Microleakage of Class V Composite Restorations Placed with Etch-and-Rinse and Self-etching Adhesives Before and After Thermocycling

Juan Ignacio Rosales-Leal

Purpose: To evaluate the sealing ability of etch-and-rinse and self-etching adhesives in Class V cavities before and after thermocycling in vitro.

Materials and Methods: Etch-and-rinse adhesives (Prime & Bond NT [P&B], XP Bond [XPB], Scotchbond 1 XT [SBX], Syntac [SYN]) and self-etching adhesives (Xeno III [XNO], i-Bond [IBO], Clearfil SE Bond [CLF]) were used. A microleakage test was performed to evaluate marginal sealing. Seventy molars were divided into seven groups according to the adhesive used. Class V cavities were restored and each group was divided into two subgroups. One group was water immersed for 24 h and the other was thermocycled. Then, specimens were immersed in fuchsin and sectioned. Microleakage and dentin permeability were recorded on occlusal and gingival walls and data were statistically analyzed.

Results: Etch-and-rinse adhesives provided perfect occlusal sealing. Self-etching adhesives obtained slight occlusal leakage. In the gingival wall, XNO and CLF showed the lowest leakage, followed by XPB and SBX, then P&B. SYN and IBO exhibited the highest leakage. All SE adhesives and XPB provided sealed dentinal tubules. Thermocycling did not affect the occlusal sealing but reduced the gingival sealing when P&B, SYN, XNO, CLF, and IBO were used.

Conclusion: In enamel, marginal leakage was prevented when phosphoric acid was used. Self-etching adhesives promoted slight occlusal leakage. The gingival sealing was poorer than the occlusal sealing. XNO, CLF followed by XPB obtained the best gingival sealing. Thermocycling did not affect the occlusal bonding but reduced the gingival sealing, except when XPB and SBX were used.

Keywords: adhesives, Class V sealing, thermocycling, in vitro.

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Sealing of a cavity is one of the most important requirements for the durability of a composite restoration. Microleakage of a restoration may be the starting point of secondary caries and the treatment failure. The bond needs to be hermetic but also durable over time. In vitro microleakage tests offer very useful data about the sealing behavior of adhesives. A microleakage test provides information about the sealing of the interface and the dentin tubule sealing (dentin permeability). Results are close to clinical reality because extracted human teeth and clinical protocols are used.

Two different classes of adhesives are currently used. Etch-and-rinse adhesives require a separate acid-etching step prior to the adhesive infiltration that promotes an aggressive substrate treatment. Self-etching adhesives etch and infiltrate at the same time, but their acidity is less than that of phosphoric acid, resulting in less etching depth.

More information is necessary about the sealing ability of current adhesives (etch-and-rinse vs self-etching) and the effect of aging on the durability of the sealing. The purpose of this work was to evaluate the in vitro sealing ability of etch-and-rinse and self-etching adhesives in Class V cavities before and after thermocycling.

MATERIALS AND METHODS

The adhesives used are described in Table 1. Seventy third molars were divided into 7 groups as a function of the adhesive used (Table 1). In each specimen, two Class V Cavities (3 x 2 x 2 mm [depth] with a 1-mm 45-degree enamel

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## Table 1 Materials tested

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Manufacturer</th>
<th>Components</th>
<th>Directions for use</th>
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<tbody>
<tr>
<td>Prime &amp; Bond NT lot 0503000835 (P&amp;B)</td>
<td>Dentsply DeTrey; Konstanz, Germany</td>
<td>Conditioner: DeTrey Conditioner (36% phosphoric acid) Adhesive: di- and tri- methacrylate, amorphous functionalized silica, PENTA, cetyl amine hydrofluoride, acetone, photoinitiators, stabilizers Composite: Esthet-X (microhybrid)</td>
<td>Etch the cavity for 15 s, wash and dry but do not desiccate. Apply the adhesive and wait for 20 s. Dry and polymerize for 10 s. Apply composite and polymerize for 20 s.</td>
</tr>
<tr>
<td>XP Bond lot 0503004020 (XPB)</td>
<td>Dentsply, DeTrey; Konstanz, Germany</td>
<td>Conditioner: DeTrey Conditioner (36% phosphoric acid) Adhesive: tertiary butanol, HEMA, PENTA, TCB, UDMA, TEG-DMA; butylated benzeneediol, ethyl 4-dimethylaminobenzoate, camphoro-quinone, nanofillers Composite: Ceram-X mono (nano composite)</td>
<td>Etch the cavity for 15 s, wash and dry but do not desiccate. Apply adhesive and wait for 20 s. Dry and polymerize for 10 s. Apply composite and polymerize for 20 s.</td>
</tr>
<tr>
<td>Adper ScotchBond 1 XT lot 177215 (SBX)</td>
<td>3M St Paul, MN, USA</td>
<td>Conditioner: etchant (37% phosphoric acid) Adhesive: Ethanol, water, bis-GMA, HEMA, UDMA, glycerol-DMA, silanized silica Composite: Filtek Suprême (nano composite)</td>
<td>Etch cavity for 15 s, wash and dry (do not desiccate). Apply adhesive and wait for 15 s. Air dry and light cure for 10 s. Place composite and light cure for 20 s.</td>
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<tr>
<td>i-Bond lot 010082 (iBO)</td>
<td>Heraeus Kulzer; Hanau, Germany</td>
<td>Adhesive: acetone, water, methacrylate resins, glutaraldehyde Composite: Venus (microhybrid)</td>
<td>Apply a copious amount to the cavity. Apply two additional coats. Wait for 30 s. Dry and polymerize for 20 s. Apply composite and polymerize for 20 s.</td>
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HEMA: 2-hydroxyethyl methacrylate; PENTA: dipentaerythrit pentaacrylate monophosphonate; TCB: carboxylic acid modified dimethacrylate; TEG-DMA: triethylene glycol dimethacrylate; pyro-EMA: phosphoric acid modified methacrylate; PEM-F: monofluorophosphazene-modified methacrylate; MDP: 10-methacryloxydecyl dilhydrogen phosphate; UDMA: urethane dimethacrylate; bis-GMA: bis-phenoL A diglycidyl methacrylate.
bevel) were prepared with diamond-coated #330 burs at high speed under water cooling. Cavities were filled following manufacturer’s instructions for use. The filling material was placed in two increments. The LED unit SmartLite PS (Dentsply DeTrey; Konstanz, Germany) (light output: 830 mW/cm²) was used for polymerization. The restoration was finished and polished with abrasive disks. Restored teeth were divided into two subgroups. In one group, teeth were kept in water at 37°C for 24 h. In the other group, teeth were thermocycled 4000 times between water baths at 5°C and 55°C with a dwell time in each bath of 30 s. After sealing the roots with IRM (Dentsply DeTrey), the teeth were covered with two coats of nail varnish, leaving a 1-mm varnish-free margin around the restoration. Specimens were then immersed in a 0.5% water solution of basic fuchsin for 24 h and rinsed for 5 min with water. After this, specimens were embedded in acrylic resin, and 3 buccolingual slices of approx 1 mm thickness were obtained from each specimen (15 slices resulting in 30 margins in dentin and enamel, per adhesive and aging condition). Slices were coded and randomly examined independently under the microscope in a blinded fashion. The grade of microleakage at occlusal and gingival walls was categorized as follows: 0: hermetic seal, no leakage; 1: mild microleakage, dye on no more than half of the wall; 2: moderate microleakage, dye on more than half of the wall but not including the axial wall; 3: massive microleakage, dye on the entire wall, including the axial wall. Data obtained represent the percentage of each leakage score in all the analyzed slices. Dentin permeability was evaluated as negative (absence of dye solution in dentin tissue) or positive (presence of dye solution in dentin tissue). Microleakage analysis was performed with the nonparametric Kruskal-Wallis H-test and Mann-Whitney U-test (p < 0.05), Fisher’s Exact test was used to evaluate dentin permeability (p < 0.05).

RESULTS

Figures 1 and 2 show the microleakage and dentin permeability data. The occlusal wall exhibited less leakage than the gingival wall. Etch-and-rinse adhesives achieved hermetic occlusal sealing, while self-etching adhesives showed slight occlusal leakage. Occlusal dentin permeability was negative for all the adhesives tested. Thermocycling did not affect the occlusal sealing.

On the gingival wall, XNO and CLF obtained the lowest leakage, followed by XPB, SBX, and P&B, while SYN and IBO showed the highest leakage. Thermocycling did not affect marginal seal in dentin for XPB and SBX, with the latter showing more leakage after thermocycling compared to XPB. However, marginal seal of P&B, SYN, XNO, IBO, and CLF was influenced by thermocycling. Dentin permeability was negative for XPB, XNO, IBO and CLF. Positive dentin permeability was observed when P&B, SBX or SYN were used. Thermocycling increased the dentin positive permeability of P&B and SYN groups.

DISCUSSION

This in-vitro test provoked microleakage for all adhesives used. Three main factors could affect the sealing. One factor is the composite polymerization shrinkage that induces stress at the bonding interface.8 This stress can potentially
break the bond and facilitate leakage. Another factor is that the substrate is a biological tissue, which makes adhesion difficult. The third factor is the adhesive itself: the chemical composition plays an important role in achieving a strong, durable, and biologically compatible bond.

Etch-and-rinse adhesives obtained hermetic sealing on the occlusal wall, which is surrounded by enamel. Enamel is mainly composed of minerals with no significant amount of organic compounds or water. Use of phosphoric acid leads to pronounced etching depth. The adhesive covers a highly irregular mineral surface with no water, creating a hermetic and strong bond. In contrast, self-etching adhesives exhibited slight leakage. In general, the pH of acidic primers is higher than that of phosphoric acid, resulting in a less pronounced etching effect. The consequence in this in vitro study was a weaker bond with slight leakage. In agreement with other studies, sealing was better along occlusal than gingival margins. The gingival wall is formed by dentin, which consists of minerals, organic compounds (mainly collagen fibers), and water. In addition, dentin tubules cross the tissue and are full of water. After phosphoric acid treatment, there is a deep demineralization front in which the collagen network is exposed, dentin tubules are opened, and water content is increased. Etch-and-rinse primers have to infiltrate the exposed collagen, replace the water, and seal the tubules. Therefore, sealing is complicated, and this histological finding explains the higher leakage and positive permeability when etch-and-rinse adhesives are used. XPB was the best etch-and-rinse adhesive in this study, and always sealed the dentin tubules. This adhesive uses tertiary butanol as a solvent and provides an increase in resin content. After polymerization, the bonding layer will be thicker and consists of a dense polymer matrix that promotes better sealing.

Self-etching adhesives (except IBO) obtained higher dentin sealing capability than etch-and-rinse adhesives. To etch and infiltrate at the same time assures proper covering of the demineralized dentin and avoids the problem of water, which etch-and-rinse adhesives have after phosphoric etching. The lower primer acidity promotes less tubule opening, and then tubule sealing was demonstrated to be easier. The consequence is that self-etching adhesives always sealed the dentin tubules and yielded lower leakage than etch-and-rinse adhesives. In accordance with others, IBO allowed more leakage than other adhesives tested. Despite the higher leakage, IBO sealed always the dentin tubules.

Evaluation of the dentin permeability to the dye solution shows the adhesive ability to seal the dentin tubules. Within the limits of a microleakage study (not being a nanoleakage evaluation), it can be concluded that if there is leakage but no positive permeability, the interface failure will be located over the intact dentin. In a study in vivo, it was shown that when bacteria progressed between the sealed dentin and the composite, a low level of pulp inflammation was found. However, if the bacteria progress into the dentin tubules, the inflammatory activity is higher. Therefore, it is desirable to achieve sealing of dentinal tubules.

Thermocycling is an easy method to age restorations and results in the highest clinically relevant stress. As was demonstrated in others studies, thermocycling did not affect the occlusal sealing and showed that the enamel junction is resistant to aging, due to the mineral nature of the tissue which ensures a better and more durable bond.

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**Fig 2** Percentage of cases with positive and negative permeability on the occlusal wall (O) and on the gingival wall (G) after 24-h water immersion (24 h) and 4000 cycles of thermocycling (4000 c). Columns with the same letter are not statistically significantly different (p > 0.05).
trast, the dentin bond was susceptible to damage and the sealing was reduced after thermocycling.18 The larger proportion of organic compounds and water in the dentin makes the union weaker for all the adhesives tested, which would explain aging.11,17 Thermocycling accelerates the aging by hydrolytic degradation of the hydrophilic components in the bonding system.12,22 In addition, repetitive contraction/expansion stress at the bonding interface may lead to cracks that propagate along the interface and cause in- and outflow of fluids.5 When considering distribution of microleakage grades in all obtained slices, only XPB was not affected by thermocycling.

Clinically, when a cavity is surrounded by enamel, the phosphoric acid will ensure a hermetic and durable bond. When the cavity margins are in dentin, improvements are necessary, but there are some self-etching adhesives (XNO, CLF) or etch-and-rinse adhesives (XPB) that obtain excellent results with no positive dentin permeability and low leakage. Perhaps more acidic primers could improve the sealing of composite resin restorations.

CONCLUSIONS

In enamel, no microleakage was found when phosphoric acid was used, but self-etching adhesives showed slight leakage. The gingival sealing was inferior to the occlusal sealing. XNO and CLF obtained the best gingival sealing, followed by XPB, SBX, P&B, and IBO, with SYN demonstrating the worst sealing. Self-etching adhesives and XPB were able to hermetically seal the dentin tubules (negative permeability). Thermocycling did not affect the occlusal bonding or XPB gingival sealing.

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Clinical relevance: Phosphoric acid ensures hermetic and durable union on enamel. Although there are some excellent adhesives, it is necessary to improve the dentin sealing.