

Effect of sodium ascorbate on the bond strength of silorane and methacrylate composites after vital bleaching

Eda Güler^(a)
Nihan Gönülol^(a)
Özgün Yusuf Özyılmaz^(b)
Ali Çağın Yücel^(c)

^(a)Department of Restorative Dentistry, Faculty of Dentistry, Ondokuz Mayıs University, Samsun, Turkey.

^(b)Department of Prosthodontic Dentistry, Faculty of Dentistry, Selçuk University, Konya, Turkey.

^(c)Department of Endodontics, Faculty of Dentistry, Ondokuz Mayıs University, Samsun, Turkey.

Abstract: We investigated the effect of sodium ascorbate (SA) on the microtensile bond strengths (MTBSs) of different composites to bovine enamel after vital bleaching with hydrogen peroxide (HP) or carbamide peroxide (CP). Thirty bovine incisors were randomly divided into five groups and treated with no bleaching application (control), 35% HP alone, 35% HP + 10% SA for 10 minutes (HP+SA), 16% CP alone, or 16% CP + 10% SA for 10 minutes (CP+SA). Specimens were restored with Silorane adhesive and Filtek Silorane composite (designated as S/group) or with Clearfil SE bond and Filtek Supreme XT (designated as F/group). Composite build-up was created on the enamel. Sectioned specimens ($n = 10$ per group; 1 mm² cross-sectional area) were created and stressed in a universal testing machine at 1 mm/min crosshead speed. The application of 10% SA immediately after bleaching with 16% CP or 35% HP increased the enamel MTBS, regardless of the adhesive/composite resin used. The resulting MTBS values were similar to those of the control groups. Use of 16% CP and 35% HP alone decreased the enamel MTBS, regardless of the adhesive/composite resin used, with $F/CP+SA = F/HP+SA = F/CP = S/CP+SA = S/HP+SA = S/C > S/CP = S/HP = F/CP = F/HP$ ($p < 0.05$). We concluded that the application of SA for 10 minutes immediately after vital bleaching increases the enamel BS for dimethacrylate- and silorane-based composites.

Descriptors: Dental Restoration Failure; Dental Debonding; Composite Resins.

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Corresponding Author:
Eda Güler
E-mail: edaguler@omu.edu.tr

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Introduction

Whereas extrinsic tooth discoloration can be removed by prophylactic cleaning procedures, intrinsic tooth staining requires chemical bleaching.¹ Vital tooth bleaching is a widespread procedure for which several techniques and materials have been developed² (e.g., in-office or power bleaching, dentist-controlled home bleaching, and market bleaching products).³ This treatment causes porosity and an over-etched appearance along with loss of prismatic enamel structure, calcium loss and changes in the organic component of enamel. Furthermore, bleaching treatments adversely affect the bond strength (BS) of composite to enamel when bonding is immediately performed after bleaching.⁴ This decreased BS has been related to the presence of residual hydrogen peroxide (HP) in the interprismatic spaces,^{5,6} which can prevent adequate infiltration

of the bonding agent into the dental structures and inhibit its polymerization.⁷

The use of antioxidant agents after bleaching has been suggested to avoid compromising BS.⁸⁻¹⁰ Bio-compatible reducing agents, such as ascorbic acid and its salts, neutralize residual free radicals and decrease various oxidative compounds.¹¹ If antioxidant treatment of bleached enamel before bonding can reverse the reduced BS of composite resin, then this approach could be used as an alternative to delaying the restorative procedure after bleaching.¹²

A resin system was recently developed that is based on the ring-opening polymerization of silorane molecules containing both siloxane and oxirane, rather than the free radical polymerization of dimethacrylate (DMA) monomers. However, the influence of bleaching on the BS of silorane-based materials to the bleached substrate is unclear.¹³ Several studies have evaluated the effects of bleaching on the BS of composite resins, indicating that silorane- and DMA-based resin systems may exhibit similar BS values.^{13,14}

We performed a study to investigate the neutralizing effect of 10% sodium ascorbate (SA) solution on the microtensile BS (MTBS) of silorane-based and DMA-based composite resins to enamel after bleaching with different vital bleaching procedures. The hypothesis tested was that SA would affect the MTBS of both composite types with different monomer structures after vital bleaching.

Methodology

Thirty bovine incisors were used in this study. The tooth surfaces were cleaned via scaling to remove any soft tissue. Crowns were separated from the roots 1 mm apically to the cemento-enamel junction using a diamond saw under water cooling. Enamel surfaces were obtained by trimming the labial surfaces of the crown section to create standard enamel slabs (8 × 8 mm). The slab surfaces were flattened using aluminum oxide abrasive papers (300–600 grit) under water irrigation. All enamel surfaces were examined with a stereomicroscope (Nikon SMZ 1500, Tokyo, Japan). Any specimens with cracks or defects were excluded. The slabs were randomly divided into five groups and treated with no

bleaching (group C, control), 35% HP (group HP), 35% HP followed by 10% SA for 10 minutes (group HP+SA), 16% carbamide peroxide (CP; group CP), or 16% CP followed by 10% SA for 10 minutes (group CP+SA).

Bleaching procedures

In groups HP and HP+SA, an office-bleaching solution of 35% HP (H35 Total Blanc Office; Nova DFL, Rio de Janeiro, Brazil) was applied to the enamel surfaces for 30 minutes twice a day (at 9 am and 5 pm) at 3-day intervals. The effectiveness was increased by stirring the gel every 5 minutes. In groups CP and CP+SA, a home-bleaching solution of 16% CP gel (Zaris White & Brite; 3M ESPE, St. Paul, USA) was applied to the enamel surfaces for 8 hours per day for 7 days, according to the manufacturer's instructions.

The samples were stored in artificial saliva (1.160 g/L NaCl, 0.600 g/L CaCl₂, 0.600 g/L KH₂PO₄, 1,491 g/L KCl, 0.050 g/L NaF, and 100 mL distilled ionized water). In the groups HP+SA and CP+SA, the surfaces were treated with 10% SA for 10 minutes after the bleaching procedures. To prepare the solution, 10% SA (ascorbic acid sodium salt) was prepared by a method in which 98% SA was dissolved in purified water by mixing at room temperature (pH 7.5). Specimens were rinsed with distilled water and dried with compressed air.

Bonding procedures

The samples were divided into two subgroups. In the first subgroup (designated as S/group), Silorane Adhesive System (3M ESPE) was applied to the flattened enamel surfaces. Primer was applied to the surface, which was scrubbed for 15 seconds, dried under a gentle stream of air, and cured for 10 seconds with an LED light-curing unit (LCU; Hilux LEDMAX; Benlioglu Dental Co., Ankara, Turkey) at 500 mW/cm². The bond was applied to the surface, gently air dried, and cured for 10 seconds with the same LCU. Filtek Silorane (3M ESPE) was used for a composite buildup (~3 mm high) on the enamel surfaces, in two increments. Each increment was cured for 40 seconds using the same LCU.

In the second subgroup (designated as F/group),

Table 1 - Materials used in this study.

Product	Composition	Manufacturer	LOT Number
Clearfil SE Bond	Primer: MDP, HEMA, hydrophilic aliphatic dimethacrylate, di-camphorquinone, N,N-diethanol p-toluidine, water Bond: MDP, bis-GMA, HEMA, hydrophobic aliphatic dimethacrylate, di-camphorquinone, N,N-diethanol p-toluidine, colloidal silica	Kuraray, Okayama, JAPAN	01034A
Filtek Silorane Adhesive System	Primer: HEMA, bis-GMA, water, ethanol, phosphoric acid methacryloxyhexylesters, silane treated silica, copolymer of acrylic and itaconic acid, ethyl methacrylate, 1,6 hexanediol dimethacrylate di-camphorquinone, Bond: substituted dimethacrylate, silane treated silica, TEGDMA, phosphoric acid methacryloxyhexylesters, di-camphorquinone, 1,6 hexanediol dimethacrylate	3M ESPE, St. Paul, USA	180901
Filtek Silorane	0.47 µm quartz-yttrium fluoride, siloranes	3M ESPE, St. Paul, USA	321126
Filtek Supreme XT	zirconia/silica, bis-GMA, UDMA, TEGDMA, bis-EMA	3M ESPE, St. Paul, USA	3910A2D
Zaris White & Brite	16% carbamide peroxide	3M ESPE, St. Paul, USA	12424
H35 Total Blanc Office	35% hydrogen peroxide	Nova DFL, Rio de Janeiro, Brazil	11101552

the Clearfil SE bond (Kuraray, Okayama, Japan) two-step self-etch adhesive system was applied to the enamel surfaces without acid etching. Primer was applied to the surface for 20 seconds and dried under a stream of air. The bond was applied, gently air dried, and cured for 10 seconds using the same LCU. Filtek Supreme XT (3M ESPE) was used for composite buildup (~3 mm) in two increments and cured for 20 seconds using the same LCU. The materials used in this study are listed in Table 1.

MTBS testing

Prepared specimens were stored in distilled water at 37°C for 24 hours prior to MTBS testing. Specimens were mounted on acrylic resin blocks, sectioned into serial slabs, and sectioned into sticks (~1 mm² cross-sectional area). An average of 10 sticks was obtained for each group. Each stick was attached to a movable jig that was affixed to the MTBS tester with cyanoacrylate adhesive. The specimens were stressed in a universal testing machine (Bisco, Schaumburg, USA) at a crosshead speed of 1 mm/min. The load at failure (in N) was recorded, and the BS (in MPa) was calculated. Failure modes were evaluated by a single operator with a stereomicroscope at 80× magnification. Failure modes were classified as adhesive (between the adhesive and enamel), cohesive within the substrate (enamel or

composite resins), or mixed (adhesive and cohesive fractures occurred simultaneously).

Statistical analysis

The Shapiro-Wilk normality test indicated normal data distributions for all groups ($p > 0.05$). Two-way analyses of variance (ANOVA) were performed with the SPSS for Windows statistical software (version 12.0.1; SPSS Inc., Chicago, USA) to evaluate the BS values between restorative materials and bleached enamel. Means were compared by the Dunnett test ($\alpha < 0.05$). The pretesting failures were not included in the statistical analyses.

Results

The MTBS values were significantly different by two-way ANOVA ($p < 0.01$). Table 2 displays the mean BS values, standard deviations, and group differences for all groups. No differences were detected between the composite groups ($p = 0.397$). The SA treatment caused differences between the groups ($p < 0.01$). Significant interactions were found between the composite and SA-treated groups ($p = 0.017$).

No differences ($p = 0.291$) in BS values were observed between the groups S/CP (7.6 MPa), S/HP (9.4 MPa), F/CP (9.8 MPa), and F/HP (9.9 MPa). Similarly, no differences ($p = 0.070$) in BS values

Table 2 - Mean bond strengths (MPa), standard deviation and group differences.

Composite	Treatment	Interactions	n	Mean (MPa) ± SD
Filtek Silorane	Control	S/C	10	16.4 ± 3.68 ^b
	HP	S/HP	10	9.4 ± 0.92 ^a
	CP	S/CP	10	7.6 ± 1.54 ^a
	HP+SA	S/HP+SA	10	15.0 ± 1.39 ^b
	CP+SA	S/CP+SA	10	15.0 ± 2.79 ^b
Filtek Supreme XT	Control	F/C	10	14.9 ± 1.13 ^b
	HP	F/HP	10	9.9 ± 0.96 ^a
	CP	F/CP	10	9.8 ± 1.87 ^a
	HP+SA	F/HP+SA	10	13.5 ± 2.48 ^b
	CP+SA	F/CP+SA	10	13.5 ± 2.42 ^b

* The different letters indicate dissimilarity of groups ($p < 0.05$).

Table 3 - Failure modes of fractures.

		Adhesive	Cohesive	Mix
Filtek Supreme XT	Control	42.8%	28.5%	28.5%
	F/HP	92.8%	0	7.1%
	F/HP+SA	64.2%	14.2%	7.1%
	F/CP	85.7%	7.1%	7.1%
	F/CP+SA	64.2%	14.2%	21.4%
Filtek Silorane	Control	71.4%	14.2%	14.2%
	S/HP	92.8%	0	7.1%
	S/HP+SA	50%	28.5%	21.4%
	S/CP	57.1%	28.5%	14.2%
	S/CP+SA	71.4%	21.4%	7.1%

were observed among the groups F/CP+SA (13.5 MPa), F/HP+SA (13.5 MPa), F/CP (14.9 MPa), S/CP+SA (15.0 MPa), S/HP+SA (15.0 MPa), and S/CP (16.4 MPa). The latter groups demonstrated higher BS values than the former groups ($p < 0.05$). Table 3 shows the failure modes obtained with the MTBS test.

Discussion

We evaluated the effect of 10% SA treatment on the MTBSs of different composites to bovine enamel after vital bleaching procedures. The immediate application of SA solution increased the MTBS values in all groups. Therefore, the null hypothesis of this

study was accepted.

In this study, a currently used silorane adhesive system was evaluated on flattened enamel. Lima *et al.*¹³ evaluated the influence of a bleaching treatment on the BS of DMA- and silorane-based composites to bovine enamel and found similar BS values for both restorative systems. Can-Karabulut and Karabulut¹⁵ reported similar BS results for the Silorane Adhesive and Clearfil SE Bond systems in bleached and nonbleached enamel. Similarly, we observed no significant difference in terms of adhesive systems among the tested groups.

For vital bleaching treatments, oxygenating agents such as CP and HP are commonly used.¹⁶ HP breaks down to water, oxygen ions, hydrogen ions, and free radicals. The diffused oxygen reacts with the stained molecules, which starts the bleaching process.¹⁷ CP is used as a vehicle for transporting H₂O₂. First, CP breaks down to uric acid, ammonia, and H₂O₂, which forms H₂O and O₂. The oxygen takes electrons to bleach the enamel.¹⁶ Bleaching agents have been evaluated at different concentrations. In particular, 10% to 16% CP and 30% to 36% HP have been preferred in many studies.¹⁸ In the present study, 35% HP and 16% CP were used.

The influences of HP and CP on enamel and dentin properties have been largely investigated.¹⁹ Due to the chemical properties of the bleaching agents, the mineral content of the dental hard tissue could change.²⁰ Enamel bleaching immediately before bonding with adhesive systems might decrease the BS between materials. Thus, for BS to be established, restorative procedures are not recommended anywhere from 24 hours to 4 weeks after bleaching.²¹ To minimize the time that is required for restoration after bleaching, treatment with ascorbic acid can be preferred.

Ascorbic acid and its salt are well-known antioxidants that can reduce various oxidative compounds, especially free radicals.¹¹ Kimyai and Valizadeh¹⁰ found significantly higher BS values with DMA-based composites in SA-treated groups after 10% CP application. Freire *et al.*²² indicated that a 10% concentration of SA removed the residual peroxide up to 2 minutes after bleaching with 35% HP and increased the bond strength of DMA-based

composite resins to enamel. Silva *et al.*²³ reported that bleached enamel without antioxidant application demonstrated the lowest BS values among the tested groups. Similarly, we found the highest BS values in the control and SA-treated groups, for both types of composite resin, when compared to the untreated groups. To our knowledge, no other study has examined the effect of SA on the BS of silorane-based composites after bleaching. The results of this study showed that SA application after bleaching treatment similarly increased the BS values of the silorane- and DMA-based composite groups.

Silorane-based composites have a different monomer structure (oxiranes and siloxanes) from traditional methacrylate chemistry, and they require a special adhesive system. This system includes a two-step self-etching procedure; etching procedures are not recommended. In particular, because it provided a similar application procedure, the Clearfil SE bond two-step self-etch adhesive system, which does not require an etching step, was used in this study. Lopes *et al.*²⁴ reported that only the Clearfil SE bond system, which includes 10-methacryloxydecyl dihydrogen phosphate functional monomers in its composition, achieved a high enamel BS similar to that of etch-and-rinse systems. This monomer has hydrolytic stability and forms additional chemical interac-

tions with dentin and enamel that contribute to its superior bonding.²⁵ Therefore, in this study, an etching procedure was not considered necessary, due to the superior properties of the adhesive system.

Although using SA significantly increased the BS values after bleaching, the failure modes were mainly adhesive in all of the tested groups. Kahveci and Belli²⁶ stated that higher BS values produce more cohesion and fewer adhesive failures. Phosphoric acid has been proven to produce deeper and more retentive microporosities than even the strongest self-etching adhesive. Thus, the ability of the etch-and-rinse system to establish a valid micromechanical interlocking at the interface with enamel was also manifested by the higher frequency of cohesive failures.²⁷ The use of self-etching adhesives on enamel in this study resulted in lower BS values and, consequently, mostly adhesive failures in all groups.

Conclusions

Within the limitations of the current study, we concluded that the application of 10% SA solution increased the MTBS values to enamel in all of the tested groups. After vital bleaching procedures, the immediate application of SA solution can be helpful for increasing the BS to enamel for both silorane- and DMA-based composites.

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