




Bond strength of resin composite to enamel submitted to at-home desensitizer and bleaching agents

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Aim: This study evaluated the effect of a desensitizer agent (DES) during bleaching with 10% carbamide peroxide (CP) on enamel microshear bond strength (μ SBS). **Methods:** Sixty bovine incisors were obtained and randomly distributed into groups (n=15): (C) Control: no desensitizing or bleaching, (DES) desensitizing gel application, (CP) bleaching with 10% CP and (CP/DES) bleaching with 10% CP combined with DES. Bleaching was performed for 6 h/day for 14 consecutive days. DES was applied for 8 h only on the 7th and 14th days of therapy. Specimens were stored in artificial saliva among the CP or DES applications and submitted to μ SBS testing at three postrestoration times (n=5): 24 h, 7 days, and 14 days after bleaching using a universal testing machine. Failure modes were observed under a stereomicroscope. Data were analyzed by two-way ANOVA and Tukey's test ($\alpha=5\%$). **Results:** Immediately after bleaching (24 h), CP promoted lower μ SBS than the C and DES groups ($p<0.05$) but with no differences from the CP/DES. μ SBS increased in the DES, CP, and CP/DES groups ($p<0.05$) when bonding was performed for 7 or 14 days elapsed from bleaching. CP/DES exhibited the highest μ SBS among the groups 14 days after bleaching ($p<0.05$). Cohesive failure in enamel was predominant in the CP groups, while adhesive failure was mostly observed for the other groups. **Conclusion:** The use of a desensitizer during at-home bleaching maintained the enamel immediate bond strength, and its application favored bonding when the restoration was delayed for 14 days.

Keywords: Tooth bleaching. Dental enamel. Shear strength.



Introduction

Tooth bleaching is a widespread technique commonly performed and desired by patients due to its conservative approach and efficacy in providing color alterations^{1,2}. Carbamide (CP) and hydrogen (HP) peroxides are active compounds of bleaching agents, and their concentrations vary according to the bleaching technique³. In-office bleaching uses high concentrations of CP or HP (higher than 25%), while the home-applied technique uses lower CP (up to 22%) or HP (up to 10%) concentrations. Although a highly concentrated agent could accelerate the bleaching effect, both methods achieve acceptable outcomes^{1,3}. Studies have reported that at-home bleaching resulted in mild tooth sensitivity⁴, but a recent systematic study revealed that the high variation among bleaching protocols could not confirm that at-home bleaching yielded a lower risk or intensity of sensitivity⁵. Therefore, the management of this clinical symptom still represents a challenge during tooth bleaching, even when performed at lower concentrations.

In vitro studies have reported an increase in enamel surface roughness⁶ and a decrease in microhardness⁷ and immediate enamel bond strength⁸ following at-home bleaching. These alterations to the enamel surface could result in pore formation and consequent entrapment of oxygen residuals released from the peroxide agents, which could, in turn, negatively interfere with the light curing of the adhesive agent. Poor light curing could lead to a decreased number, dimension, and quality of the resin tags in the etched enamel⁹. However, despite the cause, enamel bond strength may recover by surface remineralization promoted by saliva or remineralizing solutions^{10,11}.

Although several *in vitro* studies proposed applying antioxidant agents to reverse the immediate bond strength of bleached enamel¹²⁻¹⁴, no randomized clinical trials attest to the long-term performance of restorations placed immediately after bleaching with the antioxidant application. Consequently, delaying the bonding procedures to up to 3 weeks still seems to be an effective method to guarantee satisfactory restorative results after tooth bleaching¹⁰.

In this scenario, studies investigating the effect on the enamel bond strength of potassium nitrate- and sodium fluoride-based desensitizer use before, during, or after tooth bleaching are scarce¹⁵. Although desensitizer application could be a feasible solution to overcome tooth sensitivity¹⁶ and maintain the resin composite bond strength to enamel due to its composition, a study showed that the application of a desensitizer containing potassium nitrate and sodium fluoride reduced the enamel bond strength when the bonding procedure was performed immediately after bleaching¹⁷. The authors credited the bonding reduction to the fluoride deposition on the surface, thereby compromising the adhesion between resin and teeth¹⁷. However, it is necessary to determine if a desensitizer agent influences the enamel bond strength even in delayed bonding procedures.

Based on these facts, this study evaluated the enamel bond strength at different post-restorative times after home-applied bleaching combined or not with a desensitizer agent. The null hypothesis was that the use of home-applied bleaching combined with a desensitizer agent would not influence enamel bond strength immediately or after 7 and 14 days of bleaching treatment.

MATERIAL AND METHODS

Experimental design. Sixty bovine incisors were submitted to the factors (n=5):

1. *Treatment* (four levels):
 - (C) Control: no bleaching or desensitizing treatment;
 - (DES) Enamel application of a desensitizer agent containing 0.25% sodium fluoride and 3% potassium nitrate (Ultra EZ, Ultradent Products Inc., South Jordan, UT, USA);
 - (CP) Enamel bleaching with 10% CP (Opalescence 10%, Ultradent Products);
 - (CP/DES) Enamel bleaching with 10% CP combined with the application of DES.
2. *Time elapsed after bleaching* (three levels):
 - (24 h) Immediately;
 - (7d) 7 days;
 - (14d) 14 days.

The variable responses evaluated were microshear bond strength (μ SBS, in MPa) and fracture mode analysis, observed at 40 \times magnification. Table 1 displays the composition of the materials used.

Table 1. Description of the bleaching and restorative products used in the study

Product and Manufacturer	Composition	Application mode* (*According to manufacturer)
Ultra EZ (Ultradent Products Inc., South Jordan, UT, USA)	Sodium hydroxide, sodium fluoride and potassium nitrate	The recommended treatment times can range from 15 min to 1 h. The length and number of times depend on condition, patient and clinician.
10% Opalescence (Ultradent Products)	Carbamide peroxide, sodium hydroxide and poly(acrylic acid)	Wear Opalescence 10% gel 8-10 hours or overnight. Alternatively, treatments can be from 15 minutes to several hours/day, depending on the patient's needs, level of sensitivity and day-to-day activities.
Ultra Etch (Ultradent Products)	Phosphoric acid, cobalt blue spinel aluminate and siloxane	Apply etchant to enamel and dentin (15 s). Rinse thoroughly, dry and proceed per adhesive manufacturer's instructions.
Adper Single Bond 2 Adhesive (3M Oral Care, St Paul, MN, USA)	Ethyl alcohol, BISGMA, HEMA, UDMA, EDMAB, treated silica, glycerol 1,3-dimethacrylate, copolymer of acrylic acid and itaconic acid, water, diphenyliodonium hexafluorophosphate.	Immediately after blotting, apply 2-3 consecutive coats of adhesive to etched enamel for 15 s with gentle agitation using a fully saturated applicator. Gently air thin for 5 s evaporate solvents. Light cure for 10 s.
Filtek Z350 XT Flow (3M Oral Care)	Certified silanized ceramics, dimethacrylate, BISGMA, EDMAB, TEGDMA, silane treated silica, yttrium fluoride (YbF3), polycaprolactone reacted polymer, benzotriazole, diphenyliodonium hexafluorophosphate.	Place and light cure restorative in increments (20 s for each 2.0 mm increment).

BIS-GMA: bisphenol A diglycidyl ether dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; UDMA: diurethane dimethacrylate; EDMAB: ethyl 4-dimethylaminobenzoate; TEGDMA: triethylene glycol dimethacrylate.

Specimen preparation. Sixty extracted bovine incisors, stored in 0.1% thymol solution, were cleaned with periodontal scalers, and the remaining debris was removed with a bicarbonate jet. The roots were cut at the amelocement junction, and enamel/dentin blocks (5 x 5 mm and 3 mm of thickness) were obtained from the central area of the crown using a double face diamond disc (KG Sorensen, Barueri, SP, Brazil). The blocks were embedded in polystyrene resin, and the buccal surface was flattened with #600, 800, 1200-grit silica carbide papers (3M ESPE, Sumaré, SP, Brazil) and polished with 6, 3, 1, and $\frac{1}{4}$ μm diamond pastes in a polishing machine (Arotec, Cotia, SP, Brazil).

Bleaching and desensitizing treatments. Specimens were randomly distributed into four groups according to bleaching and desensitizing treatments as described previously. A 1-mm layer of the bleaching agent (10% CP) was applied on the exposed enamel surface of groups CP and CP/DES for 6 h/day for 14 days. During bleaching, specimens were stored underneath moisture gauze at 37°C and relative humidity. After bleaching, specimens were thoroughly rinsed with deionized water and stored in artificial saliva [1.5 mM calcium (CaCl_2), 0.9 mM phosphate (NaH_2PO_4), 0.15 mM potassium chloride (KCl)], adjusted to pH 7.0 and 3.125 mL of solution for each mm^2 of exposed enamel until the next gel application^{18,19}.

The desensitizer agent in the CP/DES group was applied over the enamel, similar to the bleaching gel, for 8 h. However, desensitizer application was only performed on the 7th and 14th days of treatment. Specifically, in the CP/DES group, DES application was carried out after the bleaching procedure. After treatments, the specimens were thoroughly rinsed with deionized water and stored in artificial saliva. Desensitizer gel was also applied on the 7th and 14th days in the DES group, but no bleaching agent was applied to the specimens; instead, they were kept in artificial saliva in the remaining hours. In the C group, neither bleaching nor desensitizing treatment was performed; specimens were kept in artificial saliva throughout the 14 days of treatment. The artificial saliva was replaced daily.

Restorative procedure. The specimens were submitted to restorative procedures according to the posttreatment times (n=5): 24 h, 7 d, and 14 d. At each posttreatment time, three composite cylinders were built upon the enamel surface. Enamel was etched with 37% phosphoric acid gel (Ultra Etch, Ultradent Products Inc., South Jordan, UT, USA) for 15 s, rinsed with distilled water and air-dried. One-bottle adhesive (Adper Single Bond, 3M Oral Care, St Paul, MN, USA) was applied according to the manufacturer's instructions (Table 1) and light-cured for 20 s. A silicone matrix mold with a cylindrical configuration (0.75 mm diameter by 2.0 mm high) was placed over the treated surfaces and filled with composite resin (Filtek Z350 Flow – 3M Oral Care, St Paul, MN, USA) using a composite instrument (# 1/2, Duflex - SS White, Rio de Janeiro, RJ, Brazil). The composite was light-cured for 20 s, and the matrix molds were removed to expose the resin composite cylinders bonded to the enamel surfaces. Thus, three bonded small resin cylinders were obtained for each specimen. Composite cylinders were checked under an optical microscope (40c) (EMZ-TR, Meiji Techno Co., Saitama, Japan) to evaluate the composite and interface integrity. Cylinders with no interfacial defects, bubble inclusion or leaking of the composite were tested. The restored specimens were stored in artificial saliva at 37 °C for 24 h.

Microshear Bond Strength Testing. Specimens were fixed to the tested device with cyanoacrylate glue, and the bond strength of each composite cylinder was evaluated in a universal testing machine (4411/Instron Corp, Canton, MA, USA). The shear load (50 N) was applied to the base of the composite cylinder with a thin orthodontic wire (0.2 mm diameter) at a crosshead speed of 0.5 mm/min until failure. The μ SBS was calculated and expressed in MPa. Three bond strength measurements were recorded, and the μ SBS mean was obtained for each specimen.

Failure mode. The enamel failure modes (%) were analyzed with a stereomicroscope at 40 \times magnification (EMZ-TR, Meiji Techno Co., Saitama, Japan) and classified as follows: adhesive in enamel (AD), cohesive in enamel (COE), cohesive in resin (COR) and mixed (MIX) failures involving AD - COE or AD - COR.

Statistical analysis. The normal distribution of the values was verified by Kolmogorov-Smirnov and Lilliefors tests ($p > 0.05$), and a parametric analysis was performed. Data were analyzed by two-way ANOVA, according to the factors treatment and elapsed time following bleaching, and Tukey's test with the significance level set at 5%. SAS 9.0 software was used for all tests (SAS Institute, Cary, NC, USA).

RESULTS

Table 2 displays the results of the μ SBS test. Immediately after bleaching (24 h), CP promoted lower μ SBS than the C and DES groups ($p < 0.05$) but with no differences from CP/DES. Seven days after bleaching, the C group exhibited the lowest μ SBS values ($p < 0.05$), and DES, CP, and CP/DES displayed no significant differences ($p > 0.05$). In addition, at this time point (7 d), the μ SBS of the DES, CP, and CP/DES groups increased in comparison to that of the immediate (24 h) corresponding groups ($p < 0.05$).

Fourteen days elapsed from bleaching, CP/DES exhibited the highest μ SBS among groups, with no significant differences between the 7th and 14th days elapsed from treatment ($p > 0.05$). In addition, no differences were detected among C, DES, and CP at this time point (14 d, $p > 0.05$). C exhibited no bond strength differences, regardless of the evaluation time ($p > 0.05$). The DES and CP groups produced higher μ SBS at the 7th and 14th days elapsed from bleaching than at the immediate time point.

Table 2. μ SBS and standard deviation (MPa) values of bleached and/or desensitized teeth restored at three different elapsed times.

Groups	24 h			7d			14d		
C	11.10	(4.28)	Aa	10.53	(3.39)	Ba	11.29	(3.42)	Ba
DES	10.65	(3.10)	Ab	14.58	(2.48)	Aa	13.76	(3.70)	Ba
CP	7.99	(3.93)	Bb	14.49	(4.78)	Aa	13.65	(3.69)	Ba
CP/DES	9.13	(3.16)	ABb	13.52	(4.94)	Aa	17.00	(3.61)	Aa

According to 2-way ANOVA and Tukey's test, means followed by different letters differ statistically at 5%

Uppercase letters compare the different bleaching protocols within the same evaluation time, and lowercase letters compare the same bleaching protocol within the evaluation times.

Figure 1 depicts the failure modes (in %) of the tested groups according to the post-treatment evaluation times. Adhesive failures (AD) and cohesive failures at enamel (COE) were the most predominant failures found, regardless of the treatment or the evaluation time. Adhesive failures (AD) were predominantly observed for the C (40 - 60%) and DES (30 - 50%) groups, while cohesive failures (COE) were observed for the CP (60 - 70%) and CP/DES (40 - 60%) groups, regardless of the postevaluation times. Mixed failures (MIX) were detected in all groups (10 - 30%), and cohesive failure at the resin was detected for DES, CP (7 days), and CP/DES (14 d) at 10%.

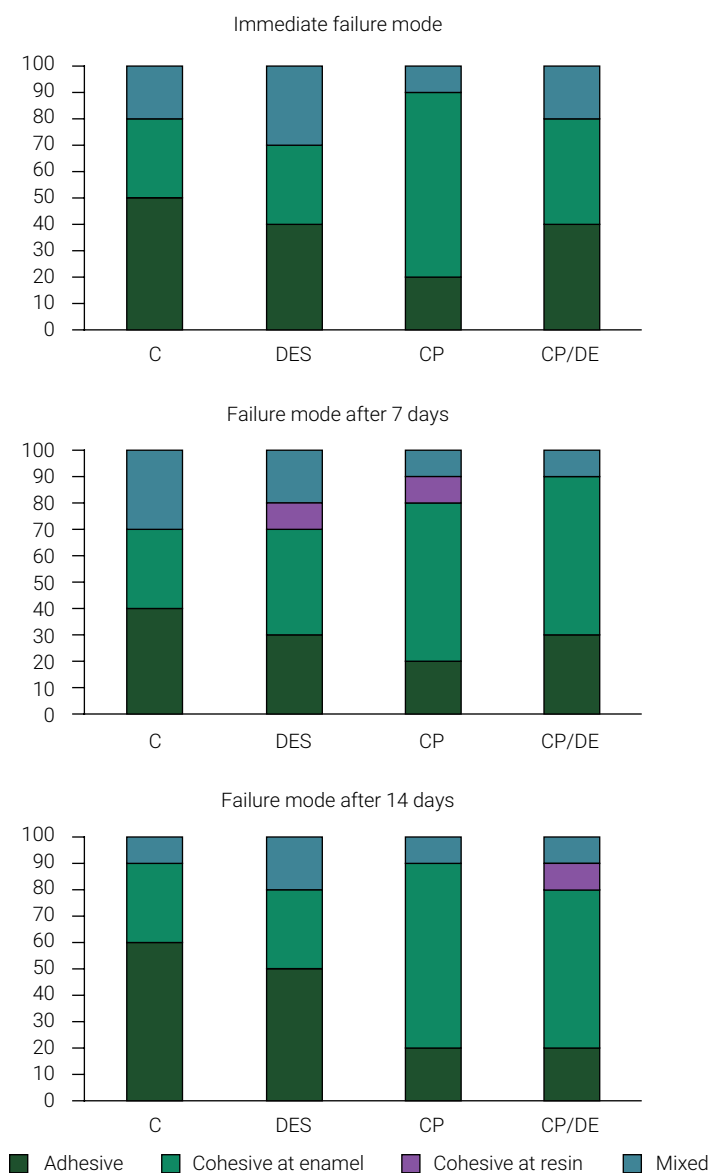


Figure 1. Failure mode distribution (%) of the groups according to the postponement of bonding (24 h – immediate, 7 d and 14 d). C: control; DES: desensitizer; CP: carbamide peroxide; CP/DES: carbamide peroxide and desensitizer.

DISCUSSION

A potassium- and nitrate-based desensitizer combined with CP maintained the enamel μ SBS and displayed values higher than those of the other groups when bonding was performed 14 days after bleaching. In addition, the desensitizer alone promoted higher μ SBS than the control on the 7th day of bonding postponement. Therefore, the results rejected the null hypothesis because this protocol influenced the enamel μ SBS after bleaching. The fluoride effect in the remineralization process could explain this outcome²⁰. The addition of Ca and F to low-concentrated CP has been demonstrated to control enamel mineral loss during bleaching^{21,22}. These findings support the theory that the fluoride in the desensitizer maintained enamel integrity and hence the bond strength.

Contrary to these findings, a report from Amuk et al.¹⁷ (2018) showed that the application of a desensitizer (UltraEZ) during 22% CP bleaching decreased the bond strength. The authors stated that desensitizer deposition weakened the adhesive interface. The differences in both studies could rely on the different CP concentrations, the frequency of the desensitizer applications, and mainly by the time elapsed from the bleaching and bonding procedures. In that study, only immediate bond strength was carried out, and the desensitizer was applied daily for 14 days¹⁷. Therefore, divergences in the protocols might impact the effects of desensitizers on enamel bonding.

Even though bleaching with low-concentrated CP decreased immediate enamel μ SBS²⁰, the postponement (7 or 14 days) of bonding reversed that result. The oxygen release and entrapment in the structures during bleaching may decrease enamel/dentin bond strength²³. In addition, HP could interfere with enamel morphology due to the inorganic content changes produced during bleaching²⁴. These have been the rationale for replacing restorations on bleached teeth only after a few weeks³. Although the desensitizing gel did not increase the immediate bond strength, the results suggested that the combination of CP with a fluoride- and potassium nitrate-containing desensitizer favored bonding in the case of the restoration's postponement. Thus, the 10% CP protocol combined with DES would benefit the enamel μ SBS 14 days elapsed from bleaching.

Fluoride interacts in the tooth demineralization process when chemically soluble, increasing remineralization and reducing mineral loss²⁰. The presence of fluoride ions during an acidic challenge, promoted by a solution with pH 6.5, leads to the exchange of Ca of hydroxyapatite (HA) for fluoride, thereby forming fluorapatite (FA). As FA has a lower solubility and dissociation constant than HA, the pH will have to drop more dramatically (approximately at 4.5) to demineralize enamel²⁵. Therefore, it is likely that the fluoride in the desensitizer gel controlled the μ SBS of bleached enamel similarly to fluoride-containing bleaching agents controlling enamel mineral content²². It is worth mentioning that bleaching gels dispensed in syringes often display neutral or even acidic pH to extend the product's shelf life¹. Moreover, pH may drop over the gel's application²⁶. Therefore, contact of the enamel surface with a fluoride-containing desensitizer after acidic challenges may have reversed this condition.

Several studies have investigated enamel bond strength after at-home and in-office bleaching treatment¹⁰⁻¹³. Cavalli et al.¹⁰ (2001) evaluated the enamel bond strength

of an etch-and-rinse adhesive after bleaching with CP gels (10 to 20%) for ten days. The authors concluded that bond strength was recovered three weeks elapsed from bleaching and artificial saliva storage¹⁴. In contrast, others²⁷ reported that enamel treated with 10% CP and restored with self-etching adhesive exhibited similar bond strength to the control group 24 h after bleaching. Furthermore, bond strength after 35% HP bleaching presented similar values to untreated enamel after one week²⁷. Such discrepancies between studies^{14,27} could rely on differences in bonding and bleaching procedures. Nevertheless, no consensus exists regarding shear bond strength for bleached teeth bonded with self-etching adhesive⁹.

The predominance of cohesive failures in enamel (COE) in the CP groups at all times corroborates the action of HP byproducts on enamel. The decrease in enamel strength was previously reported even after CP treatment²⁴. According to that study, CP produced morphological alterations, with loss of enamel prism core, decreasing enamel cohesive strength²⁴. In addition, the predominance of adhesive failures of the CP/DES group at 7 and 14 days after bleaching indicates an increase in enamel resistance to fracture, which was probably promoted by remineralization. Conversely, Faria de Lacerda et al.²⁸ (2016) observed that 0.05% sodium fluoride remineralized enamel exhibited mixed failure modes for etch-and-rinsed and self-etched restorations. That study observed that the μ SBS of the F-treated group bonded with the self-etching system was lower than that of the control group²⁸. This contrast may be explained by the 8-week fluoride daily application, which may have hypermineralized the enamel.

In summary, the μ SBS behavior of bleached enamel and its recovery or even increase within the following weeks reaffirms previous data¹⁰⁻¹⁴. Due to fluoride incorporation in the tooth structure, the desensitizer may have enhanced enamel bond strengths. Since esthetic dental treatments frequently begin by tooth bleaching, these findings suggest that desensitizer application during bleaching could favor enamel bond strength of restorative procedures when postponed for two weeks.

The manufacturer recommends the desensitizer's application from 15 min to 1 h, but the frequency depends on the tooth condition, patient sensitivity, and professional decision (Table 1)²⁹. Because of that recommendation, several application times can be found in the literature (from 15 min³⁰ to overnight use³¹). In this study, the desensitizer's gel application occurred on the 7th and 14th days of bleaching for 8 h without intervals, which corresponds to 1 h 15 min of daily use. However, the prolonged application adopted is a limitation of this *in vitro* design and cannot be extrapolated to a clinical situation because of the risks of swallowing the desensitizer. Furthermore, authors have attested³² that the shear bond test also holds limitations because shear stress concentrates where the load is applied, causing an uneven tension distribution and a higher % of cohesive failures within the resin composite (COR), underestimating the μ SBS values. However, all groups exhibited a lower % of COR (10-30%), contradicting this idea and supporting the μ SBS test performed.

Clinically, a desensitizer agent containing fluoride and potassium nitrate attenuates the risk and intensity of tooth sensitivity¹⁹ during bleaching by obstructing dentin tubules and preventing depolarization of nerve fibers³³. Even though this study presents the inherent limitation of an *in vitro* design, it demonstrated that the desensitizer

might positively impact the bond strength of postponed procedures. Further investigations could attempt different protocols to determine an acceptable effect on immediate enamel bonding.

In conclusion, the combination of a desensitizer agent with bleaching gel (10% CP) upheld the enamel bond strength immediately after bleaching. The protocol combining low-concentrated CP with a desensitizer agent presented the highest bonding performance under a 14-day postponement.

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