Effect of Chemical Irrigants on the Bond Strength of a Self-Etching Adhesive to Pulp Chamber Dentin

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Abstract
The aim of this study was to evaluate the influence of endodontic irrigants on adhesion to pulp chamber dentin. Seventy crowns of bovine incisors were cut to expose the pulp chamber. Specimens were divided into seven groups, according to the irrigant solution used: G1, 0.9% sodium chloride (control); G2, 5.25% NaOCl; G3, 5.25% NaOCl + 17% EDTA; G4, 2% chlorhexidine solution; G5, 2% CHX solution + 17% EDTA; G6, 2% chlorhexidine in a gel base; and G7, 2% CHX gel + 17% EDTA. After irrigation, Clearfil SE Bond was applied to pulp chamber dentin, followed by Filtek Z250 composite. Six rectangular sticks were obtained from each specimen and dentin/resin interface was tested in tension. Bond strength means were analyzed by ANOVA and Tukey test. There was a significant decrease in bond strength associated to NaOCl, whereas chlorhexidine irrigation showed no effects on adhesion. It was concluded that endodontic irrigants affected differently bond strength to pulp chamber dentin. (J Endod 2006;32:1088–1090)

Key Words
Bond strength, irrigant solutions, pulp chamber dentin

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Effective cleaning and shaping of the root canal, as well as creation of an apical seal is an essential goal for successful endodontic treatment. However, many in vitro studies have demonstrated that root canal fillings exposed to saliva may become contaminated regardless of the materials and obturation techniques employed (1–5), and coronal leakage has been extensively demonstrated as a negative contributor to the prognosis of endodontic treatments (4, 5). Thus, despite apical leakage still being considered an important factor on endodontic failures, in the last few years more attention has been focused on procedures performed to achieve an effective coronal sealing soon after the completion of root canal therapy.

The immediate sealing of endodontically treated teeth using restorative materials is a powerful tool in preventing early coronal leakage (6–8). Among nontemporary restorative materials, dentin adhesives have been advocated for use within the pulp chamber in an attempt to work as a durable barrier against microleakage (6) hampering apical and coronal microleakage (3).

Nevertheless, adhesion to dentin may be affected by many factors. Chemical irrigants used during root canal preparation may alter the chemical composition of dentin surface and affect its interaction with materials used for coronal sealing. The adverse effects of irrigants such as sodium hypochlorite and peroxides on resin-dentin bond strength have been investigated and confirmed previously (9). Although chlorhexidine gluconate, which showed to be as effective as sodium hypochlorite against microorganisms (10, 11), has been increasingly employed as a potential chemical irrigant, its effect on resin/root canal dentin adhesion is still unclear.

Since the importance of coronal sealing right after the conclusion of endodontic treatment has been already established, this in vitro study aimed to compare the effects of different chemical irrigants, including chlorhexidine in gel and in water solution, on the microtensile bond strength of a self-etching adhesive to pulp chamber dentin. It is hypothesized that these different chemical irrigants have no effect on resin-pulp chamber dentin bond strength.

Materials and Methods
Seventy bovine incisors stored in 0.2% thymol solution were used within 6 months of extraction. Crown segments were prepared by removing the roof of the pulp chamber (6). The root was also removed 2 mm below the cemento-enamel junction. The pulp tissue was extracted carefully with a spoon excavator. The canal orifices were sealed with temporary restorative material (Coltosol, Coltène G, Altstatten, Switzerland).

Specimens were divided into seven groups, according to the chemical irrigants employed: G1, 0.9% physiologic saline solution (NaCl, control group); G2, 5.25% sodium hypochlorite (NaOCl); G3, 5.25% NaOCl + 17% ethylenediaminetetraacetic acid (EDTA); G4, 2% chlorhexidine gluconate in water solution (CHX solution); G5, 2% CHX solution + 17% EDTA; G6, 2% chlorhexidine gluconate in a gel base (CHX gel); and G7, 2% CHX gel + 17% EDTA. The pulp chamber area of each crown segment was irrigated with 10 ml of each irrigant for 30 minutes, which were renewed every 3 minutes. In the groups where EDTA was used, 1 ml of this solution was applied for 5 minutes right after the 30-minute irrigation with the primary solution. Before bonding procedures, all teeth were rinsed with 10 ml of distilled water and completely air-dried. A self-etching adhesive system, Clearfil SE Bond (Kuraray, Kurashiki, Japan) was ap
TABLE 1. Mean microtensile bond strengths (MPa) to pulp chamber dentin for different irrigation solutions*

<table>
<thead>
<tr>
<th>Groups</th>
<th>Mean</th>
<th>SD</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl (control)</td>
<td>33.90</td>
<td>1.22</td>
<td>A</td>
</tr>
<tr>
<td>NaOCl</td>
<td>21.34</td>
<td>1.31</td>
<td>B</td>
</tr>
<tr>
<td>NaOCl + EDTA</td>
<td>19.88</td>
<td>1.38</td>
<td>B</td>
</tr>
<tr>
<td>CHX solution</td>
<td>30.87</td>
<td>1.22</td>
<td>A</td>
</tr>
<tr>
<td>CHX solution + EDTA</td>
<td>32.36</td>
<td>1.35</td>
<td>A</td>
</tr>
<tr>
<td>CHX gel</td>
<td>33.01</td>
<td>1.22</td>
<td>A</td>
</tr>
<tr>
<td>CHX gel + EDTA</td>
<td>32.13</td>
<td>1.24</td>
<td>A</td>
</tr>
</tbody>
</table>

NaCl, sodium chloride; NaOCl, sodium hypochloride; EDTA, ethylenediaminetetraacetic acid; CHX, chlorhexidine gluconate.

*Means followed by the same letter did not show any statistical significant difference.

Applied to the surface of pulp chamber dentin according to manufacturer’s instructions. Three to four layers of a resin composite (Filtek Z250, 3M/ESPE, St. Paul, MN) were added to the bonded dentin (12), and each one was light cured for 40 seconds, using a halogen light-curing unit operated at 600 mW/cm². After composite filling of the pulp chamber, teeth were stored in distilled water at 37°C.

After 24 hours, teeth were removed from the water, dried, and fixed to an acrylic plate to allow creation of serial cross sections using a diamond saw (Isomet—Buehler, Lake Bluff, IL). Six rectangular sticks (0.9 ± 0.1 mm²) were obtained from the central portion of the crown segment to assure the presence of a linear resin/dentin interface. The sticks were individually attached to a testing apparatus, Geraldisi’s jig (13), with cyanoacrylate adhesive (SuperBonder Gel, Loctite Adesivos, Itapevi, Brazil) and subjected to a tensile load (Instron 4411, Canton, MA) at a crosshead speed of 0.5 mm/min until failure. The microtensile bond strengths were determined and analyzed by one-way ANOVA and Tukey’s test.

The failure modes were examined under a scanning electron microscope (JEOL–JSM 5600 LV, Noran Instruments, Tokyo, Japan) operated at 15 kV. They were classified into one of four types: Type 1, interfacial failure, located entirely between the adhesive and dentin; type 2, mixed failure, if the fracture site continued from the adhesive into either the resin composite or dentin; type 3, cohesive failure in dentin; and type 4, cohesive failure in resin composite.

Results

Microtensile bond strength means are shown in Table 1. Statistical analysis of the data showed significant differences among groups (p < 0.05). Treatment with NaOCl and NaOCl + EDTA provided significantly lower bond strength to pulp chamber dentin (p < 0.05). Conversely, experimental groups irrigated with chlorhexidine in water solution and in gel base tended to express bond strength values similar to that observed for the control group (p > 0.05).

Scanning electron microscopy revealed that mixed and interfacial failures were the most common fracture patterns observed (Table 2), regardless of experimental condition. Cohesive failures in dentin were more frequently observed in specimens irrigated with CHX solution. Cohesive failures in resin composite were rare or non-evident. The highest incidence of “zero bonds” (i.e. specimens that could not be tested because of spontaneous failure) was observed in specimens treated with NaOCl + EDTA, NaOCl, and CHX solution + EDTA.

Discussion

Bonding to pulp chamber dentin was differently affected by the endodontic chemical irrigants. The present results indicate that, when a self-etching adhesive system was employed, NaOCl had an adverse effect on the bond strength to pulp chamber dentin, which seemed not to be minimized by the associate use of EDTA. CHX in water solution or in gel base, conversely, did not affect the bond strength to pulp chamber dentin as, in general, the specimens treated with these chemical irrigants produced mean values similar to that observed for the control group, which was irrigated by a neutral and inert physiologic saline solution (Table 1). In view of these results, the anticipated hypothesis cannot be confirmed.

Sodium hypochlorite has been extensively used in endodontic therapy to provide gross debridement, disinfection, lubrication, and dissolution of tissues (14). In agreement with our findings, this powerful antimicrobial agent had been previously shown to jeopardize the polymerization of bonding resins (12, 15, 16). It is thought that NaOCl leads to oxidation of some component in the dentin matrix (17), forming protein-derived radicals (16) that would compete with the propagating vinyl free-radicals generated by the light-activation of resin adhesives, resulting in premature chain termination and incomplete polymerization (19). Furthermore, reductions in calcium and phosphorus levels (20) and in mechanical properties of dentin, such as elastic modulus, flexural strength, and microhardness (21), were reported after irrigation of root canals with 5% sodium hypochlorite, which can also contribute to a decrease in the micromechanical interaction between adhesive resins and NaOCl-treated dentin.

Chlorhexidine is a cationic bisguanide with optimal antimicrobial activity over the pH range from 5.5 to 7.0, which acts by adsorbing onto the cell walls of microorganisms and causing breakdown of intracellular components (10). Erdemir et al. (16) reported that endodontic irrigation with CHX solution significantly increased bond strength to root dentin. These authors suggested that adsorption of CHX by dentin may favor the resin infiltration into dentinal tubules, which supposedly explain the high bond strength values obtained. However, such mechanism is still unclear and needs to be tested. We preferred to consider that chlorhexidine did not affect the interaction of the self-etching adhesive system to pulp chamber dentin because it is a nonoxidizing agent.

Bonding to deep dentin, as it is found on the pulp chamber walls, can be occasionally more difficult to be achieved than to superficial dentin (22), since deeper dentin surfaces contain large tubule diameters and are not as exposed as the superficial dentin, which is more susceptible to the reaction of the adhesive. Therefore, the results indicate that deep dentin does not seem to be affected by the irrigation with endodontic chemical solutions, as the final bond strength was similar to that of superficial dentin.

TABLE 2. Modes of bond failure

<table>
<thead>
<tr>
<th>Groups</th>
<th>Failed (zero bond)</th>
<th>Type 1 and 2</th>
<th>Type 3</th>
<th>Type 4</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl (control)</td>
<td>5</td>
<td>40</td>
<td>11</td>
<td>4</td>
<td>60</td>
</tr>
<tr>
<td>NaOCl</td>
<td>10</td>
<td>34</td>
<td>12</td>
<td>4</td>
<td>60</td>
</tr>
<tr>
<td>NaOCl + EDTA</td>
<td>13</td>
<td>45</td>
<td>2</td>
<td>0</td>
<td>60</td>
</tr>
<tr>
<td>Chlorhexidine solution</td>
<td>4</td>
<td>33</td>
<td>21</td>
<td>2</td>
<td>60</td>
</tr>
<tr>
<td>Chlorhexidine solution + EDTA</td>
<td>8</td>
<td>36</td>
<td>10</td>
<td>3</td>
<td>60</td>
</tr>
<tr>
<td>Chlorhexidine gel</td>
<td>5</td>
<td>38</td>
<td>13</td>
<td>4</td>
<td>60</td>
</tr>
<tr>
<td>Chlorhexidine gel + EDTA</td>
<td>7</td>
<td>51</td>
<td>2</td>
<td>0</td>
<td>60</td>
</tr>
</tbody>
</table>

Failed (zero bond): specimens that debonded prematurely.

Type 1, interfacial failure; Type 2, mixed failure; Type 3, cohesive failure in dentin; Type 4, cohesive failure in resin composite.
ters and high tubule density, making them a more challenging bonding substrate. Although bond strength to superficial dentin had not been established in the present study, the microtensile bond test permitted to reveal that, regardless of the effect of chemical irrigants, Clearfil SE Bond self-etching system was able to achieve bond strengths to pulp-chamber dentin with a magnitude very similar to those previously reported to superficial dentin (23). However, the cavity configuration factor (C-factor) (24), showing a ratio of five bonded walls to one unbonded wall, may adversely affect the resin-dentin bond strength. In the present study, we tried to manage the harmful influence of C-factor by inserting the resin composite in incremental layers (25). Even so, 13% of all specimens (54 from 420) failed during preparation (slicing) before testing. Therefore, it can be speculated that gaps developed at the bonded interface because of polymerization shrinkage may be responsible for this considerable amount of zero bond strengths, showing that the effects of a high C-factor might not be totally compensated by using an incremental restorative technique (26), which should be further addressed.

The influence of endodontic irrigants on adhesion was tested only for Clearfil SE Bond adhesive system. Restoration of endodontically treated teeth with self-etching adhesives and composites may offer some advantages over the use of conventional total-etch dental adhesives. Self-etching adhesives have weak acids in their primer composition, resulting in less change in the dentinal wall structure than the strong acids of total-etch systems. In addition, once primer application is performed without air-drying, collapse of collagen fibrils is avoided, reducing technique-sensitivity (27). However, in view of our results, it seems reasonable that further investigation about the effects of endodontic irrigants on the bond strength of total-etch adhesive systems should be conducted.

The microtensile bond test (28) allows bond testing of small areas promoting a better stress distribution throughout the specimen and induces failures of materials that are closer to their true ultimate strengths and are mostly adhesive failures (29). However, the correlation between bond strength and microleakage is not well established, and bond strength data alone are not sufficient to evaluate the sealing ability of resins (6, 12). According to this, the rationale involving microtensile bond testing of endodontic surfaces is that a better adhesion of restorative materials to dentine increases the opportunity for good marginal sealing, longer life of the restoration, and withstanding of mechanical stresses (30, 31). In this study, endodontic irrigation with chlorhexidine solution, chlorhexidine solution associated with EDTA, and chlorhexidine gel did not show an adverse effect on adhesion of a self-etching adhesive system, and it could be concluded that these irrigant solutions are compatible with restorative restorative procedures performed with these materials. On the other hand, the use of NaOCl as an endodontic irrigant, even if associated with EDTA, should be carefully evaluated when the subsequent coronal sealing is performed using resin-based materials associated with self-etching adhesives. It is hoped that the prospective development of adhesive resin systems and bonding techniques take into consideration the specific features of the pulp chamber substrate to achieve better defense against microleakage.

Acknowledgments

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References