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Editorial

Gratitude and the Academy

We live in an amazing time! Advancements in materials and technology have occurred so rapidly that we often struggle to determine which ones are important and which ones are not. It is easy to become lost in it all. As we strive to advance amid all of the changes, sometimes things can go awry. Recently, I was in the steam room at the gym and was struck by the conversation of two other men who were vocalizing a frustration that I have felt in some situations in my professional life and that, I believe, reflects a frustration that I have seen in some other academy members.

These gentlemen were discussing how discouraging it is when someone who has benefited from the investment of many others eventually "makes the big time" and then seems to forget the contributions of those who helped enable that achievement. The conversation concerned a local man who has made it to the national scene. Even though his success can be directly attributed to the support and encouragement of others in his community, he doesn't appear to be investing in the next generation back home. As the conversation drew to a close, we all agreed that it is often easy to discern whether a person is working from a basic attitude of gratitude or one of greed. At least in his interactions with his home community, this boxer exhibited a clear attitude of greed.

It has been said that a distinguishing characteristic of a professional is having a core motivation of service to others. This is in contrast to a pure tradesman who is motivated by maximizing profit. And in one sense, this difference is wrapped up in "gratitude or greed." In those moments when I feel frustrated by things that I observe happening in our world of dentistry, I can often connect the situations to a lack of an "attitude of gratitude" on someone's part. As dentists, we must recognize that each demonstration of greed diminishes our right to be called a professional.

Hanging on my office wall is a hand painted sign that one of my daughters made for me. It says simply, "Gratitude or Greed." I look at it every time I walk into my office. Which will I demonstrate? I wish that I could say that I have never allowed greed to influence my actions and decisions. But, to the extent that I can live with gratitude, our profession will benefit. The goal of every interaction becomes one of service with gratitude; service to God, to family, to students, to patients, to the professional community, and to the community as a whole.

Maintaining this attitude of service can go a long way toward helping sort through the advances of materials and technology. Do I need the added expense of the newest and latest? Will the quality of care be enhanced for my target patient population? Will it improve my ability to serve with gratitude? Or, will it drive me toward an attitude of greed because of increased overhead with no benefit, pressuring my cash flow? Obviously, the right answer will not be the same for every individual in any given situation. Organized efforts to provide relatively unbiased information, something seen with professional academies, enhance a practitioner's ability to provide service with gratitude.

Membership in a professional academy can help members sort through some of the questions that surround the rapid changes in our world. Just as it has for many others, I know that membership in the Academy of Operative Dentistry (AOD) has helped me do that throughout the years. Unfortunately, membership in academies is dropping. Along with that decrease in membership, the ability of the academy to impact the profession through dissemination of information is being challenged.

My membership in AOD can be directly attributed to a practicing general dentist who recognized the importance of the information exchange that occurs through the professional academy. He also recognized that, during the early years of private practice, it was very difficult for me to maintain membership in those organizations that would enhance my dental knowledge and, as a result, our profession. He went

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out of his way to provide the financial resources for me to be an active member of AOD during those early years. This man acted from a position of gratitude, to the benefit of a fellow practitioner and for the good of our profession.

Are you coming along side of a young practitioner in your community? Are you willing to do whatever it takes to encourage that practitioner to be an active member of your academy? When membership stays strong, the information exchange is strengthened. Don't become like that boxer who forgot the people who helped him achieve his level of success. We all should be living with an attitude of gratitude for those in our communities—for the benefit of all.

Jeffrey A Platt, DDS, MS
Editor

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Clinical Research

Randomized Clinical Trial of Four Adhesion Strategies: 18-Month Results

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MT Ciaramicoli • VH Kiyan • CS Queiroz

Clinical Relevance

The 18-month retention rate of the two self-etch adhesives used in the present study was similar to that of two etch-and-rinse adhesives from the same manufacturer. However, the quality of enamel margins was significantly better for the two etch-and-rinse adhesives.

SUMMARY

Statement of the Problem: With Institutional Review Board approval, 39 patients who needed restoration of noncarious cervical lesions (NCCLs) were enrolled in this study. A total of 125 NCCLs were selected and randomly assigned to four groups: 1) a three-step etch-and-rinse adhesive, Adper Scotchbond Multi-Purpose (MP, 3M ESPE, St Paul, MN, USA); 2) a two-step etch-and-rinse adhesive, Adper Single Bond Plus (SB, 3M ESPE); 3) a two-step self-etch adhesive, Adper Scotchbond SE (SE, 3M ESPE); and 4) a one-step self-etch adhesive, Adper Easy Bond (EB, 3M ESPE). A nanofilled composite resin was used for all restorations. Restorations were evaluated at six months and 18 months using modified U.S. Public Health Service (USPHS) parameters.

Results: At six months after initial placement, 107 restorations (85.6% recall rate) were evaluated. At 18 months, 94 restorations (75.2% recall rate) were available for evaluation. The 6 mo/18 mo overall retention rates (%) were 100/90.9 for MP; 100/91.7 for SB; 100/90.9 for SE; and 96.4/92.3 for EB with no statistical difference between any pair of groups at each recall. Sensitivity to air decreased significantly for all adhesives from the preoperative to the postop-

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erative stage and was stable thereafter. Interfacial staining did not change statistically from baseline to six months; however, interfacial staining at the enamel margins was statistically worse at 18 months than at baseline for the two self-etch adhesives EB and SE. Marginal adaptation was statistically worse at 18 months compared with baseline only for EB. This tendency was already significant at the six-month recall.

Conclusion: Although 18-month retention was similar for the different adhesion strategies, enamel marginal deficiencies were more prevalent for the self-etch adhesives.

INTRODUCTION

Before the advent of the etch-and-rinse bonding strategy, dentin adhesives were designed not to remove the smear layer, but rather to modify it through preservation of a modified smear layer with concomitant slight demineralization of the underlying dentin surface. Despite promising laboratory results,[1] some of the earlier bonding mechanisms never resulted in satisfactory clinical results. Retention rates for early dental adhesives in noncarious cervical lesions (NCCLs) were in the range of 63% at six months[2] to 50% at one year,[3] even with enamel etching.

Recent developments in the chemistry of dentin adhesives have resulted in *in vitro* dentin bond strengths and clinical retention that approach those usually associated with enamel bonding.[4-8] In spite of different classifications of adhesives, current strategies depend on how the adhesive interacts with the smear layer. Etch-and-rinse adhesives remove the smear layer upon acid-etching, while self-etch adhesives make the smear layer permeable without removing it completely.[9]

Multi-bottle etch-and-rinse adhesives involve separate etching and rinsing steps followed by priming and application of a bonding resin. Etching and priming are considered technique-sensitive application procedures.[9] Two-step etch-and-rinse adhesives also involve a separate etching step but combine primer and adhesive resin into one solution. These two-step adhesives may need more than one application to achieve an acceptable micromechanical interlocking of monomers into the collagen-rich etched dentin.[9,10] Three-step etch-and-rinse adhesives have resulted in better laboratory and clinical performance than two-step etch-and-rinse adhesives.[11,12]

Two-step self-etch adhesives consist of nonrinsing acidic monomers dissolved in an aqueous solution and a hydrophobic resin layer as the second step. One-step self-etch adhesives lack this hydrophobic resin. The aggressiveness of acidic monomers in self-etch adhesives (therefore, their ability to demineralize dentin and enamel) depends on their pH, that is, mild, moderate, or aggressive self-etch adhesives.[13] Self-etch adhesives rely on their ability to infiltrate through smear layers and partially dissolve hydroxyapatite to generate a hybrid layer with minerals incorporated.[9] Because the preparation is not rinsed, these materials are more user-friendly as application time is reduced, compared with etch-and-rinse adhesives.

A systematic review analyzed 85 full articles and abstracts focused on the clinical effectiveness of adhesives in NCCLs.[14] The lowest retention failure rate was shared by glass ionomer–based materials and three-step etch-and-rinse adhesives, whereas the highest retention failure rate was associated with one-step self-etch adhesives. A recent clinical trial in NCCLs compared four adhesives representing all adhesion strategies.[15] Only the three-step etch-and-rinse adhesive resulted in retention rates above 90% at 18 months, which is the American Dental Association (ADA) threshold for full acceptance.[16]

Given that dentists are confused about the efficacy of different generations of dentin adhesives, this study tested the null hypothesis that there are no differences in the clinical retention of four adhesion strategies from the same manufacturer, when applied to NCCLs.

METHODS AND MATERIALS

Before participating in the study, subjects gave informed consent. Both the consent form and this research protocol were reviewed and approved by the Paulista University Institutional Review Board. All 39 subjects, with ages ranging from 22 to 78 years (average 47.6), had been referred to the Operative Dentistry Clinic for restoration of Class V lesions. All subjects received a dental examination by a member of the clinical faculty. The dental health status of patients was normal in all other respects. Patients with fewer than 20 teeth were not included in the study when all other characteristics of dental status were considered normal, including the periodontal condition. With caries as an exclusion criterion, teeth included in the study had NCCLs without undercuts. Other exclusion criteria included the following:

- History of existing tooth sensitivity
- Bruxism and visible wear facets in the posterior dentition
- Known inability to return for recall appointments

- Fractured or visibly cracked candidate tooth
- Current desensitizing therapy, including desensitizing dentifrices or other over-the-counter (OTC) products
- Long-term use of anti-inflammatory, analgesic, or psychotropic drugs
- Pregnancy or breast-feeding (potential conflicts with recall dates)
- Allergies to ingredients of resin-based restorative materials
- Orthodontic appliance treatment within the previous three months
- Abutment teeth for fixed or removable prostheses
- Teeth or supporting structures with any symptomatic pathology
- Existing periodontal disease or periodontal surgery within the previous three months

The teeth to be restored were vital (positive response to cold sensitivity test), had a normal occlusal relationship with natural dentition, and had at least one adjacent tooth contact. Cavo-surface angles were not beveled, and no retentive grooves were placed.

Materials, respective batch numbers, composition, and manufacturer's instructions for use are provided in Table 1. Approximately 91% of the lesions were classified as degree 1 or 2 on the University of North Carolina (UNC) sclerosis scale[17] and were equally distributed among the four groups. The distribution of restorations was 51.2% in the maxillary arch and 48.8% in the mandibular arch; 82.6% of restorations were placed in premolars or molars. Differences in lesion size and in other characteristics were minimal. Mean lesion volumes were not significantly different among the four restorative groups (one-way analysis of variance [ANOVA], $p=0.98$).

A total of 125 NCCLs were restored in this study. Each subject had up to four restorations placed, with each dentin adhesive applied to one tooth. The dentin adhesives were randomly assigned with separate randomization for each subject (adhesive material vs tooth): 1) a three-step etch-and-rinse adhesive Adper Scotchbond Multi-Purpose (MP, 3M ESPE, St Paul, MN, USA); 2) a two-step etch-and-rinse adhesive Adper Single Bond Plus (SB, 3M ESPE); 3) a two-step self-etch adhesive Adper Scotchbond SE (SE, 3M ESPE); and 4) a one-step self-etch adhesive Adper Easy Bond (EB, 3M ESPE). All operators had advanced training in Operative Dentistry and were individually instructed by the study coordinator on how to apply each adhesive. Operators placed at least one restoration in extracted teeth to practice the technique before performing the first actual restoration placement. The insertion protocol for each adhesive was printed and posted in each dental unit so the operator was able to easily review the instructions before and while applying each adhesive. Each operator inserted approximately the same number of restorations (± 2). Because of the specialized field of the operators, it was not possible to insert the restorations blindly. All operative procedures were performed with cotton-roll isolation without local anesthesia.

After application of the dentin adhesive, Filtek Supreme Plus (3M ESPE) was inserted in increments of 1.0–1.5 mm. Each increment was polymerized for 40 seconds with a light-curing unit (Elipar Freelight 2, 3M ESPE). The intensity of the light exceeded 500 mW/cm^2. After polymerization, finishing was accomplished with aluminum oxide disks of decreasing abrasiveness (Sof-Lex XT, 3M ESPE).

Clinical Evaluation

In addition to assessment of sensitivity immediately before insertion, postoperative sensitivity was assessed one week after the restorative procedure via telephone interview. Restorations were evaluated immediately after insertion, at six months, and at 18 months using the UNC-modified USPHS criteria[17] (*alfa*, *beta*, *charlie*) for retention, color match, interfacial staining, wear, marginal adaptation, surface texture, preoperative sensitivity (air syringe), and postoperative sensitivity (query) (Table 2). Two clinicians evaluated the restorations blindly at each recall but did not evaluate the restorations that they had inserted. In case no consensus was reached, a third clinician evaluated the restoration. To help with the evaluation, intraoral color photographs were collected at baseline and at the recall appointments. Clinical photographs consisted of digital images obtained using a Nikon D40X camera with a 200-mm Medical Nikkor lens (Nikon Inc, Melville, NY, USA). Statistical analyses included the Mann-Whitney nonparametric test to compare the performance of the four adhesives at each recall, and the McNemar nonparametric test to compare changes in each adhesive from baseline to six months and to 18 months (Statistical Package for the Social Sciences [SPSS], version 14.0, SPSS Inc, Chicago, IL, USA). The level of significance was set at $p<0.05$.

RESULTS

At six months, 107 restorations (85.6% recall rate) were evaluated. At 18 months, 94 restorations (75.2% recall rate) were available for evaluation. A summary of direct evaluations is shown in Table 3.

Table 1: *Materials, Batch Numbers, Compositions, and Instructions for Use*

Material	Composition	Instructions for Use
Adper Easy Bond (also known as Adper Easy One) Lot 359668	BisGMA; HEMA; water (10–15 Wt%); Ethanol (10–15 Wt%); phosphoric acid-6-methacryloxy-hexylesters; silane-treated silica; 1,6-hehadeniol dimethacrylate; copolymer of acrylic and itaconic acid; (dimethylamino) ethyl methacrylate, camphorquinone; 2,4,6-trimethylbenzoyldiphenylphosphine oxide	Dry the cavity by using gentle stream of air free of water and oil, or by blotting with cotton pellets. Do not overdry. Apply the adhesive with the disposable applicator for 20 s to all surfaces of the cavity. Rewet the disposable applicator as needed during application. Subsequently, air thin the liquid for approximately 5 s until the film no longer moves, indicating complete vaporization of the solvent. Cure the adhesive with a commonly used curing light for 10 s.
Adper Scotchbond SE (also known as Adper SE Plus)		
Liquid A: Lot 8BH	Liquid A (Pink wetting solution): water (80%), HEMA, surfactant, rose bengal dye	Liquid A: Apply to the cavity so that a continuous red layer is obtained on the surface.
Liquid B: Lot 8BJ	Liquid B (Adhesive): UDMA, TEGDMA, TMPTMA, HEMA phosphate and MHP, bonded zirconia nanofiller, initiator system based on camphorquinone	Liquid B: Scrub into the entire wetted surface of the bonding area during 20 s. Red color will disappear quickly, indicating that the etching components have been activated. Air-dry thoroughly for 10 s. Apply second coat to the entire bonding surface. Light air application. Light cure for 10 s.
Adper Single Bond Plus (also known as Adper Single Bond 2 or Adper Scotchbond 1XT)		
Etchant: Lot 8MP	Etchant: amorphous silica-thickened 35% phosphoric acid gel	Apply Scotchbond Etchant to tooth surface for 15 s. Rinse thoroughly for 10 s. Blot excess water using a cotton pellet or a mini-sponge. Do not air-dry! Apply 2–3 consecutive coats of adhesive for 15 s with gentle agitation using a fully saturated applicator. Gently air thin for 5 s to evaporate solvent. Light cure for 10 s.
Adhesive: Lot 9WJ	Adhesive: ethyl alcohol (25–35 Wt%); silane-treated silica (nanofiller); BisGMA; HEMA glycerol 1,3-dimethacrylate; copolymer of acrylic and itaconic acid; diurethane dimethacrylate; water (<5%)	
Adper Scotchbond Multi-Purpose		
Etchant: Lot 8MP	Etchant: amorphous silica-thickened 35% phosphoric acid gel	Apply Scotchbond etchant to enamel and dentin. Wait 15 s. Rinse for 15 s. Dry for 5 s. Apply Adper Scotchbond multi-purpose primer to etched enamel and dentin. Dry gently for 5 s. Apply Adper Scotchbond multi-purpose adhesive to primed enamel and dentin. Light cure for 10 s.
Primer: Lot 9CC	Primer: water (40–50 Wt%); HEMA (35–45 Wt%); copolymer of acrylic and itaconic acids (10–20 Wt%)	
Adhesive: Lot 9RL	Adhesive: BisGMA (60–70 Wt%); HEMA (30–40 Wt%)	
Filtek Supreme Plus (A2B: 8XA; A2E: 8GR; A3B: 8UU; A3E: 8EX; A3D: 8EK; A3.5: 8JG; A4D: 8CL	BisGMA, UDMA, TEGDMA, BisEMA, silanated silica, silanated zirconia; photoinitiators	

Bis-EMA - ethoxylated bisphenol-A dimethacrylate; BisGMA- bisphenol A diglycidyl methacrylate; HEMA; 2-hydroxyethyl methacrylate; MHP - methacryloyloxyhexyl phosphate; TEGDMA - triethyleneglycol-dimethacrylate; TMPTMA - trimethylolpropane trimethacrylate; UDMA - urethane dimethacrylate.

Table 2: *UNC-Modified USPHS Direct Evaluation Criteria*	
Color Match	Alfa = No mismatch in room light in 3–4 s
	(margins exempted from grading)
	(interfacial staining should not affect grading)
	Bravo = Perceptible mismatch (but clinically acceptable)
	Charlie = Esthetically unacceptable (clinically unacceptable)
Interfacial Staining	Alfa = None
	Bravo = Superficial staining (removable, usually localized)
	Charlie = Deep staining (not removable, generalized)
Recurrent Caries	Alfa = None
	Charlie = Present
Wear	Alfa = No perceptible wear (or only localized wear)
	Bravo = Generalized wear (but clinically acceptable)
	(<50% of margins are detectable)
	(catches explorer going from material to tooth)
	Charlie = Wear beyond the DEJ (clinically unacceptable)
Marginal Adaptation (Ditching)	Alfa = Undetectable
	Bravo = Detectable (V-shaped defect in enamel only)
	(catches explorer going both ways)
	Charlie = Detectable (V-shaped defect to DEJ)
Surface Texture	Alfa = Smooth (better than or equal to microfilled standard)
	Bravo = Rougher than microfilled standard
	Charlie = Pitted
Postoperative Sensitivity	Alfa = None
	Charlie = Present
Retention	Alfa = Retained
	Bravo = Partially retained
	Charlie = Missing
Fracture	Alfa = None
	Bravo = Small chip, but clinically acceptable
	Charlie = Failure due to bulk restoration fracture
Other Failure	Alfa = None
	Charlie = Present

Abbreviations: DEJ, dentoenamel junction; UNC, University of North Carolina; USPHS, U.S. Public Health Service.

Two restorations were lost at 18 months for each of the adhesives EB, SE, and MP. All SB restorations were retained. Six- and 18-month retention rates (%) were 100/90.9 for MP; 100/91.7 for SB; 100/90.9 for SE; and 96.4/92.3 for EB, with no statistical difference between any pair of groups at each recall.

Interfacial staining did not change statistically from baseline to six months for any of the adhesives; however, interfacial staining around the enamel margins was statistically worse at 18 months than at baseline for the two self-etch adhesives EB and SE (both at $p<0.031$). Marginal adaptation was statistically worse at 18 months compared with baseline only for the adhesive EB ($p<0.0001$). This tendency was already significant at the six-month recall ($p<0.016$). Sensitivity to air improved significantly for all groups from preoperative conditions to one week after insertion and remained stable thereafter (Table 4). No statistical differences were noted for any of the other parameters.

Table 3: *Summary of Direct Evaluations: Percentages of Restorations With* Alfa *Scores for Each Criterion at Each Evaluation Point*

Adper Easy Bond		Adper Scotchbond SE		Evaluation Criteria	Adper Single Bond Plus		Adper Scotchbond Multi-Purpose	
6 mo	18 mo	6 mo	18 mo		6 mo	18 mo	6 mo	18 mo
28/34 = 82.4%	26/34 = 76.5%	26/30 = 86.7%	22/30 = 73.3%	Recall rate	32/32 = 100%	27/32 = 84.4%	26/29 = 89.7%	22/29 = 75.9%
27/28 = 96.4%	24/26 = 92.3%	26/26 = 100%	20/22 = 90.9%	Retention	32/32 = 100%	27/27 = 100%	26/26 = 100%	20/22 = 90.9%
27/28 = 96.4%	24/26 = 92.3%	25/26 = 96.2%	19/22 = 86.4%	Color match	31/32 = 96.9%	26/27 = 96.3%	24/26 = 92.3%	20/22 = 90.9%
26/28 = 92.9%	18/26 = 69.2%	21/26 = 80.8%	14/22 = 63.6%	Interfacial staining	32/32 = 100%	27/27 = 100%	23/26 = 88.5%	18/22 = 81.8%
27/28 = 96.4%	24/26 = 92.3%	26/26 = 100%	20/22 = 90.9%	Recurrent caries	32/32 = 100%	27/27 = 100%	26/26 = 100%	20/22 = 90.9%
27/28 = 96.4%	24/26 = 92.3%	26/26 = 100%	20/22 = 90.9%	Wear	32/32 = 100%	27/27 = 100%	26/26 = 100%	19/22 = 86.4%
21/28 = 75.0%	12/26 = 46.2%	22/26 = 84.6%	16/22 = 72.7%	Marginal adaptation	29/32 = 90.6%	23/27 = 85.1%	20/26 = 76.9%	18/22 = 81.8%
26/28 = 92.9%	22/26 = 84.6%	25/26 = 96.2%	19/22 = 86.4%	Surface texture	29/32 = 90.6%	25/27 = 95.6%	25/26 = 96.2%	19/22 = 86.4%

DISCUSSION

As per ADA guidelines, retention rates at six months must be at least 95% for provisional acceptance. At 18 months, retention rates must be at least 90% for full acceptance.[16] All four adhesives in this study fulfilled these guidelines. Therefore, we failed to reject the null hypothesis, because the 18-month retention rate of the four different adhesive strategies from the same manufacturer did not differ significantly at any recall time.

Few published clinical studies have been performed with EB or SE. A recent study[18] on NCCLs reported retention rates very similar to those of our study: 92.86%, for SB, 97.62% for SE, and 100% for EB after 12 months. Another clinical trial in Class II restorations did not result in any statistical difference between SB and SE for any of the parameters at 12 months.[19] Clinical studies with Single Bond have resulted in excellent performance up to five years.[20-22] MP has resulted in excellent clinical retention at two to three years.[23,24] In fact, three-step self-etch adhesives are the golden reference for all other adhesives.[12,14]

Among contemporary adhesives, self-etch adhesives have become popular, especially because of their user-friendliness and short application time.[14] EB, similar to many other one-step self-etch adhesives, contains phosphoric acid ester methacrylates as functional monomers. Whereas SE is a "strong" self-etch adhesive with a pH=1, EB is considered an "ultra-mild" self-etch adhesive because its pH is relatively high pH (2.4). This high pH may explain the significant deterioration of marginal adaptation from baseline to 18 months for EB. Self-etch adhesives do not etch enamel to the same depth as phosphoric acid.[7]

Both self-etch adhesives in the present study resulted in a significant increase in marginal interfacial staining around enamel margins from baseline to 18 months. This increased staining may be the result of a shallow enamel-etching pattern, because interfacial staining has been associated with a poor enamel etching ability of self-etch adhesives,[25-27] even for those considered "strong" self-etch adhesives (pH=1), such as SE.[27]

One of the drawbacks of the acidic monomers in self-etch adhesives is their instability in water.[28] SE is not

Table 4: *Incidence of Preoperative and Postoperative Sensitivity at Each Evaluation Time*

	Preoperative Sensitivity	Postoperative Sensitivity One Week Post Insertion	Sensitivity at Six Months	Sensitivity at 18 Months
Adper Easy Bond	11/34 (32.4%)	0/34 (0%)	1/28 (3.6%)	2/26 (7.7%)
		$p<0.002$[a]	NS[b]	NS[c]
Adper Scotchbond SE	8/30 (26.7%)	0/30 (0%)	1/26 (3.8%)	2/22 (9.1%)
		$p<0.008$[a]	NS[b]	NS[c]
Adper Single Bond Plus	8/32 (25.0%)	0/32 (0%)	1/27 (3.7%)	2/24 (8.3%)
		$p<0.008$[a]	NS[b]	NS[c]
Adper Scotchbond Multi-Purpose	7/29 (24.1%)	0/29 (0%)	1/26 (3.8%)	2/22 (9.1%)
		$p<0.016$[a]	NS[b]	NS[c]

[a] *Statistical significance compared with preoperative condition.*
[b] *Not significantly different from the postoperative condition.*
[c] *Not significantly different from the postoperative condition or from the six-month condition.*

a conventional two-step self-etch adhesive, as the first bottle (Liquid A) does not contain acidic monomers to condition dentin and enamel. The concept behind the two separate bottles in SE is to keep water separate from the acidic monomers to increase their shelf life. After the pink water-based solution (Liquid A, Table 1) is applied to visibly wet the preparation, the second solution, which contains the resin monomers, or Liquid B (Table 1), is vigorously mixed with the pink solution. At this point, the color changes to yellow, which means that water has triggered ionization of the acidic monomers, and etching is occurring. After air-drying to evaporate residual water, a second coat of water-free Liquid B is brushed onto the treated surface and light-cured. Ideally, this technique would prevent water from remaining inside the dentin-resin interface.

Mine and others,[29] using Transmission Electron Microscopy (TEM), showed a thick, completely demineralized and acid-resistant dentin hybrid layer as a result of the application of SE. For EB, dentin interaction was not as deep, resembling other "ultra-mild" self-etch adhesives. Regarding bond strength studies, SE has resulted in statistically higher dentin bond strengths than EB.[30] The bond strengths obtained with SE are not affected by thermal fatigue when bonded to coronal dentin[31]; this may be a result of minimal residual water left entrapped within the dentin-adhesive interface.

A recent 18-month clinical study reported a high failure rate for two one-step self-etch adhesives.[32] The addition of an extra coat of a hydrophobic bonding layer, transforming one-step into two-step self-etch adhesives, significantly improved the clinical outcome. The one-step self-etch adhesive Clearfil S^3 Bond (Kuraray America Inc, New York, NY, USA) resulted in a 77.3% retention rate at 18 months. However, when an extra layer of a hydrophobic bonding resin was added (MP adhesive), the retention rate increased to 93.4%. In the same study, iBond (Heraeus Kulzer, South Bend, IN, USA), also a one-step self-etch adhesive, resulted in a 60% retention rate at 18 months. Retention increased to 83% when a coat of the same hydrophobic resin was applied over the cured iBond. In our study, nevertheless, no differences in retention rates were noted between the one-step self-etch adhesive EB and the two-step self-etch adhesive SE. One specific factor may have accounted for the differences between the two studies. The one-step self-etch adhesive used in our study, EB, contains 1%-5% of a polyalkenoic acid copolymer, also known as the Vitrebond copolymer (3M ESPE). Mitra and others[33] reported that this copolymer bonds chemically to calcium in hydroxyapatite. This idea was corroborated by a recent clinical study[34] that found no differences in retention rate for EB with and without a hydrophobic layer

(MP adhesive) at 18 months. Both groups reported retention rates above 90%. In fact, for adhesive materials that do not require etching, chemical bonding between polycarboxylic monomers and hydroxyapatite plays an important role in the bonding mechanism.[35,36] It has been shown that two-thirds of the carboxyl groups in polyalkenoic acid are capable of bonding to hydroxyapatite.[35] Carboxylic groups replace phosphate ions on the substrate and make ionic bonds with calcium.[35] Adhesives that contain this copolymer have increased resistance to mechanical fatigue.[37]

One disadvantage of self-etch adhesives is that they do not etch enamel to the same depth as phosphoric acid.[7] In the present study, the quality of the enamel margins, specifically interfacial staining, decreased significantly for the two self-etch adhesives at 18 months. In spite of worse interfacial staining, the two self-etch adhesives resulted in similar retention rates compared with the two etch-and-rinse adhesives.

One of the limitations of this clinical investigation is that 18 months may be a short period for substantial changes to become noticeable regarding the clinical performance of dentin adhesives. Long-term clinical evaluations may better reflect the differences among the four adhesion strategies studied in this project.

CONCLUSION

In spite of enamel marginal deterioration associated with the two self-etch adhesives, the 18-month retention rates of four different adhesive strategies from the same manufacturer did not differ significantly at any recall time.

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Data Comparison Between Two Dental Spectrophotometers

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Clinical Relevance

Different color measuring devices (CMDs) are used to determine tooth color and the color of the matching restoration during the manufacturing process. This study questions whether it is possible to communicate color accurately when dentists and dental laboratories use different CMDs at different locations.

Summary

Objectives: The objective of this study was to clinically test whether the data from two different spectrophotometers, based on spot and surface measurements, can be compared.

Methods: Under standardized clinical conditions two devices (Vita Easyshade and SpectroShade-Micro) were used to record the color of three areas (cervical, middle, and incisal) per tooth for three upper maxillary anterior teeth in 102 participants. Each position was measured three times to attain an average for the CIE L*a*b* coordinates and to attain the corresponding Vita Classical shade tab integrated in the software of both devices. Vita tabs were also described as L*a*b* values using earlier published translations so that color differences (ΔE) could be calculated between them.

Results: The regression analysis between the two devices showed that the independent correlation coefficients of the L*a*b* values are low. Yet when the suggested shade codes are compared with Vita colors instead of L*a*b*, 40% of the cases were equal and 51% were clinically acceptable.

Significance: According to this study the two devices do not give a comparable shade selection output, and thus the exchange of L*a*b* values between the two spectrophotometers cannot be recommended.

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INTRODUCTION

The subjective character of visual shade determination makes color assessment one of the most complex aspects of restorative dentistry,[1-4] whereas precise

information on color is essential for the creation of an esthetic dental restoration. An escalating number of electronically based devices for tooth color determination have entered the market, compelling dental laboratories and dentists to invest in these devices. Such color measuring devices (CMDs) eliminate the subjectivity related to color determination and increase the level of consistency in the color determination process.[3,5-7] Henceforth, CMDs can be beneficial for the process of shade determination.

Different types of CMDs are based on different technologies, such as colorimetery, focal optics, and spectrophotometry. Dental colorimeters are designed to directly measure color as a function of light reflection perceived by the human eye. They use three filters corresponding to three color stimuli: red, green, and blue (RGB). Measuring tooth color is also possible by analyzing digital images, where a multitude of pixels are measured in RGB units corresponding to RGB stimuli. Spectrophotometers are devices that determine the intensity of reflected or transmitted light as a function of a light-source wavelength.[8]

The optical light settings used in CMDs can have different geometries; illumination at 0 degrees and observation at 45 degrees (0/45), illumination at 45 degrees and observation at 0 degrees (45/0), or a 0/0 degree optical geometry where the light beam and the light detector are in the exact opposite direction.[1]

There are not only different optical geometries between different CMDs but also different methods for measuring optical light[9]: spot-measurement devices and complete-tooth-measurement devices. Spot measurements are made by an optical device with an aperture of about 3–5 mm in diameter. Therefore, several recordings must be taken to obtain a more extensive shade distribution over the entire tooth surface. On the other hand, complete-tooth-measurement devices can measure the entire tooth and produce a color map of the tooth in one image.

There are several systems in which the output of the color measurements can be categorized and identified quantitatively. One of the most commonly used systems is the CIE L*a*b* color system because it approximates uniform distances between color coordinates while entirely covering the visual color space.[10,11] This system has a lightness scale, L*, ranging from 0 (black) to 100 (white), and two opposing color axes: axis a* for redness (+) and greenness (–) and axis b* for yellowness (+) and blueness (–). The output of the absolute color values, expressed as L*, a*, and b* can be translated into shade guide codes, for instance the A, B, C, and D codes of the Vita Classical (Vita, Bad Säckingen, Germany), for clinical use. It is unclear which L*a*b* values different manufacturers apply in their devices to express the different Vita shades. Moreover, the color consistency of the different Vita Shade tabs used by different manufacturers may vary, too. In a study by O'Brien and others,[12] a CIE L*a*b* translation table of the Vita shade guide was presented, as measured by a spectrophotometer. This has been, so far, the only standard where the shade tabs of the Vita shade guide are expressed as L*a*b* values.

The visual color space of teeth covers a small volume in the whole L*a*b* color space. As a consequence, the resolution of dental CMDs has to be high to be able to differentiate the whole possible color range of teeth. For the same reason, not only the resolution of the devices is of importance but also the high reproducibility of the measurement itself.[13] For instance, for most CMDs it is extremely important to keep the angle of measurement constant when repeating the measurement.

In an earlier study[8] we evaluated the repeatability and accuracy of five commercially available CMDs and concluded that, of the different CMDs, spectrophotometer measurements were the most reproducible in repeating measurements. The most reliable device *in vitro* and *in vivo* was the Easyshade (ES; Vita, Bad Säckingen, Germany),[8] which is a handheld spot-measurement device that needs to be brought into direct contact with the tooth surface when a measurement is being made. The fiber-optic tip is 5 mm in diameter and uses a pseudocircular 0/0 measuring geometry.[14] Another dental spectrophotometer that was tested in this earlier study was the SpectroShade-Micro (SS, MHT S.p.a., Verona, Italy. This device has the ability to measure the whole tooth surface and is based on illumination at 45 degrees and observation at 0 degrees (45/0).[15]

In principle, CMDs are designed to enhance communication between clinicians and dental laboratories, but the commercialization of the dental market results in the use of different devices among different professionals. However, besides the differences in working mechanisms, the basic signal of most CMDs is an electrical current originating from sensors that are transferred into color data by internal software. This can lead to possible errors when two different systems are used to determine tooth color and the color of the matching restoration

during the manufacturing process. A growing number of dental practices work with large dental laboratories abroad; hence, color has to be communicated precisely, especially in such cases where no direct contact is possible between the technician and the patient to determine color. Therefore, the question is whether it would also be possible to communicate color when dentists and dental laboratories use different CMDs at different locations. The objective was to evaluate whether the measurements of two different devices can be exchanged without resulting in a visible color difference. For this study, a color difference of $\Delta E \leq 2.0$ units was regarded as the perceptibility threshold,[12] whereas a color difference of $\Delta E \geq 3.7$ was regarded as the acceptability threshold, and thus considered clinically imperceptible.[1,12]

The hypothesis of this research was that the absolute color data, measured as a spectrum and expressed as CIE L*a*b* values, are comparable between the ES and SS spectrophotometers and therefore can be exchanged.

MATERIALS AND METHODS

The tooth color of 102 participants was measured at the Academic Centre for Dentistry in Amsterdam (ACTA, the Netherlands). A written informed consent was obtained from every subject after a full explanation of the experiment. The group consisted of 42 male and 60 female subjects, and ages ranged from 14 to 58 years (mean=23 years). Tooth color was measured under standardized clinical conditions by one operator using the Vita Easyshade and the SpectroShade-Micro CMDs.

The maxillary central and lateral incisors and the canines from the left or the right side of the maxilla were selected based on the following criteria: 1) absence of dental caries, 2) absence of restorations, 3) no previous endodontic treatment, and 4) no previous bleaching treatment or use of whitening toothpaste. Shade was recorded for all selected teeth at three sites: cervical, middle, and incisal. Thus, nine total locations were measured in 102 participants, resulting in 918 independent color measurements with the ES and SS, respectively. During measurements the participants were asked to keep their tongue in a relaxed position away from the maxillary teeth, lean their head against the headrest of the dental chair, and keep their mouth slightly opened; this was in order to prevent moving or fogging that could possibly affect the measurements. The devices were used and calibrated according to the manufacturer's instructions.

Color Measurement with Vita Easyshade

Before measuring tooth color with the ES, the selected tooth was polished using a rubber cup and polishing paste for approximately 10 seconds, after which the mouth was kept closed for at least 1 minute to allow rehydrating. A disposable infection-control polyurethane barrier (Vita Infection Control Sleeves; Vita Zahnfabrik) was used on the tip of the probe, and the device was calibrated for each participant by placing the probe with a diameter of 5 mm against a calibrated block inside the machine. Measurement proceeded by placing the probe on the previously determined area of the tooth and pressing the probe switch, taking care that the probe was not moved during a measurement and/or set at a different angle. The specific area of the tooth (Figure 1) was determined using a caliper to establish the midposition of each point and the equal distance between the three measuring areas: cervical, middle, and incisal. Tooth colors expressed in CIE L*a*b* values and the corresponding suggested Vita Classical shade codes were directly obtained for each position along the labial surface of each tooth.

Color Measurement with SpectroShade

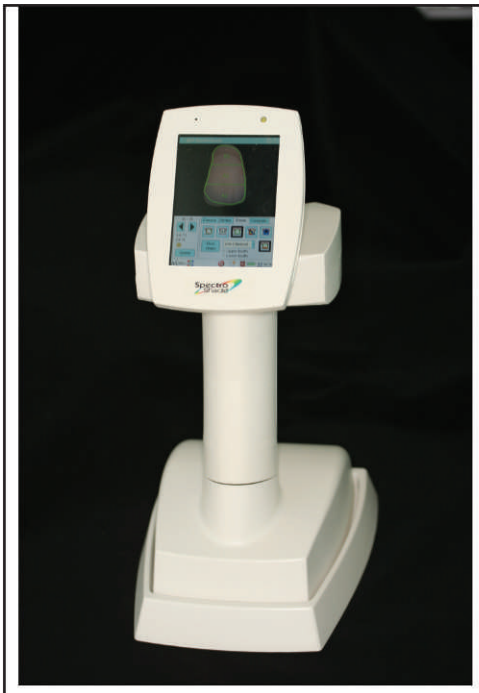
An infection-control mouthpiece and adhesive pad were placed on the optic handpiece, and then the SS was calibrated. During measurement the mouthpiece was carefully positioned over the tooth required. The screen display permitted the operator to view the whole tooth surface under the right angle, as verified by a horizontal green line (representing accurate geometry); after this the color could be recorded. After color registration of three teeth, the results were imported into the software, which automatically outlined the CIE L*a*b* values and the derived Vita Classical shade codes for each position on each tooth. The three positions were determined by locating the software tool circle with the radius of 5 mm in the cervical, middle, and incisal area and by this way approximating the same area of color measurement as used with the ES.

DATA EVALUATION

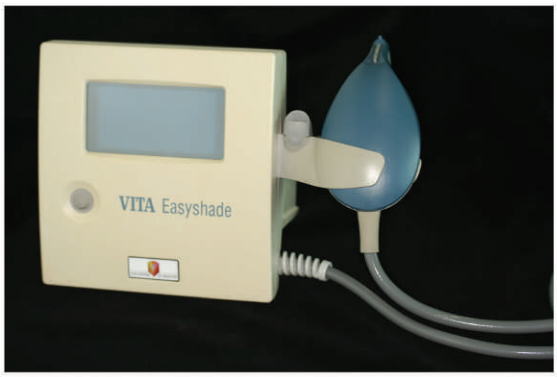
Three different methods were used to evaluate the two sets of data from the two devices:

1. Direct comparison of the measured L*a*b* values obtained with the two instruments expressed in ΔE. The following equation was used:

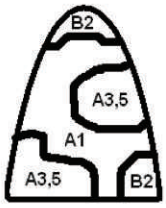
$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \qquad (1)$$



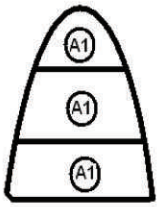
SpectroShade Micro



EasyShade



Complete tooth measurement



Spot Measurement

1 shot	3 shots
Spectrophotometer	Spectrophotometer
45/0 geometry	0/0 geometry

Figure 1. *Comparison between the two CMDs.*

In this equation the ΔL^*, Δa^*, and Δb^* are the mathematical differences between the ES and the SS L*a*b* values. In previous studies it was shown that color differences between 2.0 and 3.7 were visually detectable under clinical conditions, but they resulted in an acceptable color difference[1,12] Therefore, in this study clinically relevant data were obtained by counting the number of cases of $\Delta E > 2.0$ and $\Delta E > 3.7$, respectively.

2. To ensure that the measurements of both devices were comparable, the relation between their individual measurements at the L*, a* and b*

Table 1: *CIE L*a*b* Translation Table of the Vita Shade Guide*

	L*	a*	b*
A1	79.6	–1.6	13.1
A2	76.0	–0.1	16.7
A3	75.4	1.4	19.6
A3.5	72.3	1.5	21.8
A4	68.6	1.6	21.0
B1	78.9	–1.8	12.3
B2	76.7	–1.6	16.6
B3	74.1	0.5	22.3
B4	71.8	0.5	22.2
C1	74.2	–1.3	12.6
C2	71.0	–0.2	16.7
C3	68.8	0.0	16.7
C4	64.8	1.6	18.7
D2	75.3	–0.5	13.5
D3	72.6	0.6	16.1
D4	71.9	–1.0	17.8

levels was evaluated by comparing the obtained L*, a*, and b* values of both instruments in a linear regression analysis.

3. As differences in the outcome of the L*a*b* measurements per spectrophotometer might be taken into account in the internal software to suggest an appropriate color in Vita shades, the coinciding suggested color codes were also compared. In order to quantify this data, the coinciding suggested Vita codes given by the devices were changed into the L*a*b* values, by using the referential values to shade guide codes as published by O'Brien and others[12] (Table 1). The color differences (ΔE*) between the derived values of the Vita Classical shade guide for the two devices were also calculated according to Equation 1.

Statistical Analysis

Initially, the mean color differences between direct comparison of CIE L*a*b* values and the derived CIE L*a*b* values of the Vita Classical shade codes were analyzed with one-way analysis of variance (ANOVA). Analysis showed that the mean color difference was not normally distributed, and analysis based on Gauss distributions was not justified. Therefore, a nonparametric Mann-Whitney U test and Kruskal-Wallis one-way ANOVA on ranks with post hoc Tukey (p=0.05) were used to evaluate different data. The data sets of L*, a*, and b* values of equal specimens measured by the two different devices were subjected to a linear regression model to analyze the correlation between the obtained values of the ES and the SS. The software used for this purpose was SigmaStat 3.1 (Systat Software, Inc, Richmond, CA, USA).

RESULTS

The mean CIE L*a*b* values (n=918) obtained after measuring the three locations per tooth of three teeth in each of 102 subjects with the ES and SS, respectively, are summarized in Table 2. The mean color difference (ΔE) for all L*a*b* measurements between the two devices was 12.1 (3.0), and the medians and quartiles are summarized in Table 3.

The Vita Classical shade guide codes were converted to CIE L*a*b* parameters, and the color differences between them were calculated for both devices according to Equation 1 (see Table 1). The mean color difference (ΔE*) was 3.1 (3.3), and the medians and quartiles are summarized in Table 3. Mann-Whitney U test showed that the mean differences between CIE L*a*b* values and the converted CIE L*a*b* values of the Vita Classical shades were significantly different (T=1262876;

Table 2: *Mean CIE L*a*b* Values (Standard Deviations) Obtained for Nine Locations in 102 Subjects Measured with the Easyshade and the SpectroShade-Micro*

	L*	a*	b*
Easyshade	71.6 (2.8)	6.7 (1.6)	20.8 (3.0)
SpectroShade-Micro	80.9 (4.2)	0.1 (0.1)	26.6 (5.8)

Table 3: *Means (Standard Deviations), Medians, and Quartiles of the Calculated E Values by 3 Different Evaluation Methods Obtained for 918 Measured Locations and Number of Clinical Cases with E Cut-off Values of 2.0 and 3.7*

	ΔE (L*a*b*)	ΔE (Vita)	ΔE (Vita) Cervical	ΔE (Vita) Middle	ΔE (Vita) Incisal
Mean	13.7 (2.9)	3.1 (2.1)	3.1 (2.2)	3.1 (2.1)	3.0 (2.0)
Median	13.9	3.3	3.2	3.5	3.2
25%	12.0	1.5	1.5	1.5	1.5
75%	15.5	4.6	4.6	4.6	4.6
$\Delta E < 2.0$	0%	40.0%	41.0%	40.0%	39.7%
$\Delta E < 3.7$	0%	55.1%	55.4%	53.4%	57.0%

$p<0.001$). Furthermore, the converted CIE L*a*b* values of the Vita Classical shades per region (cervical, middle, and incisal) are summarized in Table 3. Statistical analysis showed that there were no significant differences between the three measured regions ($H=0.096$; $p=0.953$).

The linear regression plots are depicted in Figure 2, and the obtained formulas and their correlation coefficients summarized in Table 4.

DISCUSSION

The purpose of this study was to reveal whether it is possible to compare color data between two different spectrophotometers. The results show that when L*a*b* values for the same tooth area are considered, both instruments differ to such an extent that in no case was comparable color data obtained. However, the best results were achieved when the Vita code suggestions were compared. When the suggested Vita shades were compared, in 40% of the cases both devices gave an equal suggestion, and a total of 51% resulted in clinically acceptable suggestions. The fact that 49% of the cases led to an unacceptable color measurement between the two devices can be interpreted as a poor result. However, in comparison to *visual* shade determination, where shade selections matches in only 26.6% of cases[3] this might be interpreted as a valuable contribution in dental color selection.

In the present study, the data were collected by the ES, which can be categorized as a spot-measurement device, and by the SS, which is a complete-tooth-measurement device. It has been stated that the data collected by spot-measurement devices may not be entirely accurate because of the non-homogenous shade structure of the tooth, the increased potential for tooth dehydration, and errors in image capture.[16] The color measurement of the exact same spot on a curved tooth surface can also prove to be challenging, which may affect the consistency of the measurements (Figure 1).[17] However, one study explains that the spot measurements in particular are more accurate because measurements are made with the tip of the probe.[18] In contrast, in devices such as SS, software calculations of an average value for the three tooth areas may decrease accuracy of measured color. On the other hand, in contrast to the ES, the complete tooth measurement with SS presents a topographical color map of the entire tooth in only one image, making the color readings from different areas much more consistent (Figure 1). The color of human teeth has a specific distribution pattern according to the different regions of the tooth surface[19] (segment relation in color from cervical to middle and incisal). These relations in color have been established by use of a digital camera, which recorded images of the whole tooth.[20] Looking at Table 3, it is evident that the three different tooth regions do not influence the measurements taken by the two devices.

When evaluating the regression analysis between the two devices, the correlation coefficients of the L*a*b* values are independently so low that further analysis was not considered. On average, the SS assessed higher b* values than the ES (Figure 2), meaning that the SS determines the color of a tooth as being more yellow than the ES indicates. On the other hand, the ES constantly measured a much higher a* value for the same spot than the SS (Table

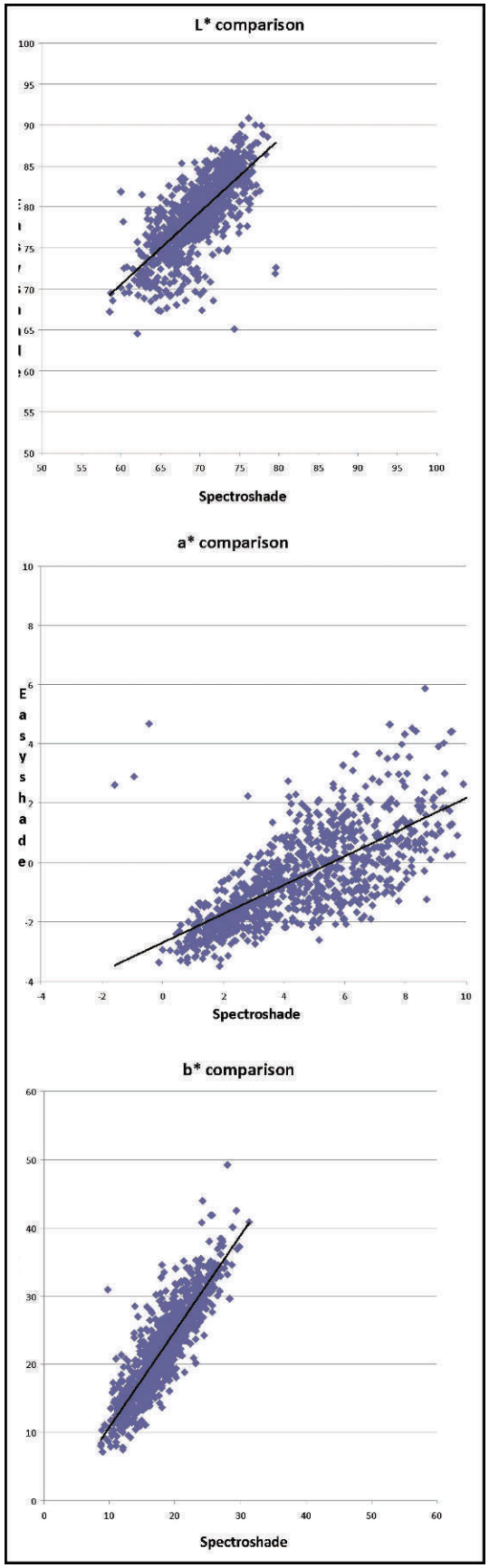


Table 4: *Regression Formulas and Correlation Coefficients for L*a*b* Data Exchange Between the Easyshade and the SpectroShade-Micro*

Color Coordinate	Regression Formula	Correlation Coefficient
L*	$L^*_{Easyshade} = L^*_{SpectroShade} * 0.88 + 17.6$	$R^2 = 0.51$
a*	$a^*_{Easyshade} = a^*_{SpectroShade} * 0.49 - 2.7$	$R^2 = 0.50$
b*	$b^*_{Easyshade} = b^*_{SpectroShade} * 1.41 - 3.3$	$R^2 = 0.76$

2), which means that the same tooth is determined as being more red with the ES. Such results could be attributable to the fact that the ES and SS have different optical geometries and that they irradiate tooth surface in different ways. The SS irradiates the tooth at an angle of 45 degrees, and the detector receives the reflected light from the tooth at the location of 0 degree. The ES irradiates the tooth surface and receives the reflected light at 0 degree. Therefore, the area of irradiation is smaller with the ES than with the SS. In previous studies it was shown that the CIE L*a*b* values, which use a smaller irradiated area, are shifted toward green and blue and to lower brightness relative to the actual color coordinates.[17,21,22] This means that the ES should have lower L*, a* and b* values than the SS. The results of this study are in agreement with these findings for L* and b* but not for a*. This might be attributable to fact that the optical geometries are different, but this has not been studied previously. The origin of the fact that CIE L*a*b* values depend on the irradiated area, could be the wavelength-dependent edge loss that occurs by small area colorimeters and spectrophotometers. It has been shown that the edge loss for green light is approximately 85% of the edge loss for red light, and this effect could have been decreased by using larger measuring areas.[17]

The corresponding CIE L*a*b* values of the Vita shade tabs originate from the study of O'Brien.[12] Although other reports have described absolute values of the Vita Classical shade tabs,[18,23-25] only O'Brien actually described the results as CIE L*a*b*

←
Figure 2. *The linear regression plots of the L*, a*, and b* values of the Easyshade and the SpectroShade obtained at 918 different points.*

values. The fact remains that even different shade guides from the same manufacturer are not identical,[26,27] which means that the CIE L*a*b* values used in this present study are specific for this study only.

One can assume that one of the reasons for the findings in this study could be the fact that the evaluation of a CMD in the oral environment is very complex. Many handling errors with the different instruments could play a role in the results. In a clinical setting, the instruments can be sensitive to the patient's movement, fogging, the angle and position of the probe, different inclinations, and different shapes of the teeth. Moreover, the accuracy of the incorporated light source can change over time, influencing the measured values.[8]

Although the L*a*b* values are absolute and standardized, they were not interchangeable between the two investigated devices. This means the dentist and the dental laboratory that work together are obliged to use the same device to communicate color between them. Manufacturers of these devices should consider putting more effort into standardization to improve the reproducibility of the data in clinical circumstances.

CONCLUSIONS

The color values (L*a*b*) of teeth, measured with two different spectrophotometers, were not comparable in this study. Therefore, the exchange of the L*a*b* values between two spectrophotometers cannot be recommended.

The two devices match each other better when the output of the tooth color is given as the closest corresponding shade tab according to the device's database. This is because the devices automatically select the closest color match from an internal database of Vita shade codes.

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Laboratory Research

Proximal Marginal Overhang of Composite Restorations in Relation to Placement Technique of Separation Rings

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Marie-Charlotte DNJM Huysmans

Clinical Relevance

A direct contact of the tines of the separation ring with the outer surface of the tooth to be restored will reduce the amount of flash/overhang formation.

SUMMARY

The aim was to investigate *in vitro* the marginal overhang in Class II composite restorations placed with various separation rings and placement techniques. A total of 180 Mesial-Occlusal [MO] preparations in artificial molar teeth were divided into nine groups (n=20). After placement of the sectional matrix, one of three ring systems was applied: Contact Matrix System (Danville Materials), Composi-Tight Gold (Garrison), and V-Ring (Triodent). In each group, rings were placed according to four different techniques (V-Ring placed with technique no. 2 only): 1) occlusally of the wedge, 2) on back end of the wedge, 3) between adjacent tooth and wedge, and 4) between treated tooth and wedge.

After application of the adhesive resin, preparations were restored with composite Clearfil AP-X (Kuraray) and polymerized in increments. After matrix removal, overhang was measured on a standardized digital macroscopic image in millimeters squared. For analysis, analysis of variance and Tukey B were used ($p<0.05$).

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For the Contact Matrix System and Composi-Tight Gold ring, the different placement techniques had a statistically significant effect on the amount of marginal overhang ($p<0.031$). The V-Ring resulted in the least marginal overhang ($p<0.001$).

None of the placement techniques and separation rings could completely prevent marginal overhang, and the placement technique has a significant influence on its occurrence.

INTRODUCTION

A dental restoration should restore form, function, and esthetics of a tooth involved and therefore prevent the occurrence of recurrent caries and periodontal diseases. Studies have shown that bulky and irregular overhanging restorations may promote periodontal diseases due to local accumulation of bacterial plaque rather than mechanical irritation. Epidemiological and clinical experimental studies have demonstrated close associations between such iatrogenic factors and the pathogenesis of local periodontal lesions.[1-7]

Despite all efforts and available techniques, placing a Class II composite restoration will result in various degrees of marginal overhang.[8-11] Circumferential matrix systems resulted in the least marginal overhang compared with sectional matrix systems, and the use of a stiffer matrix band resulted in significantly less marginal overhang compared with dead-soft matrix bands.[11] In addition, plastic matrices, considered necessary in the past, showed significantly more overhang compared with the metal bands.[9]

In order to reconstruct tight proximal contacts with Class II composite restorations, there has been a shift from circumferential to sectional matrices. These newer, precontoured sectional matrices combined with separation rings resulted in a better proximal contour[12] and tighter proximal contacts, compared with circumferential systems, in which flat matrices and no additional separation rings were used.[13]

Among different brands of separation rings, a large variety exists in the configuration of the tines (parallel, divergent, V-shaped), resulting in a different adaptation of the matrix band to the tooth surface. Furthermore, several placement techniques are applicable to keep these rings properly in place. For example, in case the clinical crown height is too short or the box is relatively wide in the buccal-lingual direction, it can be difficult or even impossible to place the ring occlusally from the wedge due to insufficient retention. A technique to provide more retention is placing the ring between the wedge and tooth surface.

As a result, each specific clinical situation offers the clinician several opportunities to place the matrix band and separation ring. However, it is unknown how the different ring configurations and positioning techniques affect the occurrence of marginal overhang. Therefore, the aim of this study was to investigate *in vitro* the marginal overhang in Class II composite restorations placed with different separation rings and ring placement techniques.

MATERIALS AND METHODS

For this study an mesial-occlusal [MO] preparation was made in artificial left first molars in the lower jaw (tooth no. 36), with the following dimensions: 5.0 mm in the bucco-lingual, 6.0 mm in the occlusal-gingival, and 1.3 mm in the mesial-distal direction. The occlusal step was 4.5 mm in buccal-lingual width, 2.5 mm deep, and 6.0 mm in mesial-distal width. The margins of the box were 1 mm supragingivally and finished butt-joint. This model was replicated using a copy-milling machine (Celay, Mikrona Technologie AG, Spreitenbach, Switzerland), resulting in 180 identical preparations. Teeth were placed in a manikin model (KaVo Dental, Biberach, Germany) and apically equipped with a stem-like anchoring system that allowed a standardized mobility of the tooth, simulating the normal physiological tooth mobility. Teeth were divided into nine different groups (n=20), each assigned to a specific ring and placement technique. A flexible sectional matrix band (Contact Matrix, Stiff Flex, Danville Materials, San Ramon, CA, USA) was placed and secured interdentally from the buccal side with a wooden wedge (Slim-Jim, Wizard Wedge, Waterpik Technologies, Ft Collins, CO, USA), after which one of three ring systems was placed. The Contact Matrix System (Danville Materials) and the Composi-Tight Gold ring (Garrison Dental Solutions, Spring Lake, MI, USA) were placed according to one of four placement techniques:

1) *Occlusally of the wedge*. On the buccal and lingual side the ring was placed occlusally of the wedge while both tines were in contact with the treated tooth as well as the adjacent tooth.
2) *On back end of the wedge*. On the buccal side the ring was placed on the back end of the shortened wedge, and both tines were in contact with the treated tooth as well as the adjacent tooth. The tine at the lingual side was placed according to technique no. 1.

Table 1: *Materials Used in the Study*

Materials	Characteristic	Manufacturers	Lot
Contact Matrix (Stiff Flex)	Sectional, flexible, and precontoured (0.05 mm)	Danville Materials, San Ramon, CA, USA	89434
Composi-Tight Gold (AU 400)	—	Garrison Dental Solutions, Spring Lake, MI, USA	18884370032
Contact Matrix ring (outward rings)	—	Danville Materials, San Ramon, CA, USA	89507
V-Ring	—	Triodent LTD, Katikati, New Zealand	3081
Wizard wedges (Slim-Jim)	—	Waterpik Technologies, Ft Collins, CO, USA	1672
PFI 49	—	Hu-Friedy, Chicago, IL, USA	—
Clearfil Photo Bond (Catalyst & Universal)	Catalyst: bisphenol A diglycidyl methacrylate (Bis-GMA), 10-methacryloyloxydecyl dihydrogen phosphate (MDP), 2-hydroxyethyl methacrylate (HEMA), hydrophobic dimethacrylate, dibenzoyl peroxide, dl-camphorquinone. Universal: N, N-di-ethanol-p-toluidine, sodium benzene sulfinate, ethanol	Kuraray Medical, Osaka, Japan	41164
Clearfil AP-X (PLT, color A3)	Barium glass and colloidal silica filler, 85.5 wt%, 70 vol%	Kuraray Medical, Osaka, Japan	0068A

3) *Between adjacent tooth and wedge*. On the buccal side the tine was situated next to the wedge at the side of the adjacent tooth, resulting in contact only with the adjacent tooth surface. The tine at the lingual side was placed according to technique no. 1.
4) *Between treated tooth and wedge*. On the buccal side the tine was placed next to the wedge at the side of the treated tooth, resulting in contact of the tine only with the treated tooth. The tine at the lingual side was placed according to technique no. 1.

Due to its configuration, the V-Ring (Triodent, Katikati, New Zealand) could only be placed according to technique no. 2 (on the back end of the wedge).

Table 1 summarizes the product profiles, lot numbers, and the characteristics of the materials used in the study. In Figure 1 the three ring systems are presented.

In all groups, after placement of the separation ring, the contact area of the matrix band was burnished with a hand instrument (PFI 49, Hu-Friedy, Chicago, IL USA) so contact was present between matrix and adjacent tooth. All cavities were restored with an adhesive and a hybrid composite (Clearfil Photo Bond and Clearfil AP-X, Kuraray Medical, Tokyo, Japan). The dual-cure adhesive system was mixed and applied in the preparation, gently air-dried, and cured for 10 seconds with a halogen polymerization unit (PolyLux II, KaVo; light intensity 600 mW/cm^2). Subsequently, the composite was injected from the preloaded tip into the cavity in three horizontal increments of 2 mm thick and adapted to the cavity walls using a hand instrument (ASH 49). Each increment was cured separately for 20 seconds from the occlusal surface. After removal of the matrix, restorations were postcured for 20 seconds from the buccal and lingual sides. Restorations were not finished or adjusted in order to prevent changes of the proximal surface. All restorations were placed in a random order by one operator, and all measurements were performed blind by an independent observer.

Figure 1. *The three ring systems used in this study. The Contact Matrix System (outward ring) of Danville Materials, the Composi-Tight Gold (AU400) of Garrison Dental Solutions, and the V-Ring of Triodent.*

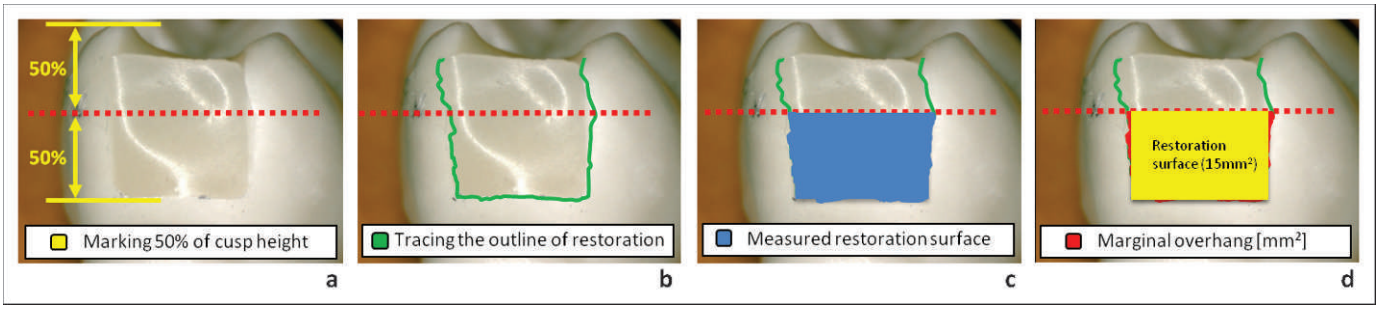

Figure 2. *Procedure for measuring the proximal marginal overhang using a stereomicroscope (7.89×). (a): Only the marginal overhang in the cervical area of the box was measured (50% box height). (b): Digital tracing of the restoration margin. (c): Showing the measured area of the restoration surface. (d): The surface of the preparation (15.0 mm^2) was subtracted from the marked surface, resulting in the marginal overhang.*

Following the restorative procedure each tooth was removed from the manikin model and placed with the box surface horizontal in a mold made of polyvinylsiloxane (Express Putty STD, 3M ESPE, Dental Products, Seefeld, Germany). With a stereomicroscope (Leica MZ 12) standardized digital images were made of the proximal surface with a magnification of 7.89×. Leica Qwin software was used to measure digitally the total proximal restoration surface (millimeters squared) by marking the margin of the restoration on the digital image (Figure 2). Only the cervical area of the box was included (50% box height: 3.0 × 5.0 mm, as previously described by Loomans and others[11]). Because all preparations were identical, it was possible for the software to automatically mark the cutoff point on the 50% box height and to include only the area beneath this cutoff line. Finally, the outer surface of the preparation (15.0 mm^2) was subtracted from the total restoration surface, resulting in the marginal overhang surface area. The marginal overhangs for the buccal and the lingual parts of the box were recorded separately in order to investigate the effect of ring positioning. Data were statistically analyzed using SPSS 14.0 (SPSS Inc, Chicago, IL, USA). To determine differences between the placement techniques for the Contact Matrix System (Danville Materials) and Composi-Tight Gold (Garrison) and to determine differences between the three separation rings at the "on back end" location, one-way analyses of variance (ANOVA) was performed, followed by the post hoc multiple comparison Tukey B ($p<0.05$).

RESULTS

Mean marginal overhang of the restorations is presented in Figure 3. For the total overhang, the values ranged between 0.81 mm^2 and 1.32 mm^2. Data were normally distributed and the measure-

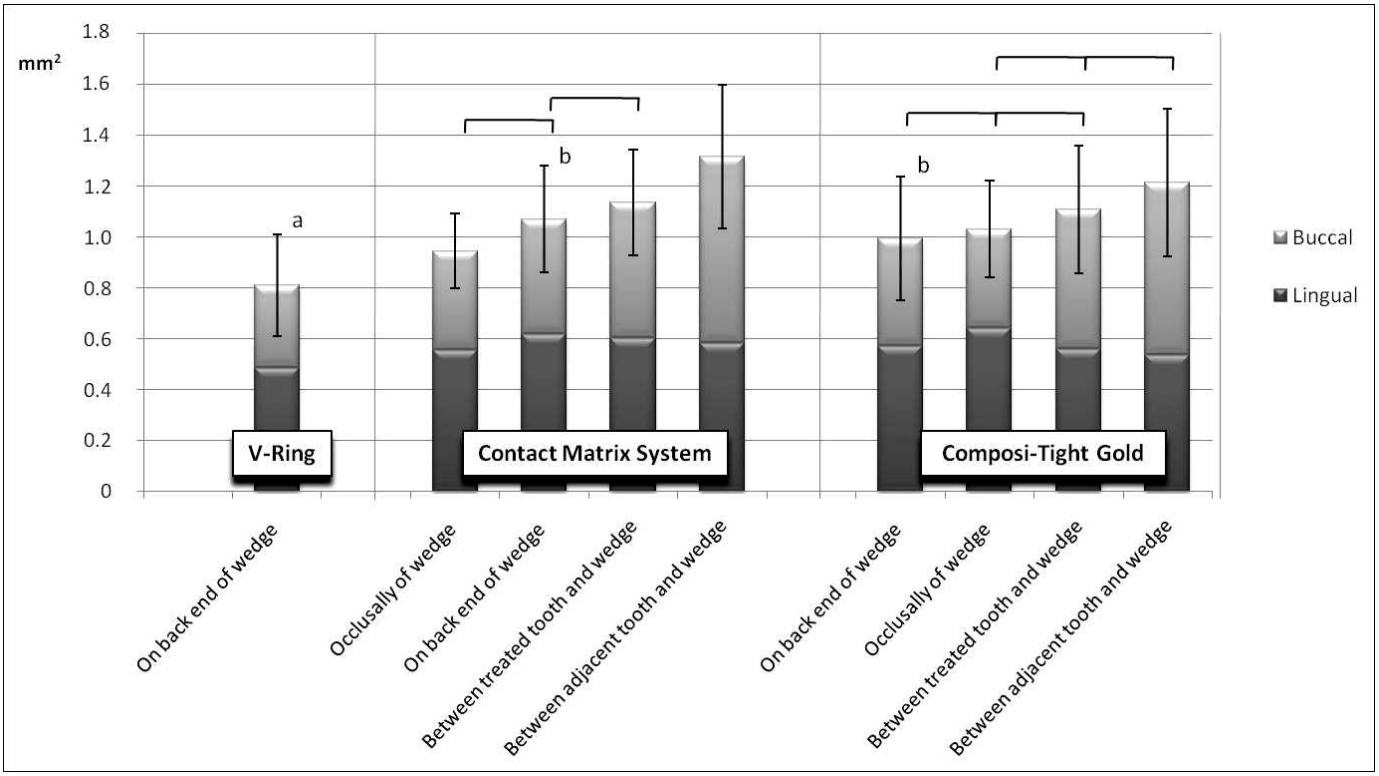

Figure 3. *Recorded proximal marginal overhang (divided by buccal and lingual) of composite (mm^2) together with the standard deviation (SD) of the total overhang, combined with an indication of statistical differences between groups (ANOVA:* $p<0.05$*). Different letters indicate statistically significant differences: only comparing the technique "on back end of wedge" (ANOVA:* $p<0.05$*).*

ment error was established by remeasuring five random specimens eight times, for an average measurement error of 0.0014 mm^2, varying between 0.0007 and 0.0024 mm^2. This resulted in a measurement error of less than 1% of the total restoration surface.

Statistically significant differences were found in the total amount of proximal overhang between the placement techniques for the Contact Matrix System (ANOVA: $p<0.001$) and Composi-Tight Gold (ANOVA: $p=0.031$). Post hoc tests demonstrated that the largest total amount of marginal overhang in the Contact Matrix group was found in the ring placed "between adjacent tooth and wedge" (1.32 ± 0.28 mm^2), which was statistically significantly larger than those placed according to the "between treated tooth and wedge" (1.14 ± 0.21 mm^2), "on back end" (1.07 ± 0.21 mm^2), and "occlusally" (0.95 ± 0.15 mm^2) techniques. No statistically significant difference was found between the "occlusally" and "on back end" techniques, nor between the "on back end" and "between treated tooth and wedge" techniques.

The post hoc tests for the Composi-Tight Gold system revealed that the largest total amount of marginal overhang was recorded for "between adjacent tooth and wedge" (1.22 ± 0.29 mm^2), which was statistically significantly different only from the "on back end" (1.00 ± 0.24 mm^2) technique. No statistically significant difference was found between the "occlusally" (1.03 ± 0.19 mm^2) and "between treated tooth and wedge" (1.11 ± 0.25 mm^2) placement techniques.

The lingual overhang formation was similar for all placement techniques regarding the Contact Matrix System (ANOVA: $p=0.365$) and Composi-Tight Gold ring (ANOVA: $p=0.083$). At the buccal side, statistically significant differences were found for both ring systems (ANOVA: $p<0.001$). Post hoc tests showed that the largest overhang formation was found with the "between adjacent tooth and wedge" placement technique for both the Contact Matrix System (0.73 ± 0.22 mm^2) and Composi-Tight Gold ring (0.70 ± 0.25 mm^2), which was statistically significantly larger than the other placement techniques.

When comparing the three ring systems for the total amount of marginal overhang using the "on back end" technique, the V-Ring (0.81 ± 0.20 mm^2)

produced the least total overhang compared with the Composi-Tight Gold ring (1.00 ± 0.24 mm^2) and Contact Matrix System (1.07 ± 0.21 mm^2) ($p<0.001$). At the buccal side also, the V-Ring (0.32 ± 0.13 mm^2) produced the least total overhang compared with the Composi-Tight Gold ring (0.43 ± 0.13 mm^2) and Contact Matrix System (0.45 ± 0.15 mm^2) ($p<0.02$). At the lingual side, a statistically significant difference was found only between V-Ring (0.49 ± 0.13 mm^2) and Contact Matrix System (0.62 ± 0.13 mm^2) ($p=0.011$).

DISCUSSION

In this study, the influence of several placement techniques of three different separation rings on the occurrence of proximal marginal overhang in Class II composite restorations was investigated. It was found that both the placement technique and type of ring had a statistically significant effect on the amount of marginal overhang.

The methodology for the present study has already been described in a previous study,[11] and as in that study, the measurement of the overhang was restricted to the cervical area, 50% of total box height of the restoration, because the overhang in this region is very difficult or even impossible to remove. The advantage of this *in vitro* model is that it gives controlled experimental conditions; however, it fails to account for the complexities of factors that are found under *in vivo* conditions.

Regarding the total amount of proximal overhang, the use of the V-Ring resulted in the least overhang compared with the Composi-Tight Gold and Contact Matrix System. This may be explained by the V-configuration of the tines in the buccal-lingual direction, leading to a better adaptation of the matrix to the tooth compared with the other systems. The advantage of this system is that the ring is placed in one very stable position; at the same time this is a disadvantage, because ring position cannot be adjusted. In a majority of clinical situations this V-Ring may be used; however, it may fail in cusp replacement situations or when clinical crown height is low, because remaining cusps of sufficient height are needed for retention. The more "basic" rings, such as BiTine ring (Dentsply Caulk, Milford, DE, USA), Composi-Tight Gold, and Contact Matrix System, offer the clinician more freedom in placement technique.

The marginal overhang was analyzed separately for buccal and lingual surfaces. Lingual overhang was comparable for all placement techniques for each ring system, which could be expected because the tines at the lingual side were always placed identically. At the buccal side, tines were placed in four different ways, resulting in statistically significant differences in marginal overhang. Tines placed "occlusally" and "on back end" resulted in the least buccal overhang, whereas tines placed "between adjacent tooth and wedge" resulted in a statistically significant larger overhang. In the situation of rings placed "occlusally" and "on back end," tines are in contact with the treated as well as the adjacent tooth. These techniques seemed to provide the best adaptation of the matrix to the tooth, thus preventing marginal overhang. Tines placed "between treated tooth and wedge" contacted only the treated tooth, resulting in pressure of the ring against the wedge away from the treated tooth, which led to a gap at the cervical area. Tines placed "between adjacent tooth and wedge" caused pressure against the wedge toward the treated tooth resulting in a good cervical adaptation. In this last situation, adaptation of the matrix to the tooth is compromised toward the occlusal side, resulting locally in a gross overhang. However, in this area excess material can be removed easily using finishing discs.

All techniques in this study resulted in some marginal overhang, and this finding is in accordance with previous studies.[8-11] It may be concluded that a complete prevention of proximal overhang is hardly achievable. Therefore, a major issue remains about how to deal with marginal overhang in composite restorations. An overhang at the buccal or lingual margin is relatively easily removed with the use of polishing discs (eg, Sof-Lex, 3M ESPE) or a special oscillating diamond finishing tip (Profin, Dentatus, or EVA, KaVo).[14] However, overhang at the cervical proximal margin is difficult to remove without damaging the tooth and/or adjacent tooth surface.

Most studies about marginal overhang are related to amalgam restorations, and the conclusions from these studies may not be generally applicable for composite restorations.[1-7] The clinical relevance of a marginal overhang might depend on the size, shape, and clinical appearance (smooth or rough and bulky) of the restoration. Rough and bulky outlines or the presence of gross overhang might result in accumulation of plaque and irritation of the epithelial attachment and might require replacement or repair of the restoration. For marginal overhang that is adhesively attached, due to the spread of bonding, and is smooth and continuous (a "flash"), no direct clinical need of removal might exist; this flash might be regarded as an extended bevel. However, the true

clinical relevance and effect of such a proximal overhang on periodontal condition is unknown.

CONCLUSIONS

- None of the placement techniques and separation rings could completely prevent marginal overhang.
- The placement technique of the separation ring has a statistically significant influence on the occurrence of marginal overhang.

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Reparability of Aged Silorane With Methacrylate-Based Resin Composite: Micro-Shear Bond Strength and Scanning Electron Microscopy Evaluation

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Clinical Relevance

Aged silorane composite restorations can be repaired with a methacrylate-based resin composite by using a phosphate-methacrylate–based adhesive as the intermediate layer.

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SUMMARY

Objectives: To evaluate the compatibility between aged siloranes and methacrylate-based composites by simulating a common repair-technique.

Methods: Twenty substrates were constructed using silorane (Filtek Silorane, 3M ESPE) and methacrylate composites (Filtek Supreme XT, 3M ESPE). Substrates were aged in 0.9% NaCl solution at 37°C for 72 hours. Silorane build-ups were constructed on silorane substrates without any intermediate layer (IL). Methacrylate build-ups were constructed on silorane substrates without any IL, with a methacrylate IL (Heliobond, Ivoclar Vivadent), or with a phosphate-methacrylate IL (Silorane System Adhesive Bond, 3M ESPE). Methacrylate build-ups were also constructed on methacrylate

substrates without any IL. The micro-shear bond strength test was carried out after thermocycling. Bond strength data were statistically analyzed using analysis of variance and Tukey post hoc tests. Failure modes were assessed by means of scanning electron microscopy observations.

Results: The silorane-methacrylate group without any IL showed the lowest bond strength values (0.4 ± 0.1 MPa). The use of a methacrylate-based IL (1.6 ± 1.7 MPa) led to a slight increase in bond strength, whereas the use of phosphate-methacrylate IL (9.1 ± 5.4 MPa) significantly increased bond strength. There was no statistically significant difference in bond strength between silorane-silorane (7.9 ± 3.6 MPa) and methacrylate-methacrylate (9.5 ± 4.1 MPa) groups without any IL.

INTRODUCTION

The use of composite resin restorative materials has been widely accepted in dental practice.[1] However, although methacrylate-based composites exhibit acceptable clinical performance, polymerization shrinkage is still a drawback.[2] Polymerization shrinkage results in volumetric contraction, causing stress in bonded restorations that can lead to clinical failure.[3-7] Recently, a new category of polymers for dental-restorative use was introduced: silorane-based composites. Polymerization of silorane-based composites occurs through a photocationic ring-opening reaction, which results in a lower polymerization contraction compared with the free radical polymerization of dimethacrylate monomers.[8,9] The volumetric shrinkage of the silorane composite was determined to be 0.9%, which is clearly the lowest value observed for the investigated materials.[9] This is in good agreement with stress measurements by Ernst and others,[10] showing the lowest stress development for siloranes among all tested composite materials.

According to the manufacturer of Filtek Silorane (3M ESPE, St Paul, MN, USA), which is the only silorane-based marketed composite, silorane composite resin can be used for direct class I and class II restorations and as a base under a methacrylate-based composite. In fact, siloranes can have an important role as a base under methacrylate composite resin in what is commonly referred to as a sandwich restoration. By replacing part of the methacrylate composite with silorane composite, it is possible to obtain lower shrinkage and consequently lower polymerization stress. In addition, more esthetically satisfactory results could be achieved by using methacrylate composite resin as an enamel restorative. According to the literature,[11,12] in order to stratify a methacrylate composite on a silorane composite, the use of a phosphate-methacrylate–based intermediate resin such as the second component (Bond) of the Silorane Adhesive System (3M ESPE) is required. The application of this hydrophobic resin coating promotes bonding not only to silorane-based composites but also to methacrylate-based materials.[13] The phosphate group reacts with oxirane, whereas the acrylate group reacts with dimethacrylate, thus resulting in the adhesion between the two composites.[11] Tezvergil-Mutluay[11] also demonstrated that the bond strength between consecutive layers of silorane composite decreased when the time of placement between consecutive layers increased. This suggests that as the chemical reactivity decays over time,[14] the bond strength could be affected. Consequently, another clinically interesting point to consider is the possibility of repairing an aged silorane restoration with a conventional methacrylate composite system. According to the manufacturer, silorane restorations can be repaired with a conventional methacrylate composite system using a dimethacrylate-based intermediate layer. On the other hand, according to the literature,[11,12] the use of a phosphate-methacrylate–based adhesive as an intermediate layer could be more appropriate. However, the studies by Tezvergil-Mutluay and others[11] and Lührs and others[12] were conducted on fresh substrates, while it has not yet been determined whether a phosphate-methacrylate–based adhesive should also be applied as an intermediate layer on aged silorane composite restorations.

Using the micro-shear bond strength test (μSBS) and scanning electron microscopy (SEM), the aim of this study was to evaluate the ability of silorane- and methacrylate-based aged composites to be repaired and to examine the compatibility between siloranes and methacrylate-based composites by simulating a common repair technique. The tested null hypotheses were 1) there is no difference in bond strength between silorane-silorane, methacrylate-methacrylate, and silorane-methacrylate combinations, and 2) similar bond strength develops between silorane-based and methacrylate-based composites regardless of the application of an intermediate bonding layer.

MATERIALS AND METHODS

The materials used in this study are listed in Table 1. A silorane composite (Filtek Silorane, A3 shade,

Table 1: *Chemical Composition and Batch Numbers of the Tested Materials*

Materials	Manufacturer	Batch No.	Material Composition
Filtek Silorane	3M, ESPE	7AU	1,3,5,7-Tetrakis (ethyl cyclohexane epoxy)
			1,3,5,7-tetramethyl cyclotetrasiloxanemethyl-bis[2-(7-oxabicyclo[4.1.0]hept-3-yl)ethyl]phenyl
Filtek Supreme	3M, ESPE	6FK	bis-GMA, Bis-EMA, TEGDMA, UDMA
Silorane System Adhesive Bond	3M, ESPE	7AJ	TEGDMA, Phosphoric acid methacryloxyhexylesters, 1,6-hexanediol dimethacrylate
Heliobond	Vivadent-Ivoclar	405316	bis-GMA, TEGDMA

Abbreviations: bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; bisGMA, bisphenol A-glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

3M ESPE, St Paul, MN, USA) and a methacrylate composite (Filtek Supreme XT, A3B shade, 3M ESPE) were used as substrate and adherent materials. As a substrate material, silorane was used in groups 1 to 4, whereas a methacrylate-based composite was used as a substrate in group 5 (Table 2). The substrates were fabricated by placing unpolymerized composite between two glass microscope slides. The material was then light polymerized for 30 seconds with a light-curing device (Astralis 10, High Power Program 1200 mW/cm^2, Vivadent-Ivoclar, Schaan, Liechtenstein). Thus, disks of about 15 mm in diameter and 1 mm in thickness were obtained. A 2-mm-diameter hole was produced near the margin of the disk using a hand piece and a parallelometer (CL-MF2002S, Heraeus-Kulzer Inc, Hanau, Germany) in order to allow for repeatable placement of the disk during the mechanical test. Substrates were aged in 0.9% NaCl solution in a light-proof container at 37°C for 72 hours and then randomly divided into five groups (n=4) according to the used method of substrate preparation (Table 2). One of the two surfaces of each disk was roughened for five seconds with P600-grit abrasive paper (WS Flex 18 C, Hermes Abrasives Ltd, Virginia Beach, VA, USA) under running water using a lapping machine (LS2, Remet, Bologna, Italy). Silorane build-ups were constructed on silorane substrates without any intermediate layer (IL; group 1). Methacrylate build-ups were constructed on silorane substrates without any IL (group 2), with a methacrylate IL (Heliobond, Ivoclar Vivadent; group 3) or

Table 2: *Descriptive Statistics of Micro-Shear Bond Strength and Distribution of Failure Modes*[a]

Group	Substrate	Intermediate Layer	Build-up	Number of Tested Specimens	Bond Strength (MPa) Mean ± SD	Fracture Mode		
						Adhesive	Cohesive	Mixed
1	F.Silorane	/	F.Silorane	27	7.9 ± 3.6[a]	12 (44%)	15 (56%)	—
2	F.Silorane	/	F.Supreme	4	0.4 ± 0.1[b]	4 (100%)	—	—
3	F.Silorane	Heliobond	F.Supreme	25	1.6 ± 1.7[b]	25 (100%)	—	—
4	F.Silorane	Silorane System Adhesive Bond	F.Supreme	30	9.1 ± 5.4[a]	19 (63%)	6 (20%)	5 (17%)
5	F.Supreme	/	F.Supreme	27	9.5 ± 4.1[a]	5 (18%)	22 (82%)	—

[a] *In the Bond Strength column, different superscript letters label statistically significant between-group differences* ($p<0.05$).

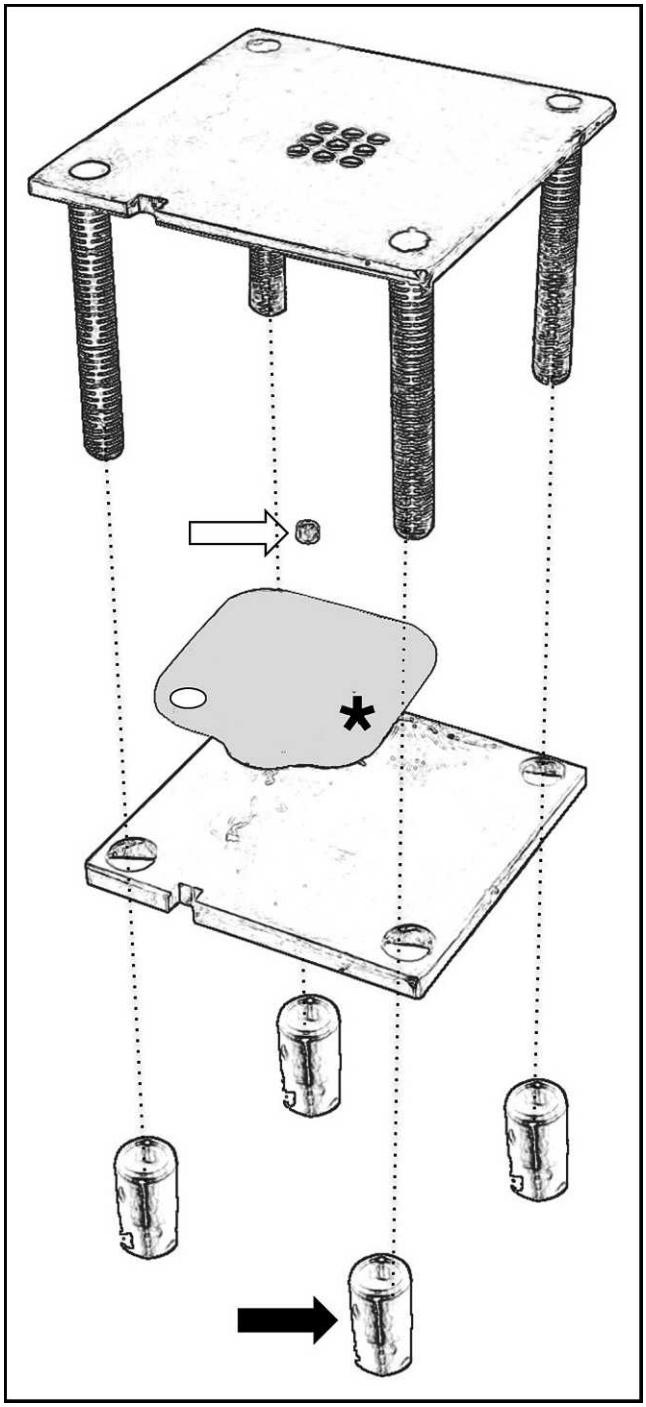

Figure 1. *Metal device for specimen construction. Silicone tubules (blank arrow) were embedded into the lower sections of the holes. A resin composite disc (asterisk) was placed between the two plates, which were then joined together with four screws (black arrow).*

with a phosphate-methacrylate IL (Silorane System Adhesive Bond; group 4). Methacrylate build-ups were also constructed on methacrylate substrates without any IL (group 5). The latter group was tested as a control.

The methacrylate IL and the silorane IL were applied to one surface of each disk by means of a micro brush, thinned with a light air stream, and light polymerized for 20 seconds.

The build-ups were constructed using a custom-made device (Figure 1), consisting of two parallel metal plates that could be joined by means of four screws. The upper plate had nine holes. Each hole was made of two sections: a lower section (1.7 mm in diameter) and an upper section (1.45 mm in diameter). Nine silicone tubules having an internal diameter of 0.7 mm, an external diameter of 1.7 mm, and a height of 0.5 mm were custom made from a silicone tube (art.30/07; Stonfo, Florence, Italy). The tubules were embedded into the holes of the lower section. Each resin substrate was placed between the two plates, which were then joined together with the four screws. The silicone tubules were then filled with the resin composite, which was subsequently light cured for 20 seconds. After opening the device, the silicone tubules were easily removed from the build-up (Figure 2). The built-up specimens were stored in 0.9% NaCl solution in a light-proof container at 37°C for 24 hours and then thermocycled (1500 cycles between 5°C and 55°C; 10-second dwell time in each 0.9% NaCl bath; LTC, LAM Technologies, Firenze, Italy). The specimens were observed under a stereomicroscope (40× magnification; SMZ-10, Nikon Corporation) to verify integrity at the build-up/substrate interface. Build-ups that showed apparent interfacial gap formation, bubble inclusion, or any other relevant defect were excluded from the study.

Specimens were subjected to a μSBS test using a universal testing machine (LMT-100, LAM Technologies). The specimen was placed on the testing machine by positioning the hole in the specimen around a pin on the moving part of the machine. The hole could freely rotate around the pin. A thin stainless-steel wire (diameter = 0.20 mm) was looped around a pin on the stationary part of the machine as well as around the resin build-up. The wire contacted half the circumference of the build-up and was gently held flush against the disk at the build-up–substrate interface. Thus, the wire loop exerted shear forces parallel to the bonded interface, at a crosshead speed of 1 mm/min until failure occurred. The μSBS was expressed in MPa by dividing the load at failure (N) by the surface area (mm^2).

Statistical Analysis of μSBS Data

As the data distribution was normal in each group (Kolmogorov–Smirnov test) and group variances

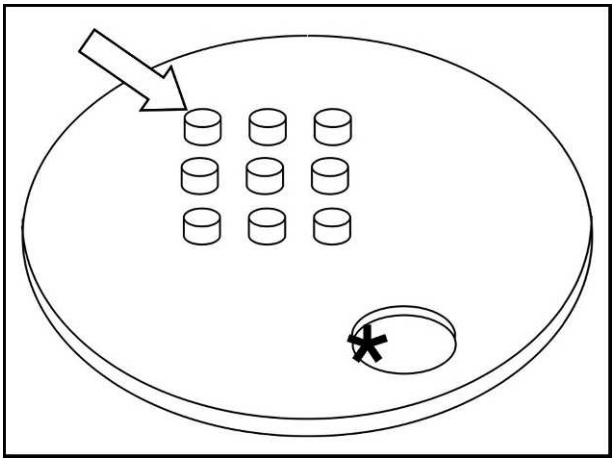

Figure 2. *A schematic representation of a specimen showing the hole near the margin of the resin composite disc (asterisk) and resin composite build-ups (blank arrow).*

were homogeneous (Levene test), the one-way analysis of variance (ANOVA) was applied, followed by the Tukey test for post hoc comparison. Between-group differences in the distribution of failure modes were statistically assessed using chi-square tests. In all the analyses, the level of significance was set at $p<0.05$ (SigmaStat 3.5 Statistical Software package, Systat Software Inc, San Jose, CA, USA).

Specimen Preparation for SEM Observation

Following the μSBS test, the substrates and the debonded build-ups were prepared for SEM analysis. The specimens were mounted on aluminum stubs with colloidal silver paint and sputter coated (SCD 005, BAL-TEC AG, Balzers, Liechtenstein) with 200-Å gold-palladium alloy (Foil Target AU, BAL-TEC AG). To determine the mode of failure, each specimen was observed under the SEM (Philips 515, Philips Co, Amsterdam, The Netherlands; 15-KV accelerating voltage) at 100× magnification. Images were acquired by means of a computerized program (Analysis 2.1, Soft Imaging System GmbH, Munster, Germany).

RESULTS

This study involved the preparation of nine build-ups on each substrate. Of 180 build-ups, 4 from group 1, seven from group 2, three from group 4, and three from group 5 either prematurely failed or accidentally detached from their base after opening the device. Under stereomicroscope observation, five build-ups from group 1, six build-ups from group 2, three build-ups from group 3, three build-ups from group 4, and six build-ups from group 5 showed

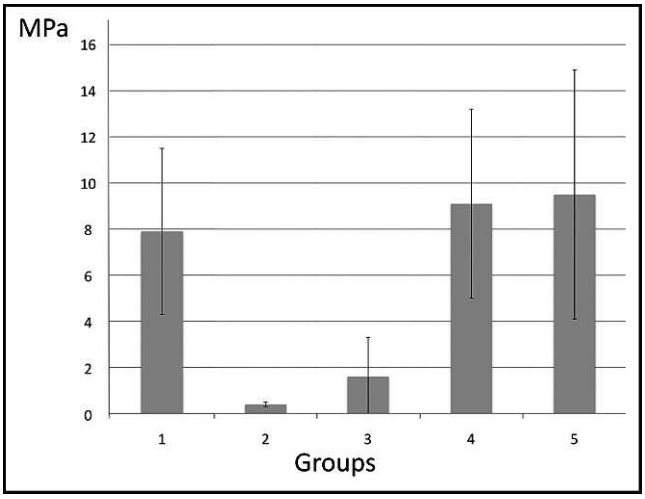

Figure 3. *The means and standard deviations of micro-shear bond strengths in different groups. Group 1: Silorane + Silorane. Group 2: Silorane + Supreme. Group 3: Silorane + Heliobond + Supreme. Group 4: Silorane + Silorane System Adhesive Bond + Supreme. Group 5: Supreme + Supreme.*

defects at the adhesive interface and were discarded. Nineteen build-ups from group 2 and eight build-ups from group 3 failed during thermocycling. Consequently, 127 build-ups (27 build-ups from group 1, four build-ups from group 2, 25 build-ups from group 3, 30 build-ups from group 4, and 27 build-ups from group 5) were subjected to μSBS testing.

μSBS Test

Descriptive statistics of μSBS are reported in Table 2 and Figure 3 along with statistically significant between-group differences. Measured bond strengths were (mean ± standard deviation) 7.9 ± 3.6 MPa for group 1, 0.4 ± 0.1 MPa for group 2, 1.6 ± 1.7 MPa for group 3, 9.1 ± 5.4 MPa for group 4, and 9.5 ± 4.1 MPa for group 5. The one-way ANOVA showed that groups differed significantly ($p<0.001$). In particular, the post hoc test revealed that the bond strengths measured in groups 1, 4, and 5 were significantly higher than those recorded in groups 2 and 3.

SEM Observation

SEM observation showed different fracture patterns among the groups (Figure 4).

All specimens of groups 2 and 3 failed adhesively. Both adhesive (44%) and cohesive (56%) failures were noticed in group 1, while failures were mostly adhesive (63%) in group 4 and mainly cohesive in group 5 (82%).

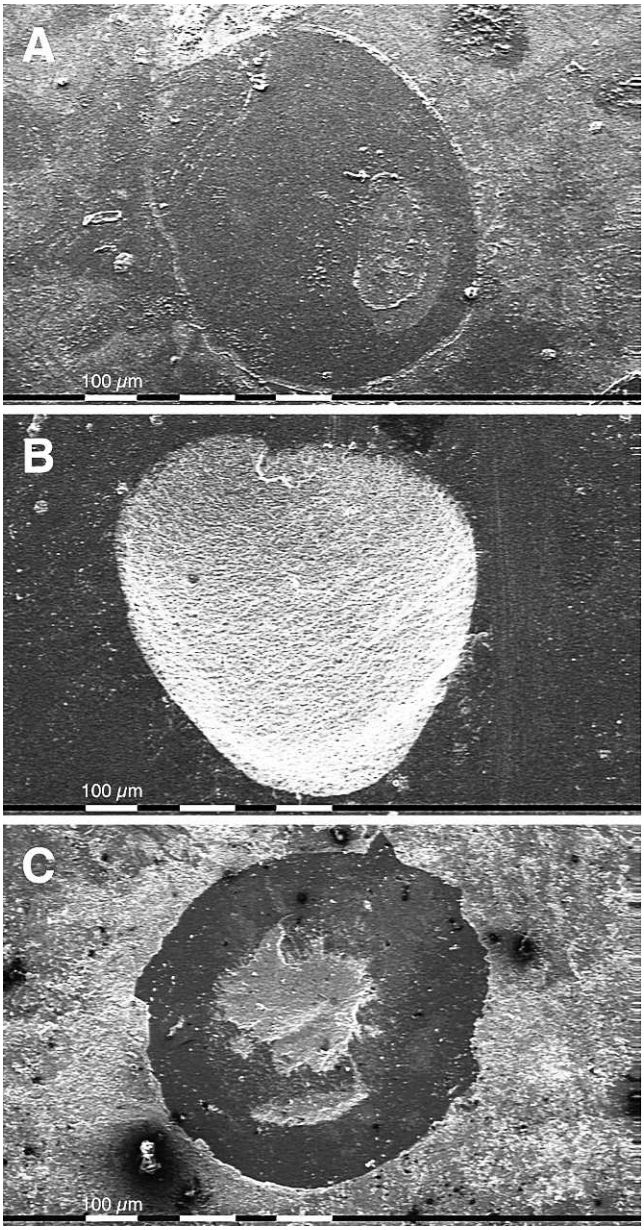

Figure 4. *Scanning electron microscopy microphotographs of the debonded surface of tested substrates (100×). (A): Adhesive failure in a silorane-silorane specimen (group 1). (B): Cohesive fracture of a methacrylate build–up, which was constructed on a methacrylate substrate (group 5). (C): Mixed fracture of a silorane build-up created on a methacrylate substrate with the interposition of a phosphate-methacrylate intermediate layer (group 4).*

The differences in failure mode distribution were statistically significant, with the exception of the comparison between groups 2 and 3, both of which exhibited exclusively adhesive failures.

DISCUSSION

This study was designed to evaluate the ability of an aged silorane-based composite to be repaired with methacrylate-based composite by simulating a common repair technique. Based on the study's findings, the first null hypothesis has to be rejected. As a matter of fact, the silorane-methacrylate group without any intermediate layer (group 2) exhibited significantly lower bond strengths than those recorded by the silorane-silorane and the methacrylate-methacrylate groups without adhesive layer (groups 1 and 5, respectively). Also, in group 2, the highest number of failures during thermocycling was recorded, and all the tested specimens failed adhesively at the silorane-methacrylate interface. No statistically significant difference in bond strength emerged between silorane-silorane (group 1) and methacrylate-methacrylate (group 5) groups. However, the two groups exhibited significantly different failure patterns. Specifically, group 5 had a predominance of cohesive failures. Also, the second formulated null hypothesis has to be rejected because the use of the phosphate-methacrylate–based adhesive as IL (group 4) significantly increased the bond strength. Statistically similar adhesion levels were instead obtained either with or without the application of a methacrylate-based IL (groups 3 and 2, respectively). The application of the phosphate-methacrylate–based IL also had an influence on specimen failure mode. It was indeed only with the interposition of this bonding material that mixed and cohesive failures occurred in silorane-methacrylate combinations. Therefore, according to the results of this study, the application of the phosphate-methacrylate–based adhesive as IL is beneficial when repairing an aged silorane-based composite with a conventional methacrylate-based composite.

The ring-opening reaction of the silorane is a cationic polymerization reaction, and no oxygen inhibition occurs on the polymerized surface.[11] Therefore, the bond between consecutive layers depends on the reactivity of the material.[11] Chemical reactivity is known to decrease over time.[14] As a result, the time interval between placement of consecutive silorane layers must not be overly long. A five-minute delay between layers results in significantly decreased bond strengths and increased percentage of adhesive failures.[11] However, the results of this study showed that the bond strength of an additional layer of silorane composite added to an aged silorane substrate is comparable to the bond strength of a layer of methacrylate composite added to an aged methacrylate resin substrate. On the other hand, the fact that the specimens in group 2 showed the lowest bond strength and the highest number of premature failures, when compared with

the other groups, suggests that there is no chemical compatibility between the aged silorane substrate and the added methacrylate resin composite. However, the use of Silorane System Adhesive Bond as an intermediate layer between the aged silorane composite and a methacrylate composite led to a significant increase in bond strength. Silorane IL is based on methacrylate chemistry with the addition of phosphate groups. The reaction of the phosphate group with oxirane and of the acrylate group with dimethacrylate might account for the recorded increase in bond strength.[11]

Numerous repair modalities have been evaluated *in vitro* for conventional methacrylate-based composites.[15-19] The treatment of methacrylate composite surfaces with a methacrylate bonding agent can be regarded as a standard procedure in today's dentistry. On the other hand, this study demonstrated that the application of a methacrylate-based IL is not beneficial when repairing an aged silorane-based composite with a conventional methacrylate-based composite. Therefore, it can be hypothesized that the repair modality for a silorane-based composite is different from that of a methacrylate-based resin composite. However, in clinical practice, the operator is blind to the type of composite resin that was originally used to restore the tooth. According to the literature,[20] Silorane System Adhesive can also be used to bond conventional methacrylate-based composites to dentin. However, the application of the Silorane System Adhesive Bond as an intermediate layer when repairing an aged methacrylate-based composite with a fresh methacrylate-based composite has not yet been tested. Further research should be carried out to allow clinicians to use Silorane System Adhesive Bond as an IL in reparations regardless of the type of composite used in the original restoration.

A μSBS test was carried out to measure the bond strength between silorane-based and methacrylate-based resin composites. This test represents a viable screening mechanism for predicting clinical performances and allows easier sample preparation as compared with other bond strength evaluation methods.[21] However, the bond strengths measured in this study were significantly lower when compared with bond strengths resulting from previous micro-shear bond test studies.[22-26] The sample preparation required for the μSBS test is not clearly and extensively described in literature.[23-34] In particular, it is not clear how the silicon tubules were held firmly on the dentin surface to prevent the resin from seeping away from the defined area at the base of the cylinder. Consequently, to avoid such inconvenience, samples were prepared by means of an especially designed, custom-made device. The lower bond strength measured in this study can be attributed to this newly devised method of sample preparation. In this study, the μSBS test was preferred to the microtensile test as it allowed easier quantification of the number of specimens that prematurely failed or accidentally detached during preparation. The number of prematurely failed, discarded specimens in each test is probably related to the aggressiveness of the preparation procedure.[35] The cutting procedure that is carried out during the microtensile test transmits vibrations to the specimens. Consequently, a common occurrence, especially if the bond strengths are relatively low (5–7 MPa),[36] is a premature failure of the specimen, which makes microtensile useless.[37-39] In this regard, because of the weak bond strength between silorane-based and methacrylate-based resin composites, the micro-shear test was preferred to microtensile as it did not require cutting after bonding, which avoided any additional stress on specimens.

CONCLUSION

A reliable bond between aged silorane composite and methacrylate composite was obtained by using a phosphate-methacrylate–based adhesive as an intermediate layer. The interfacial bond strength achieved when repairing aged silorane with silorane was similar to that obtained by repairing aged methacrylate-based composite with methacrylate-based composite.

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Cross-Compatibility of Methacrylate-Based Resin Composites and Etch-and-Rinse One-Bottle Adhesives

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Clinical Relevance

Cross-compatibility between different combinations of methacrylate-based resin composites and etch-and-rinse one-bottle adhesives was evidenced for products from different manufacturers. This represents a desirable property of adhesives as it allows the flexibility to select different composite systems based on the specific restorative needs.

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SUMMARY

Objective: To compare dentin shear bond strength (SBS) of four combinations of light-activated one-bottle adhesives and composites to determine if cross-compatibility exists, and to determine if the use of the same manufacturer's adhesive and composite results in higher SBS than systems that combine different manufacturers' products.

Methods: One hundred sixty human third molars were used for bonding (n=10). Specimens were treated with 37% phosphoric acid and one of four etch-and-rinse adhesives. Specimens were placed in a bonding jig, which was filled with one of four composites. Adhesives PQ1 (Ultradent), Excite (Ivoclar-Vivadent), Optibond Solo Plus (Kerr), and Single Bond (3M-ESPE) and composites Vit-l-Escence

(Ultradent), Four Seasons (Ivoclar-Vivadent), Premise (Kerr), and Filtek Supreme Plus (3M-ESPE) were tested. SBS was measured at 24 hours and three months with a testing machine at a speed of 1 mm/min and expressed in MPa. A three-way analysis of variance and Tukey tests were used for data analysis.

Results: Significant differences were evidenced among composites for each adhesive system ($p<0.001$) and among adhesives for each composite system ($p<0.001$). Optibond Solo Plus and PQ1 yielded significantly higher bond strengths than Single Bond and Excite for all composite systems ($p<0.05$). All combinations, with the exception of two, demonstrated a decrease in bond strength values after aging.

Conclusions: Cross-compatibility was demonstrated, indicating that etch-and-rinse one-bottle adhesive systems can be safely used with composites from different manufacturers without a compromise to the bond strength. Moreover, even higher mean SBS values were demonstrated for selective combinations of different manufacturers' products.

INTRODUCTION

A belief has been promoted by manufacturers from dental companies encouraging dentists to use their adhesive systems in combination with their resin composites as they claim that greater strength and longevity of the restorations can be obtained when using products from the same manufacturer. Clinicians often face the question as to whether incorporating an adhesive and composite from the same manufacturer will result in greater strength and longevity of restorations.

While it is well known that certain bonding agents are not compatible with self- and dual-polymerized resin composites,[1–5] it remains unclear whether compatibility issues exist between adhesives and light-activated composites. Adverse acid-base reactions as well as adhesive permeability may be responsible for any incompatibility issues that may exist.[6–8] Acidic monomers in etch-and-rinse adhesives can adversely react with basic initiators (tertiary amines) of self- or dual-polymerized composites, preventing their polymerization.[2–5] Although light-activated composites also undergo polymerization through the generation of free radicals by tertiary amines, they appear to be less affected by the acidic monomers on etch-and-rinse adhesives than self- or dual-polymerized composites. This may be the result of a more rapidly occurring initiation process, which may interrupt the acid-base reaction.[6,9]

Cross-compatibility between adhesives and resin composites made from different manufacturers represents a desirable property as it allows the dentist the flexibility to select different composite systems based on specific restorative needs. A few studies have reported higher bond strengths when an adhesive was used with a composite from the same manufacturer rather than a composite from a different manufacturer.[2–3,10] However, these findings may be the result of differences in strength among the various types of composite materials (ie, hybrids vs microfills) rather than caused by compatibility issues between the adhesive and composite systems, as suggested by some studies.[11,12] Not enough evidence is available to support the claim that combining an adhesive and a composite with somewhat different monomeric composition and made from different manufacturers will result in lower bond strengths than the use of products made from the same manufacturer. In general, it appears that cross-compatibility exists and that adhesives and composites from different manufacturers can be combined without a compromise to the bond strength.[13,14]

The literature on the topic is scarce. A literature search dating back to 1980 was conducted to identify studies related to compatibility issues between light-activated composites and adhesive systems. A few studies were identified that revealed no compatibility issues between adhesives and light-activated composites.[13–15] However, these early studies report on products that have either disappeared from the market, changed in technology, or were made by a manufacturer that is no longer in business. The authors are not aware of recent studies reporting on the cross-compatibility properties of newer commercially available adhesive systems.

The objective of this study was to compare dentin shear bond strength (SBS) of different combinations of light-activated methacrylate-based resin composites and etch-and-rinse adhesive systems made from four different manufacturers at 24 hours and three months to determine if cross-compatibility exists. Furthermore, the study aimed to determine if using an adhesive and composite from the same manufacturer results in higher bond strengths than the use of an adhesive and composite made from different manufacturers.

Table 1: *Tested Materials, Manufacturers, Category, Composition, and Batch Number as per Manufacturers' Descriptions*

Adhesive Systems				
Product (Manufacturer)	**Category**	**Organic Composition**	**Filler**	**Batch Number**
Optibond Solo Plus (Kerr, Orange, CA, USA)	Etch and rinse	Alkyl dimethacrylate resins, barium aluminoborosilicate glass, silicon dioxide, sodium hexafluorosilicate Solvent: ethanol	Barium aluminoborosilicate glass, silicon dioxide sodium hexafluorosilicate Filled 15% by wt	3267789
Excite (Ivoclar-Vivadent, Amherst, NY, USA)	Etch and rinse	HEMA, Bis-GMA, Phosphonic acid acrylate Solvent: ethanol	Silicon dioxide Filled 0.5% by wt	M06539
Single Bond (3M-ESPE, St Paul, MN, USA)	Etch and rinse	HEMA, Bis-GMA, water, PAA, Solvent: ethanol	Silica Filled 10% by wt	20090625
PQ1 (Ultradent, South Jordan, UT, USA)	Etch and rinse	Bis-GMA, methacrylate-based hydrophilic monomers Solvent: ethanol	Silica dioxide and FluorUtite Filled 40% by wt	B46NM
Resin Composites				
Product (Manufacturer)	**Category**	**Organic Composition**	**Filler**	**Batch Number**
Premise (Kerr, Orange, CA, USA)	Nanohybrid	Bis-EMA, TEGDMA	Prepolymerized filler, barium glass, silica nanoparticles (0.02-0.4 μm); filled 84% by wt	3204945
Four Seasons (Ivoclar-Vivadent, Amherst, NY, USA)	Hybrid	Bis-GMA, UDMA, TEGDMA	Barium glass, ytterbium trifluoride, Ba-Al fluorsilicate, dispersed silicon dioxide spheroid mixed oxide (0.6 μm); filled 76% by wt	L47114
Filtek Supreme Plus (3M-ESPE, St Paul, MN, USA)	Nanohybrid	Bis-EMA, Bis-GMA, UDMA, TEGDMA, Water	SiO2 Nanosilica filler, ZrO2/SiO2 Nanoclusters (0.02-0.075 μm) Filled 78.5% by wt	20080827
Vit-l-Escence (Ultradent, South Jordan, UT, USA)	Hybrid	Bis-GMA	Barium alumina silicate (0.7μm)	B4869

Abbreviations: Bis-MA, bisphenol A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; CQ, camphorquinone; PAA, polyalkenoic acid copolymer.

MATERIALS AND METHODS

Table 1 summarizes the composites and adhesives tested in this study. Adhesives and composites from four different manufacturers were selected, for a total of 16 combinations, which were all tested both at 24 hours and three months of 100% humidity storage at 37°C and thermocyling. A sample size of 10 (n=10) was selected after performing power analysis based on previous studies. This yielded a total of 320 specimens.

Specimen Preparation

One hundred sixty noncarious, unrestored human third molars were used to obtain dentin substrates for bonding. The molars were used within three months of extraction and stored in an aqueous disinfectant (0.5% chloramine T solution at 4°C) until ready to be used. The teeth were sectioned longitudinally in a mesiodistal direction using a band saw (Isomet, Buehler, Lake Bluff, IL, USA). The sectioned teeth were embedded in a chemically

polymerized methacrylate (Fastray, HJ Bosworth, Skokie, IL, USA) with the outer surface exposed. The exposed molar surface was ground flat on a model trimmer until an adequate surface of dentin was revealed. The exposed dentin was finished with 400- and 600-grit abrasive paper (Silicon carbide abrasive paper, Buehler). The prepared specimens were stored in deionized water at 4°C until ready to be bonded.

Bonding and Testing

Study groups were randomized to avoid bias relative to sequence of tooth restoration. One hour prior to bonding, the specimens were acclimatized to room temperature (23 ± 2°C). Immediately before bonding, the specimens' surfaces were slightly refinished with a 600-grit abrasive paper to expose fresh dentin. Dentin was etched with 37% phosphoric acid (Ultra-etch, Ultradent, South Jordan, UT, USA) for 15 seconds, then rinsed and blot dried for moist bonding. Each adhesive was applied following the manufacturer's instructions and polymerized for 20 seconds with a light-curing unit (Bluephase C8, Ivoclar-Vivadent, Amherst, NY, USA). A minimum power density of 800 mW/cm^2 was ensured by periodically monitoring the unit's output with a radiometer (Demetron, Kerr, Orange, CA, USA). The specimens were placed on a specially fabricated bonding jig (Ultradent) with a cylindrical mold of 2.38 mm in diameter. The mold was filled with the corresponding composite in increments no greater than 2 mm and polymerized for 40 seconds. Immediately after bonding, the specimens were stored for 24 hours in an incubator at 37°C and 100% humidity.

SBS was measured using a testing machine (Ultratester, Ultradent) at a test speed of 1 mm/min. A notched crosshead designed to match the diameter of the bonded specimen was used to apply the testing load. Specimens were stabilized in a testing jig, which was free to move to facilitate positioning under the load. The test base was then positioned so that the notched crosshead was placed against the specimen surface and the notch was fitted to the bonded specimen. The load required to debond the specimen was recorded and expressed in MPa by dividing the load by the surface area of the bonded specimen. SBS values were recorded at 24 hours, and the mean bond strength values for each study group were calculated.

Bonding procedures were repeated as previously outlined, and the specimens were stored under the same conditions for three months. Prior to bond strength testing at three months, the specimens were thermocycled for 500 cycles between 5°C and 55°C with a dwell time of 20 seconds as per recommendations from the International Organization for Standardization (ISO TR11405, 1994).

Statistical Analyses

A three-way analysis of variance (ANOVA) was used to evaluate the effect of the adhesive, composite, and time variables on bond strength. The presence of statistically significant interactions among these factors was also evaluated. A pairwise multiple comparison procedure Tukey test was used to identify differences among adhesives within the composite groups and among composites within the adhesive groups both at 24 hours and after three months of aging. In addition, a Student *t*-test was used to explore significant differences between bond strength values at 24 hours and after three months of aging for each composite-adhesive combination. A significance level of 0.05 was used for all tests.

RESULTS

Three-way ANOVA results are summarized in Table 2. Statistically significant differences were found among adhesives ($p<0.001$), composites ($p<0.001$), and the different testing times ($p<0.001$). Furthermore, statistically significant interactions between the adhesive and the composite ($p=0.003$) and between the adhesive and the testing times ($p<0.001$) were found, but no significant interaction was detected between the composite and the testing times ($p=0.092$).

Figure 1 summarizes the results of the pairwise comparison using Tukey tests. Mean SBS values for the different adhesive-composite combinations at 24 hours and three months are shown for each composite system. Significant differences were evidenced among adhesives for each composite system (letters in Figure 1). When the SBS values were compared within each adhesive system at 24 hours, Tukey tests revealed no statistically significant differences among the four composites for Optibond Solo. When bonding with Excite, a significant difference was observed between Filtek Supreme and Premise. For PQ1, Four Seasons was significantly different from Vit-l-Essence, and with Single Bond, Vit-l-Essence was found to be significantly different from Filtek Supreme and Four Seasons. The same comparison at three months revealed no statistically significant differences between the four composites for either adhesive system with the only exception of Single Bond, which showed a statistically significant difference between Filtek Supreme and Premise.

Table 2: *Three-Way Analysis of Variance Results*

Source of Variation	*df*	SS	MS	*F*	*p*
Adhesive	3	23,846.936	7948.979	152.569	<0.001
Composite	3	916.124	305.375	5.861	<0.001
Time	1	3391.059	3391.059	65.086	<0.001
Adhesive × Composite	9	1330.579	147.842	2.838	0.003
Adhesive × Time	3	2173.621	724.54	13.906	<0.001
Composite × Time	3	338.665	112.888	2.167	0.092
Adhesive × Composite × Time	9	331.666	36.852	0.707	0.702
Residual	288	15,005.043	52.101		
Total	319	47,333.692	148.381		

Abbreviations: df, *degrees of freedom; SS, sum of squares; MS: mean squares;* F, *f obtained;* p, *probability*.

Table 3 shows the differences between the mean bond strength values at 24 hours and after three months of aging for all composite-adhesive combinations. All groups showed a decrease in SBS after aging, with exception of the combinations Single Bond–Four Seasons and Single Bond–Vit-l-Essence. However, for these two groups, the differences did not show statistical significance. For the groups bonded using products from the same manufacturer, two out of four (Optibond-Premise and Excite-Four Seasons) showed significant differences in bond strength values before and after aging, and the other two (Single Bond-Filtek Supreme and PQ1-Vit-l-Essence) did not.

DISCUSSION

In this study, 16 different combinations of etch-and-rinse one-bottle adhesives and light-activated composites from four manufacturers were selected to evaluate cross-compatibility at 24 hours and three months. Cross-compatibility was demonstrated though pairwise comparisons using Tukey tests. As shown in Figure 1, even higher mean SBS values were observed for selective combinations of different manufacturers' products, indicating that cross-compatibility exists and that current etch-and-rinse one-bottle adhesive systems can be safely used with composites from different manufacturers without a compromise to the bond strength. At 24 hours, Optibond Solo Plus and PQ1 yielded significantly higher bond strengths than Single Bond and Excite for all composite systems. Optibond Solo Plus and PQ1 were not statistically different from each other. Similarly, Single Bond and Excite were not statistically different from each other. After aging, although the same behavior was observed for Optibond Solo Plus and PQ1, a few combinations remained not significant. Our findings are in agreement with studies that concluded that the use of products from the same manufacturer does not necessarily yield higher bond strengths.[13,14]

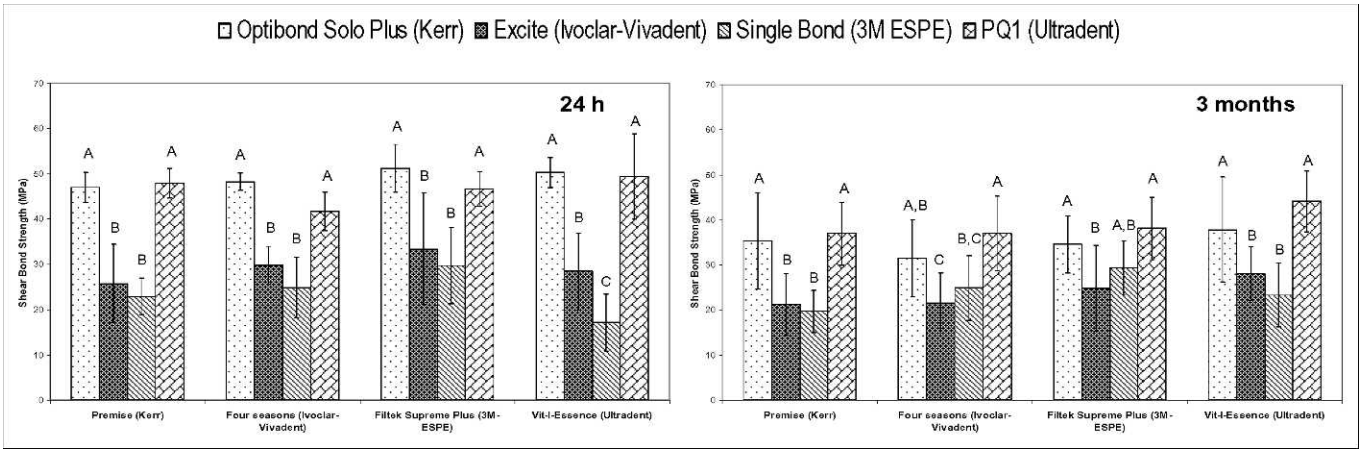

Figure 1. *Mean (SD) shear bond strength values for each of the adhesive-composite combinations. Different manufacturers are represented with different print patterns. Same letters indicate no significant differences among adhesives for each composite.*

Table 3: *Difference in Shear Bond Strength Values Between 24 Hours and Three Months of Aging (Student's t-test p-values)*[a]

	OptiBond Solo Plus (Kerr)		Excite (Ivoclar-Vivadent)		Single Bond (3M-ESPE)		PQ1 (Ultradent)	
	Difference	*p*	Difference	*p*	Difference	*p*	Difference	*p*
Premise (Kerr)	−11.6	**0.004**	−4.5	0.214	−3.2	0.119	−11.0	<**0.001**
Four Seasons (Ivoclar-Vivadent)	−16.8	<**0.001**	−8.2	**0.004**	0.1	0.987	−4.7	0.132
Filtek Supreme (3M ESPE)	−15.0	<**0.001**	−8.6	0.096	−0.4	0.911	−8.5	**0.003**
Vit-l-Escence (Ultradent)	−12.5	**0.005**	−0.4	0.910	6.2	0.053	−5.3	0.169

[a] Bold numbers indicate significant differences.

Compatibility might not be the only factor accountable for differences in bond strength values. Variations in the physical properties of the different types of composites may help explain the observed results.[11,12] Only two of the four groups that combined products from the same manufacturer (Filtek Supreme-Single Bond and Vit-l-Escence-PQ1) displayed the highest bond strength in their corresponding adhesive group.

While the debate is still ongoing as to how much strength is considered clinically acceptable bond strength, the scientific community has agreed on minimum bond strength values in the range of 17 MPa to 20 MPa in order to resist contraction stresses and obtain gap-free margins.[16] Despite the presence of statistically significant differences, all SBS values remained greater than 20 MPa, indicating that that cross-compatibility between products exists and that adhesives can be safely used with composites from different manufacturers without a compromise to their bond strength.

The formation of an adhesive interface is a complex phenomenon. The specific composition of the adhesive directly influences the quality of the resultant hybrid layer and hence its ability to resist shear forces. Compared with unfilled adhesives, filled adhesives create thicker interfacial layers, which help relieve the stresses generated during polymerization contraction, thermal changes, and occlusal loading[17] while yielding improved overall physical properties once it is polymerized. Conversely, the greater viscosity of filled adhesives may decrease momomer conversion[18] as well as limit their ability to penetrate into the demineralized dentin matrix[19] with the potential compromise to the bonded interface overtime.[20,21] Highly filled adhesives Optibond Solo Plus and PQ1 demonstrated significantly higher mean bond strength than lightly filled Excite and Single Bond irrespective of the brand of composite tested.

Current adhesives use primers based on either ethanol or acetone solvents. The type of solvent is also known to have an influence on the wetting capabilities of the adhesive and hence on the strength and stability of the hybrid layer overtime.[22] Although the four adhesives tested in this study were ethanol based, the comparatively lower bond strengths observed for Single Bond and Excite suggest that the type of solvent is only one of the many aspects influencing SBS results.

The effect of water storage on the degradation of adhesive interfaces has been the subject of numerous investigations.[23–25] Specifically, the formation of water blisters within the hybrid layer contributing to the degradation of the adhesive interface overtime has been extensively documented in the literature.[26–28] Our results are in agreement with these studies. As shown in Table 3, with the exception of two groups, all combinations showed a decrease in SBS values after three months of water storage. For seven of the 16 combinations, the differences between 24 hours and three months were statistically significant. Interestingly, the groups showing the highest bond strength values at 24 hours also showed the greatest amount of degradation after aging. However, the ranking remained the same when compared with the initial values.

Although valuable information can be obtained from laboratory studies, care should be taken not to overemphasize the results of *in vitro* investigations such as those reported in the present study. Numerous variables play a role in the immediate

and long-term behavior of adhesive interfaces, and they all deserve careful consideration.

CONCLUSIONS

Within the limitations of this *in vitro* investigation, the following conclusions may be drawn:

- Cross-compatibility was demonstrated indicating that current etch-and-rinse one-bottle adhesive systems can be safely used with composites from different manufacturers without a compromise to the bond strength. Moreover, even higher mean SBS values were demonstrated for selective combinations of different manufacturers' products.
- Optibond Solo Plus and PQ1 demonstrated superior bond strength values to all resin composites irrespective of the manufacturer.
- A decrease in SBS values was observed at three months for most combinations. However, trends similar to those observed at 24 hours remained.

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Long-Term Nanoleakage Depth and Pattern of Cervical Restorations Bonded With Different Adhesives

EH Mobarak • LE Daifalla

Clinical Relevance

A mild acetone-based single-step self-etched adhesive system may reveal less nanoleakage in the short-term interval, but unfortunately, this was not sustained after long-term storage.

SUMMARY

Purpose: This study investigated the effect of water storage on nanoleakage depth and the pattern of cervical cavities bonded with different adhesives.

Methods: For nanoleakage depth evaluation, standardized cervical cavities (2 mm in diameter) were prepared on the buccal and lingual surfaces of 36 intact human premolars. Specimens were divided into three groups (n=12) according to the three adhesive systems used: an etch-and-rinse adhesive (SBMP, Adper Scotchbond Multi-Purpose, 3M ESPE) and two single-step self-etch adhesives; one was mild and acetone based (IB-iBond, Kulzer), while the other was strong water based (PL, Adper Prompt L-Pop, 3M ESPE). All cavities were restored using Filtek Z250 (3M ESPE) resin composite. For each adhesive, specimens (n=12 with 24 restored cavities) were subdivided into three subgroups (n=4 with eight cavities) according to the storage period before examination (24 hours, three or six months). Another duplicate of teeth was prepared in the same way for nanoleakage pattern evaluation. After storage, the specimens were placed in 50%W/V silver nitrate solution for 24 hours and immersed in a photo-developing solution for eight hours. Thereafter, the specimens were sectioned buccolingually, polished, and examined by scanning electron microscopy. For nanoleakage pattern, specimens were treated in the same way as for nanoleakage depth except that they were additionally immersed in 10% EDTA for five seconds after

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polishing. Silver penetration percentage was calculated to the total length of the tooth-restoration interface. Data were analyzed with two-way analysis of variance, Kruskal-Wallis, and post hoc tests.

Results: After 24 hours, the least amount of nanoleakage depth was recorded for IB, while the highest was recorded for PL. For stored specimens, there was no significant difference among the nanoleakage depths of all adhesives. The tested adhesives recorded different nanoleakage patterns; however, there was an increase in the intensity and continuity of silver deposition by time.

Conclusions: After 24 hours, the nanoleakage depth/pattern varied with the type of adhesive used; however, after water storage, all adhesives performed equally.

INTRODUCTION

The utmost goal of bonded restorations is to achieve a marginal and internal seal. Microleakage is defined as the leakage of fluids at the tooth-restoration interface through a gap.[1] Failure to seal the restoration margin can result in postplacement sensitivity, margin staining, and recurrent caries, which are the most common reasons associated with clinical failure of adhesive restorations.[2] Fluid leakage without gap formation can also occur. Nanoleakage is an internal leakage describing the nanometer-sized spaces around the collagen fibrils within the hybrid layer.[3] Authors have also reported that nanoleakage increases with long-term storage.[4] Although there is still no clear evidence of the negative effects of nanoleakage, the existence of such a pathway in gap-free cavity margins may have potential long-term consequences for adhesion quality.[5]

A number of new adhesive systems have been developed in an attempt to obtain a reliable adhesive-restoration interface over time.[6] Current adhesive systems interact with tooth substrate, using two different strategies. The total-etch technique implies removal of the smear layer. However, incomplete expansion of collagen may impair resin infiltration and compromise bonding with those adhesives.[7] The self-etch adhesive strategy is based on the simultaneous etching, priming, and bonding to the smear-covered dental tissues.[8] When using all of these self-etch adhesive systems, less discrepancy is expected between the depth of demineralization and depth of resin infiltration. Meanwhile, in an attempt to reduce the clinical application steps as well as the technique sensitivity, single-step self-etch adhesives have been introduced in the market.[9]

Single-step self-etch adhesives vary in their acidity by virtue of the composition and concentration of polymerizable acids and acidic resin monomers.[10,11] Strong self-etch adhesives were characterized by their better etching performance on enamel and compromised bonding to dentin. Mild self-etch adhesives rely on keeping the hydroxyapatite at the interface protecting the collagen and allowing for chemical interaction. The resultant twofold micro-mechanical and chemical bonding mechanisms of mild adhesives were more promising regarding the bond strength.[7] However, the nanoleakage performance of these different types of self-adhesive systems is not yet clear. Therefore, this study was conducted to compare the nanoleakage depth and pattern of bonded cervical cavities using strong or mild self-etch adhesives over time.

MATERIALS AND METHODS

The material brand names (manufacturer), description, and composition (lot number) are listed in Table 1. For the present study, freshly extracted human premolars free from caries or any cracks were collected. A hand scaler was used to remove the remnants of the periodontal tissues and calculus if present. The selected teeth were stored in a phosphate-buffered solution containing 0.8% sodium azide for a maximum period of one week until use.[12]

Specimen Grouping and Preparation

For nanoleakage depth evaluation, 36 teeth were divided into three main groups of 12 teeth each, according to the three adhesive systems used. For each adhesive system, the teeth were subdivided into three subgroups of four teeth each, according to the storage period before examination (24 hours, three or six months). In each tooth, two cavities were prepared (buccal and lingual), yielding eight cavities for each adhesive system tested at each storage period. Another duplicate of teeth was prepared in the same way for the nanoleakage pattern evaluation.

Standardized buccal and lingual cervical cavities were prepared. For each tooth, the cemento-enamel junction (CEJ) was marked with a pencil. The mesiodistal width was measured using a precise digital caliber (Mitutoyo, Digimatic Caliper, Mitutoyo Corp, Tokyo, Japan). The cavities were designed to coincide with the points of intersection between the drawn midlines on the buccal and the lingual

Table 1: *Material Brand Names (Manufacturer), Description, Composition (Lot #), and Bonding Procedures*

Brand Name (Manufacturer)	Description	Composition (Lot #)	Bonding Procedure
Adper Scotchbond Multi-Purpose Adhesive System (3M ESPE, St Paul, MN, USA)	Three-step etch-and-rinse adhesive system	Etchant: 35% phosphoric acid (20031106)	Applied 15 seconds to enamel and dentin; rinsed thoroughly; gently air dried; dentin was left moist
		Primer: HEMA, polyalkenoic acid copolymer and water (20030408)	Applied; gently air dried for three seconds
		Adhesive: HEMA and Bis-GMA (20031106).	Applied and light cured for 20 second.
Adper Prompt L-Pop adhesive system (3M ESPE)	Two-component, single-step, self-etch adhesive system	Liquid 1 (red blister): methacrylated phosphoric esters Bis-GMA, initiators based on camphorquinone, stabilizers	Two components were mixed starting with pressing red reservoir toward yellow one; applied to enamel and dentin; rubbed for 15 seconds; gently air dried for five seconds and light cured for 20 seconds
		Liquid 2 (yellow blister): Water, HEMA, polyalkenoic acid, stabilizers (I93938)	
iBond adhesive system (Heraeus Kulzer, Hanau, Germany)	One-component single-step, self-etch adhesive system	4-methacryloxyethyltrimetallitic anhydridw (4-META), UDMA, glutardialdehyde, acetone, water, photoinitiator (010049)	Shaken three seconds before use; applied to enamel and dentin in three consecutive layers; massaged for 30 seconds; gently air dried for five seconds and light cured for 20 seconds

Abbreviations: 4-META, 4-methacryloxyethyltrimellitic anhydride; Bis-GMA, bis-phenol-A glycidyl-methacrylate; HEMA, 2-hydroxyethyl methacrylate; UDMA, urethane dimethacrylate.

surfaces and the marked CEJ. This enabled the drilling of cavities to be exactly aligned. To standardize the cavity depth and diameter at 2 mm ± 100 μm, the cavities were prepared using round stones (Komet, Brasseler, GmbH, Germany) in a successive order (ISO 012, 014, 016, 018). The cavity dimensions were ensured using the same digital caliber.

Each adhesive system was applied according to the manufacturer's instructions (Table 1). Resin composite restorative material (Filtek Z250, 3M ESPE), shade A3.5, was inserted in one increment using a plastic instrument (Dentsply, Ash, and Surrey, England). Caution was taken to minimize the excess material over the cavity margins. A polyester strip was applied, and the restorative material was light cured for 40 seconds using a light-curing unit of $\geq$500 mW/cm^2 intensity, which was checked with a radiometer (Demetron LED Radiometer, SDS, Kerr, Orange, CA, USA). After curing the excess composite, flash was removed using a lancet (Wuxi Xinda Medical Devices Co Ltd, Wuxi, Jiangsu, China). Then, finishing was done using rubber finishing points (Ivoclar Vivadent, Schaan, Liechtenstein) and a magnifying lens (Baush and Lomb Optics Co, Rochester, NY, USA) of 6× magnification to ensure that there was no composite flash at the cavity margins. The restored teeth were immersed in distilled water at 23°C ± 2°C[13] and left according to the intended storage periods (24 hours, three or six months). Water storage was changed weekly.[14] Teeth were then prepared for nanoleakage evaluation.

Nanoleakage Evaluation

After storage, customized circular pieces of adhesive tape were cut 3 mm in diameter using a 72-revolving-punch plier (General, Montreal, Canada). Each piece was placed over the restored cavity protecting the filling and 1 mm around the cavity margins. Specimens conducted for nanoleakage depth evaluation were then coated with a double layer of a fast-setting nail varnish, leaving 1 mm

Table 2: *Means and Standard Deviations for Silver Penetration Percentage Specimens Bonded With Different Adhesive Systems Over Different Storage Periods*[a]

Variable	SBMP	PL	IB	*p* Value
24 hours	5.2 ± 1.2^{aA}	9.3 ± 1.6^{bA}	4.8 ± 2.8^{aA}	≤ 0.01
Three months	11.6 ± 2.6^{aB}	12.3 ± 6.7^{aA}	11.8 ± 3.3^{aB}	≥ 0.05
Six months	12.2 ± 1.6^{aB}	11.3 ± 1.9^{aA}	9.8 ± 3.7^{aB}	≥ 0.05
p value	≤ 0.001	≥ 0.05	< 0.05	

[a] *Rows with the same lowercase superscript letters are not statistically different from each other. Columns with the same uppercase superscript letters are not statistically different from each other.*

from the bonded interface exposed. The teeth were immersed in 50% silver nitrate solution for 24 hours in total darkness, rinsed thoroughly, and immersed in a photo-developing solution for eight hours under fluorescent light. Then, the teeth were rinsed using tap water for 60 seconds as described by Tay and others.[15] After that, the teeth were sectioned through the centers of the restorations. The cut surfaces were finished and polished with fine silicon carbide abrasive papers (Buehler, Lake Bluff, IL, USA) in an ascending order (from 600 grit and up to 4000 grit) and examined using a scanning electron microscope (SEM; Philips XL30-5600MD, Eindhoven, the Netherlands) in back-scattered mode. The teeth intended for nanoleakage pattern examination were treated in the same way as those for nanoleakage depth evaluation except that they were additionally immersed in 10% EDTA for five seconds after polishing.[13]

The specimens were mounted on aluminum studs, gold sputter coated, and examined in the back-scattered electron mode at an acceleration voltage of 25 kV. Evaluation of nanoleakage depth was done using SEM. The depth of silver penetration was measured as well as the total length of the bonded interface. The percentage of the penetration depth to the total length of the bonded interface was calculated and recorded. The teeth intended for nanoleakage pattern evaluation were examined using SEM at 500× or 800× magnification.

Statistical Analysis

Data were statistically described in terms of mean ± standard deviation. Two-way analysis of variance was used to test the complex interaction between adhesive system and storage time. This was followed by a Kruskal-Wallis test for multiple comparisons and Convene Inman test for pairwise comparisons as a post hoc test. p values less than 0.05 were considered statistically significant. All statistical calculations were done using the computer programs Microsoft Excel 2007 (Microsoft Corporation, Redmond, WA, USA) and Stats Direct statistical software version 2.7.2 for MS Windows (Stats Direct Ltd, Cheshire, UK).

RESULTS

The means and standard deviations and statistical significance of the percentage of silver penetration for the three adhesive systems at the three storage periods are presented in Table 2.

Regarding the nanoleakage depth, at 24 hours, Adper Prompt L-Pop recorded a statistically significant higher percentage of silver penetration than the other materials ($p \leq 0.01$). After storage for three and six months, specimens showed a significant increase in percentage values of silver penetration for both Adper Scotchbond Multi-Purpose ($p \leq 0.001$) and iBond ($p < 0.05$), respectively. For Adper Prompt L-Pop, no significant difference ($p \geq 0.05$) was found between the three storage periods. Also, there was no statistically significant difference ($p \geq 0.05$) in the percentage of silver penetration values among the three adhesives used at the three- and six-month storage periods.

The Adper Scotchbond Multi-Purpose nanoleakage pattern revealed silver patches at the base of the hybrid layer after 24 hours (Figure 1). After storage, silver patches increased in number and size. They were found anywhere within the thickness of the hybrid layer. Silver was never found in the adhesive layer for all the specimens (Figures 2 and 3). For Adper Prompt L-Pop, a thin hybrid layer was formed. Reticular silver deposits were observed in

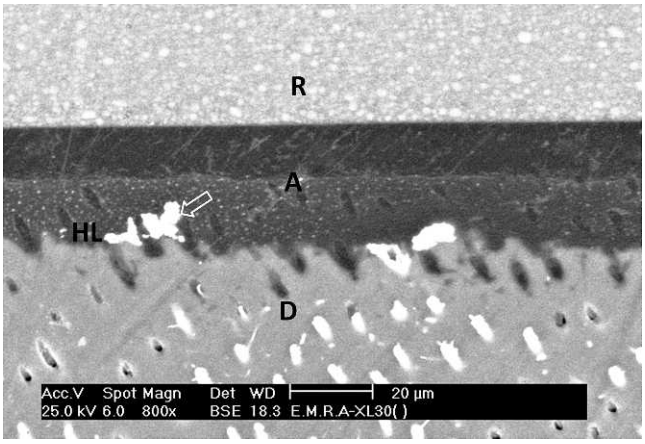

Figure 1. *Scanning electron microscope micrograph of Adper Scotchbond Multi-purpose adhesive dentin interface after 24-hour storage.*

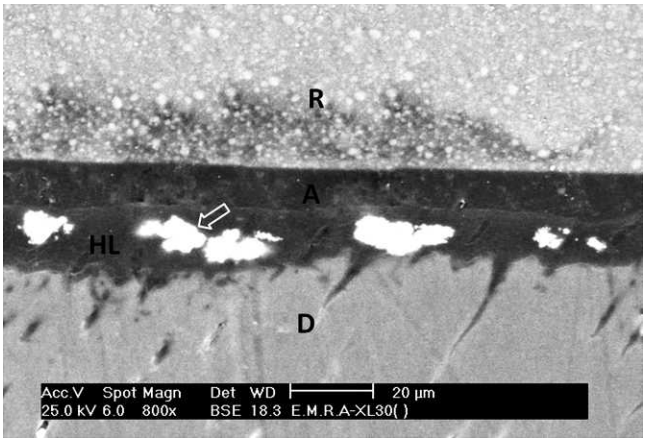

Figure 2. *Scanning electron microscope micrograph of Adper Scotchbond Multi-purpose adhesive dentin interface after three-month storage.*

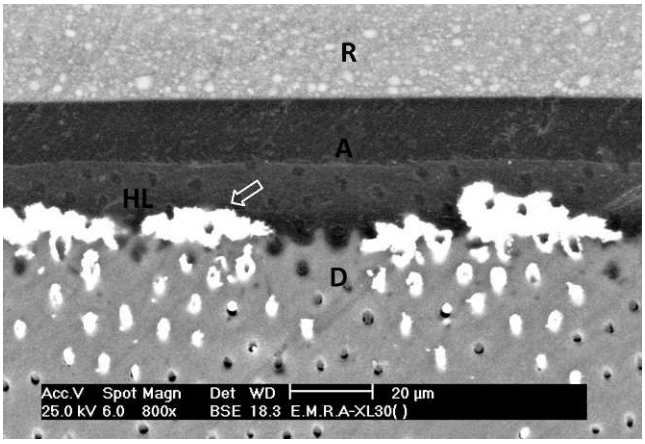

Figure 3. *Scanning electron microscope micrograph of Adper Scotchbond Multi-purpose adhesive dentin interface after six-month storage.*

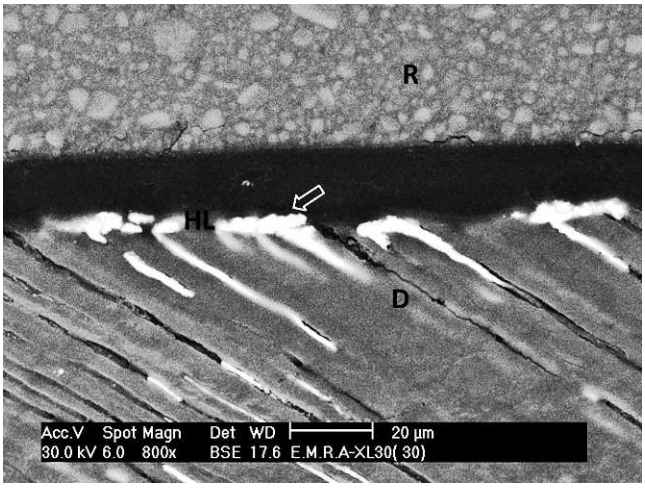

Figure 4. *Scanning electron microscope micrograph of Adper Prompt-L-Pop adhesive dentin interface after 24-hour storage.*

the 24-hour stored specimens (Figure 4). After 3-month storage, they became thicker and wider in spread (Figure 5). Upon storage for 6 months, reticular silver deposits continued to grow in width and height, and water trees became clearly observed (Figure 6). For iBond, the hybrid layer formed was very thin. Silver-stained bands (arrows) were found in the hybrid layer. No water treeing was observed in the 24-hour stored specimens (Figure 7). After aging, silver-stained bands became thicker and more continuous. Water trees (arrowheads) became more manifested within the adhesive layer (Figure 8) and increased in height and number after longer storage periods (Figure 9).

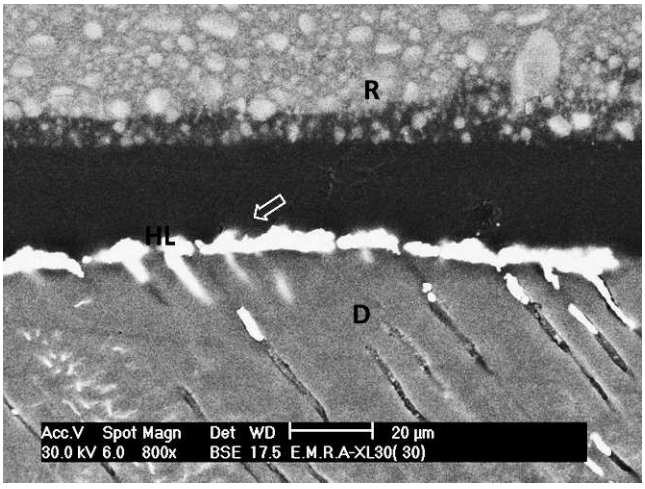

Figure 5: *Scanning electron microscope micrograph of Adper Prompt-L-Pop adhesive dentin interface after three-month storage.*

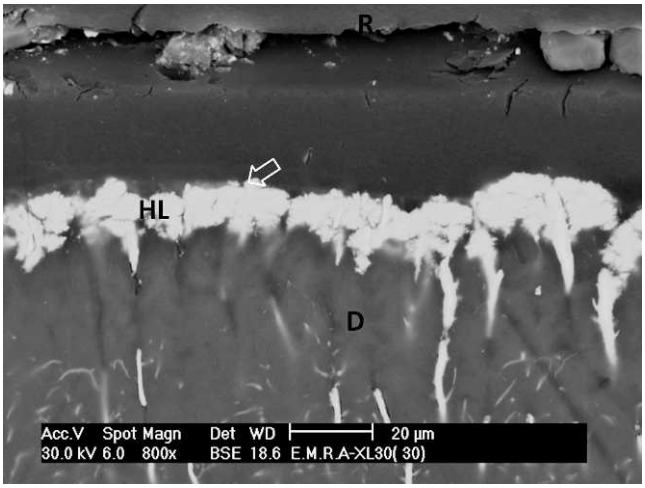

Figure 6. *Scanning electron microscope micrograph of Adper Prompt-L-Pop adhesive dentin interface after six-month storage.*

DISCUSSION

The use of bonded restorations is taking a growing role in restorative dentistry. The introduction of the acid etch technique was found to be enough to obtain acceptable sealing with enamel. However, obtaining sealed restorations at the dentin side without leakage was a more complicated target. Nanoleakage, which is a valuable criterion in the evaluation of adhesive performance, should be fully determined qualitatively as well as quantitatively.[16] In the current study, nanoleakage evaluation was carried out with high-magnification SEM by means of a back-scattered electron mode, which was reported to be better compared with the secondary electron images.[3] The present study was conducted on restored cervical cavities to evaluate nanoleakage of different adhesive strategies over different storage periods. The configurations of this cavity proved to be extremely challenging because of the high C-factor present. Most nanoleakage studies have used a flat dentin surface for bonding and nanoleakage evaluation.[4,12,13,15,17] However, cervical cavities represented a clinically relevant case.

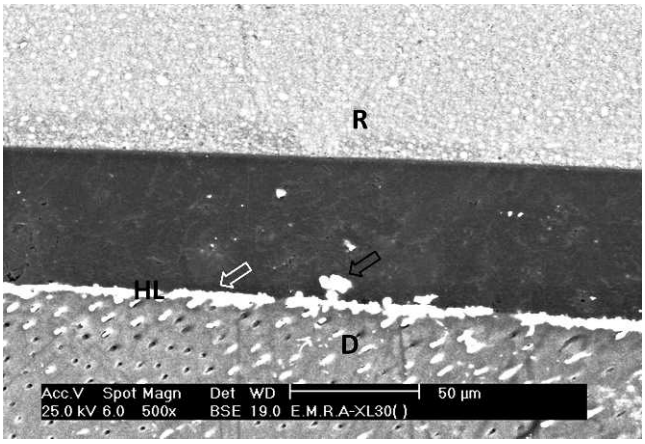

Figure 8. *Scanning electron microscope micrograph of iBond adhesive dentin interface after three-month storage.*

For nanoleakage depth evaluation, the percentage of silver penetration to total length of bonded interface was calculated. This was in accordance with Li and others[18] and Fernando de Goes and Montes[19] and in opposition to other authors[20] who graded nanoleakage with scoring. The use of a scoring system, such as 0 = 0%, 1 = 1% to 25%, 2 = 25% to 50%, 3 = 50% to 75%, and 4 = >75%, presented in previous studies, was not preferred in the current study. This was because each score other than zero represented a wide range, making it unsuitable for differentiating between the tested adhesive systems as all would meet a score 1.

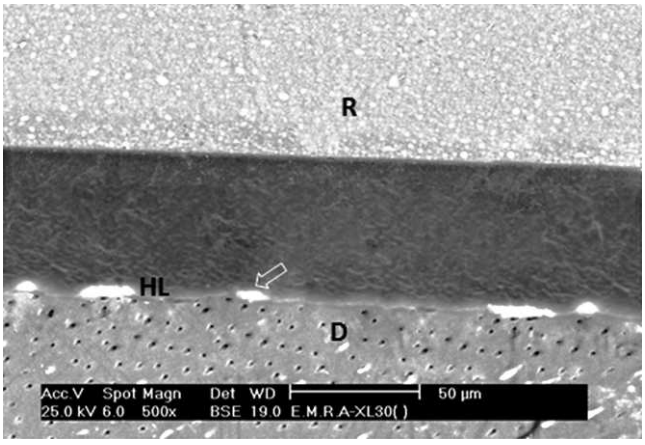

Figure 7. *Scanning electron microscope micrograph of iBond adhesive dentin interface after 24-hour storage.*

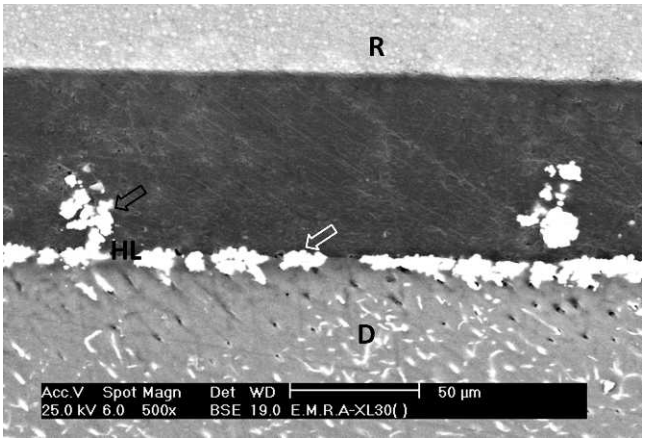

Figure 9. *Scanning electron microscope micrograph of iBond adhesive dentin interface after six-month storage.*

The observation of the photomicrographs resulting from the back-scattered electron mode revealed a difference in nanoleakage among different adhesive systems. It is generally accepted that heavy silver uptake along the interfacial layer and in the adhesive layer can be due to imperfect resin infiltration, retained water or other solvent, poor polymerization, or phase separation.[17] Regarding the etch-and-rinse adhesive technique, De Munck and others[21] stated that none of the contemporary all-in-one adhesives can compete with the more traditional multistep adhesives. However, the reported nanoleakage could be attributed to many factors. It was speculated that this represents the presence of very thin shrunken collagen fibers, which may accumulate on the dentin surface following etching.[22] This thin layer (0.2-0.3 μm) might interfere with adhesive resin infiltration, and silver ions may precipitate within this collagen smear layer. In addition, more evaporative water flux is expected with the strong air blowing recommended by the manufacturer of the used adhesives in order to evaporate the solvent and remove the excessive interfacial water.

Although the self-etch adhesive strategy is highly promising as a user-friendly and less technique-sensitive adhesive,[23] simplification and saving time may be at the expense of compromising the quality of resin-dentin bonds.[24] Reasons for reported nanoleakage are numerous. Among them is that they are too hydrophilic and act, even after polymerization, as semipermeable membranes.[25] High solvent concentration makes it impossible to obtain an adhesive resin layer of adequate thickness and free from residual solvent.[21] Phase separation and blistering that may occur during solvent evaporation due to change in the monomer-water ratio can also be a cause.[26] Additional explanations were added by Toledano and others.[27] One of them is the combination of acidic hydrophilic and hydrophobic monomers into a single step that compromises the polymerization of the adhesives.

Another investigation finding in the present study regarding self-etch adhesives was that the iBond self-etch adhesive showed significantly low nanoleakage penetration depth values after 24 hours compared with Adper Prompt L-Pop. The difference in silver deposition may be regarded as the difference in the acidity of adhesives tested and the chemical nature, which in turn affect the degree of water sorption and the bonding efficacy. The low pH (pH = 0.40 of Adper Prompt L-Pop[28]) denotes a high concentration of acidic uncured resin monomers. Another reason may be the low viscosity of Adper Prompt L-Pop that results in its spread so thin as to lead to formation of dry spots.[29] iBond (pH = 2) is an acetone-water–based adhesive,[28] whereas Adper Prompt L-Pop is water based. Water is comparatively easy to remove with the use of acetone-based adhesives because acetone increases the vapor pressure of water.[30] Also, iBond is HEMA free in contrast to Adper Prompt L-Pop. HEMA lowers the vapor pressure of water when added to a water mixture, making it more difficult to remove water from the adhesive and increasing water retention within the adhesive layer.[23]

Concerning the storage factor, specimens stored for three or six months had a significantly higher nanoleakage depth in comparison to 24-hour tested specimens for iBond and Adper Scotchbond Multi-Purpose adhesive systems. For Adper Prompt L-Pop, no significant difference was found during different storage periods. Also, the present study revealed an insignificant difference in the silver penetration percentage between three- and six-month storage regardless of the adhesive type. Such a trend has also been reported by Okuda and others.[31] They found that there was no statistically significant difference in silver penetration for the tested adhesive systems among three-, six-, and nine-month periods. These results denote that the dentin-resin interface deteriorates over time.

Bond degradation is divided into two phases: hydrolytic degradation of the collagen matrix and hydrolytic degradation of the bonding resin within the hybrid layer. Water has been claimed as one of the major causes of the collagen and resin degradation that occurs overtime. Hydrolysis is a chemical process that breaks covalent bonds between the polymers by addition of water to ester bonds, resulting in loss of resin mass.[4] Water sorption is enhanced by the presence of hydrophilic and ionic resin monomers, which in turn facilitates ion movement within a polymerized resin matrix.[32] The combined degradation of resin and collagen may increase the water content of the bonded interface, leading to a further detrimental effect on the longevity of the bond. A further contributing factor to the degradation of the hybrid layer is endogenous matrix metalloproteinases (MMP) such as MMP-2, -8, -9, and -20.[11,33,34] MMPs are slowly released from the denuded demineralized dentin matrix even in the absence of bacteria, in a way that is similar to caries progression.[33]

The nanoleakage pattern was reported to be dependent on the adhesive tested.[22] In the present

study, silver patches were seen in the hybrid layer formed by Adper Scotchbond Multi-Purpose, with the absence of silver staining in the adhesive layer. These results were in accordance with Hashimoto and others.[13] The reticular pattern of silver penetration that was observed in the hybrid layer of Adper Prompt L-Pop and the water trees in the adhesive layer were in agreement with Reis and others.[35] Silver-stained bands were found in the hybrid layer formed by iBond. Water trees were also seen in the adhesive layer of iBond. Such findings may denote that the C-factor has no effect on the nanoleakage pattern. Based on this, in the clinical situation, it is preferred that adhesive systems are hydrophilic during application and then become hydrophobic after application and completely seal the restoration margins. The dream of each researcher and clinician to have an adhesive system suitable for any case and revealing a marginal and internal seal is yet to be fulfilled.

CONCLUSIONS

Under the conditions of this study, the following can be concluded:

1. Nanoleakage is dependent not only on the application technique but also on the adhesive chemical nature.
2. Storage in distilled water increased nanoleakage depth, continuity, and intensity.

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 Prewarming and/or Delayed Light Activation on Resin-Modified Glass Ionomer Bond Strength to Tooth Structures

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Clinical Relevance

Bond strength might improve by delaying the light activation procedure when a cavity conditioner is used for bonding RMGI to enamel. Conversely, delaying the light activation and/or prewarming of RMGI compromises bond strength to dentin and should be avoided.

SUMMARY

Introduction: Recent research shows that the acid-base reaction and light-activated polymerization in resin-modified glass ionomers (RMGI) compete with and inhibit one another. In addition, extrinsic energy would improve some properties of RMGI. This in vitro study evaluated the effect of prewarming and/or delayed light activation on bond strength of RMGI to tooth structure.

Materials and Methods: Ninety-six flat enamel and dentin surfaces of human molars were ground with sequentially finer abrasives to 600-grit silicon carbide paper. Each surface was treated with a cavity conditioner for 10 seconds, rinsed, and gently air-dried (n=12). RMGI was applied to tooth substrates according to the following protocols: group 1) according to manufacturer's instructions; group 2) a delay of two minutes in light activation; group 3) prewarming of the encapsulated material (90 seconds, 40°C); group 4) prewarming plus a delay of two minutes in light activation. After 24 hours of storage at 37°C and 500 rounds of thermocycling, the samples were tested for shear bond strength and analyzed using two-

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way analysis of variance and Tukey HSD test (α=0.05).

Results: Significant differences were observed between study groups ($p<0.05$). The highest enamel bond strength was recorded in group 2. Regarding dentin groups, the highest bond strength was recorded in group 1.

Conclusion: Within the limitations of the present study, delaying light activation might improve bond strength of RMGI to enamel; however, the standard procedure recommended by the manufacturer is the best procedure for bonding of RMGI to dentin. More investigations are necessary.

INTRODUCTION

Polyalkenoate (glass ionomer) cements (GICs) have widespread clinical uses as temporary or permanent restorations, as cavity liners or fissure sealants, or as luting agents for indirect restorations and orthodontic brackets. GICs are unique in their ability to bond to enamel and dentin and to base metals,[1] release fluoride,[2] inhibit potential caries,[3] and exhibit antibacterial activity due to their low setting pH value.[4]

Resin-modified glass ionomers (RMGIs) are composed of an acid-degradable glass and aqueous solutions of polyacid and monomeric components, including 2-hydroxyethyl methacrylate (HEMA). The setting reaction of the cement is triggered immediately after mixing in the form an acid-base reaction. Free-radical polymerization of the monomeric components is then initiated by irradiation with visible light. Each acrylate group is capable of independently participating in the chain reaction, but the final outcome is the formation of a covalently cross-linked three-dimensional network. The set cement is composed of interpenetrating networks of, say, poly (HEMA) and polyacrylate salts. This photochemical reaction reduces the moisture sensitivity and the dehydration associated with the initial stages of GIC acid-base setting reaction.[5]

As a result, the setting reaction of RMGIs is mediated by at least two different mechanisms that depend on reactant diffusion before gelation. The kinetics and setting mechanisms of each reaction might depend on and/or compete with the other.[6] Necessarily, one reaction replaces part of the other; however, the chemistry indicates that the formation of each network would separately inhibit diffusion and thus the other reaction. Immediate light activation might limit the acid-base reaction and give rise to different structural properties.[6] On the other hand, delayed irradiation might limit the polymerization of resin.[6,7]

In the same context, a study recently showed that the polymerization behavior of RMGIs is under the influence of the power density of the curing unit.[8] Yelamanchili and Darvell[9] made an attempt to answer the question whether interference occurs between the components of an resin-modified glass ionomer cement (RMGIC) by changing the light-activation protocol. They reported a competitive phenomenon between network-forming reactions, leading to a sensitive balance between the two, and a critically optimum light-activation protocol: too much irradiation might be detrimental, as is delay. They recommended that the manufacturer's instructions regarding the duration of irradiation should be followed so that an optimal result would be achieved.

On the other hand, recent data show that a moderate increase in composite resin temperature increases its flowability, which is advantageous in composite resin placement, leading to better adaptation with cavity walls and outline.[10–14] Furthermore, recent research indicates that there is a higher degree of composite resin conversion[11] when it is cured at slightly higher temperatures. One study[15] showed that prewarming of composite resin might have a detrimental effect on restoration margins as it increases polymerization shrinkage. Conversely, the results of a recent study by Fróes-Salgado and others[16] showed that prewarming of composite resins before light activation in clinical situations does not alter their mechanical properties and monomer conversion but enhances composite resin adaptation to cavity walls. A more recent study by Deb and others[17] revealed that prewarming of dental composite resins enhances their flowability and conversion degree. The linear polymerization shrinkage increases parallel with an increase in conversion degree; however, the flexural strengths remain unchanged. They reported that prewarming of composite resins might have clinical advantages during placement and adaptation of the material to the cavity walls.

Regarding prewarming protocols of GICs and RMGIs, an earlier study has shown that accelerated setting reaction in RMGI by means of heat or ultrasound waves shortens the setting reaction time of RMGI and significantly increases the bond strength to enamel.[7] According to O'Brein and others,[18] application of an external energy source by prewarming the capsules, by exposing the surface to a high-irradiance light-curing unit, or by ultra-

Table 1: *Materials Used in the Study, Their Compositions, and Mode of Their Applications According to the Manufacturer Instructions*

Material name and manufacturer	Manufacturers' instructions	Material composition
Cavity Conditioner (GC, Tokyo, Japan)	Apply with a brush for 10 s, rinse thoroughly.	Polyacrylic acid (20%), aluminum chloride (3%), distilled water
Fuji II LC (Improved version) (GC, Tokyo, Japan)	Shake the capsule, push the plunger until it is flush with main body, place the capsule into a metal GC capsule applier and click the lever once, set the capsule in a mixer and mix for 10 s, load it into the applier, and inject in the prepared cavity and cure for 20 s.	Powder: fluoro alumino-silicate glass; liquid: polyacrylic acid (20%-25%); 2-hydroxyl ethyl methacrylate (30%-35%); proprietary ingredient (5%-15%); 2,2,4,trimethyl hexa methylene dicarbonate (1%-5%); powder/liquid: 0/33g/0/085 mL

sonic scaler treatment significantly improves surface hardness at initial stages of glass ionomer cement setting reaction.

RMGIs are a hybrid of glass ionomers and composite resins. It seems that no study to date has attempted to evaluate and measure the effect of delaying light-activation and prewarming on RMGIs bond strength. Therefore, the aim of the present study was to investigate the effect of a delayed light activation technique and/or prewarming of an RMGI on bond strength to tooth structures. The specific hypothesis tested in this study was that delaying light activation and/or prewarming do not influence the bond strength of an RMGI to tooth structures.

MATERIALS AND METHODS

Shear Bond Strength Evaluation

Forty-eight sound human third molars were used for the purpose of the present study. The teeth were stored in 0.2% thymol solution at 4°C and used within two months after extraction following informed patient consent, as approved by the Medical Ethics Committee of Isfahan University of Medical Sciences. The crowns of the teeth were separated from the roots, sectioned mesiodistally, and embedded in flat cylindrical acrylic resin molds in a manner that the buccal and lingual surfaces were placed horizontally. Buccal and lingual surfaces were used for enamel and dentin groups, respectively. Forty-eight buccal surfaces were ground at the vestibular enamel surface on wet silicon carbide papers up to grit 600 to achieve a flat surface. Lingual surfaces were trimmed until the dentin was exposed and then were ground on wet silicon carbide papers up to grit 600 to create flat dentinal surfaces. After preparing 96 flat enamel and dentin surfaces, the surfaces of the specimens in each group were conditioned with 20% aqueous polyalkenoic acid cavity conditioner (GC, Tokyo, Japan; Table 1) prior to the application of the restorative material. Cylindrical plastic molds with identical thicknesses (2 mm of internal diameter and a height of 1 mm; Orthorings, Ortho Organizers Inc, Carlsbad , CA, USA) were fixed on the surfaces at room temperature (22 ± 1°C). Fuji II LC RMGIC was used in the present investigation (GC Improved Version, Tokyo, Japan; Table 1), supplied in capsules and mixed according to manufacturer's instructions for 10 seconds in a mechanical mixer (CapMix 1, 3M ESPE, St Paul, MN, USA). For enamel and dentin groups, sample subgroups (n=12) were prepared as follows:

1. Control. The material was mixed, injected into the mold, and then light activated according to manufacturer's instructions.
2. Delayed light activation. The material was mixed, injected in the mold, formed, and then allowed to set without the application of light for two minutes and then light activated according to manufacturer's instructions.
3. Prewarming. RMGIC capsules were immersed in a water bath at 40 ± 1°C for 90 seconds prior to activation, mixing, and injection into the cylindrical mold.
4. Prewarming and delayed light activation. RMGIC capsules were prewarmed similar to the procedure in group 3 and then mixed, introduced into the molds, and light activated two minutes after application similar to group 2.

Fuji II LC RMGI was polymerized using a halogen light-curing unit (Coltolux 2.5, Coltene AG, Feldwiesenstrasse Altstätten/Switzerland) with a light output power of 480 mW/cm^2. For all specimens, the distance of the light curing tip from the RMGI was 1 mm. After 24 hours of storage at 37°C, the specimens were exposed to 500 rounds of thermocycling

Table 2: *Bond Strength of the Specimens in the Enamel Groups in MPa (*p=*0.037)*

Groups	Group definition	Mean	SD	CI 95% LB	CI 95% UB	Min	Max
1	Control	9.34	3.56	7.07	11.60	5.09	15.92
2	2-min delay	11.92	5.07	8.40	14.84	5.73	22.29
3	Prewarming	8.94	4.77	5.90	11.97	4.77	22.29
4	Prewarming plus 2-min delay	9.18	4.45	6.34	12.00	3.18	19.10

Abbreviations: CI, confidence interval; LB, lower bound; UB: upper bound.

between 5°C and 55°C (Mp Based, KARA1000 Inc, Tehran, Iran) with a dwell time of 30 seconds and a transfer time of 12 seconds. Subsequent to fixation, the samples were tested for shear bond strength (SBS) at a crosshead speed of 0.5 mm/min by means of a universal testing machine (Dartec, HC10, Dartec Ltd, Stourbridge, UK). SBS values were calculated by dividing the force at fracture by the initial bonded area.

Two-way analysis of variance (ANOVA) was used to analyze the effect of curing protocol and prewarming on SBS using SPSS 11.5 software. Furthermore, one-way ANOVA and Tukey HSD *post hoc* tests were used to determine differences in SBS between the groups within the materials. Statistical significance was defined at $p<0.05$. The fracture modes of RMGI cylinders on enamel and dentin surfaces were evaluated under a light microscope at 16× and classified as follows:

1. Cohesive fracture: fracture in the RMGI or dental tissue
2. Adhesive fracture: fracture in the adhesive interface
3. Mixed fracture: adhesive/cohesive fracture (Tables 3 and 4)

Interface Evaluation by Scanning Electron Microscopy

In each experimental group, two additional specimens were prepared for evaluation by scanning electron microscopy (SEM). Subsequent to preparing each tooth according to the method described previously, the specimens were prepared by sectioning each specimen. The samples were dehydrated in ascending concentrations of ethanol (50%, 70%, 95%, and 100%) for 1 hour and embedded in acrylic resin and polished down using decreasing grit abrasive papers (400, 600, 800, 1200, and 1500; Buehler Ltd, Lake Bluff, IL, USA) and 0.5 μm diamond paste (Buehler Ltd) with a polishing cloth. Between each polishing step, specimens were put in an ultrasonic device for 10 minutes. The exposed interfaces were treated with 6 N hydrochloric acid for 30 seconds followed by a 10-minute immersion in 2.5% sodium hypochlorite. Subsequent to 10-minute ultrasonication, the specimens were dehydrated for 24 hours, affixed to an aluminum mounting stub, and sputter-coated with platinum-gold to a thickness of 10 nm for analysis under SEM. Different magnifications were used to provide SEM images at a distance of 20 mm. An accelerating voltage of 15.0 kV was used for the analysis.

RESULTS

Shear bond strength (SBS) values in MPa (mean ± SD), minimum/maximum values, and 95% confidence intervals for the groups are summarized in Tables 2 and 3. ANOVA revealed significant differences in SBS values among the enamel and dentin groups ($p<0.05$). Among enamel groups, group 2 specimens showed higher SBS values compared to other groups ($p<0.05$). No significant differences were observed between groups 3 and 4 and the control group ($p>0.05$; Table 2).

Regarding dentin groups, group 1 specimens showed higher SBS values compared to other groups ($p<0.05$). No significant differences were observed

Table 3: *Bond Strength of the Specimens in the Dentinal Groups in MPa (*p=*0.018)*

Groups	Group definition	Mean	SD	CI 95% LB	CI 95% UB	Min	Max
1	Control	13.79	5.18	10.5	17.09	7.96	22.29
2	2-min delay	8.88	2.99	6.98	10.78	4.77	14.33
3	Prewarming	7.96	2.25	6.52	9.38	3.18	11.14
4	Prewarming plus 2-min delay	7.56	2.89	5.72	9.39	4.77	14.33

Abbreviations: CI, confidence interval; LB, lower bound; UB, upper bound.

Table 4: *Different Fracture Modes in the Study Groups in Enamel Specimens,* N*(%)*

Mode of fracture / Groups	Adhesive	Cohesive	Mixed	Total
1 (control)[1]	6 (50%)	4 (33.4%)	2 (16.6%)	12 (100%)
2 (2-min delay)[1]	6 (50%)	5 (41.6%)	1 (8.4%)	12 (100%)
3 (prewarming)[2]	8 (66.6%)	3 (25%)	1 (8.4%)	12 (100%)
4 (prewarming and delay)[2]	9 (75%)	2 (16.6%)	1 (8.4%)	12 (100%)

[a] *Groups with the same superscript are not statistically different (*p*>0.05).*

between groups 2 and 3 and also between groups 3 and 4 ($p>0.05$; Table 3).

Two-way ANOVA revealed that SBS values in the four enamel groups were influenced by "delaying the irradiation procedure" ($F=3.861$, $p=0.037$), "prewarming" ($F=1.195$, $p=0.028$), and "delaying the irradiation/prewarming" ($F=4.618$, $p=0.043$).

Also, two-way ANOVA revealed that SBS values in the four dentin groups were influenced by "prewarming" ($F=12.493$, $p=0.001$) and "delaying the irradiation procedure" ($F=6.866$, $p=0.012$), and "delaying the irradiation/prewarming" ($F=4.954$, $p=0.031$).

One-way ANOVA for the study groups was significant ($p<0.05$). Multiple comparisons by Tukey test for enamel and dentin groups demonstrated significantly higher SBS values in groups 1 and 2 compared to the other groups, respectively ($p<0.05$).

Table 5: *Different Fracture Modes in the Study Groups in Dentin Specimens,* N*(%)*

Mode of fracture / Groups	Adhesive	Cohesive	Mixed	Total
1 (control)[1]	5 (41.6%)	4 (33.4%)	3 (25%)	12 (100%)
2 (2-min delay)[1]	5 (41.6%)	6 (50%)	1 (8.4%)	12 (100%)
3 (prewarming)[2]	8 (66.6%)	3 (25%)	1 (8.4%)	12 (100%)
4 (prewarming & delay)[2]	7 (58.3%)	4 (33.3%)	1 (8.4%)	12 (100%)

[a] *Groups with the same superscript are not statistically different (*p*>0.05).*

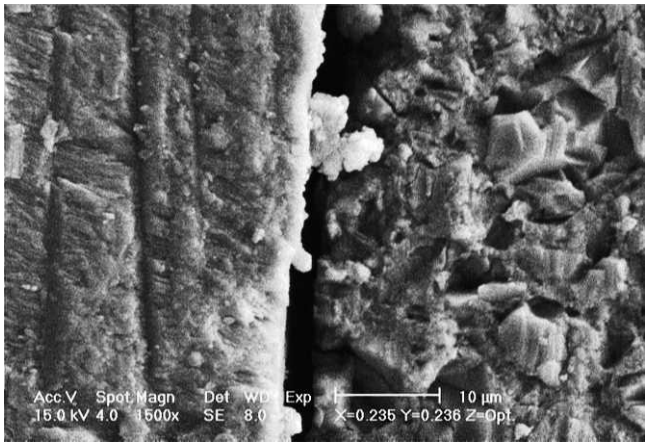

Figure 1. *Enamel/Fuji II LC interface in group 1. (Magnification = 1500×). In all the figures, the SEM photomicrographs of enamel and dentin study groups. As it appears in the photomicrographs, the resin interface area seems to be wider in groups 3 and 4. It seems that there is a better adaptation between the restorative material and tooth structures in groups 1 and 2. The glass particles seem to be larger, which might be attributed to less opportunity of the material to react with other particles and tooth structure subsequent to warming. (Magnification = 1500×).*

The fracture modes are summarized in Tables 4 and 5. According to the results, the majority of adhesive fractures were observed in groups 3 and 4 in both the enamel and the dentin group.

The SEM photomicrographs are shown in Figures 1 through 8 for enamel and dentin study groups. As it appears in the photomicrographs, the resin interface area seems to be wider in groups 3 and 4. There is a better adaptation between the restorative material and tooth structures in groups 1 and 2. The unreacted particles seem to be larger, which might be attributed to less opportunity for the material to react with tooth subsequent to heating.

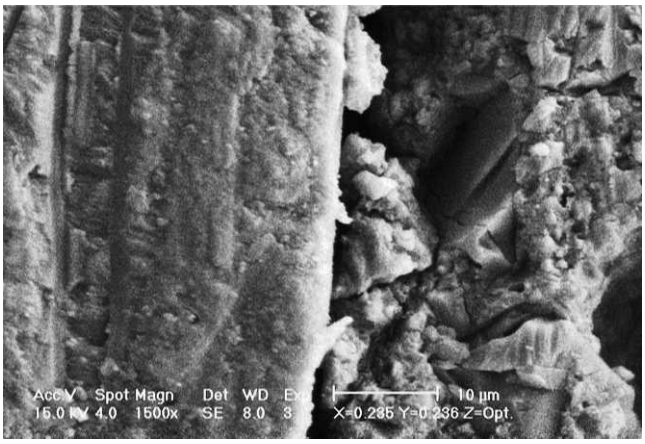

Figure 2. *Enamel/Fuji II LC interface in group 2. (Magnification = 1500×).*

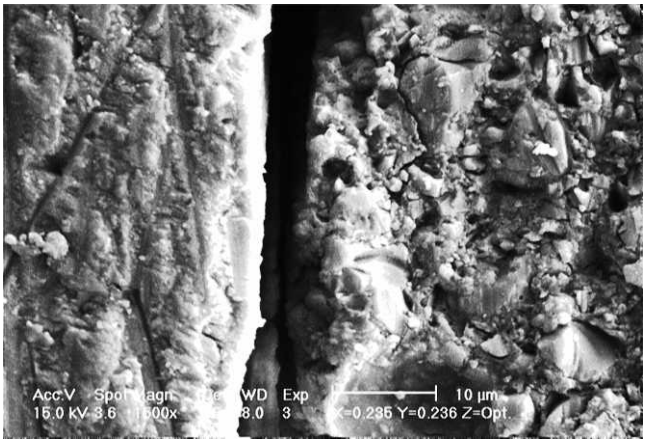

Figure 3. *Enamel/Fuji II LC interface in group 3. (Magnification = 1500×).*

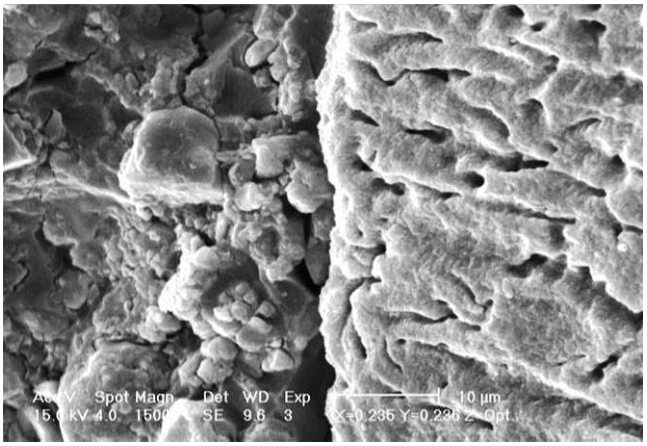

Figure 5. *Dentin/Fuji II LC interface in group 1. (Magnification = 1500×).*

DISCUSSION

Hybrid material types, such as RMGI, were developed in an attempt to improve mechanical properties, decrease setting time, and reduce moisture sensitivity of glass ionomers. However, RMGICs exhibit shrinkage and substantial water sorption because of their hydrophilic water-soluble resin content.[18]

RMGIs are a hybrid of glass ionomers and composite resins, and thus contain acid-base and polymerizable components. The setting reaction in glass ionomers is described as a series of overlapping stages.[6] Polyacrylic acid protons liberate metal ions and fluoride from the glass, forming a silica hydrogel around the glass surface. The high aqueous phase pH causes polysalt precipitates to form from the migrating ions, which act as cross-links to the polyacrylic acid chains. Setting times are approximately several minutes, although further maturation occurs over time.[19] Conversely, the resin reaction rate is much faster, although the complex photoinitiated polymerization process eventually results in a diffusion-controlled, polymer chain propagation as the concentration and mobility of monomer decrease during the formation of cross-linked matrix networks.[20–22] The final degree of conversion is dependent on monomer mobility and diffusion.[21]

In the present study, a cavity conditioner was used to remove the smear layer. The cavity conditioner (Fuji II LC I) consisted of polyacrylic acid (20%) and aluminum chloride (3%), which was applied to enamel and dentin surfaces for 10 seconds and then rinsed according to manufacturer's instructions. Earlier studies have reported that cavity conditioners are effective in improving the bond strength of Fuji II LC I restorative material.[23,24] According to the mentioned protocol, the initial bond strengths of the material under study to enamel and dentin were

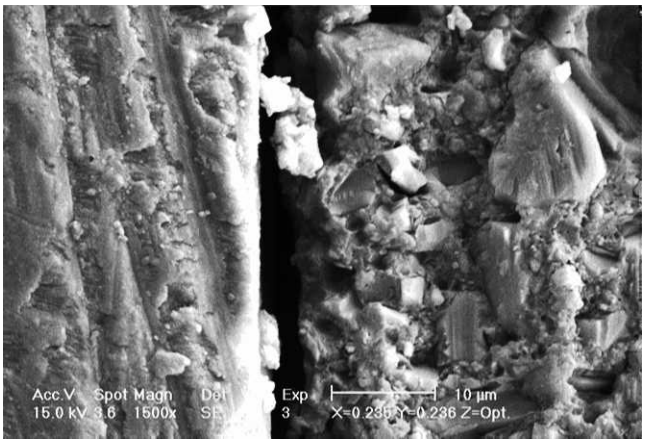

Figure 4. *Enamel/Fuji II LC interface in group 4. (Magnification = 1500×).*

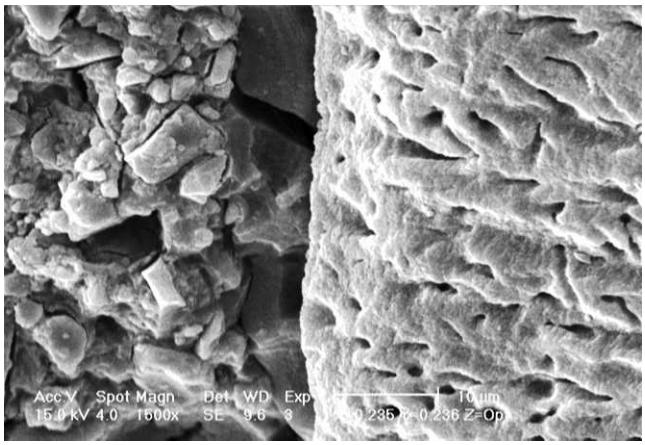

Figure 6. *Dentin/Fuji II LC interface in group 2. (Magnification = 1500×).*

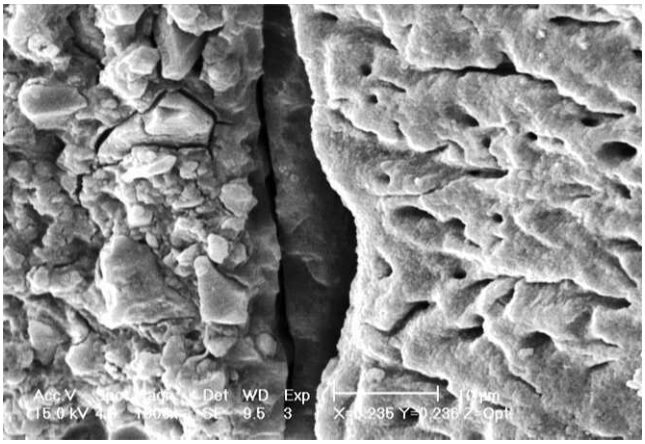

Figure 7. *Dentin/Fuji II LC interface in group 3. (Magnification = 1500×).*

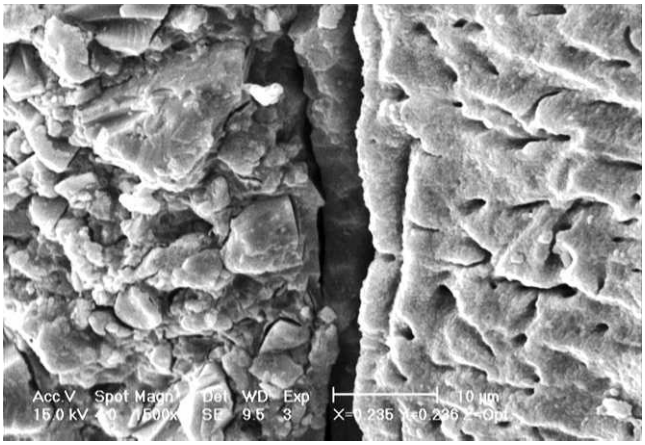

Figure 8. *Dentin/Fuji II LC interface in group 4. (Magnification = 1500×).*

9.34 ± 3.56 and 13.8 ± 5.18 MPa, respectively. The dentin bond strength values in the control group in the present study are consistent with the results of two previous studies.[25,26] In the second study,[26] the enamel and dentin bond strength values for Fuji II LC were reported to be approximately 10 and 13 MPa, respectively. Application of phosphoric acid has previously been reported to be effective in increasing the bond strength of light-cured glass ionomers to enamel.[27,28] It is probable that if a stronger acidic conditioner, such as phosphoric acid, is used for the enamel surfaces, a higher initial bond strength will be achieved when the material is light cured immediately after insertion, based on manufacturer's recommendations. In such a situation, it seems that it is better for the handling of the material to be similar to that with the composite resin, and delaying the light-activation procedure should be avoided so that acid-base reactions will not have an opportunity to proceed immediately; however, further studies are necessary in this regard.

In the present study, enamel bond strength increase and dentin bond strength decrease were observed in group 2 or groups with a two-minute delay, an increase in bond strength, that might be attributed to a greater opportunity of the material for acid-base reaction with the enamel surface, which is highly mineralized and rich in calcium and hydroxyapatite crystals. The shrinkage of glass ionomers has previously been reported to be around 3%.[5,18] Since delaying light activation has been parallel with a delay in material shrinkage and regarding the fact that a cavity conditioner with a weak acid was used in the present study, the results seem quite rational by taking into account the highly mineralized nature of enamel. As RMGIs are set by acid-base and polymerization reactions and each mechanism depends on reactant diffusion prior to gelation, it seems that the delay in RMGI light activation allows for greater acid-base reaction, reduces resin degree of polymerization[6], and results in an improvement in bond strength. However, in the case of dentin, it is probable that delaying the light-activation procedure makes the dentin surface more hydrophilic because the dentinal tubules are patent after acid conditioning with cavity conditioner. The cavity conditioner removes the smear layer, opening the dentinal tubules and making them more permeable.[22,24] Therefore, delaying the light-activation process contributes to the acid-base reaction of the material and probably decreases the concentration of the ions, decreasing the effect of the material on producing a stronger chemical bond.

In the present study, the effect of prewarming RMGI capsules on the bond strength to enamel and dentin was evaluated. Recent research has shown that conventional and light-cured glass ionomers set faster by the application of extrinsic energy sources such as ultrasonic energy or heat.[7,8,22] It is claimed that such energy application is efficacious in improving the surface topographical characteristics of glass ionomers in the early phases of setting reaction. O'Brein and others[18] evaluated the effect of application of two types of heat energy and one type of ultrasonic energy on the surface hardness of glass ionomer and reported that prewarming of glass ionomer capsules prior to mixing increases the hardness of the material compared to application of light or ultrasonic energy.

In the present study, group 3 enamel and dentin bond strengths decreased up to 30% and 45%, respectively, compared to the control groups. The

heating process in the present study was gradual: the process was carried out at 40°C for 90 seconds according to the procedure used in a study carried out by O'Brein and others.[18] This process is usually carried out at 55°C-60°C in the case of composite resin. Based on the results of the present study, the mechanical mixing of the material increased its temperature from 22°C to approximately 22.5°C. Since the material is supplied in powder and liquid forms, the prewarming process influences the temperature of both the powder and the liquid. After mechanical mixing, the temperature increased almost 0.5°C. After heating and mixing, the material temperature increased to 24°C (an increase of 2°C). Therefore, it appears that rising temperature results in a faster setting reaction, preventing the material from reacting properly with the substrate tissues. In addition, it is probable that heating helps compounds such as HEMA evaporate. Furthermore, the formation of poly-HEMA might be accelerated in the material while there is less opportunity for acid-base and free radical reactions.[18] Therefore, under the limitations of the present study, prewarming of hybrid glass ionomers is not recommended based on the results.

In the fourth group in the present study, the effect of two variables of pre-warming and delaying the light activation were evaluated. It was concluded that the effect of these two variables on the bond strength was not significant, which appears to be a logical result when the separate effect of each variable and the means of bond strength values in groups 2 and 3 and their comparison with bond strength values in group 4 are considered. Therefore, it is concluded that the prewarming variable had a negative effect on bond strength and yielded bond strength values similar to those in the control group (group 1) when it was combined with delayed light activation in group 4; this fact shows the insignificant positive effect of delaying the light-activation process compared to group 3.

Regarding dentin groups, it is probable that delaying the light-activation procedure and prewarming of the material can evaporate HEMA, help to rapidly form poly-HEMA, help the osmotic pressure and expel fluids out of the dentinal tubules, produce a more hydrophilic dentin surface with lower surface energy, dilute the ions present on the surface for acid-base reaction, and finally lead to an inappropriate surface for the polymerization of free radicals. Therefore, what happens in the case of RMGI is not recommended and is inconsistent with what happens in the case of composite resin, in which a higher flowability of composite resin is achieved, especially in the case of highly filled composite resins, resulting in a better bond strength and quality.[16,17] Simply stated, although prewarming of composite resins, especially posterior composites, has yielded positive results regarding bond strength and marginal seal,[16,17] the same results have not been achieved with RMGI.

Finally, the results of the present study showed that although the clinical behavior of RMGI regarding light activation according to manufacturer's instructions is similar to that of composite resins, changing the light-activation protocol and prewarming before mixing in particular do not necessarily yield the same results as those with composite resin. Therefore, more attention to the dual nature of the material and the theory of "network competition" is needed, especially in relation to its clinical behavior and its comparison with composite resins; in addition, further studies are necessary regarding the differences in the results with two principal tooth substrates (ie, enamel and dentin).

The results of the present study, in relation to the failure of RMGI in the groups under study, showed the effects of prewarming and prewarming/delaying light activation on increasing the rate of adhesive failures in the two enamel and dentin substrates in groups 3 and 4, which is consistent with the numerical bond strength values.

In the present study, given the limitations of SEM, the photomicrographs were not appropriate for comparison to extend the results to bond strength results. Certainly, meticulous TEM evaluations will be useful in this regard and are highly recommended.

CONCLUSIONS

Under the limitations of this in vitro study, the following may be concluded:

1. Delaying light activation of RMGI and/or prewarming prior to mixing in relation to dentin substrate cannot be recommended. The standard procedure recommended by the manufacturer yields greater bond strength.
2. Delaying the light-activation procedure and allowing for acid-base reaction in the RMGI material under study might have improved enamel bond strength after preparing enamel surfaces with a cavity conditioner, although further studies are highly recommended. Until then, it remains clinically advisable to use the conventional bonding directions recommended by the manufacturers.

Acknowledgements

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Influence of the Curing Mode on Fluoride Ion Release of Self-adhesive Resin Luting Cements in Water or During pH-Cycling Regimen

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Clinical Relevance
Within the limited experimental conditions, the self-adhesive cements provided fluoride ion release capacity. The fluoride release for all resin cements was not uniform during pH cycling, decreasing throughout the duration of the study.

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SUMMARY

This study evaluated the effects of curing modes and storage conditions on fluoride release of resin cements. In phase 1, the cumulative fluoride release rate from samples of the resin cements (Panavia F 2.0, RelyX Unicem, MaxCem, and BisCem) was quantified after 15 days storage in water (n=4). In phase 2,

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the fluoride release profiles from the same materials were analyzed during pH cycling (n=4). In this second phase, fluoride was measured at specific times (one, two, three, five, eight, and 15 days). Disk-shaped specimens were prepared (10 mm × 0.5 mm), and the materials were either light activated or allowed to autopolymerize. For both phases, the fluoride release was measured using a fluoride ion-specific electrode. The fluoride release in water was not affected by the curing mode of RelyX Unicem and Maxcem resin cements. Panavia F. 2.0 and BisCem resin cements, either light cured or autopolymerized modes, released higher amounts of fluoride in water than the other self-adhesive cements. In phase 2, the concentration of fluoride released decreased from the first day of pH cycling until the 15th day for all resin cements, for both curing modes, regardless of the storage solution used (demineralizing/remineralizing). The fluoride release rate during pH cycling by Panavia F 2.0 and MaxCem was not affected by the curing mode. The effect of the curing mode on fluoride ion release in water or during pH cycling was product dependent.

INTRODUCTION

Fluoride application to tooth structures has influenced the decline in the prevalence and severity of dental caries.[1,2] Many studies have shown that the ion is safe and effective in preventing and controlling caries development under specific dosages. Such benefits are due to its ability to inhibit the demineralization and enhance remineralization.[3-5]

A number of studies have suggested that the fluoride released from glass ionomer cements can reduce the demineralization of the surrounding dentin or enamel and favor the remineralization of lesions close to the restoration wall.[6-9] Glass ionomer cements contain calcium fluoroaluminosilicate glass, which is attacked by polyacids to release cations and fluoride ions. These ions react with polyanions to form a salt gel matrix that presents small particles of silica gel containing fluoride crystallites. The preventive and therapeutic effects of glass ionomer cements are attributed to the discharge of fluoride ions, and this releasing does not affect the physical properties of the cement.[10]

Other fluoride-containing restorative materials, such as restorative composites, resin cements, and adhesive systems, have been developed to prevent recurrent caries; however, glass ionomer cements show a fluoride-recharging ability and have demonstrated more prolonged fluoride release than these materials. Most of these resin-based materials contain different fluoride sources, and the amount of fluoride released seems to depend on the brand and other characteristics of each material.[9,11-15]

The self-adhesive resin cements were recently developed, and the main advantages of using these materials include a shorter clinical application time and technique sensitivity reduction.[16-19] In addition, self-adhesive resin cements contain different fluoride sources, and limited information is available regarding their ability to release sufficient fluoride concentrations in water or during a cariogenic challenge. Thus, the purpose of this study was to evaluate the effects of curing modes and storage conditions on fluoride release from dual-polymerizing resin cements. The research hypotheses tested were that 1) curing mode affects the amount of fluoride release in water, regardless of type of resin cement used, and 2) the fluoride release profile of resin cements is uniform during pH cycling, regardless of the resin cement and the curing mode.

METHODS AND MATERIALS

Sample Preparation

Three self-adhesive dual-polymerizing resin cements, RelyX Unicem (3M ESPE, Seefeld, Germany), MaxCem (Kerr Corp, Orange, CA, USA), and BisCem (Bisco Inc, Schuamburg, IL, USA), and one conventional dual-polymerizing cement (Panavia F 2.0, Kuraray Med, Kurashiki, Japan) were used according to the manufacturers' instructions (Table 1).

Sixteen disks of each resin cement were prepared (total = 64 samples). The mixed base and catalyst pastes were dropped into individual cylindrical silicon molds (10.0 mm diameter × 0.5 mm depth, Aquasil Ultra LV, Dentsply Caulk, Milford, DE, USA), covered with a polyester strip and pressed with one microscope glass slide. Half of the total samples (32 discs) were light cured for 40 seconds (XL 3000, 3M ESPE, St Paul, MN, USA), and the other half were allowed to autopolymerize only. Thirty-two resin cement discs were evaluated according to the cumulative fluoride release rate in water, and thirty-two samples were prepared for fluoride release profiles during the pH-cycling regimen. All resin cement samples were stored at 37°C for 48 hours (n=4). A schematic diagram of the study design is shown in Figure 1. After curing, the disk-shaped samples were removed from the silicon molds, and the excess material was removed using

Table 1: *Compositions of the Resin Cements Used in This Study*

Resin Cement	Composition
Panavia F 2.0	Paste A: 10-MDP, silanated colloidal silica, bisphenol A polyethoxy dimethacrylate, hydrophobic and hydrophilic DMA, silanized silica filler, benzoyl peroxide, dl-camphorquinone
	Paste B: hydrophobic and hydrophilic DMA, sodium 2,4,6–triisopropyl benzene sulphinate, N,N-diethanol-p-toluidine, bisphenol A polyethoxy dimethacrylate, colloidal silica, sodium fluoride, silanized barium glass filler, silanized titanium oxide
RelyX Unicem (capsules)	Power: glass powder, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxy compound
	Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators
MaxCem	Multifuncional DMAs, GPDM, proprietary Redox initiators and photoinitiators, barium, fluoroaluminosilicate, fumed silica (66 wt.%)
BisCem	Bis-GMA, uncured dimethacrylate monomer, phosphate acid monomer, glass filler

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol-A-diglycidylether dimethacrylate; DMA, dimethacrylates; GPDM, glyceroldimethacrylate dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate.

600-grit SiC paper. Samples were individually fixed to orthodontic steel wires, allowing each to be suspended in each storage media.

Cumulative Fluoride Release Rate in Water

The self-cured and light-activated samples (n=4) were placed into polyethylene vials containing 2 mL of deionized water and individually stored for 15 days. The water was not renewed daily, and 1 mL of each vial was collected for the analysis of fluoride release. TISAB III (total ionic strength adjustment buffer, Orion Research Inc, Cambridge, MA, USA) was added to the collected samples at a ratio of 1:10 (buffer:sample), and the quantification of fluoride in water was carried out by an ion-selective electrode (96-06, Orion Research Inc, Cambridge, MA, USA), connected to an ion analyzer (Orion 720 A model potentiometer, Orion Research Inc, Cambridge, MA, USA), which was previously calibrated with a series of standard solutions. The concentration of fluoride released was expressed in $\mu g/cm^2$, and the data were analyzed by Kruskal-Wallis and Dunn tests ($p<0.05$).

Fluoride Release Profile During pH-Cycling Regimen

The self-cured and light-activated samples were submitted to 15 days of a pH-cycling regimen, simulating caries development (n=4).[9,20] Each day or cycle consisted of the individual immersion of the disk-shaped specimen in demineralizing solution (1.4 mM Ca, 0.9 mM P, 0.05 m acetate buffer, pH 5.0, 2 mL per specimen) for eight hours and remineralizing solution (1.5 mM Ca, 0.9 mM P, 0.1 M Tris buffer, pH 7.0, 2 mL per specimen) for 16 hours. The solutions (demineralizing and remineralizing) were renewed daily, and 1 mL of each solution was collected at the first, second, third, fifth, eighth, and 15th days for the analysis of fluoride release, which was carried out by the same methodology previously used.

Fluoride release was also recorded in $\mu g/cm^2$. Mann-Whitney and Wilcoxon tests were used to analyze the influence of curing modes and storage media (demineralizing and remineralizing solutions) on fluoride release, respectively ($p<0.05$). The Friedman Non-Parametric Repeated Measures Comparisons analyzed the fluoride release profile during the pH-cycling regimen ($p<0.05$).

RESULTS

The results of the experimental groups and the amount of fluoride released after 15 days of storage in deionized water for each resin cement in both curing modes are displayed in Table 2. The fluoride release of RelyX Unicem and MaxCem self-adhesive resin cements was not affected by curing mode, while light activation significantly increased the fluoride releasing of Panavia F 2.0 and decreased the

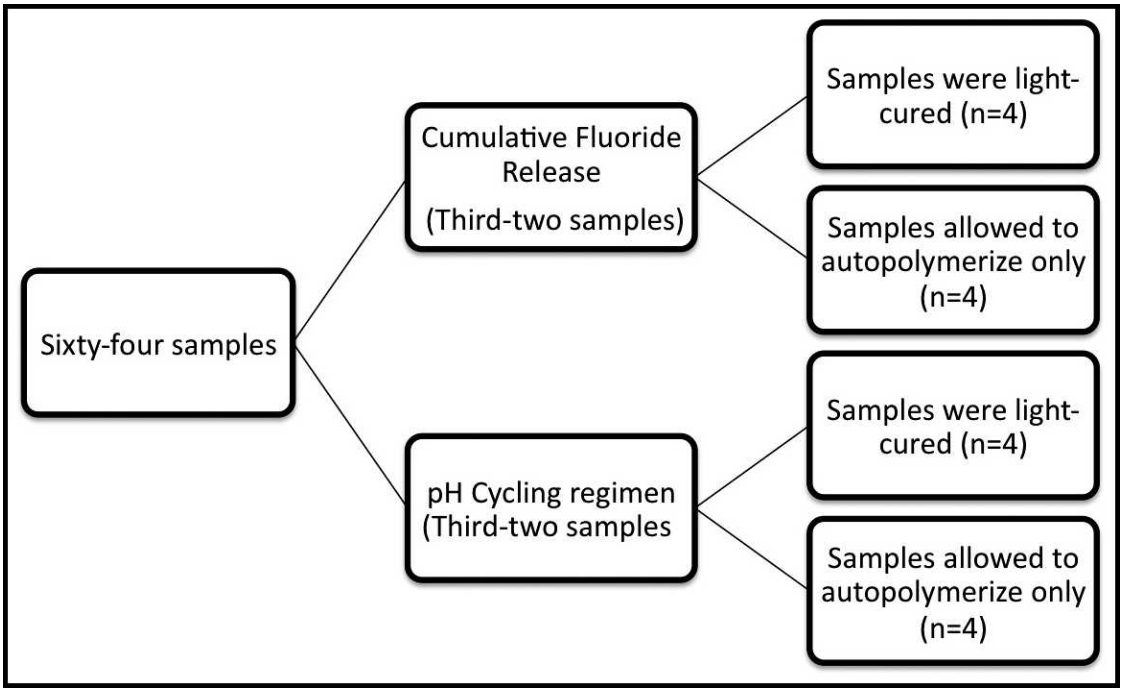

Figure 1. *Schematic diagram of study design: Four resin cements were tested in this study (Panavia 2.0, RelyX Unicem, MaxCem, and BisCem).*

releasing for BisCem self-adhesive cement. BisCem and Panavia F 2.0 released more fluoride ions in water than RelyX Unicem and MaxCem self-adhesive resin cements in both curing modes.

The fluoride release profiles during the pH-cycling regimen of the resin cements at each period are shown in Figures 2 (Panavia F 2.0, RelyX Unicem), 3 (MaxCem), and 4 (BisCem). In general, the concentration of fluoride released tended to decrease significantly from the first to the 15th day of pH cycling, regardless of the curing mode and storage solution (demineralizing/remineralizing) used.

Table 2: *Mean (Standard Deviation) of Cumulative Amounts of Fluoride Ions Released ($\mu g/cm^2$) From Resin Cements (Light or Self-cured) Over 15 Days*[a]

	Curing Mode	
Resin Cements	**Light Activated**	**Autopolymerized**
Panavia F. 2.0	9.5 (0.4) Aa	6.5 (0.1) Bb
RelyX Unicem	1.3 (0.2) Ab	1.3 (0.1) Ac
MaxCem	3.0 (0.2) Ab	2.7 (0.2) Ac
BisCem	8.9 (0.3) Ba	13.6 (0.6) Aa

[a] *Similar letters (uppercase, row; lowercase, column) are not statistically different ($p > 0.05$).*

For Panavia F 2.0 and MaxCem resin cements (Figures 2 and 4, respectively), the amount of fluoride released in the demineralizing or remineralizing was not affected by the curing mode at any time. Also, the fluoride concentration in the remineralizing solutions of these resin cements was higher than in the demineralizing solutions on the first day. At the second day, the four groups of Panavia F 2.0 resin cement released similar concentrations of fluoride until the 15th day, which tended to reduce the concentration to zero.

Fluoride-releasing behavior for RelyX Unicem during the pH-cycling regimen (Figure 3) showed that the autopolymerized samples released more fluoride ions in both demineralizing and remineralizing solutions than light-activated samples at the first day. RelyX Unicem and MaxCem self-adhesive resin cements continued to release fluoride until the 15th day, and the concentration measured in the solutions varied from 0.015 to 0.005 $\mu g/cm^2$ (Figures 3 and 4, respectively). From the second day until the 15th day, the four groups of RelyX Unicem or MaxCem cements presented similar mean values of fluoride release.

The fluoride release profile during the pH-cycling regimen for BisCem self-adhesive resin cement indicated that light activation reduced the fluoride release

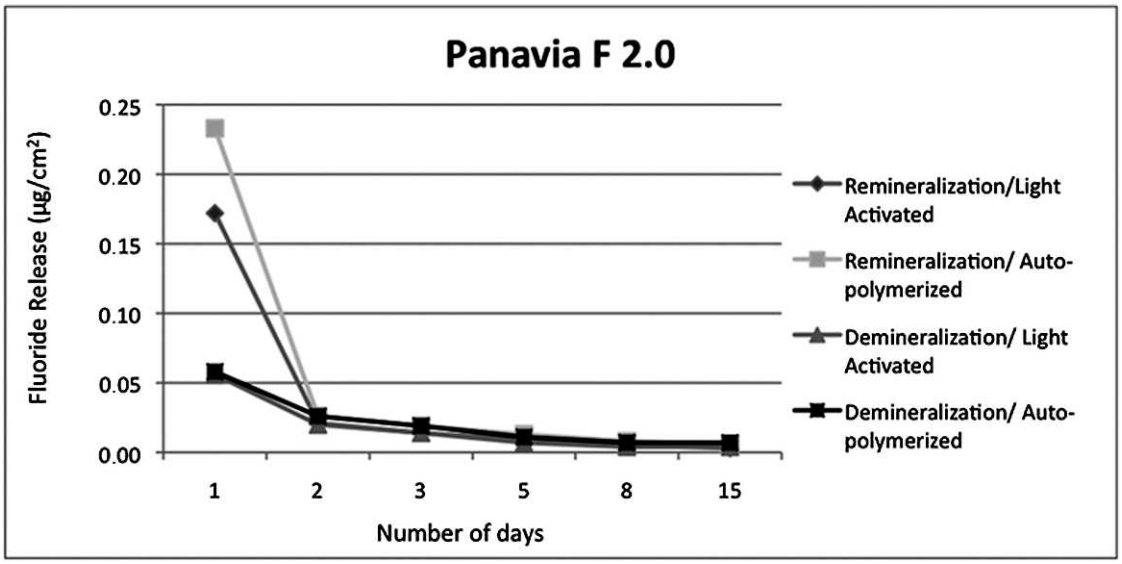

Figure 2. *Fluoride-releasing behavior for Panavia F 2.0 during the pH-cycling regimen (fluoride released [µg/cm²] as a function of elapsed time for up to 15 days).*

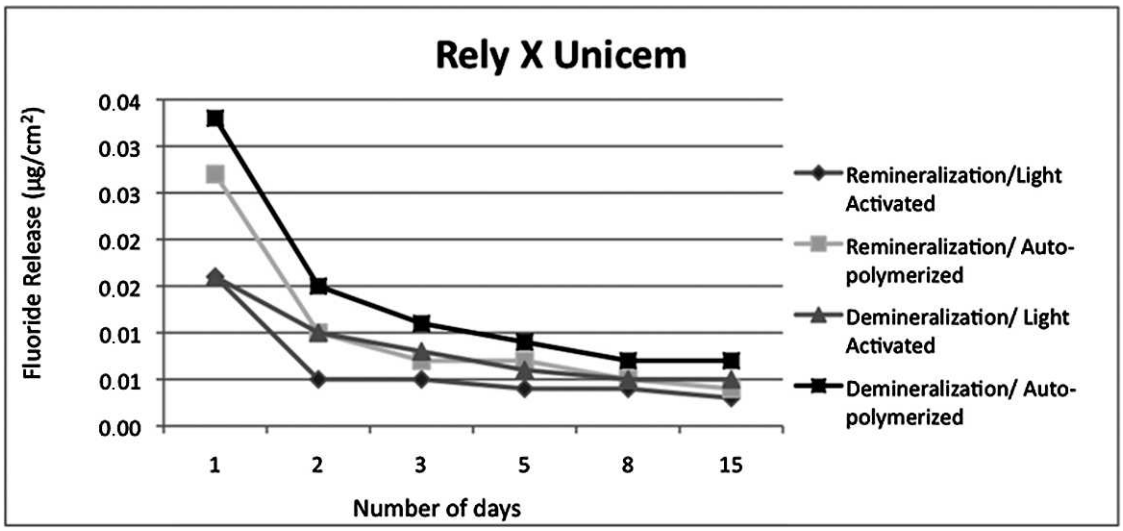

Figure 3. *Fluoride-releasing behavior for RelyX Unicem during the pH-cycling regimen (fluoride released [µg/cm²] as a function of elapsed time for up to 15 days).*

rate in both demineralizing and remineralizing solutions (Figure 5) at the first day. A low concentration of fluoride release was observed at the 15th day for all groups of BisCem cement (Figure 5).

DISCUSSION

The first hypothesis was partially accepted since fluoride release from the resin cements, RelyX Unicem and MaxCem, was not influenced by the curing modes. These cements released the lowest amount of fluoride in water, and no differences were observed between them in either curing mode. The water did not solubilize the high amount of fluoride ions from the polymerized resin matrix, even in an autopolymerizing mode that, theoretically, increases the resin matrix permeability for fluoride release.[21] On the other hand, BisCem had its fluoride release increased with the self-curing mode, and Panavia F 2.0 presented the opposite effect. BisCem and Panavia F 2.0 cements released more fluoride ions to water than RelyX Unicem and MaxCem self-adhesive resin cements in both curing modes. The composition of each resin cement, the solubility and permeability of the resin matrix, and the source,

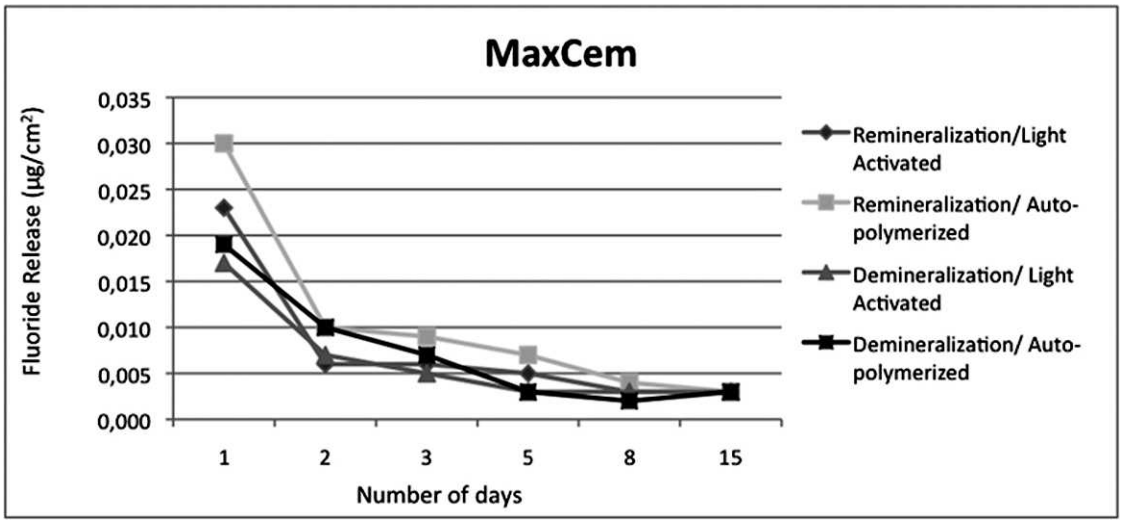

Figure 4. *Fluoride-releasing behavior for MaxCem during the pH-cycling regimen (fluoride released [µg/cm²] as a function of elapsed time for up to 15 days).*

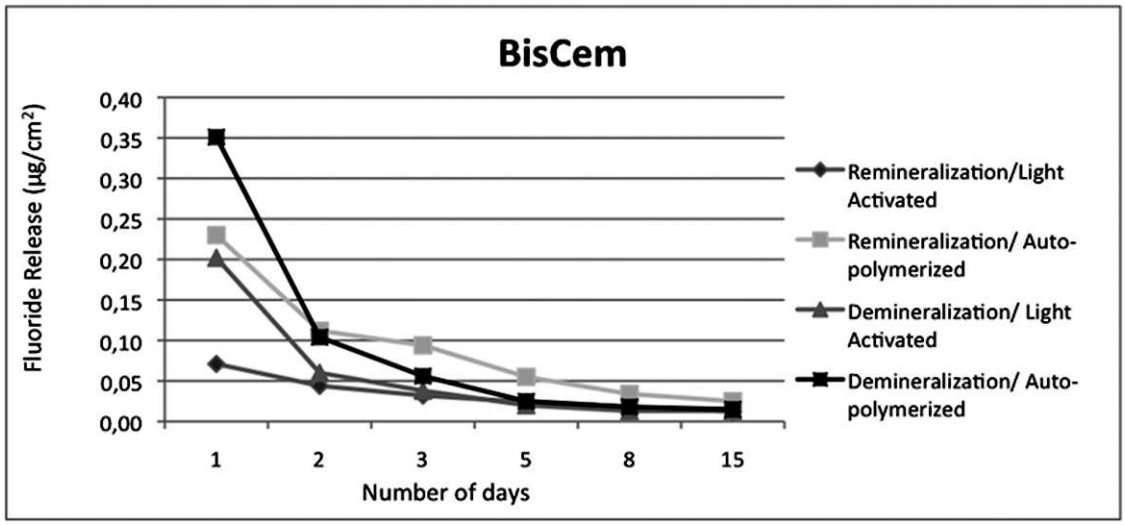

Figure 5. *Fluoride-releasing behavior for BisCem during the pH-cycling regimen (fluoride released [µg/cm²] as a function of elapsed time for up to 15 days).*

size, and concentration of the fluoride ions are important characteristics of the fluoride-releasing materials related to their ability to release the ions and, consequently, for the results of the first phase of this study.[9,14,21-23]

The fluoride source of Panavia F 2.0 is the sodium fluoride salt, which can be easily released by ionization when in contact with water or other aqueous solutions. For RelyX Unicem, the fluoride source is the glass powder, and the fluoride is available from similar acid-base reactions of glass ionomer cements.[17,24,25] MaxCem resin cement contains fluoroaluminosilicate particles that are similar to the glass powder found in RelyX Unicem. The similar fluoride source of both materials may explain the similar cumulative rate of fluoride released in water for RelyX Unicem and MaxCem (Table 2). BisCem self-adhesive resin cement contains glass fillers, which can be composed of fluoride glass. The glass fillers are presented in both base and catalyst pastes of BisCem cement and may be in relative concentration, which explains the amount of fluoride released in water.

Several studies have reported that the glass powder serves as a reservoir of fluoride.[26,27] RelyX Unicem and MaxCem self-adhesive resin cements

that contain these glass particles undergo a significant reduction in fluoride release during pH cycling, but it did not tend to zero after 15 days of cycling. For Panavia F 2.0 and BisCem cements, very low concentrations of fluoride were detected at the last day of cycling, showing that releasing did not remain constant for 15 days and the fluoride source of these materials seems to be finite.[21,22] As observed in similar fluoridated resin-based studies,[9,22,28] higher ion release was observed on the initial days for all resin cements tested in this study, demonstrating that the fluoride release was not constant or uniform during the pH-cycling regimen (Figures 2-5). Since the fluoride release decreased throughout the duration of the study, regardless of the resin cement and the curing mode used, the second research hypothesis was rejected.

In this study, the storage medium (demineralizing or remineralizing solution) influenced the amount of fluoride released only for Panavia F 2.0 and MaxCem resin cements during pH cycling. At the first day, higher amounts of fluoride ions were observed in the remineralizing solution than in the demineralizing solution. These results significantly differ from the fluoride release behavior of glass ionomer cements during pH cycling, in which the fluoride release rate increases in acidic conditions (demineralizing solutions), especially in organic acids, such as acetic, lactic, and citric acid solutions.[29-31]

Yoda and others[21] demonstrated that the curing mode and storage medium influenced the amount and rate of fluoride release from fluoridated luting cements materials (Fuji II LC glass ionomer cement, GC Corp, Tokyo, Japan, and Panavia F resin cement, Kuraray Med). The authors stated that the light curing of the dual-cure resin cement was essential to enhance the mechanical properties and increase immediate bonding to tooth structures. However, when the polymerization is only chemical, an increase of fluoride release is observed since light activation enhances cross-linking density and network quality,[32,33] resulting in a reduction of resin matrix permeability for fluoride ion release.

The effects of curing modes on the degree of conversion of the same resin cements was previously evaluated by Aguiar, and the results for RelyX Unicem and BisCem self-adhesive resin cements showed that the autopolymerizing mode yielded a lower degree of conversion value than did light activation ($p<0.05$).[34] The lower degree of conversion of self-curing mode can increase the resin matrix permeability for fluoride release, which may explain the increase of fluoride from the self-cured samples.[21] In both phases of this study (first phase, cumulative fluoride release rate in water; second phase, fluoride release profile during pH cycling), the BisCem cement released a higher fluoride concentration in self-curing mode. Conversely, the RelyX Unicem was not influenced by water media, and a higher concentration of fluoride was found for self-cured samples at the first day of pH cycling. Moreover, the acidic conditions of pH cycling possibly promoted more fluoride release. Since RelyX Unicem contains ionomeric components, this resin cement may show similar fluoride-releasing behavior as glass ionomer cements, which present an increased fluoride-releasing rate in acidic conditions.[29-31]

CONCLUSION

The analysis of fluoride release from resin-based materials is complex and depends on some clinical variables that are not present in an evaluation of glass ionomer cements. Only one resin cement (BisCem) was affected by the curing mode in both storage conditions (water and pH cycling). This resin cement released a higher amount of fluoride in a self-curing mode. The storage conditions changed the fluoride release profile of Panavia F 2.0 and MaxCem resin cements. For all tested materials, the highest fluoride release was observed at initial days of the study, tending to gradually diminish with time, according to the profile of each material.

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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Loading Standardization of Postendodontic Restorations *In Vitro*: Impact of Restorative Stage, Static Loading, and Dynamic Loading

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S Paris • M Naumann

Clinical Relevance

Dentists should read scientific papers carefully when searching for evidence for a specific treatment approach regarding postendodontic restoration, since inappropriate methodology may result in misleading clinical recommendations.

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SUMMARY

Objective: The load capability of post-restored endodontically treated teeth (ETT) can be determined at different restorative stages. It was the aim of this study to compare the load capability of ETT at these stages.

Materials and Methods: Maxillary central incisors were divided into 4 groups (n=10) and endodontically treated. Specimens were restored with: (I) only glass fiber posts (GFP); (II) GFP and composite build-up with 2 mm ferrule; (III and IV) with additional adhesively luted all-ceramic crowns. Group (I) to (III) were statically loaded, and group (IV) was exposed to thermomechanical loading (TML) and subsequent static loading.

Results: The lowest median load level of 73 N was determined for group (I). The maximum

median load value of 331 N was found for group (III). The comparison of Fmax [N] of group (I), (II) and (III) revealed significant differences between the groups ($p<0.001$). The specimens of group (IV) failed at significantly lower load values ($p<0.005$) as similarly restored specimens of group (III) which were only statically loaded. The stage of restoration and TML loading prior to static loading had a significant impact on fracture patterns ($p=0.006$).

Conclusion: Every additional restorative step torwards a final crown-restored ETT significantly increased the load capability. TML prior to load-to-fracture testing of the complete restorative complex, ie. post, core and crown, significantly decreased maximum load capability.

INTRODUCTION

In vitro studies are a valuable tool to determine mechanical properties of dental restorations and to allow preclinical risk assesment.[1,2] The ability to isolate specific variables in laboratory studies — compared with *in vivo* studies—enables the identification of parameters that are critical for clinical success.[3] Evaluation of load capability and failure mode of postendodontic restorations are commonly used as outcome parameters in *in vitro* studies.[4] Recently, numerous articles have been published investigating load behavior of endodontically treated teeth (ETT), primarily restored with adhesively luted, fiber-reinforced composite post systems.[4,5] *In vitro* tests show a significant influence of different post systems and materials on maximum load capability and failure mode. However, due to the variety of testing conditions, the results of these studies are hard to compare.[5–7] A recently published systematic review highlighted the variations of test parameters existing for *in vitro* testing of postendodontic restorations.[8] The most frequent specimen design was a combination of tooth, post, core buildup, and crown restoration (46%) followed by post-and-core restored specimens without crown (25%). Relevant recommendations derived from these studies can only be drawn when the specimens are finally restored.[9,10] However, the impact of specimen design used *in vitro* (a complex of tooth and post with or without core and with or without crown restoration) on maximum load capability is still not determined.[8]

Due to its efficiency, linear compressive loading until catastrophic failure is commonly used (~60%) to determine load capability of ETT.[8] However, this approach does not consider fatigue or aging, which are essential parameters during intraoral biodegradation of restorations of all kinds. The results and conclusions of quasi-static testing are affected, in part, by unknown variables, and correlations to clinical failure modes are unidentified.[11] Fatigue studies are assumed to be the most relevant source of information regarding the comparison of techniques and materials used for postendodontic restorations.[3] In recent studies, thermomechanical fatigue loads were used for preloading that assessed the meaning of the quasi-static load test. The most popular dynamic load test is the chewing simulation (thermomechanical loading [TML]) introduced originally for wear testing of dental biomaterials.[12] A modification of the thermomechanical protocol with an applied load of 30 N for 1.2 million cycles and simultaneous thermocycling of 10,000 cycles was specified for load testing of postendodontic restorations.[9,13–17]

It is important to note that no generally accepted test standard has been introduced to date for *in vitro* testing of postendodontic restorations. Thus, it was the aim of the present investigation to compare the maximum load capability and the mode of fracture of ETT at different restorative stages of postendodontic reconstruction. The teeth were restored either with a post, post-and-core, or post-and-core plus final crown restoration. The influence of the loading protocol, ie, with or without TML, on load capability of the crowned ETT was evaluated.

The following null hypotheses were assumed: 1) Maximum load capability of ETT is not influenced by the restorative stage; 2) Dynamic loading by TML does not influence load capability of finally crown-restored ETT.

MATERIALS AND METHODS

A total of 40 sound human maxillary central incisors with a minimum root length of 15 mm were selected. Teeth were stored for a maximum of one year in 0.1% thymol solution. To ensure an even distribution of specimen dimension among the experimental groups, the buccolingual and mesial-distal extensions at the cementoenamel junction (CEJ) were measured and the cross-sectional area was calculated as the product of these two parameters. According to these data, teeth were equally allocated to four groups ($n = 10$). Endodontic treatment was performed by gradual reaming to International Standards Organization size 60 and intermittent rinsing with 2.5% sodium hypochlorite. Roots were filled by

the lateral condensation technique with gutta-percha (Roeko, Langenau, Germany) and a sealer (AH 26, Dentsply DeTrey, Konstanz, Germany). The teeth were decoronated perpendicular to the long axis, 1 mm coronal from the most incisal point of the approximal CEJ under continuous water cooling using a diamond bur.

All specimens were blocked out with wax 2 mm below the finish line to imitate biologic width and to ensure a 2-mm ferrule preparation. To simulate a human periodontium, the roots of the teeth were covered with a 0.1-mm-thick layer of autopolymerizing silicone (Anti-Rutsch-Lack, Wenko, Wensselaer, Germany).[13] The teeth were embedded in autopolymerizing acrylic resin (Technovit 4000, Kulzer, Wehrheim, Germany), orienting their long axis facially 135° from the horizontal line.

In all groups, gutta-percha was removed (Gates Glidden burs (Dentsply Maillefer, Tulsa, USA)) leaving at least a 4-mm root canal filling apically. The root canal was prepared with a tapered drill of 1.4-mm maximum diameter (Fiberpoints Root Pins post kit, Schuetz-Dental, Rosbach, Germany) to achieve an intraradicular post length of 8 mm. All specimens received glass-fiber reinforced composite posts (Fiberpoints Root Pins Glass, Schuetz-Dental; diameter, 1.4 mm; length, 13 mm,) luted with a self-adhesive resin cement (RelyX Unicem, 3M ESPE, Seefeld, Germany) without dentin pretreatment. Posts were cleaned using 70% ethanol. The luting composite was filled in the root canal with an elongation tip by slowly pulling out the tip during cement application. Afterward the post was slowly inserted. The post was kept in place pressing the tip of the curing unit on top of the post. Light-curing was performed for two seconds (Optilux light curing unit, Demetron Research Corp, Danbury, CT, USA). Excess material was removed. Final light-curing was performed for one minute. Afterward the following protocols were performed.

Group 1 (Post Only)

The coronal portion of the post with a height of 5 mm was covered with a provisional filling material (Cavit, 3M ESPE). No further treatment was performed.

Group 2 (Post-and-Core)

After post placement, all specimens received a direct core buildup of a height of 5 mm (LuxaCore-Dual, DMG, Hamburg, Germany) using the corresponding bonding system (LuxaBond-Total Etch, DMG) according to the manufacturers' instructions.

Group 3 and Group 4 (Post-and-Core Restored With All-Ceramic Crowns)

The specimens were built up and prepared as in group 2. Twenty all-ceramic crowns (Empress II, Ivoclar Vivadent, Schaan, Liechtenstein) were fabricated in the original dimension of each tooth according to manufacturers' instructions. The crowns were adhesively cemented with a self-adhesive resin cement (RelyX Unicem, 3M ESPE).

Loading Protocol

Specimens of group 4 were exposed to dynamic loading by parallel thermal cycling and mechanical loading (TML; 1.2×10^6 cycles of mechanical loading, 3 mm palatally below incisal edge with a load between 1 and 49 N; 6000 simultaneous thermocycles between +5°C and +55°C for two minutes each in distilled water). The specimens that survived TML and all specimens of groups 1 to 3 were subjected to static linear loading in a universal material-testing machine (Zwick 1446, Roell, Ulm, Germany; cross-head speed of 1 mm/min) at an angle of 135° to tooth axis until failure occurred. Failure was defined as 10% loss of maximum applied force. For even stress distribution, a 0.3-mm-thick tinfoil was positioned between the steel piston and the specimens.

For all specimens, load capability F_{max} [N] was measured. Fracture patterns were recorded after a visual inspection (2.5× magnification).

Statistical Analysis

A nonparametric Kruskal-Wallis test was used, followed by the Mann-Whitney test as post hoc testing to study statistical differences in the maximum load capability F_{max} between the groups.

Fracture lines at or above the crestal bone level were judged as restorable. To test for differences in the fracture behavior—restorable or not restorable—among the groups, the χ^2 test was applied. All tests were two-sided with $\alpha = 5\%$.

RESULTS

Table 1 displays specimen characteristics, cycles until failure, mean load capability values (F_{max}) for all groups, and *p*-values of the statistical analysis. There was an equal distribution of the specimen characteristics within the groups. Thus, an even distribution for the risk to fail during specimen stressing can be assumed. Three specimens of group 4 failed during TML and were included with 0 N for further statistical analysis because no subsequent

Table 1: *Root Size and Length; Cycles Until Failure for TML Group 4 and Load Capability Values After Static Linear Loading for All Groups; n.p. = Not Performed*

Group	n	Cross-Section Area [a] (SD), mm^2	Root Length (SD), mm^2	TCML Early Failure, n	Cycles Until Early Failure, n	F_{max} Median Value (Min/Max), N	*P**
1	10	43.4 (4.5)	21.6 (1.6)	n.p.	—	73 (27/92)	A
2	10	43.4 (4.5)	22.5 (2.8)	n.p.	—	132 (85/377)	A
3	10	43.3 (4.6)	22.5 (1.6)	n.p.	—	331 (210/474)	A, B
4	10	43.3 (5.0)	22.9 (1.5)	3	825,847 391,799 391,902	241 (0/289)	B

[a] *at level of CEJ*
* *pairwise comparison* p-*value of* A ≤ *0.001;* B = *0.005.*

static linear loading was performable.[18] The number of cycles until failure is shown in Table 1. The F_{max} values among the four groups were significantly different ($p<0.001$) (Figure 2).

The lowest median load level of 73 N was determined for group 1 (post only). The maximum median load value of 331 N was found for group 3 (crown/no TML). The specimens of group 4 (dynamically loaded crown) failed at significantly lower load values ($p<0.005$) than similarly restored specimens of group 3 that were only statically loaded. The comparison of F_{max} [N] of groups 1, 2, and 3 revealed highly significant differences among the groups ($p<0.001$). Due to minimum F_{max} of 0 N in group 4, no significant differences were found among the groups 1, 2, and 4.

The respective fracture patterns are presented in Figure 1. The groups showed a statistically significant difference regarding the type of failure (χ^2: $p=0.006$). All fracture patterns were judged as re-restorable, given that fracture lines were located above the level of simulated crestal bone (Figure 1). In group 1 only bending of the post was observed. The post-and-core group 2 revealed mostly horizontal fractures of the composite core and two oblique fractures at the level of the finishing line palatally

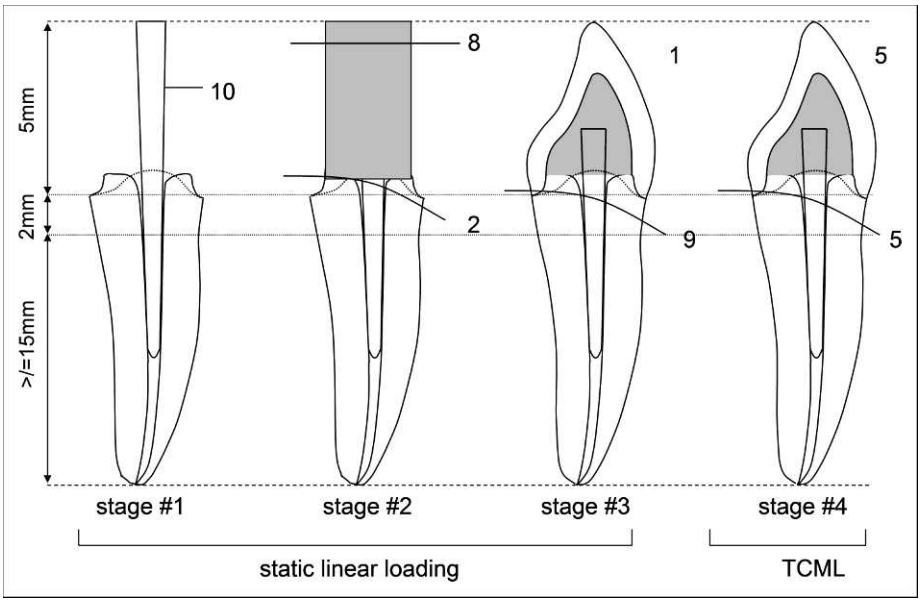

Figure 1. *Sectional view of experimental groups. Fracture lines of the specimens and frequencies of each fracture mode are indicated. Numbers lateral of the crown in groups 3 and 4 indicate crown fracture; interrupted line represents crestal bone level simulation.*

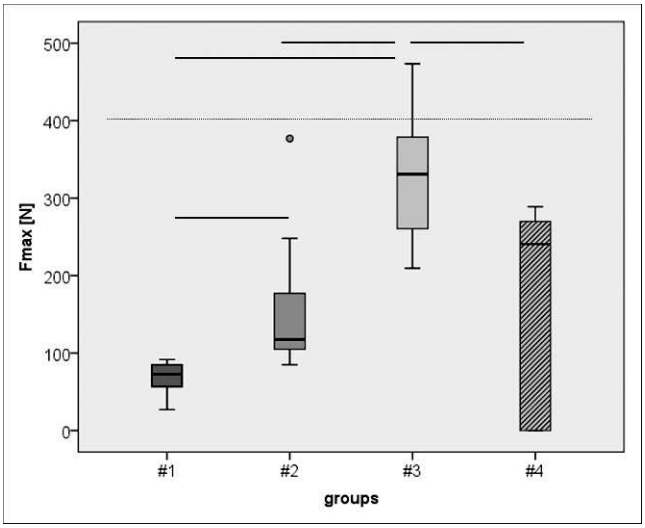

Figure 2. *Box plots illustrate the results of static linear loading (median, 25th and 75th percentiles); only group 4 was dynamically loaded before. Continuous line = $p<0.05$; interrupted line at 400 N level = maximum bite force observed clinically.*

and close to the simulated crestal bone level facially. In group 3 mainly oblique tooth fractures were found (Figure 3), whereas in group 4 an equal number of crown loosening and oblique fractures were observed. In groups 3 and 4 no glass fiber post fractured.

DISCUSSION

The present *in vitro* study was conducted to evaluate the impact of the restorative stage represented by the tested specimens and the type of loading, ie, dynamic with subsequent linear loading or linear loading only, on the maximum load capability of ETT. Specimens were restored with adhesively luted glass fiber posts only, posts and composite core buildups, or posts and composite core buildups with final crown restorations. The maximum load capability significantly depended on the simulated restorative stage of the restoration and dynamic loading (TML). A 4.5-fold linear increase of the maximum load-to-fracture from 73 N (post) up to a median value of about 331 N (post-and-core with all-ceramic crown restoration) was observed when linear loads were applied. TML prior to linear compressive loading significantly decreased the maximum load capability. Thus, both null hypotheses were rejected. The stage of restoration and dynamic loading (TML) prior to linear compressive loading had a significant impact on load capability and failure modes of post-restored ETT.

Figure 3. *Cross-section of a sample of group 3 with typical fracture line starting on the palatal aspect at the crown margin; after oblique progression of fracture line, the fracture ends facially at the level of crestal bone simulation.*

Human teeth were used because it was shown that substitutes are likely to behave differently in regard to the maximum load capability and mode of fracture.[19,20] Maxillary central incisors are an appropriate model for testing the restorative system in a worst-case scenario.[8] They can be regarded as a high-risk tooth type, given that shear forces in the anterior maxillary region are significantly higher compared with the posterior region.[21,22]

Glass fiber posts were used because *in vitro* and *in vivo* studies document their suitability when a minimum of 2-mm ferrule is provided.[23,24] Moreover, the potential of a self-adhesive cement to effectively bond to root canal dentin has been shown.[25] Esthetic requirements recommend a tooth-colored post system to improve the translucent properties of an all-ceramic crown restoration. In this context the teeth

were restored with a well-investigated all-ceramic crown system.[26,27]

The load capability at different stages of post-endodontic restoration of fiber post–restored specimens was also evaluated by Cormier and others.[28] A significant increase of the maximum load values was recorded, starting from the stage of post cementation to the stage of final crown restoration. However, the test condition provided, ie, load angulation, cross-head speed, tooth type, type of fiber post, and absence of TML as dynamic loading, limits the comparability to the present investigation. In accordance with our results, the stage of restoration caused a 2- to ~3.5-times increase of load capability, respectively, when a core buildup or a crown was added to the endodontic post.

To specify the material properties (eg, flexure strength, fracture toughness) of an endodontic post, a three-point loading test is advisable. The test arrangements used for load-to-fracture testing of ETT equates to the one-sided clamped beam test recommended for the determination of the modules of elasticity of small solid samples.[29] The load-bearing capacity, ie, the maximum load capability (F_{max}), is dependent on specimen design such as the lever arm, the specimen diameter and shape, and the moduli of elasticity of the materials involved. The concept of adhesive reconstruction of ETT is to create mechanically homogeneous units, so-called monoblocks.[30] Dependent on the number of bonding interfaces, endodontic monoblocks were classified as primary, secondary, or tertiary.[30] Irrespective of accomplishing the "ideal monoblock," the maximum load capability depends on the interaction among post, resin cement, core buildup composite, and crown restoration in order to evenly distribute loading stresses. It would be expected that with homogeneous units the increase of the maximum capability depends only on the diameter of the samples. Accordingly, the increase of the specimens' diameter from 1.8 mm (post, coronal level, group 1) to the cross-section dimension in stage of crown restoration, which averages 6.5 mm (Table 1) at the CEJ (groups 3 and 4), maximum load capability would theoretically have to increase 170 times.[31] Our results indicate only a 4.5-fold increase of F_{max}, which supports the impact of the interaction of the materials used—more precisely, the ability to appropriately bond to one another. In other words, adhesively restored ETT with post-and-core and crown restoration did not act as a monoblock. Consequently, to test the beneficial interaction of materials used for the reconstruction of ETT *in vitro,* the specimens have to be prepared in a comparable clinical manner and tested in a stage of final restoration.

The damage of the post in group 1 can be explained by bending the post during loading and fracturing the outer layer of the matrix subjected to the highest amounts of shear forces. Thus, the F_{max} values were determined by the flexural strength of the post material. In the post-and-core stage (group 2), maximum load capability did significantly increase. Due to the predominant cohesive failure of the composite core, load values were limited by the cohesive strength of the composite core material. Recently, the impact of various composite resin core materials on fracture strength of post-and-core restorations was confirmed when the same glass fiber–reinforced post was used.[32] The maximum load capability at the stage of final crown restoration is in a range comparable to that found in previous studies.[33,34]

The fracture patterns in the crown-restored groups 3 and 4 correspond to patterns found earlier.[24] There was no post fracture observed, although an unusual number of crown losses in group 4 was apparent. The fracture lines continued from the palatal aspect of the crown margin to the facial aspect of the root on the simulated crestal bone level. Due to tension palatally and compression facially caused by the palatal loading, an adhesive failure of the luting cement between tooth and restoration occurred. However, the marginal continuity of adhesively cemented lithium-disilicate ceramic crowns is more stress resistant when posts and cores were used compared with the restoration of ETT without posts even when cyclic loading was performed.[17] Recently, it was affirmed that the addition of a crown did not affect the strength of the restoration.[35] We believe that the maximum load capability is less affected by the strength of the post than by the (tensile) strength of the surrounding hard tissue that is directly correlated to its amount.[34] The most important requirement to achieve the maximum load capability of ETT is a remaining minimum of 2-mm cervical tooth structure, the so-called ferrule effect. To provide the ferrule effect, crown cementation is advisable.[36] Therefore, the evaluation of maximum load capability of postendodontic restorations requires a crowned specimen.

Additionally, our results show an influence of the TML because the maximum load capability was significantly reduced for the crowned specimens. The applied load of 49 N for 1.2×10^6 cycles as

mechanical loading with simultaneous thermocycling and the failure rate of 30% after TML are comparable to results of other studies recently published.[8,15,17,37] Repeated stress application is more clinically relevant[38] because in the oral environment mechanical and thermal stresses on the dental materials occur cyclically. Due to that, laboratory testing has moved toward laboratory simulation, which includes simultaneous thermal cycling and mechanical loading (TML) as a quasi-representation of oral condition[1] In most studies static loading was used,[8] although it is known that thermal cycling and in particular mechanical loading affect the results of load-to-fracture tests significantly.[9,39]

In principle, dynamic (cyclic) load application provokes the fatigue phenomenon, a time-dependent aging of the materials used for postendodontic reconstruction, such as core buildup composite resins, fiber-reinforced composite posts, and all-ceramic systems. For both thermocycling (TC) only and TML, a load-capability-reducing effect is evident.[40–43] As a function of the rigidity of the post, damage of the bond strength of a post bonded to root dentin was found after cyclical mechanical loading.[44] The durability of the marginal fit of fiber post and composite resin core–restored ETT significantly decreased with cyclic loading.[45] The fracture modes observed in group 4 did indicate, besides a fatigue behavior of the ceramic material initially, a debonding failure of the crown impaired by the TML.

The present investigation supports the need for standardization of *in vitro* testing embedded in a systematic approach to evaluate the materials involved in postendodontic restoration. The correlation between physical properties of the materials and the clinical behavior was only rarely shown. For the establishment of standards for postendodontic restorations further studies are needed to investigate several parameters influencing the load-to-failure behavior, as, for example, the simulation of the periodontal ligament. Some standards should be paid attention: The dental structure of interest should be "structurally representative." The test has to simulate the clinical situation as closely as possible. Best-case/worst-case scenarios are needed.[46] Thus, in the prediction of the clinical behavior of a postendodontic restorative complex more scientific work is necessary. Instead of unstandardized *in vitro* research, a validation of laboratory tests with clinical data achieved in high-level clinical studies is urgently needed.

CONCLUSION

Every additional restorative step toward a final crown-restored endodontically treated tooth increases significantly the load capability. Thermomechanical loading prior to load-to-fracture testing as performed in the present study for the complete restorative complex "post, core buildup, and crown" includes fatigue effects that significantly decrease the maximum load capability. The stage of restoration and type of loading, ie, linear or dynamic loading, should be given attention when load values of different *in vitro* studies are interpreted.

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Effect of Resin Cement System and Root Region on the Push-out Bond Strength of a Translucent Fiber Post

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Clinical Relevance

The use of self-adhesive resin cements is an option for bonding fiber-reinforced composite posts to root canal dentin. Traditional resin cements apparently provide higher bond strengths than self-etch resin cements. Because of this, the bond strength of self-adhesive resin cements to root dentin should be evaluated.

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SUMMARY

Objectives: This study evaluated the bond strength of luting systems for bonding glass fiber posts to root canal dentin. The hypothesis tested was that there are no differences in bond strength of glass fiber posts luted with different cement systems.

Methods: Forty bovine incisors were randomly assigned to five different resin cement groups (n=8). After endodontic treatment and crown removal, translucent glass fiber posts were bonded into the root canal using five different luting protocols (self-cured cement and etch-and-rinse adhesive system; dual-cured cement and etch-and-rinse adhesive system; self-cured cement and self-etch adhesive system; dual-cured cement and self-etch adhesive system; and dual-cured self-adhesive cement). Push-out bond strength was evaluated at three different radicular levels: cervical, middle,

and apical. The interface between resinous cement and the post was observed using a stereoscopic microscope.

Results: Analysis of variance showed a statistically significant difference among the cements ($p<0.05$) and the root canal thirds ($p<0.05$). The self-adhesive resinous cement had lower values of retention.

Conclusions: The resin cements used with etch-and-rinse and self-etch adhesive systems seem to be adequate for glass fiber post cementation.

INTRODUCTION

Posts and cores are frequently used in endodontically treated teeth that have suffered excessive loss of coronal tooth structure.[1,2] The choice of materials used in these cases has changed from rigid materials, such as gold and zircon dioxide, to materials that have mechanical characteristics that more closely resemble dentin, such as fiber posts and composite resins.[3,4] Use of these materials diminishes the probability of root fracture, because these failures occur particularly in the post, allowing for tooth recovery.[5]

The bonding performance of resin cements is dependent on the quality of the hybrid layer.[6-8] Some factors such as dentin morphology, bonding system, and luting cement and its cure may interfere with hybrid layer formation along the root canal walls, affecting post retention.[9,10] This hybridization is critical in the apical third of the post space because of difficulty in establishing adhesion in this area.

Various luting agents have been proposed for bonding fiber-reinforced composite (FRC) posts to root canal dentin used with self-etching or etch-and-rinse adhesive systems.[11,12] In recent years, new resin cement formulas have been developed that have a self-adhesive capacity. These cements have the advantage of not requiring any dentin pretreatment.[13]

Thus, the aim of the current study was to evaluate the bond strength of luting systems for bonding glass fiber posts to root canal dentin. The null hypotheses tested were as follows: 1) there are no differences in bond strength among the different regions of a root canal, and 2) there are no differences in bond strength among different cement systems.

METHODS AND MATERIALS

One-hundred forty freshly extracted bovine incisors with mature apices and without root curvature were utilized. A digital caliper was used to measure the teeth at three root regions: cervical, middle, and apical, in both mesiodistal and buccolingual directions. Forty teeth with similar measurements were used.

For endodontic treatment, a step-back preparation technique was used with stainless steel K-files and #2 to #4 Gates-Glidden burs (Moyco Union Broach, York, PA, USA). All enlargement procedures were followed by irrigation with 1% sodium hypochlorite. Prepared root canals were then filled with gutta-percha cones using the lateral condensation technique and AH Plus resin sealer (Dentsply, York, PA, USA). Subsequently, the filled roots were stored in distilled water at 37°C for 48 hours.

After storage, root canals were prepared to ensure a standardized space for post insertion. The crowns were removed and the canal space of each root was enlarged with a #3 Gates-Glidden, providing access for a #3 post drill, using a low-speed handpiece, to a depth of 11 mm. During preparation of the canal, 5 mm of the endodontic filling was left at the apex of each canal. The roots were randomly divided into five experimental groups (n=8). Double conicity translucent glass fiber posts (#3 White post DC, FGM, Joinville-SC-Brazil) and different resin cement systems were utilized in each group (Table 1).

In all groups, the posts were cleaned with 37% phosphoric acid for 60 seconds, followed by water-rinsing and air-drying. One coat of adhesive (Scotch Bond Multi-Purpose, 3M, St Paul, MN, USA) was applied, when necessary (only in Groups 1 and 2). To simulate clinical conditions, a wax protection barrier was applied to the external surface of the roots to prevent the passage of light.

For Groups 1 and 2, the root canal dentin was etched with 37% phosphoric acid for 15 seconds and rinsed for 30 seconds with water. After excess water was removed from the root canal with paper points, one layer of the "primer" was applied and gently air-dried for five seconds. Subsequently, the "adhesive" was applied, dried with paper points to remove the excess, and light-cured for 20 seconds with a halogen light-curing unit (Optilux 501, Kerr Co, Orange, CA, USA), with an intensity of 600 mW/cm^2. One coat of "adhesive" was also applied onto the entire post surface and light-cured for 20 seconds.

For cementation of the glass fiber posts, equal quantities of resin cement agents, base and catalyst, were mixed and applied onto the post surface and into the root canal with a periodontal probe. The post was then inserted and cemented into the root canal with light finger pressure, and excess material was

Table 1: *Groups and Composition of Luting Agents and Manufacturers*

Group	Material	Composition	Manufacturer
G1	C&B Cement	Bisphenol A diglycidylmethacrylate ethoxylated, Bisphenol A dimethacrylate silica glass frit, Bisphenol A diglycidylmethacrylate, triethyleneglycoldimethacrylate	Bisco
G2	Rely-X ARC	Silane-treated ceramic, triethylene glycol dimethacrylate (TEGDMA), Bisphenol A diglycidyl ether methacrylate (BISGMA), silane-treated silica, functionalized dimethacrylate polymer	3M
G3	Multilink	Pastes of dimethacrylates, hydroxyethyl methacrylate (HEMA), inorganic fillers, ytterbium trifluoride initiators, stabilizers and pigments, dimethacrylates, HEMA, benzoylperoxide	Ivoclar Vivadent
G4	Panavia F 2.0	Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments, others, sodium fluoride	Kuraray
G5	Rely-X U100	Glass powder, methacrylated phosphoric acid esters, triethylene glycol dimethacrylate (TEGDMA), silane-treated silica, sodium persulfate, glass powder, substituted dimethacrylate, silane-treated silica, sodium *p*-toluene sulfinate, calcium hydroxide	3M

immediately removed. For the self-cured cement (Group 1) (C&B, Bisco Inc, Schaumburg, IL, USA), seven minutes was allowed for complete polymerization. For the dual-cured cement (Group 2) (Rely-X ARC, 3M), light activation was performed through the cervical portion of the root for 40 seconds at the buccal and lingual surfaces, totaling 80 seconds of light exposure.

For Group 3, the luting procedure was carried out with self-cured material (Multilink, Ivoclar Vivadent AG, Schaan, Liechtenstein). This material was applied with the self-etching and self-curing Multilink Primer. Multilink Primer liquids A and B were mixed and applied in the root canal for 15 seconds and dried with paper points. The cement base and the catalyst were then mixed in a 1:1 volume ratio, and the same cementation procedure as already described for Group 1 was performed for this group.

For dual-cured cement used with the self-etch bonding system (Group 4) (Panavia F2.0, Kuraray Co, Ltd, Tokyo, Japan), self-etching and self-curing Primers A and B were mixed and applied into the root canal for 30 seconds and dried with paper points. The cement base and the catalyst were then mixed in a 1:1 volume ratio, and the same cementation procedure as was already described for Group 2 was performed.

Finally, for the self-adhesive resin cement (Group 5) (RelyX U100, 3M), dentin pretreatment was not necessary. The cementation procedure was carried out as previously mentioned for Group 2.

After cementation procedures were performed, specimens were stored in distilled water for 24 hours at 37°C. After the storage period, specimens were sectioned using an Isomet 1000 digital cutting machine (Buehler, Lake Bluff, IL, USA). The roots were divided into three parts, 1 mm from the cervical surface. Three 1-mm thick slabs, each separated by a 3-mm space, were obtained per root and identified as cervical, middle, and apical sections. The thickness of each root section was verified with a digital caliper (Digimess Direct, São Paulo, Brazil).

Immediately after the slabs were obtained, they were positioned on a push-out jig (1-mm diameter), which was placed on the universal testing machine (MTS 810 Material Test System, MTS, Eden Prairie, MN, USA) with a cell load of 50 kg at a crosshead speed of 0.5 mm/min until post dislodgment.

The retentive strength of the post segment was expressed in MPa. Data were analyzed using analysis of variance (ANOVA) followed by the Tukey test at a 5% level of significance ($p<0.05$) with GraphPad Prism 5 for Windows (GraphPad Software Inc, La Jolla, CA, USA) statistical software.

After push-out testing, the specimens were analyzed under stereoscopic microscopy to determine the failure mode[14]: type 1—adhesive between post and resin cement (no resin cement visible around the

Table 2: *Means (Standard Deviation) of Push-out Bond Strength in MegaPascal (MPa)**

Material	Region		
	Cervical	Middle	Apical
C&B Cement (G1)	9.5 (1.5)^{Aa}	8.6 (2.7)^{Aa}	7.7 (1.1)^{Aa}
Rely-X ARC (G2)	11.8 (2.6)^{Aa}	9.2(1.5)^{Aab}	7.7 (1.6)^{Ab}
Multilink (G3)	8.7 (2.0)^{Aa}	8.3 (1.3)^{Aa}	8.2 (0.8)^{Aa}
Panavia F 2.0 (G4)	8.3 (0.7)^{Aab}	10.2 (1.8)^{Aa}	6.7 (1.3)^{Ab}
Rely-X U100 (G5)	6.7 (1.2)^{Ba}	6.6 (1.1)^{Ba}	5.7 (2.7)^{Ba}

** Within each line, different lowercase letters mean statistically significant difference; within each column, different capital letters mean statistically significant difference (*$p<0.05$*).*

post); type 2—mixed with resin cement covering 0 to 50% of the post surface; type 3—mixed with resin cement covering between 50% and 100% of the post surface; type 4—adhesive between resin cement and root canal (post enveloped by resin cement); and type 5—cohesive in dentin.

RESULTS

Analysis of variance showed statistically significant differences among the cements ($p<0.05$) and the root region ($p<0.05$). Tukey test results are shown in Table 2.

No significant differences in bond strength were noted among groups G1 to G4. G5 (self-adhesive cement) showed statistically lower bond strength results. G2 (etch-and-rinse adhesive + dual-cured cement) demonstrated statistically lower bonding values in the apical region when compared with the cervical region. G4 (self-etching primer + dual-cured cement) had lower values in the apical third when compared with the middle third. G1 (etch-and-rinse adhesive + self-cured cement), G3 (self-etching primer + self-cured cement), and G5 (self-adhesive cement) had no significant differences along the root thirds.

The failure modes of the groups and root levels are presented in Table 3. No cohesive failure in dentin (type 5) was observed. A higher incidence of type 3 (53.3%) and type 4 failures (25%) was observed when compared with type 1 failure (6.7%). Type 2 failure was observed in 15% of cases.

Table 3: *Failure Classification at Different Levels of the Root Canal*

Group/Region	Type 1	Type 2	Type 3	Type 4	Type 5
G1					
Cervical	0	4	2	2	0
Middle	1	0	6	1	0
Apical	0	3	3	2	0
G2					
Cervical	0	1	4	3	0
Middle	1	1	4	2	0
Apical	0	4	2	2	0
G3					
Cervical	1	1	5	1	0
Middle	2	1	3	2	0
Apical	0	1	5	2	0
G4					
Cervical	1	0	7	0	0
Middle	0	1	6	1	0
Apical	1	0	5	2	0
G5					
Cervical	1	0	4	3	0
Middle	0	0	5	3	0
Apical	0	1	3	4	0
Total	8	18	64	30	0

DISCUSSION

Shear bond strength depends on the degree and stability of interfacial micromechanical interlocking and chemical adhesion between the root canal dentin, the dentin bonding agent/resin-based luting

cement/silane coupling agent, and the fiber post.[15] Microtensile pull-out and push-out tests have been traditionally used to assess the retention of posts in the root canal.[2] The push-out test is based on shear stress at the interface between dentin and cement, as well as between post and cement.[16] In the present study, the push-out test was performed 24 hours after adhesive cementation procedures because bond strength can increase during this period.[17]

The first null hypothesis, that there is no retention strength difference among the different regions, has to be rejected. Current results demonstrate that RelyX ARC (dual-cured cement) showed lower values in the apical third when compared with the cervical region. This could possibly be explained by limited light access to this region, leading to defective polymerization of the material.[18] Previous studies have shown that the use of translucent glass fiber posts may minimize this problem.[19,20] However, an increase in the ability to transmit light was insignificant for obtaining an appropriate degree of conversion of the cement, particularly in the apical region.[1,20]

Evidence indicates that a low degree of conversion does not necessarily reduce post retention.[21] The C-factor (ratio of bonded to unbonded surface areas of cavities) is critical when higher than 5.[22] In a root canal, the C-factor is always critical.[23,24] Depending on the diameter and length of the canal, the C-factor can range from 20 to 100[25] and may even exceed 200,[23] representing an unfavorable clinical situation. Shrinkage stresses in the confined space of the intact root canal can exceed cement-dentin bond strength, causing debonding.[23] In the canal apical levels, a lower degree of conversion may be an advantage, as it provides lower shrinkage stress, thereby reducing the C-factor impact.[26] This can explain the similar results with dual-cured (Rely-X ARC and Panavia) and self-cured (C&B Cement and Multilink) cements, which previously revealed low shrinkage stress.[23]

Other factors, such as moisture control in the apical region,[27] the presence of residual gutta-percha, and incomplete dentin hybridization,[28] may result in deficient sealing of the resin cement-dentin interface in the apical third. Values obtained in the apical region with the etch-and-rinse adhesive, especially when the Rely-X ARC luting agent is employed, might be due to the fact that this adhesive requires more complex procedures, and that moisture control in the apical third is compromised.

The self-adhesive cement (Rely-X U100) showed lower bond strength than the other cements. Possible deficient hybridization of dentin along the root canal walls may explain the lower results for self-adhesive cement in all thirds. Thus, the second null hypothesis, that different cements systems have no effects on the retention of glass fiber posts in root dentin, was also rejected.

Rely-X U100 has limited etching potential when compared with etch-and-rinse and self-etching adhesive systems.[11,29] This could possibly be explained by the methacrylated phosphoric esters present in this cement, which are not as effective as phosphoric acid in dissolving the thick smear layer in the root canal walls during post space preparation.[16,29] Additionally, this cement exhibits a low degree of conversion, even after light curing.[30]

Simplified adhesive systems (etch-and-rinse two-step and one-step self-etch) are incompatible with self-cured and dual-cured resin cements. This occurs by the presence of acid resin monomers in the nonpolymerized adhesive residual layer caused by oxygen inhibition, which react with the tertiary amine of the resin cement.[7,8] Moreover, these adhesives promote a permeable hybrid layer, allowing water diffusion from the dentin and forming water droplets along the adhesive resin-cement interface.[31] For this reason, a three-step etch-and-rinse adhesive system was used in this current study.

One-step self-etch adhesives were used for Multilink and Panavia resin cements because they belong to these luting systems. Nevertheless, results with these cements did not differ statistically from those seen in Groups 1 and 2. For the Panavia resin cement, this may be explained by the fact that cement tertiary amines are consumed by simplified adhesive residual acid monomers. The results for Multilink cement were similar to other non-self-adhesive cements. This can be explained by the chemical curing system that reduces shrinkage stress and consequently decreases adhesive layer injuries.[23]

Some studies have reported that the main retention mechanism of posts in root canals is not adhesive but frictional.[16,17] In the present work, excellent post adaptation to the canal walls in all regions of the root was obtained. This post adaptation, besides improving the frictional mechanism, reduces the resin cement coating, which contributes to minimizing the effects of polymerization shrinkage.

CONCLUSION

The use of resin cements to bond glass fiber posts is an attractive clinical concept. Results of this present study indicate that etch-and-rinse and self-etch

adhesive systems, when combined with resin cements, are good options for bonding glass fiber posts. Self-adhesive resin cements exhibited the lowest bond strengths to dentin. Moreover, the lowest bond strength was found in the apical third of the root canal spaces.

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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In Vitro Evaluation of the Effect of Delaying Toothbrushing With Toothpaste on Enamel Microhardness Subsequent to Bleaching the Teeth With 15% Carbamide Peroxide

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Clinical Relevance

Delaying oral hygiene procedures during bleaching does not seem to cause any change in enamel microhardness.

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SUMMARY

Changes in enamel surface microhardness as a result of bleaching with carbamide peroxide in various *in vitro* conditions have been reported. The present study evaluated the effect of oral hygiene procedures on enamel microhardness at three time intervals following bleaching with 15% carbamide peroxide. Although this was an *in vitro* study, the purpose was to address whether or not a patient's toothbrushing following at-home bleaching might affect surface changes in tooth enamel. Eighty enamel slabs were prepared from impacted human third molars that had been extracted surgically.

Subsequent to placing the specimens in acrylic resin, their surfaces were smoothed, and they were randomly divided into four equal groups. The specimens were initially evaluated for microhardness by Vickers test. The bleaching procedure was carried out for 21 days for 6 hours daily. In each group, the surfaces of specimens were brushed with toothpaste immediately, 1 hour, and 2 hours after bleaching except for the control group. The specimens were stored in artificial saliva. Enamel microhardness was again measured at the end of the bleaching period. Then the differences in enamel microhardness between the two periods were calculated. Data were analyzed with a nonparametric Kruskal-Wallis test at a significance level of $p<0.05$. The differences in the microhardness values before and after intervention between the groups were not significant ($p=0.59$). Daily oral hygiene procedures either immediately or 1 or 2 hours after daily bleaching procedures and exposing the specimens to artificial saliva during the study period produced no significant differences in enamel microhardness values.

INTRODUCTION

One of the techniques available for whitening the teeth is "at-home vital bleaching technique," with all the steps carried out by the patients at home. Except for the fabrication of the bleaching trays, the dental practitioner has nearly no control over the procedure.

Bleaching can result in structural changes on tooth surfaces. However, there is controversy over the effect of carbamide peroxide–containing bleaching agents on enamel microhardness. Some studies have demonstrated surface deterioration, formation of defects on the surface, and porosity in electron microscope studies and decreases in enamel hardness in microhardness studies.[1-5] Daily oral hygiene procedures (toothbrushing with toothpaste) are carried out by the patient during the bleaching period, which may intensify the destructive effects of the procedure on enamel surfaces.[6] On the other hand, saliva or other remineralizing agents can create an environment to help remineralize the teeth following bleaching.[7-10] Enamel microhardness is a property that can be influenced by a combination of the above-mentioned factors.

In the present study we evaluate changes in enamel microhardness subsequent to bleaching and brushing at three time intervals and storing the specimens in artificial saliva during the study period. The null hypothesis was that enamel microhardness is not influenced by the time the oral hygiene procedures are initiated after bleaching (immediately, and at 1-hour and 2-hour intervals after bleaching).

MATERIALS AND METHODS

In the present study the specimens were prepared from impacted human third molars that had been surgically extracted. The teeth were stored in a 0.5% chloramine T solution (pH=8-11) until used. Impacted third molars were included in the study because there are no changes on enamel surface in such teeth. The teeth that had any enamel surface abnormalities or had undergone cracks or fractures during surgical extraction were excluded from the study.

Subsequent to cleaning the teeth the roots were cut away at the cementoenamel junction. Two enamel slabs measuring $6 \times 4 \times 2$ mm were prepared from the middle third of the buccal and lingual aspects with the use of bilateral diamond disks (D&Z, Berlin, Germany). Eighty enamel slabs were prepared from 40 human third molars. The slabs were checked for any cracks or fractures. Water spray was used during specimen preparation to avoid dehydrating the specimens. The specimens were stored in distilled water at 37°C after cutting. Then the slabs were placed inside cold-cured acrylic resin in a cylindrical mold with a diameter of 1.5 cm, with the enamel surface on top. Subsequent to removal of the slabs from the mold, the surface of the enamel slab was prepared to a horizontal surface to be properly placed under the device which measures the microhardness; a flat-end tapered diamond bur (Teezkavan, Tehran, Iran) was used to this end. Then the enamel surface was smoothed using white aluminum oxide stones (Dura White, Shofu Dental Corp, Kyoto, Japan) under water spray. Finally, a piece of felt cloth along with 1- and 6-μm abrasive diamond pastes (Microdent, Sao Paulo, Brazil) was used for polishing the enamel surfaces.

The specimens were placed in an ultrasonic device containing distilled water for 10 minutes to remove polish debris. Then the 80 slabs were randomly divided into four groups of 20 specimens each, as follows:

- Group A (control): No hygiene procedures after bleaching
- Group B: Hygiene procedures immediately after bleaching

- Group C: Hygiene procedures 1 hour after bleaching
- Group D: Hygiene procedures 2 hours after bleaching

Microhardness values of all the specimens were measured and recorded before the study. In order to measure microhardness a 10-g force was applied for 30 seconds on the specimens by Vickers microhardness indenter (FM-700, Future Tech Corp, Tokyo, Japan). Microhardness of each specimen was measured at three separate locations 500 μm apart from each other and a mean was reported for each specimen.

Subsequent to measuring the initial microhardness, the bleaching process was instituted. To this end, a special tray was fabricated for each specimen in a vacuum apparatus; each tray was made of ethylvinyl acetate and was 1 mm thick. Then 0.02 mL of 15% carbamide peroxide gel (Opalescence PF, Ultradent Products Inc., South Jordan, UT, USA) was placed inside each tray; the tray was placed on each specimen for 6 hours daily. During the process each specimen covered with the tray was placed in a separate vial containing artificial saliva, which was replaced daily. The composition of the artificial saliva was as follows: $CaCl_2$ 1.0 mM, KH_2PO_4 3.0 mM, and NaCl 100 mM; the pH was 6.30 and was adjusted with NaOH solution.[8]After the bleaching procedure every day, the specimens were rinsed with deionized distilled water for 5 seconds. Then the subsequent steps for each group were carried out as follows:

- Group A: The specimens in this group were placed in 1 mL of artificial saliva at 37°C in an incubator for 18 hours after the bleaching and rinsing procedure.
- Group B: In this group the specimens were brushed immediately after they were bleached for 6 hours and rinsed for 5 seconds; the specimens were brushed with an electric brush (Oral-B Vitality Precision model, Oral-B Corp, Belmont, CA, USA) inside a reservoir of freshly prepared toothpaste (Opalescence whitening toothpaste, Ultradent) with one part (50 g) of toothpaste in three parts (150 g) of deionized distilled water. The brush was fixed on a bar with a clamp, and brushing was carried out once daily for 3 minutes with a typical force of 200 g. The amount of the force applied was measured with an orthodontic gauge. The brush head was made of nylon and was multitufted. A separate and specific brush was used for each specimen. The specimen was placed inside the solution which was agitated before use. The toothpaste specimen was replaced every 2 days so that a neutral pH was maintained. After daily brushing the specimens were rinsed with distilled water and stored in special containers containing artificial saliva at 37°C for the rest of the day.
- Group C: The same brushing procedure as in group B was repeated in this group except that after bleaching and rinsing, the specimens were kept in artificial saliva for 1 hour, after which the brushing procedure was carried out. Then the specimens were once again stored in artificial saliva until the next day.
- Group D: The procedure was the same as that in group C, but there was a time interval of 2 hours after bleaching for the brushing technique to begin.

The bleaching and cleaning procedures continued for 21 days in all the groups. At the end of this period the enamel microhardness values of the specimens were once again measured, recorded, and compared with the initial values. Data were analyzed with a nonparametric Kruskal-Wallis test. Statistical significance was defined at $p<0.05$.

RESULTS

Table 1 demonstrates the descriptive statistics of mean differences in microhardness values before and after intervention in the groups under study.

Before the study was initiated the means of microhardness values in the four groups were compared. As such, the nonparametric Kruskal-Wallis test showed that there were no significant differences in the means of microhardness values before intervention between the four groups ($p=0.89$).

Since data were widely distributed, logarithmic transformation of the data was considered and then a nonparametric Kruskal-Wallis test was used to evaluate the differences. The nonparametric Kruskal-Wallis test did not demonstrate any significant differences in the means of microhardness values before and after intervention between the groups under study ($p=0.59$). The linear graph and error bar of the mean differences in microhardness values before and after intervention in the groups are presented in Figure 1.

DISCUSSION

In the present *in vitro* study, an attempt was made to simulate a clinical course of an at-home bleaching procedure as exactly as possible. The procedure

Table 1: *Descriptive Statistics of Mean Differences in Microhardness Values Before and After Intervention in the Groups Under Study (ΔMH = Final Value – Baseline Value)*

Group	Mean (ΔMH)	95% Confidence Interval		Median	Lowest Value	Highest Value
A	−8.36	−41.94	25.21	−23.66	−139.33	125.33
B	−13.58	−43.00	15.83	−9.16	−176.33	79.00
C	−18.80	−34.09	−3.50	−13.16	−95.33	20.00
D	8.00	−21.32	37.32	−0.83	−116.00	152.33

lasted 21 days, 6 hours daily, with 15% carbamide peroxide. In addition, daily routine tooth brushing was carried out with the low-abrasive fluoridated toothpaste suggested by the manufacturer after each daily bleaching procedure. Artificial saliva was used to store the specimens during the bleaching period to simulate the physiologic conditions of the oral cavity. According to the results of the present study, although the changes in the enamel microhardness after 2 hours of storage of the bleached specimens in artificial saliva before brushing had an ascending trend, differences among the groups were not statistically significant.

Some studies have reported that bleaching agents can significantly decrease microhardness.[9-12] In addition, some of the complications of the use of bleaching agents on enamel include changes in the chemical composition of teeth, changes in the mineral content of dental structures such as calcium and phosphate,[13] changes in enamel fluoride content,[11] topographic changes and increase in enamel porosity, open enamel prisms, and an appearance similar to etched enamel.[9] According to some reports, the acidity of carbamide peroxide and the presence of glycerin and Carbapol influence the physicochemical structure of teeth, factors that are believed to be responsible for tooth hypersensitivity during bleaching.[14]

Despite what was previously mentioned, some studies have reported no significant differences in dental hard tissue microhardness values and other properties after bleaching with 10% and 15% carbamide peroxide.[15-18] The discrepancies in the results of various studies might be attributed to differing study designs, including different dental substrates (human vs bovine teeth); differences in microhardness testing procedures and equipment (eg, Vickers, Knoop); differences in storage conditions of the specimens between bleaching procedures (in solutions without remineralizing properties,

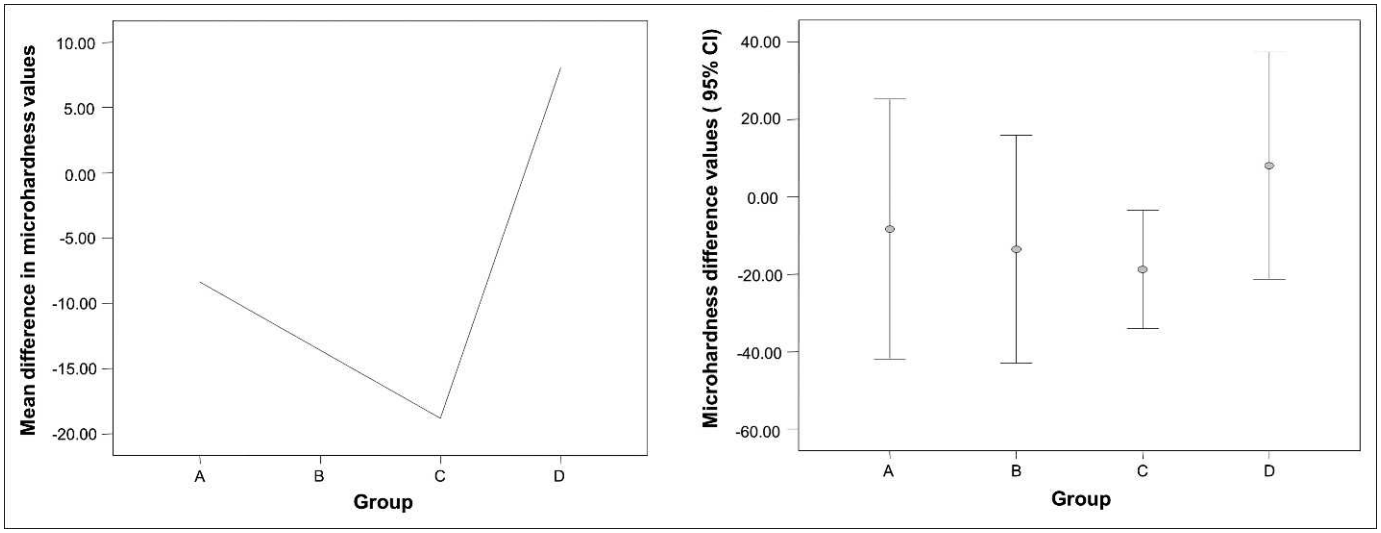

Figure 1. *The linear graph (left) and error bar (right) of differences in microhardness values before and after intervention in the groups under study.*

artificial saliva, or human saliva); fluoride use or lack thereof; the type of the study (*in vitro* or *in vivo*); the time of testing the specimens (immediately after daily bleaching sessions or after completion of the bleaching procedure); concentration and composition of the bleaching agent; and the duration of the study and the duration of each treatment session.[9,10,19]

In studies in which the duration of the bleaching procedure is long, significant decreases in enamel microhardness values have generally been reported.[11] Another factor which contributes to a decrease in microhardness is lack of a remineralization period, including lack of specimen storage in artificial saliva or the short duration of this storage process.[12,18] On the other hand, the effect of different concentrations of carbamide peroxide on enamel microhardness is dependent on the amount of hydrogen peroxide released, its pH level, and the proportion of bleached enamel organic and mineral content.[11] In the present study 15% carbamide peroxide gel (Opalescence PF) was used. This gel contains 3% potassium nitrate and 0.11 wt% (equal to 1100 ppm) fluoride ion. According to the manufacturer, incorporation of fluoride ions and potassium nitrate into the structure of this gel has aimed at reducing the odds of caries and tooth hypersensitivity during bleaching and also at improving enamel health and integrity, including improvements in its microhardness.

Changes in the mineral content of enamel surface are directly related to changes in microhardness. Remineralization increases and demineralization decreases enamel microhardness.[9] Given the fact that in the present study no differences were observed in microhardness values of the groups, it seems the probable enamel demineralization has been compensated by factors involved in remineralization. One of the factors influencing the retention of microhardness in the present study is the use of fluoridated toothpaste in the oral hygiene procedures in all groups. According to some studies, despite the probable destructive role of toothbrushing, fluoride present in the toothpaste can create a balance between remineralization and demineralization on a daily basis after the completion of the bleaching procedure.[9,20] On the other hand, the fluoride ion can prevent demineralization and microhardness decrease by forming a layer of calcium fluoride on the enamel surface.[9,21,22]

Apart from the effect of fluoridated toothpaste, artificial saliva was used in the present study as an environment to store the specimens. Saliva can have a role in remineralization and can change the oral cavity conditions after bleaching in favor of the improvements in tooth structure properties.[6] Potential agents in the saliva which serve as remineralizing agents are calcium and phosphate ions.[11] In the present study, hygiene procedures in the bleached specimens after storing them in artificial saliva for 2 hours resulted in an ascending trend in microhardness, but the change was not significant. It is probable that a longer time is necessary to produce noticeable changes.

According to a review article on the effect of bleaching agents on enamel microhardness, it has been shown that in studies in which the oral cavity conditions have been simulated, including the use of human saliva, fluoride, and fluoridated toothpastes, enamel microhardness has exhibited lower decreases during the postbleaching period compared to other studies.[19]

Another factor in the present study, which probably led to the lack of significant differences between the groups, was the type of test used to evaluate microhardness. Vickers hardness test was used in the present study. The tip of the diamond indenter of Vickers equipment penetrates into the deeper layers of enamel, which are probably not influenced by the bleaching procedure, and the enamel hardness in these layers is probably not comparable to that of the bleached enamel.[9] The pH of the bleaching agent, too, is another influential factor in the bleaching process. In the present study the pH of the bleaching agent was around 6.5. Considering that the critical pH for enamel demineralization is around 5.5,[23] and the pH of the agent in this study was higher than the critical pH, it is probable that this fact has influenced the lack of changes in microhardness in the present study. In a study in which the effect of Opalescence bleaching agent with a pH value of 6.5 was compared with that of Rembrandt having a pH value of 4.9 on enamel microhardness, Opalescence increased and Rembrandt decreased microhardness.[17]

Within the limitations of this study, postponement of daily oral hygiene procedures subsequent to bleaching with 15% carbamide peroxide does not influence enamel microhardness. It is suggested that in future studies other hardness evaluation test methods, such as Knoop, be used.

CONCLUSION

Daily oral hygiene procedures either immediately or 1 or 2 hours after daily bleaching procedures and exposing the specimens to artificial saliva during the

study period produced no significant differences in enamel microhardness values.

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Clinical Technique/Case Report

The Tucker Technique: Conservative Molar Inlays Preserving the Transverse Ridge

TA Hess • CPK Wadhwani

SUMMARY

Conservation of healthy tooth structure should be the aim of any restorative procedure. Two inlays may be an ideal choice for the treatment of maxillary molars to preserve the transverse ridge and maintain structural integrity.

INDICATIONS

When mesial and distal proximal surface restorations are indicated on the maxillary first molar that has an unaffected oblique ridge, separate two-surface cavity preparations are indicated rather than a mesio-occlusodistal preparation, inasmuch as strength of the tooth crown is significantly greater when the oblique ridge is intact.[1,2] Cast gold inlays have long been used to conservatively restore compromised tooth structure. Often the clinician is faced with two areas requiring restoration on a maxillary molar interrupted with an intact oblique ridge of ample stock. Ideally, the dentist would elect to maintain this transverse ridge to minimize the separating forces of occlusion that flex the buccal and lingual halves of the molar from one another. Various combinations of inlays are possible depending on the surfaces involved.

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TECHNIQUE

Tooth #14 (Figures 1 and 2) was observed to have an existing mesial-occlusal composite and separate occlusal composite in the distal portion of the occlusal surface. Although the restorations were still serviceable, the patient was a dental hygienist who understood that all restorations have a limited life span. The hygienist, having been informed of the clinical data available with respect to materials and techniques,[3] requested the removal and replacement of her composites with cast-gold restorations utilizing the Tucker Technique. Occlusion was evaluated, anesthesia administered, and a heavy weight rubber dam (Coltene/Whaledent, Cuyahoga Falls, OH, USA) placed. Typically, all the existing restorative material and any caries would be removed. However, in this case, the operator elected to utilize the existing composites as block-out with the knowledge that very little if any would remain after preparation with the remaining removed prior to cementation.

Initial occlusal preparation was performed using a #57 fissure carbide bur (Midwest/Dentsply International, York, PA, USA) taking care not to overextend to the buccal in the mesial-buccal aspect of the preparation. The depth of the central groove area was reduced to approximately 1.5 mm and the buccal and lingual walls 2.5 mm because of the inclines of the cusps. The #57 carbide was used to create an angulation of approximately 3–5 degrees on each of the occlusal walls, and therefore a preparation taper of 6-10 degrees was produced. A definite buccal dovetail feature was created in the buccal groove to prevent the proximal dislodgment of the casting and

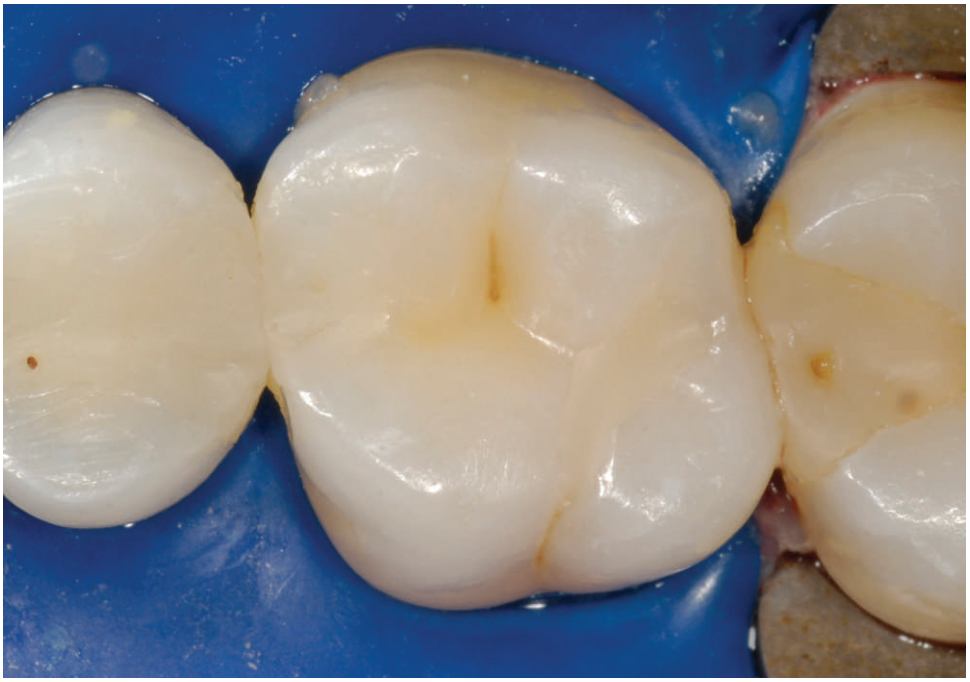

Figure 1. *Pre-operative view of tooth #14.*

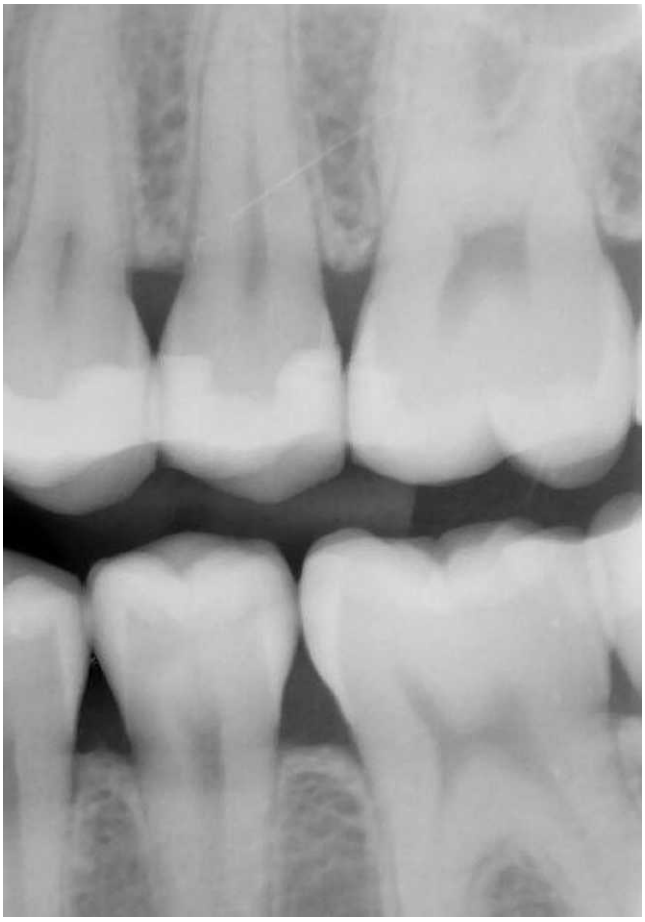

Figure 2. *Pre-operative radiograph of tooth #14.*

negate the need for an internal bevel in the proximal box. The #57 bur was used to establish a proximal box to a depth of 1.5 mm gingival to the pulpal floor or 4 mm from the proximal-occlusal cavosurface. The buccal wall of the proximal box was prepared with a 169L tapered fissure carbide bur held perpendicular to the occlusal surface to establish the mesiobuccal cavosurface and minimize extension to the buccal. The #57 carbide bur was used to prepare the mesiolingual wall of the proximal box and ensure that the axial depth of the proximal box had sufficient depth. A ½ inch sandpaper disc (E.C. Moore, Dearborn, MI, USA) was used to blend the mesiolingual with the lingual wall of the occlusal with slight flare to allow ease of finishing and create taper to be harmonious with the conservative taper of the mesiobuccal wall. The 42S chisel (Suter Dental, Chico, CA, USA) was utilized to remove any friable enamel rods and accentuate the axiogingival line angles and true the gingival floor. The axial wall was smoothed with the 43S chisel. A 0.5-mm external bevel was placed using a H248-009 beveled cylinder carbide bur (Axis, Coppell, TX, USA) and planed with the #233 Tucker gingival margin trimmer (Suter Dental).

The occlusal-lingual groove was prepared using a #7404 bur (Brassler USA, Savannah, GA, USA). This was a relatively simple preparation with a rounded pulpal floor and no line angle with draw created by

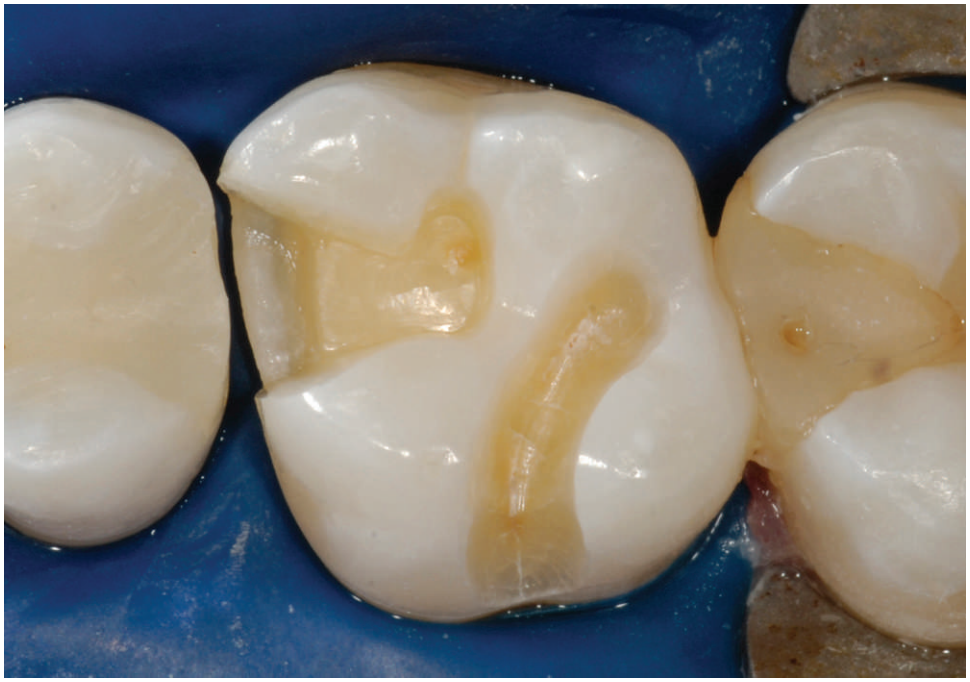

Figure 3. *Completed preparation.*

the shape of the bur. Retention was maximized by keeping the #7404 bur at a consistent perpendicular angle to the occlusal surface as the bur follows the groove. Care was taken to create enough depth in the lingual extension for bulk of gold during casting and accuracy of fit when seating and finishing. Instead of the groove being prepared as a gentle arc from occlusal to lingual, resistance form was enhanced by creating more of an acute angle from the occlusal to lingual aspect (Figure 3). Try-in prior to cementation verified the superb retention and resistance of this preparation design. It was also noted that at the

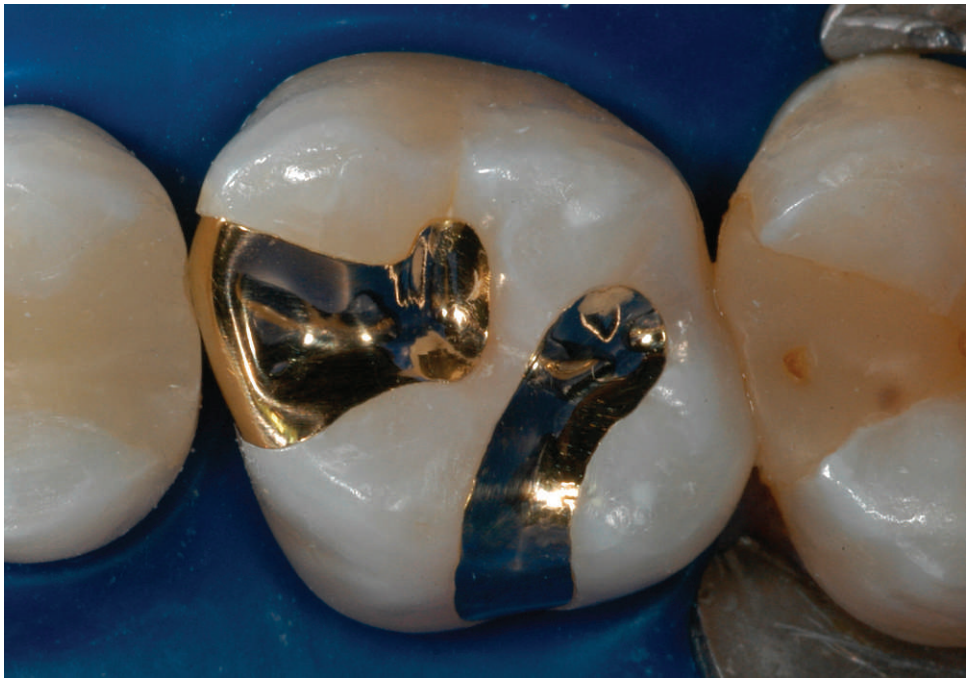

Figure 4. *Initial seating of the restoration showing the casting slightly overfinished.*

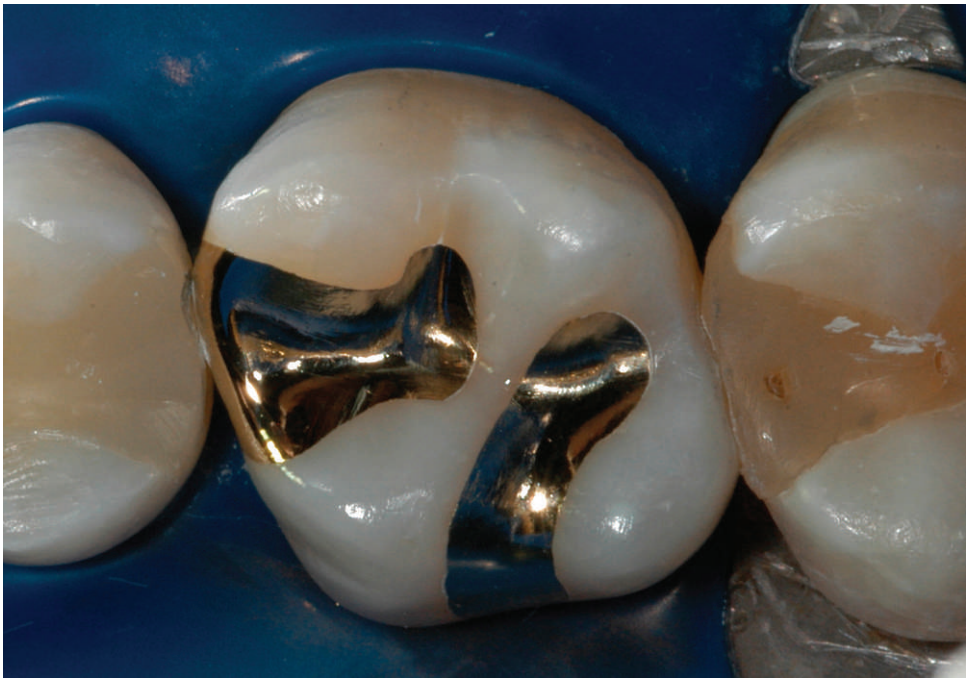

Figure 5. *Post-operative occlusal view.*

occlusal mesiobuccal cavosurface, the casting was slightly over finished and would need to be addressed with finishing (Figure 4).

Seating involved anesthesia, removal of the provisional, and placement of a heavy weight rubber dam. Castings were tried in together to verify fit and proximal contact. The castings were seated one at a time using separate mixes of zinc phosphate cement (Fleck's Cement, Myerstown, PA, USA). The zinc phosphate was slaked with a small amount of powder in the liquid until the liquid appeared clear

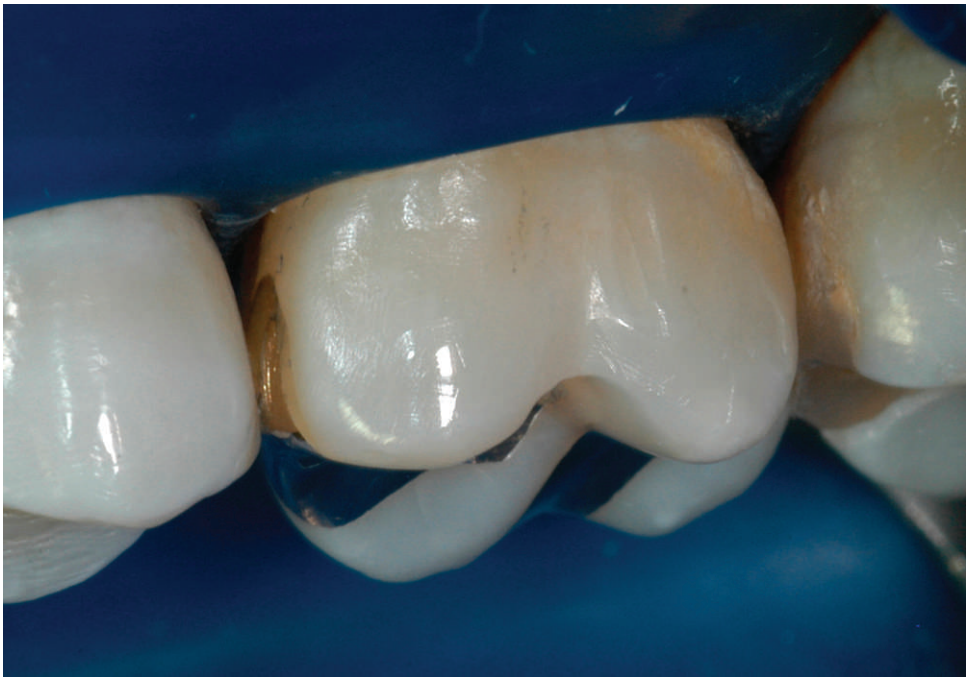

Figure 6. *Post-operative buccal view.*

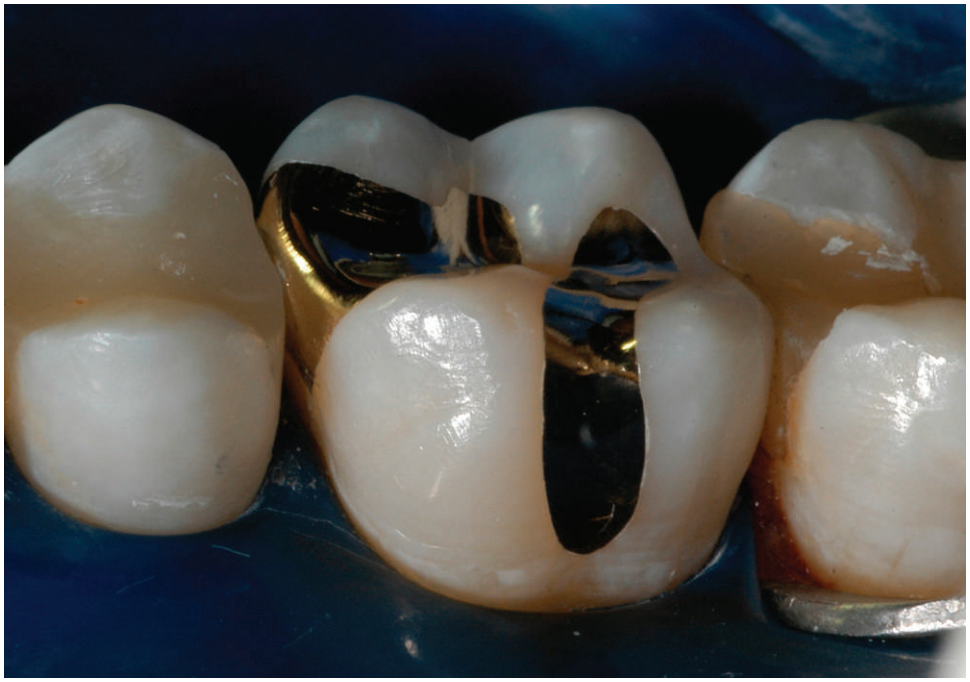

Figure 7. *Post-operative lingual view.*

and then mixed according to the manufacturer's directions. Cement was applied to the castings, and an orange wood stick was used to seat the castings along with light malleting. A shortened orange wood stick was then used between the castings and lower molar until the hydraulic pressure of cementation had dissipated. A series of sandpaper disks (medium garnet, fine sand, and fine cuttle; E.C. Moore), strips, and polishing powders were used to refine the tooth to gold interface. Often the operator will find that only the fine sand and fine cuttle discs are necessary and that the medium garnet may introduce unnecessary scratches in the gold that will require additional finishing time. In this case, no garnet discs were utilized. Wet #4 laboratory pumice (Kerr Corp, Romulus, MI, USA) was used next with a ribbed prophy cup (Young Dental, Earth City, MO, USA). A light touch rotating from casting to tooth was employed to avoid the uneven removal of tooth and gold if the pumice was used too aggressively or for too long. Next, wet 15-micron aluminum oxide powder (Micro Abrasives Corp, Westfield, MA, USA) with a new ribbed prophy cup was used. Important to note is that in between polishing steps, a thorough rinse and dry of the area should be performed to prevent incorporating scratches late in the sequence. Final polishing was performed dry with 1-micron aluminum oxide powder (Micro Abrasives Corp) and again a new ribbed prophy cup. No finishing strips were deemed necessary in this case (Figures 5 through 7).

Once the rubber dam was removed, the occlusion was checked and the patient released.

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Treatment of Invasive Cervical Resorption With Sandwich Technique Using Mineral Trioxide Aggregate: A Case Report

L Kqiku • KA Ebeleseder • K Glockner

Clinical Relevance

MTA combined with glass ionomer cement and composite resin in a "sandwich technique" showed a favourable clinical outcome for treatment of invasive cervical resorption lesions.

SUMMARY

This article presents two cases of large invasive cervical resorption (ICR) with maintenance of pulp vitality after treatment with mineral trioxide aggregate (MTA) in a sandwich technique.

Invasive cervical resorption is a relatively uncommon but aggressive form of external resorption, primarily caused by dental trauma or injury of the cervical periodontal attachment. The resorptive process does not penetrate into the root canal, and the pulp is not involved in the first phase of the resorption. This feature differentiates external resorption from internal resorption. In most cases, invasive cervical resorption is found during routine radiographic or clinical examination. Different materials have been proposed for the treatment of external cervical resorption. Therapy can be effective when it 1) removes the etiological factors and 2) interrupts the progressive resorption mechanism.

The key learning points of this article are the following: treatment strategy to arrest the cervical resorption process and to prevent further resorption without changing pulpal vitality and successful seal of invasive cervical resorption defect using MTA with a sandwich technique.

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INTRODUCTION

Definition of ICR—External root resorption is a progressive and destructive loss of hard dental tissue, initiated by a demineralized or denuded area of the root surface.[1] One of the frequent types of external root resorption is invasive cervical resorption (ICR), described in detail by Heithersay[2]: "*invasive cervical resorption* is a clinical term used to describe a relatively uncommon, insidious and aggressive form of external tooth resorption, which may occur in any tooth in the permanent dentition. The etiologic factors include: traumatic injuries, orthodontic treatment, internal bleaching, periodontal treatment, restorative treatment and idiopathic."[1-3]

Diagnosis of ICR—The diagnosis of ICR is usually achieved by radiographic images or clinical examination. Most ICRs are painless; the clinical features vary from a small root defect in the cervical region to a pink coronal discoloration or enamel lesion. The resorption is associated with inflammation of the periodontal and gingival tissues but primarily does not have any pulpal involvement because of the protective qualities of the predentin layer.[3,4]

Histopathologically, the lesions contain fibrovascular tissue with resorbing clastic cells adjacent to the dentin surface.[5] Bacteria migrating from the gingival sulcus into the dentin tubules are believed to be the resorption trigger.[6]

Therapy of ICR—Three goals have to be achieved to arrest the resorption process:

1. disinfection of the defect to eliminate the original bacterial stimulus,
2. complete seal of the defect to avoid bacterial repopulation, and
3. epithelial attachment to the filling material to avoid contact between the cervical periodontal ligament and bacteria from the gingival sulcus.

Currently, no single material exists that ideally fulfills these criteria. Mineral trioxide aggregate (MTA), glass ionomer, and resin composite have been proposed as resorptive lesion repair materials.[3,7] MTA has favorable chemical and physical properties, sealing ability, antibacterial activity, and biocompatibility and creates an ideal environment for hard-tissue healing.[8,9]

Clinically, successful treatment of ICR depends on the extent of the resorptive process, and the most preferential classification system for ICR used universally was introduced by Heithersay in 1999.[2] The four classifications are as follows.

- Class 1: a small invasive resorptive lesion near the cervical area with shallow penetration into dentin
- Class 2: a well-defined invasive resorptive lesion that has penetrated close to the coronal pulp chamber but shows little or no extension into the radicular dentin
- Class 3: a deeper invasion of dentin by resorbing tissue, involving the coronal third of the root
- Class 4: a large invasive resorptive process that has extended beyond the coronal third of the root canal.

It is important that most ICRs not be treated as a disease that requires conventional endodontic therapy since treatment can be delivered without sacrificing pulpal vitality.[10]

To achieve the three treatment goals, it is further necessary to expose the resorption lacuna orthodontically or surgically and to remove the main bulk of the granulation tissue. As the latter is just a reaction to the bacterial stimulus in the defect, complete removal as proposed in earlier literature[1] is not the main approach. Rather, it is far more important to achieve a definitive seal of the affected dentinal tubules.

This report presents the management of two clinical cases of ICR using a combination of MTA and other materials that demonstrate the outcome over a longer observation period.

CASE REPORT

Case 1

Clinical Features—A 31-year-old female patient presented to the Division of Preventive and Operative Dentistry, Endodontics, Pedodontics, and Minimally Invasive Dentistry, Medical University of Graz, in March 2006 with a gingival swelling and pink spot localized in the labial cervical area of the maxillary left central incisor (Figure 1). The medical history of the patient was not relevant for ICR: no history of orthodontic treatment, dental trauma, or bleaching. The patient reported that the gingival swelling and discoloration had increased in size during the past weeks. Intraoral examination showed that the tooth was not sensitive to percussion and responded positively to a thermal sensitivity test. In the cervical region of the labial surface, a pink discoloration under a remaining thin and fragile enamel layer was visible. The resorptive lesion advanced into the periodontal ligament re-

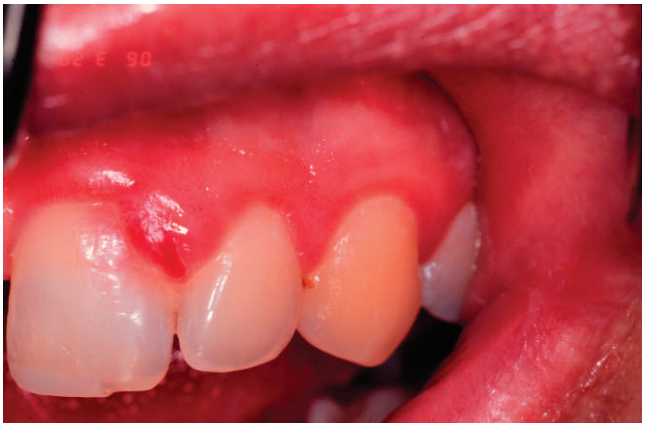

Figure 1. *Initial presentation of the case. Intraoral appearance of the gingival swelling and pink discoloration in the labial surface of tooth.*

gion, and the adjacent papilla showed an erythematous swelling. No caries or restorations were detected. A central diastema was noted. After a clinical examination, a radiograph was obtained using a digital radiographic system (Sirona Dental Systems GmbH, Bensheim, Germany). The radiographic images showed an irregular radiolucent area localized distal in the cervical third of the tooth (Figure 2). The pulp chamber and canal were not involved, and the case was classified a class 2 grade of ICR.

Management—After explaining the therapy plan to the patient, surgical treatment was performed under local infiltration anesthesia (Ultracain D-S, Sanofi-Aventis, Frankfurt, Germany). According to the considerations of Eskici,[11] a minimally invasive mucoperiosteal flap was raised. It combined a vertical incision from the mesial surface of the maxillary left central incisor with a continuing sulcular labial incision to the distal surface of the maxillary left lateral incisor (Figure 3). After the resorption lacuna had been exposed, the granulomatous tissue was removed *in toto* from the resorptive defect using an excavator instrument (Figure 4). The resorption defect was disinfected with 0.1% chlorhexidine solution and subsequently filled using a sandwich technique as follows: the first layer consisted of white MTA (Pro Root, Dentsply, Konstanz, Germany) mixed with 0.1% chlorhexidine.[12-14] This layer was not extended to the margins of the cavity (Figure 5a). The second layer consisted of glass ionomer cement (Fuji IX, Fuji, Tokyo, Japan), which filled out the whole cavity including the margins (Figure 5b). The third layer consisted of light-cured composite (bonding: Excite; composite: Artemis Enamel A2, both by Vivadent, Schaan, Liechtenstein) bonded to the intraenamel margin of the lesion (Figure 5c). After the materials had been placed, the flap was repositioned and sutured with two nonabsorbable sutures (Aesculap AG & CO. KG, Tuttlingen, Germany; Figure 6). The patient was prescribed oral cephalosporin (Ospexin 1000 mg, Sandoz GmbH, Kundl, Austria) for four days and a 0.1% chlorhexidine mouthwash (Chlorhexamed, GlaxoSmithKline Pharma GmbH, Vienna, Austria) for two weeks. At the first control visit one week after treatment, the patient was asymptomatic and the sutures were removed. No more gingival swelling was seen (Figure 7). At the six-month recall, the tooth showed no symptoms and responded normally to sensitivity tests. At the 1½-year follow-up, the radiographic images showed no pathologic changes and good adaption of the MTA to the root anatomy. The patient was free of clinical symptoms, and the pulp sensitivity tests showed a normal response

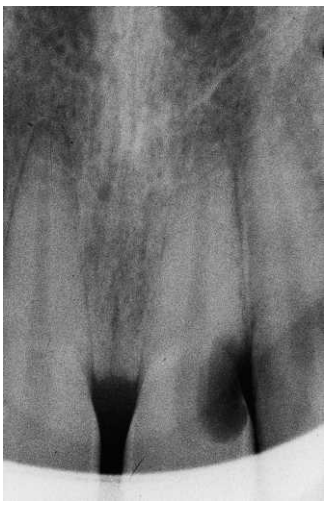

Figure 2. *Radiographic examination: irregular radiolucent area localized distal in the cervical third of maxillary left central incisor tooth.*

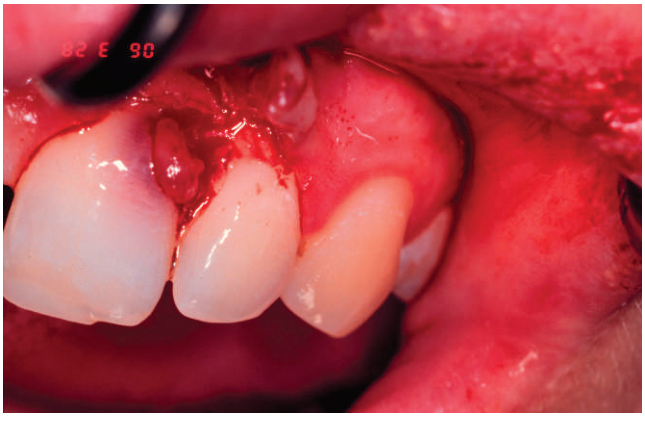

Figure 3. *Clinical/intrasurgical view after flap preparation.*

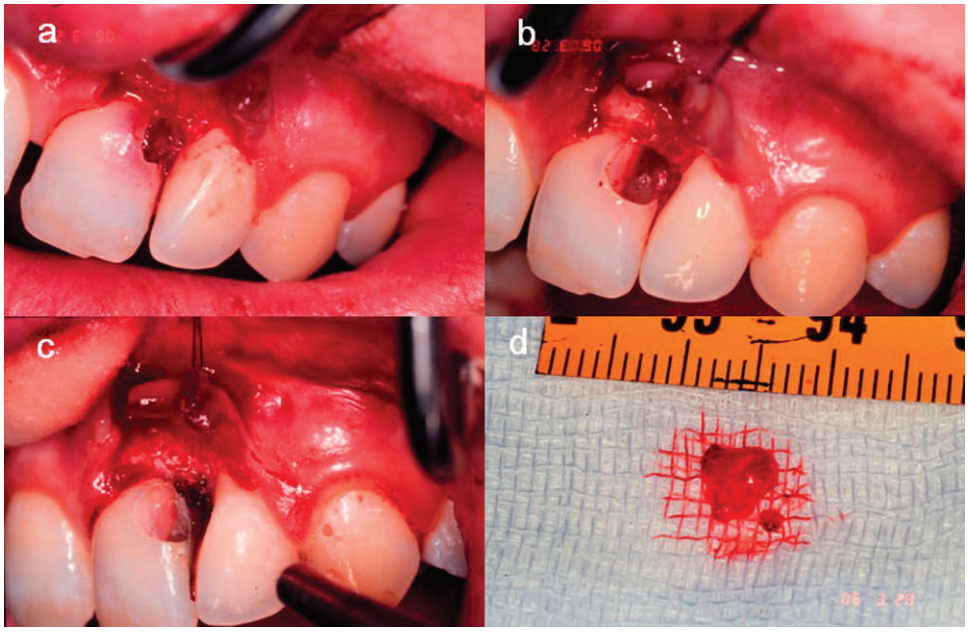

Figure 4. *Exposition of resorption defect. (a, b): Resorption defect after removal of granulation tissue. Note that the resorption defect is surrounding the intact pulp chamber. (c): Clinical view after complete cleaning of resorption defect. (d): Granulation tissue in toto.*

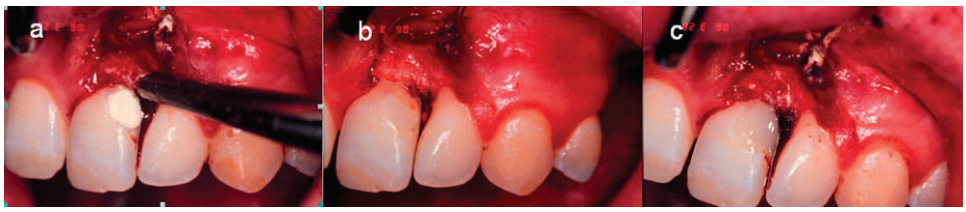

Figure 5. *The resorption defect filled with sandwich technique. (a): Layer of mineral trioxide aggregate. (b): Layer of glass ionomer cement. (c): Layer of light-cured composite.*

(Figures 8 and 9). At the four-year follow-up, the pulp responded positively to sensibility tests, no pink discoloration or caries were detected, and the radiographic examination showed no pathologic changes of the tooth and surrounding tissues (Figures 10 and 11).

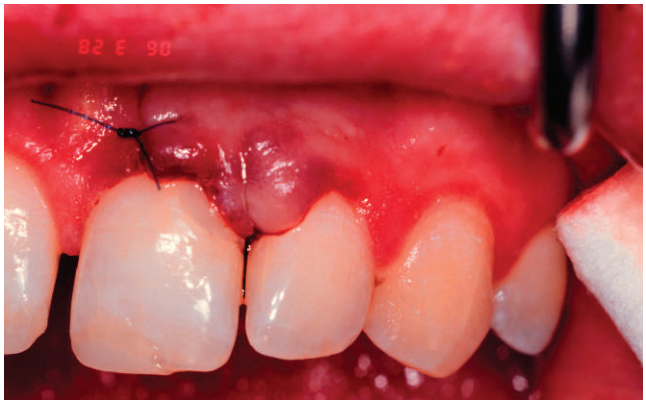

Figure 6. *Flap repositioned and sutured. Note the hyperplastic papilla due to the preexisting inflammation.*

Case 2

Medical History and Clinical Features—A 16-year-old male patient presented to the Division of Preventive and Operative Dentistry, Endodontics, Pedodontics, and Minimally Invasive Dentistry, Medical University of Graz, in January 1998 with dental trauma caused by a skateboard accident. The Periotest device revealed that 11 and 21 were mobile (subluxation), and the patient had orthodontic treatment at this time. In the initial treatment, teeth were repositioned and splinted with passive wire and composite. Systemic antibiotic coverage was achieved by oral penicillin (Augmentin 625 mg, GlaxoSmithKline Pharma GmbH) for four days. Local disinfection during the first two weeks was achieved by a 0.1% chlorhexidine mouthwash three times a day. Follow-up after four weeks consisted of

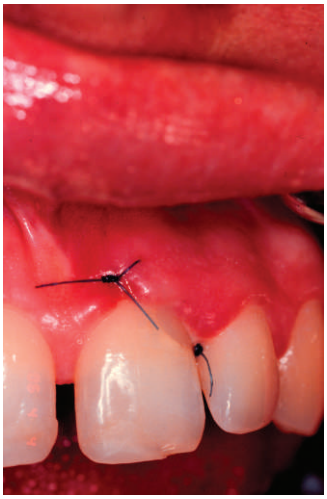

Figure 7. *Gingival healing after one week. The papilla appears to be of normal size and color.*

a clinical and radiographic evaluation. Pulp vitality testing with carbon dioxide snow was negative for both subluxated teeth. No pathologic changes were seen in radiographic images. At the six-month recall, the teeth showed no clinical symptoms and responded negatively to vitality tests. One year after initial treatment, the patient was still free of symptoms and both pulp vitality tests were positive. No further appointment was made. Ten years later, the patient was sent to our division by the local dentist because of a defect localized in the cervical region of the labial and palatal surface in the maxillary right central incisor. The radiographic images showed an irregular radiolucent area in the cervical region of this tooth (Figure 12). Cone-beam computed tomography (CBTC) was performed to confirm the diagnosis of a class 3 lesion of ICR according to Heithersay[2] on the palatal aspect of the root (Figure 13). It was composed of an external destructive part with access

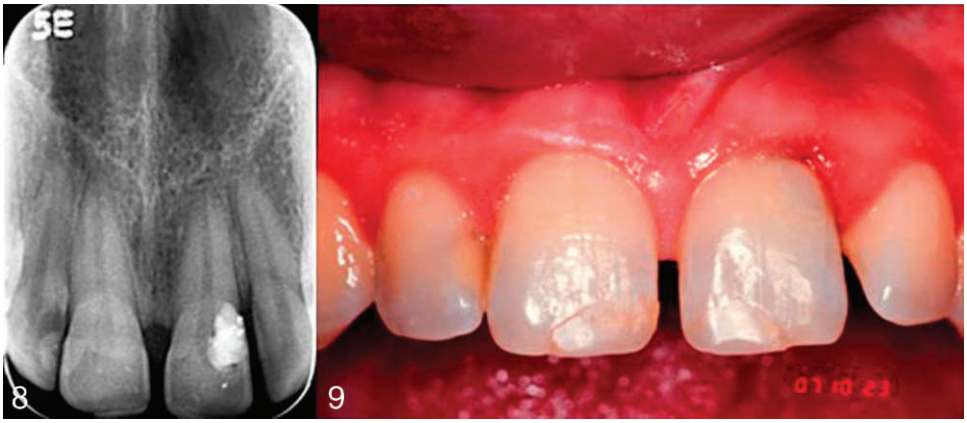

Figure 8. *Radiographic and clinical examination after 1½-year follow-up.*
Figure 9. *Radiographic and clinical examination after 1½-year follow-up.*

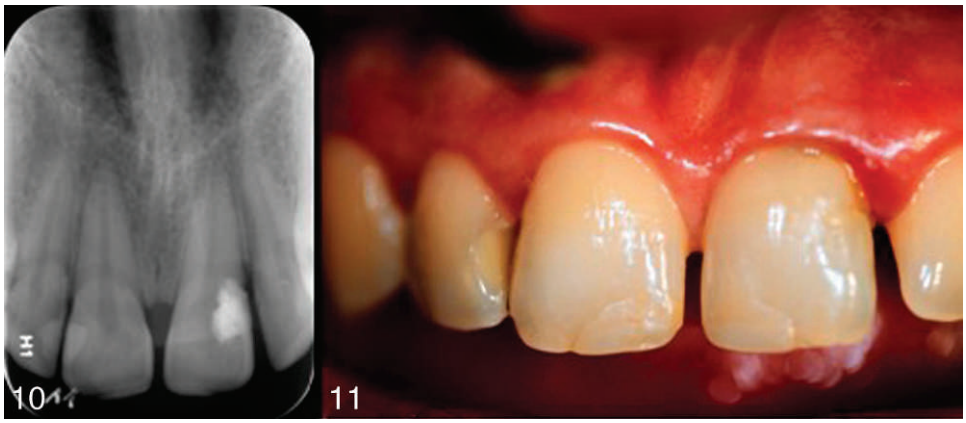

Figure 10. *Radiographic and clinical examination after four-year follow-up.*
Figure 11. *Radiographic and clinical examination after four-year follow-up.*

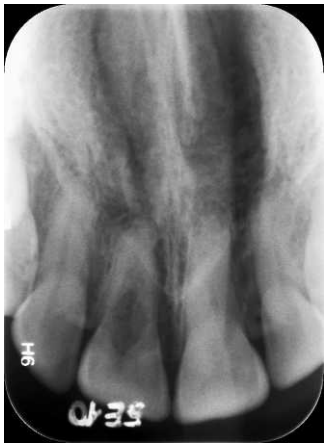

Figure 12. *Radiographic result after 11½ years, irregular radiolucent area localized in maxillary right central incisor tooth.*

to the sulcular region and a corresponding internal part with bone formation. It was decided to treat only the external part as it seemed to be the primary source of infection.

Management

Surgical treatment under local infiltration anesthesia was performed according to the method described in case 1. The patient was prescribed oral penicillin (Augmentin 625 mg, GlaxoSmithKline Pharma GmbH) for one week and a 0.1% chlorhexidine mouthwash for two weeks. At the first control visit one week after treatment, the patient was symptomless and the sutures were removed. The radiographic examination showed good adaption of the MTA to the root anatomy (Figure 14). Six months after surgical treatment, the tooth showed no further pathological symptoms. The radiographic images showed no further pathologic changes of tooth and surrounding tissues (Figure 15). Further control was scheduled after another six months.

DISCUSSION

The exact etiology of ICR is still unknown. It is supposed that ICR requires two phases: injury and stimulation.[1,15] Injury can be caused by mechanical

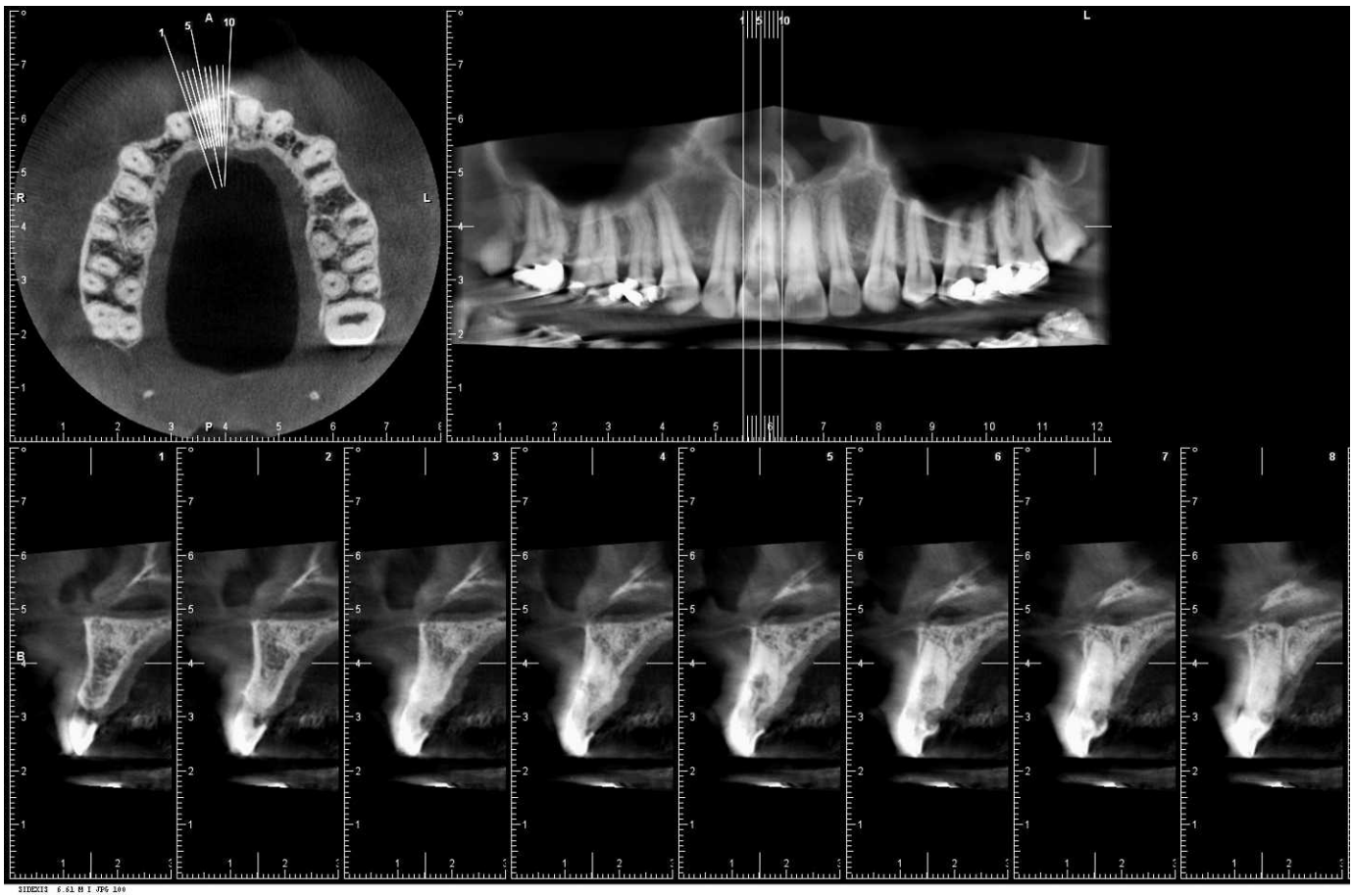

Figure 13. *Cone-beam computed tomography after 11½ years. Note the two components of the resorptive process especially in the sixth saggital section: a destructive part in the cervical-peripheral region and an underlying reparative part (bone formation) extending to the apical-central region.*

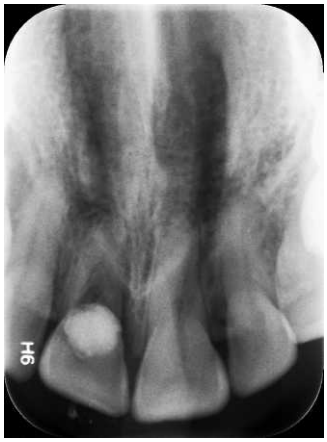

Figure 14. *Radiographic examinations immediately after surgical treatment.*

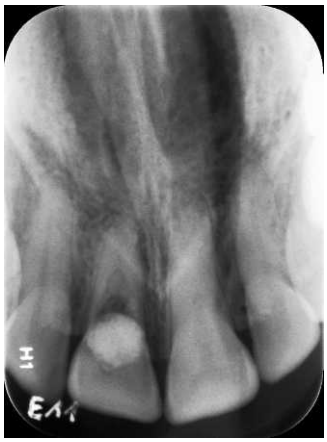

Figure 15. *Six-month follow-up.*

(orthodontics, trauma) or chemical stress (internal bleaching). Gold and Hasselgren[16] suggested that there are three environmental factors that in general may contribute to root resorption: absence of protection for the root surface, presence of vascular connective tissue, and an inflammatory stimulus. The origin of stimulation factors is different for each type of root resorption, and when these stimulating factors are identified, it may be possible to reverse the process by removing the etiological factor.[6] External resorptions associated with inflammation in the marginal tissue can present a difficult clinical situation.[10] When the infection originates from the periodontal sulcus and stimulates the resorption process, removal of the granulation tissue from the resorption lacuna and reliable sealing are necessary for repair since the elimination of micro-organisms in the periodontal sulcus is unlikely.[6] Case 1 demonstrates the outcome of sealing a resorptive defect with advanced repair materials. Although the latter showed an invasive gingival ingrowth, one week after therapy the gingiva appeared to be healed, and neither showed an inflammatory response nor recurrent aggressive ingrowth, which indicates that no further inflammatory stimulus had been present.

In previously published investigations, MTA was successfully used as a barrier between the pulp canal space and the periodontal tissue in root perforations in dogs and humans.[17-19] Hiremath and others[20] used calcium hydroxide and glass ionomer cement for the treatment of ICR and found that the tooth was without symptoms for only six months, but at the follow-up after six months, healing was incomplete. The use of MTA for treatment of ICR showed favorable healing compared with composite.[21] In this report, a case is described in which an ICR defect was sealed with dentin adhesive and composite and treatment failure was noted after six months. After retreatment with MTA, the tooth was asymptomatic at the three- and nine-month follow-up with continued pulp vitality. It can now be reported that this favorable outcome has continued for four years.

Orthodontic treatment, dental trauma, and bleaching were the most common potential predisposing factors for ICR.[22] External root resorption may occur after injury of precementum by dental trauma or ischemic necrosis of cementoblasts in the pressure zone during orthodontic treatment. Consequently, the damaged area of the root surface is colonized by hard-tissue resorbing cells. With orthodontic pressure resorption, the correct treatment is the removal of the source of the pressure.[6] In case 2, a tooth undergoing orthodontic movement was subjected to periodontal trauma, which means that the patient exhibited two of three predisposing etiologic factors for ICR. We assume that the preexisting cervical resorption lacunae (caused by the orthodontic pressure) were initiated by the traumatic event. As a result, resorption advanced although the orthodontic pressure was removed by splinting the tooth with a passive wire. Thus, the sulcular region may have been compromised with subsequent ongoing bacterial invasion that acted as an ICR stimulus.

The topical application of a 90% solution of trichloracetic acid, curettage, and restoration of resorptive lesion with glass ionomer cement has been recommended by Heithersay.[23] In the present cases, trichloracetic acid was not used because the granulomatous tissue was only a clinical symptom but not the etiological factor. Instead, MTA with its high pH was chosen as the disinfectant on one hand

and as filling material on the other hand because of its biocompatibility and its favorable potential as a pulp-capping material during vital pulp therapy.[24] It forms an apatite-like layer on its surface when it comes into contact with physiologic fluids.[25] In both cases, MTA was used to arrest cervical resorption, to prevent further resorption, and to preserve pulp vitality also in a class 3 defect, in which, according to Heithersay.[2] the pulp should be sacrificed. In case 2, the resorption was arrested although the filling did not reach the reparative portion of the resorption defect.

In previous case reports, MTA or glass ionomer cement were successfully used for treatment of ICR but not combined with other materials.[20,26-28]

CONCLUSIONS

In summary, MTA combined with glass ionomer cement and composite in a sandwich technique has not previously been reported over a long-term observation period without sacrificing pulp vitality. This report demonstrates a favorable clinical outcome for surgically accessible ICR lesions when MTA is used as a repair material in combination with a glass ionomer cement and composite resin.

(Accepted 19 May 2011)

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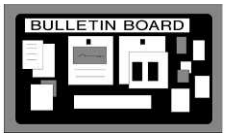

Erratum

In Operative Dentistry 36(6) 572–580, *The Effect of Bleaching Systems on the Gingiva and the Levels of IL-1β and IL-10 in Gingival Crevicular Fluid* an author name was omitted from the list of authors. Dr. Ozlem Ozer Yucel should be listed as 4th co-author with the following affiliation address: Özlem Özer Yücel, DDS, PhD., Department of Oral Pathology, Faculty of Dentistry, Gazi University, Ankara, Turkey. The Authors and Operative Dentistry apologize for this omission.

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