Shear Bond Strength Evaluation of Composite Resin on Enamel and Dentin after Nonvital Bleaching

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ABSTRACT

Background: Studies have shown that bleaching agents interfere with the adhesion of composite resin placed immediately after bleaching.

Purpose: The aim of this study was to evaluate the shear bond strengths of composite resin after four different durations since the time of nonvital bleaching (1 d and 1, 2, and 3 wk).

Materials and Methods: Two hundred seventy bovine teeth were assigned to nine groups (n = 30) according to the bleach used and the time between bleaching and restoration: group 1—sodium perborate (SP), 1 day after bleaching (AB); group 2—SP, 1 week AB; group 3—SP, 2 weeks AB; group 4—SP, 3 weeks AB; group 5—37% carbamide peroxide (CP), 1 day AB; group 6—CP, 1 week AB; group 7—CP, 2 weeks AB; group 8—CP, 3 weeks AB; group 9—control group (no treatment). After the bleaching treatment, the teeth in each group were sectioned and the enamel and dentin were separated. The teeth were cut, embedded in polyester resin, and polished to obtain flat enamel and dentin surfaces. The adhesive system was applied and a cylinder of composite resin was bonded on each flat surface. The specimens were stored in distilled water for 7 days at 37°C. The shear bond strength test was performed in a universal test machine (Emic DL-500, São José dos Pinhais, São Paulo, Brazil) at a crosshead speed of 0.5 mm/min.

Results: The data were subjected to analysis of variance, Dunnett's t-test, and Tukey's least significant difference at $p \leq .05$ and showed a statistically significant decrease in bond strengths of composite resin for enamel and dentin 1 day after the nonvital bleaching.

CLINICAL SIGNIFICANCE

The decrease in shear bond strength values is time dependent. A delay in bonding procedures for composite resin restoration is recommended. According to the results, a satisfactory time to perform the restoration of composite resin for both enamel and dentin is 2 weeks after bleaching.

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One of the factors that contributes most to the success or failure of a smile is the color of an isolated tooth or of the teeth as a whole.[1] Discoloration of nonvital teeth is an esthetic problem that often requires treatment.[2] Bleaching permits a successful esthetic outcome at minimal expense while conserving tooth structure.[3] Endodontic therapy occasionally has the undesired effect of darkening a treated tooth.[4] Nonvital tooth bleaching has been shown to be an effective and conservative

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technique.[5] Thus, the bleaching treatment, which attempts to restore the normal shade of a tooth by decolorizing the stain with an oxidizing agent,[6] is an important phase of a restorative procedure.

According to the literature, the use of highly concentrated hydrogen peroxide has occasionally been associated with the development of external root resorption.[7–9] It has been considered that the diffusion of hydrogen peroxide may initiate a local inflammatory reaction through the radicular dentin into the periodontal ligament in the presence of cementum defects.[8] Consequently, for nonvital tooth bleaching, as recommended by Spasser, use of a sodium perborate–water paste in the "walking bleach" technique has been the most popular and recommended approach.[6] The decomposition reaction of sodium perborate is slow and releases hydrogen peroxide (approximately 9%) in a low concentration, resulting in a wider margin of safety when compared with other techniques.[2,6,8,9] Another bleaching agent that has been used and considered harmless in the walking bleach technique is 37% carbamide peroxide.[10,11] Carbamide peroxide does not require the use of heat, hence reducing radicular reabsorption risks.[10]

Nonvital bleaching is often followed by the placement of esthetic restorations. Several studies have demonstrated a decrease in bond strengths of composite resin to bleached enamel and dentin after the bleaching process.[11–16] The influence on bond strength may be due to an interaction between peroxide or peroxide-related substances and the resin at or near the enamel surface.[17] Lai and Nikaido and colleagues have suggested that the reduction in bond strength in hydrogen peroxide–treated dentin could be caused by a residual solution in the collagen matrix and dentinal tubules that eventually breaks down to oxygen and water.[18,19] Oxygen release could either interfere with resin infiltration into etched dentin and enamel, or inhibit the polymerization of resins.[18,20] However, Perdigão and colleagues have suggested that the residual oxygen may not be responsible for this effect, and that changes in proteins and mineral content of the most superficial layers of enamel may be responsible for the reduced bond strengths.[21]

Previous studies have stated that an increase in the elapsed time after bleaching decreases the adverse effects of hydrogen peroxide on bonding procedures.[5,12–14,20,22–24] To achieve a good adhesion, it is necessary to know the time that has elapsed since the bleaching treatment before the restoration procedure is performed.[11] The purpose of this study was to evaluate the shear bond strengths of a composite resin after nonvital bleaching using one of two bleaching agents (sodium perborate or 37% carbamide peroxide) after four different durations since bleaching (1 d and 1, 2, and 3 wk).

MATERIALS AND METHODS

Specimen Preparation to Bleaching Treatment

Two hundred seventy freshly extracted bovine teeth were selected, cleaned, and stored in 0.1% thymol solution prior to the study. Each

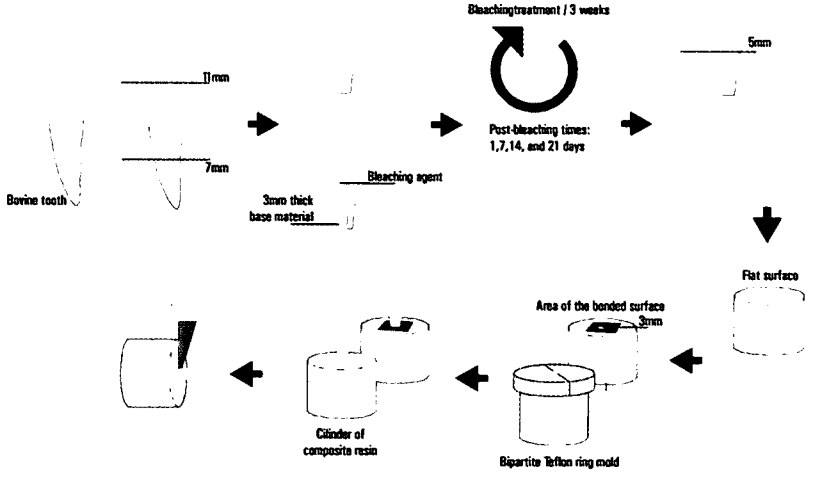

Figure 1. Methodology used in the experiment.

tooth was horizontally sectioned approximately 11 mm occlusal and 7 mm apical to the cementoenamel junction (Figure 1) using a double-faced diamond disk (KG Sorensen, Barueri, São Paulo, Brazil). The pulp was removed with a dental probe, and the pulp chamber was enlarged to a standard size with a spherical diamond bur (Metalúrgica Fava, Franco da Rocha, São Paulo, Brazil) using a low-speed handpiece (Kavo do Brasil S/A, Joinville, Santa Catarina, Brazil).

Bleaching Treatment

A 3 mm thick base material (Cavitec, DentalTEC, Joinville, Santa Catarina, Brazil) was placed in the root canal and leveled 2 mm below the cementoenamel junction to prevent apical leakage of the bleaching material during the walking bleach technique. The apical region was sealed with the adhesive system Single Bond (3M ESPE, St. Paul, MN, USA) and composite resin Fill Magic (Vigodent, Bonsucesso, Rio de Janeiro, Brazil). The teeth were stored in a humidor at 37°C prior to the bleaching treatment.

The specimens were randomly assigned to nine groups (n = 30), according to the bleaching treatment and the time delay between bleaching and restoration (Table 1). The control group was not bleached and was stored in artificial saliva at 37°C prior to the restoration. The bleaching treatment was conducted with consideration of different wait times so that all groups could be restored simultaneously.

The walking bleach technique was used for both sodium perborate (2 g/1 mL distilled water; Proderma Farmácia de Manipulação Ltda, Piracicaba, São Paulo, Brazil) and 37% carbamide peroxide gel (Super Endo Whiteness, FGM Produtos Odontológicos, Joinville, Santa Catarina, Brazil). The bleaching materials were inserted in the pulp chamber, and a 1.5 mm thick surface seal was made with a temporary material, Cavitec. The bleaching agents were changed every 7 days for 3 weeks. The teeth were stored in artificial saliva at 37°C during the bleaching period.

Specimen Preparation to Shear Bond Strength Test

After the bleaching treatment, the specimens were sectioned and the fragments obtained were embedded in a self-curing polystyrene resin (Piraglass, Piracicaba, São Paulo, Brazil). The embedded specimens were polished on a water-coolant mechanical grinder (Maxigrind, Solotest, São Paulo, São Paulo, Brazil) using Al_2O_3 sandpapers (Carborundum Abrasivos, Recife, Pernambuco, Brazil) to expose flat surfaces areas of 5 to 6 mm^2. Half the specimens of each group were ground to obtain a flat dentin surface, and the other half were ground to obtain a flat enamel surface. Subsequently, an area of 3 mm in diameter was left uncovered as a bonding site by placing a fenestrated polyvinyl chloride film with a 3 mm diameter hole over the flat surface (dentin and enamel).

Restorative Procedures

Single Bond adhesive system and Filtek Z250 (3M ESPE), a hybrid composite resin, were used according to the manufacturer's instructions for bonding procedures.

TABLE 1. BLEACHING AGENTS USED AND DURATIONS BETWEEN BLEACHING AND RESTORATION.

Group (n = 30)	Bleaching Treatment	Duration between Bleaching and Restoration
1	Sodium perborate	1 d
2	Sodium perborate	1 wk
3	Sodium perborate	2 wk
4	Sodium perborate	3 wk
5	37% carbamide peroxide	1 d
6	37% carbamide peroxide	1 wk
7	37% carbamide peroxide	2 wk
8	37% carbamide peroxide	3 wk
9	No treatment—control group	—

A piece of tape with a hole (3.0 mm in diameter) was attached to the specimen surface in all groups to limit the area of the bonded surface. The flat surface (enamel or dentin) was etched for 15 seconds with 35% phosphoric acid gel, rinsed with water for 15 seconds, and dried briefly, leaving a moist surface. Two consecutive coats of the adhesive were applied, lightly air dried for 2 seconds, and light cured (Optilux 500, Demetron-Kerr, Danbury, CT, USA) for 10 seconds.

After the application of the bonding agent, a bipartite polytef ring mold with a circular hole of 3.0 mm in diameter and 5.0 mm deep was positioned over the treated flat surface (enamel or dentin). The mold was filled in bulk technique with the composite resin Filtek Z250, light cured for 40 seconds, and then light cured again for 40 seconds in the opposite direction after the mold was removed to ensure the degree of conversion. The specimens were immersed in distilled water and stored for 1 week at 37°C before testing.

Bond Strength Test

The shear bond strength was measured in a universal test machine (Emic DL-500, São José dos Pinhais, São Paulo, Brazil). A parallel knife-edge shearing device was aligned over the bonded interface, and the specimen was loaded to failure using a crosshead speed of 0.5 mm/min. Means and standard deviations were calculated and expressed in megapascals. The data were subjected to two-way analysis of variance with an additional treatment (with the nontreated control group). Dunnett's *t*-test was applied to compare the treated groups with the control group, and Tukey's least significant difference was used to compare the results of the two bleaching agents. The data of different times (1 d, 1, 2, and 3 wk) were analyzed by polynomial regression ($\alpha = .05$) (Figure 2).

RESULTS

Mean bond strengths and standard deviations are shown in Tables 2 and 3. For enamel, Dunnett's *t*-test showed a statistically significant decrease in bond strengths for groups 1 ($p = .03$), 2 ($p = .0007$), and 5 ($p = .004$) compared with group 9, the control group (no treatment). For dentin, Dunnett's *t*-test showed a statistically significant decrease in bond strengths for group 1 ($p = .019$) compared with group 9 (no treatment). The

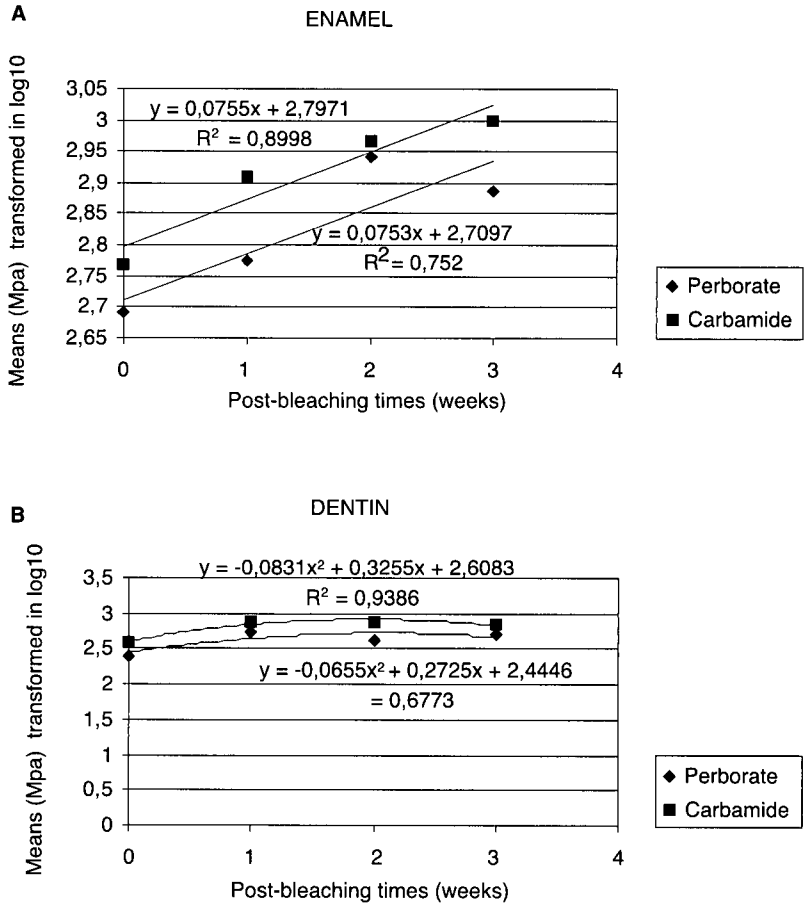

Figure 2. Polynomial regression for the time elapsed between bleaching treatments and restorations: A, *for enamel;* B, *for dentin.*

TABLE 2. MEAN VALUES OF SHEAR BOND STRENGTHS TO ENAMEL.

	Mean (SD) in MPa	
Time to Restoration*	After Sodium Perborate	After Carbamide Peroxide
1 d	14.96 (2.62)† b	16.48 (4.46)† a
1 wk	16.65 (3.36)† b	18.60 (3.85) a
2 wk	19.12 (2.98) b	20.00 (3.58) a
3 wk	18.47 (4.35) b	20.65 (5.26) a
None‡	20.07 (3.39)	

*Since bleaching with sodium perborate or carbamide peroxide.
†Indicates a statistical difference compared with the control group for both enamel and dentin. Statistical differences between bleaching agents are expressed by different letters within the rows.
‡Control group—no bleaching treatment.

results suggest that the bleaching treatment with sodium perborate and 37% carbamide peroxide interferes with the adhesion of composite resin restoration for both enamel and dentin.

Tukey's least significant difference showed higher bond strength values for teeth bleached with 37% carbamide peroxide compared with teeth bleached with sodium perborate for both enamel (p = .03) and dentin (p = .001).

The F-test revealed a statistically significant difference among groups with different durations after bleaching for both sodium perborate and 37% carbamide peroxide, regardless of the substrate (enamel and dentin). The graphics of polynomial regression demonstrated a statistically significant difference among treated groups for both enamel (p = .00018) and dentin (p = .00025) (see Figure 2). For enamel, the results suggest that after a 2-week period between the bleaching treatment (sodium perborate and carbamide peroxide) and the restorative procedure, bond strength is not significantly affected as compared with the control group. For dentin, the results suggested that after a 1-week period between the bleaching treatment with sodium perborate and the restorative procedure, bond strength is not significantly affected, and that the 37% carbamide peroxide agent does not interfere with shear bond strength values.

TABLE 3. MEAN VALUES OF SHEAR BOND STRENGTHS TO DENTIN.

	Mean (SD) in MPa	
Time to Restoration*	After Sodium Perborate	After Carbamide Peroxide
1 d	11.49 (3.09)† b	13.98 (4.85) a
1 wk	15.98 (3.45) b	18.03 (2.93) a
2 wk	14.41 (4.14) b	19.04 (4.75) a
3 wk	15.88 (5.29) b	16.98 (5.44) a
None‡	16.99 (6.80)	

*Since bleaching with sodium perborate or carbamide peroxide.
†Indicates a statistical difference compared with the control group for both enamel and dentin. Statistical differences between bleaching agents are expressed by different letters within the rows.
‡Control group—no bleaching treatment.

DISCUSSION

Nonvital bleaching treatment is a well-accepted clinical procedure. An important and necessary step after bleaching is often an esthetic restorative procedure. Several researchers have demonstrated that bleaching reactions interfere with bond strengths of adhesive systems and composite resins.[5,12,13,15,16,20,22,23,25] Shinohara and colleagues stated that nonvital bleaching increases the microleakage on dentin margins of composite resin restorations.[11]

The findings in this work have confirmed those of previous studies[5,12,13,15,16,20,22,23,25]: the bleaching treatment may indeed alter the substrate and interfere with the adhesion of composite resin restorations to both enamel and dentin. One hypothesis of how this occurs is that the presence of residual peroxides and oxygen may inhibit the polymerization process of the adhesive systems and adversely affect bond strengths.[15,26]

Our results have suggested that residual oxygen may be responsible for the decrease in bond strength.

The bleaching agent was applied in the pulp chamber, illustrating that it is able to interfere with the substrate even from a distance. Perdigão and colleagues suggested that the reduced bond strengths are due to the changes in the superficial layers of enamel.[21] Conversely, our data demonstrated that the reduction in shear bond strength values is more than a surface phenomenon—the bleaching agent spreads from dentin to enamel. Another possible explanation is that the hydrogen peroxide causes denaturation of proteins in the organic components of dentin and enamel, altering the organic-to-inorganic ratio with an increase in the inorganic component.[12]

According to the results obtained, the enamel substrate was vulnerable to the bleaching reactions. The shear bond strength values were lower compared to those of the control group (no treatment) after 2 weeks of bleaching treatment. In accordance with the results of Josey and colleagues and García-Godoy and colleagues, the characteristics of the etched enamel after bleaching seemed altered, which may have affected the bonding of composite resin to bleached enamel.[13,14] Bovine teeth were used in the present study. If an interprismatic presence of peroxide is the explanation for the adverse influence on resin adhesion, it is highly likely that this may be more marked in bovine enamel than it is in human enamel due to inherent differences in the structure and size of their interprismatic areas.[17]

Our data showed that a duration of 2 weeks after the bleaching treatment on enamel is sufficient prior to performing a restoration. Several studies have found that in vitro specimens stored in water or artificial saliva experience a complete reversal of the reduced enamel bonds.[18,24] This seems to be due to the leaching of hydrogen peroxide during the storage in artificial saliva after bleaching for both enamel and dentin. Demarco and colleagues reported an increase in the adhesion on bleached teeth after storage in distilled water. The hydrogen peroxide left on the dentin surface by the bleaching treatment loses its activity with time because of its instability.[20]

Regarding dentin, the results showed that shear bond strength returns to values similar to those of the control group 1 week after the bleaching treatment with sodium perborate. The 37% carbamide peroxide did not alter the shear bond strength of the adhesive system used. A possible explanation for the results in dentin is that the porous substrate and the peroxide residues may release the oxygen more easily than the enamel substrate does. Another reason may involve the methodology of the present study, which used the walking bleach technique. Unlike previous studies, in which the specimens were immersed in the bleaching agent or the bleaching agent was placed on the ground dentin surface,[16,20] in our study, after the bleaching treatment was finished, the specimens were polished to obtain a flat dentin surface. This procedure may have altered the dentin surface and removed residues of hydrogen peroxide. Furthermore, the reaction of carbamide peroxide is immediate, and the residues of hydrogen peroxide probably leach rapidly,[10] whereas sodium perborate releases less hydrogen peroxide and has a slower process.[27] This difference might explain the variety of results shown in several studies involving elapsed times between bleaching treatments and restorative procedures.

The present studies in the literature suggest that bleaching treatments alter bond strengths in vital and nonvital teeth. The reduction in adhesion can also induce microleakage, which is the main cause of color reversal after the bleaching procedure.[20]

Although the data showed that the technique of grinding the surface after bleaching may alter the dentin surface and remove its residues of hydrogen peroxide, this procedure is not recommended in the clinical situation because it is not a conservative technique. Therefore, a delay prior to the bonding procedure is a better alternative to achieve good adhesion on a bleached tooth.

The graphics of polynomial regression (see Figure 2) demonstrate a statistically significant difference

among groups with different durations after bleaching for both sodium perborate and 37% carbamide peroxide, regardless of the substrate (enamel and dentin). The drop in the values of shear bond strengths on dentin after 2 weeks is due to the instability of the bleaching agent components in the substrate, mainly the oxygen residues. However, these values were not statistically lower than the values in the control group.

The decrease in shear bond strength values is time dependent. A delay in bonding procedures prior to composite resin restoration is recommended. According to the results, 2 weeks is a satisfactory waiting period between bleaching and performing restorations with composite resin.

CONCLUSIONS

The following conclusions can be drawn from this study:

- The decrease in shear bond strength values after nonvital bleaching is time dependent.
- A satisfactory period of time to wait between nonvital bleaching treatment and performing a restoration with composite resin for both enamel and dentin is 2 weeks.
- Teeth bleached with 37% carbamide peroxide have higher shear bond strength values than do teeth bleached with sodium perborate.
- Bleaching agents spread from dentin to enamel, thus interfering in the resin-tooth interface.

DISCLOSURE AND ACKNOWLEDGMENTS

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COMMENTARY

SHEAR BOND STRENGTH EVALUATION OF COMPOSITE RESIN ON ENAMEL AND DENTIN AFTER NONVITAL BLEACHING

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Following whitening procedures, it is often recommended to wait at least 2 weeks prior to placing a direct esthetic restoration to allow any residual oxygen left in the tooth to be eliminated. It is well known that residual oxygen can interfere with the polymerization of the resin and thus affect the bond strength to enamel and dentin.

Occasionally following endodontic therapy, the tooth discolors and requires either the placement of a restoration or bleaching of the tooth prior to the final restorative procedure. Nonvital tooth whitening using a paste placed in the pulp chamber and left for several days is a commonly used procedure. The above article describes the effects of sodium perborate and carbamide peroxide on the bond strengths of enamel and dentin at 1 day and 1, 2, and 3 weeks after bleaching.

The study clearly demonstrates the negative effect of residual oxygen on enamel and dentin after 24 hours; however, it does not present a statistical difference in bond strengths after 1 week for either carbamide peroxide or sodium perborate. Is interesting to note the diffusion of the hydrogen peroxide into the enamel and how it affects the bond strength, at least in the first 24 hours after the bleaching procedure.

Two factors not tested in this study are the effect of bleaching on the immediate bond strength after 10 minutes or less and the effect of bleaching on microleakeage. These values are of great clinical importance since they are indicators of the potential bond strength in restorations with a high C-factor. Also unknown is the effect of bleaching on microleakage. Perhaps a future study should be performed using a push test instead of a shear test method; this might be a more clinically relevant method of evaluating the effect of bond strength on dentin.

Of great importance for future research is the evaluation of a combination of 10 to 15% carbamide peroxide and sodium perborate used over a 2- to 3-week period. This paste is fairly popular with clinicians who want to increase the whitening effect on the tooth without increasing the potential for external root resorption.

It would appear from this article and from what is described in the literature that oxygen has a deleterious effect on the initial bond strengths of enamel and dentin to composite.

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