# Effect of Postoperative Peroxide Bleaching on the Marginal Seal of Composite Restorations Bonded with Self-etch Adhesives

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## **Clinical Relevance**

If postoperative bleaching is expected, composite restorations should be bonded preferably with well-proven etch-and-rinse adhesive systems.

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## SUMMARY

The aim of this study was to determine the effect of peroxide bleaching on the marginal seal of composite restorations bonded with several adhesive systems. Combined cylindrical Class V cavities located half in enamel and half in dentin were prepared on the buccal and lingual surfaces of human molars. The cavities were bonded with the self-etch adhesives Clearfil SE-Bond (CLF), Adper Prompt (ADP), and iBond (IBO) and an etch-and-rinse adhesive Gluma Comfort Bond (GLU) and restored with a microhybrid composite Charisma. Experimental groups were treated 25 times for eight hours per day with a peroxide bleaching gel Opalescence PF 20, while the control groups were stored in distilled water for two months and then subjected to a microleakage test using a dye penetration method. Scanning electron microscopy was used to investigate the etching and penetration abilities of the adhesives and morphology of debonded restoration-enamel interfaces after the microleakage tests. Statistical analyses were performed using nonparametric Kruskal-Wallis, Mann-Whitney, and Wilcoxon tests at p=0.05. The microleakage of all GLU groups was low and not significantly affected by peroxide bleaching. Low microleakage was recorded for CLF control groups, but after bleaching, a small but significant increase in microleakage at the enamel margin indicated its sensitivity to peroxide bleaching. For ADP and IBO control groups, the microleakage at the enamel margins was significantly higher than for GLU and CLF and exceeded that at the dentin margins. Bleaching did not induce any significant changes in the microleakage. Electron microscopy analysis indicated that in our experimental setup, decreased adhesion and mechanical resistance of the ADP- and IBO-enamel interfaces could be more important than the chemical degradation effects induced by the peroxide bleaching gel.

## INTRODUCTION

Tooth discolorations caused by exogenous factors, such as smoking, absorption of pigments from foods and drinks, or frequent mouth washing with antimicrobial rinses, can be removed by peroxide bleaching. Some studies, however, show that reactive oxygen species released from peroxide products may attack not only the staining moieties captured in the enamel structure but also hard tooth tissues and reconstruction materials. In contrast, little attention has been paid to the effect of postoperative peroxide bleaching on the durability of the adhesive interface between composite restorations and tooth tissues. The results obtained by bond strength<sup>1-3</sup> and microleakage<sup>4-10</sup> measurements, however, are contradictory and do not clearly illustrate this effect.

Adhesion between the tooth tissues and restoration materials has been tested with a wide variety of experimental approaches. In bond strength measurements, the composite build-ups are often made on flattened surfaces of teeth. The configuration factor (C-factor), defined as the ratio of the bonded to unbonded surface, and thus the polymerization shrinkage stress effects, are low in such restorations.<sup>11</sup> Under these conditions, the adhesive interface is primarily challenged by the ambient environment. With microleakage tests, however, the C-factor is significantly higher, and thus the adhesive interface is stressed not only by the environment but also by shrinkage stress, which can accelerate degradation of the interface.

In a previous study,<sup>3</sup> the resistance of an adhesive interface against peroxide bleaching degradation was investigated by the shear bond strength tests at a C-factor of approximately 0.30 using four selfetch and etch-and-rinse adhesive systems that represented typical currently used adhesives associated with different working protocols. A decrease in the bond strength indicated degradation of the adhesive interface created with the one-step selfetch adhesives Adper Prompt and iBond.

In the present study, we focused on evaluating the resistance of an adhesive interface created with the same adhesive systems used in the previous bond strength test, but using the microleakage test instead. It was assumed that degradation of the adhesive interface due to peroxide bleaching would be more pronounced under a higher shrinkage stress than that observed with a bond strength measurement. The null hypothesis was that the marginal seal of the composite restorations would not be impaired by peroxide bleaching.

#### MATERIALS AND METHODS

The two-bottle, two-step Clearfil SE Bond (CLF; Kurarav Medical Inc. Okavama, Japan): the twobottle, one-step Adper Prompt (ADP; 3M ESPE AG, Seefeld, Germany); and the one-bottle, one-step iBond (IBO; Heraeus Kulzer GmbH, Hanau, Germany) self-etch adhesives were used. These adhesives were compared with that associated with the one-bottle, two-step etch-and-rinse Gluma Comfort Bond, combined with an etching gel, Gluma Etch 20 Gel (GLU; Heraeus Kulzer GmbH). All restorations were made with a microhybrid composite Charisma (shade A2, Heraeus Kulzer GmbH) to avoid any unwanted effects of varying composites. The home bleaching peroxide gel Opalescence PF 20% (Ultradent Products Inc, South Jordan, UT, USA), containing 20% carbamide peroxide, was used in the study. All materials came from the same production batches. Their composition and application protocols are summarized in Table 1. After delivery, the restorative materials were stored at 10°C to slow down hydrolysis of self-etch adhesives.<sup>12</sup> The bleaching gel was stored at 4°C and was used in the first quarter of its shelf life to minimize spontaneous peroxide gel decomposition during the testing period.

Forty human noncarious third molars extracted for orthodontic reasons were used in the study. After extraction, the teeth were cleaned and stored at a

Table 1: Materials Used and Their Working Protocols								
Material	Manufacturer	Chemical Composition	Application <sup>a</sup>					
Adhesive system								
Gluma Comfort Bond (GLU)	Heraeus Kulzer GmbH, Hanau Germany	Etchant: Gluma Etch 20 Gel (phosphoric acid 20%)	e (20 s), r, d (1-2 s), 3× b (15 s), w (15 s), d, c (20 s)					
		Bond: HEMA, 4-META, polyacid, ethanol, photoinitiators, polyacrylic acids						
Clearfil SE Bond (CLF)	Kuraray Medical Inc, Okayama, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, camphorquinone, <i>N,N</i> -diethanol- <i>p</i> -toluidine, water	p (20 s), d, b, d, c (10 s)					
		Bond: MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, <i>N,N</i> -diethanol- <i>p</i> -toluidine, silanated colloidal silica	-					
Adper Prompt (ADP)	3M ESPE AG, Seefeld, Germany	A liquid: methacrylated phosphoric esters, bis-GMA, initiators based on camphorquinone, stabilizers	m (A+B), a (15 s), d, a, d, c (10 s)					
		B liquid: water, HEMA, polyalkenoic acid, stabilizers	-					
iBond (Gluma inside) (IBO)	Heraeus Kulzer GmbH	4-META, UDMA, glutaraldehyde, acetone, water, photoinitiators, stabilizers	3× a, w (30 s), d, c (20 s)					
Composite material								
Charisma	Heraeus Kulzer, GmbH	bis-GMA, TEGDMA, UDMA, barium fluoride glass, silicon dioxide, initiators, stabilizers, pigments	c (20 s)					
Bleaching gel								
Opalescence PF 20	Ultradent Products Inc, South Jordan, UT, USA	Carbamide peroxide 20 weight %, sodium fluoride 0.25 weight % potassium nitrate	25× 8 h					
Abbreviations: bis-GMA, bisphenol methacryloyloxydecyl dihydrogen p <sup>a</sup> Application protocol with a recomm w, waiting.	A diglycidyl methacrylate; 4-ME hosphate; TEGDMA, triethylene mended time (in seconds): a, ap	TA, 4-methacryloxyethyl trimellitic anhydride; HEMA, 2-hydroxyethj e glycol dimethacrylate; UDMA, urethane dimethacrylate. plication; b, bonding; c, light curing; d, drying/spreading; e, etching; r	/l methacrylate; MDP, 10- n, mixing; p, priming; r, rinsing;					

temperature of approximately 4°C in 0.5 % chloramine-T solution for one week. They were then stored in distilled water for up to six months at this same temperature.<sup>13</sup> On the buccal and lingual tooth surfaces, 80 standardized cylindrical Class V cavities, with diameters of approximately 3 mm, depths of approximately 1.5-2.0 mm, and a C-factor of 3-4, were cut by one operator using a spherical coarse diamond bur (100  $\mu$ m, Hager & Meisinger, Neuss, Germany) under water cooling. The cavities were finished with a cylindrical tungsten carbide bur (Acurata G+K Mahnhardt Dental, Thurmansbang Germany) in a high-speed handpiece and were cooled with an air-water spray. The burs were replaced after preparation of 10 cavities. One half of the cavity was located in enamel and the other half in dentin, with a cavosurface angle of approximately 90°. No beveling was made at the cavity margins. Ten teeth, divided into experimental and control groups of 10 cavities, were randomly chosen for each adhesive system. Both the adhesive systems and the composite material were applied by the same operator, strictly following the manufacturer's recommendations. The cavities were restored incrementally, the first increment placed occlusally up to approximately half of the cavity depth and the second increment placed gingivally in contact with the first increment. The last increment restored the anatomical shape of the tooth. Each increment was polymerized for 20 seconds using an Elipar TriLight halogen lamp (3M ESPE AG) with a power intensity of 800-850 mW/cm<sup>2</sup> that was checked periodically using a calibrated handheld radiometer EVT 460 (Preciosa, Jablonec and Nisou, Czech Republic). After polymerization the restorations were slightly polished with silica-carbide sandpaper P1200 with a particle size of 15  $\mu$ m (Buehler Ltd, Lake Bluff, IL, USA) under water cooling with an Ecomet III polishing machine (Buehler Ltd) to prepare defined restoration margins without overlaps.

The experimental groups were subjected to 25 bleaching cycles. Each cycle included application of approximately 0.1 g of bleaching gel on the restoration margin. The teeth with the gel were wrapped in moisture-resistant Parafilm foil (Parafilm M, Alcan Packaging, Chicago, IL, USA) and stored in a 100% relative humidity environment. After eight hours, the peroxide gel was carefully removed from the tooth surface under running water using a soft toothbrush, and the teeth were stored in distilled water until the next application. To prevent microbial growth, each tooth was stored in 20 mL of water with approximately 100 ppm of sodium azide per 1 L of distilled water. The control teeth were stored in similarly treated distilled water (replaced every four to five days) for the two months during which the bleaching tests took place in the experimental groups. All of the exposures were performed at 37°C. After the bleaching program was finished, the apices and the surfaces of the teeth were carefully sealed with two layers of nail varnish and one layer of sticky casting wax, except for a 1-mm zone around the restoration margin. The teeth were immersed for 24 hours in a 2% methylene blue aqueous solution at 23°C and were then rinsed, dried, and fixed in polyethylene rings with the selfcuring methylmethacrylate resin Spofacryl (Spofa-Dental, Jičín, Czech Republic). The restorations were cut into three parts, in the occlusal-cervical direction, using an Isomet low-speed saw equipped with a water-cooled diamond wafering blade (Buehler Ltd).

## **Microleakage Evaluation**

The depth of dye penetration was evaluated using a Nikon SMZ 2T optical stereomicroscope with  $10-20\times$  magnification. A 5° microleakage score was implemented for enamel and dentin, with scoring criteria as follows: 0 = no dye penetration; 0.5 = penetration up to one-fourth of the cavity depth; 1 = penetration up to one-half of the cavity depth, typically equal to

the whole depth of the enamel layer on the enamel margin; 2 = penetration over one-half of the cavity depth to its floor; and 3 = penetration including the cavity floor. Evaluation was performed by three calibrated subjects, and the consensual value was considered in the case of score variances. For each restoration, the highest scores on the enamel and dentin margins were used for statistical evaluation.<sup>14</sup> With the exception of the ADP and IBO experimental groups 10 scores were obtained for the remaining groups. As a result of dye penetration through the apices one restoration had to be eliminated in each of these two groups. The Kruskal-Wallis analysis of variance tests by ranks, followed by multiple comparisons of mean ranks, were used for the identification of significant differences in the microleakage of teeth restored using different adhesives. Within each adhesive system, the Mann-Whitney U-test, corrected for ties, was used to analyze the effect of bleaching on the enamel and dentin margins. Lastly, the Wilcoxon matched-pairs test was used to evaluate differences in the microleakage observed at the enamel and dentin margins. All of the statistical analyses were performed using statistical software (Statistica 10, StatSoft Inc, Tulsa, OK, USA) with a significance level of 0.05.

# Scanning Electron Microscope (SEM) Analysis

To evaluate the etching and penetration abilities of the adhesive systems, eight (n=2) more restorations were placed in the same way as the teeth that were prepared for the microleakage tests. After 24 hours in distilled water, the restorations were sectioned into two parts in an occlusal-cervical direction, demineralized in 6 N HCl for 24 hours, and then immersed into 5% NaOCl for 10 minutes to eliminate organic substances from the composite surface.<sup>15</sup> The composite surface was rinsed with distilled water, cleaned in an ultrasound bath, air-dried, sputter-coated with gold, and examined using a SEM (SEM, Jeol 5500, Tokyo, Japan). To investigate the morphology of the enamel-composite interface created with ADP and IBO, where increased dye penetration indicated marginal failure, tooth tissues adjacent to the restoration were broken off by gentle force, and the surfaces of both the enamel and the composite were analyzed with a SEM.

#### RESULTS

Table 2 shows the distribution of the microleakage scores and the results of the statistical evaluations. The results of the Kruskal-Wallis analyses indicate Table 2

Adhesive	Enamel						Dentin							WLC Test	
	Microleakage Score			K-W Test	M-W Test	Microleakage Score			K-W Test	Test	M-W Test				
	0	0.5	1	2				0	0.5	1	2				
GLU															
BG	5	3	2	0	А		NS	3	7	0	0	A		NS	NS
W	5	2	3	0		A		3	4	3	0		A		NS
CLF															
BG	1	1	7	1	А		0.030	6	1	3	0	A		NS	0.043
W	4	3	3	0		A		4	3	3	0		A		NS
ADP															
BG	0	0	6	3	в		NS	0	8	1	0	A		NS	0.008
W	0	0	8	2		В		5	4	1	0		A		0.008
IBO															
BG	0	0	8	1	В		NS	1	6	2	0	A		NS	0.02
W	0	0	7	3		В		3	7	0	0		A		0.005

differences between BG and W groups; WLC, Wilcoxon to test differences between enamel and dentin margins. Different letters within each column indicate a significant difference, NS, nonsignificant difference; p = 0.05.

that microleakage at the enamel margin of the GLU and CLF control groups stored in water was low and significantly smaller than that of the ADP and IBO groups (p < 0.0001). With these two adhesives, scores of 1 or higher indicated microleakage within the whole thickness of the enamel layer. At the dentin margin of the control groups, no significant differences between adhesives were found (p>0.65). After bleaching, a slight yet significant (p < 0.030) increase in microleakage at the enamel margin for CLF was revealed by the Mann-Whitney U-test (Table 2). This increase, however, did not affect the differences among the adhesives, which varied in the same order (GLU=CLF<ADP=IBO) as did the control groups (p < 0.0003). At the dentin margin, no significant differences were detected after bleaching (p>0.20). The Wilcoxon pairs test revealed significantly higher microleakage at the enamel margin than at the dentin margin for the control groups of ADP (p < 0.008) and IBO (p < 0.005); these tests also demonstrated increased enamel, compared with dentin microleakage, after bleaching for the CLF (p < 0.043), ADP (p < 0.008), and IBO (p < 0.02)groups.

Figure 1 shows the morphology of the enamel and dentin interfaces, which characterize the abilities of adhesive systems to demineralize and penetrate into the tooth tissues. For GLU, well-developed, long resin tags in enamel and in dentin, with clearly visible lateral branches, were observed (Figure 1a). The resin tags formed in the ADP and CLF enamel and dentin interfaces shortened and were less distinct compared to those associated with the GLU (Figure 1b,c). In the IBO-enamel interface a shallow



Figure 1. Morphology of enamel and dentin interfaces treated with various adhesive systems. Tooth tissues removed from the composite restoration surface by HCl and NaOCI: (a) GLU with long resin tags in both enamel and dentin interfaces. Resin tags in dentin with lateral branches parallel to the Class V cavity walls; (b) ADP with short tags in the enamel; (c) CLF with less distinct resin tags and the short dentin lateral branches; (d) IBO with poorly developed resin tags in the enamel and dentin interface and extensive porosity below the enamel-dentin junction.



Figure 2. Composite-enamel surfaces of ADP bonded restorations after peroxide treatment and microleakage test, composite surfaces: (a) distinct prismatic structure on the top of the adhesive layer, (b) remnants of ADP film pulled up from the enamel structure, (c) area of adhesive film disrupted with voids. A, adhesive resin; RA, a thin film of adhesive's remnants disrupted by voids.

prismatic structure could be recognized (Figure 1d), and in the dentin interface, only a small number of poorly developed resin tags were formed (Figure 1d). With IBO, extensive porosity was observed at deeper locations of the cavities below the dentin-enamel junction (Figure 1d). Analysis of the composite and enamel surfaces, where the dye penetrated along the ADP-bonded cavity, revealed their prismatic structure (Figure 2a), indicating failure at the enameladhesive interface. However, some areas of enamel or composite surfaces were covered with remnants of a thin film of the adhesive, pulled out from the enamel structure (Figure 2b) and disrupted by small voids (Figure 2c). Similarly analyzed surfaces of the IBO restorations displayed a prism-less texture and discrete grinding grooves representative of failure at the enamel-adhesive layer interface. Below the

enamel-dentin junction extensive porosity in the adhesive layer was found (Figure 3a,b).

# DISCUSSION

Frequent esthetic bleaching of vital teeth with peroxide products may adversely affect soft and hard oral tissues,<sup>16,17</sup> restoration materials, and the quality of the marginal seal. The risk of marginal failure due to degradation of the adhesive interface by oxygen radicals is particularly relevant for selfetch systems, which are prone to increased water sorption<sup>18</sup> and thus may be more susceptible to the penetration of small oxygen molecules than are etchand-rinse systems. Scission of the three-dimensional polymer network, the addition of oxygen radicals to unpolymerized monomers' double bonds, or reaction of radicals with ester groups might accelerate the

Figure 3. Composite-enamel surfaces of IBO bonded restorations after peroxide treatment and microleakage test, composite surfaces: (a) top of the adhesive layer without prismatic structure, grinding grooves caused by cutting burs are clearly visible; (b) extensive porosity at the bottom of the adhesive layer at deep parts of cavities below the enamel-dentin junction. A, adhesive resin.

degradation of the adhesive interface in a similar fashion to that of the polymer matrix of composite materials.<sup>19</sup>

Peroxide-induced degradation of the adhesive interface, however, has not yet been fully elucidated. Inconsistent results in the literature may be due to the different adhesives and bleaching systems studied, differences in bleaching protocols, and the various test methods employed, including, typically, bond strength and microleakage measurements. To clarify some of these discrepancies, the effect of bleaching on marginal integrity was evaluated in this study using the identical adhesives, peroxide bleaching gel, and application protocols of the previous research performed using the shear bond strength measurement.<sup>3</sup> We supposed that comparison of the bond strength results and microleakage should contribute to our understanding of the effects of peroxide bleaching on the composite-tooth tissue interfaces and help to clarify differences in adhesive performance often found with these methods.<sup>20</sup> The adhesives used differed in composition and application protocols (Table 1). The self-etching systems were represented by a two-step mild aggressive CLF, in which an acidic primer and hydrophobic bond are applied to tooth tissues in separate steps. ADP is a strongly acidic one-step self-etch adhesive containing hydrophilic 2-hydroxyethyl methacrylate and hydrophobic monomers. However, volatile solvents, such as ethanol or acetone, which usually serve to remove water from an adhesive layer, are not included in its formulation (Table 1). While these two adhesives contain phosphoric acid esters as selfetch primers, a mild all-in-one adhesive IBO is based on a weak acidic organic monomer, 4-methacryloxyethyl trimellitic anhydride. In its composition acetone is used as a solvent to facilitate water removal (Table 1). These adhesives were compared with the well-proven etch-and-rinse adhesive GLU.<sup>21-23</sup> Microleakage tests performed on the control groups stored in water showed that none of the adhesives guaranteed a perfect marginal seal and that the enamel margins created with ADP and IBO were more susceptible to failure than were those of GLU and CLF. After bleaching, a small but significant increase (p < 0.030) in microleakage was found for CLF at the enamel margin only. Hence, the null hypothesis stating that bleaching would not deteriorate the marginal seal was rejected.

Similar degradation of the marginal seal due to bleaching was reported for the etch-and-rinse system Single Bond,<sup>8</sup> at the enamel margin, and for Prisma Universal Bond III<sup>7</sup> and the self-etch adhesive Prompt L-Pop,<sup>9</sup> at the dentin margin. On the other hand, no adverse effect of bleaching on marginal seal was reported for the etch-and-rinse adhesives Scotchbond and Single Bond lines<sup>4,5,9,10</sup> and selfetch adhesive iBond.<sup>9</sup>

The higher microleakage at the enamel margin than at the dentin margin found for ADP and IBO (Table 2) also differs from the results of the majority of the other related studies, which usually report a lower resistance at the dentin margin.<sup>20</sup> The factors that may affect microleakage are the location, shape, and volume of the cavity, which control the polymerization shrinkage stress and its distribution at the cavity-restoration interface.<sup>11</sup> In our experiment, standardized cylindrical Class V cavities were prepared with a cavosurface angle of 90° without enamel beveling, which improves the marginal seal<sup>24</sup> by increasing the enamel-restoration bonded area in a more favorable orientation, perpendicular to enamel prisms.<sup>25</sup> Therefore, the results of our study cannot be compared with the results in which cavities with enamel beveling were prepared. <sup>7,9</sup> Other relevant factors include the type and coarseness of preparation burs, which affect the properties of the dentin and enamel smear layers.<sup>26-28</sup> The durability of the bond between the composite restoration and tooth tissues requires optimal demineralization and infiltration of the enamel and dentin by the adhesive components. These factors may be especially significant for mild self-etch adhesives, which possess a lesser ability to deeply demineralize the enamel smear layer than do the strong self-etch or etch-and-rinse systems.<sup>28</sup> Mechanical strength of polymerized adhesives can also play a significant role in the adhesive's performance.<sup>29</sup> This strength may be deteriorated by water residues in the adhesive, which decrease its degree of polymerization, the presence of residual lowmolecular substances acting as plasticizers, or the occurrence of structural defects in the adhesive layer. For example, bubbles and voids of various origins can act as stress concentrators, <sup>30,31</sup> initiating failure of the adhesive interface. As depicted in Figure 1, the demineralization and penetration abilities of the tested adhesives differed significantly. A distinct prismatic structure, as well as the presence of long resin tags in enamel and dentin interfaces, signify the optimal demineralization and deep penetration of GLU-bonding monomers into the etched tooth structures. Therefore, we can assume that the strong demineralization capacity of the phosphoric acid etchant of GLU, along with the deep penetration of the adhesive into the microporosities in enamel and dentin, produce a good marginal seal and confer stability in both water and peroxide bleaching gel. A good marginal seal created with CLF can be attributed not only to its stronger demineralization abilities but also to its unique formation of a hydrolytically stable chemical bond between the 10-methacryloyloxydecyl dihydrogen phosphate monomer and the calcium of tooth tissue hydroxyapatite.<sup>32-34</sup> However, increased microleakage at the enamel margin after bleaching might indicate a potential degradation of the interface under a combined effect of shrinkage stresses and a peroxide bleaching gel. The lower resistance of the enamel margin sealed with ADP is difficult to

explain because the opposite behavior should be expected as a result of ADP's more pronounced demineralization and penetration properties (compared with those of CLF). Analysis of the morphology of composite and enamel surfaces after the microleakage test revealed the complicated failure behavior of this adhesive interface under shrinkage stress in both environments. Remnants of the plastically deformed film of ADP, protruding from the prismatic enamel surface (Figure 2b) and in some areas disrupted by the voids (Figure 2c), indicated lower mechanical resistance of the ADP layer. It might be caused not only by structural defects in the adhesive layer but also by inappropriate polymerization resulting from the presence of water residues in the adhesive<sup>35</sup> or incompatibility of ADP acidic components with basic amines of the composite initiation system.<sup>36</sup> If the shrinkage stress of the composite material develops faster and exceeds the mechanical strength of ADP or its bond to the tooth tissues, a marginal failure can occur before the interface is degraded by the bleaching gel. On the other hand, IBO possesses a weaker organic acid in its formulation as a self-etch primer (Table 1), and it has a limited ability to create microporosities in enamel (Figure 1d), resulting in a low resistance of the enamel margin to the shrinkage stress.

Class V restorations at the cemento-enamel junction are produced in a strongly anisotropic substrate. One part of the restoration is bonded to enamel with a high elastic modulus, while the other part is bonded to dentin with a substantially lower elastic modulus. In addition, the bond strength between the substrates and the composite restoration can differ significantly, leading to heterogeneous stress distribution at the tooth-restoration interface and a difference in its stress resistance. If the weakest link between the restoration and tooth tissues breaks, it can be expected that the configuration factor of the restoration and shrinkage stresses acting in the opposite margins will decrease. Thus, if the enamel margin fails as in restorations made with ADP and IBO, stresses at the dentin margin should decrease accordingly. At a lower shrinkage stress, degradation of the dentin interface might thus be slower. With regard to this hypothesis, our results cannot fully exclude the possibility of a degradation of the ADP and IBO interfaces by the bleaching gel found by the bond strength measurement.<sup>3</sup>

Although originally developed to evaluate the sealing ability of nonadhesive restorations, such as amalgam fillings, microleakage tests on Class V

cavities are often used for testing adhesive restorations in which the mechanical properties of substrates and their interaction with an adhesive play key roles. To eliminate the effect of heterogeneous stress distribution, Class V cavities for microleakage tests should be prepared with margins in dentin or in enamel. If modeling a clinical situation, the cavity margins should be beveled, as recommended by standard preparation procedures, and the results should be analyzed with respect to the fact that the durability of cavity margins may depend not only on the adhesive bond strength but also on other factors specific to the test set-up. Thus, the obtained results may differ from the results of other *in vitro* tests.

### CONCLUSIONS

Within the limitations of this *in vitro* study, it can be concluded that a marginal seal resistant to postoperative bleaching can be created with etch-and-rinse adhesive GLU. On the other hand, a small but significant increase in the microleakage of two-step self-etch adhesive CLF at the enamel margin might indicate its susceptibility to degradation during peroxide bleaching. Compromised bonding performance of one-step self-etch adhesives ADP and IBO could mask their degradation in peroxide bleaching gel and could be the reason for their apparent resistance in the bleaching gel environment.

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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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#### REFERENCES

- 1. Barcellos DC, Benetti P, Fernandes Junior VVB, & Valera MC (2010) Effect of carbamide peroxide bleaching gel concentration on the bond strength of dental substrates and resin composite *Operative Dentistry* **35(4)** 463-469.
- Far C, & Ruse ND (2003) Effect of bleaching on fracture toughness of composite-dentin bonds *Journal of Adhesive Dentistry* 5(3) 175-182.
- 3. Dudek M, Roubickova A, Comba L, Housova D, & Bradna P (2012) The effect of postoperative peroxide bleaching on the stability of composite to enamel and dentin bonds *Operative Dentistry* 2012 Oct 23. [Epub ahead of print].
- 4. Khoroushi M, & Fardashtaki SR (2009) Effect of lightactivated bleaching on the microleakage of Class V toothcolored restorations *Operative Dentistry* **34(5)** 565-570.

- 5. White DJ, Duschner H, & Pioch T (2008) Effect of bleaching treatments on microleakage of Class I restorations Journal of Clinical Dentistry **19(1)** 33-36.
- Moosavi H, Ghavamnasiri M, & Manari V (2009) Effect of postoperative bleaching on marginal leakage of resin composite and resin-modified glass ionomer restorations at different delayed periods of exposure to carbamide peroxide *Journal of Contemporary Dental Practice* 10(6) 9-16.
- 7. Crim GA (1992) Post-operative bleaching: Effect on microleakage American Journal of Dentistry 5(2) 109-112.
- Ulukapi H, Benderli Y, & Ulukapi I (2003) Effect of preand postoperative bleaching on marginal leakage of amalgam and composite restorations *Quintessence International* 34(7) 505-508.
- Mortazavi V, Fathi M, & Soltani F (2011) Effect of postoperative bleaching on microleakage of etch-andrinse and self-etch adhesives *Dental Research Journal* 8(1) 16-21.
- Klukowska MA, White DJ, Gibb RD, Garcia-Godoy F, Garcia-Godoy C, & Duschner H (2008) The effects of high concentration tooth whitening bleaches on microleakage of Class V composite restorations *Journal of Clinical Dentistry* 19(1) 14-17.
- 11. Feilzer AJ, De Gee AJ, & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.
- Moszner N, Salz U, & Zimmermann J (2005) Chemical aspects of self-etching enamel-dentin adhesives: A systematic review *Dental Materials* 21(10) 895-910.
- ISO-Standards (2003) ISO/TS 11405 Dental Materials— Testing of Adhesion to Tooth Structure Geneve: International Organization for Standardization 2nd edition 1-16.
- Dejou J, Sindres V, & Camps J (1996) Influence of criteria on the results of in vitro evaluation of microleakage Dental Materials 12(6) 342-349.
- 15. Perdigao J, & Geraldeli S (2003) Bonding characteristics of self-etching adhesives to intact versus prepared enamel *Journal of Esthetic and Restorative Dentistry* **15(1)** 32-41.
- Naik S, Tredwin CJ, & Scully C (2006) Hydrogen peroxide tooth-whitening (bleaching): Review of safety in relation to possible carcinogenesis *Oral Oncology* 42(7) 668-674.
- 17. Goldberg M, Grootveld M, & Lynch E (2010) Undesirable and adverse effects of tooth-whitening products: A review *Clinical Oral Investigations* **14(1)** 1-10.
- Tay FR, Pashley DH, Suh BI, Carvalho RM, & Itthagarun A (2002) Single-step adhesives are permeable membranes *Journal of Dentistry* 30(7-8) 371-382.
- Durner J, Stojanovic M, Urcan E, Spahl W, Haertel U, Hickel R, & Reichl F-X (2011) Effect of hydrogen peroxide on the three-dimensional polymer network in composites *Dental Materials* 27(6) 573-580.
- 20. Heintze SD (2007) Systematic reviews: I. The correlation between laboratory tests on marginal quality and bond strength. II. The correlation between marginal quality

and clinical outcome *Journal of Adhesive Dentistry* **9(Supplement 1)** 77-106.

- Pde Cardoso C, Lopes GC, Vieira LC, & Baratieri LN (2005) Effect of solvent type on microtensile bond strength of a total-etch one-bottle adhesive system to moist or dry dentin *Operative Dentistry* 30(3) 376-381.
- 22. Ernest CP, Holzmeier M, & Willershausen B (2004) In vitro shear bond strength of self-etching adhesives in comparison to 4th and 5th generation adhesives *Journal* of Adhesive Dentistry **6**(4) 293-299.
- 23. Bradna P, Dudek M, Vrbova R, & Housova D (2008) Comparison of bonding performance of self and total-etch adhesives on human dentin using reliability analysis *Journal of Adhesive Dentistry* 10(6) 423-429.
- 24. Swanson TK, Feigal RJ, Tantbirojn D, & Hodges JS (2008) Effect of adhesive systems and bevel on enamel margin integrity in primary and permanent teeth *Pediatric Dentistry* **30(2)** 134-140.
- Crawford PJM, Whittaker DK, & Owen GM (1987) The influence of enamel prism orientation on leakage of resinbonded restorations *Journal of Oral Rehabilitation* 14(3) 283-289.
- 26. Kihn PW, Spanganberg PA, & von Fraunhofer JA (2004) The role of cavity preparation and conditioning in the leakage of restorations *Journal of Adhesive Dentistry* **6(4)** 287-291.
- Sattabanasuk V, Vachiramon V, Qian F, & Armstrong SR (2007) Resin-dentin bond strength as related to different surface preparation methods *Journal of Dentistry* 35(6) 467-475.
- Mine A, De Munck J, Cardoso Vivan M, Van Landuyt KL, Poitevin A, Kuboki T, Yoshida Y, Suzuki K, & Van Meerbeek B (2010) Enamel-smear compromises bonding

by mild self-etch adhesives *Journal of Dental Research* **89(12)** 1505-1509.

- 29. Takahashi A, Sato Y, Uno S, Pereira PN, & Sano H (2002) Effects of mechanical properties of adhesive resins on bond strength to dentin *Dental Materials* **18(3)** 263-268.
- 30. Van Landuyt KL, De Munck J, Snauwaert J, Coutinho E, Poitevin A, Yoshida Y, Inoue S, Peumans M, Suzuki K, Lambrechts P, & Van Meerbeek B (2005) Monomersolvent phase separation in one-step self-etch adhesives *Journal of Dental Research* 84(2) 183-188.
- 31. Van Landuyt KL, Snauwaert J, De Munck J, Coutinho E, Poitevin A, Yoshida Y, Suzuki K, Lambrechts P, & Van Meerbeek B (2007) Origin of interfacial droplets with onestep adhesives *Journal of Dental Research* 86(8) 739-744.
- 32. Salz U, Zimmermann J, Zeuner F, & Moszner N (2005) Hydrolytic stability of self-etching adhesive systems Journal of Adhesive Dentistry 7(2) 107-116.
- 33. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, Inoue S, Tagawa Y, Suzuki K, De Munck J, & Van Meerbeek B (2004) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* 83(6) 454-458.
- 34. Inoue S, Koshiro K, Yoshida Y, De Munck J, Nagakane K, Suzuki K, Sano H, & Van Meerbeek B (2005) Hydrolytic stability of self-etch adhesives bonded to dentin *Journal* of Dental Research 84(12) 1160-1164.
- Jacobsen T, & Soderholm KJ (1995) Some effects of water on dentin bonding *Dental Materials* 11(2) 132-136.
- Tay FR, King NM, Suh BI, & Pashley DH (2001) Effect of delayed activation of light-cured resin composites on bonding of all-in-one adhesives *Journal of Adhesive Dentistry* 3(3) 207-225.