ORIGINAL ARTICLE

Effect of an antioxidizing agent on the shear bond strength of brackets bonded to bleached human enamel

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Introduction: The purpose of this study was to investigate the effect of antioxidant treatment and delayed bonding on the shear bond strength of metal brackets bonded with composite resin to human enamel after bleaching with carbamide peroxide (CP). **Methods:** Eighty recently extracted premolars were divided into an experimental group (n = 60), which was bleached with 10% CP, and a control group (n = 20), which was not bleached. The experimental group was further divided into 3 groups. Specimens in group 1 (n = 20) were bonded immediately after bleaching; specimens in group 2 (n = 20) were bleached, then treated with 10% sodium ascorbate, an antioxidant agent, and then bonded; group 3 specimens (n = 20) were bleached, then immersed in artificial saliva and held for 1 week before bonding. The specimens were debonded, and the enamel surfaces and bracket bases were examined with a stereomicroscope. The adhesive remnant index was used to assess the amount of resin left on the enamel surfaces after debonding. The shear bond strength data were subjected to 1-way analysis of variance. Multiple comparisons were performed with the Bonferroni test. The level of significance was established at $P < .05$ for all statistical tests. **Results:** Shear bond strength of brackets bonded immediately after bleaching with 10% CP was significantly lower than that of brackets bonded to unbleached enamel ($P < .05$). No statistically significant differences in shear bond strength were noted when the antioxidant-treated and delayed bonding groups were compared with the control group ($P > .05$). **Conclusions:** Bleaching with 10% CP immediately before bonding reduces the bond strength of composite resin to enamel. Treating the bleached enamel surface with 10% sodium ascorbate or waiting 1 week reverses the reduction. (Am J Orthod Dentofacial Orthop 2006;129:266-72)

In recent years, new materials and techniques have been introduced that offer an impressive level of predictable and successful cosmetic dental results. In 1989, Haywood and Heymann[1] published an article on patient-applied, at-home bleaching by using carbamide peroxide (CP). Since then, manufacturers have introduced numerous other at-home bleaching systems. Many of these systems use 10%, 15% to16%, or 20% to 22% CP as the active bleaching agent.[2,3]

Vital tooth bleaching has become increasingly popular, even with adolescent patients. With heightened consumer interest in whiter teeth, clinicians must become familiar with bleaching products to provide optimal solutions and treatment.

Bleaching or whitening products can be used before or after orthodontic treatment. Hintz et al[4] reported a significant clinical color difference between enamel surfaces subjected to orthodontic bonding and debonding compared with control sites after whitening with 10% CP in vitro. They recommended at least 2 to 4 weeks of continuous whitening to overcome the difference. On the other hand, once patients have their teeth bleached, they often become interested in other esthetic dental or orthodontic procedures.[5] However, studies have shown that the bond strength of adhesive restorations and resin-bonded brackets is reduced when the tooth has been bleached with an in-office or at-home technique.[6-12]

Several methods have been proposed to avoid clinical problems related to compromised bond strength after bleaching, such as removal of superficial layer of enamel,[13] pretreatment of bleached enamel with alcohol,[14] and use of adhesives containing organic solvents.[15,16] However, the most common recommendation is to delay bonding after bleaching, because the reduction of composite-resin bond strength to freshly bleached enamel has been shown to be transient.[10,17,18] The recommended postbleaching period for bonding procedures varies from 24 hours to 4 weeks.[10,19-23] A study by Lai et al[24] has shown that reduction in bond

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strength of composite resin to dentin that occurs after bleaching with hydrogen peroxide or sodium hypochloride is reversed when the antioxidant sodium ascorbate is applied to the tooth surface before bonding. In another study, Lai et al[12] found, using extracted human third molars, that, when sodium ascorbate was applied to enamel after bleaching and before composite restoration, shear bond strength of the composite returned to the control level.

If antioxidant treatment of bleached enamel before bracket bonding reverses the reduction in bond strength of composite resin, it might be an alternative to waiting and could eliminate the need to postpone bonding. The purpose of this study was to compare the effect of antioxidant treatment and delayed bonding after bleaching with CP on the shear bond strength of metal brackets bonded with composite resin to human enamel.

MATERIAL AND METHODS

Eighty recently extracted, sound, human premolars (maxillary and mandibular) were collected and placed in a solution of 0.1% thymol. The criteria for tooth selection were intact buccal enamel; no pretreatment of chemical agents such as derivatives of peroxide, acid, or alcohol; no cracks from forceps; no caries; and no restorations. Before the experiment, the teeth were debrided, washed in tap water, and embedded in standardized, $15 \times 18 \times 29$ mm^3 polyethylene molds containing self-curing resin with the crowns exposed; they were then stored in distilled water at 4°C until required. Just before bleaching, the enamel surfaces were polished with oil- and fluoride-free fine pumice and water by using a brush and a slow-speed handpiece, rinsed again, and dried with an air syringe. Subsequently, the procedure specified for each experimental group was followed. The specimens were randomly divided into a control group (n = 20) and 3 experimental groups that were bleached with 10% CP (n = 20 each). Group 1 consisted of specimens bonded immediately after bleaching. Group 2 specimens were bleached and then treated with an antioxidant (10% sodium ascorbate) just before bonding. Specimens in group 3 were bleached and then immersed and held in artificial saliva for 1 week before bonding. Specimens in the control group were not bleached but were immersed and held in artificial saliva for 1 week before bonding (Table I).

In the 3 experimental groups, a commercial, 10% CP at-home bleaching gel (Rembrandt Xtra-Comfort; Den-Mat, Santa Maria, Calif) was applied on the enamel surfaces of the embedded teeth for 8 hours a day, according to the manufacturer's instructions. The specimens were partially immersed in artificial saliva at 37°C in a glass laboratory beaker so that the enamel surfaces coated with bleaching gel did not contact the saliva. After the daily bleaching procedure, the specimens were thoroughly rinsed with water and compressed air for 30 seconds and air-dried. For the rest of the day, they were stored in 250 mL of artificial saliva. The procedure was continued for 1 week. No further procedures were done for group 1.

Table I. Treatment regimens before bonding

Group	*Treatment regimen*
Control	1 week-immersion in artificial saliva
Group 1	Bleaching with 10% CP
Group 2	Bleaching with 10% CP, then 10% sodium ascorbate treatment
Group 3	Bleaching with 10% CP, then 1 week immersion in artificial saliva

In group 2, after the bleaching, an antioxidant, 10 mL of 10% sodium ascorbate, was applied to the enamel surfaces as an irrigating solution for 10 minutes with a flow rate of 1 mL per minute under continuous agitation. The enamel surfaces were then thoroughly rinsed with distilled water for 30 seconds; later the brackets were bonded.

The specimens in group 3, after bleaching, were immersed in 250 mL of artificial saliva at 37°C and held for 1 week. The artificial saliva, with an electrolyte composition similar to that of human saliva, was prepared from 1 g sodium carboxymethylcellulose, 4.3 g xylitol, 0.1 g potassium chloride, 5 mg calcium chloride, 40 mg potassium phosphate, 1 mg potassium thiocyanate, and 100 g distilled, deionized water. The artificial saliva was changed twice daily during the 1-week period. After the specimens were removed from the artificial saliva, the enamel surfaces were rinsed with an air-water syringe for 30 seconds.

The brackets used in this study were .018-in, stainless steel, mesh based, standard edgewise, premolar brackets (Forestadent, Pforzheim, Germany). The brackets were bonded with Concise (3M Unitek, Monrovia, Calif), a chemically cured bis-GMA (bisphenol glycidyl methacrylate) composite resin. The Concise components were applied according to the manufacturer's instructions at 24°C room temperature by the same operator (H.B.). The following steps were carried out for each tooth. One drop of liquid etchant was applied to the labial enamel surface and allowed to remain for 30 seconds. The etching liquid and any remaining demineralized tooth particles were removed with an air-water syringe, applied for 10 seconds. The teeth were then dried for 10 seconds with oil-free compressed air. Equal amounts (1 drop) of enamel bond resin A and B

Fig 1. Custom shearing rod (Bulut-type shearing blade) of testing apparatus. Contour of edge (*arrows*) matches contour of bracket base (see Fig 2).

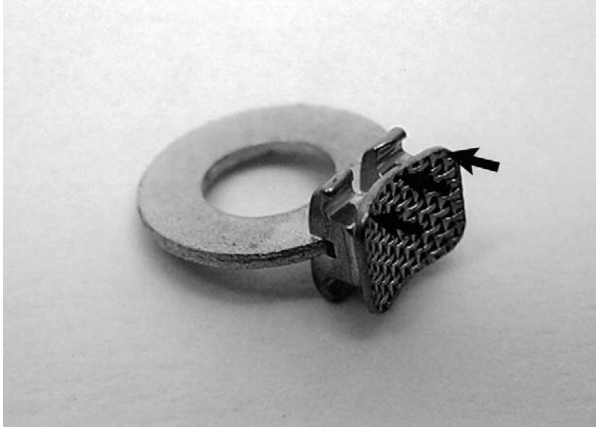

Fig 2. O-ring soldered into slot of bracket to prevent deformation during loading.

were mixed thoroughly (5 seconds) and applied to the etched surfaces in a thin coat. Immediately after that, the bonding paste was prepared by mixing equal portions of pastes A and B for 15 seconds and placed on the bracket base so that the base was fully covered. Then the brackets were seated and positioned firmly in the middle third of the labial enamel surface. Excess resin was removed with a scaler. After bonding, the specimens in all groups were stored in distilled water at 37°C for 24 hours and then subjected to 200 thermal cycles in 2 thermally controlled baths of streaming tap water maintained at 5°C and 55°C, respectively, with a dwell time of 30 seconds in each temperature.

The shear bond strength was measured with an LR 5K universal testing machine (Lloyd Instruments, Hampshire, United Kingdom). To minimize misalignment of the testing apparatus, a custom, knife-edge shearing rod was used (Fig 1). The specimens were secured and precisely aligned toward the shearing blade in the desired position by using the movable platform of a vise in order to obtain the nearest load location to enamel. The long axis of the bracket was positioned parallel to the plunger of the testing machine. To prevent deformation of bracket base and wings during loading, .018-in, metal O-rings were previously soldered into the bracket slots (Fig 2). The crosshead speed was set at 1 mm per minute, and the direction of the plunger was gingivo-occlusal. The load at failure was monitored and continuously recorded by a Dapmat (V2.31, 40/0224, Lloyd Instruments, Hampshire, United Kingdom) software system. Because of uncontrolled variables, the data of applied load to specimens were standardized by dividing the force-to-failure value by the area of the bracket base and expressed in megapascals (MPa) (bracket base dimensions were provided by Forestadent).

Fracture analysis of the bonded enamel surface was done with a stereo microscope (Olympus Co, Tokyo, Japan) at × 16 magnification. In addition, selected fractured specimens were examined under a scanning electron microscope (SEM) ([Jeol/JLM-5200, Tokyo, Japan) at between × 10 and × 1000 magnification to show the characteristics of the debonded surfaces. Failures were classified according to the adhesive remnant index (ARI) (Table II).

According to the Kolmogorov-Smirnov test of normality, the shear bond strength data of the groups were normally distributed ($P > .05$). They were subjected to 1-way analysis of variance. Pair-wise comparisons of the groups were made with the Bonferroni test. The level of significance was established at $P < .05$ for all statistical tests. Statistical analysis of ARI scores was carried out with the Kruskal-Wallis test and Mann-Whitney U test with the Bonferroni correction. The predetermined level of significance ($P < .05$) was reset at $P < .0083$ after the Bonferroni correction. Statistical analyses were processed with the SPSS 10.0 software system (SPSS, Chicago, Ill).

RESULTS

Shear bond strengths in MPa (mean ± SD) for the groups are shown in Table III. One-way analysis of variance showed significant differences in bond strength among the 4 groups ($P < .05$). A Bonferroni test (Table III) showed that shear bond strength of brackets bonded immediately after bleaching with 10% CP was signifi-

Table II. Location of adhesive layer after bracket failure

Condition	n	ARI score*				Enamel fractures
		0	1	2	3	
Not bleached (control)	20	0	3	13	4	0
Bleached (group 1)	20	11	9	0	0	0
Bleached + 10% sodium ascorbate treatment (group 2)	20	0	7	13	0	0
Bleached + artificial saliva immersion (group 3)	20	0	5	15	0	0

ARI scores: 0 = no adhesive left on tooth surface; failure between adhesive and enamel; *1* = less than half of adhesive left on tooth surface; *2* = half or more adhesive left on tooth; *3* = all adhesive left on tooth surface; failure between adhesive and bracket base.
*Observations of 2 examiners.

Table III. Shear bond strengths (MPa)

Group	N	Mean*	SD	Min	Max
Control	20	20.6[a]	2.9	17.0	26.1
1	20	14.2[b]	2.4	9.6	18.0
2	20	19.9[a]	2.8	16.8	26.9
3	20	19.7[a]	2.7	16.3	26.1

*Values with same letter are not significantly different at $P < .05$.

cantly lower than that of unbleached enamel ($P < .05$). For the bleaching groups, when the antioxidant-treated and saliva-immersed (delayed bonding) groups were compared with the control group, there was no statistically significant difference with respect to shear bond strength ($P > .05$). This showed that both antioxidant treatment and artificial saliva immersion were significantly effective in increasing the shear bond strength of brackets bonded to bleached enamel. For the 2 bleaching groups, when the antioxidant-treated and saliva-immersed groups were compared, no statistically significant difference was observed ($P > .05$).

To assess the amount of resin left on the enamel surfaces after debonding, the ARI was used. The Kruskal-Wallis test of fracture analysis data showed that there were statistically significant differences among the 4 groups ($P < .05$). Pair-wise comparison of groups with the Mann-Whitney U test demonstrated that group 1 was significantly different from the other 3 groups ($P < .0083$), whereas the other groups showed no difference when compared with each other.

In all groups, no damage of enamel or specimens was observed after debonding. SEM analysis of fractured specimens in the shearing test showed that, in group 1, adhesive resin on the bleached enamel surface appeared to be granular and porous (Fig 3), but, in the other groups, it appeared more uniform and pore-free (Fig 4).

DISCUSSION

In this study, enamel surfaces were treated chemically, aiming to break the adhesion at the composite-enamel interface. Therefore, specimens were subjected to a shear-peel test. Shear stresses can be applied to brackets by means of either a loop[25-28] or a steel rod with a flattened end.[29-35] The loading method can influence relative strength measurements.[36]According to Katona,[37] stresses tend to be highest near the location of force application. Consequently, to prevent excessive distancing of the test apparatus (point of force application) from the specimen (which can alter the magnitude of resin layer stress), a special, knife-edge shearing rod (Bulut-type shearing blade; Fig 1) was developed for this study and applied to the specimens unconventionally in the gingivo-occlusal direction. Via this method, a parallel application of the shearing force at closest range to the tooth surface was provided (contoured shearing blade, contoured bracket base, and contoured enamel surface were as parallel as possible to the plunger), and no damage to either specimens or enamel occurred.

The high incidence of enamel fracture usually observed in shear tests[38,39] and other debonding procedures[40-43] was not observed in this study. Although we used an approved orthodontic adhesive, no damage to the enamel or the specimens was observed in any group. The specially constructed shearing blade, the gingivo-occlusal direction of the testing apparatus plunger, and the low crosshead speed appeared to be appropriate for effective shear-peel tests of brackets, if a shearing blade can be created for the bracket used.

There have been a number of reports on the interaction between bleaching agent and bond strength of composite resin to enamel. Several authors have reported a significant decrease in the bond strength of composite resin to CP-bleached enamel when compared with unbleached enamel.[7,8,10,11,13,16,19,44] The results of our study demonstrated that the reduction in shear bond strength of brackets immediately after bleaching was significant when compared with the control group. These results agree with those of Cavalli et al,[22] who showed that CP bleaching agents in the range of 10% to 20% adversely

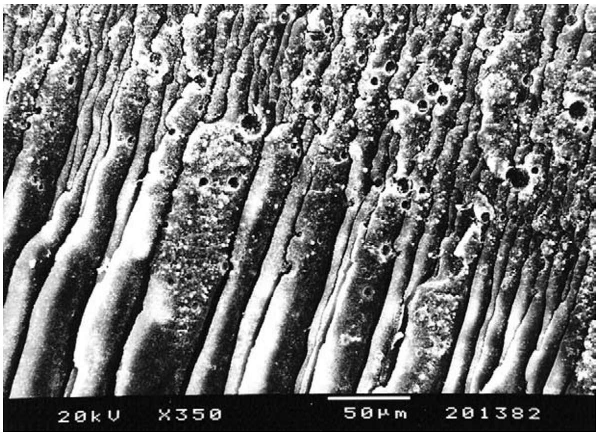

Fig 3. Appearance of adhesive resin on enamel surface of debonded specimen from group 1 (immediate bonding). Note bubble-like formation in resin layer (original magnification × 350).

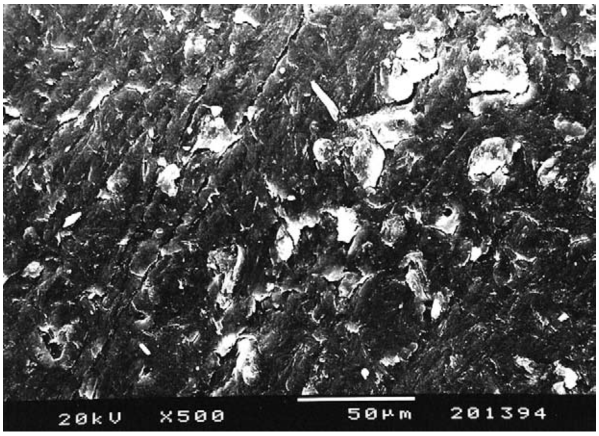

Fig 4. Appearance of adhesive resin on enamel surface of debonded specimen from control group (original magnification × 500).

affect the bond strength of composite resin to enamel when bonding is performed immediately after bleaching. Turkun and Kaya[45]investigated the effect of different concentrations (10%, 16%, and 20%) of CP on shear bond strength of resin composite to bleached bovine enamel. They showed that all 3 concentrations of CP reduced the shear bond strength, but higher concentrations produced greater reductions.

Studies of physical alterations after bleaching have been done to explain the decrease in enamel bond strength caused by CP. Several authors have suggested increased porosity of the enamel as manifested by an overetched appearance with loss of prismatic structure.[11,44] In addition, loss of calcium, decrease in microhardness, and alterations in the organic substance might also be factors in the decrease of enamel bond strengths.[46,47] It has also been proposed that residual oxygen from the bleaching agent inhibits resin polymerization.[6,15,19,20] Titley et al[6] reported that, under SEM, interfaces between resin and bleached enamel are substantially different from those formed between resin and unbleached enamel. In the bleached specimens, large areas of the enamel surface were free of resin, and, when tags were present, they were fragmented and poorly defined, and they had penetrated to a lesser depth than in the unbleached controls. Dishman et al[20] also indicated that the quality of a composite bond is compromised by a smaller number of resin tags. SEM observations suggested an association between a high concentration of voids in the bond area and low mean bond strengths.[17] In another study by Titley et al,[19] SEM examinations of interfaces between resin and bleached enamel displayed a granular and porous aspect with a bubbly appearance. It has been suggested that these might be due to gaseous bubbling, possibly the result of oxidization of peroxide trapped in the subsurface layer of the enamel. This suggestion is corroborated by the study of Turkun and Kaya.[45] We share the opinion that the bubbly appearance of the resin might be caused by residual oxygen in the enamel structure.

Our results support previous studies showing that immersion of in vitro specimens in distilled water or artificial saliva resulted in a complete reversal of the reduced enamel bond strength.[10,18,19,22,44] This might be due to residual oxygen from the bleaching material being removed by the immersion process. It has been noted that, under clinical conditions, saliva might have a similar action after bleaching. However, the amount of time required to restore bond strength to the prebleaching level is controversial. Although there is considerable variation in the recommended postbleaching time, most researchers have advised a delay in bonding of 1 week,[10,17,18,48] in agreement with our own study.

Emphasis was placed on neutralizing the oxygen by application of an antioxidating agent. Ascorbic acid and its salts are well-known antioxidants that can reduce various oxidative compounds, especially free radicals.[49,50] Previous studies have demonstrated the protective effect of ascorbic acid in vivo against hydrogen peroxide–induced damage in biologic systems.[51,52] In this study, treatment of the bleached enamel with 10% sodium ascorbate before bonding appeared to restore the reduced shear bond strength of metal brackets bonded with composite resin to enamel for a concentration of 10% CP. These results concur with those of recent studies,[12,45,53] which have shown that reduced bond strength of composite resin to bleached enamel was effectively reversed by antioxidant treatment. In the study by Lai et al,[12] bleached specimens were immersed in 10% sodium ascorbate solution for 3 hours. Nevertheless, in 2 more recent studies,[45,53] the duration of antioxidant treatment was 10 minutes. We used this application time for the antioxidant because it is reasonable in clinical conditions. During the 10-minute treatment period, sodium ascorbate solution was continuously refreshed, and the enamel surface was agitated with a sterile brush.

Zhao et al[54] demonstrated that under certain conditions, peroxide ions take the place of the hydroxyl radicals in the apatite lattice and produce peroxide-apatite. They also showed that when peroxide ions decompose, substituted hydroxyl radicals reenter the apatite lattice, resulting in elimination of the structural aberrations caused by incorporation of the peroxide ions. Lai et al[12] hypothesized that the incorporation process of peroxide ions might also be reversed by an antioxidant. Furthermore, they suggested that sodium ascorbate allows free-radical polymerization of the adhesive resin to proceed without premature termination by restoring the altered redox potential of the oxidized bonding substrate and hence reverses the compromised bonding.[24]

Because of poor reports in the literature, further investigation into developing a factory-made antioxidant agent is warranted in this area.

CONCLUSIONS

1. Bleaching with 10% CP immediately before bonding reduces the bond strength of composite resin to enamel.
2. Delaying bonding for 1 week after bleaching reverses the reduced bond strength.
3. Treating the bleached enamel surface with 10% sodium ascorbate also reverses the reduced bond strength and could be an alternative to delayed bonding, especially when orthodontic treatment must be started immediately after bleaching.

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