Influence of Provisional Cements on Ultimate Bond Strength of Indirect Composite Restorations to Dentin

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\textbf{Purpose:} The aim of this study was to evaluate the effect of provisional cements on the adhesion of resin bonded indirect restorations and determine the best method for avoiding adverse effects.

\textbf{Materials and Methods:} Forty-five bovine incisors were selected, and the enamel removed with a 600-grit SiC abrasive disk to expose superficial dentin. Provisional restorations of acrylic resin were cemented with three different provisional cements: calcium hydroxide cement, Dycal (HC); cement containing zinc oxide-eugenol, Provy (ZOE); zinc oxide eugenol-free cement, TempBond NE (ZNE). The specimens were stored at 100% humidity, 37°C. Then provisional restorations were removed with: (1) hand scaler for 10 s; (2) pumice-water slurry for 10 s; (3) aluminum oxide sandblasting for 10 s. The indirect restorations were subsequently cemented with Single Bond and Rely-X ARC. The teeth were sectioned, 4 slices per tooth \((n = 16)\), and each slice trimmed with a diamond bur to obtain an adhesion area of 1 mm\(^2\). The microtensile bond strength test was performed with a universal testing machine (Instron-4411) at a crosshead speed of 0.5 mm/min. The results were analyzed with ANOVA, followed by Tukey’s test \((p < 0.05)\).

\textbf{Results:} ANOVA showed significant differences in the interaction between provisional cement and dentin cleaning method; in general, aluminum oxide sandblasting provided the highest values of bond strength and calcium hydroxide the lowest.

\textbf{Conclusion:} The type of provisional cement and its method of removal can affect the adhesion of resin-bonded indirect restorations.

\textbf{Key words:} provisional cements, dentin cleaning method.

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The intensive search for esthetic procedures has stimulated changes in restorative dentistry.\textsuperscript{45} Buonocore\textsuperscript{3} laid the foundation for adhesive dentistry, and Nakabayashi and co-workers\textsuperscript{26} described the hybrid layer, an interdiffusion zone between resinous monomers of the adhesive system and the conditioned dentin surface.\textsuperscript{9} This type of adhesion to dentin is believed to achieve bond strength values of ca 20 to 30 MPa\textsuperscript{33,39} or higher.\textsuperscript{22}

Different factors can affect the bond strength to tooth structure: exposed dentin intertubular area, diameter and number of exposed tubules, number and extension of secondary lateral tubules, capability of the dentinal bonding system to moisten and infiltrate the conditioned dentinal surface, ie, surface tension, and surface energy of the tooth substrate.\textsuperscript{6,13} When a procedure makes the surface energy of the tooth substrate higher than surface tension of the adhesive system, the consequence is a better dentin wettability. This facilitates the infiltration of the adhesive system into the collagen fiber net exposed by acid etching, which is the chief mechanism of adhesion to dentin.\textsuperscript{10,43,44}

The presence of surface impurities, such as components and residue of provisional luting agents\textsuperscript{2} not
effectively removed from the dentinal substrate, might impair the infiltration of the adhesive system or even inhibit the polymerization of resinous monomers. Eugenol, 2-methoxy-4-allyphenol, a component of provisional materials used in indirect restoration fixation and cavity sealing, has been shown to adversely affect many of the physical properties of composites. This substance softens composite resin, decreases transverse bond strength, decreases surface hardness, increases surface discoloration and roughness, decreases the shear bond strength of resin to resin, and has also been shown to increase the gap width between the dentin bonding agent and the tooth.

The negative influence of provisional cements, with or without eugenol, on the bond strength of composites to tooth substrates is believed to be related either to the presence of provisional cement residues not completely removed before cementation or to the effect of eugenol on the mechanical properties of the composites. Thus, the effect of the dentin cleaning method is a factor which may interfere with dentin adhesion. Mechanical cleaning of tooth substrate is common, although some authors question it, recommending a combination of chemical and mechanical cleaning to remove not only cement residues but also residual eugenol that has infiltrated the dentin.

Since the literature provides contradictory information on this topic, the aim of this study was to verify the influence of different provisional eugenol-containing or eugenol-free cements, and different methods of cleaning dentinal substrate on the ultimate bond strength of resinous cement to bovine dentin.

**MATERIALS AND METHODS**

Forty-five freshly extracted, intact, sound bovine incisors were selected and stored in 0.2% thymol solution until use. Residual soft tissue was mechanically removed and the teeth were cleaned with a pumice-water slurry and stored in 0.9% NaCl. The specimen preparation for the microtensile bond strength test followed the method of Sano et al (Fig 1). The teeth were sectioned about 2 mm below the cementoenamel junction and the buccal surface was ground with 600-grit silicon carbide paper under a stream of running water to form a flat superficial dentin surface, which was gently trimmed for 5 s with a #1090 diamond bur (Kg Sorensen, São Paulo, Brazil) to standardize the hybrid layer formation. The teeth were fixed on an acrylic support, keeping the ground surface upward, parallel to the support. Provisional restorations were made in a silicon matrix (3 x 3 x 5 mm) with acrylic resin (Dencor, Jet Classic, São Paulo, Brazil). After that, these restorations were fixed with the following provisional cements: G1, calcium hydroxide cement (Dycal, Dentsply/Caulk, Milford, DE, USA); G2, zinc oxide-eugenol cement (Provy, Herpo/Dentsply, São Paulo, Brazil); G3, zinc oxide eugenol-free cement (TempBond NE, Kerr, Orange, CA, USA), resulting in 15 teeth per group. Materials used in this study are listed in Table 1. The cements were mixed according to the manufacturers’ instructions and applied on the restoration base, which was seated over a delimited dentinal area and allowed to set under a load of 500 g. Excess cement was removed and the specimens stored at 37°C and 100% humidity for 7 days.

After the period of storage, the provisional restorations were removed and the dentin was cleaned by the follow-
ing methods: A, hand scaler for 10 s; B, pumice-water slurry for 10 s; C, sandblasting with 50-µm aluminum oxide particles for 5 s at a pressure of 4 bars and a source-to-sample distance of 2 cm (Microjato Plus, Bio-Art, São Paulo, Brazil). Five samples from each provisional cement group were cleaned with only one of the different cleaning methods. The definitive laboratory composite restorations were made with Filtek Z-250 (3M-ESPE, St Paul, MN, USA) in a silicon matrix (3 x 3 x 5 mm), incrementally light cured with a XL-3000 device (3M-ESPE) and postpolymerized with a multifocal laboratory light source, EDG-LUX (EDG, São Paulo, Brazil), for 7 min. The dentinal surface was washed with water spray for 5 s, dried and acid etched with 35% phosphoric acid (per volume) for 15 s; then it was washed with water spray for 15 s and the water excess removed with an autoclaved tissue paper. The adhesive system Single Bond (3M-ESPE) was applied and left untouched for 20 s, followed by a second application. The adhesive system was polymerized for 20 s with XL-3000. A dual-curing resinous cement, Rely-X ARC (3M-ESPE), was mixed and applied on the base of the composite restoration, which was then seated under a load of 500 g for 5 min over the delimited dentinal area previously occupied by the provisional restoration. The cement excess was removed and the polymerization performed for 40 s on each side of the indirect restoration. The samples were stored in 37°C and 100% humidity for 24 h. The teeth were sectioned on a serial cutter (ImpTech PC10, Equilam, Diadema, SP, Brazil) with a diamond disk (Extect 4”, 0.12 x 0.12) producing 4 slices of 1 mm per tooth (n = 16). Each slice was trimmed with a #1090 diamond bur in order to obtain a 1-mm² adhesion area.

The samples were stored at 37°C and 100% humidity. The samples were fixed in a universal testing machine (Instron-4411, Instron Testing Instruments, Canton, MA, USA) on a microtensile support with a cyanoacrylate adhesive (Super Bonder; Loctite, São Paulo, Brazil) and its accelerator (Loctite 7452). The microtensile bond strength test was performed at a crosshead speed of 0.5 mm/min until failure. The ultimate bond strength of each sample was recorded in kg/force and converted to MPa.

The bond strength values were submitted to a statistical analysis to verify whether a parametric analysis could be used. As data presented a normal and homogeneous distribution, two-way ANOVA (3 x 3) and Tukey’s test (p < 0.05) were employed.

RESULTS

Two-way ANOVA (p < 0.05) showed statistically significant differences among the types of provisional cements and the tooth substrate cleaning methods; it also showed a significant interaction between these factors. Then Tukey’s test was applied (p < 0.05) to the interaction of these two factors. Concerning the provisional cements, all groups had a similar behavior irrespective of the tooth substrate cleaning method: sandblasting with aluminum oxide achieved significantly higher values of bond strength than mechanical cleaning with a hand scaler, and these two cleaning methods did not differ statistically from pumice-water slurry (Table 2). Concerning the tooth substrate cleaning methods, each group of provisional cement showed one behavior: the groups cleaned by aluminum oxide sandblasting did not show differences among themselves; the groups cleaned by pumice-water slurry showed higher values of bond strength for G2 and lower values for G1, and both groups were similar to the one fixed with zinc oxide eugenol-free cement (G3). The groups cleaned with the hand scaler showed higher values of bond strength for G3, lower values for G1, and both groups were similar to G2 (Table 3).

DISCUSSION

A great number of studies have been conducted to evaluate the effect of eugenol or cement residues on the adhesive procedures, resulting in highly contradictory statements. Therefore, this study aimed to elucidate the influence of provisional cements, with or without eugenol, on the bond strength of indirect composite restorations. In addition, a dentin cleaning method was sought that would provide higher bond strength.

It has been showed that a chelating reaction occurs when zinc oxide is mixed with eugenol, resulting in grains of zinc oxide embedded in a zinc eugenolate matrix. Since all of the eugenol reacts, none is available for diffusion through dentin. However, due to the water content in the dentinal tubules, eugenol becomes able to penetrate dentin, achieving a concentration of about 10⁻² molar adjacent to a ZOE/dentin interface. It is still unclear whether this eugenol concentration can cause any ad-
verse effect on the adhesive procedures.\textsuperscript{11} Powell et al\textsuperscript{34} reported that the effects occurring at the eugenol-composite interface may be too small to cause deterioration of the composite’s bulk properties. Kustz et al\textsuperscript{17} showed that the eugenol-containing sealer had no effect on bond strength of tooth-colored posts. Jung et al\textsuperscript{15} found that acid etching removes about 10 µm of superficial enamel, where eugenol from provisional cements should be found; thus, eugenol might not affect the bond strength. Besides this, eugenol is able to demineralize dentin, releasing calcium, which lowers its diffusion rate through dentin and may decrease the release rate from dentin towards a composite material as well. Acid etching demineralizes dentin more deeply than does eugenol, and removes residual eugenol.\textsuperscript{11} It should be remembered that the effect of eugenol is documented,\textsuperscript{21,24,25,42} but it was absent after 7 days of sample storage in this study.

Tjan and Nemetz,\textsuperscript{42} however, reported lower bond strength for indirect restorations, even after 7 days of sample storage. They applied 0.04 ml of pure eugenol on the dental substrate, rendering a relative eugenol quantity higher than the one obtained when the same eugenol quantity is mixed with zinc oxide. When eugenol is one of the components of provisional cements, it is largely consumed during the material’s setting; after that, any remaining eugenol is able either to penetrate dentin\textsuperscript{11} or be eliminated into the oral environment. Mayer et al\textsuperscript{24} reported that lower bond strengths were related to the eugenol effects and not to cement residues. When they used pure eugenol instead of provisional cements containing eugenol, bond strength was reduced. In addition, when the definitive insertion was performed 24 h after having used a provisional cement, there was no negative effect on bond strength.

In this study, after the period of 7 days, the eugenol from provisional cements did not affect bond strength to dentin. Therefore, the association of mechanical and chemical cleaning methods is not necessary, since normally a definitive restoration is fixed 7 to 10 days after the fixation of the provisional one, the period of time necessary for the laboratory to make the restoration. In spite of the fact that this study did not employ a control group, the bond strength always achieved acceptable values, irrespective of the cleaning method, assuming effective adhesion to human dentin lies between 20 and 30 MPa\textsuperscript{33,39} or even higher.\textsuperscript{22} Mak et al\textsuperscript{22} obtained values of 34.50 (± 7.60) MPa for bond strength of indirect restorations cemented with Single Bond and Rely-X ARC on human teeth, which normally present higher values of bond strength than bovine teeth (15.70 ± 4.70).\textsuperscript{27} This study achieved high bond strength values on bovine dentin, similar to the ones obtained on human teeth on previous studies,\textsuperscript{22} justifying the employment of only an indirect technique in the groups. Thus, the experimental groups were based on only definitive cementation after the usage of a provisional cement, because in a clinical situation that involves a conventional indirect restoration, it is always necessary to place a provisional restoration.

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Bond strength means (MPa) and standard deviations for all cements, varying the cleaning methods (Tukey’s test, p &lt; 0.05)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Provisional cement</td>
<td>Cleaning methods</td>
</tr>
<tr>
<td>G3</td>
<td>Zinc oxide-eugenol-free</td>
</tr>
<tr>
<td>Pumice-water slurry</td>
<td>25.39 (3.10) ab</td>
</tr>
<tr>
<td>Hand scaler</td>
<td>24.25 (4.06) b</td>
</tr>
<tr>
<td>G2</td>
<td>Zinc oxide-eugenol</td>
</tr>
<tr>
<td>Pumice-water slurry</td>
<td>26.10 (5.63) ab</td>
</tr>
<tr>
<td>Hand scaler</td>
<td>20.73 (5.29) b</td>
</tr>
<tr>
<td>G1</td>
<td>Calcium hydroxide</td>
</tr>
<tr>
<td>Pumice-water slurry</td>
<td>20.36 (3.99) ab</td>
</tr>
<tr>
<td>Hand scaler</td>
<td>15.98 (3.22) b</td>
</tr>
</tbody>
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Different letters represent statistically significant differences.

<table>
<thead>
<tr>
<th>Table 3</th>
<th>Bond strength means (MPa) and standard deviations for cleaning methods, varying the types of cements (Tukey’s test, p &lt; 0.05)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Provisional cement</td>
<td>Cleaning methods</td>
</tr>
<tr>
<td>Al₂O₃ oxide sandblasting</td>
<td>Zinc oxide-eugenol-free</td>
</tr>
<tr>
<td>Zinc oxide-eugenol</td>
<td>28.31 (1.77) A</td>
</tr>
<tr>
<td>Calcium hydroxide</td>
<td>27.21 (3.71) A</td>
</tr>
<tr>
<td>Pumice-water slurry</td>
<td>Zinc oxide-eugenol-free</td>
</tr>
<tr>
<td>Zinc oxide-eugenol</td>
<td>26.10 (5.63) a</td>
</tr>
<tr>
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<td>20.36 (3.99) B</td>
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<tr>
<td>Calcium hydroxide</td>
<td>15.98 (3.22) B</td>
</tr>
</tbody>
</table>

Different letters represent statistically significant differences.
As stated before, the reduced bond strength of composites to tooth substrates after the usage of a provisional cement is believed to be related either to the presence of provisional cement residues not completely removed before cementation\textsuperscript{15,32,40,46} or to the effect of eugenol on the mechanical properties of the composites.\textsuperscript{2,21,25} Since eugenol is not believed to reduce dentin wettability,\textsuperscript{37} some authors report that even after mechanical cleaning or pumice slurry and 37% phosphoric acid etching, cement residues still remain, reducing the dentin wettability and affecting the hybrid layer formation.\textsuperscript{32,40,41,46} Thus, better removal of cement residues should mean better bond strength.

Higher bond strength values were achieved when aluminum oxide sandblasting was used as the cleaning dentin method. Some studies report a higher bond strength when acid etching is preceded by aluminum sandblasting.\textsuperscript{14,18} However, other research groups report similar bond strengths when acid etching is associated with aluminum sandblasting or when it is performed by itself.\textsuperscript{7,20,28,36} Aluminum sandblasting was effective in removing cement residues, resulting in better dentin wettability, and consequently facilitating the infiltration of the adhesive system into the dentin after acid etching. This fact also supports the hypothesis that effective cement residue removal is more important than the effect of the residual eugenol, as previously reported by Paul et al.,\textsuperscript{32} Woody et al.,\textsuperscript{46} and Terata et al.\textsuperscript{40}

Among the provisional cements used in this study, the calcium hydroxide cement seems to promote a higher retentive strength for conventional provisional\textsuperscript{229} or definitive implant restorations.\textsuperscript{1} Higher retentive strength yields greater difficulty in later cement removal. This fact explains the lower bond strength values in this group and again supports the importance of the dentin cleaning method. According to the manufacturers, zinc oxide cements have a lower retentive strength in order to facilitate the penetration of resin monomers on tooth substrates. J Biomed Mater Res 1982;16:225-230. However, the cement type does not seem to affect dentinal adhesion if the correct cleaning method is employed, as was shown by the high bond strength values achieved for the three cements after aluminum sandblasting. This is also supported by Ganss and Jung,\textsuperscript{11} who found the eugenol from provisional cements not to play an extremely important role.

**CONCLUSION**

Employing the proper cleaning method seems to be enough to ensure that the adhesive procedures will not be compromised. Further research is needed to evaluate the influence of eugenol or the cement residue on bond strength during a shorter period. Nevertheless, the clinician must pay careful attention to the dentin cleaning method before adhesively inserting the restoration.

**REFERENCES**


Clinical relevance: In terms of assuring optimal bond strength, the dentin cleaning method seems to be more important than the provisional cement type used before definitive cementation of an indirect resin restoration.