The Effect of Nonvital Bleaching on the Shear Bond Strength of Composite Resin Using Three Adhesive Systems

Mirela Sanee Shinoharaa/Alessandra Rezende Perisa/José Augusto Rodriguesa/Luiz André Freire Pimentab/Gláucia Maria Bovi Ambrosanoc

Purpose: To evaluate the effect of nonvital bleaching on the shear bond strength of composite resin using three adhesive systems.

Materials and Methods: Two hundred seventy bovine teeth were assigned to 3 groups (n = 90): SP, paste of sodium perborate and water; CP, 37% carbamide peroxide; CO, control group (no treatment). After the bleaching treatment, the teeth in each group were cut into enamel (E) and dentin (D) sections. The teeth were embedded in polyester resin and polished in order to obtain a flat E or D surface. Each group was divided into 6 subgroups (n = 15) according to substrate (E and D) and adhesive system: SB, Single Bond (solvent: water/alcohol); PB, Prime & Bond NT (solvent: acetone); CLF, Clearfil SE Bond (solvent: water). The adhesive system was applied on each flat surface according to the manufacturer's instructions and a cylinder of composite resin Z-250 was overlaid. The specimens were stored in distilled water for 7 days at 37°C. The SBS test was performed in a universal testing machine with crosshead speed of 0.5 mm/min. The data obtained (in MPa) were statistically analyzed with two-way ANOVA and Tukey's test (p ≤ 0.05).

Results: The bleaching treatment influenced the SBS, regardless of the adhesive system used. On enamel, the SP and CP bleaching treatments reduced the SBS values. On dentin, the SP bleaching agent reduced the SBS values, but the CP bleaching did not.

Conclusion: Nonvital bleaching treatment with sodium perborate may adversely affect shear bond strength of composite resin for both enamel and dentin. Similar effects can be expected from 37% carbamide peroxide bleaching agent on enamel but not dentin bonding strength. The use of water/alcohol and acetone-based adhesive systems cannot reverse the effects of bleaching treatments on bond strengths.

Key words: bleaching agent, bond strength, adhesive system.

J Adhes Dent 2004; 6: 205–209. Submitted for publication: 10.06.03; accepted for publication: 23.09.03.

Discoloration of anterior teeth is a serious esthetic problem in restorative dentistry, and it requires effective treatment. Bleaching is the most conservative and economical alternative for improving the appearance of the discolored nonvital tooth. In this technique, the bleaching agent is sealed in the pulp chamber for 1 week. This procedure is repeated 3 to 4 times, and each time, fresh bleaching agent replaces the previous one.

The use of sodium perborate mixed with water is the most accepted bleaching agent to reduce the risk of cervical root resorption. Sodium perborate slowly releases
MATERIALS AND METHODS

Bleaching Treatment

Two hundred seventy freshly extracted bovine teeth were selected, cleaned, and stored in 0.1% thymol solution prior to the study. Each tooth was horizontally sectioned approximately 11 mm occlusally and 7 mm apically to the cementoenamel junction using a double-faced diamond disk (KG Sorensen, Barueri, SP, Brazil). The pulps were removed with a dental probe, and the pulp chamber was enlarged to a standard size using a spherical diamond bur (#1016HL, Metalúrgica Fava, Franco da Rocha, SP, Brazil) in a low-speed handpiece (Kavo do Brasil S/A, Joinville, SC, Brazil).

A 3-mm-thick base material (IRM, Dentsply, Petrópolis, RJ, Brazil) was packed into the root canal to a level 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 epoxy resin (Araldite Ciba Especialidades Química, Taboão da Serra, SP, Brazil). The teeth were stored in a humidor at 37°C prior to bleaching treatment.

The specimens were randomly assigned into 3 groups (n = 90) according to the bleaching agent used: PS, sodium perborate; PC, 37% carbamide peroxide; CO, control (not bleached). The teeth were stored in artificial saliva at 37°C before restoration.

The walking bleach technique was used with both bleaching agents: sodium perborate (Proderma Farmácia de Manipulação, Piracicaba, SP, Brazil) 2 g/1 ml water, and 37% carbamide peroxide gel (Super Endo Whiteness, FG Produtos Odontológicos, Joinville, SC, Brazil). The bleaching materials were inserted in the pulp chamber, and a 1.5-mm-thick surface seal was made with a temporary material (IRM). The bleaching agents were changed every 7 days for three weeks. The teeth were stored in artificial saliva at 37°C during the bleaching period.

Specimen Preparation for Shear Bond Strength Test

After the bleaching treatment, the specimens were sectioned and the sections obtained were embedded in artificial material (IRM). The bleaching agents were changed every 7 days for three weeks. The teeth were stored in artificial saliva at 37°C during the bleaching period.

Restorative Procedures

Subsequently, the specimens of each group (PC, PS, and CO) were divided into 6 subgroups (n = 15) according to the adhesive system used: Single Bond (SB) (3M, St Paul, MN, USA); Clearfil SE Bond (CLF) (Kuraray, Osaka, Japan); Prime & Bond NT (PB) (Dentsply, Petrópolis, RJ, Brazil), and untreated flat surface (dentin and enamel). Each adhesive system was applied according to the manufacturer’s instructions (Table 1).

After the application of the bonding agent, a bipartite Teflon ring mold with a circular hole of 3.0 mm in diameter and 5.0 mm in depth was positioned over the treated

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Table 1 Application procedures of adhesive systems

<table>
<thead>
<tr>
<th>Adhesive System</th>
<th>Procedures</th>
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</thead>
<tbody>
<tr>
<td>Single Bond (SB)</td>
<td>Etching (15 s), rinse (15 s), blot dry, apply adhesive in 2 consecutive coats, air thin gently (2 to 5 s), light cure (10 s)</td>
</tr>
<tr>
<td>Clearfil SE Bond (CLF)</td>
<td>Light air drying, apply Primer, wait 20 s, evaporate with light air flow, apply Bond, gently air blow, light cure (10 s)</td>
</tr>
<tr>
<td>Prime &amp; Bond NT (PB)</td>
<td>Etching (15 s), rinse (15 s), blot dry, apply 1 coat of the adhesive, air thin gently (2 to 5 s), light cure (10 s)</td>
</tr>
</tbody>
</table>

hydrogen peroxide in low concentration. Another bleaching agent that can be used is 37% carbamide peroxide, which is considered efficient and harmless when applied in the walking bleach technique. According to the literature, the use of hydrogen peroxide for bleaching has occasionally been associated with the development of external root resorption.

In nonvital teeth, the bleaching agent diffuses from the pulp chamber dentin into the enamel, producing an oxidation reaction in the discolored dentin which results in whitening. After the bleaching treatment, previous restorations may need replacement to achieve a correct shade match.

Several studies have shown the adverse effects of bleaching treatment on bond strength between composite resin and tooth substrate. Some authors have suggested that a reduction in bonding strength of composite resin in bleached teeth may be due to the presence of active chemicals from bleaching. Residual oxygen may be responsible for the inhibition of resin polymerization and increase in resin porosity.

Several researchers have found that optimal bond strengths can be achieved with a time delay after the bleaching. According to some findings, the use of alcohol-based bonding agents may decrease the effect on the strength of composite resin after nonvital bleaching treatment. Several studies have shown the adverse effects of bleaching treatment on bond strength when restorative work is to be completed immediately after bleaching. The presence of alcohol may counteract any residual water and oxygen from the bleaching agent.

The purpose of this study was to evaluate the effect of three different adhesive systems on the shear bond strength of composite resin after nonvital bleaching treatment.

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flat surface (enamel and dentin). The mold was filled in bulk technique with composite resin Z-250 (3M) and light cured (Optilux 500/Demetron-Kerr, Danbury, CT, USA) for 40 s, and then light cured again for an additional 40 s from the opposite direction after the mold was removed, in order to ensure a maximum degree of conversion.

The specimens were immersed in distilled water and stored for one week at 37°C before testing.

**Bond Strength Test**
The shear bond strength was measured in a universal testing machine (Emic DL-500, São José dos Pinhais, SP, Brazil). A parallel knife-edge shearing device was aligned over the bonded interface and the force was loaded to failure, using a crosshead speed of 0.5 mm/min. Means and standard deviations were calculated with units expressed in MPa. The data were subjected to two-way ANOVA and Tukey’s test ($\alpha = 0.05$).

Workflow is depicted in Figure 1.

**RESULTS**
Mean bond strengths and standard deviations on enamel and dentin are shown in Tables 2 and 3, resp.

The two-way ANOVA showed no significant interaction between bleaching agent and the adhesive system used on SBS for either enamel or dentin. Statistically significant differences among the groups were indicated ($p \leq 0.05$). The differences were evaluated using Tukey’s test.

On enamel, the bleaching treatments with SP and CP reduced the bond strength values. On dentin, the bleaching treatment with SP reduced the bond strength values, but CP did not.

The results indicated that the bleaching treatment on enamel and dentin reduced bond strengths, independent of the adhesive system used. Single Bond and Clearfil SE Bond showed significantly higher mean bond strengths than did Prime & Bond NT for bleached and unbleached dental tissues.

**DISCUSSION**
After nonvital bleaching treatment, the restorative procedure is important to achieve a pleasant appearance, since the color of composite resin must be compatible with the bleached tooth. Moreover, the adhesion of the composite resin restoration to the tooth should be able to prevent microleakage and withstand the forces exerted during mastication.

Previous investigations have demonstrated that the bleaching agent interferes with the adhesion of composite resin restoration to dental tissues and also contributes to the susceptibility of the tooth surface to microleakage. Our results have confirmed those of other studies. The bleaching treatment interferes with the shear bond strength of composite resin restorations independent of the adhesive system used.

In this study, three adhesive systems were evaluated immediately after nonvital bleaching treatment. According to some researchers, the type of bonding agent could improve the adhesion on bleached teeth. Kalili et al suggested that the application of an alcohol-based adhesive system may have been able to minimize the inhibitory effects of the bleaching treatment by the interaction of alcohol with residual oxygen and counteract any residual water and oxygen from the bleaching agent. An interaction of high-pressure solvents (acetone and ethanol)
with the higher concentration of water inside the enamel microstructure after bleaching is alleged to occur.21
According to the results obtained, Single Bond and Clearfil SE Bond showed significantly higher mean bond strength values than that obtained with Prime & Bond NT for bleached and unbleached teeth; however, there was no interaction between bleaching agent and the adhesive system used.

Some studies have suggested that the presence of residual peroxides and oxygen may be responsible for the decrease in bond strength.30,31 The hypothesis of Toko et al is that the adverse effect of hydrogen peroxide may be attributed to the removal of the nonfibrous organic content within the tooth substance.33 Hydrogen peroxide has been suspected to cause denaturation of proteins in the organic components of dentin and enamel, altering the organic:inorganic ratio with an increase in organic component.3

Rotstein et al23 indicated that most bleaching agents cause changes in the levels of calcium, phosphorus, sulfur, and potassium in the tissues. Calcium and phosphorus are present in the hydroxyapatite crystal, the main building block of dental hard tissues. Changes in Ca:P ratio indicate alterations in the organic components of hydroxyapatite.23 It seems that bleaching agents may adversely affect dental hard tissues. Perinka et al22 have found that the dentinal characteristics (dentin thickness, hardness, and calcium concentration) might influence the bond strength to dentin.22

Our data demonstrated that both bleaching agents (sodium perborate and 37% carbamide peroxide) reduced shear bond strengths to enamel when compared to the control group (no treatment). Titley et al,31 in an electron microscopic scanning evaluation, showed that the resin tags in 35% hydrogen peroxide treated enamel were sparse, shorter, poorly defined, and structurally incomplete.

Some authors have suggested that the decrease in bond strength may be due to bleaching agent residues inhibiting resin polymerization,30,35 and the released oxygen could interfere with the resin infiltration into etched enamel.11,30,31 Conversely, Perdigão et al21 stated that the residual oxygen may not be responsible for this effect. The changes in proteins and in mineral content of the most superficial layers of enamel may be responsible for reduced bond strengths.

On dentin, the use of SP decreased shear bond strength values. Lai et al17 have suggested that the reduction in bond strength in hydrogen peroxide-treated dentin could be caused by residual solution in the collagen matrix and dentinal tubules that occasionally broke down to oxygen and water. Liberation of oxygen could either interfere with resin infiltration into etched dentin, or inhibit polymerization of resins.17

Carbamide peroxide did not interfere in the adhesion of composite resin with dentin. The reaction of carbamide peroxide is immediate, and it is probable that the residues of hydrogen peroxide leach rapidly.5 Sodium perborate releases less hydrogen peroxide and has a slower reaction speed.13 Furthermore, dentin is a porous substrate, and the peroxide residues of CP may release the oxygen more easily to the dentin tubules than to enamel. Nakamichi et al demonstrated that bovine coronal dentin possessed larger dentin tubules than did human dentin.19 Therefore, the bovine dentin is more porous and the peroxide residues could spread more easily than in human dentin.

Physical properties of dentin vary with distance from the pulp.7,20 The effect of the bleaching agent is likely to be more pronounced in the inner dentin, decreasing as it approaches the dentinocemental junction.7 In this study, only the outer dentin was evaluated. The procedure of polishing specimens to obtain a flat outer dentin surface could have removed residues of hydrogen peroxide and decreased the action of bleaching agent on bonding strength. According to Chng et al,7 the variation in bleaching agent effect in terms of the dentin loca-

### Table 2 Mean (SD) shear bond strengths in MPa to enamel

<table>
<thead>
<tr>
<th>Bleach</th>
<th>Single Bond</th>
<th>Clearfil SE Bond</th>
<th>Prime &amp; Bond NT</th>
</tr>
</thead>
<tbody>
<tr>
<td>37% carbamide peroxide</td>
<td>20.23 (6.30) B a</td>
<td>19.14 (9.23) B a</td>
<td>11.93 (5.74) B b</td>
</tr>
<tr>
<td>Sodium perborate</td>
<td>16.56 (5.96) B a</td>
<td>21.62 (4.72) B a</td>
<td>10.92 (5.10) B b</td>
</tr>
<tr>
<td>Control (no treatment)</td>
<td>21.44 (5.01) A a</td>
<td>23.69 (5.73) A a</td>
<td>17.46 (7.42) A b</td>
</tr>
</tbody>
</table>

Groups that were statistically different from each other according to two-way ANOVA and Tukey tests are indicated by different capital letters (per column) and small letters (per row).

### Table 3 Mean (SD) shear bond strengths in MPa to dentin

<table>
<thead>
<tr>
<th>Bleach</th>
<th>Single Bond</th>
<th>Clearfil SE Bond</th>
<th>Prime &amp; Bond NT</th>
</tr>
</thead>
<tbody>
<tr>
<td>37% carbamide peroxide</td>
<td>19.88 (5.56) A a</td>
<td>18.54 (6.20) A a</td>
<td>15.41 (6.13) A b</td>
</tr>
<tr>
<td>Sodium perborate</td>
<td>14.55 (2.58) B a</td>
<td>18.41 (4.69) B a</td>
<td>8.50 (4.56) B b</td>
</tr>
<tr>
<td>Control (no treatment)</td>
<td>19.09 (3.30) A a</td>
<td>22.29 (5.11) A a</td>
<td>11.40 (4.58) A b</td>
</tr>
</tbody>
</table>

Groups that were statistically different from each other according to two-way ANOVA and Tukey’s test are indicated by different capital letters (per column) and small letters (per row).
tion is likely to be related to several factors, such as the pH of the bleaching agents, the buffering capacity of dentin, and the increasing diameter and density of dentinal tubules near the pulp.

Another study in our laboratory showed that nonvital bleaching increased the microleakage in restorations with dentin margins, but not in those with enamel margins. It should be emphasized that the different methods of bleaching and adhesion employed in the different studies – such as the concentration of the bleaching agent, the technique of bleaching treatment, the preparation of the substrate, the enamel or dentin substrate, the period of bleaching, the test used, and the performance of the restoration – may influence the results obtained and their comparability.

Even with these differences, the literature has demonstrated that restoration procedures performed after bleaching require caution. Several studies have confirmed that composite resin restoration performed immediately after bleaching treatment results in shear bond strength decreases on enamel and dentin. This seems to be due to the leakage of hydrogen peroxide into the water. There is a delay in bonding procedures for the leaching of hydrogen peroxide into the water. There is likely to be related to several factors, such as the period of bleaching, the test used, and the performance of the restoration – may influence the results obtained and their comparability.

REFERENCES


Clinical relevance: Since bleaching interferes with bonding, a time delay between bleaching and bonding procedures is recommended when composite resins are used.