Title
Influence of endodontic irrigants on resin sealer bond strength to radicular dentin.

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Abstract

The present study evaluated the influence of 2% chlorhexidine and 2.5% sodium hypochlorite on the resin sealer/dentin interface bond strength of AH Plus/gutta-percha and Epiphany/Resilon. Seventy-two extracted bovine incisors were randomly distributed into 6 groups according to irrigant and sealers: GS + AH = physiologic saline solution + AH Plus/gutta-percha; GS + Ep = physiologic saline solution + Epiphany/Resilon; GH + AH = 2.5% sodium hypochlorite (NaOCl) + AH Plus/gutta-percha; GH + Ep = 2.5% NaOCl + Epiphany/Resilon; GC + AH = 2% chlorhexidine (CHX) + AH Plus/gutta-percha; and GC + Ep = 2% CHX + Epiphany/Resilon. After 7 days at 37°C and 100% humidity, the roots were cut transversally on the long axis of the tooth in 0.8 mm (±0.09)-thick slices; these slices were then subjected to the push-out test. Data were analyzed using a 2-way ANOVA and Tukey tests at 5% significance. The AH Plus/gutta-percha groups showed significantly higher bond strength than the Epiphany/Resilon groups, regardless of the irrigant used (p<0.001). Sodium hypochlorite adversely affected bond strength in the AH Plus group, whereas chlorhexidine did not influence the push-out bond strength of either sealer (p<0.05). Two percent chlorhexidine did not adversely affect the bond strength of the sealers, whereas 2.5% sodium hypochlorite solution damaged AH Plus/gutta-percha bond strength.

Key words: Chlorhexidine — Sodium hypochlorite — Endodontics — Bond strength — Sealers

Introduction

Several resin-based sealer materials have been developed in an attempt to minimize leakage by improving the effectiveness of the seal between the filling material and the root canal walls. Different monomers were used in the
development of resin-based sealers. AH Plus® (De Trey-Dentsply, Konstanz, Germany) is a two-component sealer based on an epoxy resin; it is used in combination with gutta-percha points. Epiphany SE self-etch (Pentron Clinical Technologies, Wallingford, CT, USA) is a dual-curable self-etching methacrylate resin sealer that is used in association with Resilon points (Resilon Research LLC, Madison, CT, USA), a thermoplastic synthetic polyester polymer-based material that replaces gutta-percha. A related advantage of the Epiphany system could be its ability to seal the canal, creating a monoblock between the sealer and point materials.

Nevertheless, chemical irrigants used during root canal preparation may alter the chemical composition of the dentin surface as well as the interaction between the dentin and resin-based sealer. The use of sodium hypochlorite, despite its disinfective properties, could affect penetration of the resin sealer in dentin and its polymerization. Furthermore, sodium hypochlorite is a deproteinizing agent that can degenerate dentin by collagen dissolution.

The use of chlorhexidine (CHX) as an endodontic irrigant has been proposed, presenting an effective antimicrobial potential that can produce similar results to sodium hypochlorite. However, its effects in radicular
dentin and, consequently, in the bond strength of resin sealers, are unclear. The purpose of this study was to evaluate the influence of 2% chlorhexidine and 2.5% sodium hypochlorite on the resin sealer/dentin interface bond strength of AH Plus/gutta-percha and Epiphany/Resilon.

### Materials and Methods

1. **Specimen preparation**
   
   This study was approved by the University Committee on Ethics (protocol no.18.986). Seventy-two extracted central bovine incisors were selected for this study. Immediately after extraction, the teeth were stored in distilled water at 4°C for no more than 4 months. Inclusion criteria of the roots were as follows: canals up to 3 mm in cervical diameter and at least 15 mm in root length. External debris was removed using a scalpel blade. The crown surface of each tooth was sectioned below the cement-enamel junction perpendicular to the long axis of the tooth with a slow speed saw under water irrigation. The length of the roots was standardized to 15 mm. Procedures are described in Fig. 1.

2. **Endodontic treatment**
   
   The root canals were prepared with K-files (Maillefer®, Ballaigues, Swiss) using the step-back preparation technique. All the roots had 15 mm of working length; a master apical file, sizes #45 and #80, was used. The roots were then randomly divided into 6 groups of 12 roots each according to the different irrigants and sealers used (Table 1). The root canals were irrigated using a syringe and a 27-gauge needle with 1 ml irrigant between each file. EDTA was left in the canals for 3 min and then removed using the same irrigant used in the irrigation.

   The root canals were then dried with paper points and obturated with the correspondent sealer of each group. The sealers were manipulated according to the manufacturer’s protocol, but the filling methods were standardized using two techniques: cold lateral compaction and vertical compaction; the excess was seared off with a hot instrument. After Epiphany/Resilon obturation, each root was photocured (Smartlite™ PS, De Trey-Dentsply, Konstanz, Germany) for 40 sec at a distance of 5 mm from the cervical surface with irradiance of 600 mW/cm².

### Table 1 Experimental groups: Irrigation and obturation type

<table>
<thead>
<tr>
<th>Groups</th>
<th>Irrigation</th>
<th>Obturation</th>
</tr>
</thead>
<tbody>
<tr>
<td>G_S_AH</td>
<td>Physiologic saline solution (Pexon, Viamão, RS)</td>
<td>AH Plus/gutta-percha (De Trey-Dentsply, Konstanz, Germany)</td>
</tr>
<tr>
<td>G_S_Ap</td>
<td>Physiologic saline solution (Pexon, Viamão, RS)</td>
<td>Epiphany/Resilon (Pentron Clinical Technologies, Wallingford, CT, USA)</td>
</tr>
<tr>
<td>G_H_AH</td>
<td>2.5% NaOCl + 17% EDTA (Iodontosul, POA, RS; Biodinâmica Quim. Farm. LTDA, Ibiporã, PR)</td>
<td>AH Plus/gutta-percha (De Trey-Dentsply, Konstanz, Germany)</td>
</tr>
<tr>
<td>G_H_Ap</td>
<td>2.5% NaOCl + 17% EDTA (Iodontosul, POA, RS; Biodinâmica Quim. Farm. LTDA, Ibiporã, PR)</td>
<td>Epiphany/Resilon (Pentron Clinical Technologies, Wallingford, CT, USA)</td>
</tr>
<tr>
<td>G_C_AH</td>
<td>2% gel CHX + 17% EDTA (Extrato Puro, POA, RS; Biodinâmica Quim. Farm. LTDA, Ibiporã, PR)</td>
<td>AH Plus/gutta-percha (De Trey-Dentsply, Konstanz, Germany)</td>
</tr>
<tr>
<td>G_C_Ap</td>
<td>2% gel CHX + 17% EDTA (Extrato Puro, POA, RS; Biodinâmica Quim. Farm. LTDA, Ibiporã, PR)</td>
<td>Epiphany/Resilon (Pentron Clinical Technologies, Wallingford, CT, USA)</td>
</tr>
</tbody>
</table>
3. Push-out test

The teeth from all the groups were stored at 37°C and 100% humidity for 7 days in order to allow the sealers to set. Afterwards, the roots were sectioned on the long axis using a precision cutting machine (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under constant water coolant. Each root was sectioned into approximately 9 slices. The 2 mm portion of the apex was discarded due to the small diameter of the canal. Slices measured 0.8 mm (±0.09) in thickness, and the cervical and apical diameters were measured using a digital caliper.

The slices were stored in bottles filled with 1.5 ml distilled water for 2 days. Afterwards, each section was marked on its apical side and positioned on a base with a central hole in a universal testing machine (DL2000, EMIC, São José dos Pinhais, PR, Brazil).

The push-out test was performed by applying a compressive load to the apical side of each slice using a cylindrical plunger attached to the upper portion of the testing machine with a crosshead speed of 1 mm/min. The load upon failure was recorded in Newtons (N) and divided by the bond area (mm²) to express the bond strength in megapascals (MPa). The total bonding area for each slice was calculated using the formula: \( \pi(R+r) \left[ h + (R-r)^2 \right]^{0.5} \), where \( R \) represents the coronal radius, \( r \) is the apical radius, and \( h \) is the thickness of the slice.

Data were analyzed using a two-way ANOVA (irrigant and sealer — and sealer and depth) and post hoc tests using the Tukey multiple comparison test at a significance level of 5%.

Results

Push-out bond strength means (in MPa) and standard deviations are shown in Table 2. AH Plus/gutta-percha groups showed significantly higher bond strength than the Epiphany/Resilon groups, regardless of the irrigating substance used (p<0.001).

Sodium hypochlorite adversely affected the bond strength of the AH Plus group, whereas chlorhexidine did not influence the push-out bond strength of the groups of either sealer (p<0.05).

Push-out bond strength means at different depths are shown in Table 3. As the depth increased, bond strength decreased in the Epiphany sealer groups. Apical third values

<table>
<thead>
<tr>
<th>Irrigants</th>
<th>AH Plus/gutta-percha</th>
<th>Epiphany/Resilon</th>
</tr>
</thead>
<tbody>
<tr>
<td>Physiologic Saline Solution</td>
<td>2.9 (±0.68) Aab</td>
<td>0.9 (±0.32) Bb</td>
</tr>
<tr>
<td>NaOCl + EDTA</td>
<td>2.4 (±0.57) Ab</td>
<td>1.3 (±0.43) Ba</td>
</tr>
<tr>
<td>CHX + EDTA</td>
<td>3.0 (±0.47) Aa</td>
<td>1.3 (±0.31) Ba</td>
</tr>
</tbody>
</table>

Table 3 Mean (± standard deviation) of bond strength (MPa) in different root portions, according to cement

<table>
<thead>
<tr>
<th>Root third</th>
<th>AH Plus</th>
<th>Epiphany</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cervical</td>
<td>2.5 (±0.6) Aa</td>
<td>1.4 (±0.4) A</td>
</tr>
<tr>
<td>Medium</td>
<td>2.8 (±0.8) Aa</td>
<td>0.9 (±0.7) A</td>
</tr>
<tr>
<td>Apical</td>
<td>3.5 (±1.1) Aa</td>
<td>0.6 (±0.6) A</td>
</tr>
</tbody>
</table>

Different capital letters indicate statistical difference between root portion in same cement (p<0.05).
were significantly lower than the cervical third (p<0.05). Among the AH Plus sealer groups, no significant difference was shown among the thirds (p>0.05).

Mean values followed by different capital letters in the same line indicate statistically significant differences. Mean values followed by different small letters in the same column indicate statistically significant differences (p<0.05).

Discussion

Adhesion of the root canal filling material to dentinal walls is of paramount importance in endodontic treatment because it could help to avoid both fluid percolation between the spaces of the obturation and the displacement of the material during procedures. Adhesion to the root dentin walls can be evaluated by the push-out bond strength test, which yields a correlation between bond strength and the sealing ability of resin cements. In this study, both the type of sealer used and the irrigant solution influenced the push-out bond strength of the root canal obturation.

The results showed that AH Plus/gutta-percha had significantly better bond strength than Epiphany/Resilon, regardless of the irrigant used. Epiphany has presented lower bond strength, even with the use of EDTA, which could play an important role with this sealer because of its self-etching characteristic and the hybrid layer formation that occurs in this kind of sealer. The removal of the smear layer created during instrumentation could lead to greater sealer penetration into the exposed tubules, which may increase adhesion.

The Epiphany composition is based on methacrylates (i.e., Ethoxylated bisphenol A dimethacrylate — BisEMA) and presents a dual cure polymerization system. Besides the chemical curing mode, the sealer presents a photoinitiator system that creates the necessity for light irradiation of the material. The depth of light penetration at the canal roots through the material could explain this result; the polymerization at the root apical third could be lower than the cervical third, as shown in Table 3. The degree of conversion of methacrylate resin materials (i.e., epiphany) increases with higher levels of irradiant energy. Initial free radical generation of dimethacrylate monomers is directly related to the irradiance absorbed by the composite. The rate of polymerization of the resin composites is proportional to the square root of the absorbed light intensity and photoinitiator concentration.

Besides the low light intensity as depth increases, shrinkage stress due to polymerization could lead to weakened bond strength of the cement. Since AH Plus is an epoxy-based endodontic sealer and presents no photopolymerization system on its composition, it is believed that homogeneous polymerization occurs, leading to higher mean values of bond strength along the canal root. Furthermore, chemical polymerization occurs at a low rate, delaying the gel point state and allowing for shrinkage stress relaxation, and avoiding a decrease in bond strength. Moreover, the results of this study showed no significant difference in bond strength between G_{H+Ep} and G_{C+E}, suggesting that the low values in the Epiphany groups were not related to the irrigating solution used.

Accordingly, a comparative evaluation of the adhesion of Epiphany and AH Plus endodontic sealers to human root dentin treated with 1% NaOCl or 1% NaOCl + 17% EDTA using the push-out test have shown that AH Plus sealer presented greater adhesion to dentin than Epiphany, regardless of the treatment of the root canal walls. However, the bond strength of AH Plus was adversely affected by NaOCl + EDTA irrigation (G_{H+AH}) in our study. NaOCl has been shown to damage the organic components of dentin — mainly through collagen dissolution — and can hinder the formation of a consistent hybrid layer. Furthermore, NaOCl breaks down into sodium chloride and oxygen, which can interfere with resin sealer polymerization, leading to strong inhibition at the resin/dentin interface and decreasing bond strength.
When the Epiphany group was irrigated with the physiologic solution, the bond strength mean was significantly lower than that of the groups that were irrigated with both NaOCl and CHX. This correlates with the presence of EDTA in the final irrigation of the test groups. The final irrigation with EDTA could promote a more favorable hybridization; thus, the test groups could better support the stress of polymerization, leading to fewer adhesive failures.

In this study, CHX did not adversely affect the bond strength of the AH Plus and Epiphany sealers to the radicular dentin. However, we believe that its influence on bond strength should be evaluated in long-term experiments in the future. Chlorhexidine has the potential to inhibit matrix metalloproteinases, a group of enzymes that regulate the physiologic and pathologic metabolism of collagen-based tissues, in addition to its adsorption properties in dentinal tubules. Based on restorative dentistry studies, CHX has been shown to be beneficial to the longevity of resin restorations. Therefore, CHX’s effects on radicular dentin when using resin sealers must be studied over a long-term period.

Conclusion

Within the methodology of this study, 2% CHX irrigation did not influence sealer bond strength, while 2.5% NaOCl solution influenced epoxy-based sealer bond strength.

Acknowledgements

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References


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