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Immediate Bonding to Bleached Enamel

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Clinical Relevance

When immediate bonding to bleached enamel is to be implemented, organic-solvent based adhesives are not capable of eliminating or reducing the adverse effect of bleaching systems on bond strength.

SUMMARY

This research sought to determine the shear bond strength, degree of resin infiltration and failure mode when organic solvent-based adhesives (acetone or ethanol) were used in immediate bonding to enamel bleached with 10% carbamide peroxide or 38% hydrogen peroxide systems. Seventy-two non-carious bovine incisors were randomly assigned to three groups of 24 specimens each—control group (deionized water), 38% hydrogen peroxide bleach group and 10% carbamide peroxide bleach group. Each group was further subdivided into two subgroups of 12 specimens each according to the adhesive system used to bond the resin composite to enamel surfaces. The two adhesive systems used were Single Bond, an ethanol-based adhesive, and One Step, an acetone-based adhesive. The shear bond strengths of 38% hydrogen peroxide and 10% carbamide peroxide were significantly lower compared to the non-bleached controls. Fractography revealed an adhesive failure mode in all specimens. Qualitative comparisons of resin tags present in the bleached and unbleached specimens using scanning electron microscopy (SEM) revealed few, thin and fragmented resin tags when 38% hydrogen peroxide and 10% carbamide peroxide were used.

INTRODUCTION

The advent of bleaching materials in the late 1980s represented an important milestone in cosmetic dentistry. Materials such as hydrogen peroxide, sodium perborate and carbamide peroxide are generally used for the bleaching processes. The ability of these products to lighten tooth color is evident, but the safety of some of these oxidizing agents is a subject of concern. Pulpal irritation (Robertson & Melfi, 1980; Seale, McIntosh & Taylor, 1981), changes in the tooth struc-

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ture (Ledoux & others, 1985; Rotstein, Lehr & Gedalia, 1992; Lewinstein & others, 1994), microleakage of restorations (Crim, 1992a,b), external root resorption (Stokes & others, 1992; Titley, Torneck & Ruse, 1992; Titley & others, 1993), reduced bond strength of resin composites (Cvitko & others, 1991; Kalili & Yoshida, 1993; Barghi & Godwin, 1994; Cavalli & others, 2001; Glasspoole, Erickson & Davidson, 2001; Lai & others, 2002) and other alterations (Harrington & Natkin, 1979; Weitzman & others, 1986; Herrin, Squier & Rubright, 1987; Rotstein & others, 1991) have been associated with these agents.

Several studies have shown a reduction in enamel bond strength when the bonding procedure is carried out immediately and up to one week after vital bleaching with hydrogen peroxide and carbamide peroxide-based bleaching agents (Titley & others, 1992; García-Godoy & others, 1993; Dishman, Covey & Baughan, 1994; Miles & others, 1994). This reduction in enamel bond strength has become a concern in cosmetic dentistry with regard to clinical operative procedures that involve resin bonding, such as porcelain veneers, composite veneers or future composite restorations, where vital bleaching is often considered a first step to improving the appearance of teeth prior to applying a bonded restoration. A period of up to three weeks is required before resin-enamel bond strengths return to values obtained for unbleached enamel (Cavalli & others, 2001).

One of the mechanisms proposed in the literature that may account for this reduction in bond strength is the presence of residual oxygen, which inhibits free-radical polymerization (Kalili & Yoshida, 1993; Barghi & Godwin, 1994). The oxygen in hydrogen peroxide is believed to be absorbed into enamel and dentin and is then released through surface diffusion and accumulates within the enamel structure, causing the inhibition of resin polymerization, which affects the bond strength of resin composite to enamel. Data from several experiments have shown that the use of organic solvent-based adhesives (acetone or ethanol) may result in less compromised composite bond strength when restorative work is to be completed immediately after bleaching, eliminating the need for any treatment delays or enamel resurfacing (Kalili & Yoshida, 1993; Barghi & Godwin, 1994; Sung & others, 1999). However, conflicts were noted among these studies regarding the most effective solvent. Lack of standardization and methodological differences did not allow for accurate conclusions about interactions between organic solvent-based adhesives and bleached enamel.

The antagonistic role of oxygen as an active ingredient in bleaching agents and as a polymerization inhibitor provided the background and significance for this investigation.

The fact that studies investigating the effect of organic solvent-based adhesives on bleached enamel are conflicting merits further investigation in this area. The question as to whether these adhesives can be equally efficient at reversing the effect of high and low concentrations of hydrogen peroxide is raised when immediate bonding is needed. As a result, this study was designed to compare and investigate the effect of acetone-based and ethanol-based adhesives on the shear bond strength of resin composite bonded immediately to enamel that has been bleached with 10% carbamide peroxide or 38% hydrogen peroxide. Scanning electron microscopy was used to help elucidate the effect of such adhesives on the ultramorphological structure of the resin/bleached-enamel interface.

METHODS AND MATERIALS

A) Experimental Factors

The two factors studied were: 1) treatment regimens (10% carbamide peroxide, 38% hydrogen peroxide [experimental groups] and deionized water [control group]) and 2) adhesive systems (acetone-based and ethanol-based adhesives).

B) Experimental Design

Seventy-two non-carious bovine incisors obtained from a local slaughterhouse were used in this investigation. The specimens were randomly assigned to three groups of 24 specimens each: Control Group (deionized water), 38% Hydrogen Peroxide Bleach Group (Opalescence Xtra Boost; Ultradent, South Jordan, UT, USA) and 10% Carbamide Peroxide Bleach Group (Opalescence; Ultradent). Each group was further subdivided into two subgroups of 12 specimens each, according to the two adhesive systems being used to bond the resin composite to enamel surfaces—Single Bond (3M ESPE Dental Products, St Paul, MN, USA), an ethanol-based single-bottle adhesive and One Step (BISCO, Inc, Schaumburg, IL, USA), an acetone-based single-bottle adhesive.

C) Specimen Preparation

Bovine teeth were stored in an aqueous solution of 0.5% chloramine-T (9.0 g sodium chloride and 5.0 g chloramine-trihydrate dissolved in 1000 ml distilled water) for a maximum of one week, thereafter kept in deionized water and refrigerated at 4°C, then used within one month of extraction (Hegedus & others, 1999). To minimize deterioration, the storage medium was replaced every week. One investigator fabricated all the specimens. The roots were separated from their crown using a low-speed saw (ISOMET, Buehler, Lake Bluff, IL, USA). The crown segments were fixed, with the exposed enamel down, on double-sided adhesive tape on a glass slide. They were then embedded in self-curing resin by placing a mold that was open on both ends on the adhesive tape and filling the mold with the mixed resin. The labial surfaces were ground flat using 400-grit and 600-grit sandpaper on a polishing machine (Ecomet 3 variable speed Grinder-Polisher, Buehler) to expose a flat enamel surface large enough for the bonding procedures. The specimens were then examined under an optical microscope (Universal Large Research Microscope, Zeiss Instrument Inc, Oberkochen, Germany) to eliminate any cracked teeth, and the teeth were kept in deionized water at room temperature for one week prior to the experimental procedures.

Exposure of the Specimens to the Bleaching Systems

The specimens were randomly assigned to three groups:

Experimental Group, 10% CP—the enamel surfaces were subjected to one application of a bleaching regimen of Opalescence for six hours per day for five consecutive days. This regimen is consistent with recommendations provided by the manufacturers of many 10% CP bleaching systems on the market. Approximately 0.1 ml of bleaching agent was applied to the enamel surface of each specimen, and the specimens were kept separately in closed plastic containers. After the bleaching period, bleaching gel was removed under deionized running water for one minute per specimen. During the remainder of the day, the specimens were individually kept in deionized water (Lai & others, 2002) that was changed daily after bleaching.

Experimental Group, 38% HP—the enamel surfaces were subjected to a 30-minute treatment session with Opalescence Xtra Boost. An approximately 0.5 to 1.0-mm thick layer of bleaching agent was applied to the enamel surface of each specimen. The gel was then removed under deionized running water for one minute per specimen.

Control Group—the specimens in the control group were kept in deionized water for 24 hours. During the bleaching cycles and after daily bleaching, the experimental and control specimens were placed in a humid atmosphere at 37°C.

Bonding Procedures

After treatment, all specimens were bonded immediately. The adhesive system application protocols were conducted according to the manufacturer's instructions. The bonded surfaces were light-cured for 10 seconds using a visible light-curing unit (Optilux 501, Kerr Inc, Orange, CA, USA). The adequacy of light intensity in the 540-570 nm wavelength range was assessed every 10 curing cycles using the built-in radiometer. A stainless steel washer (4.13-4.15 mm internal diameter and 1-mm thick) was placed on the enamel surface and dental composite (Supreme, Enamel shade A1E, 3M ESPE Dental Products) was inserted into the inner diameter of the washer in one increment. The enamel surface was then light-activated for 20 seconds. The washers were coated with a releasing agent to prevent them from bonding to the adhesive resin, and the teeth were then stored in deionized water at 37°C for 24 hours.

D) Shear Bond Strength Evaluation

Shear testing was conducted following storage for 24 hours. The specimens were mounted in a custom jig, then loaded to failure in shear, using a mechanical test machine (Model 858 Mini Bionix II, MTS Systems Corporation, Eden Prairie, MN, USA). A steel blade was placed on the rim of the washer parallel to the tooth surface, allowing the washer to distribute the loading force around the composite button. The specimen was loaded at a constant crosshead speed of 0.5 mm/minute until a maximum was recorded in the load-deflection curve. Bond strengths were calculated using the load required to debond the specimen and area of resin composite. Mean bond strengths and standard deviations were calculated. The results were analyzed using a computer program (SPSS 12 for Windows, SPSS Incorporated, Chicago, IL, USA) with two-way analysis of variance (treatment regimen vs adhesives) followed by multiple comparisons with Tukey's test to determine if significant differences in bond strengths existed among treatment subgroups at the 0.05 significance level.

E) Ultramorphological Evaluation & Scanning Electron Microscopy

The resin and enamel interfaces showing resin penetration were examined using SEM (JSM 6300, JEOL, Japan). One representative fractured specimen from each group was studied. Each specimen was cross-sectioned and polished on 600 grit silicon carbide papers. The sections were etched for 60 seconds with 50% (v/v) nitric acid, rinsed thoroughly with deionized water and left to desiccate overnight at room temperature. The specimens were sputter-coated with gold, then mounted on aluminum SEM stubs with the cross-sectioned surfaces exposed for examination by SEM at magnifications of 1300, 1500 and 3000x using 15 kV accelerating voltage. Representative images were captured digitally and stored in computer files.

F) Failure Mode Analysis (Fractographic Analysis)

The debonded surfaces were examined at 20x magnification using a stereomicroscope (Stereomicroscope SR, Zeiss; Oberkochen, West Germany). Determination of the failure mode was made by one investigator and recorded as a percentage of the bonding area. Failure was identified as "adhesive" when more than 75% of the bonding area appeared to be clean enamel, "cohesive in resin composite" when more than 75% of the bonding area was covered with remnants of resin composite, "cohesive in enamel" if a portion of the tooth was removed in the debonding of the material and "mixed failure" when between 25% and 75% of the failure was both adhesive and cohesive.

Table 1: *Two-way Analysis of Variance (ANOVA) for Shear Bond Strengths*

Treatment	Sum of Squares	Df	Mean Square	F	Sig	Observed Power
Bleach (A)	1563.9	2	781.9	56.3	<0.001	1.00
Adhesive (B)	33.3	1	33.3	2.4	0.12	0.34
(A) x (B)	24.6	2	12.3	0.8	0.41	0.20
Error	906.0	66	13.7			
Total	2527.8	71				

Table 2: *Shear Bond Strength (MPa)*

Treatment	Adhesive	Mean ± sd	n
Carbamide Peroxide	Single Bond	(9.4 ± 4.4)^{bc}	12
	One Step	(10.2 ± 3.2)^{b}	12
Hydrogen Peroxide	Single Bond	(5.2 ± 2.9)^{c}	12
	One Step	(8.2 ± 2.9)^{bc}	12
Deionized Water	Single Bond	(17.6 ± 4.0)^{a}	12
	One Step	(17.9 ± 4.2)^{a}	12

Means with the same superscript are not significantly different at (p<0.05)
n = number of tested specimens
sd = standard deviation

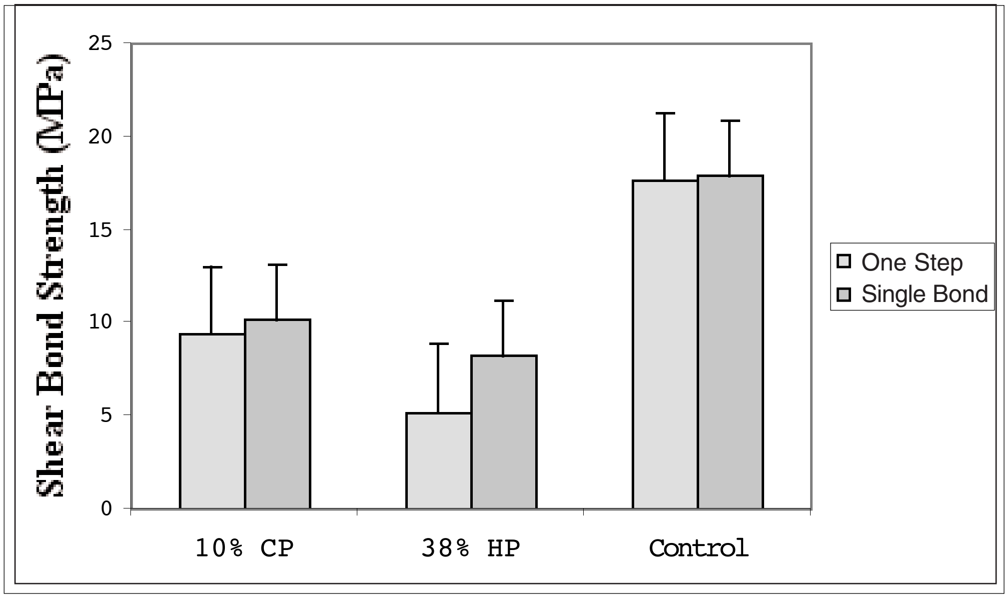

Figure 1. *Graph of shear bond strength (MPa) between treatment regimens and adhesive systems grouped by treatment regimens. Bars show means and Error Bars show 95% CL.*

RESULTS

Shear Bond Strength

The analysis of variance (Table 1) indicated a statistically significant difference among the three treatment regimens ($p<0.001$) but not between the adhesives ($p=0.12$). There was no significant interaction between the two factors ($p=0.41$). Control/ethanol and control/acetone subgroups had the highest bond strength values compared to all other subgroups (Table 2) and were not statistically different from each other ($p \geq 0.05$).

Effect of Adhesive System

For each treatment regimen, the subgroup fabricated using One Step adhesive (acetone-based) had a higher bond strength than the subgroup fabricated using Single Bond adhesive (ethanol-based), but the difference was not statistically significant. For both adhesives, bond strengths were significantly reduced when bonding to enamel bleached with either 10% carbamide peroxide or 38% hydrogen peroxide. The 38% hydrogen peroxide/ethanol subgroup had the lowest bond strength value among all subgroups (Figure 1).

Effect of Bleaching System

For the 10% carbamide peroxide and 38% hydrogen peroxide-bleached subgroups, bond strengths were significantly lower than that of the non-bleached controls ($p<0.001$), and the bond strengths for 10% carbamide peroxide were significantly higher than for 38% hydrogen peroxide ($p=0.01$).

Failure Analysis

All specimens exhibited 100% adhesive failure. Examination of the fractured surfaces using the stereomicroscope did not reveal any breakaway of the enamel or any apparent residual composite on the enamel surface. Most specimens displayed areas of pooled adhesive resin around the periphery of the bonded zone.

Scanning Electron Microscopic Evaluation

Figures 2 through 7 show the representative photomicrographs of resin penetration of the six tested subgroups. The resin tags, which could clearly be seen penetrating the enamel in all specimens, were long, thick, dense and coagulated in the control (non-bleached) specimens (Figures 2 and 3). Figure 4 depicts few, but long resin tags, and areas devoid of resin tags in the 10% Carbamide peroxide/One Step specimen. When

Single Bond was used with 10% carbamide peroxide, the resin tags appeared short, and there were few of them (Figure 5). Fewer resin tags could be seen penetrating enamel in the hydrogen peroxide specimens (Figures 6 and 7).

DISCUSSION

In this study, there was no interaction between bond strength to bleached enamel and the bonding agents used. The bleached groups, when treated with either ethanol-based or acetone-based adhesives, demonstrated a statistically significant decrease in bond strengths compared to the non-bleached group. This observation contradicts the hypothesis and findings of Barghi and Godwin (1994) and Sung and others (1999), that stated adhesives containing organic solvents may reverse the drop in bond strength found immediately after bleaching.

It has been suggested that acetone is the best solvent for carrying resin into the conditioned tooth surface, because of its effective role as a "water chaser" that displaces water from the tooth surface. This role is supported by experimental evidence (Gwinnett & Matsui, 1967; Kanca, 1992; Jacobsen & Soderhold, 1995; Swift, Perdigão & Heymann, 1998). For each treatment regimen in this study, the subgroup fabricated using One Step adhesive (acetone-based) had higher bond strength than the subgroup fabricated using Single Bond adhesive (ethanol-based), but the difference in bond strengths was statistically insignificant. Despite these facts, the observed power for adhesive systems factor was found to be 0.34, which means that the chance of detecting a difference between adhesives, if there actually was a difference, was only 34%. Based on this, it cannot be determined with a high degree of certainty whether adhesives are different or similar in their effect on bond strength.

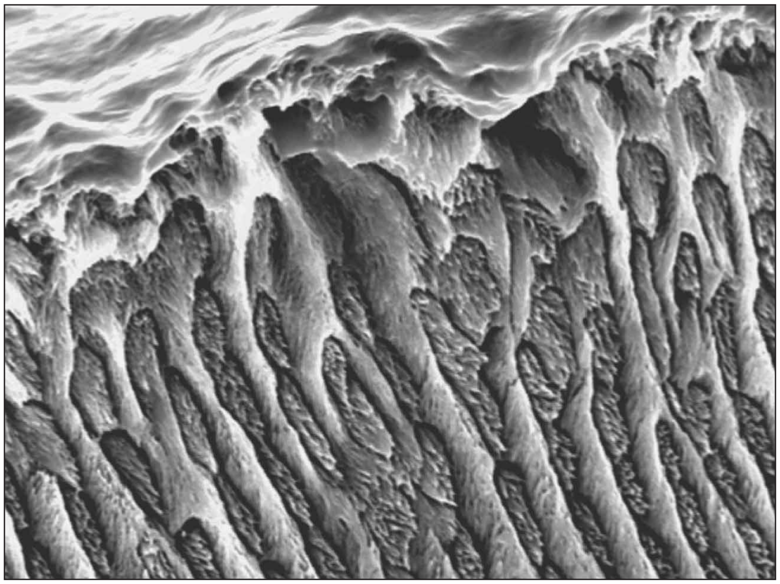

Figure 2. *SEM photograph (1900x) of Control/One Step. Arrows—long and thick resin tags.*

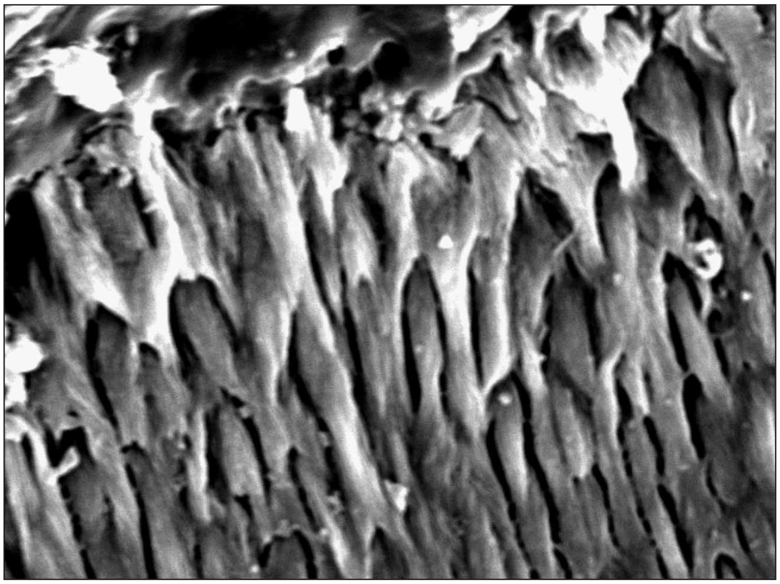

Figure 3. *SEM photomicrograph (1900x) of Control/Single Bond, revealing long, thick and coagulated resin tags.*

Conflicts, and a lack of accurate description of the experiment conditions, were noted among studies in the literature. Barghi and Godwin (1994), who found that use of a dentin adhesive containing acetone without the use of other pretreatment regimens eliminated the adverse effects of office bleaching on the composite-enamel bond, did not mention whether bonding to bleached enamel was carried out immediately post-bleaching. Sung and others (1999) reported the reduction in bond strength to be more pronounced in bleached groups that used acetone-based adhesives, while an ethanol-based adhesive demonstrated no statistical decrease in bond strength between the bleached and unbleached groups. The clinical implication of a study by Sung and others (1999) was that alcohol-based adhesives may result in less compromised bond strength when restorative work is to be completed immediately after bleaching. However, in that study, the bleached specimens were stored in saline, then resin composite bonding was initiated five days post-bleaching. Sung and others (1999) incorrectly referred to this as immediate bonding. Studies that stored specimens in either deionized water or artificial saliva for at least 24 hours after bleaching demonstrated that restored bond strength values were not significantly different from unbleached controls (Titley & others, 1993; Dishman & others, 1994; Josey & others, 1996). It may be that, during the post-bleaching stage before bonding, the storage medium acted to dilute the effect of residual oxygen from the bleaching agent (Sung & others, 1999). The formation of oxygen comes from residual hydrogen peroxide; therefore, one source of discrepancy between past studies and this study may be due to the duration of the post-bleaching period before bonding and the presence or absence of a substance that could remove or neutralize oxygen that affects polymerization.

The results of this study were consistent with those of Lai and others (2002), who reported no difference between the effects of acetone-based and ethanol-based adhesive. Lai and others also found that, for both adhesives, bond strengths were reduced significantly when bonded to carbamide peroxide-bleached enamel.

The cause of the greater adverse effect by office bleaching on bonding reported in the current study could be attributed to the potent 38% hydrogen peroxide concentration, while the 10% carbamide peroxide solution has a 3.0% to 3.5% equivalent concentration of hydrogen peroxide, ammonia and carbonic acid when it decomposes intraorally (Crim, 1992a,b; Rodrigues & others, 2001). These systems have been reported to be less acidic, because of the presence of ammonia and carbon dioxide, both of which are products of the urea breakdown cycle (Croll & Cavanaugh, 1986; Bishara, Denehy & Goepferd, 1987; Chen & others, 1989; Haywood & others, 1990).

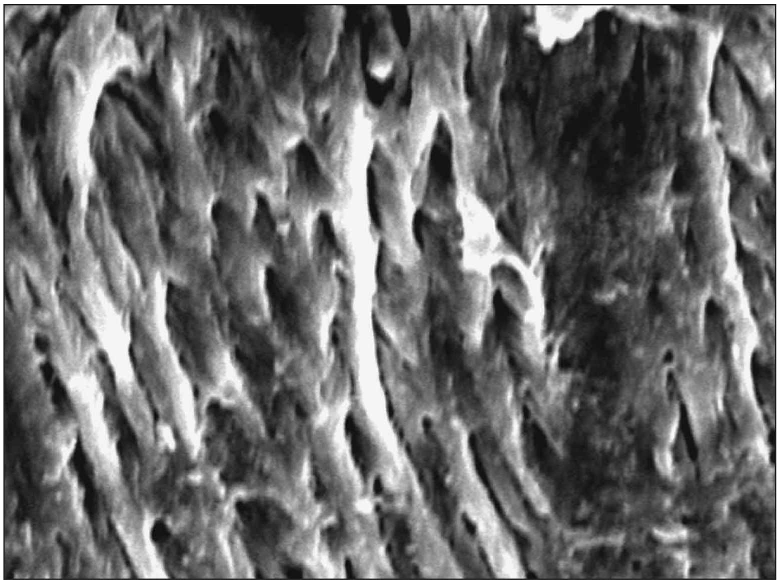

Figure 4. *SEM photomicrograph (1900x) of 10% Carbamide Peroxide/One Step. Arrow—area devoid of resin tags.*

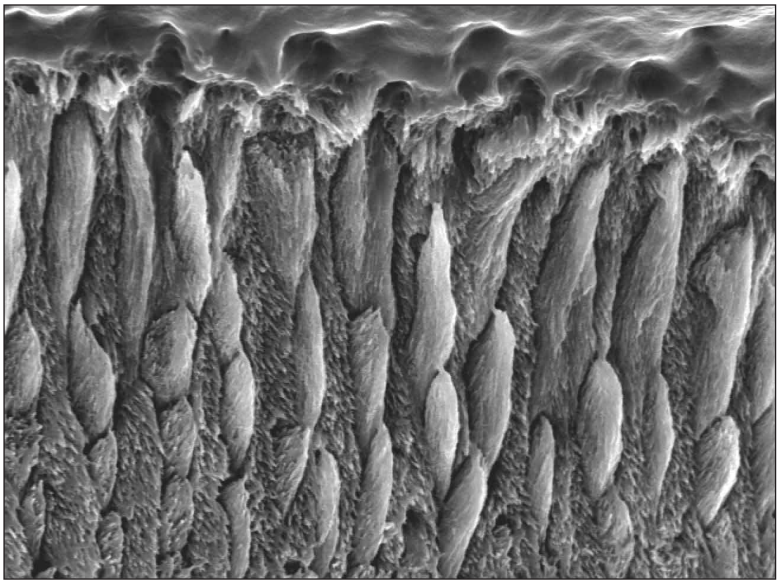

Figure 5. *SEM photomicrograph (1900x) of 10% Carbamide Peroxide/Single Bond. Arrows—short and few resin tags.*

As reported in the literature, bleaching agents may cause alterations to the chemistry and morphology of hard dental tissues. Previous studies suggested that the subsurface enamel organic matrix is altered by the oxidizing effect of peroxide radicals in hydrogen peroxide and by urea and peroxide radicals in carbamide peroxide bleaching agents (Seghi & Denry, 1992; Hegedus & others, 1999). These alterations in organic substance might result in changes in the mechanical properties of enamel and dentin, such as fracture toughness (Hegedus & others, 1999). Since fracture toughness is an indicator of the ability of enamel to resist crack propagation, changes in this property would only lead to decreased bond strengths. Likewise, morphological alterations in enamel, resulting in a poor bonding surface due to the loss of mineral content and/or increased porosity and/or alterations in the organic matrix, were proposed as another possible reason for reduced bond strength (Perdigão & others, 1998; Hegedus & others, 1999). Different hydrogen peroxide concentrations and pH levels of the bleaching systems are believed to affect the topography of the enamel surface differently, since loss of minerals can be related to the acidic properties of bleaching agents (Basting, Rodrigues Jr & Serra, 2001). Hegedus and others (1999) found that 30% hydrogen peroxide solution caused more severe changes in enamel surfaces than carbamide peroxide gel. Although carbamide peroxide bleaching produced morphological alterations (McGuckin, Babin & Meyer, 1992; Shannon & others, 1993; Ben-Amar & others, 1995; Josey & others, 1996) that were seen using TEM (Perdigão & others, 1998; Lai & others, 2002), these changes were only slight when a 10-16% concentration was used compared with a 35% concentration (Oltu & Gurgan, 2000). Based on

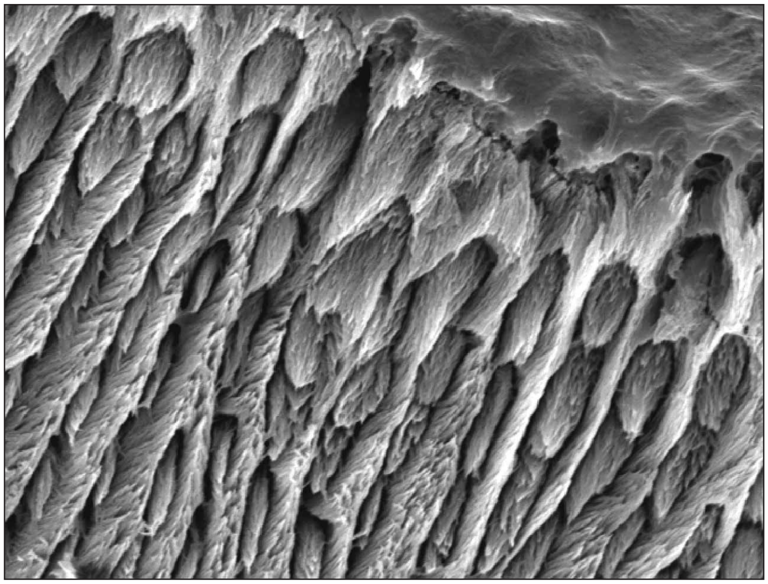

Figure 6. *SEM photomicrograph (1900x) of 38% Hydrogen Peroxide/One Step. Arrows—etch pattern with exposed interrod porosity. Circle—fragmented resin tags.*

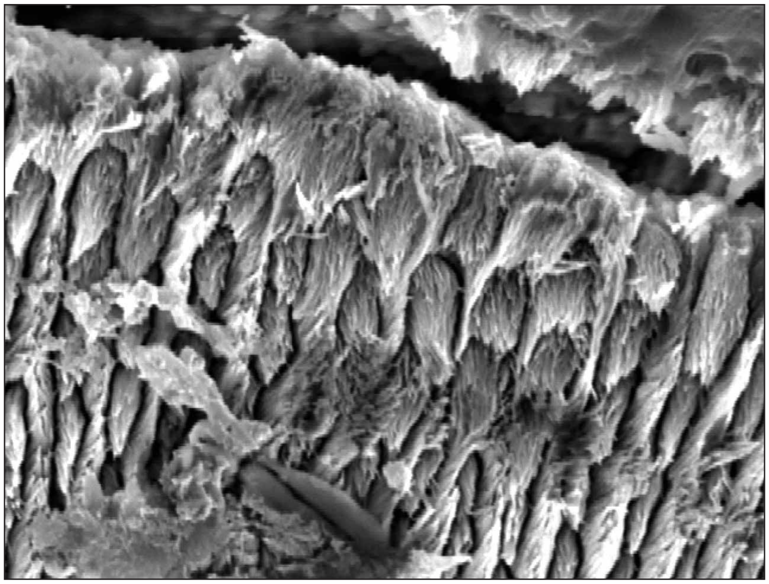

Figure 7. *SEM photomicrograph (1900x) of 38% Hydrogen Peroxide/Single Bond. Circle—fragmented resin tags; arrow—gap in the adhesive resin layer that might be an artifact attributed to the laboratory processing during specimen preparation for SEM.*

these findings and the previous discussion, 38% hydrogen peroxide is expected to have a more deleterious effect on enamel. Although 10% carbamide peroxide had a significantly higher bond strength than 38% hydrogen peroxide, the authors of this study found that carbamide peroxide had a significantly adverse effect on immediate bonding compared to the non-bleached group. This could be explained by prolonged exposure to hydrogen peroxide and, perhaps, a deeper penetration of oxygen.

Failure analysis of the specimens revealed adhesive failure, making it is possible to state that there was no correlation between the failure mode and bond strength values. Since the strongest bonding group (no bleach group) had an adhesive failure (bond strength was less than tooth-enamel strength) and, assuming that the enamel has the same bond strength in all specimens, if the strongest bond was weaker than enamel, then, of course, the bond in the other groups would also be weaker than enamel. Hence, the other groups also exhibited adhesive failure.

SEM studies generated significant information on the factors contributing to the less reliable bonding of adhesive resins to bleached enamel. Specimen preparation by etching the crosscut section of enamel and resin with nitric acid was in accordance with recommendations made in an earlier reported investigation (Glasspoole & others, 2001). The exposure of enamel to a low-concentration inorganic acid defined the structural components of the enamel and highlighted the presence of nonacid-soluble materials that penetrated its surface (Gwinnett & Buonocore, 1965). The resin penetration of enamel is characterized by the presence of polymerized, tag-like extensions of resin in and around the etched enamel prisms. These extensions promote an effective, permanent mechanical attachment, and the quality of the resin tags, their incidence and their depth of penetration influence the bond strength of the resin to the enamel surface (Gwinnett & Matsui, 1967).

This investigation made qualitative observational comparisons of the resin tags present in bleached and unbleached specimens. Differences in the number and depth of the tags occurred within each specimen and within groups of specimens. Therefore, a quantitative evaluation of tag morphology was difficult and beyond the current objective of this study. The authors' intentions were to subjectively compare the typical appearance of tags in bleached enamel with those in unbleached enamel, with the hope that the differences, if any, might help to explain why the bleaching of enamel leads to a marked reduction in bond strength under some circumstances. The information gained from the SEM images from this study was consistent with previous SEM studies (Haywood & others, 1990; Titley & others, 1991; McGuckin & others, 1992; Shannon & others, 1993; Ernst, Marroquin & Willershausen-Zonnchen, 1996; Josey & others, 1996; Lai & others, 2002).

In this study, SEM images of unbleached subgroups revealed numerous long, thick tags, which was consistent with their highly significant bond strengths. On the other hand, resin interfaces with 38% hydrogen peroxide and 10% carbamide peroxide-bleached enamel showed a few short, fragmented resin tags compared to interfaces with unbleached enamel. However, the 10% carbamide peroxide/acetone image depicted a few thin, long resin tags. The same image also showed that areas devoid of resin tags were left in the hybridized area of the enamel, which may have been the result of insufficient penetration of resin into the bleached enamel surface. The concentration of hydrogen peroxide itself may be responsible for some damage to the hydroxyapatite crystallites of the enamel rods, which has been observed in the 38% hydrogen peroxide images (Zalkind & others, 1996). It was also noted that these changes induced in the enamel rod morphology were not related to the type of adhesive resin used. In consideration of these findings, it is reasonable to assume that the suboptimal resin tag formation and the significantly reduced bond strengths of the bleached groups were not affected by the type of adhesive system.

CONCLUSIONS

Under the circumstances of this study, it was found that:

1. Compromised bond strength was not reversed when acetone-based or ethanol-based adhesives were used in the immediate bonding of resin composite to enamel bleached with 10% carbamide peroxide or 38% hydrogen peroxide.
2. For both bleaching systems, the bond strengths were reduced between 43% and 70% when compared to the unbleached subgroups.

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