Shear Bond Strengths of Self-etching Adhesives to Caries-affected Dentin on the Gingival Wall

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Received August 23, 2005/Accepted November 25, 2005

The purpose of this study was to evaluate the bonding ability of five current self-etching adhesives to caries-affected dentin on the gingival wall. Seventy extracted human molars with approximal dentin caries were employed in this study. In order to obtain caries-affected dentin on the gingival wall, grinding was performed under running water. Following which, specimens mounted in acrylic blocks and composite resins of the bonding systems were bonded to dentin with plastic rings and then debonded by shear bond strength. With Optibond Solo Plus Self-Etch, bonding to caries-affected dentin showed higher shear bond strength than AQ Bond, Tyrian SPE & One-Step Plus, and Prompt-L-Pop (p<0.05). Further, the bond strengths of Clearfil SE Bond and Optibond Solo Plus Self-Etch to sound dentin were higher than those of Prompt-L-Pop, AQ Bond, and Tyrian SPE & One-Step Plus (p<0.05). In conclusion, besides micromechanical interlocking through hybrid layer formation, bond strength of self-etch adhesives to dentin may be increased from additional chemical interaction between the functional monomer and residual hydroxyapatite. The results of this study confirmed that differences in bond strength among self-etching adhesives to both caries-affected and sound dentin were due to chemical composition rather than acidity.

Key words: Self-etch adhesives, Caries-affected dentin, Gingival wall

INTRODUCTION

Resin composite materials are increasingly used as alternatives to amalgam for Class II cavities in posterior teeth. Satisfactory esthetics and longevity have been reported in smaller restorations, but in larger restorations long-term success is more difficult to attain. Poor marginal adaptation and considerable leakage have been shown in vitro in cavities with the cervical margin located at or below the cervicoenamel junction. Furthermore, dentinal tubule orientation on the gingival wall of approximal cavities is different from that on the pulp chamber as a substrate. In contrast to Class I cavities, the gingival wall of Class II cavities have the disadvantage of limited sight. As a result, dentists have considerable difficulties in cavity removal and in producing Class II restorations with good adaptation. It is speculated that the caries-affected dentin in this area may be the reason behind the decrease in bond strength of resin composite materials.

Caries-affected dentin, the hard, sometimes stained dentin beneath excavated carious lesions that often forms a portion of many cavity preparations. It is not normal dentin, because the tubules are occluded with mineral crystals, but it is bacteria-free. However, when self-etch or total-etch adhesives are used on caries-affected dentin, several potential problems may affect their bonding and sealing efficacy. Caries-affected dentin is partially demineralized, and carious intertubular dentin exhibits a higher degree of porosity than sound intertubular dentin due to mineral loss. In addition, hybrid layers in caries-affected dentin are thicker than those in sound dentin, suggesting easier diffusion of acidic conditioners and adhesive monomers due to increased porosity in the intertubular dentin. Conversely, resin infiltration into dentinal tubules is severely hampered by the presence of acid-resistant mineral casts within dentinal tubules of both caries-affected and caries-infected dentin. This can cause a decline in resin retention, particularly when relatively mild-acting self-etching primers are used.

A number of adhesive systems have been developed in an attempt to reduce the steps of and simplify the clinical bonding procedures. A self-etch approach involves either a one- or two-step application procedure. The self-etch effect should be ascribed to the monomers which contain one or more carboxylic or phosphate acid groups. Depending on the degree of etching aggressiveness, self-etching adhesives can be categorized as “strong” or “mild.” “Strong” self-etch adhesives usually have a pH of 1 or below. This high acidity results in rather deep demineralization effects. “Mild” self-etch systems, in general,
have a pH of about 2 and demineralize dentin only up to a depth of 1 μm. Recently, some newly marketed adhesives cannot be classified as "mild" or "strong" two-step self-etching adhesives. The pH of their self-etching primers is about 1.5 based on their action with dentin. As such, authors have referred to them as "intermediary strong" two-step self-etch adhesives.

Good bonding to dentin on the gingival wall is important on two fronts: it ensures the longevity of a restoration and prevents marginal microleakage and secondary caries. Against this background, it is necessary to compare the bond strength to both caries-affected and sound dentin on the gingival wall of approximal cavities. However, only scant information is available concerning dentin on the gingival wall. The purpose of this study, therefore, was to investigate the bond strength of five different self-etching/self-priming adhesive systems to caries-affected and sound dentinal tissues on gingival wall by a shear bond test.

MATERIALS AND METHODS

Seventy extracted human molars—with only mesial or distal approximal dentin caries—stored in isotonic saline at +4°C were employed in this study. The gingival wall of caries lesions was about 1 mm above cementoenamel junction (Fig. 1A). By means of a low-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under running water, all teeth were cut perpendicular to long axis of the tooth from the gingival margin of carious lesion to expose a flat bonding surface. To remove caries-infected dentin, grinding with 320-grit silicon carbide abrasive papers was performed under running water using the combined criteria of visual examination, degree of hardness to a sharp excavator, and staining with a caries detector solution (Kuraray Co. Ltd, Osaka, Japan). Relatively soft, dark-red-stained dentin was classified as caries-infected dentin, while discolored, harder dentin that was stained pink was classified as caries-affected. Yellow, hard dentin was classified as sound dentin. Both caries-affected and sound dentin surfaces were obtained from the same tooth (Fig. 1B, Fig. 2). Caries-affected and sound dentin surfaces at mutual gingival walls of the same tooth were on the same level (Fig. 2). Diameter of the caries-affected dentin area used was measured by a digital caliper (Mitutoyo, Tokyo, Japan) before bonding process. When the diameter was less than 3 mm (Fig. 2), these teeth were not used in this study.

Roots of teeth were mounted in 3-cm diameter circular molds using chemically cured acrylic resin. The teeth were placed such that the bonding surface was parallel to the base of the mold. Caries-affected and sound dentin surfaces at mutual gingival walls of the same tooth were hand polished with 320-grit silicon carbide abrasive papers under running water before bonding procedure. The surfaces were re-examined after polishing to ensure that its orientation was not altered.

The teeth were randomly divided into five groups, each containing 14 teeth. As for self-etch adhesives, they can be subdivided according to their application procedure and acidity (thus their etching aggressiveness). In terms of application procedure, two-step and simplified one-step self-etch adhesives exist. In terms of acidity, three categories of self-etch adhesives are available: mild (pH 2), intermediary (pH 1.5), and strong (pH 1). In this study, the adhesives used were divided into three groups: mild (AQ Bond, Clearfil SE Bond, One-Step Plus Tyran SPE), intermediary (Optibond Solo Plus SE), and strong (Prompt-L-Pop). Manufacturers, primer acidity measurements, bonding components, adhesive procedures, and respective resin composite

Fig. 1 Tooth with approximal caries (A) and cylindrical composite attachments at mutual gingival walls of the tooth (B).

Fig. 2 Caries-affected and sound dentin surfaces at gingival walls on the same level of a tooth.
materials of the five commercially available self-etching adhesive systems used in this study are shown in Table 1.

After completion of the bonding procedures according to manufacturers' instructions, composite resins were added to the surface by packing the material into cylindrical-shaped plastic molds with an internal diameter of 2.3 mm and height of 3 mm (Ultradent, Utah, USA) (Fig. 1B). Excess composite was carefully removed from periphery of molds with an explorer. Bonding agents and composites were cured with a curing light (Hilux 250, Benlioglu, Ankara, Turkey) according to the stipulated curing time, where light intensity was at least 400 mW/cm².

Specimens were then stored in distilled water at 37 °C until the specimens were applied to a shear bond test in a universal testing machine (Testometric Micro 500, Lancashire, England). A notched blade apparatus (Ultradent, Utah, USA) was placed directly over the resin stub flushed against the tooth, and a cross-head speed of 1 mm/min was applied to each composite block until failure occurred. To determine the shear bond strength in MPa, maximum load (N) was divided by the cross-sectional area of the bonded composite block.

Failure mode of each fractured specimen was determined by means of an optical stereomicroscope at ×20 magnification (Olympus SZ4045 TRP, Tokyo, Japan).

### Table 1 Manufacturers, primer acidity, components, and application procedures of the test materials used in the study

<table>
<thead>
<tr>
<th>Bonding system</th>
<th>Primer acidity</th>
<th>Bonding components</th>
<th>Composite</th>
<th>Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prompt-L-Pop (PLP)</td>
<td>Strong</td>
<td>Water, methacrylated pentaacrylate, phosphoric acid esters, phosphine oxide, stabilizer fluoride complex, parabenes</td>
<td>Pertac II f (15s), a, e (20s)</td>
<td></td>
</tr>
<tr>
<td>3M ESPE, St. Paul, MN, USA</td>
<td>pH=0.70</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AQ Bond (AQB)</td>
<td>Mild</td>
<td>4-META, acidic methacrylate monomer, 40% acetone</td>
<td>Metafil-AP d (30s), a, d, a, e (10s)</td>
<td></td>
</tr>
<tr>
<td>Sun Medical, Japan</td>
<td>pH=2.30</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Clearfil SE Bond (CSE)</td>
<td>Mild</td>
<td>Primer: MDP, HEMA, hydrophilic dimethacrylate, dL-Camphoroquinone, N,N-Diethanol-p-toluidine, water</td>
<td>Clearfil AP-X c (20s), a</td>
<td></td>
</tr>
<tr>
<td>Kuraray, Osaka, Japan</td>
<td>pH=2.04</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Optibond Solo Plus</td>
<td>Intermediary</td>
<td>Primer: HEMA, GPDM, MMEP, ethanol, water</td>
<td>Point 4 c, a</td>
<td></td>
</tr>
<tr>
<td>Self-Etch (OSP)</td>
<td>pH=1.20</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kerr, Orange, CA, USA</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>One-Step Plus/Tyrian SPE (TSPE)</td>
<td>Mild</td>
<td>Adhesive: Bis-GMA, HEMA, GPDM, silicate glass filler</td>
<td>d, a, e (10s)</td>
<td></td>
</tr>
<tr>
<td>Bisco Dental, Schaumburgh, IL, USA</td>
<td>Primer A, pH=5.60</td>
<td>Primer A: Ethanol;</td>
<td>a</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Primer B, pH=0.80</td>
<td>Primer B: 2-Acrylamido-2-methyl propanesulfonic acid, Bis (2-(methacryloxy)ethyl) phosphate, ethanol</td>
<td>Pyramid b, c (2 coats), a</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

Procedure steps: a, air-dry; b, mix primer; c, apply primer; d, apply adhesive; e, light cure; f, apply self-etching

Abbreviations: Bis-GMA, bisphenyl-glycidyl-methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloxydecyl dihydrogen phosphate; HPMA, hydroxypropylmethacrylate; PENTA, dipentaerythritol pentaacrylate phosphoric acid; TEQDMA, triethylene glycol dimethacrylate; GPDM, glycerophosphoric acid dimethacrylate; MMEP, mono (2-methacryloxy ethyl) phthalate; BPDM, biphenyl dimethacrylate
BONDING TO CARIES-AFFECTED DENTIN

The Japanese Society for Dental Materials and Devices (JSDMD)

Failure modes were designated as adhesive fracture (if 90-100% of the bonded interface failed between the dentin and composite resin), cohesive fracture (if 90-100% of the failure was in the composite resin or dentin), or mixed fracture (if failure was partially adhesive and partially cohesive resin fracture and/or dentin fracture).

Since the data were normally distributed, two-way ANOVA and Tukey's HSD post-hoc tests were used to test the differences in shear bond strength to both caries-affected and sound dentin among the materials. Paired samples t-test was then used to test the differences in shear bond strength for each adhesive material between caries-affected and sound dentin in the same tooth. Frequency of fracture modes was analyzed using Kruskal-Wallis and Mann-Whitney U tests. Level of statistical significance was set at 0.05.

RESULTS

Table 2 summarizes the results of shear bond test. Among the different types of bonding system, ANOVA analysis showed significant differences in the mean values of bond strength to both caries-affected (p<0.05) and sound dentin (p<0.05). Among the materials applied to sound dentin, CSE and OSP showed higher shear bond strengths than PLP, AQB, and TSPE (p<0.05). As for bond strength to caries-affected dentin, CSE showed the highest bond strength (p<0.05). Following which, OSP showed higher shear bond strength than AQB, TSPE, and PLP (p<0.05) for bonding to caries-affected dentin.

Statistically significant differences were found between the bond strengths to caries-affected and sound dentin for all materials used (p<0.05). While CSE, OSP, and PLP showed high bond strength to sound dentin, AQB and TSPE showed high bond strength to caries-affected dentin.

Table 3 summarizes the failure patterns of the specimens to both caries-affected and sound dentin. Cohesive failures were rarely observed. In terms of adhesive failure, AQB and TSPE showed this failure pattern for both caries-affected and sound dentin. With PLP, adhesive failure percentage was 71% for caries-affected dentin and 100% for sound dentin. With OSP, adhesive failure was approximately 50% for both caries-affected and sound dentin. With CSE, adhesive failure of 64% was observed for sound dentin. However, for caries-affected dentin, adhesive failure percentage was 36% — lower than that of mixed failure percentage (57%).

DISCUSSION

Orientation of dentinal tubules differs at the occlusal and gingival walls of a cavity. Therefore in this study, the bond strength of five self-etch adhesives was investigated on the gingival walls of approximal caries lesions. With CSE, OSP, and PLP, the bond strength to sound dentin was higher than that to caries-affected dentin. Conversely, the bond strengths of AQB and TSPE to caries-affected dentin were

Table 2 Shear bond strength values (Mean±SD, MPa) of materials to caries-affected and sound dentin (n=14)

<table>
<thead>
<tr>
<th>Bonding agent</th>
<th>Sound dentin*</th>
<th>Significance (P)**</th>
<th>Caries-affected dentin*</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSE</td>
<td>34.09±5.49a</td>
<td>0.012</td>
<td>29.25±4.28a</td>
</tr>
<tr>
<td>OSP</td>
<td>29.25±6.39b</td>
<td>0.005</td>
<td>22.95±7.91b</td>
</tr>
<tr>
<td>PLP</td>
<td>16.02±4.29c</td>
<td>0.000</td>
<td>10.91±3.09b</td>
</tr>
<tr>
<td>AQB</td>
<td>7.02±2.02d</td>
<td>0.000</td>
<td>12.28±4.03c</td>
</tr>
<tr>
<td>TSPE</td>
<td>7.44±3.20d</td>
<td>0.000</td>
<td>11.15±2.91a</td>
</tr>
</tbody>
</table>

* Same letters in the same column indicate no statistical differences according to Tukey's HSD test.

** Differences between caries-affected and sound dentin according to paired samples t-test.

Table 3 Failure modes of the bonding agents used after shear bond testing (A: Adhesive, M: Mixed, C: Cohesive)

<table>
<thead>
<tr>
<th>Bonding system</th>
<th>Sound dentin</th>
<th>Caries-affected dentin</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A (%)</td>
<td>M (%)</td>
</tr>
<tr>
<td>CSE</td>
<td>9 (64)</td>
<td>3 (22)</td>
</tr>
<tr>
<td>OSP</td>
<td>7 (50)</td>
<td>6 (43)</td>
</tr>
<tr>
<td>PLP</td>
<td>14 (100)</td>
<td>0</td>
</tr>
<tr>
<td>AQB</td>
<td>14 (100)</td>
<td>0</td>
</tr>
<tr>
<td>TSPE</td>
<td>13 (93)</td>
<td>1 (7)</td>
</tr>
</tbody>
</table>

Same letters in the same column indicate no statistical differences (p>0.05).
higher than those to sound dentin. On the overall, the bond strengths of AQB and TSPE were significantly lower than those of CSE and OSP to both dentin types.

To create strong adhesion as well as perfect sealing of the enveloped collagen fibers, it is essential that there is complete penetration of resin monomers into the demineralized dentin\(^2\). It is theoretically possible for adhesive resin monomers to penetrate further into less mineralized-and more porous, caries-affected demineralized dentin. On this note, there is concern that some acidic conditioners may not be strong enough to etch sclerotic or caries-affected dentin adequately. Further, in caries-affected intertubular dentin, the mineral occupying the interfibrillar space may be different from that of normal apatite due to cyclic demineralization-remineralization\(^1\). This difference may influence hybrid layer formation\(^3\), as well as chemical interaction with carboxylic and phosphate derivatives of methacrylates\(^4\). Therefore, the primary cause for lower bond strength to caries-affected dentin is probably due to the different components of each material rather than the acidity of self-etching primer systems used in this study.

"Strong" self-etch adhesives usually have a pH of 1 or below. This high acidity results in rather deep demineralization effects\(^1\). Dentin collagen is exposed and nearly all hydroxyapatite is dissolved. Consequently, the underlying bonding mechanism of strong self-etch adhesives is primarily diffusion-based, similar to etch-and-rinse approach. Such lower pH self-etch adhesives have often been documented with rather low bond strength values—especially at dentin, and quite a high number of pre-test failures was recorded when testing was done following a microtensile bond strength approach\(^5\). In this study, PLP—a "strong" self-etch adhesive containing methacrylated phosphoric acid esters as an adhesion promoter—showed low shear bond strengths to both caries-affected and sound dentin when compared to "mild" CSE and "intermediary" OSP.

Generally, "mild"-self-etch systems have a pH of about 2 and they superficially demineralize the dentin surface. This superficial demineralization occurs only partially, keeping residual hydroxyapatite still attached to collagen. Nevertheless, sufficient surface porosity is created to obtain micromechanical interlocking through hybridization. The preservation of hydroxyapatite on collagen fibers may serve as a receptor for additional chemical bonding\(^6\). The CSE bonding system included MDP—which has been suggested to facilitate chemical bonding with hydroxyapatite. As such, CSE indicated the highest bond strength to both dentin types. Further, it should be highlighted again that the hydroxyapatite of caries-affected dentin may be different from that of normal apatite due to cyclic demineralization-remineralization\(^1\).

The pH value of "intermediary strong" self-etching primers is about 1.5. OSP, as an "intermediary strong" self-etching primer, showed lower bond strengths to both caries-affected and sound dentin than CSE (a "mild" self-etch adhesive).

Self-etching adhesives contain adhesion promoting agents in both their primer and adhesive components. With OSP, the etching component was glycerophosphoric acid dimethacrylate (GPDM), a phosphate ester. With CSE, the adhesion promoting agent used was MDP. In both cases, these monomers must have played an important role in yielding higher bond strengths for these bonding systems. The results of this study confirmed those of Sengun et al.\(^5\), suggesting that the strength of adhesion to dentin depends on two factors: type of adhesive system used and type of dentin. Yoshiyama et al.\(^6\) measured the bond strength of two resin bonding systems to sound and caries-affected dentin. Using Fluro Bond and Single Bond, they obtained lower bond strengths to caries-affected dentin than to normal dentin. Similarly, Nakajima et al.\(^7\) indicated that bond strength to caries-affected dentin with Single Bond was always lower than that to normal dentin, regardless of the strength of phosphoric acid. By means of the moist bonding technique (ART Bond), they also found that there were no significant differences in bond strength between sound and caries-affected dentin.

In this study, two-bottle systems CSE and OSP showed also higher shear bond strengths to both caries-affected and sound dentin than one-bottle systems, AQB and PLP. As for two-bottle, self-etch/self-priming adhesive system TSPE, it showed lower bond strengths than those of CSE and OSP in this study. We speculated that this could be due to the high pH value of this material.

Based on the results of this study, it was shown that the acidity of self-etching bonding systems did not directly influence their bonding ability to dentin. Although CSE and AQB had similar acidity, they yielded different bond strength values. Likewise, PLP and TSPE—with similar acidity—showed different bond strengths to sound dentin, but not caries-affected dentin. Therefore, with regard to bonding ability to dentin, components such as adhesion promoting agent of self-etch adhesive systems may play a more critical role than acidity.

**CONCLUSION**

The results of this study confirmed that differences in bond strength of self-etching adhesives to both caries-affected and sound dentin were due to their chemical composition rather than acidity. In particular, these results also indicated that the performance of an adhesive may depend on the composition of a
specific ingredient, such as the adhesion promoting agent. Furthermore, it should be noted that primer treatment on both dentin surfaces may cause changes in two aspects: bond strength to dentin and the structure of hybrid layer on each dentin surface. On this note, further research is needed to investigate the effect of self-etch systems’ chemical bonding efficiency.

ACKNOWLEDGEMENTS

The authors profusely thank Mr Yusuf Kitiz and Mr Turgut Endes from KOSGEB for their most generous technical assistance in this study.

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