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Laboratory Research

Effect of Postoperative Peroxide Bleaching on the Stability of Composite to Enamel and Dentin Bonds

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Clinical Relevance

The increased risk of adhesive bond degradation by bleaching should be considered when simplified self-etching adhesives are used. This should be taken into consideration while planning bleaching of adhesively reconstructed teeth.

SUMMARY

This study investigated the effect of peroxide bleaching gel on the durability of the adhesive bond between composite material, enamel, and dentin created with the etch-and-rinse adhesive Gluma Comfort Bond (GLU) and with the self-etch adhesives Clearfil SE Bond (CLE), Adper Prompt (ADP), and iBond (IBO). The adhesives were applied to flattened enamel and dentin of extracted human molars and built up with a microhybrid composite (Charisma). After 25 eight-hour cycles of bleaching with a 20% carbamide peroxide bleaching gel (Opalescence PF 20), the shear bond strength was measured and compared with one-day and two-month control specimens stored in water. The data were analyzed using nonparametric Mann-Whitney and Kruskal-Wallis statistics ($p<0.05$). Detailed fractographic analysis was performed using scanning electron microscopy. The bleaching gel significantly decreased the bond strength on both enamel and dentin for the simplified single-step self-etch adhesives ADP and IBO and markedly affected a

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fracture pattern of ADP specimens at the periphery of their bonded area. The results of our study indicate that the durability of adhesive restorations can be detrimentally influenced by carbamide peroxide bleaching and that different adhesives show varying sensitivity levels to the bleaching gel.

INTRODUCTION

The stability of the adhesive bond between composite restorations and tooth tissue is one of the main factors determining restoration longevity. This bond is challenged by mechanical stresses; water penetration, which causes plasticization and hydrolysis of its resinous components; and degradation of collagen fibers in the dentin hybrid layer.[1] In addition, chemical agents, such as those contained in foodstuffs, drinks, mouth rinses, disinfectants,[2,3] and peroxide bleaching gels, may affect bond longevity. Vital bleaching is now widely demanded by the public and ranks among the most frequent cosmetic treatments in dentistry. Office-administered and self-administered bleaching gels contain hydrogen peroxide or its precursor, carbamide peroxide, as the active ingredient in concentrations ranging from 3% to 35% of hydrogen peroxide equivalent. It is supposed that hydroxyl radicals (HO·), perhydroxyl radicals (HOO·), perhydroxyl anions (HOO–), and superoxide anions (OO·–) released from peroxides attack double bonds of chromophore molecules captured within tooth tissues.[4-6] The change in double-bond conjugation causes a shift in the absorption spectrum of chromophore molecules and thus bleaching of tooth tissues. Because of the high reactivity and nonspecific nature of these oxygen radicals, they may have a side effect on tooth tissues,[7,8] reconstruction materials,[9-11] and the bond between them, which is usually the most susceptible to degradation.

To date, most studies have focused on the preoperative influence of bleaching gels on adhesion of composites to enamel. It has been shown that freshly bleached enamel cannot establish a full-value bond to composites because residues of peroxide and oxygen in the enamel structure inhibit free radical polymerization of acrylate or methacrylate monomers.[6] Fewer studies have dealt with the influence of bleaching gels on the adhesive bond of previously prepared composite restorations. This effect was analyzed using different methods, including measurement of bond strength,[12,13] fracture toughness,[14] and microleakage.[15] The results of these studies are ambiguous, however. Some authors reported compromised adhesion to enamel, but not to dentin,[12] while others described a decreased adhesion to dentin[14] or to both tissues[13] after bleaching.

The aim of our study was to investigate the effect of repeated application of carbamide peroxide bleaching gel on the enamel-composite and dentin-composite shear bond strength and fractographic analysis of four contemporary etch-and-rinse and self-etch adhesive systems. The null hypothesis tested was that repeated bleaching has no effect on enamel-composite and dentin-composite bond strength.

METHODS AND MATERIALS

Materials

Four adhesive systems were chosen: 1) etch-and-rinse two-step Gluma Comfort Bond with Gluma Etch 20 Gel (GLU, Heraeus Kulzer GmbH, Hanau, Germany), 2) self-etch two-step Clearfil SE Bond (CLE, Kuraray Medical Inc, Okayama, Japan), 3) self-etch one-step Adper Prompt (ADP, 3M ESPE AG, Seefeld, Germany), and 4) self-etch all-in-one iBond (IBO, Heraeus Kulzer GmbH), all combined with a Charisma microhybrid composite (Heraeus Kulzer GmbH). Their composition and working protocols are summarized in Table 1. The adhesive systems and the composite material were applied strictly according to the manufacturer's instructions and light-cured with a halogen lamp (Elipar Trilight, 3M ESPE AG). The lamp output power of 800 mW/cm^2 was monitored at regular intervals by an integrated radiometer. Bleaching was performed with a 20% carbamide peroxide bleaching gel (Opalescence PF 20, Ultradent Products Inc, South Jordan, UT, USA), which is routinely used for home bleaching. The gel, which has an equivalent concentration of 6.6% of hydrogen peroxide, was stored at 4°C and used in the first quarter of its shelf life to minimize its decomposition and the risk of decreased carbamide peroxide concentration.

Design of the Study

Sound human third molars extracted for orthodontic reasons were used in this study. After extraction, the teeth were cleaned and stored for one week in a 0.5% chloramine-T aqueous solution and then in distilled water at 4°C as recommended by International Organization for Standards Technical Specification 11405:2003. The teeth were used within six months after extraction. The tooth roots were cut off by an Isomet low-speed saw using a diamond wafering blade in water spray (Buehler Ltd, Lake Bluff, IL,

Table 1: *Materials and Their Working Protocols*

Material	Manufacturer	Chemical Composition	Application[a]
Adhesive System			
Gluma Comfort Bond (GLU)	Heraeus Kulzer GmbH, Hanau, Germany	*Etchant:* Gluma Etch 20 Gel (phosphoric acid 20%) *Bond:* HEMA, 4-META, polyacid, ethanol, photoinitiators, polyacrylic acids	e (20 s), r, d (1–2 s), 3× b (15 s), w (15 s), d, c (20 s)
Clearfil SE Bond (CLE)	Kuraray Mediacal Inc, Okayama, Japan	*Primer:* MDP, HEMA, hydrophilic dimethacrylate, camphorquinone, N,N-diethanol-p-toluidine, water *Bond:* MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, N,N-diethanol-p-toluidine, silanated colloidal silica	p (20 s), d, b, d, c (10 s)
Adper Prompt (ADP)	3M ESPE AG, Seefeld, Germany	*A liquid:* methacrylated phosphoric esters, bis-GMA, initiators based on camphorquinone, stabilizers *B liquid:* water, HEMA, polyalkenoic acid, stabilizers	m (A+B), a (15 s), d, a, d, c (10 s)
iBond (IBO)	Heraeus Kulzer GmbH	4-META, UDMA, glutaraldehyde, acetone, water, photoinitiators, stabilizers	3× a, w (30 s), d, c (20 s)
Composite			
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 percentage, sodium fluoride 0.25 weight percentage potassium nitrate	25× 8 hours

Abbreviations: bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 4-META, 4-methacryloxyethyl trimellitic anhydride;
TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *Application protocol: a, application; b, bonding; c, curing; d, drying/spreading; e, etching; m, mixing; p, priming; r, rinsing; w waiting.*

USA), and the teeth were fixed in a stainless steel ring using the Spofacryl self-curing resin (SpofaDental as, Jicin, Czech Republic).

The 240 teeth were randomly divided into enamel and dentin groups (n=120) for shear bond strength testing. In the enamel group, the buccal or lingual enamel was ground with 600 grit P1200 SiC paper (Buehler Ltd) to yield a flat enamel surface of approximately 3–4 mm in diameter. In the dentin group, enamel was removed by a low-speed saw to expose dentin. A standard smear layer was created by grinding dentin with SiC paper P1200. The dentin and enamel groups were each divided into four subgroups (n=30) to test the four adhesive systems. These subgroups were further divided into two control groups (n=10), one group stored for one day and one group stored for two months in distilled water, and one experimental group (n=10) that was exposed to the bleaching gel 25 times for eight hours daily over two months.

Twelve more specimens with exposed enamel and dentin were prepared to characterize the conditioning ability of the adhesives.

Shear Bond Strength Test

After the adhesive system was applied on enamel or dentin and light-cured, composite build-ups were prepared using a cylindrical polyethylene mould (3.5 mm in diameter, 2 mm high) placed on the tooth surface and filled with one portion of composite and light-cured for 20 seconds. All the specimens were immersed in distilled water for one day, after which the mold was carefully removed. With the experimental groups, approximately 0.1 g of the bleaching gel was applied around the adhesive interface, and

Table 2: *Enamel Shear Bond Strength Results (Mpa) Expressed as Mean and (SD)*

Adhesive	**Water**				**Bleaching Gel**	
	1 Day		**2 Months**			
GLU	24.3	(4.0)	23.3	(3.8)	20.2	(2.9)
CLE	18.3	(5.4)	19.7	(3.6)	22.0	(1.5)
ADP	20.4	(2.0)	16.9	(2.6)	11.2	(3.3)
IBO	13.0	(1.9)	14.3	(4.3)	8.9	(3.1)

the specimens were then wrapped in Parafilm foil (Parafilm M, Alcan Packaging, Chicago, IL, USA) with a small piece of wet absorbent cotton around the stainless steel ring to keep the standard level of moisture. After eight hours, the bleaching gel was carefully removed using distilled water and a soft toothbrush, and the specimens were left in distilled water until the next application of bleaching gel. The two control groups were stored in distilled water for one day and for two months, respectively. The specimens were kept in an incubator at 37°C under all storage conditions. After being exposed, the specimens were fixed in a shear bond testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) and stressed with a universal testing machine (AGS-G, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.75 mm/min. Shear bond strength (MPa) was calculated by dividing the force at the fracture by the area of the bonded interface.

Microscopy of Fractured Surfaces

Fractographic analysis was performed using a stereomicroscope (SMZ 2T, Nikon, Tokyo, Japan) at magnifications from 10× to 60×. A detailed analysis was made by a scanning electron microscope (SEM, JSM 5500-LV, Jeol, Tokyo, Japan) at magnifications from 20× to 6000×. Before SEM observation, the specimens were left to dry for one week at room temperature and then sputter-coated with gold (JFC-1200 Fine coater, Jeol). According to the location of fracture line, the fractures were assigned to one of the following categories: 1) interfacial fractures located between the substrates at the dentin/enamel-adhesive resin interface plus fractures in the adhesive resin and the hybrid layer; 2) cohesive fractures in the substrates where fractures propagated through the tooth tissues or composite material; 3) mixed mode of fracture, including interfacial and cohesive fractures. The area fraction of interfacial and the cohesive fracture category on the dentin side were measured using image analysis software (SigmaScan 5, SPSS Inc, Chicago, IL, USA) on SEM images at 25× magnification.

Conditioning Properties of Adhesive Systems

The priming components of each adhesive system were applied onto enamel and dentin according to the manufacturer's recommendation (Table 1). After rinsing with water (GLU) or acetone (CLE, ADP, IBO) to remove the adhesive monomers, the specimens were air-dried and analyzed using an SEM.

Statistical Analysis

To compare the initial bonding performance of the adhesives, statistical analysis of the shear bond strengths of one-day control groups was carried out using nonparametric Kruskal-Wallis multiple comparisons of mean ranks. To analyze the effect of bleaching gel on enamel and dentin bond strength of each adhesive system, the difference between control groups stored in distilled water for one day and for two months was first determined using the nonparametric Mann-Whitney U test. If no significant time effect was revealed, the bond strength data measured in water were pooled and compared with those of the experimental bleached group using the same statistics. In contrast, the bleached group was compared with the specimens stored in distilled water for two months. All the statistical analyses were performed at a confidence level of 95% and calculated using statistical software (Statistica 7.1, StatSoft Inc, Tulsa, OK, USA).

RESULTS

Shear Bond Strength

The mean shear bond strength values and standard deviations (SDs), together with nonparametric characteristics represented by median, first and third quartiles, and minimum and maximum values on enamel and dentin for each group, are shown in Tables 2 and 3 and Figures 1 and 2. As shown, the one-day bond strength on enamel decreased as follows: GLU > ADP > CLE > IBO, with the GLU ($p<0.00001$) and ADP ($p<0.002$) significantly exceeding the IBO bond strength. On dentin, the bond strength decreased as follows: CLE > GLU > ADP > IBO, and the IBO bond strength was significantly lower ($p<0.03$) than the CLE bond strength. After two months in water, the adhesives showed low

Table 3: *Dentin Shear Bond Strength Results (Mpa) Expressed as Mean and (SD)*

Adhesive	Water				Bleaching Gel	
	1 Day		2 Months			
GLU	22.3	(2.1)	24.1	(2.7)	19.6	(4.8)
CLE	25.0	(4.2)	26.9	(3.9)	22.0	(1.8)
ADP	20.7	(2.9)	17.2	(4.4)	14.5	(2.1)
IBO	17.5	(6.7)	20.0	(3.9)	13.4	(2.3)

decrease or increase in bond strength compared with the one-day groups (Tables 2 and 3). Statistical evaluation (Figures 1 and 2) showed that, except for ADP on dentin, the differences between the two-month and one-day groups were not significant. Therefore, the bond strengths of these systems measured in water were pooled for the subsequent statistical evaluation of the bleaching gel effect. With ADP on dentin, however, the two-month group was used as a control. Statistical analysis showed that GLU and CLE bond strength on enamel and dentin changed significantly after bleaching compared with water ($p = 0.02\text{-}0.03$) (Figures 1 and 2). With ADP and IBO, the bond strength on enamel and dentin dropped significantly after exposure to bleaching gel with lower values of p=0.003–0.0005, which unambiguously indicated adhesive joint degradation.

Fractographic Analysis

Enamel—After exposure to water and bleaching gel, the fractured surfaces of GLU, CLE, and IBO showed a typical prismatic texture with grooves resulting from abrasive particles of SiC papers and small areas of adhesive resin remnants (Figure 3), suggesting failure predominantly at the enamel-adhesive resin interface. With ADP groups in water, large areas of cohesively fractured adhesive resin with cracks perpendicular to the load direction were found (Figure 4A). After bleaching, however, there were two irregular zones of failure: an outer rim and a central zone (Figure 4C). Although the fracture character of the central zone was not different from that of the control groups, at the outer rim debris of composite, filler particles and small voids were found after particle debonding (Figure 4D), which indicated a failure between the top of the adhesive resin and the composite. Similar voids were observed on the composite side of the outer rim and on the composite build-up areas directly exposed to the bleaching gel, indicating filler particle debonding from the composite matrix.

Dentin—With the GLU and CLE groups, a high number of mixed fractures were found within composite or dentin after water storage. Although the extent of cohesive fractures within the substrate in CLE was retained after bleaching, it decreased

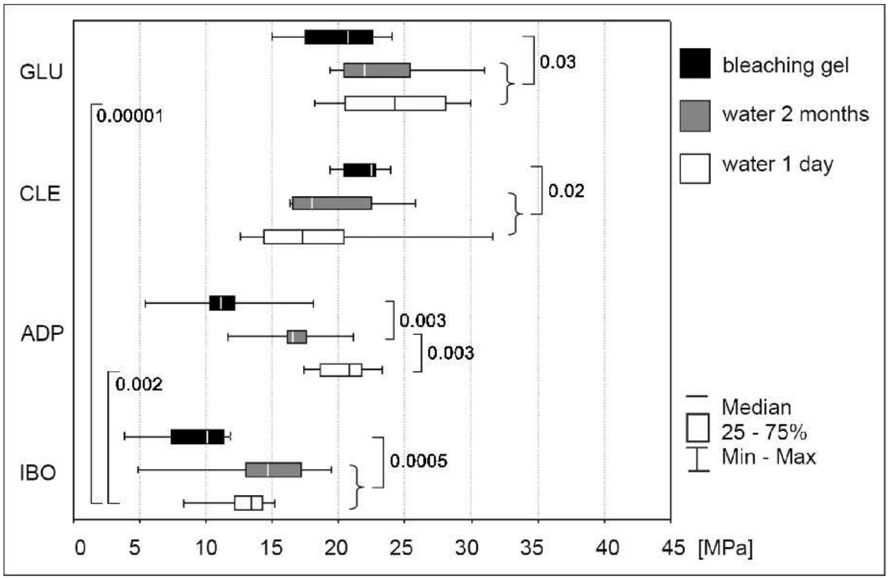

Figure 1. Enamel shear bond strength of the adhesive systems in the tested environments. Braces indicate pooled data. Statistically significant differences are marked by vertical lines and corresponding p values.

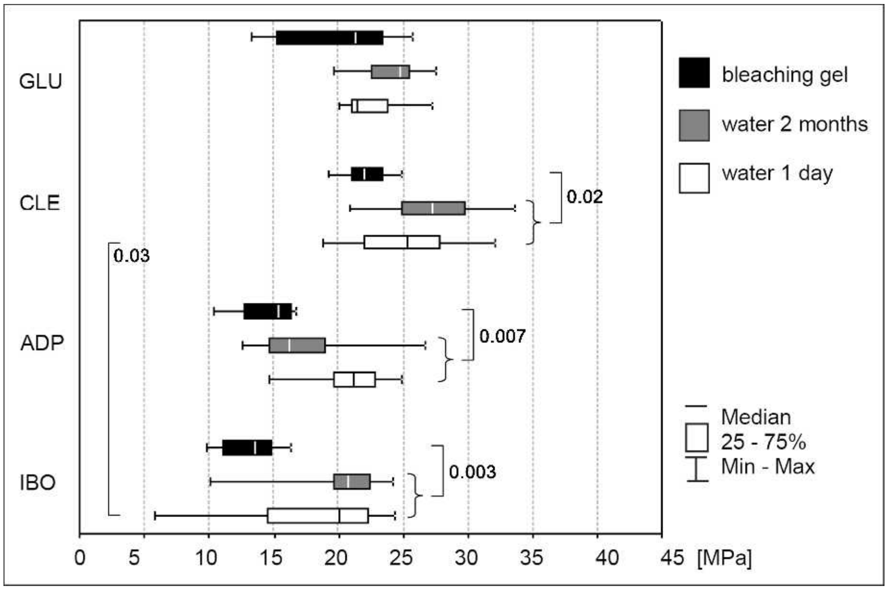

Figure 2. Dentin shear bond strength of the adhesive systems in the tested environments. Braces indicate pooled data. Statistically significant differences are marked by vertical lines and corresponding p values.

with GLU in favor of the interfacial type (Fig 7). Different behavior was found with self-etch adhesives ADP and IBO. With ADP, cohesive fractures within the adhesive resin or interfacial fractures between adhesive resin and composite occurred after water storage. After exposure to bleaching gel, the outer rim and central zone were observed on the fractured surface as on enamel, but with different fracture characteristics (Figure 5A). The central zone (Figure 5B) showed a similar fracture character as it did after water storage. In the outer rim, however, the fracture line passed through the adhesive resin but it had smooth fractured surfaces and no pronounced morphologic features (Figure 5C). With IBO in water, we observed areas of fractures between dentin and adhesive resin with resin tags in tubuli orifices (Figure 6A) and areas of cohesive fractures within adhesive resin with composite filler particles on top (Figure 6B). There were voids at the composite-adhesive resin interface on some specimens (Figure 6C), as frequently found in similar adhesives.[16] After the bleaching, the fractures between dentin and adhesive resin prevailed (Figure 6D).

Conditioning Properties of Adhesive Systems

The enamel and dentin surfaces after priming are shown in Figure 8. Gluma Etch 20 Gel etched the dental tissues most aggressively as the typical prismatic enamel structure and opened dentinal tubuli were clearly visible on the etched surfaces. An aggressive etching was also found for ADP, where wide open tubuli and areas of etched enamel with prismatic structure resembled the pattern created with phosphoric acid. However, less distinguishable enamel prisms in some areas suggested sensitivity of its conditioning properties on the enamel structure. On the other hand, enamel covered with the smear layer with grooves originating from the abrasive particles of SiC paper were typical of the lower etching ability of mild CLE and IBO adhesives. Even though the smear layer on dentin was removed and the dentin tubuli opened, deep grooves on its surface confirm their lower demineralization properties.

DISCUSSION

Many studies have shown that long-term exposure to water decreases the bond strength between tooth tissues and composite materials. The extent and mechanism of bond degradation largely depends on the composition of the adhesive system, its application protocol, the substrate to be bonded, and the length of water exposure.[1, 17-19] Much less information, however, is available showing how chemical substances or products intended for use in the oral cavity affect adhesive bond durability. Such products include, among others, peroxide bleaching gels for vital bleaching, which are one of the most frequently used cosmetic oral cavity treatments today. Three studies[12-14] dealing with the postoperative effect of bleaching gels on bond strength provide controver-

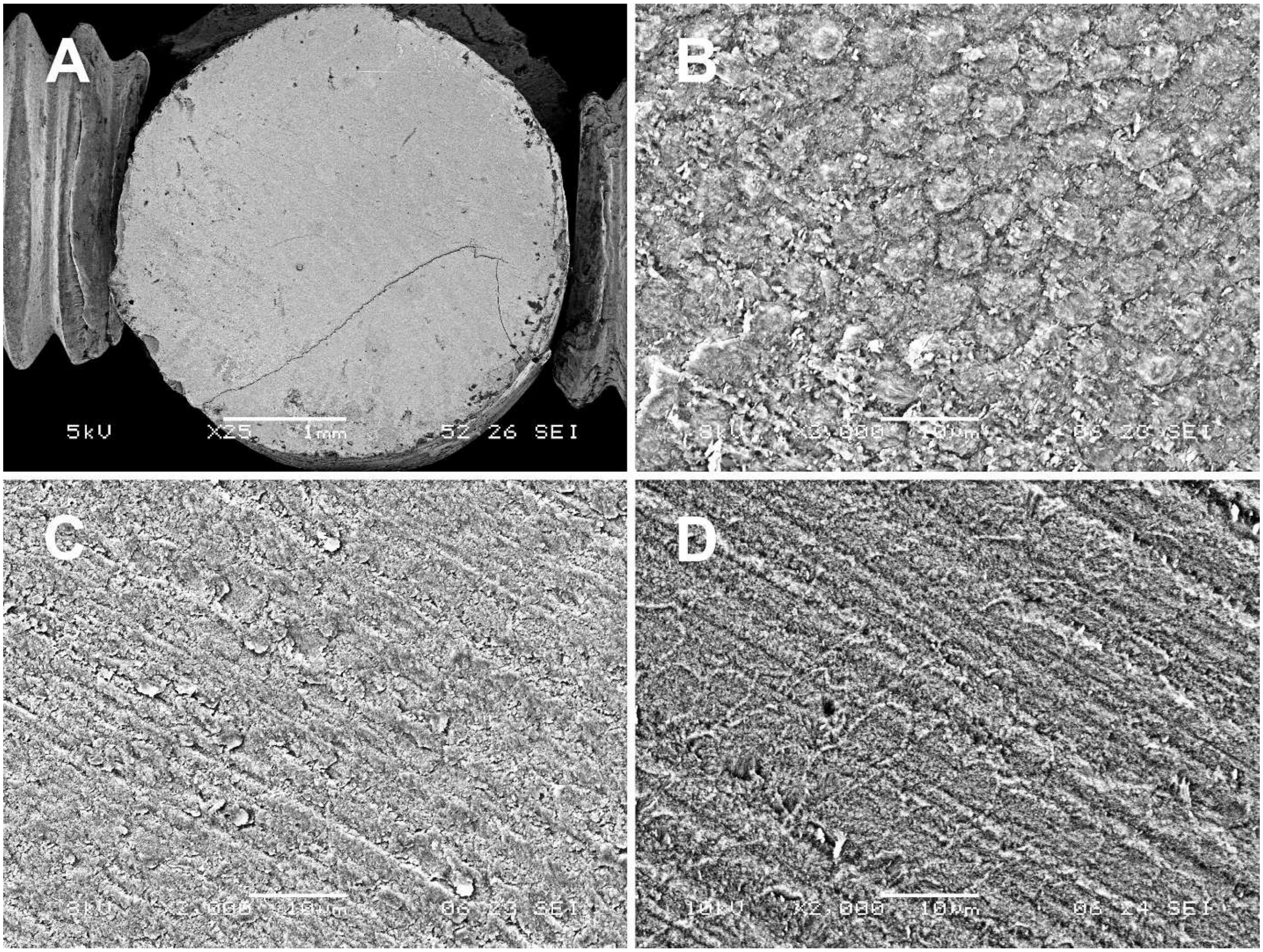

Figure 3. Representative enamel-composite fracture surfaces of GLU, CLE, and IBO specimens after water and bleaching gel expositions. (A): GLU, composite side, with fracture between the adhesive layer and enamel (25×). (B): GLU, enamel side, with enamel prisms clearly visible (2000×). (C): CLE, enamel side, surface covered with the remnants of smear layer with grinding grooves and shallow depressions resembling prismatic structure (2000×). (D): IBO, enamel side, prism borders on the grooved enamel surface (2000×).

sial results. Cavalli and others[12] used a microtensile bond strength method to study the resistance of enamel-composite and dentin-composite bonds created with the two-step etch-and-rinse Single Bond and the two-step self-etch Clearfil SE Bond. After bleaching with a 10% carbamide peroxide gel for six hours daily over two weeks, a decrease in the enamel-composite bond strength for Clearfil SE Bond was found. No significant influence of the bleaching gel on the dentin-composite bond strength was reported. On the other hand, Barcellos and others,[13] using a two-step etch-and-rinse Adper Single Bond 2, observed a decrease in the microtensile bond strength to bovine enamel and dentin after treatment with bleaching gel containing 15% and 20% carbamide peroxide for six hours daily for two weeks. Degradation of the dentin-composite bond created with the two-step etch-and-rinse Single Bond was reported by Far and Ruse,[14] who tested its resistance to bleaching gels of increasing carbamide peroxide concentrations by measuring fracture toughness. Their results show that carbamide peroxide gels at concentrations higher than 11% compromised fracture toughness of the dentin-composite joint after 70 hours of cumulative exposure. A change in fracture mode from cohesive fracture within the adhesive resin to fracture between dentin and adhesive resin interface suggested degradation at the dentin-adhesive interface.

As bond strength and its susceptibility to degradation depend on the adhesive system,[20-22] a few typical adhesives with different working protocols

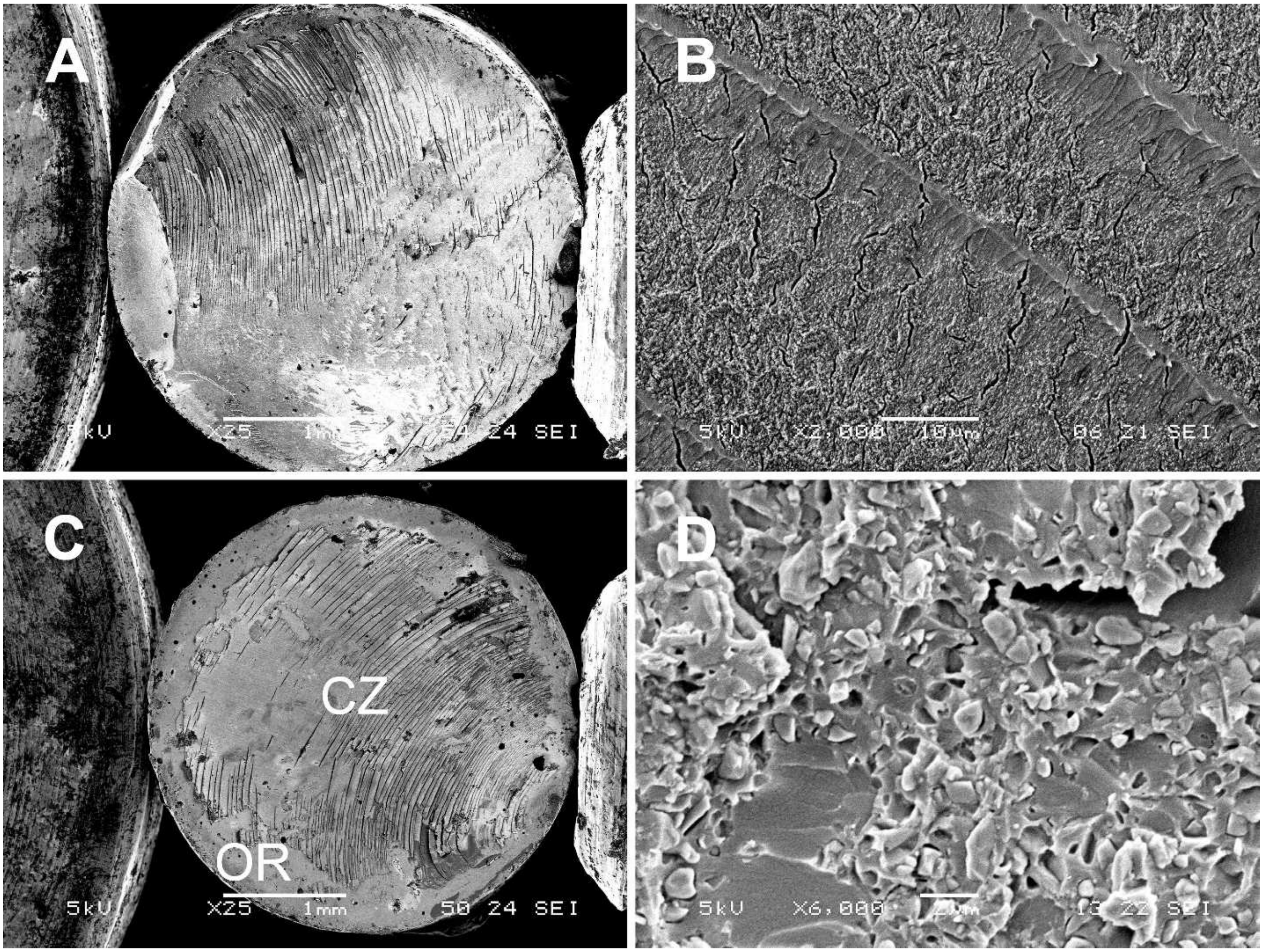

Figure 4. Representative enamel-composite fracture surfaces of ADP specimens. (A): Water, two months, composite side, fracture between adhesive resin and enamel (25×). (B): Enamel side corresponding to (A), enamel prisms and remnants of adhesive resin (2000×). (C): Bleaching gel, composite side, the outer rim and the central zone (25×). (D): Bleaching gel, enamel side, detail of the outer rim, top of the adhesive resin layer covered with remnants of composite material and voids after debonded composite filler particles (6000×). OR, the outer rim; CZ, central zone.

representing the state-of-the-art of current adhesive technology were chosen in our study. The bleaching gel containing 20% of carbamide peroxide as a representative of safe in-office and home systems[23] was applied in 25 cycles each for eight hours as recommended by the manufacturer for night bleaching. A statistical analysis revealed differences in the resistance of the bond strength of individual adhesive systems to the bleaching gel (Figures 1 and 2). Etch-and-rinse adhesive GLU and self-etch CLE, which rank among the most reliable adhesive systems proven *in vitro* and in clinical tests,[24,25] showed slight but significant changes in bond strength on enamel and, in the case of CLE, on dentin (Figures 1 and 2). Fractographic analysis revealed predominantly interfacial fractures between enamel and adhesive resin and mixed fractures within dentin or within composite on dentin groups; however, there was no pronounced change after bleaching gel application (Figure 7). Mixed fractures, including substrates, occur frequently in the shear bond strength measurement, resulting from uneven stress distribution in this experimental setup and differences in the mechanical properties of dentin and a composite.[26,27] In these cases, bond strength should be evaluated with care as the basic requirements on interfacial fracture are not met and the calculated bond strength will therefore not truly reflect the actual bond strength. With these adhesive systems,

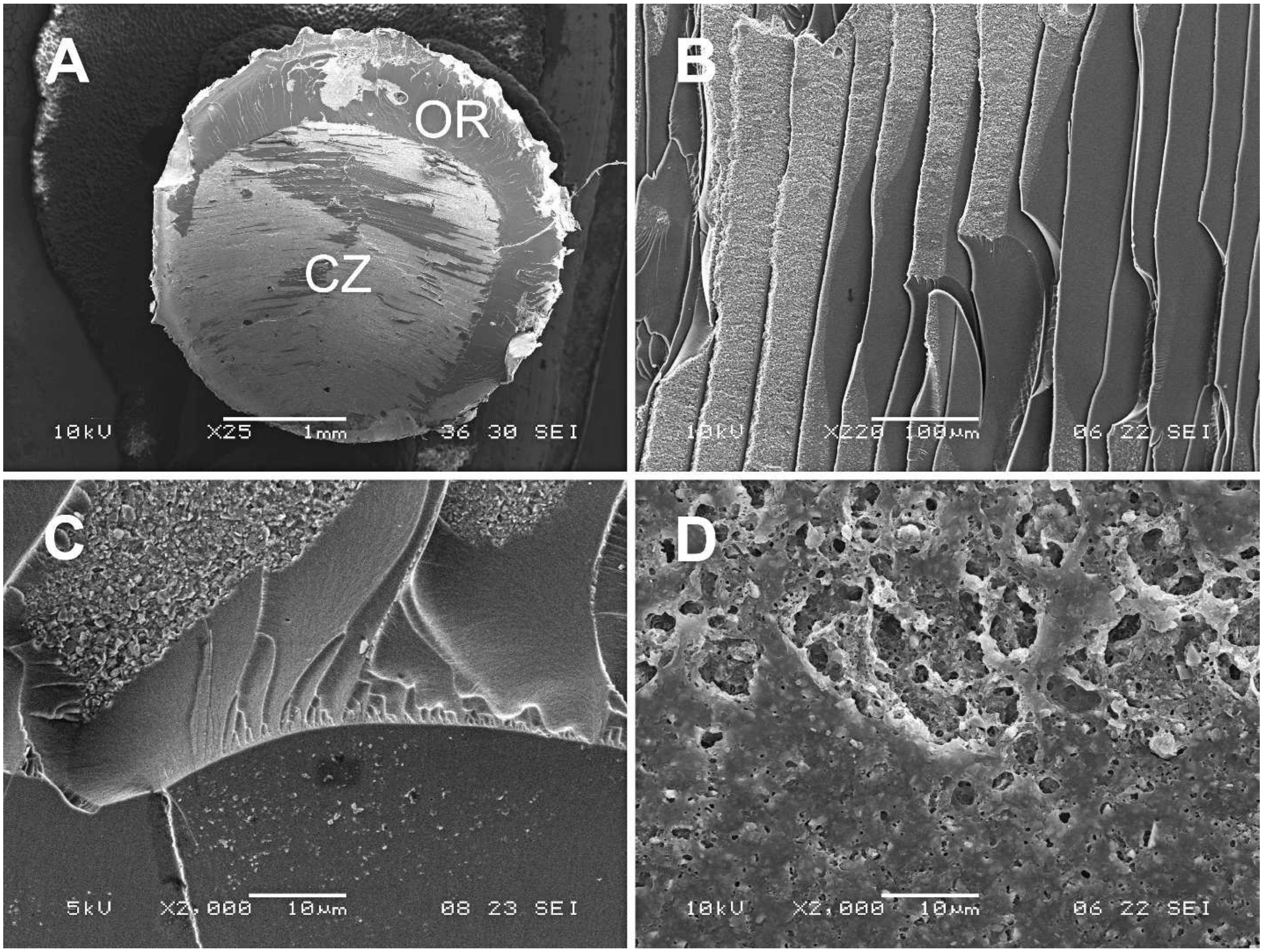

Figure 5. Representative dentin-composite fracture surfaces of ADP specimens after bleaching. (A): Composite side, the outer rim (OR) and the central zone (CZ) (25×). (B): Dentin side, detail of the CZ, cohesive fracture within the adhesive layer, and composite filler particles on its top (220×). (C): composite side, border line between the OR and the CZ, cohesive fracture in adhesive layer and fracture extending into composite material (2000×). (D): composite side, small region of voids in the OR (2000×).

the statistical significance p=0.02–0.03, close to the critical value p=0.05, and a considerable number of mixed fractures within both substrates do not allow for a reliable and unambiguous conclusion concerning the effect of a bleaching gel on their bond strength.

On the other hand, a significant decrease in the bond strength of ADP and IBO after bleaching with a low statistical significance p=0.007–0.0005 (Figures 1 and 2) indicates bond degradation that was more pronounced than in water. With ADP, bond strength degradation was also reflected in the pronounced change of fracture pattern at the periphery of specimens in close contact with the bleaching gel. In this area of irregular depth, fractures located at the adhesive resin-composite interface on enamel or within adhesive resin on dentin were found (Figure 4). This outer rim, which resembled the fractured surfaces of adhesive joints exposed to degradation in NaOCl solutions,[28] indicated gradual penetration of the bleaching gel reactive components into the adhesive joint and its degradation. The irregular depth of the outer rim might have resulted from the different thickness of the adhesive layer or an imperfectly defined adhesive edge during the preparation of composite build-ups onto tooth tissue surfaces.

These results show that peroxide bleaching gels may induce degradation of adhesive bonds between the composite material and tooth tissues. Its extent, however, depends largely on the composition of the adhesive system. Hence, the null hypothesis on

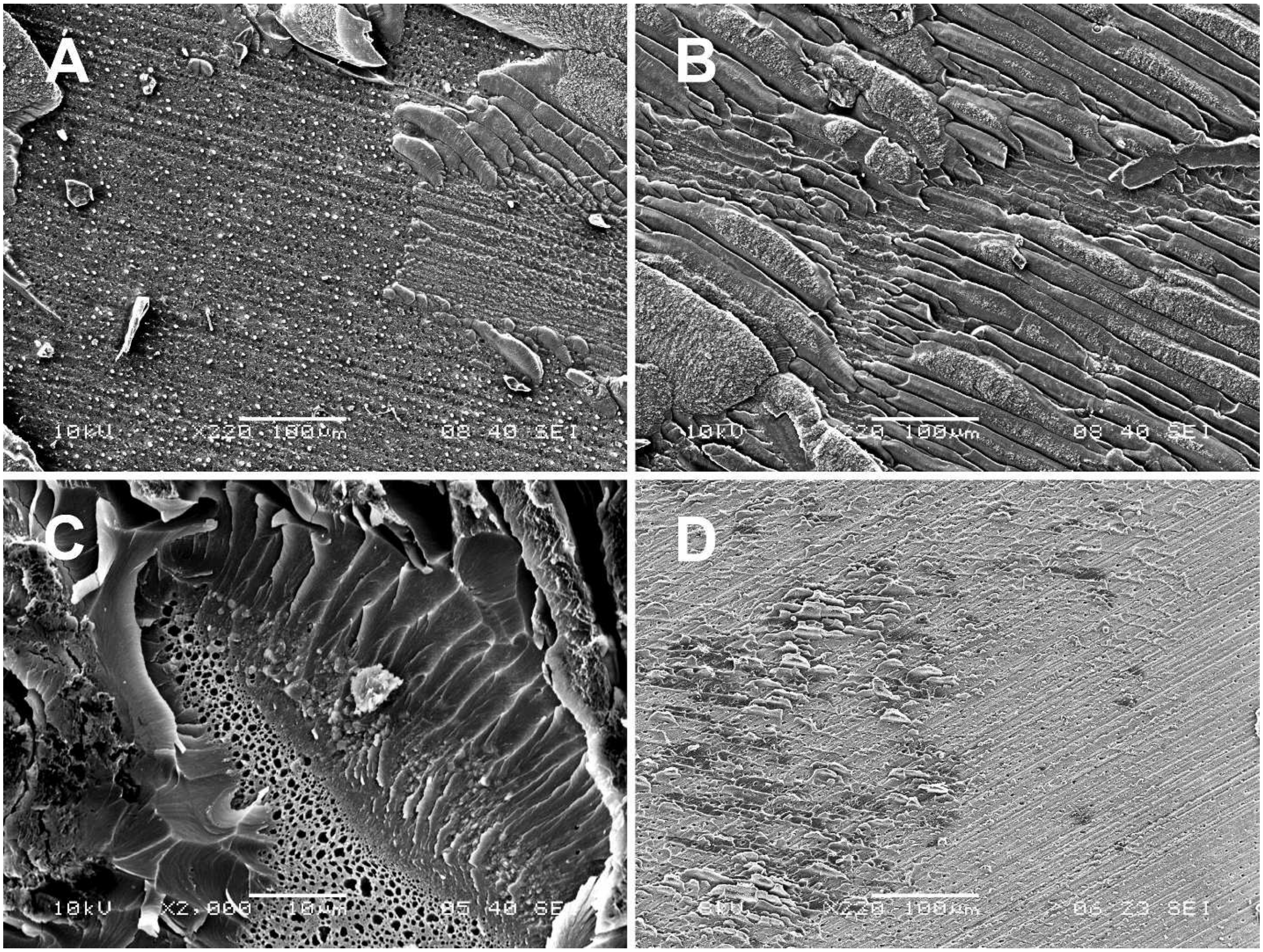

Figure 6. Representative dentin-composite fracture surfaces of IBO specimens. (A): Water, one day, dentin side, fracture between dentin and adhesive resin with grooves created by grinding particles and small areas of cohesively failed adhesive resin (220×). (B): Water, one day, dentin side, cohesive fracture within adhesive resin with composite filler particles on its top (220×). (C): Water, one day, composite side, small region of voids at composite-adhesive resin interface (2000×). (D): Bleaching gel, dentin side, fracture between dentin and adhesive resin with grooves from grinding and remnants of adhesive resin (220×).

resistance of adhesive joints to the peroxide bleaching gels can be rejected.

The bond strength studies revealed acceptable long-term stability of enamel-composite bond created with etch-and-rinse[29,30] and decreased stability of the bond created with self-etch adhesives.[31] The dentin-composite bond stability, on the other hand, is usually lower than that of the enamel-composite bond. The bottom of the dentin hybrid layer, particularly improperly impregnated collagen fibrils, is the most frequently attacked zone. Hydrolysis of collagen fibrils, their enzymatic decomposition, and adhesive resin hydrolysis are assumed to be the main mechanisms of dentin bond degradation[32] and occur after a few months of water storage.[30] Lower resistance of some self-etch adhesives arises not only from the presence of incompletely impregnated collagen fibrils,[33] as with etch-and-rinse adhesives, but also from their considerable hydrophilic properties, which allow water to penetrate into the adhesive joint,[34] causing water treeing,[35] swelling, and plasticization of the adhesive polymer matrix.[34] With these adhesives, defects that arise because of imperfect polymerization in the presence of water,[36] voids, blisters, and water droplets resulting from phase separation of non-miscible components of some adhesives or from penetration of water from dentin to adhesive-composite interface[37,38] should be considered. Such defects may act as stress concentrators, accelerat-

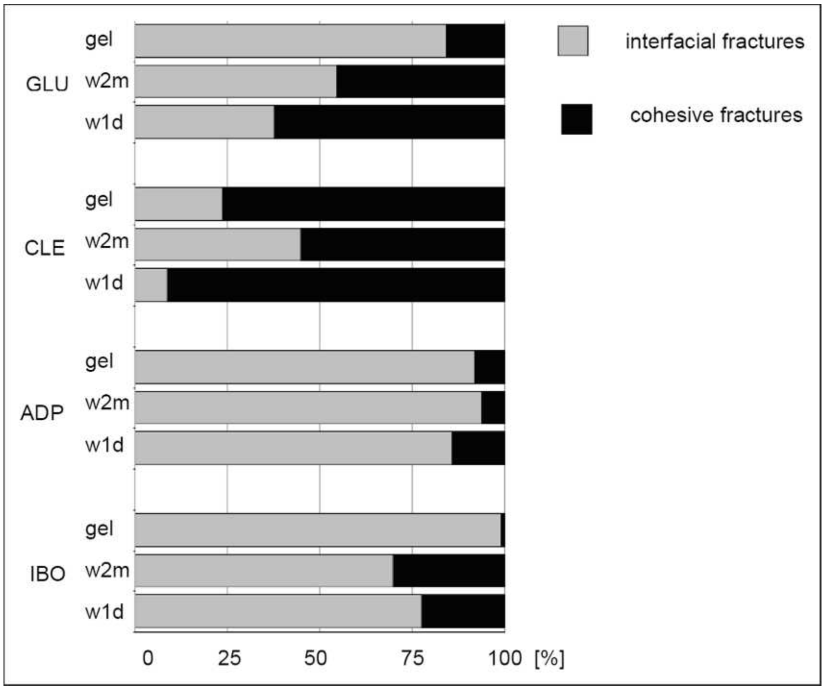

Figure 7. Distribution of fracture categories for dentin groups expressed as area percentage. Bleaching gel (gel); water, two months (w2m); water, one day (w1d).

ing water penetration and diminishing adhesive joint mechanical strength. In addition, the bond strength of adhesive systems also depends on their ability to remove and demineralize the smear layer and subsurface tooth tissues after tooth preparation. We assume that the GLU high bond strength and high bond resistance to bleaching and water resulted from optimally etched enamel and dentin surfaces with numerous microretentions created with a phosphoric acid etching (Figure 8). With self-etch CLE, which has weaker etching properties (Figure 8), a chemical bond between the MDP monomer and the hydroxyapatite of tooth tissues[39] may be a key factor contributing to its high bond strength and resistance to bleaching gel. In addition, a hydrophobic resin of this two-step system might seal primed tissues and form a barrier against water and active substances penetrating from the bleaching gel in the adhesive joint.[21] The lower bond resistance of one-step self-etch adhesive ADP to the bleaching gel is not surprising. This adhesive contains water necessary for the ionization of acidic monomers and 2-hydroxyethyl methacrylate (HEMA), which dissolves hydrophilic and hydrophobic components, enabling a homogeneous solution to be formed. Because of its high boiling point, HEMA is not as effective in removing water from the adhesive applied on dental tissues as the acetone or ethanol contained in other systems; thus, water retained in the adhesive structure might decrease its degree of polymerization[36,40] Such a system will be prone to water sorption and to degradation, as reflected in the decrease in ADP bond strength even after storage in water for two months. For the predecessor of ADP, the adhesive Prompt L-Pop (3M ESPE AG, Seefeld, Germany), which had similar composition, it was reported that because of the reaction of its acidic components with basic amines present in the composite photoinitiation system, composite polymerization may have been inhibited, resulting in decreases in bond strength.[41] Although such decreases did not occur until a few minutes after their contact, it might have started as soon as the uncured composite with a camphorquinone-amine photoinitiating system was applied on incompletely polymerized adhesive resin. In the enamel group, the outer rim surface covered with filler particles (Figure 4D) suggests that this region of the ADP resin-composite interface was the primary site of the bleaching gel penetration and attack. In contrast, a smooth fracture surface of the outer rim in the dentin group indicated a fracture inside the adhesive resin (Figure 5C). This may be due to a higher water content absorbed by ADP from wet dentin, easier penetration of the reactive bleaching gel compo-

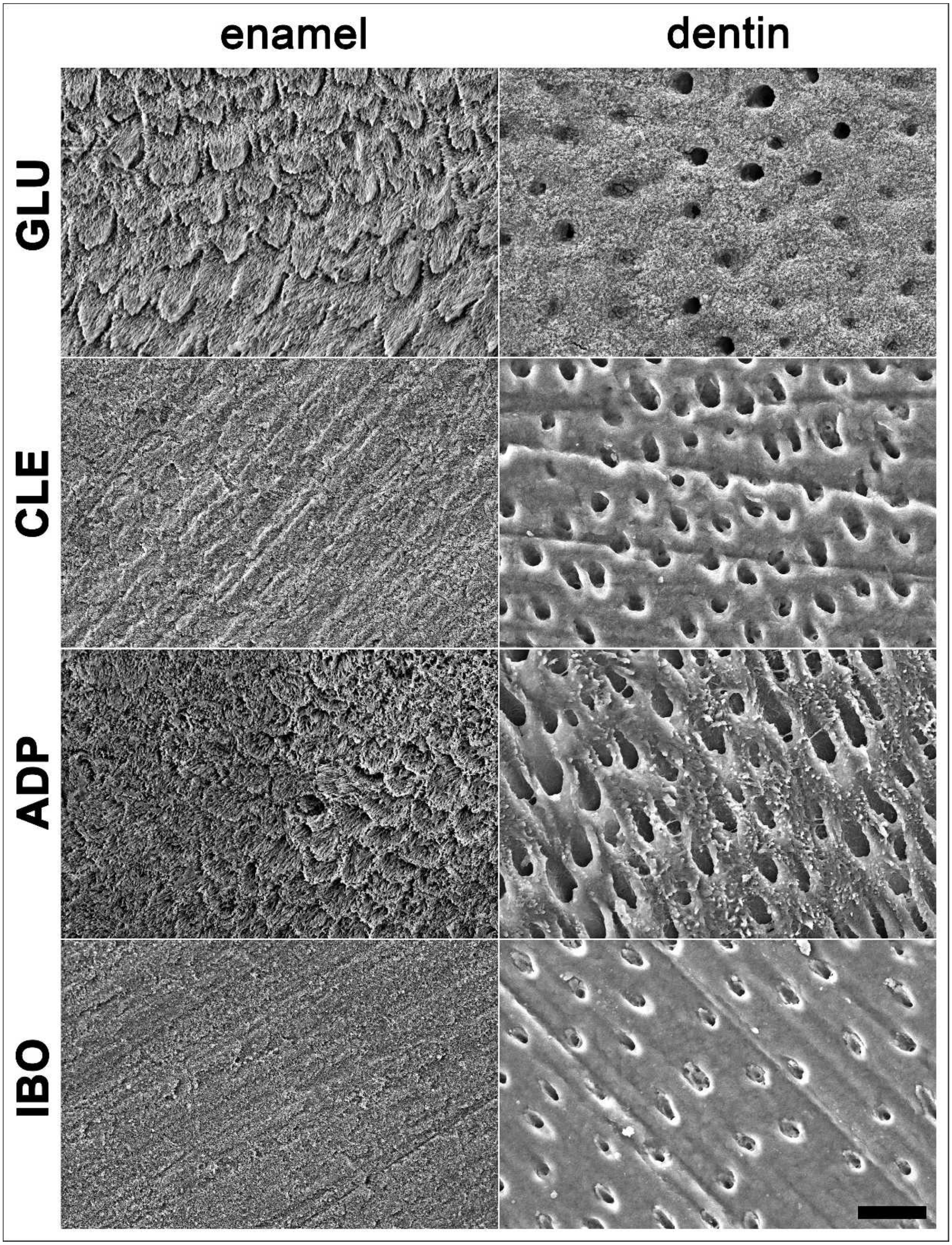

Figure 8. Morphology of enamel and dentin after conditioning. GLU Etch 20 was applied for 20 seconds. Note the prismatic structure of enamel. Dentin with open dentinal tubuli is covered with pyrogenic SiO_2 particles used as a thickening agent. CLE primer was applied for 20 seconds. SiC paper grinding grooves are visible on both enamel and dentin. Smear layer covers enamel but dentinal tubuli are opened. ADP was applied for 15 + 10 seconds. The smear layer is removed, and enamel prisms and open dentinal tubuli are visible. IBO was applied for 30 seconds. The smear layer is not removed from enamel, and dentinal tubuli with visible orifices and grinding grooves are clearly apparent. The bar length indicates 10 μm.

nents through the adhesive structure, and its fast oxidative degradation.[42] The question arises as to why similar behavior was not observed with IBO, too. It contains the less aggressive organic 4-methacryloxyethyl trimellitic anhydride (4-META) primer, which has demineralization properties that are not as efficient as those of ADP, which is obvious from its inability to remove the smear layer from highly mineralized enamel (Figure 8) and its initial shear bond strength being the lowest of all tested adhesives. Predominantly enamel or dentin-adhesive resin interface failure suggested that, as a result of the lower demineralization efficiency of IBO, this interface was the weakest region of the IBO adhesive joint and the most susceptible to degradation.

CONCLUSIONS

Our study has shown that carbamide peroxide bleaching gel may cause degradation of adhesive bonds. However, the type of the adhesive system used seems to be the factor determining the susceptibility of the adhesive bond to the influence of bleaching gel.

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Conflict of Interest

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

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