Long-term bonding durability of four dentin adhesive systems

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We attempted to estimate the in vitro bonding durability of four adhesive systems to dentin. The tensile bond strength (TBS) was determined for specimens immersed for 1 day, 1 month, 3 months, 6 months, 1 year and 2 years. The two etching systems used were Scotchbond MP and Single Bond, and the two self-etching primer systems were Fluoro-bond and Linerbond IIΣ. The bovine dentin surfaces were polished with SiC paper up to 600 grit before application. TBS was significantly reduced with the Scotchbond MP system at 1 year, and degradation of the dentin surface was seen after 6 months. TBS was stable and adhesive failures were observed with the Single Bond system during all test periods. TBS was significantly decreased with the Fluoro-bond system, and the hybridized smear layer was observed at 1 year. With the Linerbond IIΣ system, the TBS was stable during all test periods and there were fractured surfaces that included mixed failure of the adhesive, and cohesive failure in sound dentin. We concluded that the demineralized dentin and hybridized smear layer compromised the bond strength of the composite resin to dentin, and that the remaining demineralized dentin was less durable than the hybridized smear layer in the subsurface of the dentin surface created with the SiC 600 grit sandpaper. (J Osaka Dent Univ 2006; 40: 19-23)

Key words: Composite resin; Dentin; Bonding durability

INTRODUCTION

Adhesive composite resin restorations are often used to treat caries in the anterior teeth and molars. Adhesion of composite resin to dentin became a reality in the mid 80's with introduction of the Gluma system¹,² that employed a dentin primer. Since then, there have been marked advances in the adhesive system, and three-step systems consisting of an acid etchant, a dentin primer and a bonding resin became widely available.³,⁴ Today, more refined two-step systems are most common. One such system is an acid etching-priming adhesive type system⁵,⁶ that incorporates a wet technique and combines a dentin primer and a bonding resin. Another is a self-etching primer type system⁷,⁸ that eliminates acid etching by adding an acidic monomer to a primer. However, because these systems are relatively new, clinicians are not certain about their long-term durability. We conducted tensile bond strength tests following long-term water immersion to ascertain the bonding durability of two acid-etching systems and two self-etching primer systems.

MATERIALS AND METHODS

Table 1 shows the systems used in the present study. The extracted bovine teeth were polished using SiC paper up to #600 grit to prepare flat dentin surfaces. On each surface, a brass mold with an internal diameter of 3.0 mm was attached using double-sided adhesive tape. Composite resin was applied and cured by irradiating light for 40 seconds, according to the instructions provided in the manual for each system. After storing in water at 37°C for 1 day, 1 month, 3 months, 6 months, 1...
year or 2 years, tensile bond strength was measured at a crosshead speed of 0.3 mm/min, and the mean and standard deviation were calculated for ten samples. One-way ANOVA was used to statistically compare bond strengths at different periods of immersion. After the test, fractured surfaces were gold-coated and analyzed by SEM (JSM-5610 LV, JEOL Inc., Tokyo) at ×2,000 magnification.

RESULTS

Table 2 shows the results of the tensile bond strength test, and Figures 1 through 4 show the SEM images of fractured dentin surfaces after the tensile test. With the Scotchbond MP system, the bond strength after 1 day of water immersion was 14.8 MPa, and there was no significant difference in this value up to 6 months. However, after 1 year, the bond strength decreased significantly to 2.8 MPa. SEM of fractured dentin surfaces showed adhesive failures up to 3 months. However, after 6 months, almost all of the failures were cohesive and in the demineralized dentin. The interface crumbled, and in many areas, resin tags appeared loose (Fig. 1).

With the Single Bond system, the bond strength after 1 day of water immersion was 14.1 MPa, and there was no significant decrease after 2 years. At 2 years, failures were for the most part adhesive in nature (Fig. 2). With the Fluorobond system, the bond strength after 1 day of water immersion was 23.2 MPa. While no significant decrease was seen for 6 months, there was a significant decrease to 15.5 MPa after 1 year. After 6 months, the failures were primarily mixed failures in adhesion, and cohesive failures in the bonding resin. However, after 1 year, adhesive failures were more common (Fig. 3).

With the Linerbond ILΣ system, the bond strength

Table 1 Materials

<table>
<thead>
<tr>
<th>Scotchbond MP</th>
<th>Etchant</th>
<th>Primer</th>
<th>Adhesive</th>
<th>Composite resin</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 M ESPE, St Paul, MN, USA</td>
<td>35% H 3 PO 4 (6 PU)</td>
<td>Polyalkenate-copolymer, HEMA, water (5 KP)</td>
<td>HEMA, Bis-GMA (6 DC)</td>
<td>Restorative Z 100 Shade A 3 (5 GJ)</td>
</tr>
<tr>
<td>Single Bond</td>
<td>Etchant</td>
<td>Priming adhesive</td>
<td>Composite resin</td>
<td></td>
</tr>
<tr>
<td>3 M ESPE, St Paul, MN, USA</td>
<td>35% H 3 PO 4 (7 RJ)</td>
<td>Polyalkenate copolymer, HEMA, Bis-GMA, water, ethanol (7 AF)</td>
<td>Dry, light 10 sec</td>
<td>Restorative Z 100 Shade A 3 (6 MJ)</td>
</tr>
<tr>
<td>Fluorobond Shofu Inc., Kyoto, Japan</td>
<td>Self-etching primer</td>
<td>Adhesive</td>
<td>Composite resin</td>
<td></td>
</tr>
<tr>
<td>A (059728) B (109845)</td>
<td>4-AET, HEMA, water</td>
<td>4-AET, HEMA, UDMA, Filler (039723)</td>
<td>Light 10 sec</td>
<td>LITE-FIL II A Shade A 3 (129160) Light 40 sec</td>
</tr>
<tr>
<td>Clearfil Linerbond II Σ Kuraray Inc., Tokyo</td>
<td>Self-etching primer</td>
<td>Adhesive</td>
<td>Composite resin</td>
<td></td>
</tr>
<tr>
<td>MDP, HEMA, N, N-Diethanol</td>
<td>MDP, HEMA, Bis-GMA, N, N-Diethanol p-toluidine, water</td>
<td>Filler (039723)</td>
<td>Light 20 sec</td>
<td>Clearfil APX Shade A 3 (0449) Light 40 sec</td>
</tr>
</tbody>
</table>

HEMA: 2-Hydroxyethyl methacrylate, Bis-GMA: Bisphenol-blycidyl methacrylate, 4-AET: 4-Acryloxyethyltrimellitic acid, UDMA: Urethan di-methacryloyloxydecyl dihydrogen phosphate.

Table 2 Tensile bond strength to dentin

<table>
<thead>
<tr>
<th>Time</th>
<th>Scotchbond MP</th>
<th>Single Bond</th>
<th>Fluorobond</th>
<th>Linerbond II Σ</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 day</td>
<td>14.8±5.3</td>
<td>14.1±3.9</td>
<td>23.2±5.1</td>
<td>24.2±5.0</td>
</tr>
<tr>
<td>1 month</td>
<td>15.0±6.3</td>
<td>12.3±2.8</td>
<td>22.0±7.2</td>
<td>26.9±8.8</td>
</tr>
<tr>
<td>3 months</td>
<td>9.0±3.0</td>
<td>11.2±3.3</td>
<td>20.5±3.9</td>
<td>22.2±5.6</td>
</tr>
<tr>
<td>6 months</td>
<td>10.5±1.7</td>
<td>13.8±5.3</td>
<td>23.0±3.7</td>
<td>25.0±4.3</td>
</tr>
<tr>
<td>1 year</td>
<td>2.8±2.2</td>
<td>13.7±3.2</td>
<td>15.5±4.1</td>
<td>21.7±4.2</td>
</tr>
<tr>
<td>2 years</td>
<td>3.0±2.6</td>
<td>13.6±4.1</td>
<td>15.9±3.0</td>
<td>24.8±3.6</td>
</tr>
</tbody>
</table>

MPa, Mean±SD, Vertical bars indicate no significant difference (p>0.05).
Fig. 1 Fractured dentin surface of Scotchbond MP (SEM, ×2000).
A: Adhesive failure, DD: Cohesive failure in demineralized dentin.

Fig. 2 Fractured dentin surface of Single Bond (SEM, ×2000).

Fig. 3 Fractured dentin surface of Fluorobond (SEM, ×2000).
HSL: Hybridized smear layer, BR: Cohesive failure in bonding resin.
after 1 day of water immersion was 24.2 MPa. There was no significant change even after 2 years. Failures at all time points were primarily mixed adhesive failures and failures of cohesion in sound dentin (Fig. 4).

**DISCUSSION**

Adhesion of composite resin to dentin has rapidly advanced with the introduction of dentin primers. Since that time, resin systems adhering to both enamel and dentin have become indispensable in dental practice. Dentin adhesion is thought to be achieved by fairly complicated processes, which include smear-layer processing and hybrid layer formation. It has not been clarified whether the initial adhesive strength can be maintained for a long period of time. Therefore, in the present study, we prepared specimens using either an acid-etching system or a self-etching primer system, and measured the tensile bond strength after long-term water immersion. SEM observations were also performed to analyze the fractured surfaces.

Scotchbond MP is a conventional three-step system. After 6 months of water immersion, specimens bonded with this system showed marked interface degradation on the fractured surfaces, and there was significant decrease in the bond strength after 1 year. With adhesive systems in which dentin is treated using an acid etchant, there is a demineralized layer not impregnated by resin below the hybrid layer. Studies have reported that adhesion is compromised when this area is hydrolyzed after being subjected to long-term water immersion. The main reason for this is that the collagen fiber layer of demineralized dentin contracts after acid etching owing to the air-drying that follows water-rinsing. This hinders penetration of the bonding monomer.

While the role of a dentin primer is to expand this shrunken collagen layer again, our results suggest that it is difficult for conventional three-step systems to recover this layer to its original state.

However, with Single Bond, which is a two-step system based on phosphoric acid etching in which a wet technique is used after rinsing, there was no significant decrease in bond strength to dentin after 2 years of water immersion. Wet bonding suppresses drying after acid etching and rinsing. This helps to prevent contraction of the demineralized collagen layer. The use of an alcohol or acetone solvent in a bonding resin facilitates penetration of the bonding monomer through water substitution. Although hydrolysis of the remaining demineralized dentin occurs as quickly as 1 month, it was rarely seen in the samples we examined, even after 2 years. This supports the effectiveness of wet bonding.

On the other hand, the Fluorobond system does not involve phosphoric acid etchant; it is a two-step system utilizing a self-etching primer. With Fluorobond, the bond strength after 1 day was high.
(23.2 MPa). Although this level was maintained after 6 months, it decreased significantly to 15.5 MPa after 1 year, at which time the fractured surfaces showed many areas that appeared to be smear layers remaining in the interfaces. These hybridized smear layers are considered fragile parts of an adhesion system. When these areas were hydrolyzed, the bond strength decreased after 1 year. However, even with the decrease, the bond strength remained at 15 MPa, and fractured surfaces showed no dentin degradation. As a result, we concluded that the hybridized smear layer in self-etching primer systems is more durable than the demineralized dentin in acid-etching systems.

Like Fluoro bond, Linerbond IIE is a two-step self-etching primer system. Its bond strength was maintained above 20 MPa after 2 years, and fractured surfaces mostly consisted of mixed failures of adhesion and failures of cohesion in sound dentin. Failures did not occur in the hybridized smear layer. Therefore, compared with Fluoro bond, Linerbond IIE either removed the smear layer better or the bonding monomer penetrated the dentinal substrates better. In either case, the result was suppression of formation of the hybridized smear layer. The above findings suggest that dentin adhesion is stable for a longer period of time with self-etching primer systems than with acid etching systems. However, it has been reported that the ability of a self-etching primer to remove a smear layer is affected by cutting conditions, and that if a dentin surface is covered by a thick smear layer, acid etching with powerful demineralization may be more effective than treatment with self-etching primer. This point requires further investigation.

REFERENCES