, shade, and translucency
	Charlie	Mismatch between restoration and tooth structure outside the normal range of color, shade, and translucency
Fracture	Alpha	No evidence of fracture
	Bravo	Evidence of fracture

restorations showed unacceptable defect at the restoration margin, needing replacement (one restoration from each ceramic system). A general success rate of 87% was recorded for IPS Empress and 93.3% for Duceram. The Fisher exact test revealed no significant difference between Duceram and IPS Empress ceramic systems for all aspects evaluated at different recall appointments ($p>0.05$). Regarding the influence of the covariables (inlay × onlay; premolar × molar) on the clinical behavior of ceramic restorations, the Fisher exact test showed no statistical difference ($p>0.05$).

Table 2: *Alpha Results of IPS Empress (IPS) and Duceram (D) Ceramics According to Modified United States Public Health Service Criteria at Baseline, and at One, Two, Three, and Five Years (n = number of restorations evaluated)*

Recall	Baseline (n=86) 100%		1 Year (n=86) 100%		2 Years (n=86) 100%		3 Years (n=79) 92%		5 Years (n=62) 72%	
	IPS	D	IPS	D	IPS	D	IPS	D	IPS	D
Criterion										
Postoperative sensitivity	97.6	92.1	100	100	100	100	100	100	93.8	100
Secondary caries	100	100	100	100	100	100	97.5	97.4	96.9	96.7
Fracture	100	100	100	100	100	100	100	100	87.5	100
Color match	97.7	90.5	95.5	90.5	95.4	90.5	95.0	89.7	37.5	56.7
Marginal discoloration	100	100	75.0	88.1	68.2	76.2	62.5	64.1	21.9	23.3
Marginal integrity	100	100	88.6	90.5	81.8	88.1	77.5	84.6	31.3	33.3
Surface texture	97.7	88.1	97.7	88.1	97.7	85.7	82.5	56.4	25.0	10.0

The McNemar chi-square test was used to draw a comparison between baseline and five-year recall data for each ceramic system. Significant differences in relation to marginal discoloration, marginal integrity, surface texture, and color match were detected between the baseline and five-year recall ($p<0.001$) for the IPS Empress ceramic. For the Duceram ceramic, a significant difference was found in relation to marginal discoloration, marginal integrity, and surface texture ($p<0.001$).

DISCUSSION

The null hypothesis that there would be no difference in clinical performance between the two ceramic systems was supported. At five-year evaluation of adhesively luted ceramic inlay and onlay restorations, no significant difference between Duceram and IPS Empress ceramic systems was found for any aspect evaluated at different recall appointments ($p>0.05$). This result is in accordance with a previous study that reported no significant difference between the conventional porcelain (Vitadur Alpha) and the leucite-reinforced porcelain (IPS Empress) at five-year evaluation.[21] Regarding the conventional ceramic systems, it has been reported that the main disadvantage of fired ceramic restorations is the degree of microporosities and inhomogeneities between the ceramic particles due to the fabrication technique used to process these restorations.[22] On the other hand, the IPS Empress system has been considered a more homogeneous ceramic through the use of precerammed ingots and has also shown superior flexure strength in laboratory tests when compared to less reinforced ceramics.[23] However, the clinical relevance of such a difference between the two systems could not be upheld in this study at five-year evaluation. Both systems presented satisfactory results with a success rate of 87% for IPS Empress and 93.3% for Duceram.

Other studies with a similar time frame of evaluation have reported overall success rates of between 90% and 95% of ceramic restorations investigated.[7,9,21,24,25] In these previous studies, the main cause of failure was associated with bulk fracture of the ceramic restoration and secondary caries.

In the present study, 67 restorations were evaluated at five years. The sample size may not be large, but it is comparable to previous studies[7,9,11,13,25] in which one operator carried out all of the restorative treatments, rather than multiple operators carrying out treatments at multiple centers. The dropout rate with regard to the number of restorations was 22% (19 restorations), and 26% with respect to the number of patients (nine patients). Of the nine patients who could not participate in the five-year recall, four had moved to another city, three could not be reached by telephone nor e-mail, and two did

not want to participate. At five-year recall, eight restorations needed to be replaced (11.9%). Four IPS Empress restorations suffered cohesive bulk fractures (6%), two restorations presented secondary caries (one from IPS and one from Duceram) and two restorations showed unacceptable defects at the restoration margin needing replacement (one restoration from each ceramic system). Bulk fracture is still considered one common problem reported in clinical trials.[26] The failure rate associated with bulk fracture in the present study is in accordance with other studies that performed their evaluations in the same time frame (Arnelund and others,[21] 8% after five years; Naeselius and others,[27] 7.3% after four years; Krämer and others,[9] 5.5% after four years). Molin and Karlsson[7] investigated the five-year performance of three inlay ceramic systems (CAD-CAM, Vita Cerec-Siemens; a conventional porcelain buildup sintering technique, Mirage, Myron; and a glass ceramic casting high-pressure technique, IPS Empress, Ivoclar Vivadent) and reported that among the 60 ceramic inlays placed, five inlays (8%) fractured within the five-year follow-up period, four being from the IPS Empress system and one from the Cerec system.

Unlike metals, resin composites, and dental tissues, ceramic materials are unable to endure elastic deformation to the same level. Ceramic materials possess a high modulus of elasticity and low flexural strength, which is a limiting property of brittle materials.[28]

Fischer and others[29] evaluated the long-term failure probability of different ceramic materials using a computational method and reported a high tendency for failure of the IPS Empress system (2.6% after one year, 4.6% after five years, and 6.0% after 10 years, respectively). Previous clinical studies that have investigated the IPS Empress system have reported different failure rates due to fracture in different periods of evaluation (Studer and others,[26] 2.3% after three years; Galiatsatos and Bergou,[11] 3.1% after six years; Frankenberger and others,[10] 16% after 12 years). However, it is relevant to take in account that factors other than the ceramic material may have an influence on the restoration survival. According to Martin and Jedynakiewicz,[30] the most common reasons for all-ceramic restoration failures are fracture of the ceramic, fracture of the supporting tooth, postoperative hypersensitivity, and wear of the resin luting agent. In their study, the main reason for fracture was usually related to either excessive occlusal loads or insufficient ceramic thickness. The recommended minimum thickness of 1.5 mm should always be observed in order to improve strength. According to van Dijken and others[31] a cuspal overlay should permit a material thickness of ideally 2 mm. Also, the presence of cracks on the ceramic restoration can lead to fracture of the ceramic material by crack propagation under excessive tensile stresses. These cracks can be produced by finishing and polishing procedures, by processing, or by intrinsic defects in the structure of the material.[32] So, a careful finishing procedure should be employed when adjusting the occlusal surface of the restoration, since it has been reported that cracks as small as 25 μm can lead to fracture of the ceramic restoration under function.[28]

Drawing a comparison between the data collected at the five-year recall appointments, no fracture was observed until five-year evaluation. Secondary caries were observed in one restoration from each system at three-year recall, and one more for each system at five-year recall. It is interesting to notice that the number of Alpha scores for marginal discoloration, marginal integrity, and surface texture has declined significantly for both systems ($p<0.001$), from the baseline to all other subsequent follow-ups. Color match showed a significantly reduced performance for the IPS Empress system ($p<0.001$) after five-year evaluation. The explanation may be related to the fading of the extrinsic painting used on the external surface of these IPS ceramic restorations to enhance the esthetic result. In this system, a precerammed monochromatic ingot is selected, heated, and pressed to process the ceramic restoration using the traditional lost-wax technique, while for the conventional porcelain buildup sintering technique, layers with different degrees of translucencies and opacities can be employed.

Because of esthetic reasons, there is a tendency nowadays to select ceramic materials, rather than gold, when dealing with partial restorations. Wagner and others[33] evaluated the performance of gold and ceramic onlays for about seven years and found no statistical difference between the two restorative groups. Federlin and others[34] have reported similar survival rates for both gold and ceramic onlays at five and a half years of evaluation; however, they verified a statistically significant decrease of Alfa ratings for anatomic form and marginal discoloration criteria on the ceramic restorations.

In relation to marginal discoloration and marginal integrity, significant differences were detected between the baseline and the five-year recall data. After five years, significant reduction on Alpha scores was observed for marginal discoloration,

marginal integrity, color match, and surface texture (McNemar chi-square test, $p<0.001$). Several studies have reported a significant increase of marginal discoloration and decrease of marginal integrity over time.[7–13] Frankenberger and others[35] verified that 94% of the surviving restorations exhibited marginal deficiencies after six years. Deterioration and wear of the resin luting agent has been considered a contributory factor in the marginal deterioration of the ceramic restorations.[7,8,11,13,35] This fact was associated with a very high modulus of elasticity of the ceramic material, which during masticatory forces transmits stress to the cement, whose modulus of elasticity is lower.[8,14] Although marginal discoloration has been regarded as a common phenomenon addressed in clinical trials, it has not been considered critical for the clinical performance of the ceramic restorations.[8,11–13]

Some studies[35,36] have considered postoperative sensitivity as another clinical complication; however, it was not an issue in the present study. In this study, all deep areas and undercuts were covered with a resin-modified glass ionomer (Vitremer, 3M ESPE) before final preparation to protect the exposed deep dentin and to help standardize the depth of the pulpal floor (from 1.5 to 2.0 mm). All restorations were adhesively cemented with the three-step etch and rinse Syntac adhesive and Variolink II resin cement. Krämer and others[9] used the same cementation protocol employed in the present study and reported low sensitivity with the use of Syntac adhesive and Variolink resin cement.

The occurrence of dropped participants is inevitable in clinical trials. In the present study 35 patients, representing 86 restorations (100%) were evaluated at the two-year recall. At the five-year examination, 62 restorations in 26 patients were evaluated (74.28%), totalling 32 IPS Empress restorations (72.72%) and 30 Duceram restorations (71.42%).

Comparing inlay vs onlay restoration types and in relation to premolar and molar regions, no statistically significant differences were observed. These results are in accordance with Arnelund and others,[21] whose clinical evaluations revealed no significant difference between ceramic inlays and onlays after five-year evaluation. Manhart and others,[37] however, observed significantly higher failure rates for inlays placed in molars compared with premolars. In the present study, the fractures occurred in the molar region for both inlay and onlay restorations; however, this result was not statistically significant. In addition, despite the fact that molars are usually subjected to more intense chewing forces, this negative effect did not result in significant influence at the five-year recall evaluation.

CONCLUSIONS

It can be concluded that the adhesively bonded ceramic restorations presented satisfactory results at five-year evaluation. The null hypothesis was not rejected, since no significant differences were noticed between the two ceramic systems. The advantages of these partial all-ceramic inlay and onlay restorations include less tooth destruction; avoidance of subgingival restoration margins, usually required by crowns; and good esthetic results.

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Conflict of Interest

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

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Noncarious Cervical Lesions Restored with Three Different Tooth-Colored Materials: Two-Year Results

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Clinical Relevance

Microfilled composite, nanohybrid composite, and compomer give similar results in treatment of noncarious cervical lesions within a two-year evaluation period.

SUMMARY

Introduction: **The aim of this two-year prospective clinical study was to evaluate and compare the clinical performance of three different adhesive esthetic materials in noncarious cervical lesions.**

Material and Methods: **A total of 90 restorations (30 per material) were placed in 30 patients who ranged in age between 18 and 50 years and of both genders, by a single operator with no previous preparation. The restoration of noncarious cervical lesions was done with either a microfilled composite (Esthet.X/Dentsply/De Trey, Konstanz, Germany, and Prime&Bond NT/Dentsply/De Trey), a nanohybrid composite (TetricEvoCeram/Vivadent, Schaan, Liechtenstein, and AdheSE/Vivadent), or a compomer (Dyract eXtra/Dentsply/De Trey and Xeno III Dentsply/De Trey). All restorations were evaluated by independent examiners using a modified US Public Health Service criteria at baseline and after 12 and 24 months for six clinical categories. Data were analyzed statistically by Pearson's chi-**

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square or the Fisher's exact test at 5% significance level ($p<0.05$).

Results: **Results showed that most of the restorations were clinically satisfactory after 12 and 24 months, with no statistically significant differences among the three groups for all evaluated criteria.**

Conclusion: **Treatment of noncarious cervical lesions using composite and compomer materials, combined with the appropriate adhesive systems and properly implemented restorative procedures, gives satisfactory results after a two-year evaluation period.**

INTRODUCTION

Noncarious cervical lesions (NCCLs) represent irreversible loss of hard tooth tissue in the cervical zone of teeth. They may have different forms, from shallow to deep and huge wedge-shaped defects that may be flat, concave, or acute angled. NCCLs are initially located in enamel; however, they progress slowly into the dentin and gradually lead to dentinal sclerosis. Dentinal sclerosis is formed as a response to low-intensity chronic stimuli and as the consequence of physiological aging, consistent with the fact that NCCLs occur more in the older population. However, there is also a high prevalence of noncarious cervical lesions that affect children and adolescents.[1]

If the occurrence of NCCLs is more progressive, these lesions show a marked hypersensitivity, causing discomfort to the patient; this situation lasts until the dentinal tubules are closed. When these lesions become sclerotic from mineral deposits that occlude the dentin tubules, the tooth becomes insensitive to stimuli.[2–4]

In the treatment of NCCLs, clinicians most commonly use composites, glass-ionomer cements, or a combination of two restorative materials with the appropriate adhesive systems. Micromechanical retention, preserving tooth structure, good esthetics, and functional features are all aspects when making the choice of material. Esthetic materials and adhesive systems are constantly being improved in order to enhance adhesion to hard dental tissues, improve esthetic features, reduce polymerization contraction, and simplify the clinical procedures. To get a good bond between the adhesive and dental structures, it is necessary that the enamel and dentin surfaces are appropriately prepared, the adhesive system has a low viscosity, and the treated surface has a low surface tension.[5,6]

The conditioning of dentin significantly affects the quality of adhesive bonding. For the successful restoration of NCCLs, it is necessary to possess a good knowledge of micromorphological characteristics of the sclerotic dentin, which is the basic bonding substrate with the restorative material. Sclerotic, or vitreous dentin, is shiny and dark with a homogenic surface, is considerably tougher when probing, and contains denatured collagen, which significantly hinders the formation of adhesive interlocking.[7,8] The literature shows that the quality of adhesion to sclerotic dentin is weaker when compared to nonsclerotic dentin, as the conditioning of sclerotic dentin is unpredictable because of the higher degree of mineralization and almost complete obliteration of the dentinal tubules, resulting in a lower penetration of adhesives.[8,9] The hybrid layer (resin-reinforced dentin zone) is significantly thinner in sclerotic dentin when compared with normal dentin.[3,9]

The frequent localization of NCCL margins in cementum and/or dentin makes their treatment more difficult, thus making the cervical restoration margins more susceptible to microleakage. The flow of microorganisms and oral fluid due to microleakage may cause marginal discoloration of restorations, postoperative sensitivity, secondary caries, and irreversible pulp disease.[6,7,9]

The aim of this two-year prospective clinical study was to investigate the clinical performance of adhesive esthetic materials for dental restorations in the treatment of NCCLs using modified US Public Health Service (USPHS) criteria.

MATERIALS AND METHODS

The Selection of Patients and Teeth for Restoration

This prospective clinical study lasted 24 months, during which the clinical evaluation of the treatment of NCCLs with different esthetic materials was carried out. The study included 30 patients, aged between 18 and 50 years, and of both sexes. After getting familiar with the type and purpose of the study, the respondents gave their written consent for their participation. The protocol of tests and written consent of the patients were reviewed and approved by the Ethics Committee of the Medical Faculty Novi Sad.

The criteria for inclusion of patients in the study, along with the clinical diagnosis of noncarious cervical lesions, were satisfactory oral hygiene, low caries index, preserved vitality of teeth, and the

absence of periodontitis, periradicular lesions, traumatic occlusion, bruxism, and wear facets, whereas the criteria for excluding patients from the study were anamnestic data indicating pulp pathology of teeth scheduled for inclusion in the study, diagnosed caries with cervical defects, and patients with mobile or fixed prosthetic restorations in the immediate environment of the tooth restorations to be observed in the study.

A detailed dental history was taken from all patients, along with an oral examination, recording the dental and periodontal status, occlusal relationships, movements of the lower jaw, and temporomandibular joint examination.

The final sample consisted of 30 patients, 90 teeth with NCCLs that had a minimum depth of 1 mm, and each patient obtained at least two different restorations and no more than three restorations of the same material, which is in accordance with the recommendations of the American Dental Association on testing adhesive restorative materials in clinical trials.[10]

After the formation of the sample, according to the above-mentioned criteria, there was an additional division of the subjects into three groups, depending on the materials used in the treatment of NCCLs.

The study used the following materials and adhesive systems:

1. Microfilled composite Esthet.X (Dentsply/De Trey, Konstanz, Germany) with a two-step etch & rinse adhesive system, Prime&Bond NT (Dentsply/De Trey), hereinafter EstX-P&B.
2. Nanohybrid composite TetricEvoCeram (Vivadent, Schaan, Liechtenstein) with a two-component two-step self-etching adhesive system, AdheSE (Vivadent), hereinafter TeC-AdSE.
3. Compomer Dyract eXtra (Dentsply/De Trey) with a two-component one-step self-etching adhesive system, Xeno III (Dentsply/De Trey), hereinafter DyeX-XeIII.

Clinical Protocol of NCCL Restoration

Immediately before the restorative procedures, the color of of the teeth to be restored was determined because of the subsequent dehydration of dental tissue and changes of the optical properties of enamel during treatment. The surfaces of the NCCLs were mechanically cleaned with a rotating brush and prophylactic paste without fluoride (Nupro Cups without fluoride, Dentsply/De Trey). It is imortant to emphasize that the lesions were not prepared with any cutting instruments, following the guidelines of the American Dental Association Acceptance program for dentin and enamel adhesive materials, which do not allow the placement of bevels.[10] Isolation was achieved using a cheek retractor, cotton rolls, a saliva ejector, and retraction cord (Ultrapak knitted retraction cord # 1, Ultradent Inc., South Jordan, UT) placed in the gingival sulcus of the treated tooth.

The cavities were restored in accordance with the manufacturer's instructions for each tested material as follows:

1. EstX-P&B. Enamel and dentin surfaces were treated with 36% orthophosphoric acid (DeTrey Conditioner 36 Conditioning & Etching Gel, Dentsply/De Trey) for 20 and 10 seconds, respectively. The cavity was rinsed with water spray for 20 seconds and then dried slightly, taking care to avoid overdrying the dentin. The adhesive system (Prime&Bond NT, Dentsply/De Trey) was applied to the conditioned surface of the cavity using the applicator for 20 seconds, slightly dried, and light polymerized using a SmartLite PS Pen-Style High-Power LED Curing Light (Dentsply/De Trey) for 10 seconds. The composite, Esthet.X (Dentsply/De Trey), was placed into the cavity in two layers, and each layer was polymerized for 20 seconds with the same light source.
2. TeC-AdSE. The surface of each NCCL was first treated with AdheSE Primer (Vivadent) 30 seconds, using the applicator. Excess primer was dried with an air spray until the liquid film on the cavity surface was no longer visible. A thin layer of AdheSE Bond (Vivadent) was applied to the entire dentin and was light polymerized (SmartLite PS Pen-Style High-Power LED Curing Light, Dentsply/De Trey) for 10 seconds. The composite, TetricEvoCeram (Vivadent), was placed in the cavity in two layers, with each layer polymerized for 20 seconds with the same light source.
3. DyeX-XeIII. The adhesive system, Xeno III (Dentsply/De Trey), was previously prepared by mixing liquid A and liquid B in a separate mixing bowl for 5 seconds and then applied to the prepared cavity surface (not overdried). After 20 seconds, it was gently dried by an air spray and polymerized with the SmartLite PS Pen-Style High-Power LED Curing Light (Dentsply/De Trey) for 10 seconds. Dyract eXtra (Dentsply/De Trey) was placed in the cavity in two layers, with each layer polymerized for 20 seconds with the same light source.

Table 1: *Modified USPHS criteria for six clinical categories*

Category	Grade	Criterion
Retention	Alpha (A)	Retained
	Charlie (C)	Partially retained or missing
Marginal integrity	Alpha (A)	Closely adapted, no visible crevice
	Bravo (B)	Visible crevice, explorer will penetrate
	Charlie (C)	Crevice in which dentin is exposed
Marginal discoloration	Alpha (A)	No discoloration
	Bravo (B)	Superficial staining (without axial penetration)
	Charlie (C)	Deep staining (with axial penetration)
Wear	Alpha (A)	Continuous
	Bravo (B)	Discontinuous, no dentin exposed
	Charlie (C)	Discontinuous, dentin exposed
Postoperative sensitivity	Alpha (A)	None
	Charlie (C)	Present
Recurrent caries	Alpha (A)	No caries present
	Charlie (C)	Caries present

The removal of excess material and finishing of the restoration was carried out using diamond burs of different grain fineness, in a dry working area for better visibility and accuracy in order not to damage the enamel. Polishing was done after seven days with the Enhance Finishing and Polishing System (Dentsply/De Trey).

Clinical Evaluation

The operator who placed the restorations did not take part in the clinical evaluation of the test results. That part was carried out by operators who were not familiar with the materials used in restoring the NCCLs, thus forming a double-blind study. All of the restorations were recorded with a digital camera (Nikon Digital Camera D 3000, Nikon Corp., Tokyo, Japan) at each examination. The evaluation of results was done using the modified USPHS criteria (Table 1), and the following were evaluated: retention (R), marginal integrity (MI), marginal discoloration (MD), wear (W), postoperative sensitivity (PS), and secondary caries (SC). The rating A (Alpha) was used to mark the best quality restorations, B (Bravo) minor change, and C (Charlie) an unsatisfactory quality of the restoration. To record the findings, forms were used to include the following: patient's name, the tooth on which the restoration was placed, and the criteria for assessing the quality of the restorations. The forms were completed immediately after finishing the restorations and at clinical evaluations after 12 and 24 months.

The statistical analysis of each criterion among the tested materials was performed using Pearson's chi-square or the Fisher's exact test at a 5% significance level ($p<0.05$).

RESULTS

At baseline, all ratings were 100% Alpha. Recall examinations for all the patients were performed after 12 and 24 months. The results are shown in Table 2.

For *retention rate* after 24 months, there was a loss of six (20%) EstX-P&B, five (16.7%) TeC-AdSE, and five (16.7%) DyeX-XeIII restorations. Regarding *marginal integrity* after 24 months, there were seven (29.2%) EstX-P&B, 10 (40%) TeC-AdSE, and six (24%) DyeX-XeIII restorations that were evaluated Bravo (visible cracks, with no exposed dentin). Charlie ratings (cracks with exposed dentin) were given to four (16.7%) EstX-P&B, five (20%) TeC-AdSE, and two (8%) DyeX-XeIII restorations.

Regarding *marginal discoloration* after 24 months, Bravo ratings (surface discoloration with no axial penetration) were given to six (25%) EstX-P&B, 10 (40%) TeC-AdSE, and seven (28%) DyeX-XeIII restorations. Ratings of Charlie (deep discoloration of the axial penetration) were not observed in any of the restorations placed.

A Bravo rating (discontinuous wear without dentin exposure), for *wear* after 24 months, was given to two (8.3%) EstX-P&B, four (16%) TeC-AdSE, and two (8%) DyeX-Xe III restorations. Charlie ratings (discontinuous wear with dentin exposure time) were not recorded in any of the restorations placed at the end of the evaluation period.

Table 2: *Clinical analysis of restorations of cervical lesions after 12 and 24 months results (cont.)*

Category	Material	After 12 Months			After 24 Months		
		A	B	C	A	B	C
Retention	EstX-P&B	28[a] (93.3)[b]	0	2[a] (6.7) [b]	24[a] (80.0)[b]	0	6[a] (20.0)[b]
	TeC-AdSE	28[a] (93.3)[b]	0	2[a] (6.7)[b]	25[a] (83.3)[b]	0	5[a] (16.7)[b]
	DyeX-Xe III	28[a] (93.3)[b]	0	2[a] (6.7)[b]	25[a] (83.3)[b]	0	5[a] (16.7)[b]
			Fisher, p=1			Fisher, p=0.927	
Marginal integrity	EstX-P&B	21[a] (75.0)[b]	5[a] (17.9)[b]	2[a] (7.1)[b]	13[a] (54.2)[b]	7[a] (29.2)[b]	4[a] (16.7)[b]
	TeC-AdSE	18[a] (64.3)[b]	8[a] (28.6)[b]	2[a] (7.1)[b]	10[a] (40.0)[b]	10[a] (40.0)[b]	5[a] (20.0)[b]
	DyeX-Xe III	25[a] (89.3)[b]	3[a] (10.7)[b]	0	17[a] (68.0)[b]	6[a] (24.0)[b]	2[a] (8.0)[b]
			Fisher, p=0.237			Fisher, p=0.383	
			Chi^2=5.53			Chi^2=4.17	
Marginal discoloration	EstX-P&B	25[a] (89.3) [b]	3[a] (10.7)[b]	0	18[a] (75.0)[b]	6[a] (25.0)[b]	0
	TeC-AdSE	22[a] (78.6)[b]	6[a] (21.4)[b]	0	15[a] (60.0)[b]	10[a] (40.0)[b]	0
	DyeX-Xe III	23[a] (82.1)[b]	5[a] (17.9)[b]	0	18[a] (72.0)[b]	7[a] (28.0)[b]	0
			Fisher, p=0.548			Fisher, p=0.484	
	EstX-P&B	27[a] (96.4)[b]	1[a] (3.6)[b]	0	22[a] (91.7)[b]	2[a] (8.3)[b]	0
Wear	TeC-AdSE	25[a] (89.3)[b]	3[a] (10.7)[b]	0	21[a] (84.0)[b]	4[a] (16.0)[b]	0
	DyeX-Xe III	27[a] (96.4)[b]	1[a] (3.6)[b]	0	23[a] (92.0)[b]	2[a] (8.0)[b]	0
			Fisher, p=0.427			Fisher, p=0.588	
Postoperative sensitivity	EstX-P&B	23[a] (82.1)[b]	0	5[a] (17.9)[b]	24[a] (100)[b]	0	0
	TeC-AdSE	26[a] (92.9)[b]	0	2[a] (7.1)[b]	25[a] (100)[b]	0	0
	DyeX-Xe III	27[a] (96.4)[b]	0	1[a] (3.6)[b]	25[a] (100)[b]	0	0
			Fisher, p=0.166			Fisher, p=1	

After 12 months, *postoperative sensitivity* was present (grade Charlie) in five (17.9%) EstX-P&B, two (7.1%) TeC-AdSE, and one (3.6%) of the DyeX-Xe III restorations. After 24 months, there was a complete regression of postoperative sensitivity (rating change from Charlie to Alpha). *Secondary caries* was not registered in any restoration after the two-year evaluation period. When comparing the

Table 2: *Continued*

Category	Material	After 12 Months			After 24 Months		
		A	B	C	A	B	C
Recurrent caries	*EstX-P&B*	28[a] (100)[b]	0	0	24[a] (100)[b]	0	0
	TeC-AdSE	28[a] (100)[b]	0	0	25[a] (100)[b]	0	0
	DyeX-Xe III	28[a] (100)[b]	0	0	25[a] (100)[b]	0	0
		Fisher, p=1			Fisher, p=1		

Abbreviations: A, Alpha; B, Bravo; C, Charlie; EstX-P&B, Esthet.X + Prime&Bond NT; TeC AdSE, TetricEvoCeram + AdheSE; DyeX-Xe III, Dyract eXtra + Xeno III; Fisher, Fisher exact test; Chi^2, Pearson chi-square test.
[a] The values denote the number of restorations receiving respective scores for each criterion.
[b] Figures in parentheses indicate percentages.

results obtained after 12 and 24 months, no statistically significant differences in any of the criteria among the groups were noticed.

DISCUSSION

Unclear etiology, pathogenesis, diagnosis, and selection of restorative procedures for NCCLs represent a major problem in dentistry and a frequent subject of discussion, as there are still many doubts and contradictions. NCCLs are also a challenge for every clinician because of the difficulties in their restoration.[3]

There are many studies analyzing the *in vitro* behavior of materials while simulating the optimal oral environment. Laboratory tests, although easier, quicker, and more convenient, cannot replace clinical studies, nor can they predict the clinical performance of restorative materials *in vivo*. Therefore, clinical studies are the most reliable when it comes to drawing conclusions about the quality of restorative materials.[11,12] Rapid technological developments and the emergence of new materials on the market, as well as the time required for clinical trials and publication of the results, have significantly reduced the number of published clinical studies dealing with the quality of restorative materials. Likewise, there is an overt tendency to shorten the *in vivo* evaluation period to one year, although observance for an extended period of time is more desirable.[4]

Various tests are used to assess the quality of materials for final restorations, such as the Ryge protocol, the CDA system, and modified USPHS criteria. Modified USPHS criteria are widely accepted and are suitable for a long-term clinical evaluation of restorations and also for comparing the results of different studies.[13] The fault with the modified USPHS criteria, as stated by Hayashi and Wilson, is a frequent overlap of Alpha with Bravo ratings for criteria, such as marginal integrity, marginal discoloration, and restoration wear. The assessment criteria should be better standardized to provide better uniformity of examiners and reliable results; until then, they should be treated cautiously.[14]

Since NCCLs do not have a retentive shape and are not prepared, they represent an appropriate model for testing adhesion of materials to dental tissue. Most researchers use this feature, and today many studies are drawing conclusions about the adhesive properties of materials precisely by examining them on NCCLs.[5,15]

In the current two-year clinical study, with a sample size of 30 subjects, the number (30 for each esthetic restorative material) and distribution of restorations were in accordance with the recommendations of the American Dental Association regarding the clinical examinations of restorative materials.[10]

The retention of restorations is the key criterion by which clinical efficacy of the applied adhesive systems and restorative materials are estimated. This is the most reliable diagnostic criterion and the most obvious sign of a failed restoration since it does not depend on the examiner's subjective assessment.[13]

In the present study, the retention rate after the two-year evaluation period was not statistically

significant from the 12-month evaluation and was between 80% and 83.3% depending on the material. Pollington,[16] while examining composites (Pertac-II) and compomers (Hytac) in combination with a self-etching adhesive system (Prompt L-Pop) placed on NCCLs, obtained a retention rate of 86.6% for the composite and 86.7% for compomers after 36 months. In a one-year clinical study, Burrow and Tyas[17] tested the single-phase two-component adhesive One-Up Bond F, which belongs to the all-in-one adhesive group, in combination with the composite material Palfique Estelite for restoring NCCLs. At the end of their evaluation period, the retention rate was 100%, which is a tremendous deviation from the results of a similar clinical study that tested a similar single-phase two-component adhesive system (Prompt L-Pop, 3M-ESPE, St Paul, MN, USA) where after one year the retention rate was 65%.[18] It is difficult to compare the durability of the restoration of NCCLs with other clinical studies since many factors affect the retention of restorations. The differences in the obtained results can be attributed to differences in the morphology of the cavity, variability, and operator skill; type of occlusion; binding capacity of the restorative system; and the polymerization of the restorative materials.[11,16,18] As a patient ages, dentin becomes more sclerotic, the frequency of NCCLs is higher, and the retention of restorations is decreasing, as shown by Bayne and others.[19] Those authors found that the percentage of the loss of restorations in patients aged 21–40, 41–60, and 61–80 was 31%, 62%, and 75%, respectively.[19] Baratieri and others[20] examined the effect of enamel beveling on the retention of Class V composite restorations. After a three-year clinical evaluation, those authors concluded that beveled margins did not contribute to increased retention rates. Beveling the enamel as a way of improving retention is incompatible with the concept of maximum preservation of tooth structure and preventing further structure loss, which is the basis of contemporary dentistry.[5,21]

The occurrence of inadequate marginal adaptation (ie, the existence of marginal defects) is a sign of degradation of adhesive bonding that leads to the clinical failure of restorations.[21] Polymerization contraction, a different coefficient of thermal expansion between the material, and dental structure and occlusal loading are all potential causes of marginal cracks.[7,21] The quality of marginal attachment depends largely on the type of adhesive system; the physical, mechanical, and viscoelastic properties of materials; and the techniques of restoration. While examining adhesive systems in their extensive one-year clinical study, Van Merbeek and others[22] indicated that failure in all tested adhesive systems showed inadequate margin closure. The differences among results in the literature are largely the consequence of the lack of universal criteria in evaluation, but there is also the possibility of errors during the sensitive restorative procedures. The violation of the marginal integrity may be the result of inadequate finishing and polishing, dimension changes during the polymerization, and/or absorption of water as well as the hygroscopic expansion of the glass-ion components of restorations.[7,21]

The occurrence of marginal discoloration is closely associated with the formation of marginal defects. In the current study, after a two-year evaluation period, the percentage of marginal discoloration and Bravo ratings (surface discoloration with no axial penetration) was higher in group 2 (TeC-AdSE) at 40%, while the restorations in groups 1 (EstX-P&B) and 3 (DyeX-Xe III) presented Bravo ratings of 25% and 28%, respectively. Approximately 70% of the marginal discoloration presented on the mesial and distal margins of the restorations, which are difficult to reach in order to finish the restorations correctly. This leads to the conclusion that marginal discoloration is more likely caused by the accumulation of pigments on the retained steps or cracks than by microleakage.[23,24] In a very large clinical study, Di Lenarda and others[25] examined the marginal discoloration of esthetic restorations. After 48 months, they observed discolouration in 40% of the cervical restorations that were placed without etching the enamel, whereas this change was noticed in 16.7% of the restorations in cases when the total etching technique of enamel was applied, which represents a statistically significant difference between the two studied groups. The above-mentioned phenomenon is associated with inferior adhesive bonding of single-phase self-etching adhesive systems and enamel regarding the conventional three-phase adhesive means.[26] The wear criterion had no statistical significance in the current study.

In the present study, postoperative tooth sensitivity, which was present after 12 months, fully withdrew after two years. Perdigao and others[27] found that the increased sensitivity at the beginning of the evaluation results from retraction of the gingiva and tooth root surface exposure, which occurs immediately after placing a restoration or after its finishing and polishing. Sensitivity that was created immediately after placement of the restoration may be the result of mechanical damage of the

gingiva during finishing and polishing or excess material that was left in contact with soft tissues. Gingivitis will be reversible if the surface of the restoration is well polished, without the existence of steps and uneven parts.

After a two-year evaluation period, secondary caries did not occur in any of the three tested groups. Patients with NCCLs are usually characterized by a low caries index and good oral hygiene, especially after remotivation and training that was performed prior to the restorative procedures; this can explain the absence of secondary caries in this current study.

CONCLUSION

After a two-year evaluation period, no statistically significant differences were observed in any of the criteria of the surveyed group of adhesive esthetic materials. Restoration of NCCLs can be carried out in a satisfying manner using composite and compomer materials in combination with the appropriate adhesive system and appropriate restorative procedures. In addition to the proper selection of restorative materials, the elimination of etiological factors, occlusal balance, and good oral hygiene are also important factors for the longevity and quality of restorations.

Conflict of Interest Declaration

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

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Three-Month Evaluation of Vital Tooth Bleaching Using Light Units—A Randomized Clinical Study

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Clinical Relevance

Light acceleration of the bleaching process, using a laser or halogen light, does not seem to be more beneficial than chemical activation with regard to the stability of tooth color over a period of three months.

SUMMARY

The aim of this study was to evaluate the color stability of vital bleaching using a halogen unit, laser, or only chemical activation up to three months after treatment. A total of 60 patients were divided into three groups, and their teeth were bleached with 38% hydrogen peroxide using three methods: acceleration of the bleaching process with halogen (eight minutes), laser (30 seconds), or chemical activation only. All teeth were bleached a maximum of four times (4 × 15 minutes) until a change of six shade tabs took place. The color was evaluated both visually and with a spectrophotometer before bleaching, immediately after bleaching, and one and three months after bleaching. Directly after bleaching, the use of halogen showed better results than laser ($p \leq 0.05$). One and three months after bleaching, no significant difference was found between the tested methods relative to the shade change, independent of the method of shade evaluation ($p > 0.05$). As far as the color stability is concerned, bleaching with halogen resulted in stable color throughout the three months ($p > 0.05$), whereas the other two methods resulted in whiter teeth after one and three months compared with the color directly after bleaching ($p \leq 0.05$). Bleaching with laser needed more time than halogen for the desired

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shade change ($p \leq 0.05$). Although directly after treatment bleaching with halogen resulted in better results, one and three months after bleaching the kind of acceleration used in the bleaching process did not have any effect on the esthetic results.

INTRODUCTION

The esthetics of the smile and teeth, including tooth color, has become of great importance to patients, resulting in increased requests for tooth bleaching. There are several products and methods described for bleaching of vital teeth, including different concentrations of bleaching agents, times of application, application modes, and the kind of acceleration used with the bleaching agent (ie, by means of chemical activation or using light energy).[1,2] In-office vital tooth bleaching is one of the most popular bleaching methods and is based on the application of 25%-40% hydrogen peroxide products on the external tooth surfaces. The bleaching mechanism is believed to be due to the penetration of hydrogen peroxide into the tooth and the production of free radicals that can oxidize organic stains.[1,3]

The clinical effectiveness of bleaching has been demonstrated extensively.[1,4–6] However, it must be mentioned that usually several applications of the bleaching agent are necessary[7,8] in order to achieve the desired esthetic results. Therefore, the total treatment time needed can be extensive. In order to achieve the same results in a shorter application time, an increase in the efficiency of the bleaching procedure by stimulating the dissociation of the hydrogen peroxide would be helpful. Therefore, recently the use of different light units such as halogen curing lights, LEDs, diode lasers, argon lasers, and plasma arc lamps has been introduced for vital tooth bleaching in order to achieve a better activation of the hydrogen peroxide, resulting in better esthetic results.[8] However, the *in vitro* findings in the literature concerning the efficacy of using light units for the acceleration of the bleaching agents seem to be controversial.[9–13] In previous studies[9–11] it has been shown that bleaching in combination with light units can achieve better results than bleaching with chemical activation. Some other authors[12,13] have shown that the use of light units for acceleration of the bleaching agent was not beneficial compared with the conventional chemical activation. Even if the efficacy of bleaching agents with light units might be beneficial concerning the acceleration of the bleaching agent and, therefore, the esthetic results achieved after the bleaching procedure, their use is still questionable concerning the generation of heat by the light sources, with the danger of causing pulp necrosis.[8] Light-curing units that produce high energy have been shown to result in higher intrapulpal temperature change.[14]

Although the efficacy of tooth bleaching with light units has been widely studied *in vitro*,[6–11] only a few *in vivo* studies[12,15–17] exist concerning the efficacy of vital tooth bleaching by using light units, not giving a clear conclusion about the beneficial use of light acceleration of the bleaching process. Some of these studies[12,17] showed no difference between bleaching with and without light units, whereas Alomari and others[16] found in their study that the use of light units increase the efficacy of in-office bleaching for a short period of time.

The differences in the bleaching methodologies in the published studies, the controversial results, and the difficulty of a standardized color evaluation make further research in this field necessary. Therefore, the aim of the present study was to evaluate the efficacy of the bleaching with regard to the color stability of an in-office bleaching agent after using a halogen unit or laser compared with bleaching without light units over a period of three months, using two different kinds of color evaluation. The hypothesis made was that all three bleaching methods can achieve the same color change and that the color achieved after the end of the bleaching procedure can remain stable for all methods over the period of three months, independent of the method used for the color evaluation.

MATERIALS AND METHODS

For the present *in vivo* study, 60 volunteers/patients were selected. The study was approved by the Ethics Committee of the University of Freiburg, Freiburg, Germany. All patients treated in the study signed an informed consent form after full explanation of the project. The inclusion criteria were as follows: the participants should be healthy; they should not be pregnant; they should not smoke; their teeth should not have been bleached before; and they should be between 18 and 70 years old. Additionally, the color of the upper canines should be so dark that a change of six tab shades, according to the Vita shade guide (Table 1), could take place. For this purpose, the shade guide tabs were arranged from B1 to C4, corresponding to a grade of whitening from 1 to 16.[6,18] Any patients with sensitive teeth, caries, or

Table 1: *VITA Shade Guide With 16 Shades Ranked From the Lightest Color on the Left to the Darkest Color on the Right*

B1	A1	B2	D2	A2	C1	C2	D4	A3	D3	B3	A3,5	B4	C3	A4	C4
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16

composite fillings on the upper canines were excluded from the study.

The patients were randomly divided into three groups (n=20). The tested groups were

1. bleaching without using light to accelerate the bleaching process;
2. bleaching with a halogen unit (Beyond Technology Corp,BEYOND European Headquarters, Berlin, Germany);for eight minutes
3. bleaching with a laser unit (KaVo Dental GmbH, Biberach, Germany) for 30 seconds.

Table 2 provides detailed information about the units used for acceleration of the bleaching process. For the bleaching procedure, the same bleaching agent was used for all the tested groups: Opalescence Boost (38% hydrogen peroxide) (Ultradent Products Inc, South Jordan, UT, USA).

Before bleaching, the teeth were polished with a fluoride-free paste. The bleaching procedure took place for a maximum of 60 minutes (4 × 15-minute sessions). After this, the bleaching procedure was terminated even if the change of six shade tabs was not achieved. Additionally, the bleaching procedure was stopped at the onset of sensitivity or pain and the color change achieved at this point was used for the study.

The light sources were applied at the beginning of each bleaching cycle, meaning that each 15-minute cycle was accompanied by the respective light application, according to the group to which it belonged. Therefore, in the case of the group using the halogen unit, it was applied during the first eight minutes of each 15-minute cycle of the bleaching procedure, and during the remaining seven minutes of each cycle, no light was used. In the case of laser, it was applied for 30 seconds at the beginning of each bleaching cycle.

For the shade evaluation, the color of both canines in the upper jaw was used. The shade change was determined by one examiner with two different techniques: visual evaluation with the VITA shade guide (Table 1); and spectrophotometric evaluation using the tooth vita shade reading from a VITA Easyshade (VITA Zahnfabrik H Rauter GmbH & Co KG, Bad Säckingen, Germany) in the "single tooth shade" mode.

The examiner was previously calibrated for both color selection methods. The shade evaluation took place at the following time periods: before polishing, before bleaching (baseline for evaluation of the bleaching effect), after bleaching, and one and three months after the end of the bleaching procedure. At each tested time point, the visual shade evaluation was performed first and then the digital one.

For each time point, the shade change for each tooth was compared with the baseline. The end of the bleaching procedure was determined according to the shade tab change as identified by the spectrophotometer.

After the bleaching session, patients were advised to avoid the use of red balsamic vinegar, drinking of corrosive drinks, and drinking dark beverages like tea, coffee, red wine, and juices that could stain the teeth for the first three days after bleaching. Oral hygiene instructions included the recommendation

Table 2: *Information on the Light Units Used for the Acceleration of the Bleaching Process*

Light Units					
Name	**Manufacturer**	**Type**	**Power output**	**Wavelength**	**Application Time**
Beyond Whitening Accelerator	Beyond Technology Corp, BEYOND European Headquarters, Berlin, Germany	Halogen	150 W	480–520 nm	8 min
GENTLEray 980	KaVo Dental GmbH, Biberach, Germany	Diode laser	6 W	980 nm	30 s

Table 3: *Mean Values of Shade Changes (Δ-Values) ± Standard Deviations for All Groups and at Each Tested Time Compared With Baseline*

Color Change (Δ Mean Values ± Standard Deviation)					
Bleaching Type	**Shade Evaluation**	**After Tooth Polishing**	**After Bleaching**	**1 mo After Bleaching**	**3 mo After Bleaching**
Bleaching without light	Visual	0.0 ± 0.0	1.75 ± 2.7	2.9 ± 2.9	4.55 ± 1.7
	Digital	0.15 ± 0.4	4.8 ± 3.7	7.25 ± 2.9	7.05 ± 2.3
Bleaching with laser	Visual	0.15 ± 0.6	1.15 ± 1.7	3.65 ± 2.5	4 ± 2.17
	Digital	0.05 ± 0.2	2.15 ± 2.4	6.75 ± 3.2	6.7 ± 2.8
Bleaching with halogen	Visual	0.15 ± 0.6	5.5 ± 2.94	4.85 ± 2.18	4.95 ± 2.3
	Digital	0.25 ± 1.1	6.1 ± 1.9	5.55 ± 2.2	5.1 ± 2.1

to use a medium or soft toothbrush and toothpaste with a low abrasive ability for the duration of the study.

Statistical Analysis

A linear mixed model was fitted with a random intercept (subject=patient). The continuous response variable was modeled as a linear function of time, kind of bleaching, the time-group interaction, and the baseline values as explanatory variables, separately for color evaluation. Variance components were used as a covariance structure. Least-square means and pairwise differences were calculated and *p*-values were adjusted by the method of Tukey-Kramer. All calculations were performed with the statistical software SAS system version 9.1 (SAS, Cary, NC, USA) using the PROC MIXED PROCEDURE. The significance level was set at $\alpha=0.05$.

RESULTS

The mean age of the participants in the study was 27.64 ± 5 years. During the bleaching procedure, four patients in the laser group complained of sensitivity/pain. In these cases, the bleaching procedure was terminated and the shade change and bleaching time were used in the study. For these patients, directly after bleaching, two thin layers of Seal & Protect (Dentsply DeTrey GmbH, Konstanz, Germany) were applied on the bleached teeth in order to reduce the sensitivity. These patients were asked to return to the clinic the day after bleaching in order to evaluate the sensitivity. No sensitivity was present one day after bleaching. No sensitivity was observed after bleaching with the halogen unit or after bleaching without light units. In the present study, no patients were lost and all 60 patients appeared at their recall appointments.

Polishing of the teeth before bleaching did not result in any significant change of color, having no influence on the results of the bleaching methods (Table 3).

The statistical analysis was performed separately for each kind of color evaluation (visual and digital with spectrophotometer). The color changes of each group at each tested time for both types of color evaluation are given in Table 3. The digital evaluation revealed in all cases a greater color shade change compared with the visual evaluation, at each tested time and for all the bleaching methods used. Table 4 provides the results of the pairwise analysis with the Tukey test, comparing the shade change after one and three months with the one after the bleaching procedure, giving important information concerning the stability of the tooth color throughout the study period.

Figures 1–3 illustrate the color change achieved with each bleaching method for both evaluation methods up to three months after the bleaching procedure. Bleaching with halogen showed the greatest shade change directly after bleaching, followed by bleaching without using a light unit and then by bleaching with laser, independent of the color evaluation method. The results at the other two

Table 4: *The Pairwise Analysis of the Tested Bleaching Methods at Each Tested Point of Time, According to Tukey-Kramer Test*

Tukey-Kramer Test (Adjusted *p*-Values)*				
Color Evaluation	**Pairs Compared**	**Directly After Bleaching**	**One mo After Bleaching**	**Three mo After Bleaching**
Visual	Bleaching without light vs bleaching with laser	0.2784	0.9255	0.0578
	Bleaching without light vs bleaching with halogen	0.6610	0.9749	0.1455
	Bleaching with halogen vs bleaching with laser	0.0127	0.9997	0.6455
Digital	Bleaching without light vs bleaching with laser	0.0041	0.8013	0.6267
	Bleaching without light vs bleaching with halogen	0.2047	0.1630	0.1466
	Bleaching with halogen vs bleaching with laser	<0.0001	0.4531	0.1315

** Significance at the level of $\alpha=0.05$.*

tested time points differed between the visual and digital evaluation. The digital evaluation showed bleaching without light>laser>halogen at one month and three months after the bleaching, whereas the visual evaluation showed halogen>laser>without light after one month and halogen-without light >laser at three months after the bleaching.

Visual Evaluation

Bleaching Without Light Unit—The time had a significant effect on the color change ($p=0.0035$). At each tested period the teeth were significantly whiter compared with their initial color ($p\leq0.05$). The Tukey test showed no significant difference between the color after bleaching and one month later ($p\leq0.05$), but the tooth color three months after the bleaching was significantly whiter than directly afterward.

Bleaching With Laser—The time had a significant effect on the color change ($p<0.0001$). At each tested period the teeth were significantly whiter compared with their initial color ($p\leq0.05$). The Tukey test showed that the color one month after the bleaching was significantly whiter ($p\leq0.05$) than the one right after bleaching, but no further significant change took place during the next two months ($p>0.05$). The color remained stable during the last tested period.

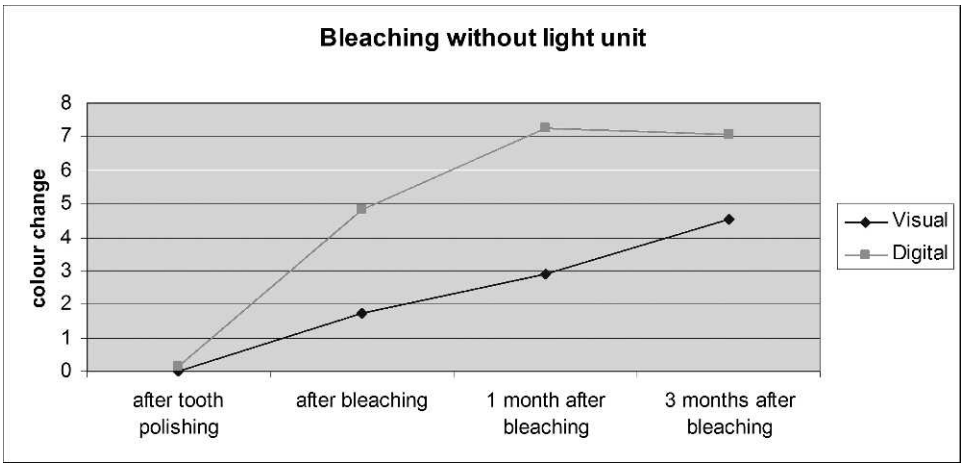

Figure 1. *The color change achieved (according to the VITA shade guide) with bleaching without a light unit is presented for both color evaluation methods up to three months after the bleaching procedure.*

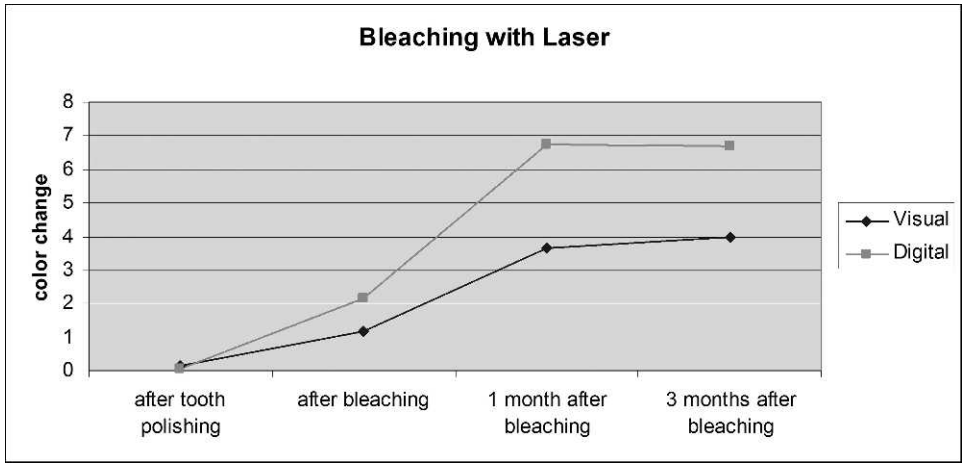

Figure 2. *The color change achieved (according to the VITA shade guide) with bleaching with laser is presented for both color evaluation methods up to three months after the bleaching procedure.*

Bleaching With Halogen Unit—Although it was shown that separately for each tested time period the teeth were significantly whiter compared with their initial color ($p \leq 0.05$), the statistical analysis showed that generally the time did not affect the color change ($p = 0.6754$). According to the Tukey test, the tooth color remained stable over the three months after the bleaching procedure ($p > 0.05$).

Digital Evaluation

Bleaching Without Light Unit—The time had a significant effect on the color change ($p = 0.0241$). At each tested period the teeth were significantly whiter compared with their initial color ($p \leq 0.05$). The Tukey test showed a significant effect on the tooth color through the first month after the bleaching procedure ($p \leq 0.05$). After this time point, the color remained stable up to three months after the bleaching.

Bleaching With Laser—The time had a significant effect on the color change ($p < 0.0001$). At each tested period the teeth were significantly whiter compared with their initial color ($p \leq 0.05$). The Tukey test showed a significant effect on the tooth color through the first month after the bleaching procedure ($p \leq 0.05$). After this time point, the color remained stable up to three months after the bleaching.

Bleaching With Halogen Unit—Although it was shown that separately for each tested time period the teeth were significantly whiter compared with their initial color ($p \leq 0.05$), the statistical analysis showed that generally the time did not affect the color change ($p = 0.1710$). This was also supported by the Tukey test.

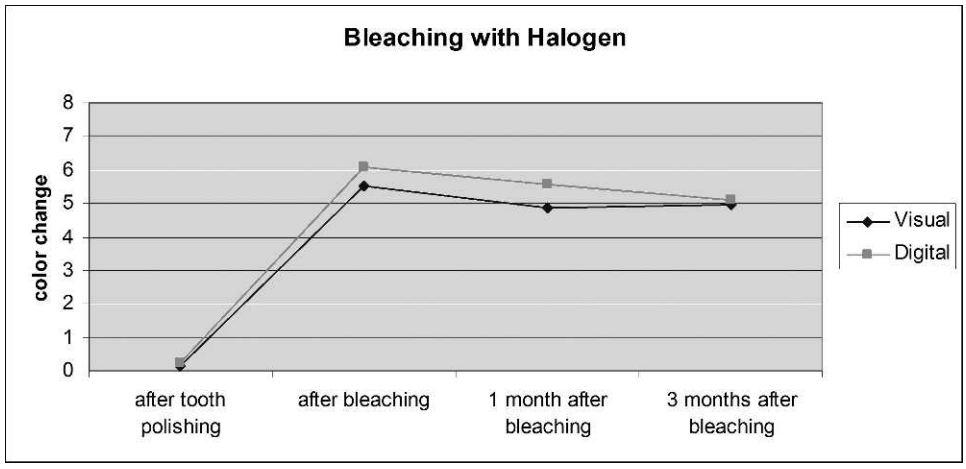

Figure 3. *The color change achieved (according to the VITA shade guide) with bleaching with the halogen unit is presented for both color evaluation methods up to three months after the bleaching procedure.*

Table 5: *The Pairwise Analysis of the Tooth Color at the Different Tested Time Points for Each Bleaching Method*

Tukey Test (Adjusted *p*-Values)*				
Color Evaluation	**Pairs Compared**	**Bleaching Without Light**	**Bleaching With Laser**	**Bleaching With Halogen**
Visual	After bleaching vs 1 mo	0.3245	0.0016	0.6888
	After bleaching vs 3 mo	0.0024	0.0003	0.7653
	1 mo vs 3 mo	0.1042	0.8657	0.9911
Digital	After bleaching vs 1 mo	0.0363	<0.0001	0.5498
	After bleaching vs 3 mo	0.0592	<0.0001	0.1463
	1 mo vs 3 mo	0.9766	0.9983	0.6689

** Significance at the level of $\alpha=0.05$.*

The results of the pairwise analysis of the three bleaching methods tested with the Tukey-Kramer test for each tested time and for both kinds of color evaluation are given in Table 5.

After comparing the three different bleaching methods at each tested time point concerning the shade change, the following were found for the two different methods of color evaluation:

Visual Evaluation—A significant difference was found among the three different bleaching methods directly after the bleaching procedure ($p=0.0011$). At this time point, bleaching with halogen resulted in better results compared with bleaching with laser ($p=0.0127$). One month and three months after the end of bleaching, no significant difference was observed ($p>0.05$).

Digital Evaluation—A significant difference was found among the three different bleaching methods directly after the bleaching procedure ($p<0.0001$). At this time, the Tukey-Kramer test showed that bleaching without a light unit ($p=0.0041$) and bleaching with halogen ($p<0.0001$) resulted in significantly whiter teeth compared with bleaching with laser. One and three months after the end of bleaching, no significant difference was observed ($p>0.05$).

As far as the application time was concerned, a significant difference was found among the three bleaching methods ($p=0.0244$). Longer application time was needed for bleaching with a laser, followed by bleaching without a light unit and then by bleaching with halogen. However, the pairwise analysis with the Tukey test showed no significant difference between bleaching without a light unit and each of the methods using light acceleration of the bleaching process (laser: $p=0.2288$; halogen: $p=0.5157$). A significant difference was shown between the two groups using a light unit ($p=0.0188$). The application times for the three bleaching methods are given in Table 6.

DISCUSSION

In the present *in vivo* study, three different methods were used for tooth bleaching. For all three methods the same bleaching agent (38% hydrogen peroxide) was used, making the comparison of the three methods easier. Four cycles of bleaching, 15 minutes each, was the maximum bleaching time that took place in the present study, in accord with the study

Table 6: *The Bleaching Time of Each Group Needed to Achieve a Whitening of Six Tab Shades*

Acceleration Technique	**Bleaching Time (min)***
Bleaching without light	48.75 ± 12.76[ab]
Bleaching with laser	53.25 ± 12.38[a]
Bleaching with halogen	45.75 ± 11.38[b]

** The use of the same letter does not show any significant difference. Different letter shows significant difference:* $p \leq 0.05$.

of Auschill and others,[19] who found that 3.15 cycles of 15 minutes each were necessary in order to achieve the desired six Vita shade-guide tab changes. The mean age of the volunteers who participated in the study was 27.64 years, making the comparison among the tested groups easier. The age of the patients can influence the results of bleaching because the teeth of older patients are more difficult to whiten than those of younger people, due to the different kind of color changes that occur during the maturation stage of the teeth.

In the present study, only bleaching with laser resulted in tooth sensitivity during the bleaching procedure. This is in contrast to the results of Gurgan and others[17] who found similar esthetic results between the groups with and without light acceleration of the bleaching process; however, they found lower tooth and gingiva sensitivity in the case of diode laser. The pulp temperature rise after application of the laser[20] in combination with the fact that 30 seconds was used as the application time for the laser treatment might be the reason for the sensitivity mentioned. However, this sensitivity existed only on the first day of bleaching and no further symptoms were observed.

Among the three different test methods, bleaching with the halogen unit showed the best results directly after bleaching, followed by bleaching without a light unit. Bleaching with laser revealed the smallest shade change among the groups directly after bleaching. The two different kinds of color evaluation made it difficult to generalize the present results. According to the digital evaluation, only bleaching with the halogen unit achieved the six-tab change directly after the bleaching procedure. Although no significant difference in esthetic results was found between bleaching with halogen and bleaching without a light unit, bleaching with halogen was more efficient directly after bleaching, whereas bleaching without a light unit achieved better results over time, resulting at the end of the three-month period in a similar color change as that achieved with the halogen unit. This is in agreement with the study of Alomari and others,[16] who found a beneficial effect for bleaching with a blue light-curing unit directly after the bleaching procedure, but they could show that this was only for a short period of time and did not affect the long-term results. However, better esthetic results directly after bleaching would probably be beneficial in daily clinical practice in terms of the patients' satisfaction after the bleaching treatment. Our findings are similar to those of Lima and others[13] who found in their study that bleaching in combination with a halogen unit presented the same or higher efficacy than bleaching without using some extra light unit. Not only was the shade-tab change higher in the case of the halogen unit, but additionally the mean time needed to achieve this color change was significantly shorter than the time needed for bleaching with the laser unit. The high power output of the halogen unit used and the presence of the photosensitive agent (beta-carotene) in the bleaching agent might have been the reason for the better results achieved with the halogen unit compared with the laser. The addition of beta-carotene to the bleaching product is supposed to improve its ability to absorb blue light.[13]

Not only the power of radiation but also the wavelength of the laser influences the mechanism of laser systems for bleaching purposes.[8] The poor esthetic results observed in the group treated with laser compared with that treated with the halogen unit might be due to the wavelength of the laser, which is, at 980 nm, far greater than the 400–500 nm at which the beta-carotene strongly absorbs. The wavelength of the halogen unit is better suited to the absorption spectrum of the beta-carotene. This could explain the results of the present study concerning the difference between the two tested light units. Although it can be considered that the use of a diode laser with another wavelength might have resulted in better results, the fact that diode lasers with a wavelength of 980 nm are among the lasers recommended by the Food and Drug Administration[21,22] for tooth bleaching makes the situation tested in the present study clinically relevant because no exact information is given for dental clinicians concerning the appropriate combination of bleaching product and laser unit. The combination of bleaching products containing beta-carotene with lasers with high wavelengths is often seen in the literature.[23–25]

Between bleaching with halogen and bleaching without using some light unit, similar application times were necessary in order to achieve similar esthetic results, according to the statistical analysis. Although the acceleration of the bleaching process with the halogen unit showed good esthetic results directly after bleaching, the fact that the pulp temperature rose during bleaching with a light unit[20] makes chemical activation of the bleaching agent more attractive for daily clinical practice.

In the present study, the laser showed the poorest results directly after bleaching especially in comparison to bleaching with the halogen unit. In the study

of Gontijo and others,[26] no significant difference was found between bleaching with laser and bleaching with a halogen unit after treating teeth that had undergone root canals. Kashima-Tanaka and others[27] showed in their study that the amounts of hydroxide that were generated from the hydrogen peroxide were higher in the case of bleaching by using a plasma arc lamp and halogen unit than by using a laser. However, the different methodologies used among the published studies make their comparison very difficult. Additionally, the light units used among the studies were never the same, making such a comparison very critical. Not all halogen units had the same power output and not all the lasers used in the studies for acceleration of the bleaching process had the same characteristics.

In our study, additional color evaluations took place one and three months after the end of the bleaching procedure. The stability over time of tooth color after bleaching is one of the major concerns of patients after the end of the bleaching treatment. The esthetic results achieved with bleaching accelerated with the halogen unit remained stable over the three months. In contrast to this, bleaching without a light unit and acceleration with the laser resulted in whiter teeth after one month and three months compared with the color achieved directly after bleaching. Therefore, the hypothesis made at the beginning of the study concerning the whitening efficacy of the three bleaching methods and the color stability cannot be accepted.

Our results are in contrast to previous studies[3,12,13,28] that showed a decrease of the color change and darker teeth some time after the bleaching procedures compared with the results achieved directly after bleaching. Wiegand and others[3] found that over a period of 12 months the teeth darkened, but they did not return to the baseline color. In the study of Lima and others,[13] one month after bleaching a color regression was observed; whereas, in the study of Marsio and others,[12] the same results were found after observation of six months. In an older study of Rosenstiel and others,[28] the color regression was seen even seven days after the end of bleaching. These authors[12,13,28] suggested that the single bleaching treatment used might be responsible for the color regression, and multiple treatment sessions were recommended. Alomari and others[16] found that the use of light acceleration of the bleaching agents increased their efficacy only for a short period of time and did not affect the long-term results of the bleaching agents.

A reversible tooth dehydration was thought[14,29,30] to be the reason for the whitening effect of the light on the bleaching efficacy. This could not be confirmed in the present study. The esthetic results remained stable or were enhanced over time.

Given the hypothesis of Greenwall[31] that during bleaching the tooth is filled with oxygen and is dehydrated from the oxidative process, changing the optical qualities of the tooth might offer some explanation of further whitening effect observed in the present study after one and three months. According to Greenwall,[31] after a period of two weeks the oxygen had dissipated and the rehydrated tooth demonstrated the actual lightened effect.

An additional parameter that might have influenced the results of the present study was the viscosity of the bleaching agent used. Opalescence Boost with 38% hydrogen has replaced last year's version of the bleaching agent Opalescence Xtra Boost with the same concentration of hydrogen peroxide. Although no chemical changes have been reported to take place according to the manufacturer, the texture and the consistency of the bleaching agent differs from that of the previous product; it is more viscous than before. The composition and the viscosity of the bleaching agent have an effect on the diffusion of the hydrogen peroxide and, therefore, on the bleaching procedure.[32–34] Bleaching agents with high viscosity show a higher peroxide diffusion compared with less viscous materials.[34] This change in the viscosity of the bleaching agent might be responsible for the different behavior during and particularly after the bleaching procedure because the viscosity of the material influences the peroxide-release kinetics.

One important point of the present study was the comparison of the visual color estimation with the digital one. First the visual shade was determined, and then the digital evaluation took place. Tooth color evaluation in daily clinical practice is of great importance for the esthetic result of any dental treatment. It is rather difficult due to several parameters that can influence color estimation, such as daylight, time of the day, and the color of the patient's clothes. The use of a device for the tooth color evaluation can make the daily dental practice easier. Several devices, such as spectrophotometers or cameras, have been marketed for this purpose. It has been mentioned that a spectrophotometer generates a highly accurate spectral curve indicating the exact L*a*b* values.[35] The Commission international de l'éclairage (CIE) 1976 L* a* b* system is adequately related to human eye color

perception in all three dimensions of color space.[36] The L* values depict the sample lightness, whereas the a* and b* values depict the chroma of the samples. The a* values are a measure of redness (positive a*) or greenness (negative a*). The b* value is a measure of yellowness (positive b*) or blueness (negative b*). The a* and b* values approach zero for neutral colors (white, grays). Unfortunately, there are some disadvantages associated with the use of spectrophotometry-based instruments.[35] Fogging of the optical device can occur, which can lead to inaccurate readings. There are properties of teeth that complicate the use of spectrophotometers. Translucency, an inherent property of teeth, is abstract and intangible and is currently difficult to measure and standardize. Furthermore, the curved surface of the tooth may be problematic because it might negatively impact the uniform reflectance of light to the spectrophotometer. Translucency can be measured when using a spheric optical spectrophotometer designed for industry and research that captures light reflectance from an object in three dimensions. However, this type of instrument is not designed for clinical use because the object to be measured must be placed within the chamber of the spectrophotometer. According to Lath and others,[37] an image system is a reliable alternative measurement method validated against spectrophotometry for stain removal *in vitro* and can provide full color measurement. However, digital evaluation has not been so widely tested as the spectrophotometer. In the present study, the Vita Easyshade intraoral dental spectrophotometer was used. According to the manufacturer, it is a self-contained, portable, digital shade-matching device that is compatible with the 26 Vita System 3D-Master shades, the three Vita System 3D-Master Bleaching shades, and the 16 Vitapan Classical shades. In the literature, the same spectrophotometer has been used in several studies[12,38–41] over the years to evaluate tooth color. Meireles and others[41] evaluated in their study the validity and reliability of the visual assessment of tooth color using the same spectrophotometer used in the present study. In their study, the Vita Easyshade was used as the criterion standard. The sensitivity and specificity of the visual assessment with respect to the criterion standard was 86.9% and 81.9%, respectively, leading to the conclusion that the visual assessment of tooth color using the VITA Classical shade guide is a valid method with good reliability. In the present study, the visual evaluation of the tooth color resulted in darker colors compared with the digital evaluation. As shown in Figures 1–3, only in the case of the halogen unit did the visual and digital evaluation seem to show the same color tab change. Although the estimation of the absolute color differed between the two kinds of evaluation for the other two bleaching methods, the statistical analysis revealed the same results for bleaching with laser concerning the color change to the end of the three-month period. As far as bleaching without any light unit is concerned, the results differed among visual estimation and spectrophotometer, and only those for the three-month period showed similar statistical results. Additionally, these two different kinds of shade evaluation revealed the same statistical results for the one-month and three-month observation. Only directly after bleaching were the different kinds of color evaluation found to result in different conclusions. Therefore, according to the findings of the present study the use of a spectrophotometer seems to be more objective concerning the observation of tooth color, and, therefore, the hypothesis made at the beginning of the study concerning the two different methods for color evaluation cannot be accepted. Additionally, the fact that the visual observation revealed darker values compared with the spectrophotometer leads to the hypothesis that human observations seem to be more critical concerning the esthetic results of tooth bleaching.

CONCLUSIONS

Within the limitations of the present study, bleaching with the halogen unit showed a better whitening effect directly after bleaching compared with bleaching with the laser. Bleaching with the halogen unit was not beneficial compared with chemical activation of the bleaching agent. The whitening effect after bleaching with a laser or bleaching without any light unit increased during one and three months compared with the results achieved directly after bleaching. After a period of three months, the esthetic results achieved were similar for all tested bleaching methods. Therefore, it can be concluded that the use of light to accelerate the process of bleaching is not important for esthetic results with regard to long-term whitening effects.

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 Repeated Preheating Cycles on Flexural Strength of Resin Composites

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Clinical Relevance

Highly repeated preheating cycles seem to negatively influence the flexural strength of resin composites. Assuming dental clinicians are aware they are using the same composite syringe for more than 20 cavities and a preheating procedure is steadily adopted, then the use of single-use composite compules instead of syringes should be adopted.

SUMMARY

The aim of this study was to assess the flexural strengths of three resin composites prepared at room temperature or cured after 20 or 40 cycles of preheating to a temperature of 45°C. Three resin composites were evaluated: Enamel Plus HFO (Micerium) (HFO), Enamel Plus HRi (Micerium) (HRi), Opallis + (FGM) (OPA). One group of specimens for each composite was fabricated under ambient laboratory conditions, whereas in the other groups, the composites were cured after 20 or 40 preheating cycles to a temperature of 45°C in a preheating device. Ten specimens were prepared for each group. A three-point bending test was performed using a universal testing machine at a crosshead speed of 0.5 mm/min. Data were analyzed with a two-way analysis of variance (ANOVA) test and a Games-Howell test ($\alpha = 0.05$). The two-way ANOVA showed that both the material and the number of heating cycles were significant factors, able to influence the flexural strength values ($p<0.05$). However, there was not a statistically significant interaction ($p>0.05$). For all three composites flexural strengths were not affected after 20 preheating cycles in comparison with the control groups (0 preheating cycles) but were, however, significantly decreased

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Table 1: *Summary of the Resin Composites Tested*

Material (Group)	Shade	Composition	Total Content of Filler	Particle Size	Classification
Enamel Plus HFO (HFO)	UD3	UDMA, Bis-GMA, 1,4-butandioldimethacrylate; glass filler, highly dispersed silicone dioxide	75% by weight (53% by volume)	Glass filler: mean particle size of 0.7 μm; highly dispersed silicone dioxide: mean particle size of 0.04 μm	Microhybrid
Enamel Plus HRi (HRi)	UE2	UDMA Bis-GMA, 1,4-butandioldimethacrylate; surface-treated nano zirconium oxide particles with high refractive index (12% by weight); new type of filling glass with high refractive index (68% by weight)	80% by weight (63% by volume)	Glass filler: mean particle size of 1 μm; nano zirconium oxide particles: mean particle size of 20 nm	Nanofilled
Opallis + (OPA)	EA3	Bis-GMA monomers, Bis-EMA, TEGDMA, UDMA; barium-aluminum, silanized silicate, silicon dioxide, camphoroquinone, accelerators, stabilizers, and pigments	78.5% to 79.8% by weight (57% by volume)	Between 40 nm and 3.0 μm, with a mean particle size of 0.5 μm	Microhybrid

Abbreviations: UDMA: diurethane dimethacrylate; Bis-GMA: Iso-propyliden-bis (2(3)-hydroxy-3(2)-4(phenoxy)propyl)-bis (methacrylate) or bisphenol A diglycidyl methacrylate; Bis-EMA: bisphenol A diglycidyl methacrylate ethoxylated; TEGDMA: triethylene glycol dimethacrylate

when 40 prewarming cycles were conducted. The HRi and OPA groups had the highest flexural strengths, with no statistically significant differences among them. HFO presented significantly lower flexural strengths in comparison with HRi.

INTRODUCTION

Handling characteristics such as paste viscosity, packability, stickiness, and polishability play a critical role for composite resins used in restorative dentistry.[1] To achieve a perfectly sealed, long-lasting restoration, material adaptation to cavity walls is of primary importance.[2] In fact, recent literature suggests that there are benefits in increasing the flowability of composite resins by raising the temperature of the composite before placement and thus obtaining a better adaptation in the cavity.[3–14] Warming resin-based restorative materials prior to placement and contouring enhances composite adaptation to preparation walls by decreasing the viscosity of unpolymerized resin composite paste. Preheating may be achieved by placing compules, or syringes, of the resin composite material into commercially available preheating devices that operate at a temperature range of 39°C-68°C. Some *in vitro* studies using commercially available resin composites indicate a significant increase in conversion with an increasing curing temperature, as well as an increase in both polymerization and conversion rates seen at maximum cure rate.[3,4] However, in a recent *in vivo* study, Rueggeberg and others[15] showed that warmed composite lost heat quickly once removed from the heating device and inserted into a tooth preparation. This study indicated that the composite temperature (preheated using a 60°C preheating setting) remained only 6°C to 8°C above intraoral temperature once it was injected; only a slight increase in monomer conversion of preheated composite compared with that of room temperature (RT) material was recorded. From these findings, the authors suggest using the current preheating techniques, being aware of their limitations and with the intent to improve the ease of handling and composite placement.[15] Deb and others[14] showed that the cytocompatibility of composites after preheating remains unaffected. Many studies[8,11,14] disclosed that preheating protocols did not have any harmful effect on the mechanical properties of resin composite materials. However, all the *in vitro* studies in the literature have compared the me-

Table 1: Extended.

Batch No.	Manufacturer
2009000372	Micerium, Avegno, Genova, Italy
2010009717	Micerium, Avegno, Genova, Italy
061208	FGM Produtos Odontológicos, Joinville, Brazil

chanical properties of resin composites cured at RT with those of the same materials cured after a preheating cycle to a determinate temperature. To the extent of the authors' knowledge, only one study analyzed the effect of repeated preheating and cooling cycles, as well as extended periods of preheating on composite cure.[2] This information could be of extreme importance because the same composite syringe can clinically undergo numerous preheating cycles before it is completely consumed. Daronch and others[2] reported that neither prolonged preheating nor repeated (10 continuous cycles of preheating and cooling) compule heating affected the degree of conversion of preheated composites compared with composites maintained at RT. On the basis of these findings, it seems interesting to assess whether the mechanical properties of cured composite can be affected if the number of preheating and cooling cycles are increased to the maximum number that a composite syringe is expected to clinically undergo.

The aim of this *in vitro* study was to assess the flexural strengths of three resin composites prepared at RT or cured after 20 or 40 preheating cycles to a temperature of 45°C. The formulated null hypotheses tested were that the flexural strengths would not be affected by 1) composite selection or by 2) preheating procedures.

METHODS AND MATERIALS

Three resin composites were evaluated in this study: Enamel Plus HFO (Micerium, Avegno, Genova, Italy) (HFO group), Enamel Plus HRi (Micerium) (HRi group), and Opallis + (FGM, Produtos Odontológicos, Joinville, Brazil) (OPA group). Their specifications are given in Table 1. One group of specimens of each material was fabricated under ambient laboratory conditions (21°C ± 1°C), whereas in the other groups the composites were cured after 20 or 40 preheating cycles to a temperature of 45°C in a commercially available preheating device (ENA HEAT composite heating conditioner, Micerium; batch no. SN C1102004).

Preliminary tests were carried out on the three materials to evaluate the heating and cooling times needed at RT (21°C ± 1°C). Temperature variations of the materials were monitored with a digital multimeter equipped with a temperature microprobe (GBC KDM 350, KON EL CO SpA, Milano, Italy). Maximum composite temperature attained was 48.5°C, with the preheating device preset to 55°C. However, after the first 12 minutes (time needed to heat the resin composites to a temperature of 45°C), further small increases in composite temperature required several minutes of heating. Following the results of these preliminary tests, the time needed to heat the resin composites to 45°C (about 12 minutes) was considered to be the most clinically acceptable time to allow the composite to reach a temperature as close as possible to that of the heating device. The same time was required to return the composites to 21°C. Then, each preheating cycle in this study consisted of 12 minutes composite heating in a heating device and 12 minutes of composite cooling at RT.

Ten specimens for each group (n=10) were then prepared using a stainless steel mold with the dimensions specified by the ISO 4049/2000 specification[16] (25 × 2 × 2 mm), positioned over a polyester strip. The materials were inserted into rectangular molds at RT (control groups) or after 20 or 40 preheating cycles. Resin composites were packed into the mold, covered by an acrylate strip, and smoothed with a glass slide to achieve a uniform surface finish. Three overlapping sections of the composite were light cured for 20 seconds with a curing light (Bluephase C8, with a 800 mW/cm^2 output, Ivoclar Vivadent AG, Schaan, Liechtenstein). The final temperatures of the composites before insertion into the mold were gauged with the digital multimeter (KON EL CO). The mean time between removing composite from the heating device

Table 2: *Mean Values (Standard Deviations) Achieved in the Different Groups According to Heating Cycles*

		Groups			*Total*
		HFO	***HRi***	***OPA***	
Heating cycles	0	110.26 (19.73)	126.68 (25.27)	121.98 (10.88)	119.64[a] (20.12)
	20	112.72 (23.79)	128.76 (14.28)	116.01 (17.28)	119.16[a] (19.52)
	40	80.21 (24.59)	95.90 (25.69)	93.96 (6.51)	90.02[b] (18.74)
	Total	101.06[b] (24.59)	117.11[a] (26.46)	110.65[a,b] (17.11)	

[a] *Same superscripted letters indicate means that were not statistically different (p>0.05).*

and light polymerization was approximately 40 seconds for all tests. After irradiation, the excess material on the specimens was carefully removed with a scalpel blade. Specimen dimensions were measured using a digital caliper (series 500 Caliper, Mitutoyo America Corp, Aurora, IL, USA). The specimens were placed into deionized water at 37°C for 24 hours. A three-point bending test was then performed using a computer-controlled universal testing machine (LMT 150, LAM Technologies Electronic Equipment, Firenze, Italy) at a crosshead speed of 0.5 mm/min. The maximum loads were obtained, and the flexural strength (FS) was calculated in megapascals by using the following formula: $FS = 3FL / (2BH^2)$, where F is the maximum load (in newtons), L is the distance between the supports (in millimeters), B is the width of the specimen (in millimeters), and H is the height (in millimeters). The data were statistically analyzed. A two-way analysis of variance (ANOVA) test was performed to analyze the influence of the two factors under investigation (number of heating cycles and material) on the flexural strength mean values. Given that the homogeneity of the variances could not be assumed (Levene test), a Games-Howell test was chosen for post hoc multiple comparisons, with the significance level set at $\alpha = 0.05$.

RESULTS

The two-way ANOVA showed that both the material (p=0.006) and the number of heating cycles (p=0.000) were significant factors, able to influence the flexural strength values. However, there was not a statistically significant interaction between them (p=0.881). As a consequence, pairwise comparisons among the marginal means of the significant main effects were performed. Mean values achieved in the different groups, together with the marginal means and the statistical significance, are shown in Table 2. For all three composites, flexural strengths were not affected after 20 prewarming cycles in comparison with the control groups (0 preheating cycles) but were significantly decreased when 40 prewarming cycles were conducted. For the material, the HRi and OPA groups had the highest flexural strengths with no statistically significant differences among them. HFO presented significantly lower flexural strengths in comparison with HRi.

DISCUSSION

This study showed that flexural strengths of the three different composites tested were significantly affected by composite selection and by repeated composite preheating cycles. Therefore, both the null hypotheses of the present investigation have to be rejected. The three composites had a similar behavior: After 20 prewarming cycles flexural strengths were not affected if compared with the unheated group. However, when 40 prewarming cycles were conducted before light curing, the mean flexural strengths of the composites tested showed a significant decrease. In a clinical situation, the composite can be prewarmed to gain some benefits, such as increased flow and easier adaptation to the cavity.[3–14] The use of temperature to improve flow avoids some of the possible problems associated with a flowable resin material, such as the ongoing release of unreacted monomer and less favorable physical characteristics.[14] Fróes-Salgado and others[12] recently demonstrated that preheated composites showed better marginal adaptation compared with RT composites. Blalock and others[5] showed that the extent of flow varies between brands and resin composite classifications without any correlation

between composite resin classification, filler content, or shape. The decrease in viscosity offered by preheated resin composite never reaches the low levels of a RT flowable composite.[5]

The results of the present study have an important clinical significance, given that there is a consensus in the literature on the absence of harmful effects of preheating procedures on the mechanical properties of resin composites.[8,12,14] Uctasli and others[8] showed that different preheating protocols (40°C, 45°C, or 50°C) do not have any harmful effect on the flexural strength and flexural modulus of tested composite materials. However, the majority of previous studies did not consider repeated preheating cycles. From this point of view, the present study is not in opposition to previous studies[8,12,14] because its findings also suggest that after 20 preheating cycles, the tested composites mechanical properties are not negatively influenced by the heating procedure. The present study is in accordance also with the findings from Daronch and others,[2] who reported that neither prolonged preheating nor 10 repeated continuous preheating cycles (cycles of 15 minutes from RT to 60°C) affected the degree of conversion of preheated composites. The study tested three different commercially available resin composites. For each composite tested, monomer conversion, either after repeated or extended preheating, remained equivalent to RT values, indicating that no resin polymerizable components were lost upon heating nor was there any degradation of monomer during the different heating treatments.[2] However, in clinical use, a standard composite syringe can be used to fill more than 20 cavities especially if a multi-shade layering technique is steadily adopted. From the results of the present study, in these cases the mechanical properties of the preheated resin composites would be decreased.

In this study, flexural strength was investigated to compare the composite groups. Flexural strength is a fundamental mechanical property for brittle materials, although the results cannot be directly extrapolated to the clinical behavior without considering some other aspects, namely flaw distribution[17] and the structural reliability of the material.[18] Nonetheless, the *in vitro* three-point bending flexural test is recommended by the ISO 4049/2000 specification[16] for polymer-based materials and is widely used for comparative purposes.[19,20] The resin-based composites tested showed significant differences in flexural results: the nanofilled resin composite (HRi) showed the highest mean flexural strengths. This is in contrast with other research studies[8,21] that reported higher flexural strength values for microhybrid resin composite compared with nanohybrid resin composite. The present results are probably related to the different filler loading of the composites tested (Table 2). It has been shown that resin composite filler volume fraction and filler load level have a strong correlation to the materials' strength and elastic modulus, as well as fracture toughness.[22,23] Kim and others[24] found that the mechanical properties of resin composites are related to their filler content. According to these findings, in the present study resin composite with the highest filler loading (HRi) exhibited the highest flexural strength; the composite with the lowest filler content (HFO) presented significantly lower flexural strengths in comparison with HRi.

In this study, a slightly lower composite temperature was achieved compared with that of the heating source. This would be expected, as already reported,[2] because composites are filled with inorganic particles and organic resins that function as thermal insulators. For this reason, it was decided to test the composites warmed to 45°C after a standard clinically acceptable preheating time.

In conclusion, from the results of this study it can be assumed that highly repeated preheating cycles seem to negatively influence the flexural strength of resin composites. If dental clinicians are aware that they are using the same composite syringe in more than 20 cavities with a steadily adopted preheating procedure, then the adoption of single-use composite compules instead of syringes would be considered preferable, according to the findings in this study.

Conflict of Interest Declaration

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

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Effect of Bur Roughness on Bond to Sclerotic Dentin With Self-etch Adhesive Systems

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Clinical Relevance

The use of diamond burs with different roughness did not increase the bond strength of self-etch systems and etching pattern in sclerotic dentin. Clinicians should avoid using this procedure when applying self-etch adhesive.

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ABSTRACT

Purpose: **To evaluate the effect of bur roughness on bond strength values and conditioner pattern of two-step self-etch adhesives applied on sclerotic dentin.**

Methods: **The roots of 48 bovine incisors were removed and the crowns were divided into four groups: the control group (CO) teeth were left untreated or the teeth were slightly roughened with coarse-, medium-, or fine-grit diamond burs. Next, the teeth were subdivided and Clearfil SE Bond (CSE) and Adper SE Bond (ASE) were applied according to the manufacturers' instructions. Composite resin (Opallis) buildups were incrementally constructed on the bonded surfaces. After storage for 24 hours in distilled water at 37°C, the teeth were sectioned into sticks (area of 0.8 mm^2). The sticks were stressed until failure by tensile forces (0.5 mm/min). Additionally, eight bovine teeth were treated as previously described, and after adhesive application, the surface was rinsed off and examined by scanning electron**

microscopy to measure the relative number of open tubules (OT). Data (MPa) were analyzed by two-way analysis of variance and Tukey test ($p=0.05$).

Results: CO showed the highest bond strength values ($p<0.05$). As regards OT, the lowest mean was observed for CO ($p<0.05$) and the highest was found after application of CSE or ASE ($p<0.05$).

INTRODUCTION

The basic bonding mechanism to dental substrates is essentially an exchange process involving replacement of the minerals removed from hard dental tissue by resin monomers, which upon setting become micromechanically interlocked in the porosities created. Presently two bonding mechanisms with resin monomers are used with modern adhesive systems: the etch-and-rinse and the self-etch approaches.[1,2]

Regardless of the bonding strategy, bonding to pathologically altered substrates such as sclerotic dentin generally leads to compromised bonding.[3,4] Sclerotic dentin has partially or totally obliterated dentinal tubules as a result of the continuous deposition of peritubular dentin,[5,6] and it is generally present in noncarious cervical lesions.[3,4,6,7] The micromorphological features of this altered dentin substrate are potential obstacles to resin infiltration, which include the hypermineralized surface layer, an additional partially mineralized surface bacterial layer, and intratubular mineral casts that are comparatively more acid-resistant.[3,8,9]

In vitro studies[4,7,10,11] have demonstrated that for etch-and-rinse adhesives, bond strength values in sclerotic dentin are 25%-40% lower than that achieved in sound dentin as a result of the presence of an acid-resistant hypermineralized surface layer. The causes of this mineral deposition are multifactorial, including occlusal stresses, chronic stimuli of low intensity and high frequency, and bacterial colonization.[6]

Hypermineralization inside the dentinal tubules also hinders the formation of resin tags and thus promotes the formation of a thinner and less homogeneous hybrid layer.[8,9,12,13] It has been suggested[3,4,8] that removal of the upper hypermineralized surface layer by bur grinding or stronger acids offers a possible strategy with which to improve micromechanical retention in sclerotic dentin. Several researchers have investigated the effect of these approaches (ie, increasing phosphoric acid conditioning time[10,14-16] and roughening the dentin surface[10,17,18] when using etch-and-rinse systems) and have obtained controversial results.

With regard to self-etch adhesives, phosphoric acid pretreatment[4,13] or roughening the sclerotic surface with diamond burs has also been reported[13] to increase the hybrid layer thickness. Nevertheless, the formation of a thicker hybrid layer cannot be interpreted as a material advantage because the thickness of this layer is not related to high bond strength values.[2] To the best of our knowledge, the effect of roughening dentin with a bur on the bond strength of self-etch adhesives to sclerotic dentin has not yet been investigated. Therefore, the aim of this study was to evaluate the effect of different diamond bur grits on the bond strength of self-etch systems to sclerotic dentin and the etching pattern produced by self-etch systems on the diamond bur–treated dentin surfaces.

MATERIALS AND METHODS

Tooth Selection and Preparation

Fifty-six extracted bovine incisors with exposed sclerotic dentin, clinically characterized by a vitreous appearance,[12,19] were used in this study. They were extracted from the mandibles of three-year-old animals who had been slaughtered on a commercial scale for meat consumption. After harvesting, they were stored in distilled water at 4°C for no longer than one week before being used in this experiment. The roots of all teeth were sectioned and the coronal pulp was removed.

Experimental Design and Restorative Procedure

Forty teeth were then embedded in chemically activated resin, with the exposed dentin surfaces parallel to the horizontal plane. Next, the dentin surfaces of all teeth were cleaned with a detergent (Tergentol, Inodon, Porto Alegre, RS, Brazil). The teeth were randomly assigned into eight experimental groups (n=5) according to the combination of the main factors "adhesive system" (two levels) and "surface treatment" (four levels).

In the control group, dentin surfaces were left untreated. For experimental groups the teeth were slightly roughened with a coarse-grit (#1035, KG Sorensen, São Paulo, SP, Brazil; mean particle size 151 μm), medium-grit (#1035, KG Sorensen; mean particle size 91 μm), or fine-grit (#1035F, KG Sorensen; mean particle size 46 μm) diamond bur mounted in a high-speed hand-piece under water

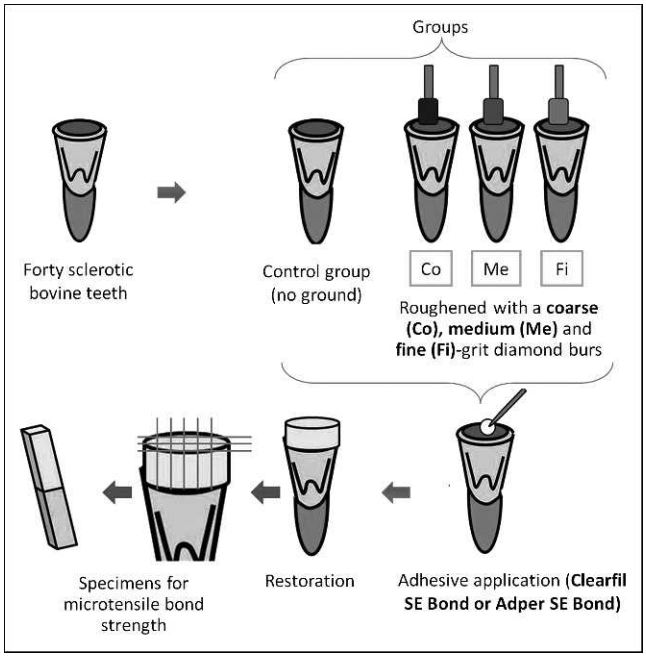

Figure 1. *Flowchart of the microtensile bond strength test.*

cooling for a period of 10 seconds (Figure 1). The teeth were prepared with 10 passes with the bur across the dentin surface under copious air-water spray. Before performing the sample surface preparation, the single operator was training on the surface of an analytical balance to determine the equivalent manual pressure that would be placed on the surface during surface preparation (Mettler, type H6, Columbus, OH, USA). The pressure applied was equivalent to approximately 300 ± 55 g.

After this, the adhesive systems Clearfil SE Bond (CSE; Kuraray, Okayama, Japan) and Adper SE Plus (ASE; 3M ESPE, St Paul, MN, USA) were applied according to the manufacturers' instructions (Table 1). After the bonding procedure, all teeth received a microhybrid composite restoration (Opallis, FGM Produtos Odontológicos, Joinville, SC, Brazil) in two increments of 2 mm that were each light polymerized for 40 seconds using a halogen light-curing unit set at 400 mW/cm^2 (VIP, Bisco, Schaumburg, IL, USA), without use of a matrix, as conventionally performed for the microtensile bond strength test.[11,13] The specimens were stored in water at 37°C for 24 hours.

Microtensile Bond Strength Test

The specimens were cut perpendicularly with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) to obtain five to 10 resin-dentin beams (0.8 mm × 0.8 mm cross-sectional dimensions on average) from each tooth for microtensile testing (μTBS) (Figure 1). The final width and thickness of the bonded area were measured to the nearest 0.01 mm with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). Specimens were attached to a Geraldelli jig[20] with cyanoacrylate adhesive and stressed under tension (Kratos Dinamometros, Co-

Table 1: *Adhesive Systems, Batch Number, Composition, and Application Mode*

Adhesive Systems	Composition	Mode of Application
Clearfil SE Bond (Kuraray, Okayama, Japan); Lot No. 00795A	Primer: MDP, HEMA, dimethacrylate monomer, water, catalyst Bond: MDP, HEMA, dimethacrylate monomer, microfiller, catalyst	1. Apply primer and leave for 20 s. 2. Dry thoroughly with mild air flow. 3. Apply bond. 4. Gentle air flow. 5. Light polymerize for 10 s at 400 mW/cm^2.
Adper SE Plus (3M ESPE, St Paul, MN, USA); Lot No. 8BG/8BF	Bottle A: water, HEMA, surfactant, pink colorant Bottle B: UDMA, TEGDMA, TMPTMA, HEMA phosphates, MHP, bonded zirconia nanofiller, initiator system based on camphorquinone	1. Apply one coat of bottle A. This goes on pink, acting as a guide to achieving full coverage when applying the adhesive. 2. Apply bottle B and the pink color disappears, indicating where the adhesive is placed. 3. Agitate for 20 s. 4. Air-dry for 10 s. 5. Light polymerize for 10 s at 400 mW/cm^2.

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; MDP, methacryloyloxydecyl dihydrogenphosphate; MHP, methacrylated phosphates; TEGDMA, triethylene glycol dimethacrylate; TMPTMA, hydrophobic trimethacrylate; UDMA, urethane dimethacrylate or 1,6-di(methacryloyloxyethylcarbamoyl)-3,30,5-trimethylhexaan.

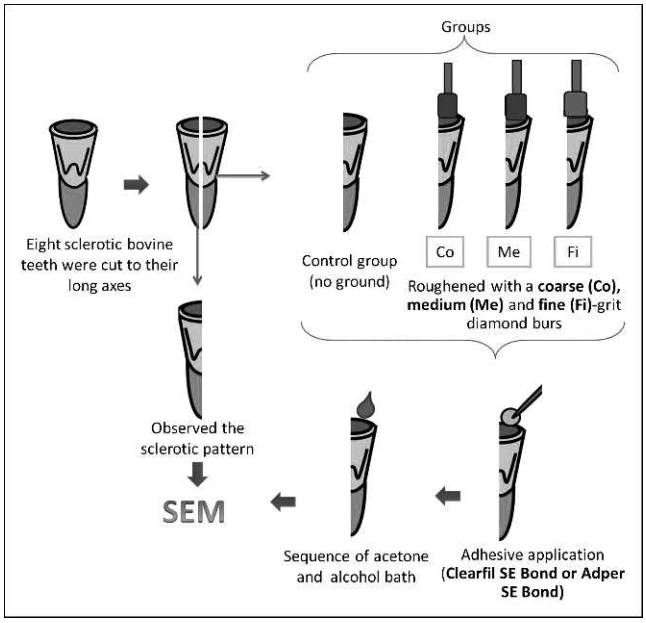

Figure 2. *Flowchart of the etching pattern examined by scanning electron microscopy (SEM).*

tia, São Paulo, Brazil) at 0.5 mm/min until failure. Bond strengths were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure mode of the specimens was classified as adhesive/mixed if failure occurred at the resin-dentin bond interface with or without cohesive failure of the neighboring substrates and as cohesive if the failure occurred at the substrate (resin or dentin). The classification was done under a stereomicroscope at 100× magnification (Olympus SZ40, Tokyo, Japan).

Statistical Analysis

All bond strength values obtained from the same tooth were averaged, and the tooth was considered the statistical unit of the present investigation.[21,22] The data were then submitted to two-way analysis of variance (ANOVA) (adhesive vs surface treatment) and post hoc Tukey tests. The level of significance was preset at α=0.05.

Etching Pattern Examined by Scanning Electron Microscopy (SEM)

A total of 16 bovine teeth were used for this part of the experiment. The teeth were cut perpendicular to their long axes using a slow-speed diamond saw (Isomet) in order to obtain two dentin halves. They were then randomly divided into two lots of halves: one half was used to evaluate the degree of dentin obliteration (control group), while the other half was treated according to one of the four following groups (n=2): 1) adhesive only application (without bur) and slightly roughened with a 2) coarse-grit, 3) medium-grit, or 3) fine-grit diamond bur, followed in each case by adhesive application. The adhesive application was performed as described in the "Experimental Design and Restorative Procedure" section. However, the adhesives were not light-cured after application (Table 1). Each surface was rinsed off with a sequence of acetone bath (five minutes), deionized water (five minutes), 96% alcohol bath (five minutes), and deionized water (five minutes) in order to remove all resin monomers from the surface (Figure 2).[23,24]

Specimens were then fixed in 2.5% glutaraldehyde in 0.1 M phosphate-buffered solution for 24 hours at room temperature before being dehydrated in ascending grades of ethanol and submitted to chemical drying in hexamethyldisilazane (SSX-550, Shimadzu, Tokyo, Japan). Finally, the samples were sputter-coated with gold (Sputtering SCD050, Bal-Tec, Balzers, Liechtenstein) and examined by SEM (SSX-550, Shimadzu) at 12 kV operated in secondary electron mode.

For the SEM analysis, three pictures were taken of each half. All of the electron micrographs were taken at the same working distance using the same magnification.[19] The measurement was performed as follows: the total area of each image was recorded using the UTHSCSA ImageTool 3.0 software (Department of Dental Diagnostic Science at The University of Texas Health Science Center, San Antonio, TX, USA) by a blinded researcher. Then, in the same image, the open tubule area was delineated by a software tool, summed, and the relative ratio between the open tubule area vs total area was calculated to give the relative percentage of open tubule area of each specimen. The three readings were averaged for statistical purposes. The relative percentage of open tubule area was evaluated by two-way ANOVA (adhesive vs surface treatment) and post hoc Tukey tests. The level of significance was preset at α=0.05.

RESULTS

Microtensile Bond Strength Test

Five to 10 resin-dentin specimens were obtained from each tooth. The failure modes of all experimental groups are shown in Table 2. The majority of the specimens (86.6%) presented adhesive/mixed failures. Dentin and resin failures were observed in 1.5% and 5.9% of the specimens, respectively. A small number of premature failures (6.0%) were observed.

Table 2: *Number of Specimens and Percentage (%) of Failure Modes for All Experimental Groups*

Adhesive	Failure Type	Control	Diamond Bur Granulation		
			Coarse	Medium	Fine
Clearfil SE Bond	A/M	17 (10.8)	37 (23.4)	40 (25.3)	39 (24.6)
	D	3 (2.0)	0 (0)	0 (0)	0 (0)
	R	7 (4.4)	3 (2.0)	0 (0)	0 (0)
	PF	1 (0.6)	5 (3.0)	4 (2.6)	2 (1.3)
Adper SE Bond	A/M	25 (22.5)	24 (21.6)	23 (20.7)	27 (24.4)
	D	0 (0)	0 (0)	1 (0.9)	0 (0)
	R	1 (0.9)	2 (1.8)	2 (1.8)	1 (0.9)
	PF	0 (0)	3 (2.7)	1 (0.9)	1 (0.9)

Abbreviations: A/M, adhesive or mixed failure; D, dentin cohesive failure; PF, premature failures; R, resin cohesive failure.

With regard to resin-dentin bond strength, only the main factor surface treatment was statistically significant ($p=0.022$). For both adhesives, the highest mean bond strength values were observed for the control group. However, this group was similar to the groups treated with coarse- and fine-grit diamond burs ($p>0.05$). The lowest resin-dentin bond strength was found when the medium-grit diamond bur was used ($p<0.05$) (Table 3).

Etching Pattern

Table 4 shows the relative percentage of open tubule area for all groups. Statistical analysis revealed that the cross-product interaction adhesive vs surface treatment was not statistically significant ($p=0.43$); however, both main factors were statistically significant ($p=0.006$ and $p=0.0001$, respectively, for adhesive and surface treatment).

For both adhesives, the lowest mean percentage of open tubules was observed for the control group before treatment in comparison with all groups ($p<0.05$). However, the use of primer significantly increased the percentage of open tubules for all groups ($p<0.05$). The use of a diamond bur showed an intermediate value of open tubules, regardless of the diamond bur coarseness ($p>0.05$). As regards adhesives, Clearfil SE Bond showed higher mean percentage values of open tubules in comparison to Adper SE Bond ($p<0.05$).

Table 3: *Mean Values and Standard Deviation of Microtensile Bond Strength (MPa) and Statistical Analysis[a] of All Experimental Groups*

Adhesive	Control A	Diamond Bur Granulation		
		Coarse A	Medium B	Fine A
Clearfil SE Bond	32.5 ± 10.7	26.3 ± 3.4	25.6 ± 3.6	24.1 ± 5.3
Adper SE Bond	30.1 ± 7.5	23.1 ± 4.7	16.0 ± 5.2	27.7 ± 5.6

[a] *Groups identified with the same capital letter are not statistically different (Tukey test, $p>0.05$).*

Table 4: *Mean Values and Standard Deviations of the Relative Percentage of Open Tubule Area (%) and Statistical Analysis[a] for All Experimental Groups*

Adhesive	Before Treatment	Control	Diamond Bur Granulation		
			Coarse	Medium	Fine
Clearfil SE Bond (a)	5.5 ± 2.6 A	22.8 ± 4.6 C	13.7 ± 4.3 B	10.4 ± 1.8 B	13.1 ± 5.1 B
Adper SE Bond (b)	4.5 ± 2.4 A	21.1 ± 4.6 C	9.3 ± 4.0 B	9.4 ± 2.2 B	8.7 ± 3.2 B

[a] *Groups with the same capital or lowercase parenthetical letters are not statistically different (Tukey test, $p>0.05$).*

Figure 3 shows SEM of the dentin surfaces of all the experimental groups. Very superficial open dentinal tubules were observed in the adhesive-only application (without bur) control groups (Figure 3C,D) in comparison to the other experimental conditions, in which diamond burs were used (Figure 3E-J). That is, the primers of the adhesive system used were unable to condition the dentin surface after it was roughened with the bur (Figure 3E-J).

DISCUSSION

The difficulty of obtaining human teeth with sclerotic features represents the reason why we opted to use bovine teeth in the present study. Previous studies revealed that both human and bovine dentin substrates have a similar morphology,[19,25-27] and superficial similarity could point at the possibility of using bovine sclerotic dentin as a replacement for human sclerotic dentin in bond strength tests, which allows researchers to use teeth from bovine animals to evaluate the behavior of adhesives systems.[28,29]

The results of the present investigation showed that the use of diamond burs of different grit sizes to roughen the sclerotic dentin did not improve the bond strength between the sclerotic dentin and self-etch adhesives. The findings of previous literature indicated that diamond bur roughening would produce a more irregular surface and a greater area of intertubular dentin available for adhesion.[3,10] Furthermore, Eliguzeloglu and others[13] showed that tag formation produced by self-etch adhesives was much more pronounced when the sclerotic dentin

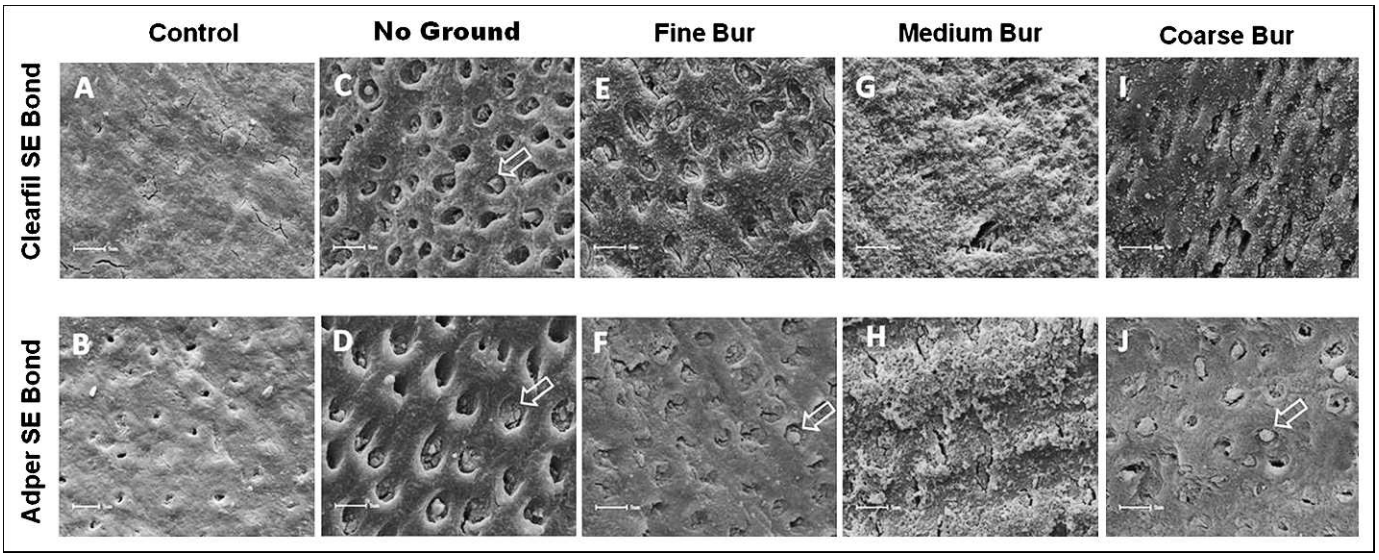

Figure 3. *Scanning electron micrographs of the sclerotic dentin surfaces in a control group before treatment (A and B) and after adhesive application with and without bur (Clearfil SE Bond [C, E, G, and I] and Adper SE Bond [D, F, H, and I]). Observe that in sclerotic dentin, complete obliteration of dentin tubules was shown (A and B). However, a large number of dentin tubules were exposed after application of the adhesive systems (C, Clearfil SE Bond; and D, Adper SE Bond). Note that in these groups, sclerotic dentin with "sclerotic" casts extending from open dentin tubules was shown (arrows). The surface of the sclerotic dentin after being roughened with different diamond burs (E, F: fine; G, H: medium; and I, J: coarse) was shown to be partially covered.*

had been previously roughened with a diamond bur prior to adhesive application.

Although the results of the present investigation are not in agreement with those of previous studies, we cannot rule out the fact that the authors of those other studies have only evaluated the bonding performance micromorphologically. It has been demonstrated that the hybrid layer thickness[3,10] or the amount of tag formation[13] are not correlated with improved resin-dentin bond strength.[2]

In fact, mechanical preparation may lower the thickness of the hypermineralized surface layer and remove some of the residual sclerotic casts that superficially obliterate the dentin tubules, but a tenacious smear layer is invariably formed along the lesion surface, consisting of hypermineralized remnants that are likewise resistant to acid dissolution,[4] and this can be observed in Figure 1.

The features of this smear layer, such as thickness and coarseness, depend on the bur used for cavity preparation.[30,31] This has led to investigators recommending the use of extra-fine diamond burs[30] or tungsten carbide burs[31] for cavity preparation of sound dentin, as they produce thinner smear layers. While this may yield improved bond strength of self-etch systems to sound dentin, this does not seem to be applicable to sclerotic dentin, according to the results of the present study. It was observed that the bond strength after bur roughness did not improve, and in some cases, a decrease in bond strength was found. There are no clear explanations for the lesser results found for the medium diamond bur, and future studies need to be conducted to test this hypothesis.

The formation of a smear layer that consists of acid-resistant hypermineralized dentin chips and whitlockite crystals derived from the sclerotic casts creates additional diffusion barriers to self-etch adhesives.[3] The retention of diffusion barriers, either in the form of an intact hypermineralized layer or an acid-resistant smear layer, resulted in some decline in the success of bonding to sclerotic dentin.[32]

Although this issue is somewhat controversial in the literature, there are studies[31,33-36] indicating that thick smear layers may hinder the infiltration of resin monomers into the underlying substrate and reduce the resin-dentin bond strength values produced by self-etch systems. Thus, the findings of this study indicate that when self-etch adhesives are to be applied to sclerotic dentin, clinicians should avoid cavity preparation, particularly with the use of a medium-grit diamond bur. This may be feasible when restoring noncarious cervical lesions. If no cavity preparation is performed, the sclerotic dentin surface is devoid of smear layer and the hypermineralized layer is left intact.[4] In this case, although it does not increase the bond strength values, there are a larger number of tubules available for adhesion.

However, this may not be the case with sclerotic dentin beneath caries lesions, where cavity preparation, in most of the cases, is mandatory. Other clinical approaches should be investigated in order to improve the bond strength of self-etch systems and instrumented sclerotic dentin. For instance, when observing the results of the etch-and-rinse adhesive, roughening/removal of the sclerotic superficial dentin was of help in obtaining more uniform etching and resin infiltration into intertubular sclerotic dentin,[17] and this procedure is responsible for improving the bond strength to sclerotic dentin.[10] Thus, the adjunctive use of an acid solution on ground sclerotic dentin before applying a self-etch adhesive will probably improve the bond strength, as has been suggested by several authors.[1,3,4] Future studies need to be conducted to test this hypothesis.

With regard to the adhesive system tested in the present study, the smear layer thickness produced in sound dentin had no influence on the microtensile bond strength values for the mild two-step self-etch adhesive, such as Clearfil SE Bond.[33-36] Adper SE Plus (also known as Adper Scotchbond SE in Europe) is a new material, and there is little information available on it; however, recent studies[37,38] have indicated that the bond strength values to sound dentin are similar between them, a finding that is in agreement with the present results in sclerotic dentin.

CONCLUSIONS

The bonding performance of self-etch systems in sclerotic dentin cannot be improved by roughening the dentin with diamond burs. Depending on the grit size of the diamond bur used, a decline in the bond strength of self-etch systems may occur when they are applied to sclerotic dentin.

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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Bond Durability of Adhesives Containing Modified-monomer With/Without-fluoride After Aging in Artificial Saliva and Under Intrapulpal Pressure Simulation

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Clinical Relevance

Adhesives with hydrolytically stable modified monomers could be a promising approach to enhance bonding durability.

SUMMARY

Objective: **To evaluate the dentin bond strength durability of adhesives containing modified-monomer with/without-fluoride after storage in artificial saliva and under intrapulpal pressure simulation (IPPS).**

Materials and Methods: **The occlusal enamel of 48 freshly extracted teeth was trimmed to expose midcoronal dentin. Roots were sectioned to expose the pulp chamber and to connect the specimens to the pulpal-pressure assembly. Specimens were assigned into four groups (n=12) according to adhesive system utilized: a two-step etch-and-rinse adhesive system (SB, Adper Single Bond 2, 3M ESPE), a two-step self-etch adhesive system (CSE, Clearfil SE Bond, Kuraray Medical Inc), and two single-step self-etch adhesives with the same modified monomer (bis-acrylamide)—one with fluoride (AOF, AdheSE One F, Ivoclar-Vivadent) and the other without (AO, AdheSE One,**

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Ivoclar-Vivadent). Bonding was carried out while the specimens were subjected to 15-mm Hg IPPS. Resin composite (Valux Plus, 3M ESPE) buildups were made. After curing, specimens were aged in artificial saliva and under 20-mm Hg IPPS at 37°C in a specially constructed incubator either for 24 hours or six months prior to testing. Bonded specimens (n=6/group) were sectioned into sticks (n=24/group) with a cross section of 0.9 ± 0.01 mm^2 and subjected to microtensile bond strength (μTBS) testing using a universal testing machine. Data were statistically analyzed using two-way analysis of variance (ANOVA) with repeated measures, one-way ANOVA tests, and a *t*-test ($p<0.05$). Failure modes were determined using a scanning electron microscope.

Results: **The μTBS values of SB and CSE fell significantly after six-month storage in artificial saliva and under IPPS, yet these values remained significantly higher than those for the other two adhesives with modified monomers. There was no significant difference in the bond strength values between fluoride-containing and fluoride-free self-etch adhesive systems (AOF and AO) after 24 hours or six months. Modes of failure were mainly adhesive and mixed.**

Conclusions: **Based on the results of this study, 1) Fluoride addition did not affect dentin bond durability; and 2) despite the fact that the single-step adhesive system with modified monomer showed stability, bond strengths associated with these systems remained lower than those of multistep adhesive systems.**

INTRODUCTION

Patient demand for esthetic restorations has generated an interest in the advancement of bonded restorations.[1] Several adhesive systems have been used for this purpose, ranging from etch-and-rinse adhesives to the more simplified self-etch adhesives that simultaneously condition and prime the dentin. This approach eliminates the rinsing phase, which not only reduces the clinical application time but also significantly decreases the technique sensitivity or the risk of making errors during application.[2] Current developments have focused on improving several aspects in the formulation of these adhesive systems to enhance their durability.

In order to control secondary caries, which is one of the common factors that limits the bond durability,[3] modifications in the adhesive systems' formulation, such that they were characterized by antibacterial activity, were considered beneficial. From this point of view, versions of adhesive systems containing fluoride in composition have been introduced. Fluoride has been added to the adhesive system as a therapeutic agent that supports the inhibition of secondary caries,[4] dentin demineralization, and inhibition of endogenous enzymes that attack the components of the hybrid layer,[4] thus allowing for the maintenance of bond durability.[5]

It has also been claimed[6] that (di)methacrylates are hydrolytically unstable in the acidic aqueous solutions of simplified self-etch adhesives, which may result in lower bond strengths and compromised bond durability. Therefore, monofunctional and cross-linking modified monomers have been developed, such as acrylamides, that offer increased hydrolytic stability[7] and that are less viscous and soluble than UDMA and bisphenol A glycidyl methacrylate (Bis-GMA).[8] Adjunctive use of the hydrolytically stable monomer bis-acrylamide and fluoride in an adhesive system could be a beneficial approach against various bond challenges in the oral environment.

Clinical evaluation of bonding durability under complex oral environments is highly recommended; however, *in vitro* testing is required to elucidate the specific factors that cause bond deterioration over time. In order to diminish the distance between *in vivo* and *in vitro* conditions, challenging the adhesive interface under a simulated oral environment at different time periods should be conducted.[9] The absence of an outward fluid flow through dentinal tubules has represented the most critical difference between clinical and laboratory conditions.[10] As a consequence, it is necessary to employ pulpal pressure simulation when adhesive systems are tested *in vitro*.

Thus, the objective of this study was to evaluate the dentin bond durability of fluoride-free and fluoride-containing adhesive systems that contain a modified monomer formulation in their chemistry, compared to a two-step etch-and-rinse adhesive system and a two-step self-etch adhesive system after storage in artificial saliva at 37°C and under intrapulpal pressure simulation. The null hypothesis was that there was no significant difference among the adhesive systems after six months of storage in artificial saliva with simulated intrapulpal pressure simulation.

MATERIALS AND METHODS

Specimen Preparation

Sound human third-molar teeth, extracted from patients with an age group of 18–20 years, were stored in phosphate buffer solution containing 0.2% sodium azide at 4°C. The teeth were collected from the patients after the protocol was approved by the Faculty of Dentistry's Ethics Committee at Cairo University, Egypt. All teeth were used within one month after extraction. Each tooth was trimmed perpendicular to its long axis, exposing the dentin, using a slow-speed diamond saw sectioning machine (Buehler Isomet Low Speed Saw, Lake Bluff, IL, USA) under water coolant. A second cut was made parallel to the occlusal surface, 2 mm below the cemento-enamel junction, thereby exposing the pulp chamber. Remnants of pulp tissue in the pulp chamber were removed using an excavator (Carl Martin GmbH, Solingen, Germany) without touching the walls of the pulp chamber.[11] Dentin surfaces were then wet-polished with 600-grit silica carbide paper to create a standard surface roughness and smear layer. The specimens (n=48) were connected to the intrapulpal pressure assembly during bonding and storage following the same procedures described by Mobarak.[12]

Restorative Procedures

Prepared specimens were divided into four groups (n=12) according to the adhesive systems utilized: Adper Single Bond 2 (SB; two-step etch-and-rinse adhesive system, 3M ESPE Dental Products, St Paul, MN, USA), Clearfil SE Bond (CSE; two-step self-etch adhesive system, Kuraray, Tokyo, Japan), AdheSE One (AO; single-step non–fluoride containing self-etch adhesive system, Ivoclar Vivadent, Schaan, Liechtenstein), and AdheSE One F (AOF; single-step fluoride-containing self-etch adhesive system, Ivoclar Vivadent). Material specifications, manufacturers, compositions (batch numbers), and application procedures are listed in Table 1. Resin composite (Valux Plus, 3M ESPE) of shade A1 was applied in two increments of 2 mm each. Each increment was polymerized for 40 seconds using Bluephase C5 (Ivoclar Vivadent) with an intensity $\geq$500 mW/cm^2. Light intensity was checked using a LED radiometer (Kerr Dental Specialties, West Collins Orange, CA, USA). Specimens were then immersed in artificial saliva[13] for 24 hours or six months at 37°C in a specially constructed large incubator to accommodate the intrapulpal pressure assembly.

Microtensile Bond Strength (μTBS) Testing

After storage of the bonded teeth, each tooth was longitudinally sectioned in both mesio-to-distal and buccal-to-lingual directions across the bonded interface to obtain multiple sticks of approximately 0.9 $\pm$ 0.01 mm^2 for the μTBS test. From each tooth, the central sticks were collected. A digital caliber was used to check the cross-sectional area and length of the sticks. Sticks of similar length and remaining dentin thickness were tested, which resulted in a total of 24 sticks for each subgroup. Each stick was fixed to the attachment with a cyanoacrylate adhesive (Rocket Heavy, Corona, CA, USA) and stressed in tension using a universal testing machine (Lloyd Instruments Ltd, Ametek Company, West Sussex, UK) at a cross-head speed of 0.5 mm/min until failure. The tensile force at failure was recorded and converted to tensile stress in MPa units using computer software (Nexygen-MT, Lloyd Instruments). Sticks that failed before testing were counted as 0 Mpa,[14,15] while those with premature cohesive failure were discarded and were not included in the calculations.[16] Two-way analysis of variance (ANOVA) with repeated measure was used to compare the effects of adhesive system, storage time, and their interaction. One-way ANOVA was used to test the effect of differences in adhesive systems on the bond strength values at each storage time. This was followed by the Bonferroni post hoc test for pairwise comparison. A *t*-test was used to compare between 24-hour and six-month μTBS mean values for each adhesive system. The significance level was set at $p \leq 0.05$. Data were analyzed using the SPSS Program for Windows (Release 15 for MS Windows,SPSS Inc, Chicago, IL, USA).

Fractured sides of all specimens were inspected under a scanning electron microscope (SEM) (515; Philips, Einhoven, The Netherlands) at different magnifications. Failure modes were evaluated at 100× and were classified into six types, as follows: type 1: Adhesive failure at the dentin side; type 2: Cohesive failure in the adhesive layer; type 3: Mixed failure (adhesive failure at the dentin side and cohesive failure in the adhesive layer); type 4: Mixed all (adhesive at the dentin side, cohesive failure in the adhesive and cohesive in resin composite); type 5: Cohesive failure in resin composite; and type 6: Cohesive failure in dentin. The frequency of each mode of failure was calculated for each subgroup.[17]

RESULTS

Two-way ANOVA with repeated measures revealed that there were statistically significant effects for

Table 1: *Material Specifications, Manufacturers, Compositions, Application Procedures, and Batch Numbers*

Material	Specification	Manufacturer	Composition (Batch Number)	Application Procedures
Adper SingleBond 2	Two-step etch-and-rinse adhesive system	3M ESPE Dental Products, St Paul, MN, USA	Etchant: 35% phosphoric acid, colloidal silica (7523)	Etching: apply for 15 s, water rinsing for 10 s, then blot excess water with minisponge (visibly moist surface)
			Adhesive: Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymers of polyacrylic and polyitaconic acids, silica nanofillers (51202)	Adhesive: apply with gentle agitation for 15 s, gently air-thin for 5 s, and light-cure for 10 s
Clearfil SE Bond	Two-step self-etch adhesive system	Kuraray Medical Inc, Sakazu, Kurashiki, Okayama, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, DL-camphorquinone, *N,N*-diethanol-*p*-toluidine, and water (00999A)	Priming: Apply onto the visibly moist dentin surface, then leave undisturbed for 20 s and dry with oil-free mild air flow for 5 s
			Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, DL-camphorquinone, *N,N*-diethanol-*p*-toluidine, and silanated colloidal silica (01486A)	Bonding: Apply one coat of Clearfil SE bond and gently oil-free air-thin for 2 s, then light-cure for 10 s.
AdheSE One	Single-step non–fluoride containing self-etch adhesive system	Ivoclar Vivadent AG, Schaan, Liechtenstein	Derivatives of bis-acrylamide, water, bis-methacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxyl alkyl methacrylamide, silicon dioxide, catalysts, and stabilizer (K41349)	Apply onto the visibly moist prepared tooth and leave for 30 s. Disperse excess amount with a strong stream of air until there is no longer any movement of the material, then light-cure for 10 s.
AdheSE One F	Single-step fluoride -ntaining self-etch adhesive system	Ivoclar Vivadent AG, Schaan, Liechtenstein	Derivatives of bis-acrylamide, water, alcohol, bis-metacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxyl alkyl methacrylamide, alkyl sulfonic acid acrylamide, silicon dioxide, initiators, stabilizers, and potassium fluoride (251379)	Follow same application procedures as AdheSE One adhesive system

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

adhesive systems ($p<0.001$) and storage time ($p<0.00001$) but not for their interaction ($p=0.33$). The means and standard deviations (SDs) of the μTBS values of all tested groups are presented in Table 2. One-way ANOVA indicated that there was a significant difference among the adhesive systems when tested after 24 hours ($p<0.0001$) and after six-month storage ($p=0.003$). As shown in Table 2, a Bonferroni post hoc test revealed that AO and AOF were significantly lower than SB and CSE at 24 hours as well as at six months. With regard to the effect of storage time, the *t*-test revealed a significant decrease in bond strength values for SB ($p<0.00001$) and CSE ($p=0.0001$) adhesive systems but not for AO ($p=0.89$) and AOF ($p=0.75$) after six-months of storage. In all groups, the major mode of failure was adhesive at the dentin side (type 1), followed by mixed failure (type 3). Cohesive failures in resin composite (type 5) and in dentin (type 6) were very few (Figure 1). Representative SEM micrographs for some modes of failure are presented in Figures 2 and 3.

DISCUSSION

The μTBS results of both tested adhesive systems, two-step etch-and-rinse (SB) and self-etch (CSE),

Table 2: *Microtensile Bond Strength (μTBS) Values [Mean (standard deviation, SD)] in MPa of Tested Adhesive Systems*[a]

Storage Periods	Adhesive Systems μTBS Values [Mean (SD)]				
	Adper Single Bond 2	Clearfil SE Bond	AdheSE One F	AdheSE One F	*P*-Value
24 h	34.87 (4.9) aA [Ptf/tnt=0/24]	38.11 (5.1) aA [Ptf/tnt=0/24]	16.22 (4.9) bA [Ptf/tnt=5/24]	15.78 (3.9) bA [Ptf/tnt=5/24]	<0.0001
6 mo	20.69 (3.1) aB [Ptf/tnt=0/24]	22.67 (1.8) aB [Ptf/tnt=0/24]	15.60 (1.7) aA [Ptf/tnt=6/24]	14.84 (1.9) aA [Ptf/tnt=6/24]	0.003
p-value	<0.0001	<0.0001	0.89	0.75	

[a] *[ptf/tnt=pretest failure/total number of tested sticks]. Within rows, means with different lowercase letters are statistically significantly different (p≤0.05, Bonferroni test); Within columns, means with different small capital letters are statistically significantly different (p≤0.05,* t-test).

revealed significantly higher values compared to the tested single-step self-etch adhesive systems (AO and AOF) after 24-hour and six-month storage times in artificial saliva at 37°C under simulated intrapulpal pressure. This finding correlates with that of Belli and others,[16] who found that SB and CSE were significantly higher than AO under simulated intrapulpal pressure at the 24-hour storage point.

Several reasons could be behind the recorded low bond strength of these single-step self-etch adhesives. One of them is that the combination of acidic hydrophilic and hydrophobic monomers into a single solution may compromise the function of each component.[18,19] It should be noted that AO and AOF have relatively high acidity (pH=1.5).[20] Another reason is that for such types of adhesive systems, a relatively high concentration of solvent is required to keep the monomers blended into the solution. Hence, air-drying of the solvent may not be able to accomplish significant solvent evaporation,[21] which leads to the retention of a greater amount of water in the adhesive layer, even after light-curing. Increased water concentration may indeed dilute the adhesive monomers and lower their inward rate of diffusion as well as limit the degree of polymerization.[22] In the present study, intrapulpal pressure was simulated during bonding and storage, which could intensify the negative effect of residual water retention.

Additionally, Margvelashvili and others[20] reported that the AO adhesive system is an acetone-free

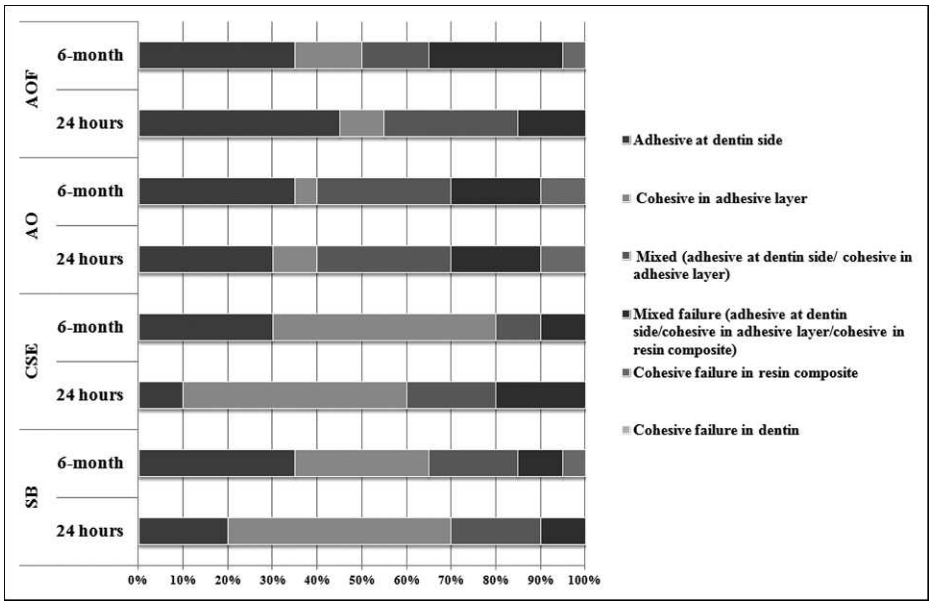

Figure 1. *Modes of failure of tested adhesives.*

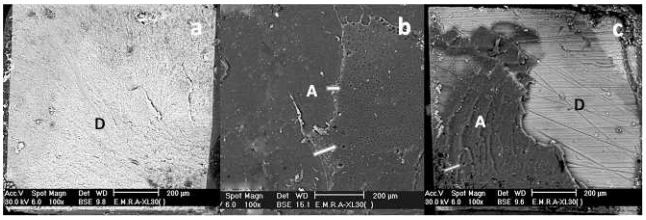

Figure 2. *SEM photomicrography of representative fractured AO specimens after six-month storage showing type 1, adhesive at the dentin side (a); type 2, cohesive failure in the adhesive layer (b); and type 3, mixed failure (adhesive at the dentin side [AD] and cohesive at the adhesive layer [CA]) (c). White arrows denote voids in the adhesive layer.*

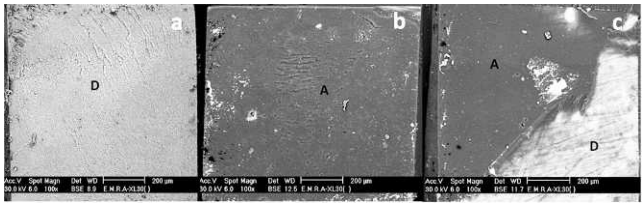

Figure 3. *SEM photomicrography of representative fractured AOF specimens after six-month storage showing type 1, adhesive at the dentin side (a); type 2, cohesive failure in the adhesive layer (b); and type 3, mixed failure (adhesive at the dentin side [AD] and cohesive at the adhesive layer [CA]) (c).*

adhesive system so that, based on the manufacture's claim, the probability of solvent/monomer phase separation was eliminated. In reality, the phase separation that occurred between the hydrophilic and the hydrophobic components of this adhesive cannot be discounted. A varying infiltration gradient can be established as a consequence of phase separation within the adhesive and/or as a result of the difference in molecular weight or affinity to dentin.[19,23] SEM photomicrographs of the AO fractured sticks support the above-mentioned explanations wherein type 2 failure (cohesive failure in the adhesive layer) blisters (droplets) were observed. These droplets were usually detected with single-step self-etch adhesives and were attributed to water sorption from the moist dentin substrate through an osmotic process caused by a gradient imbalance at the interfaces.

The μTBS results of the tested adhesive systems after six-months of storage in artificial saliva and under intrapulpal pressure support the rejection of the null hypothesis. This was clearly shown with SB and CSE, for which μTBS values were significantly decreased after six months of storage. Campos and others[24] agreed with these findings; however, they used thermo-mechanical stressing in addition to the intrapulpal pressure simulation. The decrease in bonding effectiveness of such adhesives may be caused by the degradation of interface components *via* hydrolysis (mainly resin and/or collagen).[2] In etch-and-rinse adhesive systems, there is a discrepancy between the depth of demineralization and resin infiltration, which leads to a zone of hydroxyapatite-depleted collagen fibrils that are left exposed and unsupported.[24,25] The mineralized dentin matrix contains many endogenous enzymes (for example, alkaline phosphatase and metalloproteases) that may be released and activated during etching or more slowly during water storage. These enzymes could attack the denuded collagen,[26] the ester bonds of resins,[27] or both. Thus, the demineralized dentin at the bottom of the hybrid layer would become a weak link in the bonding interface over time.[28] Furthermore, Pashley and others[29] reported that bonding of contemporary etch-and-rinse adhesive systems to water-saturated dentin leaves a very thin layer of water or hydrogel between the infiltrated adhesive and the collagen fibrils of the matrix. This layer seems to provide a fluid-filled continuum from resin tags into the base of the hybrid layer through lateral branches of dentinal tubules throughout the full thickness of the hybrid layer. In addition, permitting the diffusion of water molecules from dentin across the adhesive layer reduces the frictional forces between the polymer chains, causing what is known as plasticization.[27] The aforementioned explanation is supported by the detected shift in the mode of failure patterns of the Adper Single Bond 2 toward a more complex interfacial fracture after six months of storage under intrapulpal pressure.

The decrease in bond strength of CSE after six-months of storage under simulated intrapulpal pressure simulation supports the findings of Abdalla and others.[30] This reduction in bond strength may be attributed to slow water sorption of the adhesive, which decreases its mechanical properties and its bond strength. Hosaka and others[31] suggested that if the acidity of the self-etch adhesives is unable to etch through both smear layer and smear plugs then hybridized smear plug material could prevent water from seeping from the dentinal tubules into the adhesive during bonding. However, the hybridized smear plugs may be porous enough to permit sufficient seepage of water to elute unreacted monomers or oligomers from the bonded interface over the next 90 days of pulpal perfusion, replacing their volume with water that would weaken the adhesion.[32] In addition to 2-hydroxyethyl methacrylate (HEMA), CSE primer contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer,

which is believed to increase its wetting to moist dentin on one side. Nevertheless, it is hydrophilic and, hence, more prone to hydrolysis.[33]

Interestingly, there was no significant difference in the bond strength values between fluoride-containing and fluoride-free self-etch adhesive systems (AOF and AO) after 24 hours or six months of storage. This finding opposes those of previous researchers,[5,34] who reported stability of fluoride-containing adhesive. This might be explained by the fact that in our study, the AOF we used contains potassium fluoride fillers rather than sodium fluoride fillers, in which case the higher molecular weight of potassium (58.0967 g/mol), compared to sodium (41.89 g/mol), could have played a role. In particular, those that reported bond stability used an adhesive system with sodium fluoride fillers that are characterized by their easy solubility.[35] Therefore, it might be expected that using an adhesive system containing fillers with higher molecular weight might decelerate the fluoride release and, consequently, might not exceptionally stabilize the bond strength over the tested period. It should also be noted that the release of fluoride from the adhesive is time dependent. Thus, further investigation is required to determine whether the possible positive effect of fluoride-containing adhesives on the bond durability can be elucidated over a longer duration than that tested in the present study. Interfacial compositional analysis is also required to detect the release of fluoride and its reaction with the hybrid layer. Moreover, there is still a possibility that the amount of fluoride present in the tested adhesive system was not adequate to reveal a positive effect. The quantity of fluoride that should be included in the adhesive system in order to obtain a significant effect on the bond durability is not yet known.

In the present study, AO and AOF adhesives maintained their stability from 24 hours to six months under intrapulpal pressure storage. The bond stability of these adhesives can be attributed to their altered chemical composition, as they contained acrylamido alkylsulfonic acid that would contribute to a chemical interaction with the tooth substance through the phosphoric ester group. In addition, they contained bifunctional acrylic amides, which are responsible for the formation of the polymer network. Such hydrolytic stability may possibly protect the bond from long-term water degradation.[36] Our AO results corroborate with those of Belli and others,[16] who tested this adhesive under simulated intrapulpal pressure for one year and reported its low but stable bond strength. Meanwhile, the results of this study raised some points of concern that require further investigation. One point is that despite the recorded stability of the adhesives with modified monomer (AO and AOF), they revealed significantly lower μTBS values than did the other multistep adhesives, even after six months. Thus, further investigation is required to determine whether the recorded low bond strength of adhesives with modified monomer (AO and AOF) would remain stable while that of the other adhesives (CSE and SB) will continue to degrade over time, compensating for the large gap that was recorded initially in favor of CSE and SB bond strength. Another negative aspect recorded for these adhesives in both the present study and that of Belli and others[16] was the large number of pre-test failures. The μTBS test procedure is difficult to perform, time consuming, and extremely technique-sensitive, especially in regard to stick preparation, which can result in pre-test failures. However, the presence of the pre-test failure with these two adhesives in particular may point out that these adhesives produce nonuniform hybridization with dentin. This calls for additional research to enhance the homogeneous bonding of these adhesive systems to dentin.

CONCLUSIONS

Based on the results of this study, the following conclusions were drawn:

1. Fluoride addition did not affect dentin bond durability.
2. Despite the fact that the single-step adhesive system with modified monomer showed stability, bond strength remained lower than that associated with the multistep adhesive systems.

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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Efficacy of Mouth Rinses and Toothpaste on Tooth Whitening

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Clinical Relevance

Several tooth whitening products are available to consumers on the market. This study questions whether the use of whitening mouth rinses and toothpaste can result in bleaching efficacy similar to that of the 10% carbamide peroxide at-home bleaching technique.

SUMMARY

Objectives: **People increasingly desire tooth whitening. Considering the wide range of whitening products on the market, this study evaluated the efficacy of whitening toothpastes and mouth rinses compared with the 10% carbamide peroxide (CP) whitening gel.**

Methods: **We obtained 120 cylindrical specimens from bovine teeth, which were darkened for 24 hours in a coffee solution. The color measurement was performed by a spectrophotometer using the CIE L*a*b* system, and specimens were divided into six groups according to the use of the following agents: group 1, conventional fluoridated toothpaste; group 2, Close Up White Now; group 3, Listerine Whitening; group 4, Colgate Plax Whitening; group 5, experimental mouth rinse with Plasdone; and group 6, 10% CP Whiteness Perfect. After the simulation of 12 weeks of treatment for groups 1 to 5 and 14 days of treatment for group 6, the specimens were subjected to a new color reading.**

Results: **Data were subjected to one-way analysis of variance (α=0.05), which showed significant differences among groups after 12 weeks for ΔE (p=0.001). Results of the Tukey test revealed that groups 3, 4, and 6 presented significantly higher color alteration than groups 1, 2, and 5.**

Conclusions: **The whitening toothpaste Close Up White Now and the experimental mouth rinse with Plasdone showed similar color alteration as conventional toothpaste after a 12-**

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week treatment simulation. These groups presented significantly lower color alteration compared with whitening mouth rinses Listerine and Colgate Plax Whitening, which showed similar results to those observed after 14 days of bleaching with 10% CP treatment.

INTRODUCTION

Today, patients are demanding more than a healthy mouth; they want a perfect smile.[1] Esthetics has become central; thus, treatments that focus on esthetics have been gaining more visibility and interest in all society.

The color of permanent teeth is mainly determined by the dentin and modified by the thickness and translucence of enamel. The deposition of a variety of pigments into or onto the tooth may change its color. These changes are usually classified as intrinsic or extrinsic, depending on the source of the stain. Therefore, attempts to improve the color of teeth should be directed to each type of stain.[2]

Tooth discoloration may cause social and cosmetic problems for patients. Thus, dentists and patients spend large amounts of money and time trying to improve the appearance of teeth.[3] Therefore, the demand for whitening treatments has grown considerably in recent years, as they are not invasive and are relatively simple to carry out. There are three fundamental approaches of tooth whitening: dentist-supervised home bleaching; in-office bleaching; and mass market products, also known as over-the-counter (OTC) products.[4,5]

The mechanism of action of mass market products are mainly two: bleaching intrinsic stains by using oxidizing agents that break down the pigments in the tooth structure, and removing and controlling extrinsic stains using abrasive agents. OTC products usually contain low levels of whitening agent (3%-6% hydrogen peroxide) that are self-applied to the teeth via gum shields, strips, paint-on products, toothpastes, or mouth rinse products. The removal and control of extrinsic stains are via so-called whitening toothpastes that contain abrasives and chemicals to maximize cleaning. Some products claim to possess optical brighteners, which are dyes that deposit on the teeth and result in an increase in the measurement and perception of tooth whiteness.[5–7]

Considering the wide range of mass market products and the scarce evidence of their efficacy, the aim of this study was to evaluate the effectiveness of four commercially available whitening products (one toothpaste and three mouth rinses) compared with the 10% carbamide peroxide whitening gel used for dentist-supervised home bleaching. The null hypothesis tested is that the whitening products tested have no effect in the color change of teeth.

MATERIALS AND METHODS

Specimens Preparation

The specimens were prepared according to the method described by Wiegand and others.[8,9] From 60 extracted nondamaged bovine incisors, we obtained up to two specimens of 3 mm in diameter and 2.2 mm in height from the buccal surface with a trephine drill.

The dentin and enamel thickness were standardized at 1 mm each by polishing with aluminum oxide abrasive papers (1200-grit FEPA-P, Struers, Ballerup, Denmark) in a polishing device (DP-10, Panambra Industrial e Técnica SA, São Paulo, Brazil). The enamel surface was polished with sequential abrasive papers (1200, 2400, and 4000-grit, Struers), applied for 20 seconds each.

The specimens were placed on a silicon support, leaving only the enamel exposed, and were immersed in a coffee solution prepared with 25 g of soluble coffee (Nescafé Tradição, Nestlé Brazil Ltda, Araras, SP, Brazil) in 100 mL water for 24 hours.[3] After this, they were rinsed in deionized water and ultrasonically cleaned for 2 minutes. The color measurement was then performed.

Color Measurement

Color reading of each specimen was performed at standardized ambient condition using a Spectrophotometer CM 2600d (Minolta, Osaka, Japan). The color and spectral distribution were measured according to the CIE L*a*b* system, using Spectra Magic NX software CM-S100w (Konica Minolta, Osaka, Japan). The D65 illuminant standard was set, with the reflectance mode and ultraviolet light included. The angle of observation was set to 2°, and the specular component was included. The specimens were slightly dried with absorbent paper and immediately placed into an individually prepared white rubber holder containing a hole the same size as the specimen.

A standard white background (Ceram, Staffordshire, UK) and an optical coupling using 400 polyethyleneglycol were used. The device was set to make three consecutive readings, automatically calculating the mean values of L*a*b*, as established by Commission Internationale de l'Éclair-

Table 1: *Products, Manufacturers, and Their Components*

Product	Manufacturer	Components
Close Up White Now	Unilever, Ipojuca, PE, Brazil	Sorbitol, aqua, silica PEG-32, sodium lauryl sulfate, aroma, cellulose gum, sodium fluoride, sodium saccharin, PVM/MA copolymer, trisodium phosphate, MICA, CI 74160, limonene
Colgate Fluoridated Toothpaste	Colgate-Palmolive, São Bernardo do Campo, SP, Brazil	Calcium carbonate, aqua, sorbitol, sodium lauryl sulfate, sodium monoflouophosphate, cellulose gum, aroma, tetrasodium pyrophosphate, sodium silicate, sodium saccharin, methylparaben, propylparaben
Listerine Whitening	KIK Custom Products, Etocicoke, Canada	Water, 8% alcohol, hydrogen peroxide 2%, sodium phosphate, poloxamer 407, sodium lauryl sulfate, sodium citrate, mint aroma, menthol, eucalyptol, sodium saccharin, sucralose
Plasdone 2%	International Specialty Products, Wayne, New Jersey, USA	Polyvinylpyrrolidone K29/32, water
Colgate Plax Whitening	Colgate-Palmolive, São Bernardo do Campo, SP, Brazil	Water, sorbitol, ethanol, hydrogen peroxide 1.5%, poloxamer 338, polysorbate 20, methyl salicylate, menthol, sodium saccharin, CI 42090
Whiteness Perfect 10%	FGM, Joinville, SC, Brazil	Carbamide peroxide, neutralized carbopol, potassium nitrate, sodium fluoride, humectant (glycol), deionized water

age.[10] The L* value of each specimen was used for stratified allocation of all samples among the experimental groups.

Group Divisions

Table 1 shows all products used in this study, including manufacturers and their components. Six groups of 20 specimens each were divided according to the proposed treatment:

- Group 1: Brushing with conventional fluoridated toothpaste (Colgate Fluoridated Toothpaste), the negative control.
- Group 2: Brushing with whitening toothpaste (Close Up White Now).
- Group 3: Immersion in whitening mouth rinse (Listerine Whitening - hydrogen peroxide 2%) for 1 minute, followed by brushing with conventional fluoridated toothpaste.
- Group 4: Immersion in whitening mouth rinse (Colgate Plax Whitening - hydrogen peroxide 1.5%) for 1 minute, followed by brushing with conventional fluoridated toothpaste and new immersion in Colgate Plax Whitening for 1 minute.
- Group 5: Immersion in experimental whitening mouth rinse prepared using 2 g polyvinylpyrrolidone (K29/32 Plasdone 2%) diluted in 100 mL of distilled water for 1 minute, followed by brushing with conventional fluoridated toothpaste.
- Group 6: Application of whitening gel (Whiteness Perfect - 10% carbamide peroxide) for 2 hours and immersion in artificial saliva for 22 hours. The procedure was performed once a day for 14 days and served as the positive control.

For brushing, stained specimens were brushed with soft electric toothbrushes. Eighty-four cycles of brushing were performed using 10 mL of a suspension containing 33% toothpaste[11] and artificial saliva. This was meant to simulate a six-week treatment period of two daily brushings of two minutes each.[12] Specimens were rinsed with water and color was measured. Then, 84 additional cycles were performed, corresponding to a 12-week treatment period, and the final color was measured.

The mouth rinses were used according to the manufacturer's recommendation. Artificial saliva was prepared according to the formulation of Gohring and others.[13] Specimens were stored in artificial saliva in the intermediate periods between testing procedures for all groups.

With L*a*b* values after the darkening of teeth (baseline values) and the values obtained after different periods of tested treatments, it was possible

Table 2: *Means and Standard Deviations Relative to Evaluated Parameters at Each Time*

Groups	ΔE		ΔL		Δb	
	6 weeks	12 weeks	6 weeks	12 weeks	6 weeks	12 weeks
1	3.92 ± 1.66[a]	3.47 ± 1.00[a]	–0.47 ± 2.70[abc]	–0.20 ± 1.36[a]	–2.59 ± 1.84[a]	–3.15 ± 0.96[a]
2	3.34 ± 1.09[a]	3.45 ± 0.99[a]	–0.79 ± 1.21[ac]	–0.27 ± 1.16[a]	–3.03 ± 1.04[a]	–3.22 ± 0.99[a]
3	4.49 ± v1.17[a]	6.12v ± 1.31[b]	1.12 ± 2.00[ab]	3.71 ± 2.18[bc]	–3.86 ±v1.09[ab]	–4.20 ± 1.70[ab]
4	4.36 ± 0.81[a]	6.30 ± 1.95[b]	1.15 ± 1.56[ab]	4.96 ± 2.70[c]	–3.82 ± 1.08[ab]	–3.05 ± 1.46[a]
5	4.28 ± v1.94[a]	3.93 ± 1.73[a]	–1.39 ± 2.48[c]	0.27 ± 2.53[a]	–3.38 ± 1.63[a]	–3.02 ± 1.65[a]
6	6.24 ± 2.73[b]	6.24 ± 2.73[b]	1.49 ± 3.22[b]	2.54 ± 2.43[b]	–5.07 ± 2.74[b]	–5.07 ± 2.74[b]

[a-c] *For a given time, values with different letter designations are significantly different ($p<0.05$).*

to calculate ΔE and then determine the efficacy after simulated treatments of 6 (ΔE1) and 12 weeks (ΔE2), according to the following formula:

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

On the other hand, Δb was calculated by subtracting the value of b* obtained after darkening and after different simulated periods of treatments, resulting in Δb1 for 6 weeks and Δb2 for 12 weeks. The same was done for ΔL and Δa.

For group 6, the ΔE, ΔL, Δa, and Δb values were calculated after 14 days of treatment. The data were statistically analyzed using one-way analysis of variance (ANOVA) to compare the color of darkened specimens with the color obtained after the use of different tested products for a simulated 6 and 12 weeks of treatment in groups 1 to 5, and 14 days in group 6. For identifying the differences, a Tukey test was applied ($\alpha = 0.05$).

RESULTS

The application of one-way ANOVA revealed significant differences among groups for ΔE, ΔL, and Δb for both 6 and 12 weeks (p=0.0001). For Δa, there were no significant differences for groups evaluated after the simulated 6 weeks (p=0.2434) and 12 weeks (p=0.1903).

Results of the Tukey test for ΔE1 and ΔE2 are shown in Table 2. After the simulated 6 weeks of treatment, groups 1 to 5 showed no significant differences among themselves and significantly lower color change than that obtained by group 6. After the simulated 12 weeks of treatment, groups 3 and 4 reached color changes similar to that of group 6.

The Tukey test for Δb1 revealed that specimens of group 6 presented significant differences compared with groups 1, 2 and 5 but did not show significant differences compared with groups 3 and 4. After the simulated 12 weeks of treatment, group 6 showed similar Δb values compared with group 3 and significantly higher values than other groups (Table 2).

After the simulated 6 weeks of treatment, group 6 presented significant differences of ΔL means compared with groups 2 and 5. After the simulated 12 weeks of treatment, groups 3, 4, and 6 showed better results than all other groups (Table 2).

DISCUSSION

Many substances have been used to produce staining *in vitro*, with coffee, red wine, and tea showing the best results.[14,15] In this study, a 24-hour coffee darkening period was chosen because maximum staining was obtained in a previous study with this time.[3]

We evaluated the effects of four whitening products in color of bovine teeth: three commercially available and one experimentally manipulated. Such products were compared with the 10% carbamide peroxide whitening gel (positive control) and to brushing with toothpaste without bleaching substances (negative control).

In the color space L*a*b*, L* indicates lightness, and a* and b* represent chromaticity coordinates: +a* indicates the red direction, –a* indicates the green direction, +b* indicates the yellow direction, and –b* indicates the blue direction. The increase in the a* and b* direction means that the point moves away from the center and increases color saturation.[16]

The toothpaste Close Up White Now has Blue Covarine Foam Technology[17] as the active agent, which has been shown to be deposited onto the tooth surface, altering the optical properties of the tooth by shifting the b* parameter from yellow to blue, which results in a visual perception of whitened tooth.[6] The efficacy of silica as an abrasive for gradual removal of extrinsic stains has been demonstrated previously.[18] Nevertheless, in the present study, the whitening blue covarine-based dentifrice presented no significantly different results for ΔE, ΔL, and Δb parameters compared with conventional fluoridated toothpaste.

Polyvinylpyrrolidone (PVP) (Plasdone K-29/32, ISP, Wayne, New Jersey, USA) is a water-soluble homopolymer, and it can be presented in various molecular weights and with several applications. PVP forms complexes with catechins, just as it does with many other compounds that cause discoloration, removing them from enamel. Although this polymer is thought to bind and remove stains in several oral care applications and to inhibit stain redeposition,[19] in this study, the mouth rinse manipulated with Plasdone K29/32 presented results that did not differ statistically from brushing with conventional fluoridated toothpaste. Because few studies have investigated this agent, it is suggested that further studies be performed with Peroxydone (ISP), which is the PVP associated with hydrogen peroxide.

The effectiveness of whitening mouthwashes are not discussed enough in literature. In a previous study, no significant bleaching effect was observed with the use of different peroxide-based whitening rinses on stained teeth after a 21-day treatment period.[20] On the other hand, Hasturk and others[21] showed that a 1.5% hydrogen peroxide mouth rinse used for 6 months was effective in reducing gingivitis and whitening teeth. In this study we obtained similar results with Colgate Plax Whitening and Listerine Whitening, as they showed color alteration similar to the 10% carbamide peroxide whitening gel.

Although Listerine Whitening and Plax Whitening have been effective as tooth whitening agents, care should be taken as the active agent in both mouth rinses is hydrogen peroxide. Because there is a concern regarding the possible tumor-promoting ability of this agent with the tobacco carcinogen DMBA (9,10-dimethyl-1,2-benzanthracene), patients should avoid alcohol and smoking during the treatment.[22] Nevertheless, hydrogen peroxide is present in low concentrations in both products (1.5% in Plax Whitening and 2% in the Listerine Whitening), which would not damage the mucosa.[21] Indeed, in a previous study, strong evidence for the safety of low-concentration, hydrogen peroxide–containing products was suggested, with no damage in oral hard and soft tissues and no significant risk of adverse long-term effects.[23]

The 10% carbamide peroxide was used as positive control because this substance has proved in previous studies to be safe and effective for dentist-supervised home bleaching. In addition, the bleaching effect caused by carbamide peroxide has been stable and long lasting.[24,25] Although 10% carbamide peroxide results in release of 3.5% hydrogen peroxide,[25] which is greater than that found in the mouth rinses tested, it is a safer alternative because it is applied in the form of gel in a tray, thus restricting the contact of hydrogen peroxide to the teeth and minimizing contact to adjacent gums. On the other hand, the mouth rinse is in contact with all the oral mucosa.

This *in vitro* study showed that simulated 12 weeks of treatment with mouthwashes containing hydrogen peroxide at low concentrations (Listerine Whitening and Plax Whitening) had results similar to treatment with 10% carbamide peroxide for 14 days. However, clinical studies are needed to confirm these results, as dilution of mouth rinses by the presence of saliva may alter their whitening effect. In addition, the longevity and stability of the whitening achieved with these products also need to be further investigated.

CONCLUSION

Within the limitations of this *in vitro* study, it can be concluded that

- The use of Listerine and Colgate Plax Whitening mouth rinses for 12 weeks presented similar color alteration compared with 14 days of 10% carbamide peroxide bleaching.
- The use of fluoridated conventional toothpaste, whitening toothpaste White Now, and experimental mouth rinse with Plasdone for 12 weeks showed

lower bleaching results for ΔE and ΔL parameters compared with other groups,.

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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Effect of Different Adhesion Strategies on Bond Strength of Resin Composite to Composite-dentin Complex

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Clinical Relevance

When composite materials are to be repaired next to dentin, preferably the substrate composite and repair composite should be of the same type. Conditioning the substrate composite with silica coating and silanization after etching the dentin add to the repair strength of the composite-dentin complex when compared to the success of silane application only.

ABSTRACT

Service life of discolored and abraded resin composite restorations could be prolonged by repair or relayering actions. Composite-composite adhesion can be achieved successfully using some surface conditioning methods, but the most effective adhesion protocol for relayering is not known when the composite restorations are surrounded with dentin. This study evaluated the effect of three adhesion strategies on the bond strength of resin composite to the composite-dentin complex. Intact maxillary central incisors (N=72, n=8 per subgroup) were collected and the coronal parts of the teeth were embedded in autopolymerized poly(methyl tfr54methacrylate) surrounded by a polyvinyl chloride cylinder. Cylindrical cavities (diameter: 2.6 mm; depth: 2 mm) were opened in the middle of the labial surfaces of the teeth using a standard diamond bur, and the specimens were randomly divided into three groups. Two types of resin composite, namely microhybrid (Quadrant Anterior Shine; AS) and nanohybrid (Grandio; G), were photo-polymerized incrementally in the cavities according to each manufacturer's

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recommendations. The composite-enamel surfaces were ground finished to 1200-grit silicone carbide paper until the dentin was exposed. The surfaces of the substrate composites and the surrounding dentin were conditioned according to one of the following adhesion protocols: protocol 1: acid-etching (dentin) + silica coating (composite) + silanization (composite) + primer (dentin) + bonding agent (dentin + composite); protocol 2: silica coating (composite) + acid-etching (dentin) + silanization (composite) + primer (dentin) + bonding agent (dentin + composite); and protocol 3: acid-etching (dentin) + primer (dentin) + silanization (composite) + bonding agent (dentin + composite). Applied primer and bonding agents were the corresponding materials of the composite manufacturer. Silica coating (CoJet sand, 30 μm) was achieved using a chairside air-abrasion device (distance: 10 mm; duration: four seconds in circular motion). After conditioning protocols, the repair resin was adhered to the substrate surfaces using transparent polyethylene molds (diameter: 3.6 mm) incrementally and photo-polymerized. The substrate-adherend combinations were as follows: AS-AS, G-G, AS-G. Shear force was applied to the adhesive interface in a Universal Testing Machine (crosshead speed: 1 mm/min). The types of failures were further evaluated and categorized as follows: 1) cohesive in the composite substrate and 2) adhesive at the interface. Bond strength values (MPa) were statistically analyzed using two-way analysis of variance and least significant difference post hoc tests (α=0.05). Significant effects of the adhesion strategy (p=0.006) and the composite type (p=0.000) were found. Interaction terms were not significant (p=0.292). Regardless of the substrate-adherend combination, protocol 1 (17–22 MPa) showed significantly higher results than did protocols 2 (15–17 MPa) and 3 (11–17 MPa) (p=0.028, p=0.002, respectively). The highest results were obtained from the G-G combination after all three protocols (17–22 MPa). The incidence of cohesive failures was more common when the substrate and the adherend were the same composite type (AS-AS: 87.5%, 87.5%, 75%; G-G: 100%, 75%, 50% for protocols 1, 2, and 3, respectively). When substrate and adherend were used interchangeably, adhesive failures were more frequent (25%, 50%, and 100% for protocol 1, 2, and 3, respectively). When the substrate and the adherend are of the same type, greater repair strength could be expected. In the repair of composites next to the dentin, depending on the composite type, conditioning the composite with silica coating and silanization after etching the dentin adds to the repair strength compared to the results obtained with silane application only.

INTRODUCTION

Resin-based composite materials (hereafter referred to as composites) are widely used in restorative dentistry.[1–3] Composites are becoming more durable with advances in the filler particles, monomer matrices, improved adhesive systems, and polymerization devices.[4] However, failures of composite restorations are still being reported in clinical studies,[5–7] with failure rates ranging between 5% and 45% during an observation period of up to five years. Ageing of such materials is often a consequence of mechanical/physical degradation mechanisms such as wear, abrasion, and fatigue or is due to chemical degradation mechanisms such as enzymatic, hydrolytic, acidic, or temperature-related breakdown.[6,8–10] While physical and chemical degradation phenomena occur as a function of time and are considered to represent late failures, early failures could occur as a result of mishandling of the material, failure to master the matrix technique, improper finishing and polishing procedures, or a mismatch between the restored tooth and the adjacent one.[1,11]

Complete replacement of failed restorations is usually costly and time consuming.[1,10–14] Moreover, removing tooth-colored restorations may lead to removal of intact dental tissues and induce destructive changes in odontoblasts.[15,16] When composite restorations fail as a result of discoloration, microleakage, ditching at the margins, delamination, or simple fracture, restorations need to be repaired or replaced.[3,7,15,16] Partial replacement is often preferable when possible. This can be achieved by adding a new layer of composite onto an existing one.

High bond strength is desired between the prepolymerized and the new composite layer for clinical durability.[1,2,4,9] Adhesion between two composite layers is achieved in the presence of an oxygen-inhibited layer of the unpolymerized resin.[17,18] Since prepolymerized composites contain fewer free radicals on their surfaces, several methods have been suggested[1,2,4,10–12,19,20] to improve the composite-composite adhesion through surface roughening

using airborne particle abrasion and etching agents such as acidulated phosphate fluoride, hydrofluoric acid, phosphoric acid or through the use of intermediate adhesive resins (IARs). Although promising results were obtained with some of these surface conditioning methods in earlier studies, adhesion tests were often performed on composite surfaces only.[1,2,4,18,20–23] In fact, in clinical situations, except in the complete relayering of a composite veneer, composite restorations such as Class IV, V, or occlusal composite fillings are surrounded with enamel and/or dentin.[1,14,24] Typically some amount of composite and the surrounding enamel are removed to create space for the new layering composite.

The success of composite-composite adhesion depends on the chemical composition of the surface, surface roughness,[25,26] wettability of the IARs or the new composite layer,[27] and the surface conditioning procedures applied.[28–31] During layering, composite materials are exposed to atmospheric oxygen, creating an oxygen-enriched surface layer that remains unpolymerized.[2,18] Though the oxygen-inhibited layer is viscous, it contains unreacted C=C bonds.[32–34] The unreacted C=C bonds of the functional groups on the surface of the polymerized resin matrix enables the monomer of the new resin composite to bond to it.[35] While authors of some studies[28,36] reported enhanced repair bond strength with the use of an IAR, others[25,32,36,37] claimed better results with physico-chemical conditioning of the composite surface. One example of the latter is the chairside tribochemical silica coating that creates both micromechanical and chemical reaction sites on the composite surfaces. In this technique, the surface is air-abraded with silica-coated alumina particles; this is followed by the application of a silane coupling agent and an IAR.[25,32,36,37] The application of silane on the composite was suggested[29] to improve the wettability of the fillers on the composite surface and, consequently, adhesion of the composite.

Successful adhesion to dentin has been established over the last two decades.[5] When composite repairs are performed next to dentin, it can be anticipated that the adhesion to the dentin could be sufficient such that additional conditioning of the composite may not be required. On the other hand, initial conditioning of the composite may impair the adhesion to the dentin. Hence, the detrimental effects of the conditioning sequence on the composite-dentin complex is not known. Furthermore, earlier studies[11,21,30,38] were often performed using the same type of composite as the substrate and adherent materials. This may not always be possible given the clinical situation since often the underlying composite type is not known unless the operator registers the type of the composite in the patient file.

Composite formulations have changed constantly over the years, and numerous brands of composites have been introduced in the dental market. The improvements in filler technology led to the development of the nanohybrid or nanofilled composites. As a result of their higher degree of conversion, as opposed to hybrid or microhybrid composites,[39] repair bond strength could be impaired for nanohybrid composites.

The objectives of this study, therefore, were to compare the effect of three adhesion strategies on the bond strength of resin composites to the composite-dentin complex and to evaluate the failure types. The null hypotheses tested were that different 1) adhesion strategies and 2) substrate-adherent combinations would not affect the bond strength.

MATERIALS AND METHODS

The brands, manufacturers, chemical compositions, and batch numbers of the materials used in this study are listed in Table 1.

Specimen Preparation

Intact maxillary central incisors (N=72, n=8 per subgroup) were collected and the roots of the teeth were removed under coolant water. The coronal parts of the teeth were embedded in autopolymerized poly(methyl methacrylate) (PMMA, Autoplast, Candulor AG, Altstätten, Switzerland) surrounded by a polyvinyl chloride (PVC) cylinder. The enamel surfaces were ground finished to 1200-grit silicone carbide paper under water until dentin was exposed. Cylindrical cavities (diameter: 2.6 mm; depth: 2 mm) were opened in the middle of the labial surfaces of the teeth using a standard bur, and the specimens were randomly divided into three groups. Two types of resin composites, namely, microhybrid (Quadrant Anterior Shine; AS) and nanohybrid (Grandio; G), were photo-polymerized incrementally in the cavities according to each manufacturer's recommendations. Each increment was photo-polymerized with a halogen polymerization unit (Demetron LC, SDS Kerr, Orange, CA, USA) for 40 seconds from a constant distance of 2 mm from the surface. Light intensity was 800 mW/cm^2 as verified by a radiometer (Demetron LC, Kerr).

Table 1: *The Brands, Manufacturers, Chemical Compositions, and Batch Numbers of the Materials Used in This Study*

Brand	Manufacturer	Chemical Composition	Batch Number
Resin composites			
Quadrant Anterior Shine (AS) (Microhybrid)	Cavex GmbH & Co KG, Haarlem, The Netherlands	bis-GMA, diurethane dimethacrylate, silica, silicate glass, and fluoride-containing fillers (63 v%)	010100
Grandio (G) (Nanohybrid)	Voco GmbH, Cuxhaven, Germany	bis-GMA dimethacrylate, UDMA, TEGDMA, glass-ceramic, SiO_2-containing filler (71.4 v%)	621332
Tribochemical silica coating kit			
CoJet-Sand	3M ESPE AG, Seefeld, Germany	Aluminum trioxide particles coated with silica, particle size: 30 μm	165092
ESPE-Sil	3M ESPE AG	3-Methacryloxypropyltrimethoxysilane, ethanol	152745
Intermediate adhesive resins			
Quadrant Unibond (for AS)	Cavex	bis-GMA, TEGDMA, silicate glass fillers, silica, polycarboxylic acid, camphorquinone	10049
Solobond Plus (for G)	Voco	bis-GMA, TEGDMA, HEMA, camphorquinone	591583

Abbreviations: bis-GMA, bisphenol A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate or 1,6-di(methacryloyloxyethylcarbamoyl)-3,30,5-trimethylhexaan.

Surface Conditioning Protocols

The surfaces of the substrate composites and the surrounding dentin were conditioned according to one of the following adhesion protocols: protocol 1: acid-etching (dentin) + silica coating (composite) + silanization (composite) + primer (dentin) + bonding agent (dentin + composite); protocol 2: silica coating (composite) + acid-etching (dentin) + silanization (composite) + primer (dentin) + bonding agent (dentin + composite); and protocol 3: acid-etching (dentin) + primer (dentin) + silanization (composite) + bonding agent (dentin + composite).

The primer and the bonding agents were the materials of the corresponding composite manufacturers. Silica coating was achieved using a chairside air-abrasion device (Dento-Prep, RØNVIG A/S, Daugaard, Denmark) filled with 30-μm alumina particles coated with silica (CoJet-Sand, 3M ESPE AG, Seefeld, Germany) from a distance of approximately 10 mm at a pressure of 2.5 bars for four seconds. Following surface conditioning, the remnants of sand particles were gently air-blown. After conditioning protocols, the repair resin was adhered to the substrate surfaces using transparent polyethylene molds with an inner diameter of 3.6 mm and a height of 5 mm and photo-polymerized. The same operator carried out the bonding procedures in accordance with the manufacturers' instructions throughout the experiments. The composite was packed against the substrate incrementally with a hand instrument, and each layer was photo-polymerized for 40 seconds. After polymerization, the polyethylene molds were gently removed from the test specimens. The substrate-adherent combinations were as follows: AS-AS, G-G, and AS-G.

Testing Procedure and Failure Analysis

Specimens were mounted in the jig of the Universal Testing Machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany), and the shear force was applied to the adhesive interface until failure occurred. The load was applied to the adhesive interface, as close as possible to the surface of the substrate. The

Table 2: *Results of Two-way Analysis of Variance for the Composite Types, Adhesion Protocols, and the Interaction Terms According to Bond Strength Data (* p<0.05)*

Source of Variation	DF	SS	MS	F	p
Composite type	2	466.312	233.156	9.758	0.000*
Adhesion protocol type	2	267.545	133.772	5.599	0.006*
Interaction (composite*protocol)	4	121.139	30.285	1.267	0.292
Error	1505.315	1505.315	23.894		
Total	22,234.509	22,234.509			

** Statistically significant difference at the level of α=0.05.*
Abbreviations:DF, degrees of freedom; SS, sum of squares; MS, mean square.

specimens were loaded at a crosshead speed of 1 mm/min, and the stress-strain curve was analyzed with the software program (Zwick ROELL). Subsequently, digital photos (Canon Ixus 40, Canon Inc, Tokyo, Japan) were taken from the substrate surfaces, and specimens were evaluated under optical microscope (MP 320, Carl Zeiss, Jena, Germany). The types of failures were categorized as 1) cohesive failure in the composite substrate and 2) adhesive failure at the interface.

From four separate specimens, cold field emission scanning electron microscope (SEM; JSM-6301F, Jeol Instruments, Tokyo, Japan) images were taken at 25 kV at a magnification of 10,000×. Surfaces were first sputter-coated with a 3-nm-thick layer of gold/palladium (80/20) prior to examination.

Statistical Analysis

Statistical analysis was performed using SPSS 11.0 software for Windows (SPSS Inc, Chicago, IL, USA). Bond strength data (MPa) were submitted to two-way analysis of variance. Multiple comparisons were made with the least significant difference (LSD) post hoc test (α=0.05), with the shear bond strength as the dependent factor and adhesion protocols and the composite material types as the independent factors. Values of $p<0.05$ were considered to be statistically significant in all tests. Power analysis was performed using a statistical software package (Stata, StataCorp, College Station, TX, USA).

RESULTS

Significant effects of the adhesion strategy (p=0.006) and the composite type (p=0.000) were found on the bond strength results. Interaction terms were not significant (p=0.292) (Table 2). Regardless of the substrate-adherent combination, protocol 1 (17–22 MPa) showed significantly higher results than did protocol 2 (15–17 MPa) (p=0.028) and 3 (11–17 MPa) (p=0.002). Protocol 3 presented the lowest results (11–17 MPa). The highest results were obtained from the G-G combination for all three protocols (17–22 MPa) (Table 3; Figure 1). The power of the study was calculated to be 82% (confidence interval, 95%).

The incidence of cohesive failures was more common when the substrate and the adherent were the same composite type (AS-AS: 87.5%, 87.5%, 75%; G-G: 100%, 75%, 50% for protocols 1, 2, and 3, respectively) (Figure 2). When substrate and adherent composites were used interchanged (AS-G), adhesive failures were more frequent (72.5%, 12.5%, 100% for protocols 1, 2, and 3, respectively) (Table 4).

Figure 3a-d shows SEM images of the composites before (left panel) and after (right panel) silica coating. The microfilled composite, AS, clearly showed larger filler particles than did nanohybrid G. It should be noted that these regions need not necessarily be at the surface but may also be slightly underneath the surface, covered by a thin resin matrix layer. After silica coating, similarly rough surfaces were evident in all composites.

DISCUSSION

This study was undertaken in order to investigate the effect of different repair protocols on the adhesion of composites to composite-dentin complexes using the shear bond strength test. This type of test produces a combination of shear, tensile, and

Table 3: *The Mean Bond Strength Values (MPa) With Standard Deviations (±SD) for Composite Combinations After Adhesion Protocols*[a]

Adhesion Protocol	AS-AS	G-G	AS-G
Protocol 1	17.5 ± 4.8 ^{aA}	22.3 ± 6.8 ^{aA}	17.7 ± 4.1 ^{aA}
Protocol 2	15.3 ± 3.2 ^{abA}	17.6 ± 5.4 ^{aA}	15.1 ± 6.7 ^{abA}
Protocol 3	11.7 ± 3.2 ^{bA}	17.7 ± 4.1 ^{aB}	11.4 ± 3.0 ^{bA}

Abbreviations: AS, Quadrant Anterior Shine; G, Grandio.
[a] *The same uppercase superscripted letters in the column (least significant difference [LSD] test, α=0.05) and capital letters in the row indicate no significant differences (LSD test, α=0.05). For group abbreviations see Table 1.*

compressive stresses that often occurs during chewing function.[14] Since it is technically impossible to study adhesion on complex assemblies of dentin-composite or enamel-composite using tensile or microtensile tests,[40] the shear test was employed.

The surface conditioning methods indicated for composite repairs are often based on mechanical roughening with burs,[9,10,37] airborne particle abrasion,[1,4] or use of etching agents[9,10,13] followed by application of adhesive systems.[1,4,20] Particle deposition techniques increase the surface area, and adhesion of the adherent composite is achieved through mechanical interlocking.[1,4,32] This retentive surface texture also favors the surface wettability of the composite.[5,19] Based on previous favorable findings,[1,4,20,22,25,29,40] in this study, composite specimens were conditioned using silica coating and silanization. In this method, after air-abrasion with 30-μm alumina particles coated with silica,[4,22] the surfaces are coated with silane that makes the surface more reactive for the methacrylate groups of the repair resin.

The results of this study showed a significant influence of the adhesion protocol. Therefore, the first hypothesis—that the different adhesion strategies would not affect the bond strength—could be rejected. The highest mean bond strength was obtained using protocol 1, in which the dentin was etched and the composite was then silica-coated. After etching the dentin, dentinal tubules could be expected to be closed by the sand particles, and as a consequence, the bond strength would be impaired when compared to the bond strength associated with the other protocols. Since this was not the case, an adverse effect of air-abrasion could not be confirmed, but it must be also noted that in this current study, the dentin surrounding the composite was limited. The reason for the choice of 0.5 mm of dentin surrounding the composite was that it imitated the

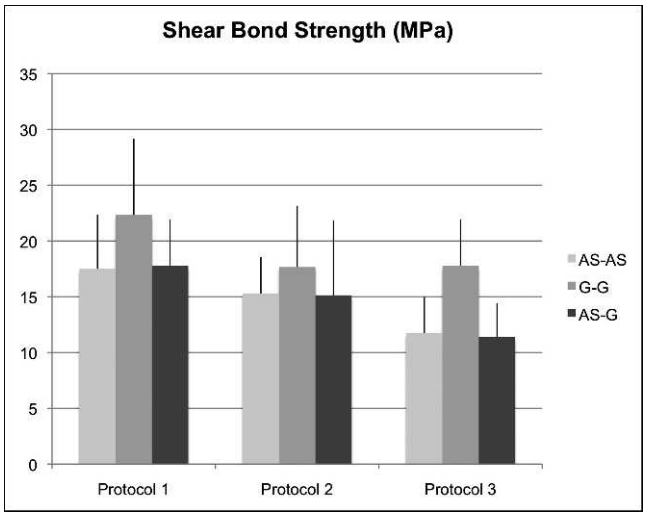

Figure 1. *The mean shear bond strength values (MPa) of composites to composite-dentin complex after three adhesion protocols.*

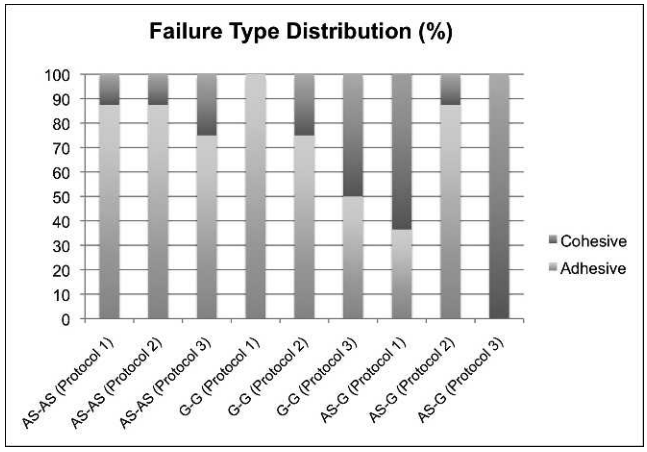

Figure 2. *Distribution of failure types in percentage analyzed after shear bond strength test depending on the substrate-adherent combinations after three adhesion protocols.*

Table 4: *Distribution and Frequency of Failure Types per Experimental Group Analyzed After Bond Strength Test*

Adhesion Protocol	Failure Type	AS-AS	G-G	AS-G
1	A	87.5	100	37.5
	B	12.5	0	72.5
2	A	87.5	75	87.5
	B	12.5	25	12.5
3	A	75	50	0
	B	25	50	100

Abbreviations: [Failure Type] A, cohesive failure in the composite substrate; AS, Quadrant Anterior Shine; [Failure Type] B, adhesive failure at the interface; G, Grandio.

tooth surface after beveling, since clinical over-contouring of the enamel/dentin surfaces during repairs is avoided. However, in the case of ceramic inlay, onlay, or overlay fractures, the exposed dentin surfaces may be wider. This aspect requires further investigation in situations in which the surrounding dentin is wider than 0.5 mm.

Considering the adhesion protocols regardless of the composite type, it can be stated that physico-chemical conditioning the composite surface adds to the adhesive strength of the adherent. When the composite surfaces were only silane-coated but not physico-chemically conditioned (protocol 3), the mean bond strength was lower. This is in contrast to the results of two previous studies[19,41] in which silane application was found to be sufficient for composite repairs. Silanes are molecules with two functional groups. While silanol groups react with the inorganic filler particles of the resin, organo-functional groups react with the methacrylate groups in the adhesive system. A covalent bond may be established between the monomers in the adhesive system and the inorganic filler particles in the composite using the silanes.[42] When no fillers are exposed on the substrate surface, the silane has to react with the monomer matrix only, which could be the situation in previous studies. Furthermore, the wettability capacity of the substrate-adherent combinations as well as IAR types could also have played a role in the variation seen in the results. The existence of the surrounding dentin did not increase the adhesion of the composite. In this group, the results were lower than in a previous report[40] in which only composites were repaired without any approximating dentin. Nevertheless, considering the high incidence of adhesive failures after using

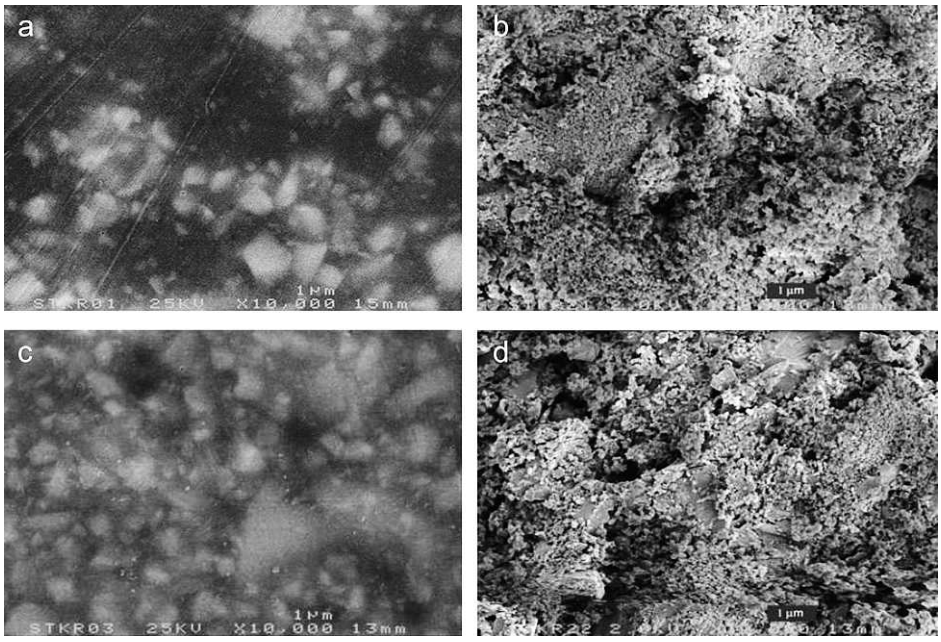

Figure 3. *(a-d) SEM micrographs (10,000×) of the two composites Quadrant Anterior Shine (a) before and (b) after silica-coating and Grandio (c) before and (d) after silica-coating. Bar marker indicates 1 μm.*

protocol 3, there seems to be a need for surface conditioning for micromechanical retention.

The composite type also significantly affected the results of this study. Therefore, the second hypothesis—that the different substrate-adherent combinations would not affect the bond strength—was also rejected. In clinical practice, the type of composite for immediate repairs is usually the same substrate. However, in some cases, as a result of better color choices, another type of composite may be chosen for relayering. In this case, the substrate and the adherent composites would be different. In addition, in some situations, if the patient already has a composite restoration, the information on the substrate type may not be traced. In such situations, a different repair composite is used. The results of this study showed that adhesion of different substrate-adherent combination (AS-G) resulted in the highest incidence of adhesive failures. This indicates that the adhesion did not reach the cohesive strength of the substrate composite. Since both composites are basically methacrylate-based materials, one reason for the adhesive failures could be the variation between the surface wettability properties of the AS (microhybrid) and G (nanohybrid) composites.[1] During the experiments, it was noted that the G composite was less viscous than AS. It is likely that surface contact was not ideal between AS and G. On the other hand, the high mean repair strength could be due to better compatibility or a greater number of free radicals available on the G substrate in the G-G combination. In future studies, the degree of conversion in correlation with the repair bond strength needs to be evaluated using Raman spectroscopy.

The bond strength values for clinically durable repairs are not known to date, but adhesion to enamel is usually considered the gold standard.[5,7] In all groups, the results ranged between 11 and 22 MPa. These values were comparable to or lower than those of previous studies[1,2,4,10,40] in which composite-composite adhesion was tested. Rinastiti and others[3] found shear bond strengths for AS and G using IARs for immediate repairs of 15.0 ± 6.6 MPa and 15.8 ± 5.9 MPa, respectively. Similar to the findings of this study, tribochemical silica coating increased the immediate repair strength of AS and G to 25.0 ± 8.5 MPa and 26.3 ± 7.9 MPa, respectively. The results obtained for G are in accordance with the findings of this study, whereas the results for AS are not in agreement with those of this study. The reason for this could be a cross-contamination effect of the conditioning method on the tooth substance. It was not practically possible to avoid the effect of air-abrasion on the dentin. Similarly, when dentin was etched, during washing and rinsing the composite surface also received some phosphoric acid. This cross-contamination might have decreased the bond results. There is certainly a needfor adhesives that could condition multiple surfaces with different natures (ie, tooth-restoration surfaces) at the same time. The obtained results need to be verified in clinical studies.

Substrate-adherent combinations were not aged, which could be considered a limitation of this study. Polymeric materials tend to absorb water and degrade when they are exposed to hydrothermal aging conditions.[29,34] Therefore, the effect of the studied protocols may vary when the composite combinations are aged. This aspect needs to be further investigated.

CONCLUSIONS

From this study, the following can be concluded:

- Increased repair strength was obtained with a high incidence of cohesive failures in the substrate when the substrate and the repair composite are of the same type.
- The nanohybrid composite tested presented higher repair strength compared to the microhybrid composite.
- For durable repair of the composite-dentin complex, after etching the dentin, neighboring composite surfaces need to be activated with air-abrasion and silanization. This procedure presented better results than did silane-only application on the composite.

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Conflict of Interest

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

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Pulpal and Periapical Response After Restoration of Deep Cavities in Dogs' Teeth With Filtek Silorane and Filtek Supreme XT Systems

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Clinical Relevance

Both the silorane and methacrylate resin systems showed good tissue compatibility, suggesting that their placement in contact with deep dentin in clinical procedures may be appropriate.

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SUMMARY

Objective: **This study evaluated, histopathologically, the pulpal and periapical response to a silorane-based resin (Filtek Silorane) and a methacrylate-based nanoparticle resin (Filtek Supreme XT) in deep cavities in dogs, having zinc oxide and eugenol-based cement (ZOE) as a control.**

Methods: **The tooth/bone blocks were collected after 10 and 90 days and processed for microscopic analysis of the dentin, pulp, and periapical tissues using a score system. Data were analyzed statistically by Kruskal-Wallis and Dunn post-test (α=0.05).**

Results: **At 10 days, the pulp, connective tissue, and periodontal ligament showed normal characteristics. No resorption areas were observed. Both resins caused significantly less ($p<0.05$) periapical and pulpal inflammatory**

response than ZOE. At 90 days, for all materials, the connective pulp tissue was healthy and dense, with a normal blood vessel system. The apical and periapical region had normal structure and thickness.

Conclusions: **The use of the Filtek Silorane and the Filtek Supreme XT resins caused no adverse pulpal and periapical reactions after restoration of deep dentin cavities *in vivo*.**

INTRODUCTION

Reactive components released from conventional composite resins, such as unreacted monomers, may induce toxicity or inflammatory tissue reactions depending on their aggressiveness[1,2] and the amount of dentin remaining on the cavity floor.[3] In contact with exposed pulp, adhesive systems may also unchain an unfavorable tissue reaction, causing pulp necrosis and apical periodontitis.[4]

The results of cell culture studies have shown that methacrylate and dimethacrylate monomers, commonly used in restorative polymeric technology, may affect the recruitment of leukocytes in inflammation sites by decreasing the expression of intercellular adhesion molecules[5] and inducing enzymatic activity and expression of growth factors and cellular cytokines.[6] In addition, resin monomers suppress the mitochondrial activity of macrophages and alter their inflammatory responses.[7,8]

In the last few years, methacrylate-based resins with modifications in their composition and structure have been developed, such as Filtek Supreme XT. This resin is a nanocomposite recently launched by 3M ESPE (St Paul, MN, USA) that contains only nanoparticles and nanoclusters as inorganic fillers. The nanoparticles are monodispersed, nonagglomerated silica particles, while the nanoclusters are spheroidal agglomerates consisting of nano-sized silica and zirconia particles. The micron-sized porous cluster is infiltrated with silane coupling agents to allow chemical bonding with the organic matrix. Although adequate physical properties have been attributed to these resins (eg, improved surface polishing),[9] toxic effects have been observed in cell cultures.[10]

On the other hand, the occurrence of polymerization shrinkage and the release of chemical products from methacrylate-based systems have led to the development of resinous materials with improvement of polymerization technology, wear resistance, esthetics, and adhesive properties.[11] Recently, a silorane-based composite resin with a distinctive polymerization characteristic to reduce polymerization shrinkage has been introduced to the market (Filtek Silorane, 3M ESPE). Silorane matrix is formed by the cationic ring-opening polymerization of the silorane monomers. A silorane molecule represents a hybrid that is made of both siloxane and oxirane structural moieties. Silorane technology has afforded a highly hydrophobic restorative material with reduced polymerization shrinkage,[12,13] more balanced volumetric stress, higher ambient light stability,[12] and insolubility in biologic fluids.[14]

Although several recent studies have evaluated physicochemical properties of silorane-based resins,[13-17] research on their biologic effects is quite limited, being based mostly on *in vitro* and cell culture studies.[5,10,18] To the best of our knowledge, the *in vivo* pulpal and periapical response to these new restorative systems has not yet been investigated.

The present study evaluated the *in vivo* pulpal and periapical response to a silorane-based resin system (Filtek Silorane) and a methacrylate-based nanoparticle resin (Filtek Supreme XT) after restoration of deep cavities prepared in dogs' teeth.

MATERIALS AND METHODS

All experiments were conducted strictly in accordance with the guidelines of the university's Ethics Committee for the Care and Use of Laboratory Animals. The study design and histologic parameters for pulpal and periapical tissue reaction evaluation were in accordance with the protocol recommended by the International Organization for Standardization for biologic evaluation of dental materials (ISO standard 7405:1997).[19]

The second and third maxillary premolars and the second, third, and fourth mandibular premolars of five mongrel dogs aged 12–18 months and weighing 15 kg on average were selected for the study, providing 50 teeth. The dogs were maintained in quarantine and received vermifuges, vitamin supplements, and antirabic and triple vaccines to put them in adequate health conditions for the experiment. The dogs were housed with free access to water and standard lab chow during the whole course of the study.

The 50 teeth were randomly distributed into six groups: groups I and IV (experimental) (n=10 teeth/group)—restoration with Filtek Silorane system (3M ESPE) for 10 and 90 days, respectively; groups II and V (experimental) (n=10 teeth/group)—restoration with Filtek Supreme XT system (3M ESPE) for

10 and 90 days, respectively; groups III and VI (control) (n=5 teeth/group)—restoration with zinc oxide and eugenol-based cement (ZOE) (Caulk IRM Intermediate Restorative Material, Dentsply, Milford, DE, USA) for 10 and 90 days, respectively.

The animals were preanesthetized with an endovenous injection of levomepromazine (Neozine; 1 mg/kg body weight; Aventis Pharma, São Paulo, SP, Brazil) 15 minutes before the operative procedures and then anesthetized with an endovenous injection of tiletamine hydrochloride and zolazepam hydrochloride (0.1 mL/kg body weight; Zoletil 50, Virbac do Brazil Ind e Com, São Paulo, Brazil) to facilitate the passage of an endotracheal tube. Inhalation anesthesia with Isoflurane (Abbott Laboratories, Saint-Laurent, Quebec, Canada) was delivered using an inhalation anesthesia apparatus (Takaoka KT-20, Takaoka Ind e Com, São Paulo, Brazil). Throughout the duration of the operative procedures, the animals were maintained on isotonic saline solution (0.9% NaCl; Glicolabor Indústria Farmacêutica, Ribeirão Preto, SP, Brazil).

Standardized radiographs of the teeth to be treated were taken using custom-made film-holding devices and size 2 periapical films (Ultraspeed, Eastman Kodak Co, Rochester, NY, USA). An exposure time of 1 second was used with the x-ray equipment (Heliodent, Siemens, Siemens Medical Systems, Iselin, NJ, USA) operating at 60 kVp and 10 mA. The exposed films were processed in an automated processing machine.

All teeth were isolated with a rubber dam, and the operative field was disinfected with 3% hydrogen peroxide followed by the application of 1% chlorhexidine gluconate. After pumice/rubber cup prophylaxis, Class V cavities were prepared on the buccal surface of each tooth with a sterile #1015 bur (KG Sorensen, São Paulo, SP, Brazil) in a high-speed handpiece under abundant air/water spray cooling. Cavity depth was standardized based on the size of the active part of the #1015 bur and the mean thickness of the enamel and dentin of canine teeth, leaving a remaining dentin thickness in the cavity floor between 0.5 and 1.0 mm. The burs were used with intermittent movements and without pressure to avoid overheating. Burs were replaced every four preparations to maintain a good cutting efficiency. The cavities were abundantly irrigated with sterile saline (Glicolabor Indústria Farmacêutica Ltda, Ribeirão Preto, SP, Brazil) to remove debris and enamel and dentin chips, and were dried with cotton pellets.

The cavities were restored with the materials corresponding to each group (I to VI), according to the manufacturers' instructions. In groups I and IV, Filtek Silorane self-etch primer was applied actively for 15 seconds with a disposable microbrush tip, followed by gentle air dispersion and light curing for 10 seconds with a halogen source (Ultralux Electronic, Dabi Atlante, Ribeirão Preto, SP, Brazil) with light intensity of 450 mW/cm^2 as measured with a curing radiometer (Demetron, Kerr Corp, Danbury, CT, USA). A layer of Filtek Silorane adhesive bond was applied, followed by gentle air thinning and 10 seconds of light curing. The cavities were restored with the composite resin using an incremental technique. In groups II and V, Scotchbond Etchant was applied for 15 seconds, rinsed for 10 seconds, and excess water was blotted using a cotton pellet. Two to three consecutive coats of Adper Single Bond 2 Plus Adhesive were applied for 15 seconds with gentle agitation using a fully saturated applicator, gently air thinned for 5 seconds to evaporate solvent, and light-cured for 10 seconds. In groups III and VI, ZOE was prepared and taken to the cavities following the conventional technique.

As all variables should be evaluated in the same animal and in the different dental quadrants, each hemiarch was alternately subjected to the different experimental protocols. Each hemiarch was radiographed, and the animals were maintained under observation during the whole experiment.

At the predetermined experimental periods (10 and 90 days postoperatively), the teeth of each group were radiographed, and the animals were euthanized with a lethal intravenous overdose of sodium pentobarbital. The maxilla and mandible were dissected, reduced in volume, and the restored teeth were removed in blocks (tooth/bone) using a water-cooled diamond saw. The specimens were fixed in a 10% buffered formalin solution for 48 hours at room temperature, demineralized in 10% EDTA, pH 7.4, during approximately 30 days, subjected to routine histologic processing, and embedded in paraffin.

Five-micrometer-thick slides were serially cut longitudinally in a buccolingual direction and were stained with hematoxylin and eosin, Mallory's Trichrome, and Brown and Brenn staining techniques. A single calibrated observer blinded to the treatment groups performed all examinations in a subjective manner using an optical microscope (Axio Imager.M1, Carl Zeiss, Gottingen, Germany) coupled to a digital camera (AxioCam MRc5, Carl Zeiss). The pulpal and periapical response to the tested

materials was evaluated in front of the whole extension of the pulpal wall of the cavity based on the histopathologic parameters described by Silva and others.[4,20] The characteristics of dentin, pulp, and periapical tissues were evaluated according to the following scores: Odontoblast layer: 1 - present, 2 - absent; Inflammatory pulp response: 1 - absent, 2 - mild, 3 - moderate, 4 - severe; Mineralized tissue resorption: 1 - absent, 2 - mild, 3 - moderate, 4 - severe; and Bacteria: 1 - present, 2 - absent. The data were analyzed statistically by Kruskal-Wallis and Dunn post-test using Graph Pad Prism 5.0 (Graph Software Inc, San Diego, CA, USA). A significance level of 5% was set for all analyses.

The thickness of remaining dentin between the pulpal wall of the cavity and the pulp chamber roof was measured in micrometers in images obtained from three slides per specimen at 5× magnification. In all teeth, the remaining dentin thickness was standardized in three regions: at half of the length of the pulpal wall of the cavity and at two regions equidistant between this point and the lateral wall in the right and in the left.

For the radiographic analysis, three calibrated observers (kappa=0.9) examined the periapical radiographs taken before, 10 days and 90 days after the operative procedures as to the presence or absence of lamina dura, areas of periapical bone rarefaction, and internal and external root resorption.

RESULTS

After the 10- and 90-day experimental periods, all fillings were still present in all teeth of the six groups. The mean thickness of remaining dentin between the pulpal wall of the cavity and the pulp chamber roof was 530 μm (standard deviation = 225 μm).

Groups I and II: Filtek Silorane and Filtek Supreme XT (10 Days)

As these groups presented similar histopathologic features, the results are presented together as follows. In all 10 teeth, the pulp tissue was normal, exhibiting an odontoblast layer with vacuolization areas under the remaining dentin layer. In the superficial portion of the pulp, the connective tissue was normal with mild vascular congestion and spindle-shaped fibroblasts. The apical region was normal, with a uniform cementum layer and cementoblasts on the entire surface. The periodontal ligament was normal with intense presence of fibers and absence of inflammatory cells. There were no areas of mineralized tissue resorption. Bacteria were not detected.

Group III: ZOE (10 Days)

In the five teeth of this group the histopathologic features were similar to those described in groups I and II, except for discrete presence of mononuclear inflammatory cell in the pulp core, close to the remaining dentin layer. Bacteria were not detected.

Groups IV and V: Filtek Silorane and Filtek Supreme XT (90 Days)

As these groups presented similar histopathologic features, the results are presented together as follows. One tooth of group IV and two teeth of group V had accidental pulp exposure during cavity preparation, reducing the number of teeth to nine and eight, respectively. In all teeth of both groups, the pulp tissue was healthy, exhibiting a normal odontoblast layer underneath the remaining dentin layer and along the root canal walls. The connective tissue was dense, with a normal vascular system. The apical and periapical region had normal structure and thickness. There were no areas of mineralized tissue resorption or inflammatory cells. Figures 1 and 2 present sets of panels of microscopic images of these groups. The histopathologic features observed at 90 days are also representative of those observed at 10 days. Bacteria were not detected, as illustrated in Figures 1D and 2C.

Group VI: ZOE (90 Days)

At 90 days, the five teeth restored with ZOE presented similar histopathologic features to those observed in the 10-day period, except for the absence of inflammatory cells in the pulp tissue adjacent to the remaining dentin.

Statistical Analysis

Statistically significant difference was found only for periapical and pulpal inflammatory response at 10 days: Filtek Silorane and Filtek Supreme XT had a similar behavior and both materials differed significantly from ZOE ($p<0.05$). The distribution of teeth according to the scores attributed to the histopathologic parameters in the four groups and the statistical analysis are presented in Table 1.

Radiographic Analysis

There were no alterations regarding the integrity of the lamina dura, presence of areas of periapical bone rarefaction, or presence of internal or external root

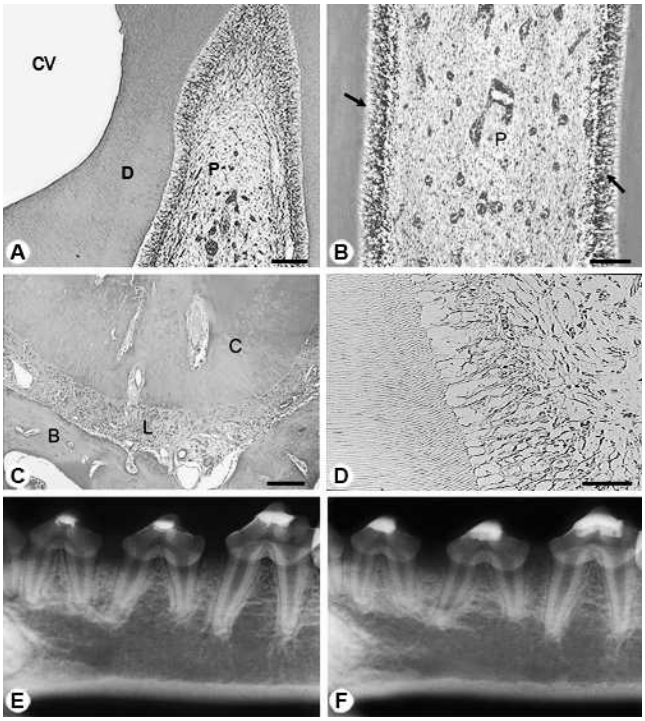

Figure 1. *Filtek Silorane system (90 days). (A): Cavity (CV), normal dentin (D), and pulp tissue (P). Hematoxylin Eosin (H.E.), 100× magnification. Scale bar: 50 μm. (B): Pulp tissue (P) with accentuate fibroblast population, vessels, and odontoblast layer (arrows). H.E., 200× magnification. Scale bar: 20 μm. (C): Apical and periapical region. Normal periodontal ligament (L), cementum (C), and alveolar bone (B). H.E., 100× magnification. Scale bar: 20 μm. (D): Representative Brown- and Brenn-stained images illustrating the absence of bacteria. 400× magnification. Scale bar: 50 μm. (E): Radiographic image immediately after the operative procedures. (F): Radiographic image 90 days after the operative procedures, showing no alterations.*

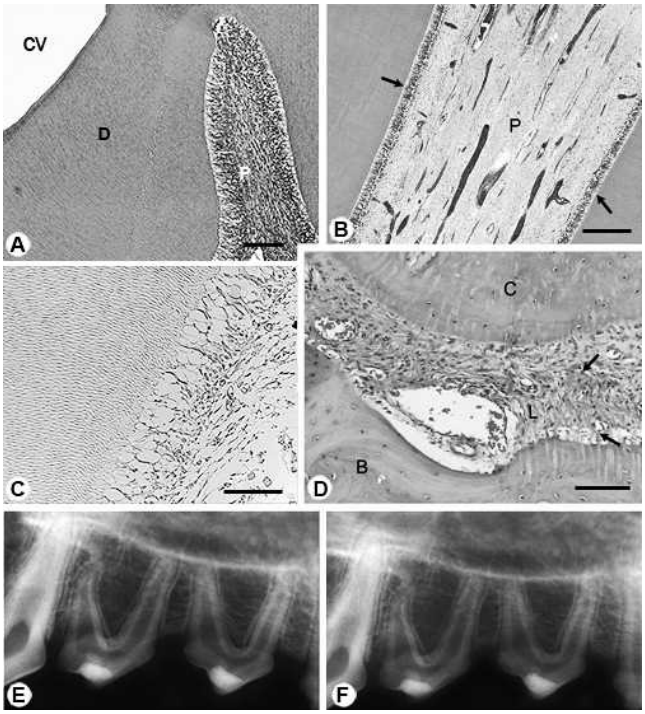

Figure 2. *Filtek Supreme XT (90 days). (A): Cavity (CV), normal dentin (D), and pulp tissue (P). H.E., 100× magnification. Scale bar: 50 μm. (B): Normal pulp (P) and odontoblast layer (arrows). H.E., 100× magnification. Scale bar: 20 μm. (C): Representative Brown- and Brenn-stained images illustrating the absence of bacteria. 400× magnification. Scale bar: 50 μm. (D): Interwoven collagen fibers (arrows) in the periodontal ligament (L), normal cementum (C), and alveolar bone (B). H.E., 200× magnification. Scale bar: 20 μm. (E): Radiographic image immediately after the operative procedures. (F): Radiographic image 90 days after the operative procedures, showing no alterations.*

resorption in any specimen at either of the two experimental periods.

Pulp Exposure (Groups IV and V)

In the teeth restored with Filtek Silorane and Filtek Supreme XT in which the pulp was accidentally exposed during cavity preparation, the odontoblast layer was absent and the pulp tissue, if present, was necrotic along the canal extension. In the root apex, cementum surface was free of cementoblasts and had a markedly irregular appearance due to resorption. The periodontal ligament was widened, exhibiting intense fiber dissociation, edema, and mononuclear and polymorphonuclear inflammatory cell infiltrate. Microorganisms were not observed.

DISCUSSION

As restorations are frequently placed in deep cavities, the transdentinal diffusion of products released from restorative materials towards the dental pulp may cause pathologic alterations, depending on the size of the molecule, material composition, the surface available for diffusion, dentin tubule permeability, and remaining dentin thickness. For these reasons, deep cavities were prepared to evaluate the pulpal and periapical tissue compatibility of Filtek Supreme and Filtek Silorane resin systems.

Pulp reaction to cavity preparation may vary from a mild inflammatory response associated with slight tissue disorganization to partial pulp necrosis or complete pulp breakdown. Several factors such as air drying of exposed dentin, heat generation by continuous cutting during cavity preparation, or inadequate water cooling, among others, have been considered as responsible for eliciting pulp damage.[21,22] In the present study, the cavities were prepared under copious air/water spray cooling and with intermittent movements using high-speed burs

Table 1: *Distribution of Teeth According to the Scores Attributed to the Odontoblast Layer, Periapical and Pulpal Inflammatory Response, Mineralized Tissue Resorption and Presence of Bacteria in Groups I (Filtek Silorane; 10 Days), II (Filtek Supreme XT; 10 Days), III (ZOE; 10 Days), IV (Filtek Silorane; 90 days), V (Filtek Supreme XT; 90 days), and VI (ZOE; 90 days)*[a]

Histopathological Parameters	Scores	Group					
		I	II	III	IV	V	VI
Odontoblast layer	Intact (score 1)	10	10	5	9*	8*	5
	Absent (score 2)	0	0	0	0	0	0
	Statistical analysis	a	a	a	a	a	a
Periapical and pulpal inflammatory response	Absent (score 1)	10	10	0	9	8	5
	Mild (score 2)	0	0	10	0	0	0
	Moderate (score 3)	0	0	0	0	0	0
	Severe (score 4)	0	0	0	0	0	0
	Statistical analysis	a	a	b	a	a	a
Mineralized tissue resorption	Absent (score 1)	10	10	5	9	8	5
	Mild (score 2)	0	0	0	0	0	0
	Moderate (score 3)	0	0	0	0	0	0
	Severe (score 4)	0	0	0	0	0	0
	Statistical analysis	a	a	a	a	a	a
Bacteria	Present (score 1)	10	10	5	9	8	5
	Absent (score 2)	0	0	0	0	0	0
	Statistical analysis	a	a	a	a	a	a

[a] *Same letters indicate no statistically significant difference, $\alpha=0.05$.*
** One tooth of group IV and two teeth of group V had accidental pulp exposure during cavity preparation, reducing the number of specimens to nine and eight teeth, respectively.*

with good cutting efficiency. These cautions probably minimized the thermal aggressions, since no significant pathologic alterations were found in the 10-day period in either of the groups.

Another parameter that may have an impact on the results is the evaluation period. In the present study, the restorations were made at two different time points of an 80-day interval (10 and 90 days) to allow the collection of the teeth at the same time. The rationale for this sampling schedule design was primordially to have restorations of both experimental periods placed in the same animal. Another methodological design would be to perform the restorative work for all animals at baseline and then conduct the sacrifice/collection of teeth according to

the preset schedule at 10 days and 90 days. However, the use of this sampling schedule (ie, place the restorations in different animals allocated to each experimental period) would have some important implications that were taken into consideration during preparation of the study. Firstly, the fact that dogs are not isogenic animals would bring more interferences and biases to the results. Having a material placed in one animal sacrificed at 10 days and having the same material placed in a different animal sacrificed at 90 days would disregard the individual factors inherent to each animal, as every animal responds differently to the experimental conditions and evaluation times. Secondly, there would be ethical implications because the number of animals to be sacrificed for collection of teeth at 10 days and then at 90 days should be considerably increased. Finally, the overall costs of the study would be significantly increased.

Filtek Supreme XT

In the last few years, methacrylate-based resins with modifications in their composition and structure have been developed, such as the nanocomposite Filtek Supreme XT. Although these resins have better physical properties, their cytotoxicity in cell cultures has been demonstrated.[10] Filtek Supreme XT has also been shown to have a high monomer release rate[23] and cytotoxicity to L929 fibroblasts.[24] Although the characterization of tissue response is a key factor to substantiate the clinical use of new dental materials, no histopathologic study has yet evaluated pulp response *in vivo* after restoration of deep cavities with Filtek Supreme XT resin. In the present study, there was good pulpal and periapical response to the placement of this material in deep cavities. This possibly occurred because Filtek Supreme XT is composed of nanoparticles and nanoclusters,[9] which may reduce the interstitial space and, consequently, the amount of organic matrix capable of releasing methacrylates, thus causing less tissue irritation than conventional methacrylate-based resins.

System Filtek Silorane

According to Ilie and Hickel,[16] the macromechanical, micromechanical, and nanomechanical properties of silorane-based resins are comparable to those of methacrylate-based resins. Cell culture studies investigating the biologic properties of silorane-based resins have shown no cytotoxic effects[5] and low mutagenic potential.[18] Another study[10] evaluating the *in vitro* cytotoxicity of different resins in Balb/c 3T3 mouse fibroblast cell cultures during eight weeks found that the methacrylate-free resin Hermes, precursor of Filtek Silorane, presented lower cytotoxicity than methacrylate-based resins and was similar to that of Teflon (control). However, to date, there is no published study evaluating the *in vivo* pulpal and periapical response to the use of Filtek Silorane as a restorative material in deep cavities. In the present study, the pulp tissue had an adequate response to the placement of this material in deep dentin at both evaluation periods. Materials with new polymerization modes are promising for reducing the release of uncured components and, consequently, their cytotoxicity.[10] The results of the present study may have great clinical significance because, in addition to its claimed low polymerization shrinkage, which improves its clinical performance, Filtek Silorane showed tissue compatibility when placed in contact with deep dentin *in vivo*.

Pulp Exposure (Groups IV and V)

It has been demonstrated that, even in the absence of bacterial contamination, direct pulp capping with adhesive systems causes an inflammatory reaction,[4,25] disruption, or disappearance of the odontoblast layer, pulp necrosis,[26] and absence of dentin barrier formation.[27] Gerzina and Hume[28] observed that the hybrid layer produced with methacrylate-based adhesive systems cannot be considered an impermeable barrier because it does not prevent the diffusion of monomers to the pulp. In our study, the teeth in which cavities with accidental pulp exposure were restored with Filtek Supreme XT and Filtek Silorane presented pulp necrosis, periodontal ligament widening, edema, intense mononuclear and polymorphonuclear inflammatory cell infiltrate, bone resorption, and cemental resorption, even in the absence of microorganisms. These results indicate that these materials should not be placed in direct contact with exposed pulp.

ZOE

The teeth restored with ZOE did not present significant pulpal alterations, except for the presence of inflammatory cell infiltrate in the 10-day period, which was more intense that that observed in the teeth restored with the resins. These results are in agreement with those of Murray and others[29] who reported that the use of ZOE for indirect pulp capping did not cause intense pulp reactions.

CONCLUSION

Based on the findings of this *in vivo* study, it may be concluded that the use the silorane-based (Filtek Silorane) and the methacrylate-based nanoparticle (Filtek Supreme XT) resins caused no adverse pulpal and periapical reactions when placed in deep dentin cavities. Further research at other research levels and with longer evaluation periods, including clinical trials, are needed to support the clinical use of these materials.

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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Phosphoric Acid-Etching Promotes Bond Strength and Formation of Acid-Base Resistant Zone on Enamel

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Clinical Relevance

The present study was undertaken to examine the effect of phosphoric acid (PA) etching on the bond strength and acid-base resistant zone (ABRZ) formation of a two-step self-etching adhesive to enamel. Taking both bond strength and ABRZ formation into consideration, we recommend bonding enamel by the means of directly applying Clearfil SE Bond adhesive after 35% PA etching.

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SUMMARY

This study examined the effect of phosphoric acid (PA) etching on the bond strength and acid-base resistant zone (ABRZ) formation of a two-step self-etching adhesive (SEA) system to enamel. An etch-and-rinse adhesive (EAR) system Single Bond (SB) and a two-step SEA system Clearfil SE Bond (SE) were used. Human teeth were randomly divided into four groups according to different adhesive treatments: 1) SB; 2) SE; 3) 35% PA etching→SE primer→SE adhesive (PA/SEp+a); (4) 35% PA etching→SE adhesive (PA/SEa). Microshear bond strength to enamel was measured and then statistically analyzed using one-way analysis of variance and the Tukey honestly significant difference test. The failure mode was recorded and analyzed by χ^2 test. The etching pattern of the enamel surface was observed with scanning electron microscope (SEM). The

bonded interface was exposed to a demineralizing solution (pH=4.5) for 4.5 hours and then 5% sodium hypochlorite with ultrasonication for 30 minutes. After argon-ion etching, the interfacial ultrastructure was observed using SEM. The microshear bond strength to enamel of the SE group was significantly lower ($p<0.05$) than that of the three PA-etched groups, although the latter three were not significantly different from one another. The ABRZ was detected in all the groups. In morphological observation, the ABRZ in the three PA-etched groups were obviously thicker compared with the SE group with an irregular wave-shaped edge.

INTRODUCTION

In recent years, bonding to tooth substrates with self-etching adhesive (SEA) systems has gained popularity. Because these systems contain specific acidic monomers to condition and prime tooth substrates simultaneously, separate etching and water-rinsing steps are eliminated, reducing the application procedures and technique sensitivity.[1] Furthermore, it has been shown that some functional monomers in SEA can chemically interact with the hydroxyapatite in the tooth demineralized layer within a clinically manageable time.[2–7] Hypothetically, this chemical interaction can improve the bonding performance and degradation resistance of the bonding interface.[8–10]

Nevertheless, unlike the predominant position of SEA in dentin bonding, bonding to enamel with SEA seems to be a controversial issue. The literature does not provide a straightforward answer whether SEA bonded to enamel can resist the mechanical and chemical challenges as well as the conventional etch-and-rinse (ERA) systems can. Some studies indicated that contemporary SEA could be used as a satisfactory alternative when bonding to enamel,[11–13] whereas other published work demonstrated that enamel bonding using SEA was inferior to that of ERA systems, probably related to the comparatively moderate etching capacity of most SEA systems.[14–17] They demineralized enamel mildly, resulting in shallow intercrystallite resin infiltration and a lack of interprismatic resin tag formation.[18–19]

Given that enamel bonding is believed to be primarily based on the micromechanical interlocking structure of adhesive resin into microporosities, it seems logical that the etching procedure should influence the bonding performance of an adhesive. The adjunctive use of phosphoric-acid (PA) etching is recommended to improve the bonding of some SEA systems by facilitating enamel dissolution.[20,21] It was repeatedly demonstrated that, with this method, the enamel bond strength significantly increased. In most of these studies,[20,21] the enamel surface was PA etched, rinsed, and then treated with the recommended procedures of commercial SEA systems. This procedure is beyond dispute for those single-step SEAs with a mild pH. However, questions arise for two-step SEA, which involves two application steps: the conditioning of tooth susbstrates with a self-etching primer, followed by the application of a bonding resin. It is still not clear, after enamel is PA-etched, whether a self-etching primer ought to be used and to what extent a self-etching primer contributes to the overall bond strength.

The acid-base resistant zone (ABRZ), a structural layer formed on the tooth bonding interface, has been confirmed by many studies.[22–27] Because this layer can resist acid and base challenges, it might play an important role in the prevention of secondary caries. In previous studies, the effects of various dental materials on the formation of dentin ABRZ were assessed. It has been shown that morphology of dentin ABRZ was highly adhesive-material dependent and that dentin ABRZ formed in SEA systems but not in ERA systems.[24,25,28] This phenomenon might be related to the aggressive etching capacity of PA, demineralizing dentin deeper than the infiltration of bonding resin. The incompletely sealed interface may facilitate acid penetration and give rise to demineralization below the hybrid layer. For enamel bonding substrate, we first reported[23] the formation of ABRZ with a two-step SEA system, Clearfil SE Bond, which contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP) as the acidic functional monomer. However, it has not been investigated whether there is an ABRZ when enamel is etched with aggressive PA. Concerns emerge regarding the morphology of enamel ABRZ using the combination of PA and SEA.

Based on these considerations, the present study was undertaken to examine the effect of PA etching on the bond strength and ABRZ formation of a two-step SEA to enamel. The null hypotheses tested were: 1) PA etching would not affect the bonding performance of the two-step SEA to enamel; 2) PA etching would not influence the formation and morphology of ABRZ when enamel was treated with SEA.

Table 1: *Composition and Instructions of the Adhesive Systems According to the Manufacturers*

Materials	Manufacturer		Composition	Directions
Single Bond (etch and rinse)	3M-ESPE, St Paul, MN, USA	Etchant pH = 0.7	35% phosphoric acid	Apply etchant for 15 s; water rinse and air dry; apply two coats of adhesive; blow gently for 5 s; light cure 10 s.
		Adhesive	Bis-GMA, HEMA, polyalkenoic acid copolymer, water, ethanol, dimethacrylates	
Clearfil SE Bond (self-etch)	Kuraray Medical, Tokyo, Japan	Primer pH = 20	MDP, water, HEMA, hydrophilic dimethacrylate, camphorquinone, N,N-diethanol p-toluidine	Apply primer and leave for 20 s; moderate air dry for 10 s; apply bond and leave for 10 s; gently air blow for 10 s; light cure 10 s.
		Adhesive	MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, N,N-diethanol p-toluidine, silanated colloidal silica	

Abbreviations: Bis-GMA(bisphenol-A glycidyldimethacrylate), *HEMA(*hydroxyethyl methylacrylate*), MDP (*10-methacryloxydecyl dihydrogen phosphate*).*

MATERIALS AND METHODS

Twenty noncarious human third molars and 30 human premolars were collected after the individuals' informed consent was obtained under a protocol approved by the Institutional Review Board of Tokyo Medical and Dental University. The teeth were stored in water at 4°C and used within one month after extraction.

Two adhesive systems were used in this study (Table 1): an ERA system Single Bond (3M ESPE, St Paul, MN, USA) and a two-step SEA system Clearfil SE Bond (Kuraray Medical, Tokyo, Japan). For PA etching, K-etchant gel (35% PA, Kuraray Medical) was used. A conventional tungsten-halogen light-curing unit (XL3000, 3M-ESPE) at 600 mW/cm^2 power density was used for the light curing of specimens.

Microshear Bond Test

A 2-mm-thick cuspal enamel slice was sectioned from each molar using a low-speed diamond saw (Buehler Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA). Flat enamel surfaces were created by wet-grinding of the slices with 240-grit SiC paper (Buehler Ecomet V, Buehler Ltd). In order to standardize the smear layer, the ground enamel surfaces were wet-polished with 600-grit SiC paper for one minute.

The slices were randomly divided into four groups according to adhesive treatment as follows (Table 2): 1) Single Bond (SB); 2) Clearfil SE Bond (SE); 3) PA etching and rinsing, followed by Clearfil SE Bond primer and adhesive (PA/SEp+a); and 4) PA etching and rinsing, directly followed by Clearfil SE Bond adhesive (PA/SEa). Prior to the adhesive resin polymerization, a tygon tube (R-3603, Norton Performance Plastic Co, Cleveland, OH, USA) with an internal diameter of 0.8 mm and a height of 0.5 mm was placed on the bonded area. After the adhesive was light cured, a hybrid resin composite Clearfil AP-X (shade A3, Kuraray Medical) was injected into the tube and polymerized for 40 seconds. All bonded specimens were stored in distilled water at 37°C for 24 hours before testing.

After the storage period, the tygon tube was removed to reveal a composite cylinder with a cross-sectional area of 0.5 ± 0.02 mm^2. The specimens were examined under a stereomicroscope (Olympus, Tokyo, Japan) at 20× magnification to exclude specimens with any detectable interfacial defects. Each specimen was attached to a jig (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) using a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA) and placed in a universal testing machine (EZ-Test-500N, Shimadzu, Kyoto, Japan). A shear force was applied to each specimen using a thin wire loop at a crosshead speed of 1.0 mm/min until failure occurred.

The mean bond strengths were calculated then statistically analyzed using one-way analysis of variance (ANOVA) and the Tukey honestly significant difference test at the significance level of $\alpha = 0.05$ (n=10). The mode of failure was determined light-microscopically at 20× magnification using a

Table 2: *Application Procedures of the Groups Tested*

Groups	Code	Application Procedures
Single Bond	SB	As recommended by manufacturer
Clearfil SE Bond	SE	As recommended by manufacturer
PA etching/Clearfil SE Bond primer/Clearfil SE Bond adhesive	PA/SEp+a	1 Etch enamel with 35% PA for 15 s, water rinse, and air dry;
		2 Apply Clearfil SE Bond primer and leave for 20 s, moderate air dry for 10 s;
		3 Apply Clearfil SE Bond adhesive and leave for 10 s, gently air blow for 10 s, light cure for 10 s.
PA etching/Clearfil SE Bond adhesive	PA/SEa	1 Etch enamel with 35% PA for 15 s, water rinse and air dry;
		2 Apply Clearfil SE Bond adhesive and leave for 10 s, gently air blow for 10 s, light cure for 10 s.

stereomicroscope. Failure modes were categorized as three types: (A) adhesive failure; (C) cohesive failure; and (AC) mixed failure. The distribution of failure mode was analyzed by Pearson χ^2 test at a significance level of $\alpha = 0.05$.

Scanning Electron Microscope Observation of the Enamel Surface After Etching

Buccal enamel surfaces of six premolars were wet-polished with 600-grit SiC paper until a flat surface was reached. They were randomly divided into three groups, conditioned with the following procedures, respectively: 1) 35% PA for 15 seconds, rinsing; 2) Clearfil SE Bond primer for 20 seconds; (3) 35% PA for 15 seconds, rinsing, followed by Clearfil SE Bond primer for 20 seconds. For the latter two groups, the treated surface was immediately rinsed with an ascending series of ethanol (30%, 50%, 70%, and 95%) for one minute each and further cleaned ultrasonically in absolute acetone for one minute in order to completely dissolve the primer and dehydrate the specimens for scanning electron microscope (SEM) observation. After air drying, samples were coated with gold for SEM observation of the surface texture and etching pattern.

Preparation and Observation of the ABRZ

Sample preparation and observation for enamel ABRZ were performed according to our previous study.[23] Crowns of premolars were sectioned mesiodistally. Crowns with buccal surfaces facing out were embedded in epoxy resin (Epoxicure Resin, Buehler). After 24 hours, they were wet-polished with 600-grit SiC paper until flat midcoronal buccal enamel surfaces were exposed. Twenty-four of the prepared enamel discs were randomly divided into four groups. For each group, the enamel surfaces were treated according to the procedures mentioned previously (Table 2). A flowable composite Metafil Flow (shade A3, Sun Medical, Moriyama, Japan) was then applied on the top and cured for 20 seconds.

After storage in distilled water at 37°C for 24 hours, each specimen was sectioned perpendicular to the bonding interface into two halves and embedded in the epoxy resin overnight. They were wet-polished with SiC paper to 1200-grit to standardize the surface, and then subjected to an acid-base challenge to create artificial secondary caries, according to the conditions of our previous study.[23] Super Bond C&B (Sun Medical) was applied to the treated surface without acid etching, to prevent wear of the interface. Then the specimens were cut perpendicular to the interface, polished with diamond pastes and argon-ion etched in order. Following gold-sputter coating, the interface was observed using SEM (JSM- 5310LV, JEOL, Tokyo, Japan).

RESULTS

Microshear Bond Strength

The microshear bond strength values and failure modes are shown in Table 3. The SE group showed significantly lower bond strength compared with the other three groups (one-way ANOVA, $p<0.05$). For groups SB, PA/SEp+a, and PA/SEa, the bond

Table 3: *Bond Strength and Failure Mode of the Four Groups (*n = *10)*

Group	Microshear Bond Strength (MPa)	Failure Mode[a]		
		A	C	AC
SB	59.61 (4.50) b[b]	0	6	4
SE	43.08 (3.73) a	2	2	6
PA/SEp+a	60.13 (6.06) b	2	4	4
PA/SEa	57.74 (5.21) b	0	5	5

Abbreviations: A, adhesive failure; C, cohesive failure in enamel or composite; AC, mixed failure.
[a] There was no significant difference in the distribution of failure modes (χ^2 test, $p>0.05$).
[b] Groups having similar letters are not significantly different (one-way ANOVA).

strength showed no significant difference ($p>0.05$). The distribution of failure mode among groups was not significantly different by the Pearson χ^2 test ($p>0.05$).

SEM Observation of Enamel Etching Pattern

Enamel etching patterns are shown in Figures 1–3. With PA etching (Figure 1), the enamel prism cores were preferentially attacked, with simultaneous conservation of the marginal area. The spatial repetition of these regular patterns created a multitude of deep pits, forming a honeycomb structure. With SE primer treatment (Figure 2), the surface exhibited a very shallow and nonuniform etching pattern. The enamel prism core was slightly dissolved. Unetched patches and polishing scratches could be clearly seen on the surface. When the enamel surface was etched with PA, followed by SE primer treatment, preferential dissolution of prism boundaries instead of the prism core was observed (Figure 3).

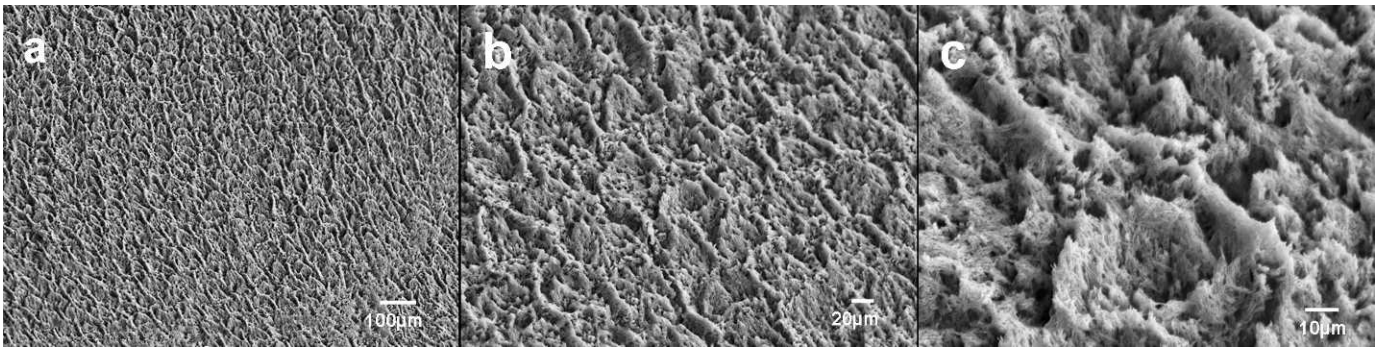

Figure 1. *SEM observation of the enamel etching pattern after 35% PA treatment. (a) Spatial repetition of regular patterns created a multitude of deep pits, forming a honeycomb structure. (b) Enamel prism cores were preferentially attacked, with simultaneous conservation of the marginal area. (c) Widely spaced crystallites were observed, forming a highly porous surface.*

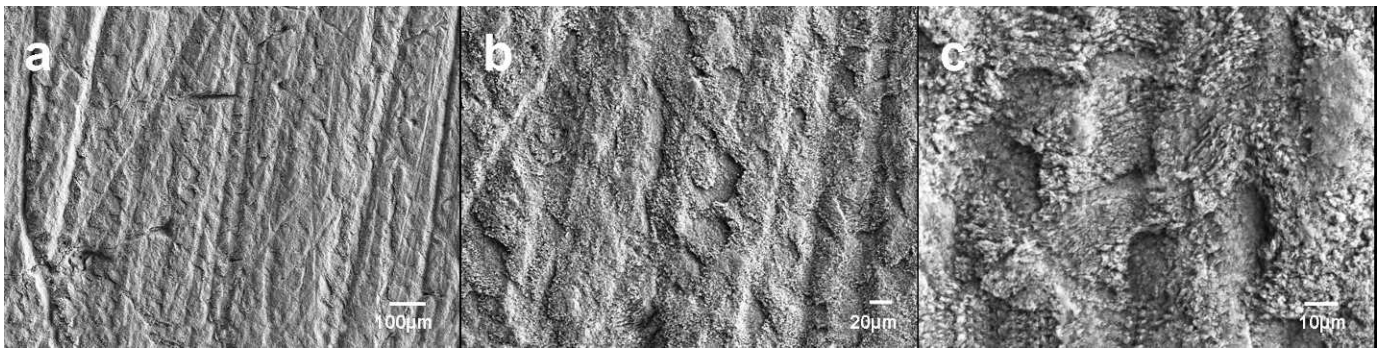

Figure 2. *SEM observation of the enamel etching pattern after SE primer treatment. (a) The surface exhibited a shallow and nonuniform etching pattern. Unetched patches and polishing scratches could be seen clearly. (b) Predominant dissolution of enamel prism core was observed. (c) Densely arranged crystallites were identified on the surface.*

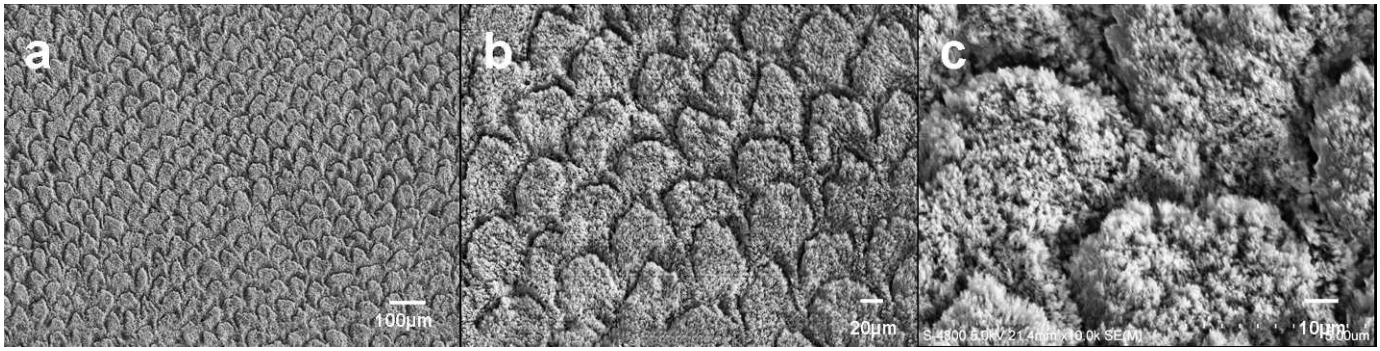

Figure 3. *SEM observation of the enamel etching pattern of PA/SEa group. (a) Spatial repetition of regular patterns created a multitude of deep grooves on the entire surface, forming a fish-scale structure. (b) Preferential dissolution of prism boundaries was observed. (c) Crystallites could be identified on the surface.*

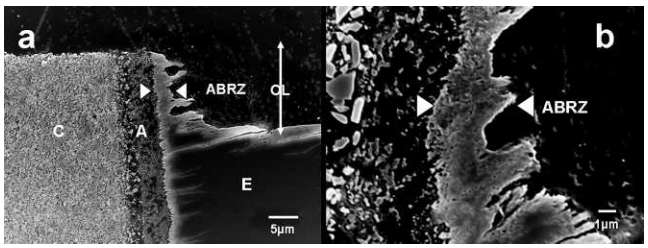

Figure 4. *Morphology of enamel-adhesive interface after acid-base challenge in SB group. OL, outer lesion; C, resin composite; A, adhesive layer; E, enamel; ABRZ, acid-base resistant zone. The ABRZ was pointed between triangles. The edge of the ABRZ toward the top of the OL showed an irregular and wavelike shape. Thickness of the zone was approximately 3 μm.*

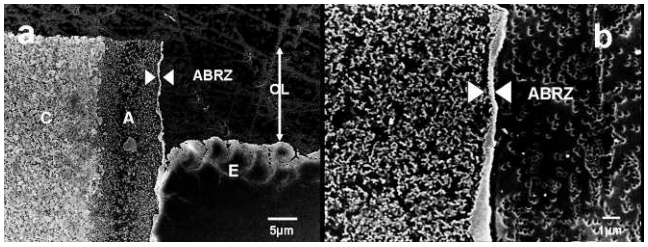

Figure 5. *Morphology of the enamel-adhesive interface after acid-base challenge in the SE group. The ABRZ was pointed between triangles. The edge of the ABRZ was relatively regular. Thickness of the zone was about 0.5 μm.*

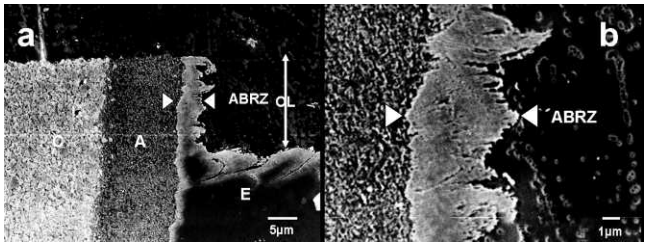

Figure 6. *Morphology of enamel-adhesive interface after acid-base challenge in PA/SEp+a group. The ABRZ was pointed between triangles. The edge of the ABRZ showed an irregular and wavelike shape, similar to that of SB. However, thickness of the zone was increased approximately 5 μm.*

SEM Observation of ABRZ Morphology

SEM observation of the enamel-adhesive interface after the acid-base challenge is shown in Figures 4–7. The top surface of the composite and adhesive was not remodeled in all specimens. An outer lesion (OL), defined as the mineral loss due to the acid-base challenge, ranged from 10 to 15 μm; and an electron-dense zone along the bonding interface beneath the adhesive resin at the OL front, coincident with enamel ABRZ, was observed in all groups.

The ABRZ in the SE group was the thinnest (Figure 5), about 0.5 μm thick. The edge of this zone toward the overlying OL was relatively regular. Under a higher magnification (60,000×), the zone consisted of densely arranged grainlike crystals (Figure 8b). For the other three groups (SB, PA/SEp+a, and PA/SEa) (Figures 4, 6, and 7), the edge of the ABRZ toward the top of the OL showed an irregular and wavelike shape. The thickness of the ABRZ was obviously increased and reached approximately 3, 5, and 5 μm at the wave trough for SB, PA/SEp+a, and PA/SEa, respectively. Examination under 30,000× magnification revealed that the ABRZ consisted of densely arranged crystals (Figure 8a).

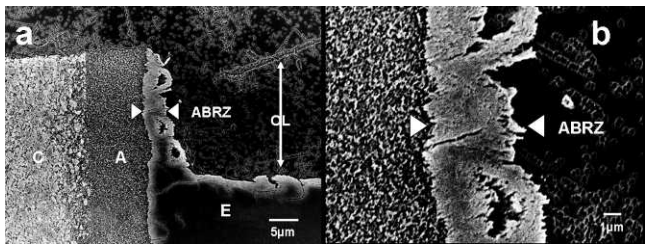

Figure 7. *Morphology of the enamel-adhesive interface after acid-base challenge in the PA/SEa group. The ABRZ was pointed between triangles. ABRZ morphology was similar to that of the PA/SEp+a group.*

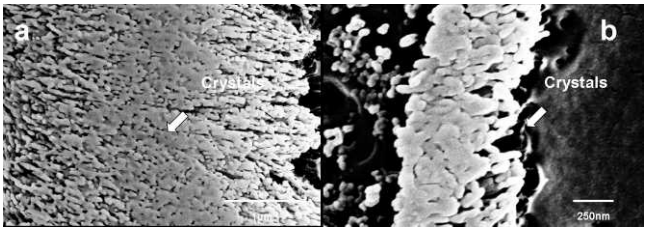

Figure 8. *Ultrastructure of the ABRZ under high magnification. (a) SB group (30,000×); (b) SE group (60,000×). It revealed that the ABRZ consisted of densely arranged crystals.*

DISCUSSION

PA etching is recommended by some manufacturers to improve the bonding performance of SEA systems to enamel. However, after enamel is etched with PA, the resistance of the bonding interface to mechanical and chemical challenges has not been fully elucidated. From this perspective, microshear bond strength and ABRZ formation of the enamel bonding interface were investigated in the present study. Two different adhesive systems were selected: an ERA system SB and an SEA system SE. SB is a two-step ERA system that requires pre-etching with 35% PA and water rinsing to remove the smear layer and demineralize the underlying substrates. SE is a two-step self-etching primer adhesive system. An acidic functional monomer, MDP, is present in the primer, which demineralizes the smear layer and underlying substrates simultaneously. Moreover, with this system, the formation of the ABRZ along the enamel bonding interface had been confirmed in our previous research.

In this study, the SE group showed lower bond strength than the PA-etched groups, which is in agreement with several other studies.[14,15] The most plausible explanation might be related to the different etching capacities of SE primer and 35% PA. Compared with the mild etching pattern of the SE primer, PA etching promotes the porosity of the demineralized enamel, resulting in increased resin interlocking and micromechanical retention. In spite of the weak correlation between enamel etching depth and bond strength found in the literature, the particular etching pattern achieved by 35% PA was believed to play an important role in enamel bonding. Erickson and others[14] showed that when enamel is etched with PA, the following adhesive resin penetration is fairly extensive, the structure is quite three dimensional, and the transition from resin to sound enamel is extended over a few microns. Such an interface may be more resistant to crack propagation than the relatively planar interface obtained with SEA systems. However, it should also be noted that only crosscut enamel was evaluated in the current study. Shimada and others reported[29] that, although the bond strength of SB to crosscut enamel was significantly higher than that of SE, its bonding performance was drastically influenced by the enamel prism orientation. On the contrary, the enamel bonding of SE is stable for any orientation of enamel. They suggested that PA etching was too aggressive for the parallel-cut enamel. Therefore, the results in this study may not be extrapolated to an experimental setup with a different enamel prism orientation.

Because PA etching could increase the bond strength of SEA to enamel, some researchers[21] recommended extending a two-step SEA into a three-step adhesive. However, as far as this study was concerned, after enamel was etched with PA (groups PA/SEp+a and PA/SEa), whether a SE primer was applied or not, neither the bond strength nor the ABRZ morphology were affected. The primary functions of primer in two-step SEA systems include two aspects.[6] One is to condition the surface of bonding substrates, creating spaces for resin penetration; the other is to prime the demineralized layer, promoting the infiltration of hydrophobic monomers into tooth tissue. Etching with PA could substitute for the former function of self-etching primer. Because enamel contains a very small amount of water and organics, the hydrophobic resin monomers could deeply and completely infiltrate into the demineralized layer without primer application. In addition, it was suggested that the bonding agent alone may have better mechanical properties compared with the mixture of the water-containing primer and the bonding agent, which may affect the durability of the bond.[30] However, whether eliminating the step of a self-etching primer on etched enamel is effective for the other two-step SEA systems should be further investigated.

The dentin ABRZ was confirmed with SEA systems only. In previous studies, [24,25,28] when dentin was treated with SB, no ABRZ could be distinguished. Researchers presumed that it might be related to the aggressive etching capacity of PA in SB. After PA etching and rinsing, the dentin surface gets completely demineralized to a certain depth. However, due to the hydrophilic property of bonding resin and the existence of water in dentin, the bottom of the demineralized dentin could not be completely infiltrated by hydrophobic resin, creating a weak collagenous band at the base of the hybrid layer. During the acid-base challenge, the weak area

is vulnerable to acid-base attack, promoting the demineralization of dentin below the hybrid layer. On the contrary, in the present study, when enamel was treated by PA (groups SB, PA/SEp+a, and PA/SEa), the ABRZ could be clearly detected beneath the bonding interface. As is known, enamel has a higher mineral content with a matrix structure different from a dentin collagen network. Even after etching with a strong acid like PA, what remains is not a collagen matrix but rather a mineral framework. Hydroxyapatite (HAp) crystals are distributed all over the demineralized layer. These crystals either may be directly wrapped and protected by polymerized resin components or may chemically interact with specific functional monomers of the adhesive systems, producing insoluble calcium salts. As a result, a layer resistant against an acid-base attack forms on the PA-etched enamel bonding interface.

In the SE group, the ABRZ was the thinnest, and the edge of the ABRZ toward OL showed a relatively regular shape. This may be explained by the mild etching pattern produced by the SE primer. As shown in SEM micrographs, the SE primer etched enamel moderately, leaving shallow prismatic depressions and some intact unetched areas. When the bonding resin was applied to the poorly etched surface, monomers penetrated superficially. On the other hand, when enamel was PA etched, the thickness of the ABRZ was obviously increased, and its edge showed a wavelike shape. SEM examination confirmed that PA produced an aggressive etching pattern. The resin monomers could penetrate deeper into the demineralized layer, resulting in thicker ABRZ formation.

In the three PA-etched groups (SB, PA/SEp+a, and PA/SEa), the ABRZ of group SB was the thinnest. As confirmed by our former work,[23] acidic functional monomers in adhesive systems may play an important role in the formation and morphology of the ABRZ. The functional monomer contained in SE is MDP, which has been found to chemically interact with HAp intensively and stably, forming MDP-calcium salt with a low solubility. For the SB group, polyalkenoic acid copolymer was used. The relatively larger molecular size may prevent the deep penetration of resin monomers into a demineralized layer, partially contributing to the thinner ABRZ morphology. Furthermore, the chemical bonding capacity of polyalkenoic acid copolymer with HAp has been shown.[31] The carboxyl groups of the polyalkenoic acid can replace phosphate ions of the substrate and make ionic bonds with calcium ions of HAp. This mechanism is similar to 4-MET, for which the chemical interaction capacity with HAp has proved to be inferior to that of the MDP.[3,9] Therefore, it is reasonable to assume that the crystals in the SB group are less protected and the resulting polyalkenoic acid copolymer-calcium salts are prone to faster dissolution during the acid-base challenge. Further chemical analysis is necessary to prove these speculations.

CONCLUSION

In the present study, the null hypotheses were both rejected. With 35% PA etching, the bond strength of SEA system SE to enamel was significantly increased, comparable with the EAR system SB. Also, the formation of the ABRZ on enamel was promoted. Taking both bond strength and ABRZ formation into consideration, we recommend bonding enamel by the means of directly applying SE adhesive after 35% PA etching. Further studies are necessary to evaluate the effect of PA etching on one-step SEA systems to enamel.

Acknowledgments

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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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Effect of Surface Treatments on Microtensile Bond Strength of Repaired Aged Silorane Resin Composite

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Clinical Relevance

To obtain high repair bond strength on aged silorane composite, aluminum oxide sandblasting should be used as a surface treatment procedure. After aluminum oxide sandblasting, either silorane composite with the LS system adhesive or methacrylate composite with a methacrylate dental adhesive can be used.

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SUMMARY

Objective: This laboratory study compared the repaired microtensile bond strengths of aged silorane resin composite using different surface treatments and either silorane or methacrylate resin composite. Methods: One hundred eight silorane resin composite blocks (Filtek LS) were fabricated and aged by thermocycling between 8°C and 48°C (5000 cycles). A control (solid resin composite) and four surface treatment groups (no treatment, acid treatment, aluminum oxide sandblasting, and diamond bur abrasion) were tested (N=12 blocks, 108 beams/group). Each treatment group was randomly divided in half and repaired with either silorane resin composite (LS adhesive) or methacrylate resin composite (Filtek Z250/Single Bond Plus). After 24 hours in 37°C distilled water, microtensile bond

strength testing was performed using a non-trimming technique. Surface topography after surface treatment was analyzed using scanning electron microscopy (SEM). Failure mode was examined using optical microscopy (50×). Results: Weibull-distribution survival analysis revealed that aluminum oxide sandblasting followed by silorane or methacrylate resin composite and acid treatment with methacrylate resin composite provided insignificant differences from the control ($p>0.05$). All other groups were significantly lower than the control. Failure was primarily adhesive in all groups. Conclusion: Aluminum oxide sandblasting produced microtensile bond strength not different from the cohesive strength of silorane resin composite. After aluminum oxide sandblasting, aged silorane resin composite can be repaired with either silorane resin composite with LS system adhesive or methacrylate resin composite with methacrylate dental adhesive.

INTRODUCTION

One of the main disadvantages of methacrylate composite is polymerization shrinkage. There have been several attempts to overcome polymerization shrinkage such as using an incremental layering technique, placing a stress-absorbing liner, and changing the light-curing procedures.[1,2] Recently, a silorane-based material was introduced. Silorane composite has shown approximately 0.94 vol% to 1.5 vol% shrinkage[3–5] and comparable flexural strength to methacrylate composite.[3] A silorane molecule comes from the reaction of oxirane and siloxane molecules.[3,6,7] Whereas the polymerization process of methacrylate composite occurs via free radical polymerization,[8,9] the polymerization of silorane composite is generated by the cationic ring-opening polymerization of the oxirane molecule. The silorane composite has two main advantages: low polymerization shrinkage due to the ring-opening polymerization of the oxirane monomer and increased hydrophobicity because of the siloxane molecule.[10]

Silorane composite consists of four main components.[3,7] The filler is a combination of fine quartz particles and radiopaque yttrium fluoride. The polymer matrix is silorane, and the photoinitiator is camphorquinone and iodonium salt. The use of fine particular quartz contributes to good esthetic performance and mechanical stability. The quartz surface is modified with a silane layer to increase the hydrophobic character of the surface of the filler and act as the interface between filler and matrix, facilitating the reinforcement of the resin.

The average lifetime of composite restorations is 5.5 years to eight years, with an average annual failure rate of 2.2%.[11,12] Recurrent caries and discoloration are the main reasons for replacement in general dental practice.[13] Replacement frequently involves the removal of additional tooth structure to create new enamel bonding, leading to a larger restoration with further loss of tooth structure.[14]

Several methods of surface treatment have been widely used to establish adequate bond strength between aged and new layers of composite, including surface hydrofluoric acid etching, sandblasting with aluminum oxide particles, abrasion with a diamond bur followed by silica coating, and the use of intermediate bonding agents.[2,10,14–17] The bonding of new to aged composite mostly depends on micromechanical interaction, but chemical bonding is also to be taken into consideration.[18–20]

In a clinical situation, there may be no information about the chemical composition of the existing resin composite restorations. Understanding how to repair existing silorane restorations is critical because there is limited information regarding repair protocols of silorane composite. Based on this concern, the purpose of this study was to compare the repaired microtensile bond strength of aged silorane composite using different surface treatments and either silorane or methacrylate composite.

MATERIALS AND METHODS

One hundred eight silorane composite blocks (Filtek LS, shade A2, 3M ESPE, St Paul, MN, USA) were fabricated using a silicone mold (6 mm × 6 mm × 12 mm for the control and 6 mm × 6 mm × 6 mm for the test specimens). Each 2-mm-thick increment was placed using a plastic instrument and was cured for 40 seconds using a Demetron LC curing unit (Kerr, Orange, CA, USA) with an intensity of 600 mW/cm^2. The intensity of the LED curing light was monitored with a Cure Rite Visible Curing Light Meter (Dentsply, York, PA, USA). The last increment was covered with a Mylar strip to obtain a flat surface and to aid in removal of excess material. All specimens were polished using 320, 400, and 600 silicon carbide paper including the top surface in order to remove the excess and to make the surface perpendicular to the specimen's long axis. All specimens were cleaned in tap water for 10 minutes in an ultrasonic device to remove loose particles and stored in distilled water for 24 hours.

All specimens were aged by thermocycling (5000 cycles, 8°C to 48°C, dwell time of 30 seconds, transfer time of 10 seconds). All tested specimens were randomly divided into four surface treatment groups, and each group was divided into two subgroups (12 blocks for each subgroup) to repair with either silorane (group 1S, 2S, 3S, 4S) or methacrylate composite (group 1M, 2M, 3M, 4M; Table 1). The surface treatment procedure was performed as described below:

Group 1 (No Treatment)—No further treatment was performed.

Group 2 (Acid Treatment)—The aged specimen surfaces were etched with 35% phosphoric acid gel (Scotchbond Etchant, 3M ESPE) for 15 seconds for the group that was repaired with methacrylate composite (group 2M). The aged specimen surfaces were etched with LS System self-etch primer (LS System adhesive, 3M ESPE) for 15 seconds for the group that was repaired with silorane composite (group 2S).

Group 3 (Aluminum Oxide Sandblasting)—The resin composite surface was abraded for 10 seconds with an intraoral air abrasion unit (Microetcher II, Danville Engineering INC, San Ramon, CA, USA) from a distance of approximately 10 mm and perpendicular to the composite block using 50-μm aluminum oxide particles (Danville Engineering INC) with an air pressure of 60 psi.

Group 4 (Diamond Bur Abrasion)—The resin composite surface was roughened with a coarse-grit diamond bur for 10 seconds (No. 027, Brasseler, Savannah, GA, USA). A high-speed handpiece with a water spray was used. Before the roughening procedure, the operator was trained on the surface of an analytical balance (AE 100, Mettler-Toledo Inc, Columbus, OH, USA) to be able to replicate the manual pressure (approximately 4.0 ± 1.0 g)[21] that was placed on the composite surface.

Then, all specimens were rinsed with tap water, and excess water was removed with canned compressed oil-free air (Falcon Dust Off Air Duster, Branchburg, NJ, USA) and repaired with either silorane (Filtek LS, shade C2, with LS System adhesive) or methacrylate composite (Filtek Z250, shade A4, with Adper Single Bond Plus adhesive agent, 3M ESPE) according to the manufacturer's recommendation (as described below) using an incremental technique with a silicone mold (Table 2).

LS System Adhesive

After the surface treatment procedure, the LS System adhesive self-etch primer was applied on

Table 1: *Materials, Batch Number, and General Compositions*

Materials	Batch No.	General Composition
Filtek LS (3M ESPE)	N18197	*Filler:* Silanized quartz, yttrium fluoride 76 wt%
		Matrix: 3,4-Epoxycyclohexylethyl-cyclopolymethylsiloxane, Bis-3,4-epoxycyclohexylethylphenylmethyl-silane
Filtek Z250 (3M ESPE)	N163688	*Filler:* Zirconia/silica 85 wt%
		Matrix: Bis-GMA, a blend of UDMA and Bis-EMA
LS adhesive (3M ESPE)	N157377	*Self-etch primer:* Phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, camphorquinone and silane-treated silica filler, initiators, stabilizers
		Bond: Hydrophobic methacrylate, phosphorylated methacrylate, TEGDMA, silane-treated silica filler, initiators, stabilizers, camphorquinone
Adper Single Bond Plus adhesive (3M ESPE)	393173	*Etchant:* 35% phosphoric acid
		Bond: Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator system, methacrylate functional copolymer of polyacrylic and polyitaconic acids, silica particles

Abbreviations: Bis-GMA, bisphenol A diglycidyl ether diemthacrylate; EMA, bisphenol A polyethylene glycol diether dimethacrylate; HEMA, 2-hydroxyethyl methacrylate, TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Table 2: *Experimental Groups and Surface Treatment Protocol*

Group	Repair Materials	Surface Treatment	Bonding Procedure
Control	—	—	—
1S	Filtek LS	No surface treatment	LS adhesive bonding
1M	Filtek Z250	No surface treatment	Single bond application
2S	Filtek LS	LS self-etch primer	LS adhesive bonding
2M	Filtek Z250	35% Phosphoric acid etching	Single bond application
3S	Filtek LS	Aluminum oxide sandblasting	LS self-etch primer + LS adhesive bonding
3M	Filtek Z250	Aluminum oxide sandblasting	35% phosphoric acid + single bond
4S	Filtek LS	Abrasion with diamond bur	LS self-etch primer + LS adhesive bonding
4M	Filtek Z250	Abrasion with diamond bur	35% phosphoric acid + single ond

the surface-treated resin composite for 15 seconds except for the no surface treatment group and the acid treatment group because the acid treatment group was previously etched from the surface treatment. Then, all etched specimens were gently dried with canned compressed oil-free air and were cured for 10 seconds. Then, LS adhesive bonding was applied on the surface of all composite surfaces, followed by gentle air drying and 10 seconds of light cure.

Adper Single Bond Plus System Adhesive

After the surface treatment procedure, 35% phosphoric acid gel (ScotchbondEtchant, 3M ESPE) was applied to the treated composite surfaces for 15 seconds except for the no surface treatment group and the acid treatment group because the acid treatment group was previously etched from the surface treatment. Then, the etched resin composites were rinsed for 10 seconds. Excess water was removed with canned compressed oil-free air. Then, Adper Single Bond Plus adhesive agent (3M ESPE) was applied in three consecutive coats for 15 seconds on all composite surfaces with gentle agitation using a fully saturated applicator, followed by air drying for five seconds to evaporate the solvent and 10 seconds of light cure.

Then, a new layer of resin composite was applied to the aged silorane composite using an incremental technique with the aid of a silicone mold. Each 2-mm-thick increment was placed using a plastic instrument and was cured for 40 seconds. The last increment was covered with a Mylar strip to obtain a flat surface and to aid in removal of excess material.

All repaired resin composite blocks were stored in 37°C distilled water for 24 hours. Then, the composite blocks were cut using a slow-speed water-cooled saw equipped with a diamond-impregnated disk (Isomet, Buehler, Lake Bluff, IL, USA) at a speed of 300 rpm, producing nine beams per block with an average area of 0.64 mm^2 (0.8 mm $\times$ 0.8 mm) for each beam. The beams located at the periphery of block were discarded.

Microtensile Bond Strength Testing

After storing in 37°C distilled water for 24 hours, the beams were placed in a notched Geraldeli jig (ODEME Biotechnology, Joacaba, SC, Brazil) for microtensile testing using cyanoacrylate resin (Loctite Superglue Control Gel, Henkel Corporation, Avon, OH, USA). All beams were loaded in tension in a universal testing machine (MTS Sintech Renew 1123, Eden Prairie, MN, USA) until fracture at a crosshead speed of 1 mm/min.

Statistical Analysis

Comparisons between the groups for differences in microtensile bond strength were performed using a

Weibull-distribution survival analysis at the 0.05 significance level.

Scanning Electron Microscopy (SEM) Analysis

Additional specimens were fabricated, aged, treated, and prepared for SEM (model JSM-5310LV, JEOL, Peabody, MA, USA). Specimens were sputter coated with gold to a thickness of approximately 200 A° in a vacuum cold sputter (Denton Vacuum Desk II cold sputter, Moorestown, NJ, USA). Micrographs were taken at 2000× and 10,000× magnification to evaluate the surface topography created by the different surface treatments.

Failure Analysis

Fracture surfaces of the repaired groups were examined using optical microscopy at 50× magnification. The type of failure was determined to be either adhesive failure (between aged and repairing composites involving the intermediate layer), cohesive failure (within the aged or repairing composite), or mixed (combination of adhesive and cohesive failures).

RESULTS

Microtensile Bond Strength

Aluminum oxide sandblasting followed by silorane or methacrylate-based resin composite (group 3S, 3M) and acid treatment with methacrylate resin composite (group 2M) provided insignificant differences from the control ($p>0.05$; Table 3). All other groups were significantly lower than the control, and no other significant difference was found among other groups.

Type of Failure

As shown in Table 4, in most tested beams, fractures developed at the composite-composite interface (81%), followed by cohesive failure (18%) and mixed failure (1%), respectively.

SEM Analysis

SEM analysis revealed different surface topography (Figure 1) of surface-treated silorane composite. Al_2O_3 sandblasting (Figure 1G,H) produced substantial surface irregularities, which could be supported by the highest microtensile bond strength. Even though self-etch primer (Figure 1C,D) and phosphoric acid (Figure 1E,F) could remove the superficial portion of the aged composite surface with some filler particles exposed, the surface roughness of the acid treatment was less obvious. In addition, at 10,000× magnification, acid treatment with phosphoric acid (Figure 1F) showed more granular-like surface topography compared with the remaining groups. No surface treatment (Figure 1A,B) and diamond bur (Figure 1I,J) abrasion showed comparable surface topography with some irregularities resulting from polishing with silicon carbide paper, which could be supported by the comparable microtensile bond strength. Moreover, diamond bur abrasion (Figure 1J) also showed exposed filler particles, but no exposed filler was observed in the no-treatment group (Figure 1B).

Table 3: *Mean, Percentage of Cohesive Strength, and p* Value of Experimental Groups

Surface Treatment	Group	Mean Bond Strength, MPa	Percentage of Cohesive Strength	*p* Value
Control	Control	62.6 + 1.6	100	—
No treatment	1S	37.2 + 1.6	59.42	0.006*
	1M	40.8 + 1.9	65.18	0.030*
Acid etch	2S	37.3 + 2.0	59.58	0.008*
	2M	44.1 + 1.8	70.45	0.070
Sandblast	3S	50.2 + 1.6	80.19	0.230
	3M	47.8 + 1.5	76.36	0.130
Abrasion	4S	39.0 + 1.7	62.30	0.014*
	4M	37.7 + 1.6	60.22	0.007*

** Statistically significant difference ($p<0.05$) when compared with the control. There was no other statistically significant difference among other groups.*

DISCUSSION

Microtensile bond strength was chosen in the present study because the purpose of this experiment was to compare the repair bond strength that is at the interface between aged and a newly repaired composite. The bond strength values from microtensile bond strength testing are normally from adhesive failure because of a uniform stress distribution, so the bond strength value better represents the adhesive bond strength instead of

Table 4: *The Incidence of Failure Mode (%)*

Surface Treatment	Group	Type of Failure, %			
		Adhesive	Mixed	Cohesive	Did Not Break
No treatment	1S	87	0	13	0
	1M	77	0	22	1
Acid treatment	2S	80	1	19	0
	2M	82	0	18	0
Sandblast	3S	72	2	24	2
	3M	63	1	36	0
Abrasion	4S	94	0	6	0
	4M	88	6	6	0
Total		81	1	18	0

Abbreviations: M, the silorane composite (Filtek LS) was repaired with methacrylate composite (Filtek Z250); S, the silorane composite (Filtek LS) was repaired with silorane composite (Filtek LS).

cohesive strength from cohesive material failure. In this study, the microscopic study showed most failures resulted from adhesive failures. As approximately 81% of the bond strength values were associated with adhesive failure, it can be assumed that the bond strength values are highly representative of adhesive bond strength rather than cohesive bond strength.

The result of the present study is in accordance with other composite repair studies[10,14,17,21] supporting aluminum oxide sandblasting as an effective surface treatment procedure for the repair of composite restorations. Either silorane composite repaired with silorane composite (group 3S) or repaired with methacrylate composite (group 3M) showed no significant difference in repair bond strength compared with the cohesive strength of silorane composite (control). This finding indicates that aluminum oxide sandblasting provides comparable microtensile bond strength to the solid silorane specimens.

SEM images showed that aluminum oxide sandblasting provided substantial surface roughness compared with the other groups. Rodrigues and others and Costa and others explained that a high surface roughness resulting from sandblasting produced microretentive features, enhancing the surface area available for bonding.[10,21]

An interesting result was found in the no surface treatment group (group 1S, 1M). Even though the repair bond strength was significantly lower than the control, the repair bond strength in the no surface treatment group was approximately the same as or even higher (with no significant difference) than that of the diamond bur abrasion group. Both mechanical and chemical bonding might be considered as an explanation. First, polishing the top surface of the composite blocks with silicon carbide paper prior to the aging procedure produced surface roughness for further bonding. Furthermore, SEM analysis also supported the above hypothesis. SEM images showed that no surface treatment and diamond bur abrasion presented a similar pattern of surface roughness (Figure 1). Second, as described by Padipatvuthikul and Mair,[22] the surface of aged composite might contain microdefects, which can be penetrated by the unfilled resin of the bonding agent, resulting in a micromechanical retention. Third, the solvent in the adhesive bonding may soften the surface of aged composite, creating swelling and gelation of the surface and subsurface layers.[22] The monomer in the layer of the repair material might access and cross-polymerize with the unreacted functional group of silorane, creating a chemical bond. Fourth, the aging cycles may not have been sufficient to create hydrolytic degradation on the composite surface so that the repair bond strength might have come from the remaining reactivity of the material.

The use of diamond bur abrasion on the aged silorane composite resulted in significantly weaker repair bond strengths than the cohesive strength of silorane composite regardless of the repair material used. The effect of surface abrasion with a diamond bur on the repair bond strength of aged composite is debated because some studies did not show significantly improved repair bond strength[23] while others did.[24,25] Thus, this technique provides a less predictable outcome compared with aluminum oxide sandblasting.

Acid treatment, including phosphoric acid, normally shows no effect in repair bond strength.[13,26–28] In this study, it was interesting to see quite different results from the two acid etching subgroups. When repaired with Filtek Z250 (group 2M), the 35% phosphoric acid was able to produce microtensile

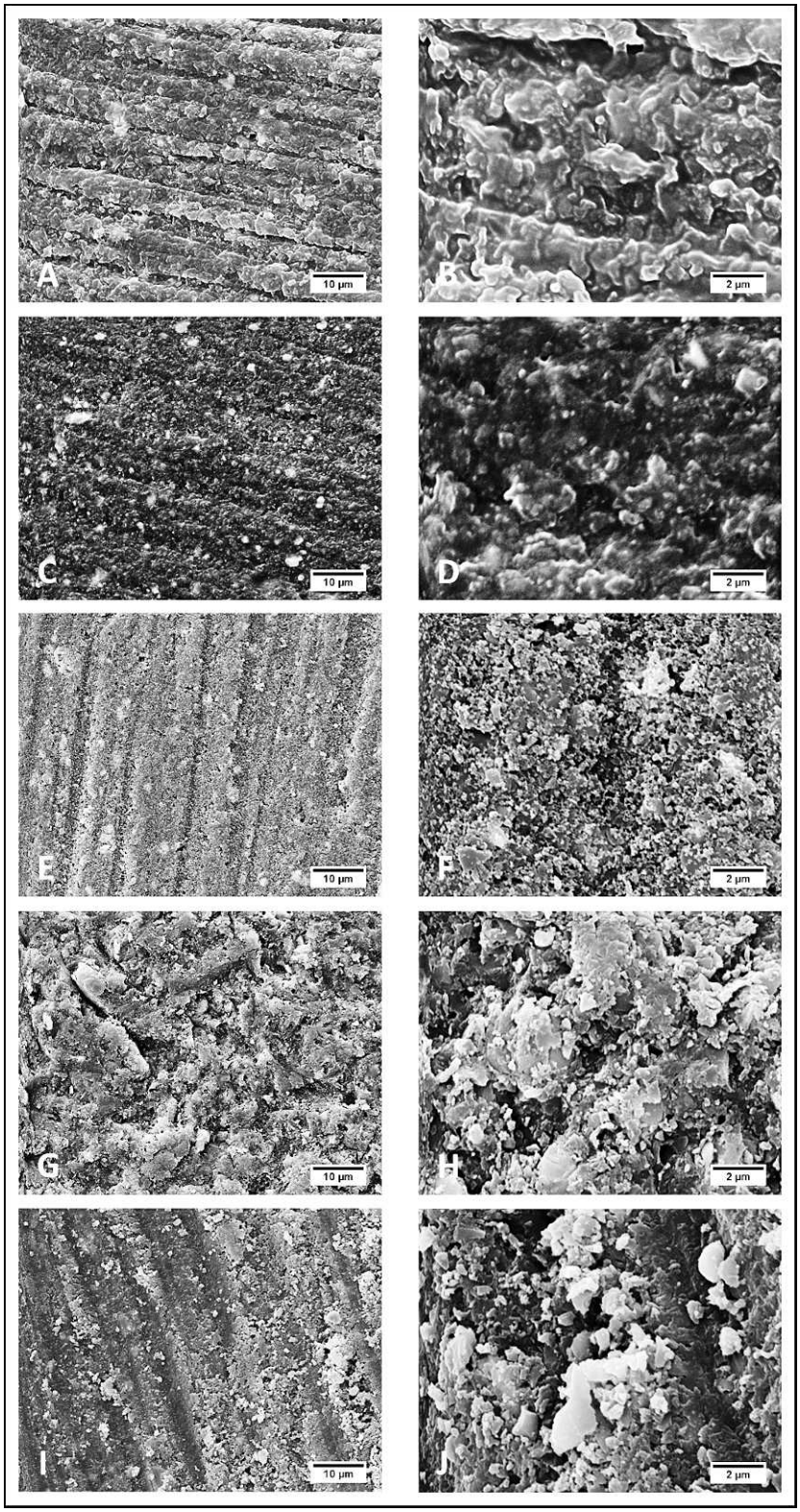

Figure 1. *The scanning electron micrographs of aged silorane composite treated with different surface treatments. (A): (2000×), (B): (10,000×): No surface treatment group; (C): (2000×), (D): (10,000×): self-etch primer; (E): (2,000×), (F): (10,000×): 35% phosphoric acid; (G): (2000×), (H): (10,000×): 50 µm Al_2O_3 sandblasting; (I): (2000X), (J): (10,000×): diamond bur abrasion. Scanning electron microscopy images showed the high surface irregularities on the silorane composite treated with aluminum oxide sandblasting. There were some exposed filler particles observed in the acid treatment groups (Figure 1C,E), Al_2O_3 sandblasting (Figure 1H), and diamond bur abrasion (Figure 1J).*

bond strength values comparable to the cohesive strength of the silorane composite. In the subgroup that was etched with LS self-etch primer and repaired with Filtek LS (group 2S), the repair bond strength was significantly lower than that of the cohesive strength of the material. Furthermore, results from subgroups 1M and 1S were interesting as well. Silorane composite repaired with methacrylate composite without surface treatment (group 1M) seemed to produce better microtensile bond strength than if silorane was used as the repair material (group 1S), even though values were not significantly different. Group 1M also resulted in higher numbers of cohesive failures, which suggested higher success in bonding of the two materials at their interfaces. From these results, methacrylate composite seems to be a better choice of repair material in the situation that an air abrasion unit is unavailable. Although the amount of bonding between methacrylate composite and the aged silorane composite without mechanical surface treatment is unknown, the chemical bonding may likely contribute to this increase in repair microtensile bond strength. In addition, with mechanically prepared surfaces, either abrasion with a diamond bur (group 4M, 4S) or abrasion with aluminum oxide air abrasion (group 3M, 3S), silorane composite seemed to work better than methacrylate composite with nominally higher repair bond strengths (even though there was no statistically significant difference). The chemical reaction between the methacrylate composite and the aged silorane composite must be further explored to explain these results.

When considering the repair material for aged silorane composite, the clinically sufficient repair bond strength is not known. It is well known that resin composite seldom fails mechanically at the junction with etched enamel. Therefore, it can be surmised that a repair microtensile bond strength that is similar to that of resin composite to etched enamel (33.8-55.6 MPa[29]) would be clinically adequate.[24] In this study, repair bond strength (37.2–50.2 MPa) was in the same range as the bond strength of resin composite to enamel. Moreover, the repair microtensile bond strength in group 3M and group 3S was comparable to the cohesive strength of the silorane resin composite. This might indicate that when using aluminum oxide sandblasting on the aged silorane composite, it can be repaired with either silorane composite with the LS system self-etch adhesive or a methacrylate composite using a methacrylate dental adhesive.

CONCLUSION

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

1. Aluminum oxide sandblasting produced microtensile bond strength comparable to the cohesive strength of silorane composite.
2. After aluminum oxide sandblasting, aged silorane composite can be repaired with either silorane composite with LS system adhesive or methacrylate composite with methacrylate dental adhesive.

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Effects of Two In-Office Bleaching Agents with Different pH on the Structure of Human Enamel: An *In Situ* and *In Vitro* Study

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Clinical Relevance

Salivary pellicle and adequate calcium and phosphate ions present in natural human saliva play pivotal roles in protecting the surface of human enamel during the process of in-office bleaching.

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SUMMARY

This study evaluated the effects of two in-office bleaching agents (Beyond and Opalescence Boost) with different pH on the structure and mechanical properties of human enamel *in vitro* and *in situ*. One hundred and eight enamel slabs were obtained from freshly extracted premolars. The specimens were randomly distributed into nine groups (n=12), and the human saliva (HS) in the volunteers' oral cavities was used to simulate the *in situ* condition: Beyond + HS, Opalescence Boost (O-Boost) + HS, Control + HS, Beyond + artificial saliva (AS), O-Boost + AS, Control + AS, Beyond + distilled water (DW), O-Boost + DW, and Control + DW. The bleaching treatments were performed on the first and eighth day,

and the total bleaching time was 90 minutes. Baseline and final surface roughness (RMS), surface morphology, microhardness, and fracture toughness (FT) were measured before the treatment and on the fifteenth day, respectively. Compared with control groups, surface alterations on enamel were found in the Beyond + AS and Beyond + DW groups under atomic force microscopy evaluation. Two-way analysis of variance and Tukey test revealed that the RMS showed significant intergroup differences for both storage condition and bleaching agent, whereas microhardness and FT revealed no significant alteration. The results indicated that in-office bleaching agents with low pH values could induce enamel morphology alterations under *in vitro* conditions. The presence of natural HS could eliminate the demineralization effect caused by low pH.

INTRODUCTION

Vital tooth bleaching has become a popular procedure for treating discolored teeth because of its simplicity and conservation property.[1] Currently, three fundamental approaches are adopted for vital tooth bleaching, which are at-home bleaching, in-office bleaching, and over-the-counter bleaching products.[2,3] Although at-home bleaching is effective and widely accepted, in-office bleaching still seems to be appropriate for the patient in some cases, such as severe or single tooth discoloration, lack of compliance, or desire for immediate whitening to recover confidence.[4]

The efficacy of in-office bleaching has been well documented, but a primary concern is that the enamel structure may be weakened by the bleaching agent.[5] Numerous studies were conducted to evaluate the safety of in-office bleaching agents on enamel. However, the results of these investigations usually conflict with each other. Some studies reported that in-office bleaching might have detrimental effects, such as alterations in surface morphology,[6,7] changes in chemical composition,[8,9] and a decrease in microhardness and fracture toughness (FT).[5,8,10,11] In contrast to these findings, there were also some investigations that held the opposite view to those observations above.[12,13] The great inconsistency in the outcome of those studies might be due to differences in study design[2,14] (type of storage condition, time of evaluation, different bleaching agents, time of application, bleaching agent pH, and so on). Among these factors, the pH of bleaching agents and storage conditions may have crucial influences on the results.

Generally, distinct bleaching agents have different pH values. Since acid bleaching agents may help to keep hydrogen peroxide stable and facilitate the bleaching process,[4] some in-office bleaching products containing highly concentrated hydrogen peroxide have a low pH. The possible adverse effects of in-office bleaching agents have been evaluated by previous studies. However, most of them focused on the concentration;[12,13,15,16] whether the pH value of in-office bleaching agents plays an important role in the bleaching treatment still remains unclear. In addition, storage condition is also an element that should be taken into account. Typically, studies on in-office bleaching use distilled water (DW) or artificial saliva (AS) as the storage condition. These models could partially influence the findings, but they had some limitations. Compared with *in vitro* studies, *in situ*/*in vivo* ones may better simulate clinical conditions and really reveal influences of bleaching agents on enamel.[2] Nevertheless, few investigations[11,13] about in-office bleaching have been done using *in situ*/*in vivo* methodologies, and none of them combined *in situ*/*in vivo* experiments with *in vitro* ones to compare the effects of different pH values and different storage conditions on human enamel.

Therefore, the primary purpose of the present study was to evaluate the effects of different pH values and different storage conditions on the structure and mechanical properties of dental enamel during the in-office bleaching process. This goal was achieved by the complementary use of atomic force microscopy (AFM), microhardness, and FT measurements. The null hypotheses in this study were that 1) the pH values of bleaching agents and storage conditions (*in vitro* and *in situ*) had no effect on the morphology alteration of human enamel during the in-office bleaching process and 2) the pH values of bleaching agents and storage condition (*in vitro* and *in situ*) had no effect on the mechanical properties of human enamel during the in-office bleaching process.

MATERIALS AND METHODS

Ethical Aspects and Volunteers

The protocol for this study was reviewed and approved by the Ethics Committee of the School and Hospital of Stomatology, Wuhan University. Four undergraduate dental students (2 men and 2 women, aged 20 to 22 years) who fulfilled the

inclusion criteria (absence of dental caries and/or periodontal disease, normal saliva flow, willing to perform bleaching treatment on the research schedule) without violating the exclusion criteria (restorations and prostheses in mouth, use of orthodontic appliances, dentin sensitivity, and smokers) were enrolled in the study after signing an informed consent form as volunteers.

Tooth Selection

The sample size was determined from the data of preliminary study in our research group. Assuming a standard type I error rate of α=0.05 and a standard type II error rate of "β=0.20 (power=0.80), a sample size of 12 enamel samples was calculated for each group to achieve more reliable results.

To achieve the calculated sample size, 54 freshly extracted orthodontic premolars were selected. All of them were examined under magnification (20×) to detect enamel cracks or fractures, carious, stains, and other defects. The teeth were cleaned thoroughly and stored in 0.2% thymol at 4°C until required.

Materials Preparation

The roots of stored teeth were separated from their crowns at the cementoenamel junction using a low-speed water-cooled diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water cooling. Two dental slabs (4 mm × 3 mm × 2 mm) were obtained from the middle third of the buccal surface of each tooth and subjected to steam sterilization to avoid bacterial contamination. Each dental slab was placed in a polyvinyl chloride matrix and fixed with a colorless translucent acrylic resin, keeping the enamel surface unsealed for bleaching applications. The specimens were serially polished by means of 600-, 1000-, 1500-, and 2000-grit SiC papers with water as a cooler to obtain flat standardized enamel surfaces. Subsequently, they were polished with diamond spray (1 μm, 0.5 μm) and polishing cloths, followed by rinsing with running water to get rid of debris layers. At last, all samples were placed in an ultrasonic cleaning machine and immersed in DW for 5 minutes to remove residual particles and smear layers. Prior to the experiment, all prepared specimens were stored in AS, which was renewed every day in a 37°C incubator for 7 days to standardize the initial conditions. The AS[17] used in this study contained calcium and phosphate with a known concentration (50 mmol/L KCl, 1.5 mmol/L Ca, 0.9 mmol/L PO_4, 20 mmol/L tri-hydroxymethyl-aminomethane, pH=7.0).

For each volunteer, a full-arch maxillary impression was obtained and a stone cast mold made based on the impression. A 0.035-inch-thick soft bleaching tray (Soft-Tray Sheets, Ultradent Products Inc, South Jordan, UT, USA) was fabricated on the cast using a vacuum tray–forming machine (Ultraform, Ultradent Products Inc) and then modified with a palatal extension.

The two in-office bleaching agents used in the current study were Opalescence Boost (O-Boost, 38% hydrogen peroxide gel, Ultradent Products, South Jordan, UT, USA) and Beyond (35% hydrogen peroxide gel, Beyond Technology Corp, Santa Clara, CA, USA). The pH values of each bleaching agent were measured by a digital pH electrode (EASY-FERM PLUS 225, Hamilton, Bonaduz, Switzerland) three times, and then mean values were recorded as their final pH values. The information about the two in-office bleaching products is shown in Table 1.

Initial Measurements

AFM Detection—Initial surface roughness and surface morphology detections were performed using atomic force microscopy. This was conducted using a Shimanzu SPM-9500J3 (Shimadzu Corp, Kyoto, Japan). AFM software SPM-Offline (version 2.30) was used to obtain the surface roughness (RMS), which represents the average of the square height difference between surface peaks and valleys (root mean square of the heights). Meanwhile, three images (10 μm × 10 μm) of each specimen were obtained.

Microhardness Test—Vickers indentation microhardness baseline values were performed using a microhardness tester (HXD-1000TMC/LCD, Taiming Inc, Shanghai, China). Three indentations were made on each specimen with 100*g* for 15 seconds. The surface area of the flattened enamel was sufficient for each indentation without interfering with each other (Figure 1).

Fracture Toughness Test—Vickers indentations with a load of 9.8 N were performed to assess baseline FT values on each enamel surface (Figure 1). These indentations for FT and cracks were then recorded immediately under a light microscope with 400-fold magnification, which belonged to the microhardness tester (HXD-1000TMC/LCD, Taiming Inc. Shanghai, China).

The diagonal lengths of each indentation were measured for the evaluation of enamel FT. The hardness values were determined according to the following expression[18]:

Table 1: *In-Office Bleaching Agents Used in the Study*

Product	Composition, pH	Activation System	Application
Opalescence Boost	38% hydrogen peroxide, fluoride, potassium nitrate, thickening agent, gel, pH=7.52	Chemically activated	First and eighth day for 45 minutes each; total of 90 minutes
Beyond	35% hydrogen peroxide, H_2O, thickening agent, gel, pH=4.03	Powerful light-emitting diodes (LED) emit a high-intensive blue light (about 480-nm wavelength)	First and eighth day for 45 minutes each; total of 90 minutes

$$H = 0.47P/a^2$$

where H=hardness, a=half the diagonal of the indentation (m), and P=applied load (MN) (9.807×10^{-6} MN).

The lengths of the cracks were measured from optical micrographs using Image J software (version 1.41). For each indentation, a circle enclosing all associated cracks (both edge and side cracks) was drawn, and its radius, developed from the center of the indentation, was taken as the maximum crack length value of the corresponding indentation. FT was then computed by means of the following formula[19]:

$$K_{IC} = 0.016(E/H)^{1/2}P/c^{3/2}$$

where K_{IC}=fracture toughness ($N/\mu m^{3/2}$), E=Young's modulus (GPa), H=Vickers hardness (GPa), P=applied load (N), and c=the maximum crack length (μm) from the center of the indentation impression. The Young's modulus of human enamel was taken at 84.1 GPa.[20]

After initial measurements, specimens were randomly divided into nine groups, according to the bleaching agents and storage conditions (n=12): group Beyond + human saliva (HS), group O-Boost + HS, group Control + HS, group Beyond + AS, group O-Boost + AS, group Control + AS, group Beyond + DW, group O-Boost + DW, and group Control + DW (Table 2).

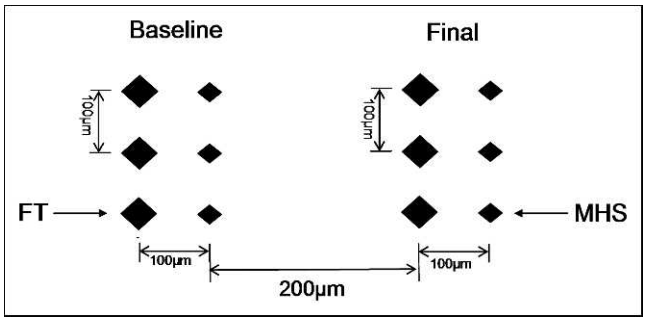

Figure 1. *Location of initial and final indentations performed for microhardness and fracture toughness test.*

Thirty-six dental slabs for *in situ* groups were removed from the polyvinyl chloride matrix by using probes and then were fixed on the palatal extension of the modified bleaching trays of four volunteers with light cure restorative material (3M ESPE, St Paul, ST, USA). Nine specimens arrayed in three columns for each volunteer: the first ones in each column were used as control specimens, and the second and third ones were the experimental specimens (Figure 2).

Once the *in situ* enamel specimens were fixed in the modified trays, they were cleaned with DW and then kept in the oral environment of volunteers for one day to form *in situ* pellicle to mimic the clinical situation. During the intraoral exposure period, the enamel slabs were cleaned every 12 hours with DW and a toothbrush without any dentifrice for a few seconds to mimic the daily oral hygiene conditions. Dentifrice was forbidden in an attempt to minimize its adverse abrasive effects on the pellicle and diminish the interference caused by other components of dentifrice, such as fluoride and desensitizing agents. Moreover, the trays were removed only for meals and stored in a 100% humidity environment to exclude the effects of food components on pellicle development.

To have a comparable model, enamel specimens for AS and DW were also exposed to the corresponding storage environment until the bleaching treatment started.

Bleaching Procedure

After one day's storage, the bleaching procedure started on the first day. Based on a usual clinical scenario, two in-office visits and three bleaching episodes for each visit were performed in this study. For the first visit, all specimens were taken out of the storage environment and dried by compressed

Table 2: *Bleaching Agent and Storage Condition in Each Group*

Storage Condition	Bleaching Agent		
	Beyond	O-Boost	Control
HS (in situ)	Beyond + HS	O-Boost + HS	Control + HS
AS (in vitro)	Beyond + AS	O-Boost + AS	Control + AS
DW (in vitro)	Beyond + DW	O-Boost + DW	Control + DW

Abbreviations: AS, artificial saliva; DW, distilled water; HS, human saliva; O-Boost, Opalescence Boost.

air for 15 seconds. Then the two bleaching agents were applied on the specimens according to the standard bleaching procedure extraorally as follows: 1) positioned the head of the matched light (Beyond Technology) upon the teeth surfaces for Beyond groups; 2) painted the specimen surfaces with corresponding whitening gels to form a 2-mm-thick layer; 3) started the timer and turned on the light at the same time, with the first bleaching episode lasting for 15 minutes at room temperature; 4) removed the bleaching agents carefully with soft cotton pellets and then repeated steps 1, 2, and 3 twice; 5) removed the bleaching gel carefully with soft cotton pellets under running DW; and 6) dried the surfaces of samples and stored *in vitro* groups in AS or DW. For the *in situ* groups, the modified bleaching trays were replaced in the oral cavity of the four volunteers as previously described. Then, the first in-office visit ended.

After seven days' storage, the second in-office visit started on the eighth day. We repeated all the procedures above and then kept samples in the corresponding storage environment for another seven days' storage.

Final Measurements

On the fifteenth day, the *in situ* samples were replaced into the polyvinyl chloride matrix, and all samples were then retested for final measurements of surface roughness RMS, surface morphology, microhardness, and FT. The experimental design is summarized in Figure 3.

The percentage of microhardness loss (PML) was calculated using the following calculation: $PML(\%) = (VHN_{(B)} - VHN_{(F)})/VHN_{(B)}$, where $VHN_{(B)}$ was the average of the baseline microhardness values and $VHN_{(F)}$ was the average of final microhardness values. The variation of RMS (ΔRMS) was expressed as follows: $\Delta RMS = RMS_{(F)} - RMS_{(B)}$, where $RMS_{(B)}$ was the average of the baseline surface roughness RMS values and $RMS_{(F)}$ was the average of final surface roughness RMS values. Similarly, the variation of FT (ΔFT) was expressed as follows: $\Delta FT = FT_{(F)} - FT_{(B)}$, where $FT_{(B)}$ was the average of the baseline FT values and $FT_{(F)}$ was the average of final FT values.

Statistical Analysis

Statistical analysis was performed by SPSS 16.0 for Windows. All statistical analyses were carried out at a significance level of 0.05. Mean values of RMS, microhardness, and FT of samples in the experiment were expressed as means ± SD. Variations for baseline and final values in each group were analyzed by analysis of variance (ANOVA). Since the storage condition and the bleaching agent were investigated as two main factors, a two-way ANOVA test and an additional Tukey *post hoc* analysis was used to analyze the effects of the two main factors on ΔRMS, PML, and ΔFT of the enamel.

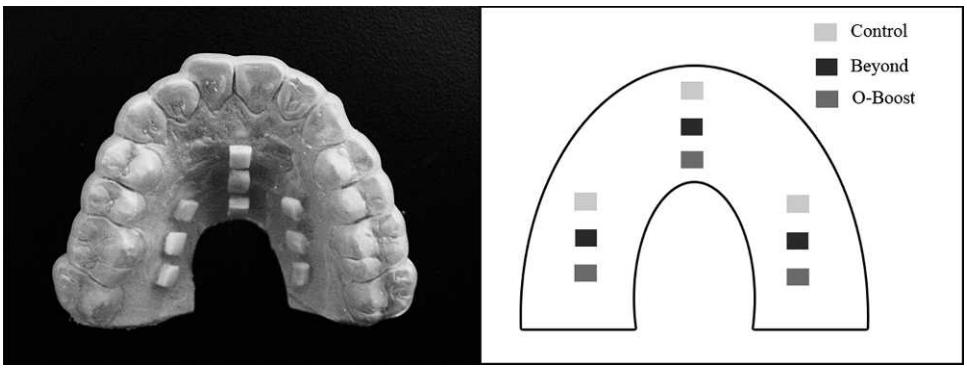

Figure 2. *Distribution of the experimental and control samples for the* in situ *condition.*

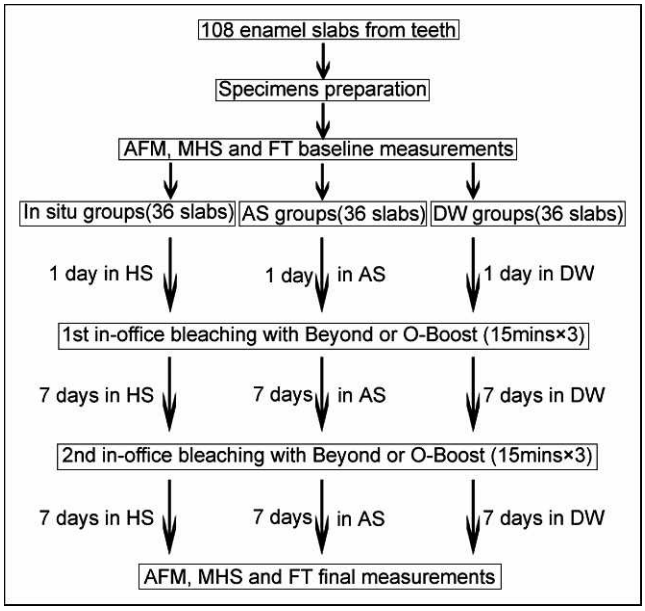

Figure 3. *Flow diagram of the experimental process in the study.*

RESULTS

AFM Detection

The morphological alterations of enamel surface were obtained by means of AFM. Figure 4 illustrates the means and standard deviations of enamel surface roughness RMS in each group. After treatment, the RMS of group Beyond + DW and group Beyond + AS increased significantly ($p<0.001$, $p<0.001$, respectively). No significant increase of RMS was found in any other groups ($p>0.05$).

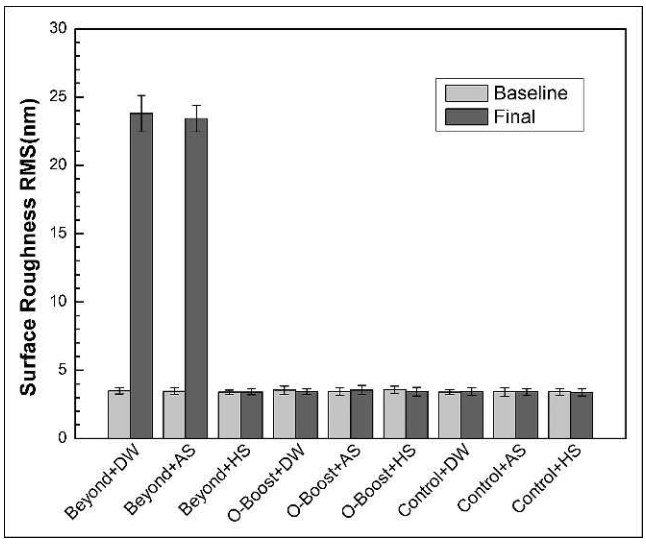

Figure 4. *RMS values following bleaching for each group. Compared with baseline RMS values, significantly higher final RMS values are shown in group Beyond + AS and group Beyond + DW.*

Table 3: *Two-Way Analysis of Variance of ΔRMS*

Source	Dependent Variable	*df*	Mean Square	*F*	*p* Value
Storage condition (A)	ΔRMS	2	551.545	1.221E3	0.000
Bleaching agent (B)	ΔRMS	2	2171.126	4.805E3	0.000
A × B	ΔRMS	4	534.185	1.182E3	0.000

Two-way ANOVA on RMS revealed statistically significant intergroup differences ($p<0.001$) for both factors and their interaction ($p<0.001$; Table 3). The additional Tukey *post hoc* test was used to clarify differences among the groups. For the factor of storage condition, the Tukey test revealed significant differences between the *in situ* condition and DW or between the *in situ* condition and AS ($p<0.001$, $p<0.001$, respectively). No significant difference was found between DW and AS ($p=0.960$). For the factor of bleaching agent, the Tukey test demonstrated significant differences between Beyond and O-Boost or between Beyond and Control ($p<0.001$, $p<0.001$, respectively). No significant difference was revealed between O-Boost and Beyond ($p=0.957$).

Representative AFM images are shown in Figures 5 and 6, and these images were in accordance with RMS data. It could be found that the grooves on the enamel surfaces became deeper and wider in the group Beyond + DW and the group Beyond + AS, and morphology alterations were more evident in the group Beyond + DW. Even some enamel rods and narrow interrod structures were seen in this group (Figure 5). To the contrary, no obvious alteration was found on enamel surfaces in the group Beyond + HS (Figure 5) and the surfaces of specimens treated by O-Boost (Figure 6). These patterns of microscopic enamel surfaces appeared to be relatively flat surfaces with some irregular and shallow grooves, resulting from the various polishing treatments.

Microhardness Test

Figure 7 provides the means and standard deviations of Vickers microhardness in each group. There was no difference between baseline and final values in all groups ($p>0.05$). Two-way ANOVA on PML revealed no statistically significant differences among the groups for both main factors ($p>0.05$) and their interactions ($p>0.05$; Table 4).

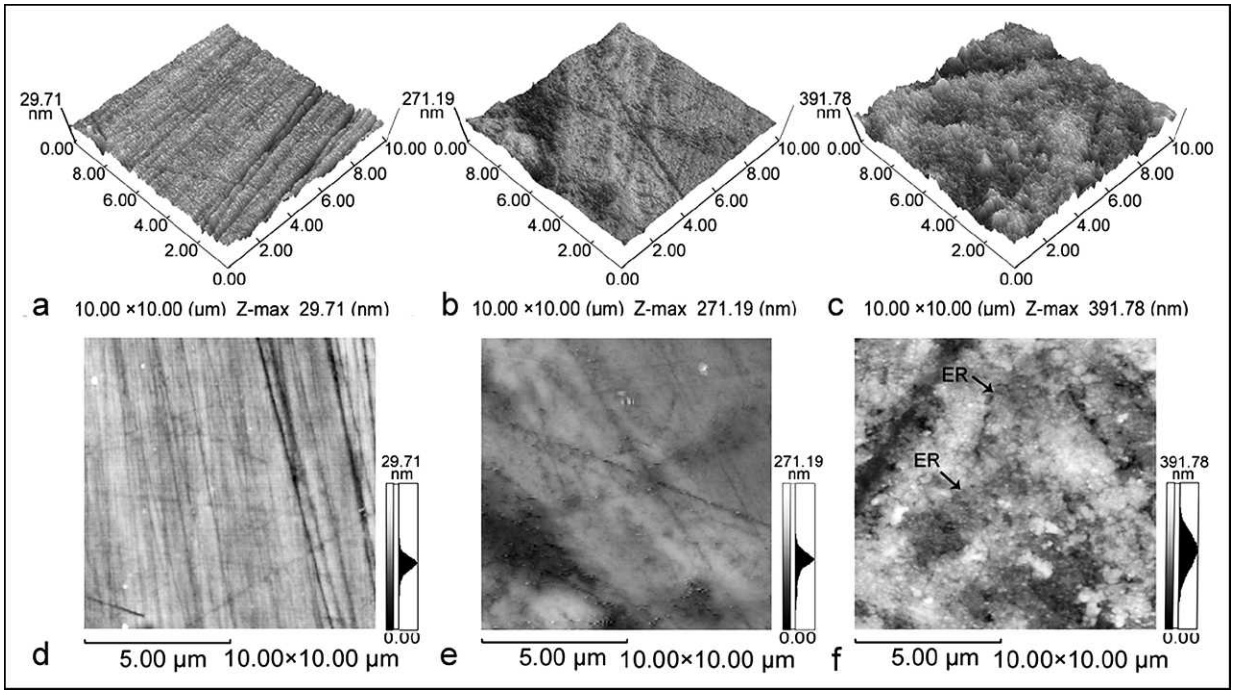

Figure 5. *Atomic force microscopy (AFM) images of specimens in the Beyond groups with three-dimensional (upper) and corresponding two-dimensional (lower) observations. (a, d) Group Beyond + HS. (b, e) Group Beyond + AS. (c, f) Group Beyond + DW. The enamel rod was depicted as ER.*

FT Test

FT values were calculated according to the aforementioned formula. Figure 8 denotes the means and standard deviations of FT in each group. After treatment, no significant decreases were found in all groups when comparing the baseline and final values ($p>0.05$). Two-way ANOVA on ΔFT exhibited

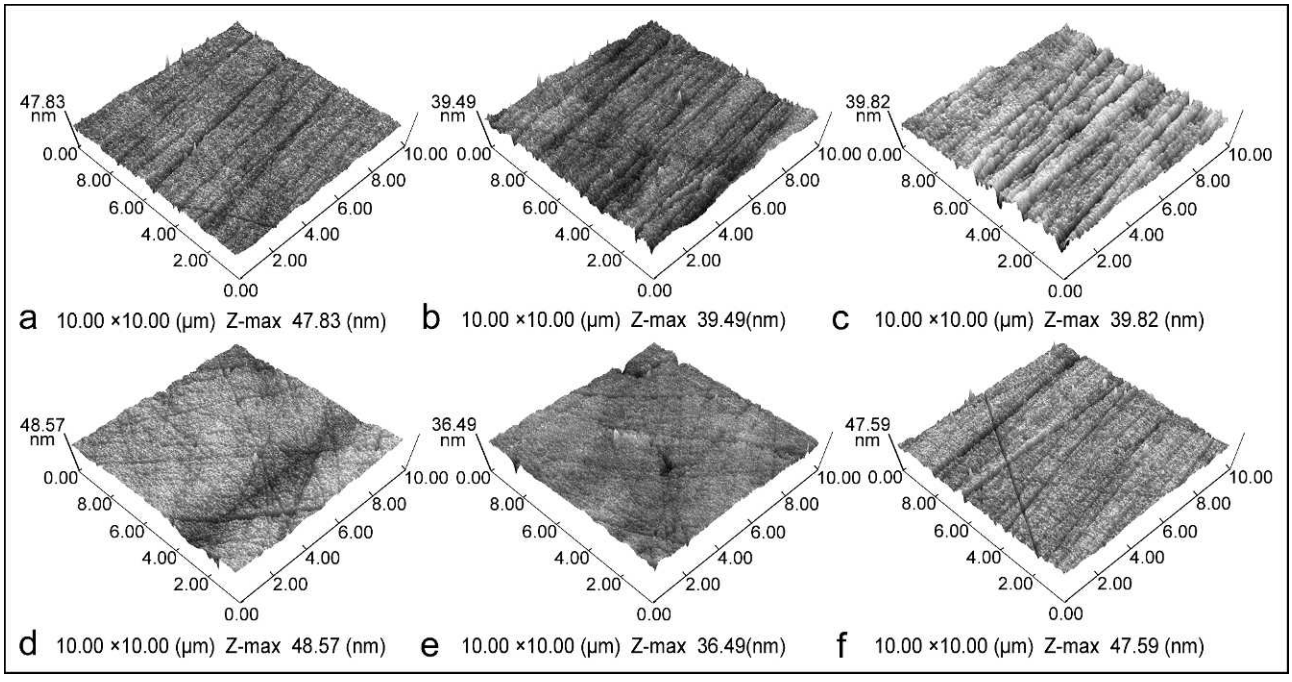

Figure 6. *Atomic force microscopy (AFM) images of specimens in O-Boost and control groups with three-dimensional observations. (a) Group O-Boost + HS. (b) Group O-Boost + AS. (c) Group O-Boost + DW. (d) Group Control + HS. (e) Group Control + AS., (f) group Control + DW.*

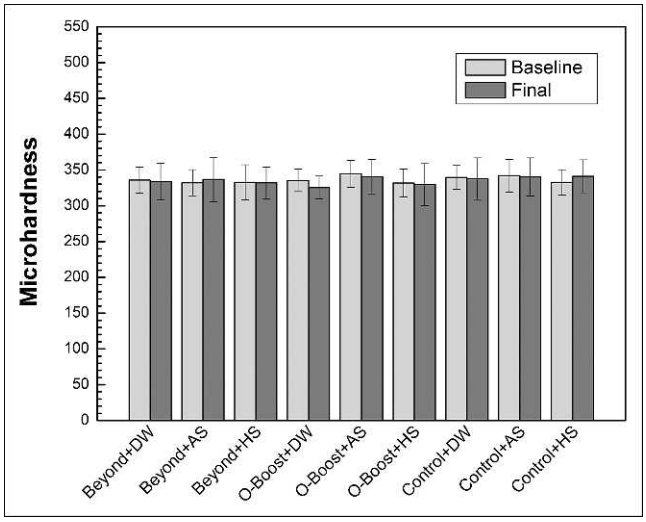

Figure 7. *Microhardness (100 g) values following bleaching for each group. No significant difference was found between the baseline and final values in each group.*

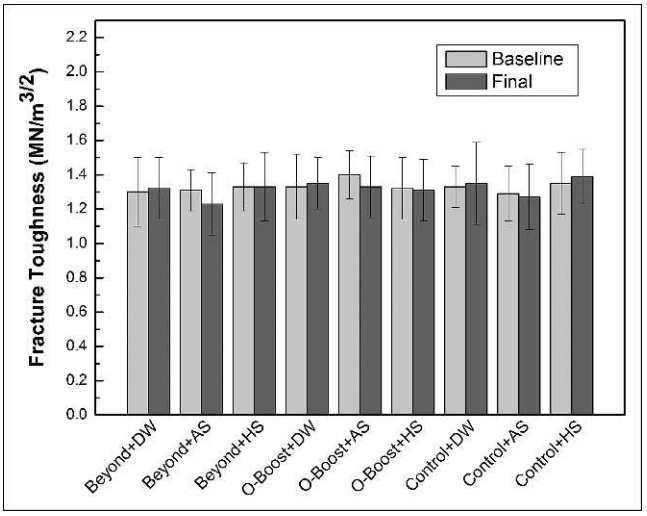

Figure 8. *Fracture toughness values following bleaching for each group. No significant difference was found between baseline and final values in each group.*

no statistically significant differences among the groups for both main factors ($p>0.05$) and their interactions ($p>0.05$; Table 5).

DISCUSSION

The results of this study indicated that bleaching agents with low pH values could induce alterations of enamel surfaces under the *in vitro* situation, while no change was observed in bleached specimens that were exposed to *in situ* conditions. Moreover, the pH values of bleaching agents and storage condition (*in vitro* and *in situ*) had no effect on the mechanical properties of human enamel after bleaching. Therefore, the first null hypothesis was partially rejected, and the second null hypothesis was accepted.

Group Beyond + DW and group Beyond + AS revealed obvious changes on enamel surfaces when compared with control groups in this study. On the contrary, little morphological modification of the enamel surface was found with the application of O-Boost in group O-Boost + DW and group O-Boost + AS. RMS results showed agreement with the morphology observations. It was claimed that the oxidative effect, pH, and composition of the bleaching agents could induce possible side effects during tooth bleaching.[5] In the current study, O-Boost and Beyond had a similar concentration of HP, but O-Boost did not result in surface alterations of specimens in DW or AS. Thus, under the same condition, the morphological alterations in Beyond groups could not be attributed to the oxidative effect of bleaching agents. Moreover, previous studies[11,21] reported that thickened agents could promote the decline of microhardness in enamel; however, it was assumed that the low pH value contained in thickened agents may be the reason for the demineralization.[21] Therefore, the pH value might be the main factor for consideration.

In the present study, the pH was 7.52 in O-Boost and 4.03 in Beyond. It has been reported that

Table 4: *Two-Way Analysis of Variance of PML*

Source	Dependent Variable	*df*	Mean Square	*F*	*p* Value
Storage condition (A)	PML	2	0.005	1.043	0.355
Bleaching agent (B)	PML	2	0.006	1.243	0.292
A × B	PML	4	0.002	0.330	0.857

Table 5: *Two-Way Analysis of Variance of ΔFT*

Source	Dependent Variable	*df*	Mean Square	*F*	*p* Value
Storage condition (A)	ΔFT	2	0.075	1.227	0.297
Bleaching agent (B)	ΔFT	2	0.005	0.077	0.926
A × B	ΔFT	4	0.009	0.149	0.963

enamel demineralization occurs when the pH falls below 5.2.[2,22,23] Thus, under the *in vitro* condition, the lower pH value may cause the demineralization of enamel surfaces. This viewpoint was supported by our previous study.[5] In that study, alkaline hydroxyapatite (HA) effectively minimized the demineralization of enamel surfaces caused by an acidic composition containing 30% HP by elevating the pH value of the HP solution and forming a protective layer for lessening the direct contact of acid HP to enamel.

By comparing AFM images obtained following treatment between group Beyond + AS and group Beyond + DW (Figure 5), typical enamel prisms and interprisms were observed in only the latter one. It indicated that some degrees of remineralization probably happened on the enamel surface in AS. This might be due to the high level of calcium and phosphate components contained in AS. These components could interact with enamel surfaces and precipitate spontaneously during the storage episode,[24,25] inducing remineralization of an existing enamel lesion.

It was of great value to note that little change was detected on the enamel surface in group Beyond + HS in this study, indicating that HS better counteracted the adverse effects of low pH compared with AS or DW. This result showed great accordance with some previous studies.[13,26]Apart from the similar calcium, phosphate, and fluoride ions in AS, HS had two other advantages that could account for its superior protective ability compared with AS.[27] One advantage was that HS with organic components might promote the formation of a pellicle layer,[2,14] which was a potentially protective surface layer that consisted mainly of salivary proteins and covered the underlying enamel surface in the oral environment.[28,29] Previous experiments[30,31] found that the *in situ*–formed short-term salivary pellicle, even within 3 minutes, could protect the enamel surface to a certain extent against demineralization. In the current study, the *in situ* samples were placed in the corresponding HS 24 hours prior to bleaching to mimic the clinical condition. Therefore, it was speculated that the salivary pellicle had formed enough thickness to offer effective protection against bleaching agents with low pH values. Another advantage of HS was that bicarbonate and phosphate-buffering systems in HS could hinder the decline of pH values and played an important role in the remineralization process when pH was within physiological limits.[26,32,33]

It was worth noting that none of the bleaching/storage regimens had any effect on the mechanical properties of the enamel specimens. This result was in contrast to several previous studies.[9,11,16,34,35] We suggested that different bleaching agents and the relatively short bleaching procedure in the current study might be the reasons for the mechanical results. These factors resulted in very shallow demineralization of the enamel surface. As the AFM images showed, the most deleterious effect was confined to less than 400 nm under the enamel surface. These relative small variations in the outermost layer of enamel were not sufficient to influence whole mechanical properties of enamel.

One limitation of the present study design should be noted. Under the clinical situation, bleaching agents are typically applied on the labial or buccal surface of the anterior teeth, whereas in the current study, we placed the enamel blocks on the palate. That was mainly attributed to the consideration of comfort and esthetic effects for the volunteers. Moreover, some previous *in situ* studies[36,37] chose the palatal model to simulate the condition of the human oral cavity and successfully proved its superiority over the *in vitro* strategy. It was possible that the action of the tongue may have removed superficial bleached enamel; however, the action of the lips may show a similar effect.

This study examined the possible enamel alterations in terms of morphology and mechanical properties. Although no morphological or mechanical alterations resulted from bleaching treatments under *in situ* conditions, these two agents might induce other alterations in enamel structure that were not apparent in this study. After all, the neutral HP agents still hold oxidizing ability for whitening efficiency. This will be an interesting direction for us to pursue in a future study.

CONCLUSION

Within the limitations of the present study, the following conclusions were drawn: In-office bleaching agents with low pH values could induce enamel morphology alterations under *in vitro* conditions. The presence of natural HS could eliminate the demineralization effect caused by low pH.

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Conflict of Interest Declaration

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

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