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## Effect of postbleaching application of an antioxidant on enamel bond strength of three different adhesives

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### Abstract

**Objectives:** The aim of this study was to compare the shear bond strength (sbs) of three different adhesives on bleached enamel immediately after bleaching, bleached/delayed for 1 week, and bleached/applied antioxidizing agent.

**Study Design:** The enamel surfaces of 144 freshly extracted incisors without any caries and restorations were flattened and divided into 12 groups. The following adhesives were investigated: Optibond FL (OFL) (three-step etch&rinse), Optibond Solo Plus (OSP) (two-step etch&rinse), Optibond All-in-One (OA) (one-step self-etch), (Kerr, Orange, USA). Unbleached enamel groups were prepared as negative controls. The remainder surfaces were bleached with 20% Opalescent PF (Ultradent, USA) 6 h/d for 5 consecutive days. Specimens were bonded immediately after bleaching, after 1 week or after using 10% sodium ascorbate gel for 6 hours. After 500 rounds of thermocycling, sbs was measured and data was analyzed with Kruskal-Wallis and Mann-Whitney U-tests ( $\alpha = 0.05$ ).

**Results:** The sbs decreased for the adhesives after bleaching except for OFL. The effect of applying sodium ascorbate subsequent to bleaching was not equal for the studied adhesives. While for OFL, sbs of the sodium ascorbate group was significantly higher than the unbleached control group, for OSP, the sodium ascorbate group had no statistically significant difference with the unbleached control group and for OA, sbs was significantly lower than the unbleached control group.

**Conclusions:** Different adhesives demonstrate different degrees of reversed bond strength subsequent to applying antioxidant. It seems the method of application and the chemical composition of the adhesives could affect the antioxidant as a reducing agent.

**Key words:** *Antioxidant, bleaching, bond strength, enamel, etch-and-rinse adhesives, self-etch adhesives.*

## Introduction

The esthetics of anterior teeth is an important consideration in modern dentistry. Introduction of vital home bleaching materials in 1989 opened up new horizons in esthetic dentistry. The capacity of such materials to reverse tooth discolorations has been confirmed (1,2). Tooth color is dependent on the intrinsic tooth color and extrinsic stains. The intrinsic tooth color is a function of reflection or absorption of light by tooth structure (1). Bleaching is carried out by extra-coronal technique for vital and non-vital teeth, and intra-coronal and sometimes intra-/extra-coronal for non-vital teeth (3). Usually hydrogen peroxide, carbamide peroxide, and sodium perborate are used for bleaching (2,4).

Although bleaching agents clearly result in a lighter color in teeth, they have complications, too: pulp sensitivity, possible changes in tooth structure, microleakage of restorations, external root resorption and decreased bond strength of resins (5). Decreased bond strength in cases in which resin bonding agents are used is of utmost importance: porcelain veneers, composite veneers, and composite restorations (6). Different reasons have been reported for such a decrease in bond strength, including changes in enamel surface roughness and increase in enamel porosity (6); residual oxygen which inhibits resin polymerization through free radical mechanism;(1) decrease in microhardness (5); changes in the mechanical properties of enamel and dentin, including fracture toughness, which is a factor involved in resisting crack formation and changes in it lead to decreased bond strength (7).

Several techniques have been suggested to compensate decreased bond strength subsequent to bleaching, including delayed bonding or a waiting period of 24 hours up to 3 weeks, which is the most common and easiest procedure; removal of surface enamel; use of acetone-containing adhesives; use of alcohol on tooth surfaces; and use of antioxidants such as gel or solution forms of sodium ascorbate (8,9).

Some studies have reported that the decrease in bond strength subsequent to bleaching in alcohol-based adhesives is less than that in acetone-based ones (10-12); some other studies have reported the opposite (13,14). Two studies have not reported any differences between these two adhesives (15,16). Some studies have compared bond quality between etch-and-rinse and self-etch adhesives subsequent to bleaching (12,16-19). The results of all these studies and more recent studies indicate that bond strength and marginal integrity compromise subsequent to bleaching is seen in all the etch-and-rinse or self-etching systems (16-23) and the higher the concentration and longer the application time, the greater the bond strength decrease (21). A delay in bonding procedure reverses the bond strength decrease (9,16,24,25).

In recent years, sodium ascorbate has been used to compensate bond strength compromise subsequent to bleaching with etch-and-rinse adhesives in some studies (8,24,25) and with self-etching adhesives in some others (20,26). In studies in which sodium ascorbate as an antioxidant have been used, bond strength has increased to the level of that in the control groups (24,25) or even higher than that (20). Sodium ascorbate can be used as a hydrogel (20) or solution (24) on tooth structure. The application time has been reported from the ten minutes (25,26) up to the 1/3 of application time of the bleaching gel (24). It has recently been reported that the use of sodium ascorbate for five minutes is sufficient for the interaction between the antioxidant and the bleaching agent (27).

Although some authors have advocated the use of etch-and-rinse adhesives in comparison to self-etching ones subsequent to bleaching (17,18,21), no studies have compared the use of an antioxidant. Therefore, the aim of the present study was to evaluate the potential of sodium ascorbate as an antioxidant to compensate bond strength decrease with the application of etch-and-rinse and self-etching adhesive systems compared to delayed bonding subsequent to bleaching.

## Materials and Methods

The present study was carried out on 144 maxillary incisors which had been extracted at most six months prior to the study and had been preserved in normal saline solution at 4°C. The teeth had no restorations, caries and cracks. One week before the experiment all tooth surfaces were cleaned of any debris and soft tissues and immersed in 0.2% thymol solution; 24 hours before the experiment the teeth were retrieved from thymol, rinsed and kept in distilled water.

In all the specimens the crown was separated from the root with a diamond bur under water spray. Then the crown was mounted in cold-curing acrylic resin horizontally in a manner in which the labial surface of the tooth was level with the acryl and the horizon as much as possible. The specimens were immersed in cold water during acryl polymerization to avoid the detrimental effect of heat on the teeth. The specimens were randomly divided into 12 groups of 12 teeth each. The groups were treated as follows:

In groups 1-3 (Negative Controls = NC) Z100 composite resin was bonded to enamel surfaces with the adhesives OptiBond FL (three-step etch-and-rinse), OptiBond Solo Plus (two-step etch-and-rinse), and OptiBond All-in-One (one-step self-etch) according to manufacturer's instructions (Kerr) (Table 1).

In groups 4-12, 20% carbamide peroxide gel (Opalescent 20% PF, Ultradent, South Jordan, UT, USA) was placed in direct contact with the prepared enamel surfaces. To this end, plastic rings were prepared and fixed

**Table 1.** Materials used in the study and mode of their applications according to the manufacturers' instructions.

Materials compositions	Manufacturers' Directions	Material name & manufacturer
Etchant: 37.5% H <sub>3</sub> PO <sub>4</sub> FL Primer: HEMA, GPDM, MMEP, Water, ethanol, CQ, BHT FL Adhesive: Bis-GMA, HEMA, GDMA, CQ, ODMAB, filler (fumed SiO <sub>2</sub> , barium aluminoborosilicat, Na <sub>2</sub> SiF <sub>6</sub> ) coupling factor A174 (48 wt% filled).	1. Etch with 37.5% phosphoric acid (15 seconds), 2. Rinse for (15 seconds) and dry (5 seconds), 3. Apply primer and rub for 15 seconds. Dry for 5 seconds, 4. Apply adhesive in a uniform thin layer, 5. Light cure for 30 seconds.	Optibond FL (Three step, etch & rinse adhesive) (Kerr, Orange, CA, USA)
Etchant: 37.5% H <sub>3</sub> PO <sub>4</sub> Adhesive : Bis-GMA, HEMA, GDMA, GPDM, ethanol, CQ, ODMAB, BHT, filler (fumed SiO <sub>2</sub> , barium aluminoborosilicate, Na <sub>2</sub> SiF <sub>6</sub> ), Coupling factor A174 (15 wt% filled).	1. Etch with 37.5% phosphoric acid (15 seconds), 2. Rinse (15 seconds) and dry (5 seconds), 3. Apply the adhesive and rub for 15 seconds. Dry for 3 seconds, 4. Light cure for 20 seconds.	Optibond Solo Plus (Two-step, etch & rinse adhesive) (Kerr, Orange, CA, USA)
Glycerol phosphate dimethacrylate, mono- and di- functional methacrylate esters, water, acetone, ethanol.	1. Shake the bottle for 10 seconds, 2. Apply the adhesive and rub for 20 seconds, 3. Repeat the procedure, 4. Air dry lightly for 5 seconds, 5. Light cure for 10 seconds.	Optibond All-in-One (one-step, self etch adhesive) (Kerr, Orange, CA, USA)
Bis-GMA, TEGDMA, Zirconium/Silica filler.	Z100 composite resin applied in 2 mm layers. Each layer light cured for 40 seconds.	Z100 Composite resin (3M ESPE)
Carbamid Proxide 20%, Potassium Nitrate, Fluoride (0.11%).	Bleaching gel for at-home bleaching, 6h/d for 5 consecutive days.	Opalescent 20% PF (Ultradent, USA)
Natrium-L(+)-ascorbat (C <sub>6</sub> H <sub>7</sub> NaO <sub>6</sub> M=198.11g/mol).	After preparing 10% hydrogel, the gel was applied on the enamel surfaces for 6 hours and then rinsed.	Sodium ascorbate gel (Appli Chem, Germany)
Abbreviations: Bis-GMA: bisphenol A diglycidyl methacrylate; BHT: butylhydroxytoluene; CQ: camphorquinone; GDMA: glycerol dimethacrylate; GPDM: glycerol phosphate dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; MMEP: mono-2-methacryloyloxyethyl phthalate; ODMAB: 2-(ethylhexyl)-4-(dimethylamino)benzoate; TEGDMA: triethylene glycol dimethacrylate		

on enamel surfaces using cellophane; the rings were 5 mm in diameter and 2 mm in height. Then bleaching gel was injected into the wells produced. After the rings were filled and it was made sure that no bubbles were present, they were covered with the second layer of cellophane to protect the bleaching gel from environmental contaminants and air.

Based on at-home bleaching technique, the bleaching agent was placed in contact with tooth surfaces for 6 hours a day for 5 consecutive days according to manufacturer's instructions. During the bleaching period the specimens were kept in an incubator at 37°C at relative humidity. The bleaching agent was rinsed from tooth surfaces for 1 minute using a water spray

after each bleaching regimen. Then the specimens were stored in relative humidity at 37°C in an incubator.

Immediately after the bleaching procedures, bonding was carried out in the three positive control (PC) groups (groups 4-6) using the three adhesive systems, OptiBond FL (OFL), OptiBond Solo Plus (OSP), and OptiBond All-in-One (OA) (Kerr, Orange, CA, USA) and Z100 composite resin (3M ESPE, USA) according to manufacturers' instructions (Table 1).

The three delayed bonding (DB) groups (groups 7-9) were kept in relative humidity at 37°C for a week after bleaching. Then bonding procedures were carried out similar to what was explained before.

In sodium ascorbate (SA) groups, (groups 10-12), 10% sodium ascorbate gel was placed in the rings similar to the rings described for the bleaching procedure, and the specimens were kept under relative humidity at 37°C for six hours (equivalent to one day of bleaching regimen). Then bonding was carried out in the manner previously described.

For bonding procedure, after the application of the adhesives according to manufacturer's instructions, transparent cylindrical molds were prepared, with a height of 4 mm and a diameter of 3 mm. Z100 composite resin was placed inside the molds in a manner in which it had a convex surface so that air would not be trapped between the tooth surface and composite resin. Then the mold was placed on the tooth and excess composite resin was removed from the periphery and from the cylinder top. Curing was carried out from three directions of top and two sides for 40 seconds from each side (a total of 120 seconds) using a Coltolux light-curing unit (Coltolux 50, Colton/Whaldent, USA) at a light intensity of 480 mW/cm<sup>2</sup>.

All the specimens were kept under relative humidity at 37°C for 24 hours subsequent to the bonding procedure. Then the plastic molds were cut away using a scalpel blade. The specimens then underwent a 500-cycle ther-

mocycling procedure at 5°C/55°C with a dwell time of 20 seconds and a transfer time of 12 seconds.

Finally, the specimens underwent a shearing bond strength test using a universal testing machine (Dartec, HC10, Dartec Ltd, Stourbridge, UK). Each specimen was fixed on the jig in a manner in which the composite cylinder was parallel with the horizon, and the equipment blade was placed adjacent to the composite-tooth interface perpendicular to composite resin at a strain rate of 1 mm/min.

The force was applied until the composite resin was detached from the tooth surface and the force was measured in Newton (N). In order to convert the force to MPa, the value (in Newton) was divided into the composite resin surface area and then the means of shearing bond strengths for each group were calculated. Data was analyzed using Kruskal-Wallis and Mann-Whitney U-tests ( $\alpha = 0.05$ ).

The fractured surface of each specimen was analyzed under a stereomicroscope at  $\times 16$  and the type of failure was determined. Failure types were classified as follows:

- Adhesive: failure at enamel-composite interface.
- Cohesive: failure in the restorative material alone or enamel alone.
- Mixed: a combination of adhesive and cohesive failures.

### Results

Means and standard deviations for the groups under study are summarized in (Table 2). Based on the results of the present study the highest and lowest bond strength values were observed in the sodium ascorbate group with OFL adhesive and in the PC group with OA adhesive, respectively. Except for the NC groups, in which OSP had a slightly higher bond strength compared to OFL, in all the other groups OFL resulted in the highest bond strength. The lowest bond strength in all the groups was related to OA.

**Table 2.** Shear bond strength values (MPa) for the three adhesive systems at four different techniques (Mean  $\pm$  Standard deviation).

Group Adhesive	NC	PC	DB	SA
OFL	20.39 $\pm$ 5.27 <sup>Aa</sup>	17.74 $\pm$ 2.30 <sup>Aa</sup>	20.06 $\pm$ 3.12 <sup>Aa</sup>	25.26 $\pm$ 4.89 <sup>Ba</sup>
OSP	21.01 $\pm$ 2.58 <sup>Aa</sup>	14.54 $\pm$ 1.47 <sup>Bb</sup>	15.98 $\pm$ 1.37 <sup>Cb</sup>	21.81 $\pm$ 5.07 <sup>Aa</sup>
OA	15.79 $\pm$ 1.94 <sup>Ab</sup>	12.57 $\pm$ 1.01 <sup>Bc</sup>	14.70 $\pm$ 2.07 <sup>ACb</sup>	14.15 $\pm$ 1.64 <sup>Cb</sup>

NC: Negative Control, PC: Positive Control, DB: Delayed Bonding, SA: Sodium Ascorbate, OFL: Optibond FL, OSP: Optibond Solo Plus, OA: Optibond All-in-one). Means followed by different letters show statistical differences: ( $\alpha = 0.05$ ). Capital letters: comparison of each procedure for each adhesive (row) Lower case letters: comparison of adhesives at each procedure (column).

**Table 3.** Different failure modes in the study groups. (%)

Method of failure Adhesive Groups	Adhesive			Mixed			Cohesive		
	OFL	OSP	OA	OFL	OSP	OA	OFL	OSP	OA
NC	18%	24%	63%	57%	26%	22%	25%	50%	15%
PC	23%	63%	80%	45%	15%	20%	32%	22%	0%
SA	42%	75%	83%	21%	12%	17%	37%	13%	0%
DB	22%	20%	71%	33%	12%	11%	45%	68%	8%

(NC: Negative Control, PC: Positive Control, DB: Delayed Bonding, SA: Sodium Ascorbate, OFL: Optibond FL, OSP: Optibond Solo Plus, OA: Optibond All-in-one).

Two-by-two comparisons of the groups under study with the relevant P-values also are presented in (Table 2). In the sodium ascorbate groups the differences between OFL and OA, and between OA and OSP were significant; however, the differences between OFL and OSP were not significant.

In comparison of the bond strength of each adhesive in different techniques (Table 2), regarding OFL, the differences between the NC and sodium ascorbate groups, between the PC and sodium ascorbate groups, and between the DB and sodium ascorbate groups, were significant.

In relation to OA, the differences between PC group and the other three groups were significant.

In relation to OSP, the differences between the NC and PC groups, between the PC group and sodium ascorbate and DB groups, and between the sodium ascorbate and DB groups were significant.

The frequency of different failure modes are shown in (Table 3). As the table indicates the highest number of adhesive failures was observed in the OA adhesive. The highest numbers of enamel cohesive failures were observed in etch-and-rinse adhesives.

**Discussion**

The present study provided the opportunity to compare the effect of delayed bonding and the application of sodium ascorbate along with the use of three-step etch-and-rinse, two-step etch-and-rinse, and one-step self-etching adhesives from one manufacturer on the shearing bond strength of composite resin to sound enamel and bleached enamel. According to the results of the present study, the mean of shearing bond strength of composite resin to bleached enamel decreased immediately after bleaching. This decrease was 13%, 30%, and 20% in OFL, OPS and OA adhesives, respectively. Previous studies have reported a 25% and a 35% decrease

in enamel bond strength after bleaching with the use of etch-and-rinse adhesives of Single Bond and Prime & Bond NT (24) and self-etching adhesives Clearfil SE Bond (20), respectively. The decrease has been attributed to the residual oxygen which inhibits the polymerization of resin free radicals (14), changes in the length and number of resin tags (4,6), and changes in the surface topography of enamel (28). Other factors, including the elimination of non-fiber mineral content of tooth structure (29), changes in the calcium, phosphorus, sulfur and potassium content of tooth structure (30), and changes in the mineral and protein content of enamel surface (1), have been mentioned as causes for bond strength decrease after bleaching.

Evaluation of bleached enamel under a light microscope has revealed a darkening under the enamel surface layer, which has been mentioned as evidence of loss of enamel mineral content (28). It has been reported that some changes in the mineral matrix beneath the surface result in changes in the chemical properties of enamel and dentin, including fracture toughness (7); since fracture toughness is a determining factor for the capacity of enamel to resist crack formation, its decrease has been reported as a factor in reduced enamel bond strength (14). The quality, number and penetration depth of resin tags have an influence on the bond strength of resin to enamel (4,28).

Some studies have reported that incorporation of peroxide ions into the apatite plexus to replace hydroxyl ions and production of apatite peroxide is the reason for reduced bond strength. With time and with the catabolism of peroxide ions, the hydroxyl ions are re-incorporated into apatite structure and reverse the structural changes produced by peroxide ions (29).

In the present study, a delay of one week in the bonding procedure resulted in a considerable improvement in bond strength of adhesives in comparison to the PC

group. This difference between the NC and DB groups was significant in OA and OSP adhesives but not significant in OFL, i.e. a delay of one week was 100% significant for OFL to achieve proper bond strength. Reduced bond strength for OSP in delayed bonding was more than that in other adhesives, i.e. a delay of one week could only compensate 6% of the 30% bond strength decrease for this adhesive.

The chemical compositions of OFL and OSP are very similar. It seems the only important difference in these etch-and-rinse systems is in their pH values and their application methods. The pH of OFL is 1.8 and OSP has a pH of 2.1. It seems the higher acidic pH in the former and the application of separate etch, prime, and bonding steps in this three-step total-etch system, which has been introduced as gold standard on enamel in some previous studies (30), confirm the results of the present study. In addition, a pH of 2.5 for OA justifies the lower bond strength values of this adhesive to enamel.

The application of sodium ascorbate increased bond strength of OFL up to 33% compared to the PC group, which was even higher than that in the NC group. Recently, a similar study on Clearfil SE Bond adhesive reported a 35% increase in bond strength under similar conditions (20). Bond strength increase up to 25-35% higher than the initial bond strength subsequent to bleaching and the application of an antioxidant in the present study and the above-mentioned study (20) is a matter for reflection.

In relation to OSP, the differences between the sodium ascorbate groups and the NC group were not significant and the bond strength in the sodium ascorbate group was significantly higher than that in the PC group. In fact, regarding the two etch-and-rinse adhesives, application of sodium ascorbate restored bond strength to the initial strength but this result was weak in relation to one-step self-etching adhesive.

In the OA group, although the bond strength was significantly higher than that in the PC group after sodium ascorbate application, its difference with the unbleached group was significant, i.e. the effect of antioxidant in restoring bond strength of self-etching adhesive to the level of etch-and-rinse adhesives was not significant. The significance of the difference between the NC and sodium ascorbate groups only in relation to OA shows that with the use of this adhesive, sodium ascorbate cannot restore the bond strength to the level of that in the NC group. The results of the present study in relation to etch-and-rinse adhesives are consistent with the results of a study carried out by Lai et al. (24).

Regarding the composition of solvents in the present study, OFL has a solvent of alcohol/water; OSP has a solvent of alcohol; and OA has a solvent of water/acetone/alcohol. In the majority of the groups the best results were related to OFL followed by OSP, and OA

ranked the last. In previous studies, Kalili et al. (10) and Sung et al. (11) suggested alcohol-based adhesives for the reversal of bond strength after bleaching; however, Lai et al. (24) did not report any significant differences in bond strength between alcohol-based and acetone-based adhesives.

In the present study OA, which is a one-step self-etching adhesive, demonstrated a lower bond strength, even in an unbleached state, compared to etch-and-rinse adhesives, which is not consistent with the claims of the manufacturer; however, these claims have been confirmed in other studies (9,30). The differences between this adhesive and other adhesives in other groups were significant. A previous study has reported a significant decrease in bond strength of the self-etching Clearfil SE Bond adhesive to bleached enamel; however, for the Single Bond etch-and-rinse system this decrease was not significant (18). The discrepancies in the results have been attributed to the greater inclination of self-etching primers to be inhibited by carbamide peroxide (18). Self-etching primers have acidic resin monomers which simultaneously etch the enamel surface and penetrate it. Anyway, considering the acidity of OA (pH=2.5) it seems its weaker etching potential compared to phosphoric acid (pH=0.6) results in a weaker bond strength in the unbleached state and subsequent to bleaching procedures.

Although it has been established that simplified adhesives are easy to use and are less technique-sensitive, they have reduced bond strength (9). In self-etching systems, simultaneous etching and priming paves the way for penetration of resin monomers of the adhesive into the etched enamel (19); however, application of self-etching primers alone results in etching of enamel with insufficient depth, which might have resulted from inadequate penetration of the etching primer into enamel microporosities or deposition of calcium on enamel surface, covering the etched surface and interfering with resin penetration (30). One reason can be the oxygen residues which interfere with penetration and polymerization of resin, leading to reduced bond strength (20). However, it seems various bonding agents yield heterogeneous results regarding bond strength to dental tissues immediately after bleaching, in delayed bonding or after application of an antioxidant; therefore, further studies are required to establish proper treatment modalities for clinical applications. Ultimately, within the limitations of the present study it can be concluded that:

1. Bleaching the enamel results in reduced bond strength of composite resin to enamel; however, different adhesives demonstrate varying degrees of reduction. Generally, the use of etch-and-rinse adhesives after bleaching results in a higher bond strength compared to self-etching adhesives.
2. A delay of one week after bleaching was enough

to restore bond strength to the initial strength in OFL and OA adhesives; however, in OSP adhesive the bond strength did not return to the initial value.

3. It appears that in etch-and-rinse adhesives the use of sodium ascorbate is a more appropriate method than delayed bonding; however, in the self-etching adhesive under study there was no difference between the two methods.

4. It seems the chemical composition of some adhesives is more compatible with sodium ascorbate to compensate reduced bond strength after bleaching. More studies are required on various bonding systems.

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