Bond strength of two adhesive systems with self-etching primer

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Self-etching primers that treat both enamel and dentin have recently been developed. We evaluated the in vitro bond strength of two self-etching primer adhesive systems that bond to enamel and dentin: UniFil Bond/UniFil F (GC Inc., Tokyo, Japan) and Fluoro-bond/Beautifil (Shofu Inc., Kyoto, Japan). The facial surfaces of bovine incisors were ground with wet SiC paper up to 600 grit to prepare flat bonding surfaces on either enamel or dentin. The adhesive systems were applied according to the manufacturer's instructions, with an additional 15 seconds of phosphoric acid etching on the enamel. The tensile bond strengths were determined at a cross head speed of 0.3 mm/min after immersion in water at 37°C for one day, and thermocycling between 5°C and 55°C in water 5,000 times. The bond strength to dentin was satisfactory for both systems. However, although the bond strength to enamel was not durable, it was improved with phosphoric acid etching. These results suggest that enamel etching with phosphoric acid is recommended when using these self-etching primer adhesive systems. (J Osaka Dent Univ 2004; 38: 1–5.)

Key words: Self-etching primer; Composite resin; Bond strength

INTRODUCTION

Since the concept of modifying the enamel structure of teeth with phosphoric acid was first introduced by Michael Buonocore in 1955,1 the acid-etch technique has become a standard procedure for bonding composite resin restorations to enamel. However, it has not been as easy to achieve bonding to dentin. Early dentin bonding systems required many clinical steps to clean and prime the dentin to accept the adhesive resin.2 In an effort to simplify the bonding procedures, manufacturers have combined the dentin conditioning and priming steps with what is called "self-etching primer".3-4 Adhesive systems with self-etching primer have recently been developed and the bonding procedure has been simplified to two steps, which are simultaneously applied to both the enamel and dentin.5-9 These systems are easy to use and have potential for good clinical success.

It has been reported that self-etching primer systems produce high bond strength to dentin because they cause less damage to the dentinal surface than the acidic conditioners.5-6 However, some researchers have been concerned about lower enamel bond strength compared to conventional acid-etch systems.7-8 The purpose of this study was to determine the tensile bond strengths both to enamel and to dentin of the two self-etching primer adhesive systems. In addition, we evaluated the effect on enamel bond strength of a supplemental application of phosphoric acid etchant.

MATERIALS AND METHODS

We used 120 extracted bovine incisors that had been frozen. The facial surface of each tooth was ground with wet SiC paper up to 600 grit to prepare flat bonding surfaces. A brass mold having a 3.0 mm inner diameter was fixed on the bonding surfaces with double-sided adhesive tape to standard-
ize the bonding area. This area was then treated with one of two self-etching primer adhesive systems, either UniFil Bond/UniFil F (GC Inc., Tokyo, Japan) or Fluorobond/Beautifil (Shofu Inc., Kyoto, Japan), according to the manufacturer's instructions (Table 1). In addition, 15 seconds of phosphoric acid etching was done on the enamel using Scotchbond etching gel (3M Inc., St Paul, USA). Composite resin was placed on the tooth and light cured for 40 seconds (n=10). The tensile bond strength was determined at a cross head speed of 0.3 mm/min with a universal testing machine (IM-20, Intesco Inc., Tokyo, Japan) after immersion in 37°C water for one day (TC 0), and after thermocycling between 5°C and 55°C in water baths with a 30 seconds dwell time for 5,000 cycles (TC 5000). The data obtained were analyzed with ANOVA and Sheffe's test at the 5% level to determine statistical significance. Fractured enamel or dentin surfaces that resulted from tensile stressing were examined with a scanning electron microscope (JSM 5400, JEOL Inc., Tokyo, Japan) after gold-sputtering.

RESULTS

1. Tensile bond strength to enamel and dentin

Table 2 shows the tensile bond strength results for each system. Both of the systems demonstrated significantly degraded enamel bond strength after thermocycling (p<0.05). Acid conditioning improved the bond strength significantly (p<0.01). Thermocycling did not produce a significant decrease in the dentin bond strength.

2. Scanning electron microscope observations

Figures 1 and 2 show tensile fractured surfaces of enamel and dentin for each system. In the enamel, fractured surfaces for the UniFil Bond system were mixed failures (cohesive failure in the composite resin and adhesive failure with the enamel) without

### Table 1 Materials

<table>
<thead>
<tr>
<th>System</th>
<th>Self-etching Primer</th>
<th>Bonding resin</th>
<th>Composite resin</th>
</tr>
</thead>
<tbody>
<tr>
<td>UniFil Bond</td>
<td>4-MET, HEMA, UDMA, water, ethanol, photoinitiator (9905311) 20 sec application, air-dry</td>
<td>HEMA, UDMA, TEGDMA, primer, photoinitiator (9905311) 10 sec light cure</td>
<td>UniFil F shade A 3 40 sec light cure</td>
</tr>
<tr>
<td>(GC)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Imperva-Fluorobond</td>
<td>A: water, catalyst (070061)</td>
<td>4-AET, HEMA, UDMA, TEGDMA, primer, photoinitiator (080071) 10 sec light cure</td>
<td>Beautifil shade A 3 40 sec light cure</td>
</tr>
<tr>
<td>(Shofu)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4-MET: 4-Methacyloxyethyl trimellitic acid, HEMA: 2-Hydroxyethyl metacrylate, 4-AET: 4-Acryloxyethyl-trimellitic acid, 4-AETA: 4-Acryloxyethyltrimellitate anhydride, UDMA: Urethane di-methacrylate, TEGDMA: Triethylenglycol di-methacrylate

### Table 2 Tensile bond strength

<table>
<thead>
<tr>
<th>Tooth structure</th>
<th>Etching</th>
<th>Thermocycling</th>
<th>UniFil Bond</th>
<th>Fluorobond</th>
</tr>
</thead>
<tbody>
<tr>
<td>Enamel</td>
<td>−</td>
<td>0</td>
<td>12.8±3.0</td>
<td>12.6±6.0</td>
</tr>
<tr>
<td>Enamel</td>
<td>−</td>
<td>5000</td>
<td>7.8±2.0</td>
<td>4.5±1.0</td>
</tr>
<tr>
<td>Enamel</td>
<td>+</td>
<td>0</td>
<td>23.8±6.9</td>
<td>24.8±3.7</td>
</tr>
<tr>
<td>Enamel</td>
<td>+</td>
<td>5000</td>
<td>18.1±5.5</td>
<td>19.4±4.6</td>
</tr>
<tr>
<td>Dentin</td>
<td>−</td>
<td>0</td>
<td>19.9±3.2</td>
<td>18.9±6.9</td>
</tr>
<tr>
<td>Dentin</td>
<td>−</td>
<td>5000</td>
<td>14.4±2.6</td>
<td>13.5±5.6</td>
</tr>
</tbody>
</table>

**p<0.01, *p<0.05, NS: No significant difference**

Mean±SD (MPa)
Bond strength of self-etching primer adhesive systems

Fig. 1 Scanning electron microscope views of fractured enamel and dentin surfaces of debonded specimens treated using UniFil Bond with and without thermocycling (×30).
A: adhesive failure, BR: cohesive failure in the bonding resin, CR: cohesive failure in the composite resin, TC 0: No thermocycle, TC 5000: Thermocycle 5000 times.

Fig. 2 Scanning electron microscope views of fractured enamel and dentin surfaces of debonded specimens treated using Fluorobond with and without thermocycling (×30).
A: adhesive failure, BR: cohesive failure in the bonding resin, CR: cohesive failure in the composite resin, TC 0: No thermocycle, TC 5000: Thermocycle 5000 times.
thermocycling, and adhesive failure after thermocycling. Fractured surfaces for the Fluorobond system were mixed failures (cohesive failure in the bonding resin and adhesive failure with the tooth) without thermocycling, and adhesive failure after thermocycling. In the acid etching groups, almost all the failures were cohesive in the bonding/composite resin in both systems irrespective of thermocycling.

In the dentin, failures for the UniFil Bond system were mixed failures (cohesive failure in the composite resin and adhesive failure with the dentin) before and after thermocycling. For the Fluorobond system, mixed failures (cohesive failure in the bonding resin and adhesive failure with the tooth) were observed regardless of thermocycling.

**DISCUSSION**

We found that phosphoric acid etching of enamel prior to application of the commercial self-etching primer adhesive systems significantly increased the bond strength. The low pH of the self-etching primer creates a shallow etching pattern in enamel that might be inadequate for a stable resin tag strong enough to resist thermal stress. Accordingly, acid etching to enamel is desirable to gain stable resin tags and mechanical retention between the enamel and composite resin. In the past, there were several adhesive systems in which only enamel was treated with phosphoric acid and the dentin was then separately treated with self-etching dentin primer. But from the clinical point of view, it is very difficult to selectively etch the enamel surface alone, and it has been reported that the bonding durability of resin to dentin is damaged by phosphoric acid etching. At present, self-etching primers can be used for the simultaneous pretreatment of both the enamel and dentin. Therefore, it is not necessary to etch all lesions with phosphoric acid. A selective, moderate application of etching on the enamel of class I, II and IV cavities that are exposed to high occlusal stress should be done so as to avoid diffusion of the etchant onto the dentin surface. Although a 60-second application time has traditionally been recommended for etching enamel, recent studies have indicated that shear bond strengths and morphology were similar with only 15 seconds of etching. Therefore, we recommend the enamel be etched for 15 seconds prior to application of the self-etching primer.

However, both of the systems we tested had good bond strength to dentin without the phosphoric acid pretreatment. When the prepared dentin is treated with an acidic conditioner to remove the smear layer, demineralized dentin is created in the dentinal subsurface. Phosphoric acid decalcifies a deeper area in the dentin than the self-etching primer because of its stronger acidity. The collagen, exposed as a result of demineralization of dentin, collapses quite easily upon air drying. As a result, the interglobular spaces and pores between the exposed collagen fibrils are eliminated, thereby decreasing monomer permeability. It has been reported that the bonding agent does not infiltrate the entire dentin layer demineralized by the phosphoric acid conditioning. This creates a demineralized zone between the hybrid layer and the intact dentin. This remaining demineralized zone causes poor bonding durability between the resin and dentin and inferior clinical results. However, the acidic adhesive monomer in the self-etching primer decalciifies the dentin moderately, and simultaneously infiltrates the deepest zone of the demineralized dentin. This creates a firm, dense, hybrid layer after polymerization of the bonding resin and no demineralized zone remains. Therefore, we conclude that decalcification of the dentin with self-etching primer is desirable for a durable bond with resin.

**REFERENCES**