Microleakage of Seven Adhesive Systems in Enamel and Dentin

Camila Silveira de Araújo, DDS; Thiago Incerti da Silva, DDS; Fabricio Auto Ogliari, DDS; Sonia Saeger Meireles, DDS; Evandro Piva, DDS, MSc, PhD; Flávio Fernando Demarco, DDS, PhD

Abstract

**Aim:** The aim of this study was to evaluate the microleakage of seven adhesive systems on two substrates (enamel and dentin).

**Methods and Materials:** Class V cavities were performed in buccal and lingual surfaces of 56 bovine incisors. The cervical margin was located in dentin and the incisal margin in enamel. The specimens were randomly divided into seven groups (n=16), according to the adhesive system employed: Single Bond; Excite; One Step Plus; Gluma One Bond; Magic Bond; One Up Bond F; and One Coat Bond. The cavities were incrementally filled with a hybrid composite Filtek Z250 and polymerized with a XL 3000 light curing unit. After polishing, the specimens were submitted to thermal cycling followed by dye immersion. Leakage was evaluated under magnification (40X) based on a standard ranking. Data were subjected to statistical analysis (Kruskal-Wallis).

**Results:** Enamel margins exhibited lower leakage than dentin margins (p<0.01). The majority of the specimens were leakage-free and materials performed similarly. Conversely, in dentin most of the specimens exhibited the highest leakage degree and significant differences among materials (p<0.05) were found, with Excite exhibiting the lowest leakage degree. It was concluded enamel provided better sealing and the adhesive system was a significant factor only in dentin.

**Keywords:** Enamel adhesive, dentin adhesives, bovine, dentin, enamel, microleakage

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Introduction
Despite the significant improvement in bonding capacity, microleakage along the tooth/restoration interface remains a concern in restorative dentistry.¹ This phenomenon can cause marginal staining, post-operative sensitivity, pulpal pathology, and secondary caries.²

The basic mechanism of bonding to enamel is related to the formation of resin tags that fill the microporosities produced by acid etching the tooth surface.³ However, dentin is a less mineralized, moist tissue presenting a smear layer when it is instrumented. These characteristics impair the bonding capacity of resin materials to this substrate.⁴

The first adhesive systems attempted to carry out a chemical adhesion with components present in the smear layer with unfavorable results.⁵ Later, increased bond strength was obtained through treatment of the dentin with primers that partially removed or modified the smear layer.⁶ Total-etch adhesives completely remove the smear layer, open the dentin tubules, and demineralize the subjacent dentin leaving an exposed collagen network to be encapsulated after primer and bonding application forming a hybrid layer.⁷ In order to decrease the clinical steps and to simplify the application technique new adhesives were developed to incorporate primer and adhesive into a single bottle.⁸ Deeper demineralization is produced with total etching, but sometimes the bonding agent does not completely penetrate the dentin leaving unprotected exposed collagen which can degrade over time.⁹

An alternative approach is the use of self-etch adhesives containing an acidic-primer, which simultaneously provides the conditioning, and priming of tooth structure. The demineralization depth is lower and the bonding fully penetrates the demineralized area.⁵,⁷ Recently, all-in-one adhesives were introduced in which conditioner, primer, and bonding agents are applied simultaneously.⁹ These products are designed to simplify the bonding technique, decrease the clinical application time, and minimize the possibility of failures.

Due to the presence of different acid concentrations in the composition of self-etch and all-in-one adhesives systems,¹⁰ some studies have questioned the effectiveness of these systems to produce adequate etching over the enamel surface.¹⁰,¹¹

The purpose of this study was to evaluate the microleakage in Class V cavities with margins in enamel and dentin and restored with different adhesive systems. The null hypothesis tested was bonding with different composition and etch approaches produce similar sealing ability in enamel and dentin.

Methods and Materials
Sample Preparation
Fifty-six recently extracted bovine incisors were selected for the study. Standardized Class V cavities were prepared on the buccal and the lingual surfaces of each tooth (3 mm length, 2 mm deep, and 2 mm wide) using a #245 carbide bur (SS White, Lakewood, NJ, USA) with a high-speed handpiece and air-water coolant spray. A new bur was used after five cavity preparations to ensure high cutting efficiency. The cervical margin was located in dentin and incisal margin in enamel. The teeth were randomly assigned to seven groups (n=16 cavities) for the different adhesive systems (Table 1).

Restorative Procedures
The adhesive systems were applied according to manufacturer’s instructions, and the cavities were incrementally restored with a microhybrid resin composite (Filtek Z-250, 3M ESPE, St. Paul, MN, USA). A light-curing unit operating at
450 mW/cm² (XL 3000 - 3M ESPE) was used for polymerization. The teeth were stored in distilled water for seven days at 37°C. Polishing procedures were performed with Sof Lex XT discs (3M ESPE).

**Microleakage Assessment**

Thermal cycling was carried out for 500 cycles between 5 to 55°C (dwell time of 60 seconds). The root apexes were sealed, and two coats of fingernail varnish were applied to the entire surface except for the restorations and a 1 mm area surrounding them. Specimens were immersed in a 1% basic fuchsin solution for 24 hours followed by washing with tap water.

Three sections were obtained of each restoration in a bucco-lingual plane using a water-cooled diamond saw (KG Sorensen, SP, Brazil). The slices were examined under magnification (40x) by three calibrated examiners. When disagreement occurred, consensus was obtained. The degree of microleakage at both enamel and dentin margins was rated from 0 to 3 (Figure 1), and the higher leakage value in one of the slices was assumed as the leakage pattern of that specimen.

**Table 1. Materials used, manufacturers, and compositions.**

<table>
<thead>
<tr>
<th>Adhesive System</th>
<th>Manufacturer</th>
<th>Composition</th>
</tr>
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<tbody>
<tr>
<td>Excite DSC</td>
<td>Ivoclar Vivadent, Schaen, Liechtenstein</td>
<td>Ethanol, TEGDMA HEMA, phosphoric acid acrylate, silicon dioxide and stabilizers</td>
</tr>
<tr>
<td>Single Bond</td>
<td>3M ESPE, St. Paul, MN, USA</td>
<td>Water, ethanol, BisGMA, HEMA, dimethacrylates, initiators and polyalquenoic acid copolymer</td>
</tr>
<tr>
<td>Gluma One Bond</td>
<td>Heraeus-Kulzer GmbH, Wehrheim, Germany</td>
<td>Acetone, HEMA, UDMA, 4-META</td>
</tr>
<tr>
<td>One Coat Bond</td>
<td>Coltene/Waledent Inc, Cuyahoga Falls, OH, USA</td>
<td>UDMA, HEMA, glycerindimethacrylate, HPMA, polyalkenoat methacrylet, and amorphous silica</td>
</tr>
<tr>
<td>One Up Bond</td>
<td>Tokuyama, Tokyo, Japan</td>
<td>Water, methacryloyloxyalkyl acid phosphate, HEMA, MMA, methacryloyxundecane dicarboxylic acid, multifunctional methacrylic monomer, coumarin dye, fluorescentsilicat glass and photoinitiator (aryl borate catalyst)</td>
</tr>
<tr>
<td>One Step Plus</td>
<td>Bisco Inc., Schaumburg, IL, USA</td>
<td>Acetone, Bis-GMA, BPDM, HEMA and initiator</td>
</tr>
<tr>
<td>Magic Bond DE</td>
<td>Vigodent, Rio de Janeiro, RJ, Brazil</td>
<td>Ethanol, dimethacrylates, phosphinic acid acrylate, silicon dioxide, initiators and stabilizers</td>
</tr>
</tbody>
</table>

**Figure 1.** Scores of dye leakage observed for both substrates:
- 0 = No dye penetration
- 1 = Dye penetration up to half of the cavity depth
- 2 = Dye penetration more than half of the cavity depth
- 3 = Dye penetration arriving to the cavity floor
Statistical Analysis
Leakage data were submitted to the non-parametric Kruskal-Wallis test with the confidence level set at 95%.

Results
The leakage scores for different adhesive systems are described in Table 2. The non-parametric Kruskal-Wallis test disclosed a higher dye penetration in dentin than in enamel (p<0.01), except for Excite which exhibited a similar performance in both substrates.

All adhesives showed minimum leakage in enamel without significant differences (p>0.05) among them. As for dentin margins, the majority of the specimens presented the highest degree of leakage and significant differences were observed (p<0.05). The least leakage was detected for Excite (p<0.05), while Single Bond and Gluma One Bond provided a better marginal seal than One Coat Bond, One Step Plus, One Up Bond, and Magic Bond which presented similar leakage patterns.

Discussion
Different techniques are used for microleakage evaluation, but the most employed method is the migration of dye along the tooth/restoration interface. Although this method is a simple, economic, and fast technique, the subjectivity of reading the specimens has been noted as a shortcoming related to this methodology.

Human teeth should be the first choice for in vitro studies; however, a more preventive approach in dentistry along with ethical concerns has decreased the availability of human teeth for laboratory use. Bovine teeth have produced comparable results to human teeth in adhesion tests since bovine and human dentin are similar in morphology.

In the present study, higher leakage was detected in dentin when compared to enamel. This difference can be related to the composition of these tissues. While the enamel is almost completely mineralized, dentin presents with a lower mineral content with an organic matrix having a moist surface which impairs the bonding mechanism. Therefore, the bond strength to enamel is typically stronger and more stable than that obtained with dentin, and the leakage along the enamel/restoration interface is reduced or completely prevented. As observed in this study, despite the variations in composition of enamel, different adhesive systems have demonstrated similar leakage patterns. There was also poor sealing ability in dentin for all of the adhesive systems tested except for Excite that showed the

<table>
<thead>
<tr>
<th>Materials</th>
<th>Enamel</th>
<th>Dentin</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>0 1 2 3</td>
<td>0 1 2 3</td>
</tr>
<tr>
<td>Excite</td>
<td>12 0 0 4</td>
<td>12 2 0 0</td>
</tr>
<tr>
<td>Single Bond</td>
<td>13 1 1 1</td>
<td>1 7 4 4</td>
</tr>
<tr>
<td>Gluma One Bond</td>
<td>14 0 1 1</td>
<td>3 8 0 5</td>
</tr>
<tr>
<td>One Up Bond</td>
<td>14 1 0 1</td>
<td>1 0 2 13</td>
</tr>
<tr>
<td>One Step Plus</td>
<td>13 1 1 1</td>
<td>1 1 1 1</td>
</tr>
<tr>
<td>One Coat Bond</td>
<td>13 0 0 3</td>
<td>2 1 3 10</td>
</tr>
<tr>
<td>Magic Bond</td>
<td>14 0 0 2</td>
<td>1 1 1 13</td>
</tr>
</tbody>
</table>

Table 2. Leakage scores for different groups in enamel and dentin.
best performance in this substrate. Single Bond and Gluma One Bond exhibited intermediate results, with a better sealing than the other adhesives. Some clinical considerations could be drawn from these findings.

Despite the continuing evolution of adhesive systems, up to now no available adhesive technique can produce predictable results when the preparation margins are located in dentin. The lower bond strength obtained in dentin is not strong enough to counteract the stress developed during polymerization shrinkage which impairs the sealing capacity. The conventional Class V cavity employed in this study represents a great challenge to the adhesive systems used due to the high C-factor. The C-factor is the ratio between bonded surfaces and unbonded surfaces. An increase in C-factor could increase the shrinkage stress at the adhesive interface, thus, impairing the sealing ability. An incremental filling technique was used in this study in an attempt to reduce the total amount of shrinkage.

In the present study, a light curing unit with high energy was used to generate a faster monomer conversion. However, this could increase the shrinkage stress and compromise the sealing ability. New polymerization methods have been developed to reduce the stress generated during shrinkage allowing a better adaptation to the cavity wall.

Although the total etch adhesive systems used in this study presented a similar mechanism of action, they produced different results in dentin. These systems are based on dentin demineralization and hybrid layer formation. Sometimes the adhesive could not penetrate the entire zone of demineralized dentin leaving an unprotected collagen zone that could degrade over time causing adhesive failure and microleakage.

Another factor to consider is the solvent used in the adhesive composition. Excite presents two different solvents: water and ethanol. These two solvents may provide a better performance in the different wettability conditions of dentin, which could not be observed for adhesive systems with only one solvent. Relative to the self-etching system, Excite performed similar to the other systems in enamel. However, this adhesive system demonstrated a poor performance when used on dentin which is a similar result found with other total etch systems. This corroborates our findings of better sealing observed in enamel than in dentin for this system.

The adhesive systems were used according to manufacturers’ recommendations, but some adhesive systems would perform better with more applications than suggested by manufacturers.

Dentin permeability to the test dye can be a confusing factor in microleakage experiments on dentin, and findings should be considered with caution.

The null hypothesis tested in this study was rejected because the substrates demonstrated different leakage patterns and adhesives performed differentially in dentin.

Conclusions
Within the limitations of this study, it could be concluded:

1. There was less leakage in enamel than in dentin.
2. The adhesive systems presented good sealing ability in enamel without significant differences.
3. Reduced sealing ability was observed in dentin and significant differences were observed between materials.
References
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