Influence of zinc oxide-eugenol temporary cement on bond strength of an all-in-one adhesive system to bovine dentin

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Abstract
The objective of this study was to evaluate the influence of temporary zinc oxide-eugenol cements (ZOEs) on the microtensile bond strength (iTBS) of an all-in-one adhesive system to dentin. Buccal dentin surface of fifteen bovine incisors were exposed and polished with 600-grit SiC paper. Next, three groups were formed: Group 1 (G1), in which the dentin surface was covered with ZOE (TempCem); Group 2 (G2), in which the dentin surface was covered with temporary cement composed of zinc oxide and an aliphatic acid replacing eugenol (TempCem NE); Group 3 (G3) - no temporary cement was used. Each group was individually stored in distilled water at 37° C for 7 days, whereupon temporary cements were mechanically removed. All groups underwent the adhesive protocol with a simplified self-etching adhesive (One-UP Bond F). Four sticks per tooth were obtained, resulting in 20 sticks per group with a cross-sectional area of 0.5 mm². The iTBS test was performed with a crosshead speed of 1mm/min. The failure pattern was analyzed by SEM. The data were submitted to one-way ANOVA, at p<0.05. The mean values were 44.67 MPa for G3, followed by 41.35 MPa for G2 and 39.33 MPa for G1. There was no significant statistical difference between groups. The failure pattern was almost mixed with the adhesive layer and dentin. The eugenol presented in TempCem did not influence the iTBS test when a simplified self-etching adhesive system was used.

Key words:
zinc oxide-eugenol, dentin, microtensile bond strength, self-etching, adhesive system
Introduction
Temporary zinc oxide-eugenol cements (ZOEs) are extensively used in Dentistry. Apart from the easy preparation in clinical practice, ZOEs exhibit acceptable physical properties, and produce satisfactory biological responses. However, these cements are often replaced by resinous materials, which require hybridization to ensure the appropriate adhesion to dentin surfaces\(^1\). Polymerization of composite resins occurs by the light-activation of camphorquinone-amine system with light wavelengths of between 455 and 500 nm. The process leads to generation of free radicals that induce the conversion of monomers into polymer\(^2\). The hydroxyl groups of the eugenol molecule are predisposed to bond with the monomer free radicals, therefore decreasing its reactivity\(^3,5\). As a result, an immediate decreased bond strength is produced\(^6\), which ultimately contributes for the failure of the adhesive restoration procedure.

The adhesion to tooth substrate depends on the demineralization of dentin surfaces. Demineralization leads to the formation of micro-retentions, in which photo-polymerized monomers could diffuse into the substrate promoting a micro-mechanical interlocking. Nowadays, two categories of adhesive systems are described: ‘etch and rinse’ and self-etching adhesives\(^7\). The self-etching adhesive systems do not require acid etch as such, since they have primers composed of acid monomers that straightforwardly demineralize dentin surfaces. Here, as opposed to ‘etch and rinse’ adhesives, there is no rinsing step, decreasing technical sensitivity. Thus, the smear layer can be modified and incorporated into the adhesion process. Nevertheless, temporary cements are hardly ever thoroughly removed\(^8,9\). Moreover, some amount of eugenol is released from ZOEs, and this eugenol eventually penetrates the dentin\(^10\). All these variables can affect the adhesive polymerization, reducing reactivity\(^11\) and ultimately decreasing microtensile bond strength (µTBS) and marginal seal. The influence exerted by eugenol in composite resin restorations has been analyzed in some experimental models\(^3,4\) and has been subject of much controversy in the literature\(^5\). The null hypothesis tested in this study was that eugenol present in ZOE’s temporary cement has no influence in µTBS, at the resin-dentin interface.

Materials and Methods
Fifteen bovine incisors previously stored in distilled water at 4° C were used. Storage period did not exceed 3 months. Buccal enamel was removed using a trimmer with water irrigation. The exposed dentin was polished with 600-grit SiC paper under water irrigation for 30 s, to obtain a uniform smear layer\(^12\). The exposed area do not exceed 1.5 cm in width and 2.0 cm in length. Teeth were divided in three groups, as follows: Group 1 (G1), in which the dentin surface was covered with ZOE (TempCem, Vigodent SA, Brazil); Group 2 (G2), in which the dentin surface was covered with temporary cement composed of zinc oxide and an aliphatic acid replacing eugenol (TempCem NE, Vigodent SA, Brazil); Group 3 (G3), in which no temporary cement was used. The same length of each paste (3.0 ± 0.5 cm) of the cement was dispensed on a glass slab and mixed upon the formation of a homogeneous paste. G1 and G2 cements were individually mixed. The homogenization did not exceed 30 s. A cement layer was applied, covering all exposed dentin surface of each tooth.

The groups were individually stored in distilled water at 37° C for 7 days, whereupon temporary cements were mechanically removed using periodontal instruments (Newmar, Brazil) and Robinson brushes (Microdont, Brazil) equipped with pumice (Herjos, Vigodent SA, Brazil) followed by rinsing with distilled water. Subsequently, all tooth groups underwent the adhesive protocol with one simplified self-conditioning adhesive (One-UP Bond F, Tokuyama Dental Corporation). The adhesive was passively applied on the dried dentin surface not exceeding 6.0mm of diameter. When mixed, the One-UP Bond F adhesive system changes its color making possible the standardization of the adhesive area when applied. The material was then polymerized for 10 s using a light-curing unit (Curing Light XL 2500, 3M Dental Products, USA) at 5.0mm of distance. The power of the light-curing unit was gauged with a radiometer (Model 100, Demetron Research Group, USA), with light intensity of 550 mW/cm\(^2\). Two composite resin (Palfique Estelite, Tokuyama Dental Corporation, Japan) layers of 2mm each were applied, to ensure the total covering of the adhesive-coated surface. Each layer was polymerized during 30 s. The final dimensions of resin composite coats did not exceed 6.0mm of diameter. Teeth were then stored in distilled water at 37°C for 24h. Materials used for adhesion and restoration procedures are descript in Table 1.

For the µTBS, teeth were sectioned perpendicularly to the adhesive interface using a low-speed diamond saw under water irrigation (Isomet, Buehler Ltd, USA). Four sticks per tooth were produced resulting in twenty sticks per group. The cross-sectional area of sticks were measured using a digital caliper and resulting in a mean cross-sectional area of 0.5 (±0.1) mm\(^2\). Sticks were assembled on a device to microtensile bond strength tests with cyanoacrylate based adhesive. Microtensile bond strength tests were carried out in a universal test machine Emic DL-2000 (Emic, Brazil) at 1 mm/min equipped with a 500 N load cell. For the determination of the failure pattern, the half corresponding to dentin in each specimen was removed from the device and dehydrated in silica gel during 24 h at room temperature. All specimens (n=60) were covered with gold, examined in scanning electronic microscope (Jeol, JSM-6060, Japan) with magnification of 100x, and were classified in...
Table 1 - Materials used, procedures and manufacturers

<table>
<thead>
<tr>
<th>Name</th>
<th>Material</th>
<th>Manufacturer/Country</th>
<th>Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temp Cem</td>
<td>Temporary cement with eugenol</td>
<td>Vigodent SA, Brazil</td>
<td>3.0 (±0.5) cm length of base paste and catalyst were dispensed on a glass slab and mixed for 30s.</td>
</tr>
<tr>
<td>Temp Cem NE</td>
<td>Temporary cement without eugenol</td>
<td>Vigodent SA, Brazil</td>
<td></td>
</tr>
<tr>
<td>One-UP Bond F</td>
<td>Dental adhesive</td>
<td>Tokyuama Dental Corporation, Japan</td>
<td>One drop of liquid A and B was mixed and applied for 20s and photo-activated for 10s.</td>
</tr>
<tr>
<td>Palfique Estelite</td>
<td>Composite resin</td>
<td>Tokyuama Dental Corporation, Japan</td>
<td>Increments of 2.0 mm maximum width were photo-activated for 20s.</td>
</tr>
</tbody>
</table>

Table 2 - Mean microtensile bond strength (μTBS) and standard deviation (SD) expressed as MPa

<table>
<thead>
<tr>
<th>Group (n=20)</th>
<th>Pre-treatment</th>
<th>μTBS Mpa</th>
<th>ANOVA</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>ZOE with Eugenol</td>
<td>39.33±15.72</td>
<td></td>
</tr>
<tr>
<td>G2</td>
<td>ZOE without Eugenol</td>
<td>41.35±13.42</td>
<td>p = 0.654</td>
</tr>
<tr>
<td>G3</td>
<td>No pre-treatment</td>
<td>44.67±13.31</td>
<td></td>
</tr>
</tbody>
</table>

agreement with their failure patterns on the adhesive interface, cohesive in adhesive resin, cohesive in dentin or mixed. The obtained data were analyzed by one-way analysis of variance (ANOVA) at \( p \leq 0.05 \). The statistical unit used was the sticks.

Results
Table 1 shows the mean μTBS results for the three studied groups. No significant statistical difference was observed between these results (\( p > 0.05 \)). The failure patterns were classified as mixed for all groups (Figures 1, 2, and 3). G1 exhibited one tooth with cohesive failure only in dentin, and one tooth with cohesive failure equally only in adhesive resin.

Discussion
The desired adhesion is essentially dependent on the capacity of the adhesive to infiltrate in the conditioned dentin matrix to generate the hybrid layer\(^{13} \). Nevertheless, adhesion is subject of negative factors that decrease its extent. Such factors include the presence of residual debris on the dentin surface and the inadequate management of materials\(^{14} \). However, this study accepted the null hypothesis that eugenol in ZOE temporary cement did not influence the μTBS to dentin.

It was reported a decrease in shear bond strength triggered by eugenol, but only when the powder/liquid phase proportion of ZOE was altered\(^{15} \). Similarly, other studies have shown decrease in tensile bond strength when eugenol alone was applied on the dentin surface before the adhesive protocol\(^{6} \). Nevertheless, a study showed that shear bond
strength was decreased when the dentin surface was treated with ZOE strictly prepared according to the manufacturer’s instructions, before the adhesive protocol. The present study did not observe a statistically significant difference between the groups studied. The same findings was observed about no eugenol influence by others16-17.

It should be remembered that adhesion of ZOE occurs in aqueous medium. In the process, the oxide molecule is hydrolyzed to form an alkali that chemically reacts with eugenol, an acid compound. During the reaction, the remaining eugenol molecules are trapped inside the cement matrix. When eugenolate molecules contact water molecules, the polymer suffers hydrolysis and eugenol is released. Apart from this, eugenol molecules that did not react and were trapped by eugenolate molecules are also released due to the degradation of the temporary cement matrix15. These released eugenol molecules eventually build up on the treated surface to a concentration of $10^{-2}$ M, and on the pulp to $10^{-4}$ M18. Thus, it is possible to conjecture that eugenol, which is present on the treated surface, could alter some of the original properties of adhesives resulting in bond failures longitudinally.

Another aspect that may influence the results is the adhesive type adopted. In the present study, it was used a simplified self-etching adhesive system, in which the primer and the adhesive are mixed a little before application on the dried dentin. In this protocol, in which the surface is not washed after conditioning, the remaining eugenol residual debris apparently bond to the adhesive. These eugenol molecules would bond to the free radicals, postponing resin polymerization and reducing iTBS. Nevertheless, this was not verified with the experimental model in the present study, since no statistically significant differences among groups were observed.

Based on the experimental model adopted in the present study and in the results obtained therefrom, it is possible to conclude that the eugenol present in TempCem did not influenced iTBS when the simplified self-etching adhesive system was used.

**References**


