Microleakage of XP Bond in Class II Cavities After Artificial Aging

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**Purpose:** To determine the microleakage of etch and rinse adhesives.

**Materials and Methods:** Standardized Class II cavities were cut in 40 human molars with one proximal box limited within enamel and one proximal box extending into dentin. Teeth were assigned randomly to 5 groups (n = 8) and restored with incrementally placed composite restorations. Five combinations were tested: G1 = XP Bond + Ceram X Mono, G2 = Syntac Classic + Tetric EvoCeram, G3 = Scotchbond 1 XT + Z250, G4 = P&B NT + Ceram X mono, G5 = Optibond Solo Plus + CeramX Mono. After finishing and polishing, teeth were stored for 48 h in water at 37°C before being subjected to artificial aging by thermal stress (5/55°C, 2000x, 30 s) and mechanical loading (50 N, 50,000x). Teeth were isolated with nail varnish and immersed in 5% methylene blue for 1 h. After sectioning, specimens were evaluated for leakage (ordinal scale: 0 to 4) at enamel and dentin margins under a stereomicroscope. Results were analyzed using the Kruskal-Wallis H-test and Mann-Whitney U-test (p < 0.05).

**Results:** Statistical analysis showed significant differences among the groups in both enamel and dentin. Mean ranks (H-test) were: enamel: G2 (64.44) < G1 (66.69) < G4 (74.88) < G3 (98.25) and G5 (98.25); dentin: G3 (66.53) < G1 (74.42) < G4 (81.09) < G2 (81.84) < G5 (99.61).

**Conclusion:** Microleakage of XP Bond is at the same level as or even better than other etch-and-rinse adhesives.

**Keywords:** Class II restorations, dentin adhesives, composite, microleakage.

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Despite improvements in the formulation of modern dentin adhesive systems, the bond strength and marginal adaptation of composite resins to dentin seems still inferior and less predictable than adhesion to enamel. However, most of the cavities in clinical dentistry, especially when restorations in the posterior region of the mouth are replaced, are not limited exclusively within enamel but show a mixed type configuration with finishing lines in both enamel and dentin. In particular, the adhesive interface between tooth and restorative material at the gingival finish line has been recognized as one of the most problematic regions. While a great number of self-etching primers and adhesives have emerged on the market, which are considered less technique sensitive and less time consuming than etch-and-rinse adhesives, the latter are still considered the gold standard with respect to long-term bond strength and marginal seal, especially for the restoration of highly loaded Class II cavities or when restorations are bonded to uninstrumented tooth tissues such as sclerotic dentin in Class V lesions or virgin enamel in anterior diastema closures.

This study assessed the microleakage of a newly formulated etch-and-rinse adhesive, based on a tert-butanol solvent used for the first time, in large clinical cavities after artificial aging in comparison with well-established competitive adhesive and composite systems. The null hypothesis tested was that the type of restorative system used does not significantly affect the marginal seal.

**MATERIALS AND METHODS**

**Specimen Preparation**

Forty freshly extracted caries-free human permanent molars, stored in a 0.25% mixture of sodium azide in Ringer solution until the date of use, were used in this in vitro study. After cleaning the teeth with scalers and polishing with...
Table 1 Experimental groups and materials used

<table>
<thead>
<tr>
<th>Group</th>
<th>Adhesive</th>
<th>Solvent type of adhesive</th>
<th>Composite</th>
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</thead>
<tbody>
<tr>
<td>G1</td>
<td>XP Bond (Dentsply DeTrey; Konstanz, Germany)</td>
<td>t-butanol</td>
<td>Ceram X mono (M2)</td>
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<td></td>
<td></td>
<td></td>
<td>(Dentsply DeTrey)</td>
</tr>
<tr>
<td>G2</td>
<td>Syntac Classic (Ivoclar Vivadent; Schaan, Lichtenstein)</td>
<td>Primer: acetone-water Adhesive: water Heliobond: -</td>
<td>Tetric EvoCeram (A2) (Ivoclar Vivadent)</td>
</tr>
<tr>
<td>G3</td>
<td>Scotchbond 1 XT (3M ESPE; Seefeld, Germany)</td>
<td>Ethanol-water</td>
<td>Z250 (A2)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(3M ESPE)</td>
</tr>
<tr>
<td>G4</td>
<td>Prime &amp; Bond NT (Dentsply DeTrey; Konstanz, Germany)</td>
<td>Acetone</td>
<td>Ceram X mono (M2)</td>
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<td></td>
<td></td>
<td></td>
<td>(Dentsply DeTrey)</td>
</tr>
<tr>
<td>G5</td>
<td>OptiBond Solo Plus (Kerr-Hawe; Bioggio, Switzerland)</td>
<td>Ethanol</td>
<td>Ceram X mono (M2)</td>
</tr>
</tbody>
</table>

Pumice, standardized Class II inlay cavities (MOD) were prepared, with one proximal box limited within enamel (1 to 1.5 mm above the cemento-enamel junction) and one proximal box extending into dentin (1 to 1.5 mm below the cemento-enamel junction) (Fig 1). The cavities were 4.0 mm in width and 3 to 3.5 mm in depth at the occlusal isthmus and 5.0 mm in width at the proximal boxes. The depth of the proximal boxes in the direction of the axial pulpal walls was 1.5 mm. To achieve divergence angles between opposing walls of 10 to 12 degrees, cavities were prepared using coarse diamond burs with a slight taper (8555.314, Komet; Lemgo, Germany) in a high-speed dental handpiece with copious water spray. Fine grained diamond burs of the same shape (8855.314, Komet) were used for finishing the preparations. The internal point and line angles were rounded and enamel margins were not beveled but prepared in butt joint configuration. After visual inspection of the cavities for imperfect finish lines, the 40 prepared teeth were randomly assigned to 5 experimental groups with 8 teeth each (Table 1), corresponding to the different restorative techniques. Manufacturers’ instructions for each material were strictly followed.

For the direct composite restorations, all enamel and dentin surfaces of the cavities were conditioned with 36% phosphoric acid gel (DeTrey Conditioner 36, Dentsply DeTrey; Konstanz, Germany), starting acid application on enamel, leaving undisturbed for 15 s, then covering the dentinal preparation surfaces for an additional 15 s (total-etch technique). After thoroughly rinsing with water, the cavities were then gently dried with oil-free compressed air, taking care to avoid desiccation of the tooth substrate (moist bonding technique). Following the application and light curing of the adhesive systems, the cavities were restored with composite resin (Table 1) using a horizontal and oblique layering technique with 5 increments in the dentin-limited proximal box and 4 increments in the enamel-limited box (Fig 1). Each increment with a maximum thickness of 2 mm was light cured individually with a LED curing unit (SmartLite PS, Dentsply DeTrey) according to the manufacturer’s recommendations. The light output of the curing unit was monitored at 1065 mW/cm² with a calibrated light meter (CureRite, Dentsply DeTrey). All restorations were finished and polished immediately after placement using finishing diamond burs and polishing disks (Sof-Lex, 3M ESPE; Seefeld, Germany).

Thermocycling and Mechanical Loading

After 48 h storage in distilled water at 37°C, the restored teeth were subjected to artificial aging by thermocycling and mechanical loading. All specimens were immersed alternately in water baths at 5°C and 55°C for 2000 cycles, with a dwell time of 30 s in each bath and a transfer time of 15 s. Mechanical loading of the teeth, which were mounted on metallic specimen holders with a light-curing composite, was conducted in the Munich Oral Environment. The carefully aligned teeth were loaded in the central fossa of the restorations in axial direction with a force of 50 N for 50,000 times at a frequency of 1 Hz. The antagonist material was a De-gusit sphere (6 mm in diameter), which exhibits a hardness and wear resistance similar to natural enamel. The metal specimen holders were mounted on a hard rubber element, which allowed a sliding movement of the tooth between the first contact on an inclined plane to the central fossa. During mechanical loading, the teeth were continuously immersed in Ringer solution. This oral simulation device exhibits similar functions to the machine developed by Krejci.

Evaluation of Microleakage

The apices of the artificially aged teeth were sealed with adhesive and composite (Prime & Bond NT and Ceram-X Mono). All tooth surfaces were covered with 2 coats of nail varnish to within approximately 1 mm of the margin of the restoration. Microleakage was tested using a standardized dye penetration method. The specimens were immersed in 5% methylene blue at 37°C for 1 hour and then rinsed with tap water. Teeth were embedded in clear acrylic auto-polymerizing resin (Technovit, Kulzer; Wehrheim, Germany). All restorations were sectioned longitudinally with two parallel cuts in three fragments in mesiodistal direction with a water-cooled low-speed diamond saw (Varicut, Leco; Kirch-
hein, Germany), resulting in four readings for each specimen at the enamel and dentin adhesive interface. An ordinal scale from 0 to 4 was used to score microleakage separately at the enamel and dentin margins of each section (Fig 2). Each section was examined under a stereomicroscope (Stemi SV 11, Zeiss; Oberkochen, Germany) at 40X magnification and scored by two examiners. Consensus was forced if disagreements occurred. The results of the microleakage investigation were analyzed using the nonparametric Kruskal-Wallis H-test and post-hoc Mann-Whitney U-test at a significance level of p < 0.05.

RESULTS

Microleakage results are presented in Table 2. The Kruskal-Wallis H-test revealed statistically significant differences among the experimental groups in the enamel (p = 0.001) and dentin margins (p = 0.022) of the Class II restorations.

DISCUSSION

Microleakage studies provide adequate screening methods, possibly determining what kind of adhesive system will show acceptable clinical performance. While quantitative marginal analysis by scanning electron microscopy assesses the entire circumference of the tooth/restoration interface, it can only determine the quality of the adhesive interface at the cavosurface margin. The extension of marginal gaps towards the axial wall of restorations is commonly assessed by microleakage studies. Detection of microleakage can be accomplished with a number of techniques, including bacteria, chemical or radioactive tracer molecules, fluid permeability, and dye penetration. The most common technique is the use of dyes, the penetration of which is determined after sectioning of the specimen with a magnifying aid.

The type of solvent strongly influences the clinical application protocol of etch-and-rinse adhesive systems. Acetone-based systems only work well on a moist dentin surface as acetone is a water chaser and can lead to rather poor results on dried acid-etched dentin surfaces. On the other hand, water-based systems are not as sensitive with regard to dentin moisture content, as they have inherent rewetting properties, but require a longer evaporation time for the solvent, because water has a considerably lower vapor pressure than acetone. If the solvent is not completely evaporated before light curing the adhesive, flaws can weaken the hybrid layer, probably causing premature restoration failure. A new type of solvent for adhesives, namely, tert-butanol, was introduced for XP Bond. Tert-butanol (2-methyl-2-propanol)

| Table 2 | Microleakage frequency scores at enamel and dentin margins in the experimental groups G1 to G5 |
|         | Enamel | Dentin |
| Leakage | Mean rank (Kruskal-Wallis) | Leakage score | Mean rank (Kruskal-Wallis) |
| score   |                         |             |                         |
| G1      | 28 | 0 | 4 | 0 | 0 | 66.69 | 20 | 3 | 4 | 5 | 0 | 74.49 |
| G2      | 28 | 3 | 1 | 0 | 0 | 64.44 | 18 | 1 | 7 | 4 | 2 | 81.84 |
| G3      | 15 | 7 | 9 | 1 | 0 | 98.25 | 23 | 5 | 1 | 2 | 1 | 65.53 |
| G4      | 24 | 4 | 4 | 0 | 0 | 74.88 | 19 | 2 | 2 | 7 | 2 | 81.09 |
| G5      | 15 | 7 | 9 | 1 | 0 | 98.25 | 10 | 6 | 7 | 6 | 3 | 99.61 |

Different superscript letters indicate statistically significantly different subsets within each margin segment as determined by post-hoc multiple comparisons with the Mann-Whitney U-test (p < 0.05)
Figure 2 Scoring system for microleakage evaluation in enamel and dentin.

CONCLUSIONS

The two-step one-bottle tert-butanol-based XP Bond showed excellent microleakage results in both enamel and dentin, with the same quality as a well-established three-step etch-and-rinse system.

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REFERENCES


Clinical relevance: The new tert-butanol-based XP Bond is expected to show good clinical results due to its chemical composition and technique robustness.