



**EFFECTS OF SODIUM ASCORBATE AND ELAPSED TIME ON BOND
STRENGTH OF RESIN TO BLEACHED ENAMEL**

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ABSTRACT

After the bleaching procedure, the clinical performance of resin- based composite restorations can be influenced by potential changes due to the presence of bleaching agents in dental structure. The purpose of this study was to evaluate the effects of 10% sodium ascorbate treatment and elapsed time on microtensile bond strength of composite resin to office bleached enamel. **Materials and Methods:** 63 human molar teeth were randomly distributed into 5 groups, 1 control (n=3) and 4 experimental (n=15). Control: Unbleached; (C-): Bleached (carbamide peroxide); (C+): Bleached (carbamide peroxide) +10% sodium ascorbate; (H-): Bleached (hydrogen peroxide); (H+): Bleached (hydrogen peroxide) +10% sodium ascorbate. Based on the storage period (0 = immediate, 3, 7, 10, 14 days after bleaching), non- control groups were further divided into five subgroups (n=3) prior to composite build-up. After the restorative procedure, sectioning of the specimens was carried

out in order to obtain proper sticks. The bond strength was quantitatively evaluated by a microtensile test (0.5 mm/min), Finally ANOVA and LSD studies were set up with significance level of 5%. **Results:** The unbleached group (control) (30.869 ± 0.424) had the highest bond strength mean and was statistically similar ($p > 0.05$) to H+0 (30.242 ± 1.066), C+0 (30.548 ± 1.174) and statistically different ($p < 0.05$) from the others. It is recommended adhesive restorative procedures in bleached enamel (independent on bleaching agent) be done after bleaching with application of 10% sodium ascorbate on the same day.

Keywords: Tooth bleaching, Antioxidants, Sodium ascorbate, Hydrogen peroxide, Carbamide peroxide

INTRODUCTION

Having whiter and brighter teeth is one of the major demands for esthetic dentistry[1]. Tooth bleaching has been a treatment modality for discolored teeth since late 1870s[2]. Vital bleaching is a safe, popular, conservative and also well-accepted treatment for discolored teeth [3]. Various bleaching agents have been used to achieve favorable esthetic results reasonably quickly. Among these bleaching materials, hydrogen peroxide and carbamide peroxide have a successful history as whitening agents [4]. Further to these esthetic results for patients, it is shown that bleaching agents have some negative effects on the enamel's organic structure[5]. Loss of mineral contents, high levels of surface roughness along with a decrease in microhardness, fracture toughness and tensile bond strength can reduce enamel bond-strength (sometimes up to 75%), immediately after bleaching [5]. After the bleaching procedure, the clinical performance of resin-based composite

restorations can be influenced by potential changes due to the presence of bleaching agents in dental structure[6]. The light activation of resin materials can be inhibited by free radicals from oxygen, which remain in dental tissues [7]. Clinically, this decreased bond strength can be very important because often tooth bleaching is done prior to the application of resin-based composite [8]. Therefore, many studies were done to determine the required time for reversal of the adverse bleaching effects [5]. Most specialists recommend a waiting period of 7 days for enamel and 14 days for dentin after bleaching [9]. Some authors suggest 24-35 hour delay between teeth bleaching and the bonding procedure [10]. In some cases, the patient would like to have their teeth restored as soon as possible and therefore the mentioned delay would not be ideal. Various methods have been proposed to counter this requirement. Amongst them, application of antioxidant agents such as sodium ascorbate has shown promising

results [5]. Peroxide apatite is made of peroxide ions, which are substituted by the hydroxyl radicals in the apatite lattice. When peroxide ions break down, substituted hydroxyl radicals reenter the apatite lattice, resulting in the elimination of the structural changes caused by the incorporation of peroxide ions [11]. Antioxidant agents can restore the altered redox potential of the bleached surface and reverse the compromised bonding. Therefore, composite restorations can be done following bleaching [12]. Considering the clinical importance of this topic, the present study was carried out to evaluate the effects of the application of 10%-sodium-ascorbate and the elapsed time from bleaching on microtensile bond strength of enamel.

Specimen preparation

MATERIALS AND METHODS

Products used in this study are presented in **Table 1**.

Table 1: Products used in this study

Brand	Material type	Composition	Manufacturer
Opalescence quick 45%	Office bleaching agent	45% Carbamide Peroxide	Ultradent/USA
Opalescence Boost PF 40%	Office bleaching agent	40% Hydrogen Peroxide	Ultradent/USA
Sodium ascorbate	Sodium ascorbate	Sodium ascorbate (C ₆ H ₇ NaO ₆)	Sigma-Aldrich / Germany
Scotchbond™ etchant	37% phosphoric acid	Phosphoric acid, water, thickening, stain	3M-ESPE /USA
Adper™ single bond plus	Adhesive system	BisGMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids	3M-ESPE /USA
Filtek Z250	Composite resin	Matrix: BIS-GMA, UDMA, and Bis-EMA Filler: 60% (volume) silica/zirconia, average particle size of 0.6 μm.	3M-ESPE /USA
Bis-GMA = Bis-phenol-A-glycidylmethacrylate; UDMA = Urethane dimethacrylate; TEGMA = Triethyleneglycol methacrylate; HEMA = 2-hydroxyethyl methacrylate.			

Table 2: Experimental design considering the interval after bleaching and the application of antioxidant

Group	Office bleaching	Antioxidant	Subgroup*	Number
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63 human caries-free molar teeth, freshly extracted for periodontal reasons, were taken for this study. Fractured, cracked, and dried teeth were excluded. The specimens were cleaned with periodontal cures and ultrasound, submitted to prophylaxis with pumice and water and stored in distilled water at 37°C. Subsequently, they were randomly divided into five groups. The control group (N=3) did not receive any bleaching procedure. According to the type of bleaching agent and antioxidant application, the other four groups were assigned 15 teeth. Non-control groups were further divided into five subgroups of 3 specimens each, according to the storage period (0=immediate, 3, 7, 10, 14 days after bleaching) prior to composite build-up (Table 2).

		(SA)		
Control	-	-	-	3
C-	CP	-	+	15
C+	CP	+	+	15
H-	HP	-	+	15
H+	HP	+	+	15
CP=45% carbamide peroxide, HP= 40% hydrogen peroxide, SA= 10% sodium ascorbate, *: Post bleaching interval (0, 3,7,10,14 days), subgroups (n=3)				

Office bleaching procedure

In groups C (\pm), 45% carbamide peroxide was used (Opalescence quick 45%/Ultradent/St. Louis/USA) for 30 minutes, whereas for specimens in groups H (\pm), 40% hydrogen peroxide (Opalescence Boost PF 40%/Ultradent/ St. Louis/USA) was applied for two 20-minute intervals (i.e. a total of 40 minutes) on the enamel surface of the teeth. Finally, they were rinsed for 30 seconds with jets of water and dried with compressed air. The bleached teeth were stored in distilled water at 37°C for 3, 7, 10, 14 days depends on their elapsed time prior to bonding.

Treatment with 10% sodium ascorbate

In groups C+ and H+, after waiting period of 0 (immediately), 3, 7, 10, 14 days after bleaching, 10% sodium ascorbate (Sigma-Aldrich/Taufkirchen/Germany) was applied on bleached enamel surface of each specimen for 10 minutes. Subsequently specimens were rinsed with jets of water for 30 seconds, and dried with compressed air.

Restoration with composite resin

The enamel surface of specimens was treated with 37% phosphoric acid (Scotchbond™ etchant/ 3M-ESPE/St. Paul/

USA) for 30 seconds, rinsed for 30 seconds and air-dried. Afterwards, two layers of a fifth generation adhesive system (Adper™ single bond plus/ 3M-ESPE/St. Paul/USA) were applied to the enamel surface. Light activation was carried out for 20 seconds. Composite resin (Filtek™ Z250/3M-ESPE/St. Paul/USA) in increments of 1 mm was placed on the enamel surface and was light cured (800 mW/cm²) with an LED device (Blue Phase C5, Ivoclar Vivadent, Lichtenstein) for 20 seconds. All enamel surfaces were restored with 4-mm high resin composite. The specimens were stored in distilled water at 37°C for 24 hours before preparation for microtensile test.

Microtensile testing procedure

A precision sectioning machine was used to cut each individual specimen to obtain proper sticks (from each specimen, five sticks were prepared) with a height of 8 mm (4 mm of composite and 4 mm of enamel plus dentin) and a cross-sectional area of 1.0 ± 0.1 mm². From control and each 20 subgroups, 315 sticks were prepared. In order to assess the microtensile bond strength, each stick was individually fixed to a microtensile-testing device (Bisco,

USA) using cyanoacrylate adhesive (Mitreapel, Turkey). This is done by positioning the adhesive interface perpendicular to the direction of force applied to the specimen. The microtensile test was run at a speed of 0.5 mm/min until the fracture occurred. The force at which the fracture happened was recorded in Newton's (N). Finally, the adhesion area was measured with a digital caliper (Mitutuyo, USA) and was used to calculate the pressure on the surface in Mega Pascal's (MPa).

Following the microtensile test, as an initial assessment of the failure mode, each fragment of the ruptured stick was examined using a stereoscopic microscope (Nikon SMZ-800, USA) with 6.3x magnification. Observed fracture types were as follows:

- Adhesive fracture: Failure in adhesion, fracture at the interface
- Cohesive fracture: Dental substrate failure or restorative material failure
- Mixed fracture: Dental substrate and resin material fracture (adhesive or resin composite) in the same test specimen.

Statistical analysis

Descriptive statistics were calculated. The normality of microtensile bond-strength distribution was confirmed using a Kolmogorov-Smirnov test. Data was then analyzed by applying the analysis of variance (ANOVA). For the individual

checks LSD test was employed. The level of significance was set at 5% ($p \leq 0.05$).

RESULTS

The cross sectional areas of sticks were statistically homogenous ($0.70-1.03 \text{ mm}^2$, $P=0.62$), indicating that differences in bond strength among groups would not be due to differences in sectional area. Table 3 presents the bond-strength means, standard deviations and failure types for different experimental groups.

Figure 1 summarizes the results of the study. In Fig. 1, the microtensile bond strengths of the control teeth were uniformly high at $30.869 \pm 0.424 \text{ MPa}$ at times 0, 3, 7, 10 and 14 days because they were not bleached with either carbamide or hydrogen peroxide. The lowest bond strengths were seen in specimens bleached with hydrogen peroxide, but were not subsequently treated with sodium ascorbate. The data show that the H-O bond strengths were $23.365 \pm 0.445 \text{ MPa}$. After 3 days of storage in distilled water, the bond strengths were $23.520 \pm 0.524 \text{ MPa}$ (not significantly different from H-O). By 7 days, the bond strengths were only $24.102 \pm 1.528 \text{ MPa}$ that were still not significantly different from H-O values. By 10 days, the bond strengths were only $24.233 \pm 0.770 \text{ MPa}$. Clearly, 14 days of storage in water did not reverse the low bond strengths of hydrogen peroxide treated enamel. When enamel was bleached with carbamide peroxide but was not treated with

sodium ascorbate (C-), the specimens that were bonded immediately (C-O) gave bond strengths that were similar to those found in H-O (23.588 ± 0.566 MPa, Table 3). There were not significantly different from those of H-O. When time delays were allowed to allow dissipation of any retained peroxide, the bond strengths slowly increased to values of 26.132 ± 1.222 MPa for C-14 (Table 3, Fig. 1). When carbamide peroxide treated teeth were immediately treated with sodium ascorbate, and bonded immediately (C+O), the bond strengths were 30.548 ± 1.174 MPa (Table 3) which were not significantly different from unbleached controls (30.869 ± 0.424 MPa). However, when carbamide peroxide bleached enamel

specimens were stored for 3, 7, 10 or 14 days and then treated with sodium ascorbate, the bond strengths fell progressively (C+3, C+7, C+10 and C+14 gave values of 29.075 ± 0.932 , 28.285 ± 0.758 , 27.783 ± 0.996 and 26.813 ± 1.273 MPa, respectively). Similarly, when hydrogen peroxide bleached specimens were stored for 3, 7, 10 or 14 days to allow for peroxide dissipation prior to being treated with sodium ascorbate, the bonds strengths fell even more, especially after 14 days (Fig. 1). Microscopic analysis revealed that fracture modes were of cohesive dental (51.7%), cohesive restorative material (19.4%), adhesive (18.7%) and mixed (10.2%) failure types.

Table 3: Microtensile bond strength means (in MPa) and standard deviations and failure types after test in the different experimental groups

Experimental groups	Bond strength (MPa)	Failure mode (%)			
	Means \pm SD	Cohesive of tooth	Cohesive of composite	Adhesive	Mixed
Non bleached teeth (control)	30.869 ± 0.424 a	33.3	20.0	33.3	13.3
C-0	23.588 ± 0.566 b	46.7	6.7	20.0	26.7
C+0	30.548 ± 1.174 a	80.0	6.7	6.7	6.7
H-0	23.365 ± 0.445 bc	20.0	33.3	26.7	20.0
H+0	30.242 ± 1.066 a	53.3	20.0	13.3	13.3
C-3	24.559 ± 1.169	86.7	6.7	6.7	0.0
C+3	29.075 ± 0.932 d	46.7	26.7	13.3	13.3
H-3	23.520 ± 0.524 c	40.0	33.3	26.7	0.0
H+3	28.419 ± 1.415 d	40.0	40.0	6.7	13.3
C-7	24.881 ± 0.801	66.7	6.7	20.0	6.7
C+7	28.285 ± 0.758 e	53.3	13.3	33.3	0.0
H-7	24.102 ± 1.528 c	53.3	26.7	20.0	0.0
H+7	28.155 ± 1.706 e	53.3	26.7	20.0	0.0
C-10	25.071 ± 0.755	66.7	13.3	13.3	6.7
C+10	27.783 ± 0.996 f	46.7	20.0	26.7	6.7
H-10	24.233 ± 0.770	53.3	13.3	20.0	13.3
H+10	27.680 ± 1.950 f	66.7	26.7	0.0	6.7
C-14	26.132 ± 1.222 g	33.3	13.3	40.0	13.3
C+14	26.813 ± 1.273 g	53.3	13.3	20.0	13.3
H-14	25.168 ± 0.903 h	33.3	40.0	6.7	20.0
H+14	25.463 ± 0.605 h	60.0	0.0	20.0	20.0

Different letters indicate statistically significant differences (LSD test, $p < 0.05$)

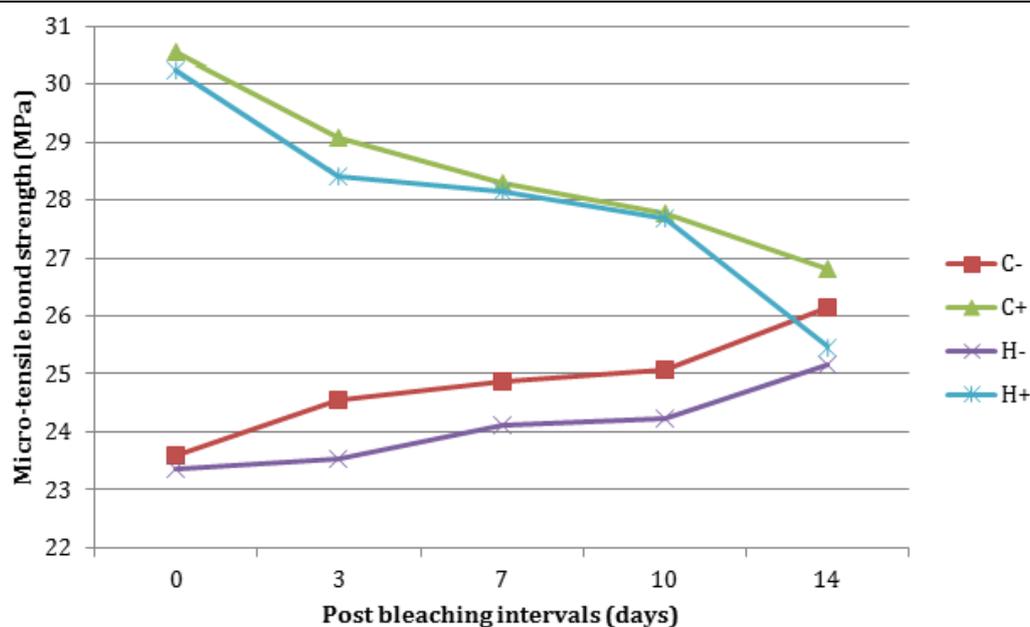


Figure 1: Mean values of microtensile bond strength in comparison to the post bleaching intervals

DISCUSSION

As far as the authors know, this is the first report to assess the effect of sodium ascorbate application on the bond-strength between resin composite and bleached enamel after different post bleaching intervals (0,3,7,10,14 days). The primary outcome of the analysis is the confirmation that the application of 10% sodium ascorbate could fully reverse the reduction of bond-strength just immediately after bleaching. However, for post-bleaching intervals of 3, 7 and 10 days, the increase in bond-strength was statistically significant but not enough to fully restore the adverse effects. For the interval of 14 days the increase was not statistically significant. We speculate that residual peroxide ions may have diffused deeper into enamel via solid state diffusion, removing it from the neutralizing effects of sodium ascorbate.

Many studies have used the microtensile test to evaluate the bond strength of resin composite to bleached teeth [13, 14]. This study was designed to assess the optimal delay period between bleaching treatment and the restorative. In many cases, the esthetic restoration of anterior teeth such as composite veneers involves enamel dental substrates; therefore, many studies evaluated the effects of bleaching agents on bond-strength between resin composites and enamel [13, 15].

Nour El-din et al, [16] concluded that the bond strength between resin and bleached enamel (38% hydrogen peroxide and 10% carbamide peroxide) was significantly lower compared to the non-bleached controls. Many authors found similar results [17, 18]. Higher levels of free radical peroxides or oxygen, alone or in combination at the bond interface, can explain the decrease in bond-strength after bleaching. This could be

due to the fact that these free radicals also contaminate the polymerization reaction and reduce bonding [19]. Lai et al, noted that the reduction of the bond strength between resin and bleached enamel is likely to be caused by a delayed release of oxygen that may affect the polymerization of resin components[20].

Additionally, morphological, [21] physical, [22] and chemical [23] changes in dental hard tissues might occur after application of bleaching agent. Perdigão et al, [24] suggested that bleaching might result in morphological alterations in most superficial enamel crystallites.

In this study, we conclude that bleaching treatment (regardless of bleaching material type), reduced the bond-strength between composite and enamel, especially when hydrogen peroxide was used. However, when the restorative procedure is done immediately after bleaching, no significant difference was observed between the behavior of the bond under these two types of material. Somehow contrary to what we observed, Patusco et al, [25] and Akin et al, [26] suggest that the use of 10% carbamide peroxide bleaching does not significantly reduce shear bond strength values. This discrepancy could be contributed to the different carbamide peroxide concentration, which was lower than what was used in our study.

According to Unluet al, [27] it is advisable

that resin composite application onto bleached enamel surfaces should be delayed at least 24 h for 10% carbamide peroxide and 1 week for 35% hydrogen peroxide. Dietrich et al, [28] observed that a delay of 1 week after bleaching decreased the adverse effects of dental bleaching agents on bonding procedures to enamel. Cavalliet al, [29] suggested that for the first two weeks post-bleaching, the bond strength of resin to enamel is low. After a three-week period, the bond-strength returns to that of the untreated control group. Similar results were found by Uysal et al, [30] after 30 days and by Mullins et al, [31] after at least 2-3 weeks, and Nascimento et al, [32] after 3 weeks. In this study, even after 14 days (\pm 10% sodium ascorbate), the bond-strength was observed to be significantly lower than that of the unbleached group. This finding was in agreement with Khoroushi and Ghazalgoo, [33] Basting et al, [34] and Liu et al [35].

According to Demarco et al, [36] hydrogen peroxide left on the surface after bleaching loses its activity with time because of its instability. An antioxidant is capable of eliminating reactive free radicals. One of the most common antioxidants is sodium ascorbate, which is used extensively in the food industry. Hence, it would be safe to use the material in the oral environment [37]. Sodium ascorbate has been used in many studies as an antioxidant agent. According to

these studies, no statistically significant difference in tensile bond-strength was noted when antioxidant-treated (immediately after bleaching procedure) groups were compared with the control group (no treatment before bonding)[38-40].

Abraham et al,[41] noted that sodium ascorbate is the only antioxidant agent able to reverse the bond-strength to enamel immediately after bleaching. Arumugamet al, [42] suggested that among the antioxidant groups, sodium ascorbate showed reasonably higher bond-strength compared to proanthocyanidin and lycopene. A similar antioxidant was used in the present study, with satisfactory results.

Kimyai and Valizadeh[43] found there is no significant difference among various forms of sodium ascorbate preparation (hydrogel and solution). According to Thapa et al,[44] 10 minutes application of 10% and 25% sodium ascorbate solutions significantly improves the bond-strength of composite resin to bleached enamel. In other studies,[45] 10 minutes application of 10% sodium ascorbate on bleached enamel was found to be advantageous.

In the present study, the treatment of bleached enamel surface with 10% sodium ascorbate solution for 10 minutes was found to be a useful clinical option for patients in need of immediate esthetic restorations following bleaching. Since sodium ascorbate oxidizes with time[46], the

solution used in this study was prepared briefly before its application.

Microscopic analysis in this study shows that fracture modes were predominantly of cohesive dental type. Furthermore, it was observed that the cohesive-tooth failure rate of bleached groups was higher than the control group. This can be due to morphological alterations of superficial enamel, which is in agreement with Perdigão et al, [24] They concluded that bleaching resulted in a significant decrease in the relative concentrations for calcium and phosphorus and triggered morphological alterations in most superficial enamel crystallites.

Finally, it is suggested that further in vitro researches using mechanical fatigue tests and clinical studies are conducted to confirm these findings.

CONCLUSION

Hydrogen peroxide reduced the bond strength of composite to enamel more than carbamide peroxide. The application of 10% sodium ascorbate could fully reverse the reduction of bond-strength only if it was used immediately after bleaching. The adhesive restoration procedure is recommended immediately after the application of 10% sodium ascorbate, or alternatively, it should be delayed more than 14 days after office bleaching.

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