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Effect of antioxidant treatment on bond strength of a luting resin to bleached enamel

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ABSTRACT

Purpose: The aim of this study was to comparatively investigate the effect of antioxidant treatment and delayed bonding after bleaching with carbamide peroxide on the shear bond strength (SBS) of a luting resin to enamel.

Materials and methods: Forty flat enamel surfaces were prepared from freshly extracted human molars using a low speed diamond saw, then divided into three bleaching groups ($n = 10$/group) and a control group ($n = 10$). Group 1 consisted of specimens bonded immediately after bleaching. Group 2 specimens were treated with an antioxidant agent, 10% sodium ascorbate, while Group 3 specimens were immersed in artificial saliva for 1 week after bleaching. Specimens in Group 4 were not bleached, but immersed in artificial saliva for 1 week before bonding. Forty ceramic blocks (Empress 2, Ivoclar) were prepared and luted to teeth using a dual-curing resin cement (Variolink II, Ivoclar). The specimens were thermocycled and the SBS tests were performed using a universal testing machine (crosshead speed: 0.5 mm/min). Fracture analysis of the bonded surfaces was done using a scanning electron microscope. Statistical analysis was carried out by Kruskall–Wallis and Mann–Whitney *U*-tests.

Results: While the samples that were immediately bonded after bleaching (Group I) demonstrated significantly lower shear bond strengths and 10% sodium ascorbate group (Group II) demonstrated significantly higher bond strengths than control group samples ($p < 0.05$), no significant differences were found among delayed bonded group and control group ($p > 0.05$).

Conclusion: Using sodium ascorbate with a concentration of 10% may be reliable for reversing the compromised bond strength.

1. Introduction

Vital tooth bleaching is a safe and well-accepted procedure for the treatment of surface and intrinsic staining of teeth.[1] In-office bleaching agents contain high concentrations of carbamide peroxide (35–37%) or hydrogen peroxide (30–35%), while at-home whiteners consist of low concentrations of both peroxides.[2,3] As vital tooth bleaching has become increasingly popular, clinicians should be aware of the outcome of the bleaching treatment and their interactions with further dental treatments, especially in terms of adhesive restorations. Following bleaching, patients often require

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additional aesthetic interventions such as, replacement of old restorations and application of laminate veneers to restore aesthetic deficiencies. However, a number of studies have shown that the bond strengths of adhesive restorations to tooth structures are reduced when the tooth has been bleached.[4–9] It is reported that this situation is related to the presence of residual peroxide, which interfered with the resin attachment and inhibited the resin polymerization.[10]

Some techniques have been suggested to solve the clinical problems related to the post-bleached compromised bond strength. Cvitko et al.[4] proposed to remove the superficial layer of enamel; Barghi and Godwin[11] treated the bleached enamel with alcohol before restoration, while Kalili et al.[12] and Sung et al.[13] suggested the use of adhesives containing organic solvents. However, the general approach is to postpone any bonding procedure for a while after bleaching since the reduction of bond strength has been shown to be temporary.[14] The waiting period for bonding procedures after bleaching has been reported to vary from 24 h to 4 weeks.[10,15–18] Reduced bonding to the bleached enamel can be reversed with 10% sodium ascorbate solution before resin bonding. A recent study conducted by Lai et al. has shown that hydrogen peroxide or sodium hypochlorite-induced reduction in bond strength of resin to enamel is reversed with the use of sodium ascorbate as an antioxidant.[6] Türkün and Kaya[19] shortened the application period of sodium ascorbate to 10 min and demonstrated that even 10 min application was enough for reversing the reduced bond strength. If the antioxidant treatment of bleached tooth before bonding reverses the reduced bond strength, it may be an alternative to the delayed bonding procedure after bleaching.[6,20,21]

Previous studies on adhesion to bleached tooth focused on restorative composite materials.[4–11] Therefore, the aim of this study was to comparatively investigate the effect of antioxidant treatment and delayed bonding after bleaching with carbamide peroxide on the shear bond strength (SBS) of a luting resin to enamel.

2. Materials and methods

2.1. *Preparation of teeth and ceramic specimens*

Forty intact and non-carious human maxillary and mandibular third molars extracted for orthodontic reasons were collected. All tooth extractions were performed at the Department of Oral Surgery, University of Ege, Izmir, Turkey, having patients signed the appropriate informed consent form approved by the university institutional review board. The teeth were cleaned of any residual tissue tags, pumiced and washed under running tap water. They were then stored in a solution of 0.1% thymol. The roots were removed from the crowns approximately 2 mm below the cemento-enamel junction using a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under copious water spray. The coronal pulps were removed and ultrasonically cleaned in deionized water. The pulp chamber was filled with light body elastomeric impression material (Xantopren VL Plus, Heraus Kulzer Gmbh & Co., KG, Dormagen, Germany). The teeth were then mounted in a plastic holder filled with autopolymerizing acrylic resin (Palapress, Vario, Heraeus Kulzer, Wehrheim, Germany) with the approximal surfaces upward, at the same level of the surface of the embedding medium to form a flat surface. Flat enamel surfaces (4 mm $\times$ 4 mm) were prepared using a water coolant slow speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA).

Forty lithia-based all-ceramic specimens (Empress 2, Ivoclar, Schaan, Liechtenstein) (diameter: 4 mm, height: 4 mm) were prepared according to the lost wax technique recommended by the manufacturer and ultrasonically cleaned for 15 min in ethanol and deionized water and divided into four groups.

2.2. *Experimental groups*

Forty specimens were randomly assigned to four groups ($n = 10$/subgroup). In each main group, the specimens were subjected to three different surface conditioning procedures: Group 1 consisted of ceramic specimens bonded to enamel surfaces immediately after bleaching. In Group 2, following bleaching, sodium ascorbate was applied to the substrate surfaces of the embedded teeth as an irrigation solution and ceramic specimens were bonded. Group 3 specimens were immersed in artificial saliva at 37 °C for 1 week after bleaching then bonded. Specimens in the control group were not bleached, but immersed in artificial saliva for 1 week before bonding.

2.3. *Bleaching procedure*

In all bleaching groups, a 10% carbamide peroxide bleaching gel (Opalescence, Ultradent, South Jordan, USA) (Batch no: B2TTV) was applied to the enamel surfaces at 100% relative humidity for 8 h in a day according to the manufacturer's instructions. The specimens were partially immersed in the artificial saliva at 37 °C in a glass laboratory beaker, in this way the enamel surfaces coated with the bleaching gel did not contact with the saliva. At the end of the daily bleaching procedure, specimens were thoroughly rinsed with an air/water spray for 30 s and air-dried. Specimens were stored in a 250-ml of artificial saliva at the remaining hours in a day. The bleaching procedure lasted 7 days. Effective bleaching was observed visually by colour changes of the specimens.

2.4. *Application of antioxidant*

Group 2 specimens were treated as follows: 10 ml of 10% sodium ascorbate (pH 7.4) was dripped on the enamel surfaces of the embedded teeth following the bleaching process and agitated with a sterile brush. After 10 min, it was washed with distilled water and dried. Sodium ascorbate solution was applied to the enamel samples for 10 min. Sodium ascorbate solution of 10 ml was applied onto the surface of the each sample. In each minute, 1 ml solution was dropped onto the sample with a syringe in order to keep the enamel surface wet.

2.5. *Artificial saliva immersion*

The specimens in Group 3 were immersed in 250 ml of artificial saliva solution at 37 °C for 7 days immediately after the bleaching process whereas those in Group 4 were only immersed in the artificial saliva for 7 days without prior bleaching. The artificial saliva solution had an electrolyte composition similar to that of human saliva. It was composed of 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 at pH 7. The artificial saliva solution was changed twice daily during the consecutive 7-day time period. After the specimens were removed from the artificial saliva, the enamel surfaces were rinsed with an air/water syringe for 30 s.

2.6. *Bonding procedure*

The ceramic specimens were etched with 4.9% hydrofluoric acid (IPS ceramic etching gel, Ivoclar, Schaan, Liechtenstein), then silanated (Monobond-S, Ivoclar, Schaan, Liechtenstein) for 60 s and dried gently and the bonding agent (Heliobond, Ivoclar, Schaan, Liechtenstein) was applied. Ceramic specimens in each group were luted to tooth surfaces by a dual-curing cement (Variolink II with Syntac adhesive system, Ivoclar, Schaan, Liechtenstein) according to the manufacturer's instructions. Each specimen was totaly light cured for 160 s circumferentially with a light-curing unit (Elipar Trilight 3M, ESPE, Germany) having an energy output exceeding 500 mW/mm^2 under a constant load of 300 g. Then, specimens were stored in distilled water at 37 C for 24 h and subjected to 1000 thermal cycles between the baths of 5 and 55 °C, with a dwell time of 30 s.

2.7. *Shear bond strength testing*

Specimens were mounted in a jig of the universal testing machine (Autograph Model AG-50kNG, Shimadzu, Japan) and a knife-edge shearing rod was applied to the ceramic/tooth interface until fracture occurred. The specimens were loaded at a crosshead speed of 0.5 mm/min. The shear bond strengths of the specimens were calculated and expressed in MPa.

Fracture analysis of the bonded enamel surface was performed using a scanning electron microscope (Jeol JSM 5200, Kyoto, Japan) at 50× and 850× magnifications. Failures were classified as adhesives (>75% of failure between tooth and restorative material), cohesive (>75% of the failure was within the restorative material), or a mixture of the two.

Statistical analysis was carried out with the SPSS 13.0 software system (SPSS Inc., Chicago, USA). The shear bond strength data of the groups were statistically analysed with the Kruskall–Wallis and Mann–Whitney *U*-tests. The fracture modes of the samples were compared by the Chi-square test. The level of significance was determined as $p = 0.05$ for all tests.

3. Results

SBS values of the specimens are shown in Table 1. While the samples that were immediately bonded after bleaching (Group I) demonstrated significantly lower shear bond strengths and 10% sodium ascorbate group (Group II) demonstrated significantly higher bond strengths than control group samples

Table 1 – SBS values of tested groups

Substrate	Mean ± S.D. (MPa)
Group 1	31.6 ± 9.3
Group 2	41.7 ± 1.9
Group 3	37.5 ± 8.3
Group 4	38.8 ± 4.1

Table 2 – The fracture modes of the tested groups

Groups	Adhesive	Mix	Cohesive
Group 1	4(%40)	5(%50)	1(%10)
Group 2	3(%30)	4(%40)	3(%30)
Group 3	3(%30)	6(%60)	1(%10)
Group 4	4(%40)	5(%50)	1(%10)

($p < 0.05$), no significant differences were found among delayed bonded group and control group ($p > 0.05$).

The fracture modes of groups are shown in Table 2. In each group, nearly half of the samples demonstrated mix fracture modes. For unbleached enamel group, mainly mixed fractures were observed (Fig. 1). The number of adhesive fracture modes was high in the immediately bonded after bleaching group (Fig. 2) which revealed the lowest mean bond strength value; whereas the number of cohesive type of fracture mode was high in the 10% sodium ascorbate group (Fig. 3) which had the highest bond strength. Exclusively mixed failures were prevalent in delayed bonded enamel group (Fig. 4). There were no significant differences among the study groups in terms of fracture modes ($p > 0.05$).

4. Discussion

Following bleaching procedures, patients often require additional aesthetic interventions such as, replacement of old restorations or application of laminate veneers to restore aesthetic deficiencies. Thus, the initial decrease in enamel bond strength after bleaching is clinically important. The bond strength of luting resins to enamel were reported to vary from 17.7 to 49.2 MPa.[22,23] However, a number of studies have shown that the bond strengths of indirect adhesive restora-

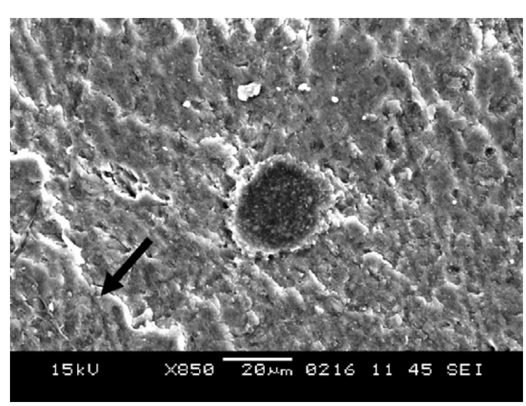

Fig. 1 – SEM image (850×) of the typical failure types for the unbleached group. Note the cohesive failure in the luting resin with layered appearance as indicated by the arrow.

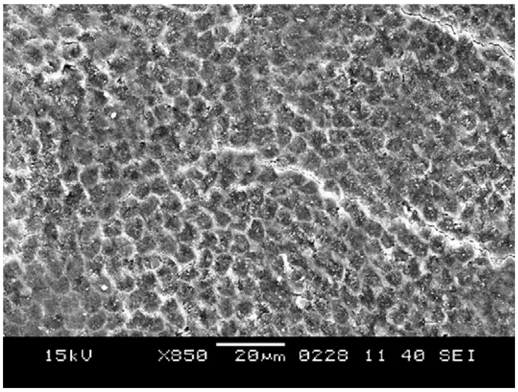

Fig. 2 – SEM image (850×) of the failure types for the bonded specimens immediately after bleaching. Etched enamel surface was observed as a honeycomb appearance with mainly adhesive failures without resin penetration.

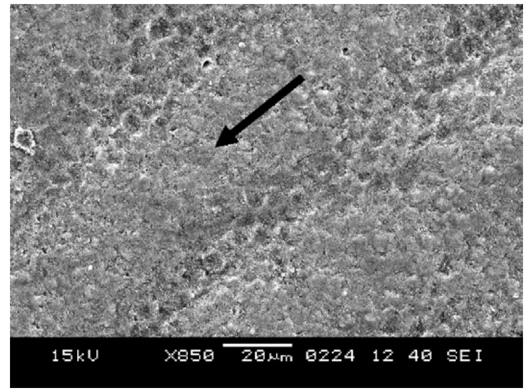

Fig. 4 – SEM image (850×) of the delayed bonded group showing exclusively mixed failures. The areas indicated by an arrow are the cohesively failed luting resin while the honeycomb appearing areas define the adhesively failed surfaces.

tions to tooth structures are reduced when the tooth has been bleached with carbamide peroxide.[4,5,7,13,24] There seems to be no literature about restoring the decreased bond strength of luting resin cements to bleached enamel by sodium ascorbate application as an antioxidant and delaying luting (1 week after bleaching) process. In this study, delayed bonding and antioxidant application following bleaching recovered the bond strength of a luting resin to enamel similar to the relevant literature on restorative composites.[5–8] Reduction in bond strength due to the bleaching has been attributed to the residual oxygen, released from the bleaching agent, that interferes with the resin infiltration into the etched enamel, or inhibits the polymerization of resin.[10,16,24] Titley et al.[10] reported that in the SEM evaluation of bleached specimens, large areas of the enamel surface were free of resin, and, when tags were present, they were fragmented and poorly defined and penetrated to a lesser depth when compared with the unbleached controls. In another study, Titley et al.[24] displayed a granular and porous aspect with a bubbly appearance at the interfaces between resin and bleached enamel with SEM examination. This porous appearance of the interfaces postulated to be gaseous bubbling from oxidizing reactions, possibly resulted from retained peroxide in the subsurface layer of the enamel.[22] This suggestion is corroborated by the present study, in which low SBS values and decreased number of resin tags were observed under SEM (Fig. 2) in the specimens that were bonded immediately after bleaching.

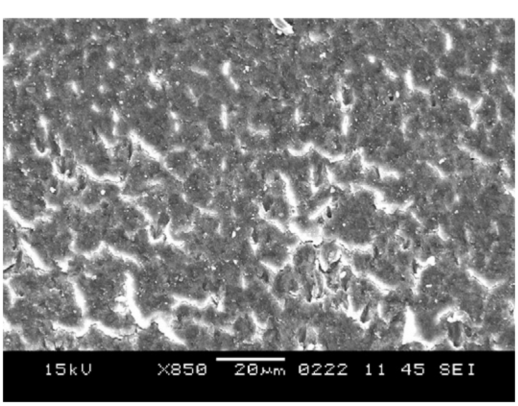

Fig. 3 – SEM image (850×) of the sodium ascorbate applied specimens, the fractures occurred as predominantly cohesive and mixed types.

Although many methods were used to reverse the compromised bond strength, emphasis was placed on neutralizing the oxygen by application of an antioxidating agent. Ascorbic acid and its sodium salt are well-known antioxidants with the capacity of reducing oxidative compounds, especially free radicals.[25–27] Lai et al.[28] immersed the bleached specimens in 10% sodium ascorbate solution for 3 h. In more recent studies,[19,20] 10 min of antioxidant treatment was tested. In all studies, sodium ascorbate treatment appeared to restore the reduced bond strength of composite to the bleached enamel samples. 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 thus reversing the compromised bonding.[6] Besides its antioxidant effect, ascorbic acid has also been shown to increase the bond strength of a chemically cured resin to tooth,[29] however, in our study a dual-curing luting resin was used and increased bond strengths were obtained, too. This might be attributed to the etching potential of ascorbic acid rather than the cement type chosen. Similar with these studies, in the present study sodium ascorbate applied specimens exhibited cohesive cracks within resin (Fig. 3) and higher SBS values compared to the other groups. Sodium ascorbate was applied for antioxidant treatment and the duration was 10 min, which is a reasonable time period for clinical application.

In this study, there were no significant differences between unbleached and delayed groups. This might be explained by the possible morphological or structural changes on enamel that were repaired during waiting period between bleaching and bonding while teeth were stored in artificial saliva.[15,7,24,14] Storing samples in artificial saliva might have contributed to the removal of the residual oxygen from the enamel surface during immersion process.

An attempt was made to provide an explanation for the structural alteration and reduction of bond strength in the presence of peroxide. It has been shown that hydroxyl radicals in the apatite lattice are substituted by peroxide ions and produce peroxide–apatite. When peroxide ions decompose, substituted hydroxyl radicals re-enter the apatite lattice, resulting in the elimination of the structural changes caused by the incorporation of peroxide ions.[30]

Post-bleaching time that is required to restore the bond strength to pre-bleached level is quite controversial. Although, there are remarkable variations among the recommended post-bleaching time periods in different studies, most of the researchers advised delays in bonding of 1 week after bleaching.[7] Thus, delaying bonding process by immersion in a remineralizing solution for 1 week seems to restore enamel properties and contribute to an optimal adhesion after bleaching. In the present study, treatment of the bleached tooth with 10% sodium ascorbate before bonding appeared to restore the reduced SBS values.

When standard bleaching protocols are considered, it is wise to wait before esthetic restoring of teeth until the colour change has been stabilized. Besides delaying bonding of restorations, application of an antioxidant might also be recommended to restore the compromised bond strength of the luting resin to bleached enamel.

5. Conclusions

Within the limitations of this study the following conclusions were drawn:

1 Bleaching with carbamide peroxide immediately before bonding reduced the SBS of the luting composite resin to enamel.
2 One week delayed bonding procedure after bleaching results in a reversal of reduced bond strength.
3 Treatment of the bleached enamel surfaces with sodium ascorbate reversed the reduced bond strength and may be an alternative to delayed bonding, especially when restoration is to be completed immediately after bleaching.

6. Clinical relevance

Treatment of the bleached teeth with 10% sodium ascorbate before bonding can be used in clinical practice for decreasing delay in fabricating esthetic restorations.

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