Effects of 6 self-etching primers on shear bond strength of orthodontic brackets

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Introduction: This study was conducted to compare the effects of 6 self-etching primers (SEPs) on the shear bond strength (SBS) of orthodontic brackets bonded with the same orthodontic composite resin. Methods: One hundred forty extracted premolars were randomly divided into 7 groups (20 per group). In group I (control), the enamel was etched with 37% phosphoric acid. In the other groups, it was conditioned with SEPs according to each manufacturer’s instructions: group II, Transbond Plus SEP (3M Unitek, Monrovia, Calif); group III, AdheSE (Ivoclar Vivadent AG, Schaan, Liechtenstein); group IV, Primers A and B (Shofu, Kyoto, Japan); group V, Clearfil Mega Bond FA (Kuraray Medical, Tokyo, Japan); group VI, Peak SE and Peak LC Bond (Ultradent Products, South Jordan, Utah); and group VII, Bond Force (Tokuyama, Osaka, Japan). All brackets were bonded with Transbond XT (3M Unitek), and the teeth were then stored (37°C, 24 hours), tested, and statistically analyzed (Scheffé, ANOVA [ P < 0.05], and Weibull analyses). The adhesive remnant index (ARI) was also recorded. Results: Group I (26.5 ± 8.1 MPa) had a significantly higher SBS value than the other groups except group II (21.1 ± 5.2 MPa). There were no significant differences among groups II, V (19.0 ± 4.3 MPa), VI (19.6 ± 5.1 MPa), and VII (18.3 ± 4.4 MPa). The values for groups I, II, and VI were significantly higher than for group III (13.4 ± 4.1 MPa), and the value for group IV (8.8 ± 2.6 MPa) was significantly lower than the values for groups I, II, V, and VII. Significant differences were found in the ARI scores. Conclusions: The SBS values of all groups might be clinically acceptable, and orthodontic brackets can be successfully bonded with Transbond XT after enamel conditioning with any of these SEPs. However, since the SEPs used in groups III and IV significantly affected the bond strength negatively, further studies are warranted to evaluate their effectiveness. (Am J Orthod Dentofacial Orthop 2009;135:424.e1–424.e7)
To find better applications for SEPs in orthodontics, we conducted this study to compare the effects of 6 SEPs on the SBS of brackets bonded with the same orthodontic composite resin. The adhesive remnant index (ARI) was used, and the enamel surfaces conditioned with 37% phosphoric acid or SEPs were observed with a scanning electron microscope (SEM).

MATERIAL AND METHODS

A total of 140 freshly extracted premolars were collected and stored in a solution of 0.2% (wt/vol) thymol. The criteria for tooth selection were similar to those described by Bishara et al." The teeth were cleansed and pumiced with fluoride-free paste (Pressage, Shofu, Kyoto, Japan) and rubber prophylactic cups (Merssage, Shofu) with a low-speed hand piece (10 seconds). The teeth were thoroughly washed with water (30 seconds) and air dried.

Stainless steel premolar brackets (0.018-in standard edgewise, Dyna-Lock, 3M Unitek, Seefeld, Germany) were used. The average surface area of the bracket base was 14.1 mm². This value was obtained by randomly measuring 10 bracket bases.4

The teeth were randomly divided into 7 groups (20 in each group).

In group I, the control, the teeth were etched with 37% phosphoric acid for 15 seconds, washed with a water spray, and dried to a chalky white appearance. The adhesive primer was applied to the etched surface, and the brackets were then put into place. In all groups, the brackets were bonded with Transbond XT (3M Unitek) for 5 seconds and slightly air-dried. The bonding agent was applied, distributed evenly and light-cured for 10 seconds.

In group II, the teeth were conditioned with Transbond Plus SEP (3M Unitek) for 5 seconds and slightly air dried.

In group III, the teeth were conditioned with AdheSE (Ivoclar Vivadent AG, Schaan, Liechtenstein). The primer was brushed onto the tooth surface for 30 seconds and gently air dried. The bonding agent was applied to the enamel surface, gently dispersed with air, and light-cured for 10 seconds.

In group IV, the teeth were conditioned with Primers A and B (Shofu) for 3 seconds and slightly air-dried.

In group V, the teeth were conditioned with Clearfil Mega Bond FA (Kuraray Medical, Tokyo, Japan). The primer was applied to the enamel surface, and 20 seconds later, the surface was dried with a gentle air flow. The bonding agent was applied, distributed evenly with the air flow, and light-cured for 10 seconds.

In group VI, the teeth were conditioned with Peak SE Primer (Ultradent Products, South Jordan, Utah). The conditioner was applied to the enamel surface for 20 seconds with a brush tip and gently air-dried for 3 seconds. Immediately, a thin coat of Peak LC Bond Resin (Ultradent) was rubbed for 10 seconds onto the etched enamel, gently air-dried, and light-cured for 10 seconds.

In group VII, the teeth were conditioned with Bond Force (Tokuyama, Osaka, Japan). The conditioner was applied and rubbed onto the enamel for 20 seconds; then the surface was gently dried with a light air flow for 5 seconds and a strong air flow for 5 seconds. The conditioner was then light-cured for 10 seconds.

A 0.017 x 0.025-in stainless steel wire was ligated into each bracket slot to reduce deformation of the bracket during debonding. The teeth were fixed in acrylic resin, and a mounting jig was used to align the facial surface of the tooth to be parallel to the force during the SBS test. The teeth were then stored in distilled water at 37°C for 24 hours.8

An occluso-gingival load was applied to produce a shear force at the bracket-tooth interface, by using the flattened end of a steel rod attached to the crosshead of a universal testing machine (EZ Graph, Shimazdu, Kyoto, Japan). The bond strengths were measured at a crosshead speed of 0.5 mm per minute, and the load at the time of fracture was recorded in newtons and converted into megapascals (MPa).

Once the brackets had been debonded, the enamel surface of each tooth was examined under 10 times magnification to determine the amount of residual adhesive on each tooth. The adhesive remnant index (ARI) scores were recorded with the following scale: 0, no adhesive left on the tooth; 1, less than half of the adhesive left on the tooth; 2, more than half of the adhesive left on the tooth; and 3, all adhesive left on the tooth, with a distinct impression made by the bracket mesh.

A SEM was used to observe the enamel surface morphology. Specimens of the enamel surfaces etched with 37% phosphoric acid and conditioned with the 6 SEPs were chemically fixed, dehydrated, freeze dried, and coated with osmium as described previously.13 The specimens were then observed with the SEM (S-4500, Hitachi, Tokyo, Japan).

Statistical analysis

Descriptive statistics including means, standard deviations, ranges, Scheffé post-hoc multiple comparisons
Table I. Descriptive statistics of the SBS values for the 7 groups

<table>
<thead>
<tr>
<th>Group (enamel conditioner)</th>
<th>n</th>
<th>Mean (MPa)</th>
<th>SD</th>
<th>Range</th>
<th>Scheffe*</th>
<th>m|</th>
<th>S0 (MPa)|</th>
<th>Pf10 (MPa)|</th>
</tr>
</thead>
<tbody>
<tr>
<td>I (37% phosphoric acid)</td>
<td>20</td>
<td>26.5</td>
<td>8.1</td>
<td>15.2-41.5</td>
<td>A</td>
<td>3.4</td>
<td>29.6</td>
<td>15.3</td>
</tr>
<tr>
<td>II (Transbond Plus SEP)</td>
<td>20</td>
<td>21.1</td>
<td>6.2</td>
<td>6.5-33.6</td>
<td>A, B</td>
<td>2.9</td>
<td>23.9</td>
<td>11.2</td>
</tr>
<tr>
<td>III (AdheSE)</td>
<td>20</td>
<td>13.4</td>
<td>4.1</td>
<td>5.9-20.9</td>
<td>C, D</td>
<td>3.0</td>
<td>15.1</td>
<td>7.2</td>
</tr>
<tr>
<td>IV (Primers A and B)</td>
<td>20</td>
<td>8.8</td>
<td>2.6</td>
<td>5.1-13.6</td>
<td>C</td>
<td>3.5</td>
<td>9.8</td>
<td>5.2</td>
</tr>
<tr>
<td>V (Clearfil Mega Bond FA)</td>
<td>20</td>
<td>19</td>
<td>4.3</td>
<td>13.3-28.5</td>
<td>B, D</td>
<td>4.6</td>
<td>20.8</td>
<td>12.8</td>
</tr>
<tr>
<td>VI (Peak SE and Peak LC)</td>
<td>20</td>
<td>19.6</td>
<td>5.1</td>
<td>9.1-30.4</td>
<td>B</td>
<td>3.7</td>
<td>21.7</td>
<td>12.0</td>
</tr>
<tr>
<td>VII (Bond Force)</td>
<td>20</td>
<td>18.3</td>
<td>4.4</td>
<td>9.3-28.7</td>
<td>B, D</td>
<td>4.1</td>
<td>20.1</td>
<td>11.7</td>
</tr>
</tbody>
</table>

*Scheffé post-hoc multiple comparisons (1-way ANOVA); *P <0.05. Groups with different letters are significantly different from each other.

\|Weibull parameters m and S0 were obtained from cumulative failure probabilities by unweighted least-squares fitting to the data shown in Figure 1. The loads for 10% probability of failure were also calculated as Pf10.

(1-way ANOVA) with significance predetermined at *P <0.05 were calculated for the SBS analysis. Similar to the methods described by Movahhed et al,\textsuperscript{18} Weibull survival analysis was also carried out to provide additional information on the safety of the bonding system performance.\textsuperscript{19} The chi-square test was used to evaluate the ARI scores.

**RESULTS**

The SBS mean values expressed in megapascals and descriptive statistics are shown in Table I. In all groups, the mean values of SBS were higher than the 6.0 to 8.0 MPa considered adequate for bonding orthodontic brackets to teeth.\textsuperscript{20,21} According to the Scheffé post-hoc multiple comparisons, group I (26.5 ± 8.1 MPa) had significantly higher SBS values than the other groups except group II (21.1 ± 6.2 MPa). There were no significant differences among groups II, V (19.0 ± 4.3 MPa), VI (19.6 ± 5.1 MPa), and VII (18.3 ± 4.4 MPa). Groups I, II, and VI had values significantly higher than group III (13.4 ± 4.1 MPa), and the value for group IV (8.8 ± 2.6 MPa) was significantly lower than values for groups I, II, V, VI, and VII. There was no significant difference between groups III and IV. On the other hand, the loads for 10% probability of failure of the Weibull analysis (Fig 1, Table I), corroborated that the SBS values for groups III and IV were lower than those for the other groups; groups III and IV had values under 8 MPa.

The scores indicating the amount of adhesive remaining after debonding are shown in Table II. The chi-square comparisons of the ARI scores among all groups (chi-square = 34.29) indicated that the groups were significantly different (*P = 0.0006). However, there were no scores of 3, and only 1 score of 2 in group VII. In other words, with the methods described in this study, the amount of residual adhesive after debonding...
Table II. Distribution frequency (and percentages) of ARI scores

<table>
<thead>
<tr>
<th>Group (enamel conditioner)</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>I (37% phosphoric acid)</td>
<td>3 (15)</td>
<td>17 (85)</td>
<td>0 (0)</td>
<td>0 (0)</td>
<td>20</td>
</tr>
<tr>
<td>II (Transbond Plus SEP)</td>
<td>6 (30)</td>
<td>14 (70)</td>
<td>0 (0)</td>
<td>0 (0)</td>
<td>20</td>
</tr>
<tr>
<td>III (AdheSE)</td>
<td>8 (40)</td>
<td>12 (60)</td>
<td>0 (0)</td>
<td>0 (0)</td>
<td>20</td>
</tr>
<tr>
<td>IV (Primers A and B)</td>
<td>18 (90)</td>
<td>2 (10)</td>
<td>0 (0)</td>
<td>0 (0)</td>
<td>20</td>
</tr>
<tr>
<td>V (Clearfil Mega Bond FA)</td>
<td>6 (30)</td>
<td>14 (70)</td>
<td>0 (0)</td>
<td>0 (0)</td>
<td>20</td>
</tr>
<tr>
<td>VI (Peak SE and Peak LC)</td>
<td>7 (35)</td>
<td>13 (65)</td>
<td>0 (0)</td>
<td>0 (0)</td>
<td>20</td>
</tr>
<tr>
<td>VII (Bond Force)</td>
<td>9 (45)</td>
<td>10 (50)</td>
<td>1 (5)</td>
<td>0 (0)</td>
<td>20</td>
</tr>
</tbody>
</table>

Chi-square, 34.293; df, 12; P = 0.0006.

was less than 25% (score 1). The scores of groups II, III, V, and VI were comparable. The lowest amount of adhesive was found in group IV, with 18 scores of 0, whereas group I had the highest ARI scores. On the other hand, there were no enamel fractures.

Figure 2 shows the enamel surfaces observed under the SEM after conditioning. The surfaces etched with 37% phosphoric acid, conditioned with Transbond Plus SEP, and Primers A and B (Fig 2, A, B, and D) show the effect of the conditioners on the enamel surface. Conversely, the enamel surfaces conditioned with AdheSE, Clearfil Mega Bond FA, Peak SE and Peak LC Bond Resin, and Bond Force (Fig 2, C, E, F, and G) show the enamel surfaces after the bond components of the SEPs had been light-cured.

DISCUSSION

Pumice prophylaxis could be an important procedure before bonding orthodontic brackets. Previous studies have shown strong evidence suggesting the need for pumice prophylaxis when using a SEP.22,23 The omission of this procedure before orthodontic bonding with a SEP has a significant effect, leading to a higher bond failure rate.23 In contrast, before conventional etching with phosphoric acid, pumicing has been shown to be unnecessary because it has no effect on in-vivo bond failure rates.24

In this study, the brackets in all groups were bonded with the same orthodontic adhesive paste to compare the effects of the 6 SEPs on the SBS. Transbond XT was selected because it has been widely tested in several studies and is commonly for direct bonding.13 Transbond XT is a traditional composite resin filled with diverse sizes of filler particles,25 and it has been shown to have suitable bond strength and properties.13,25,26

Under the conditions of this in-vitro study, the brackets bonded with any of the 6 SEPs showed higher SBS values than those minimally required for orthodontic treatment.20,21 Generally, the application of 37% phosphoric acid increases the bond strength.4 In this context, the highest SBS value yielded by group I (26.5 MPa) was expected, and this was significantly higher than that of any other group except group II, in which the enamel was conditioned with Transbond Plus SEP. There were no significant differences among groups II (Transbond Plus SEP), V (Clearfil Mega Bond FA), VI (Peak SE and Peak LC Bond), and VII (Bond Force). If the bond strength is the main concern of clinicians, Transbond Plus SEP might be routinely used for enamel conditioning. Particularly for the initial bonding (full mouth), the application of this SEP is faster than that of the others; however, the other SEPs can be appropriate alternatives.

The quick and simplified technique of a SEP for enamel conditioning has become popular in orthodontics.9 Although the SEPs used in groups III, V, VI, and VII have been marketed for use in operative dentistry, other restoration-based self-etching systems are used successfully to bond orthodontic brackets.4,16 Specifically, Clearfil Mega Bond FA, a 2-step fluoride-releasing SEP with antibacterial properties, has been sold as Clearfil Protect Bond in other countries than Japan.27,28 In agreement with this study, Clearfil Protect Bond showed suitable bond strength for bonding orthodontic brackets in previous studies,4,28,29 and it might be excellent for patients with a high risk of caries. Bond Force is a recently developed 1-bottle SEP that can be successfully used for enamel conditioning. Its application is slightly faster than 2-bottle systems, and the bond strength in group VII might be adequate for bonding orthodontic brackets. Peak SE and Peak LC Bond had suitable SBS for bonding orthodontic brackets, and this system has the advantage of the use of the conditioner within 30 days after activation. In addition, the disposable brush tips prevent cross contamination between patients; this might be particularly beneficial for rebonding brackets that failed during treatment.

The SBS for group III (AdheSE) was significantly lower than that for group I, II, or VI, and the value for group IV (Primers A and B) was significantly lower than that for group I, II, V, VI, or VII. Even so, the force might still be clinically acceptable,20,21 since the SBS values obtained with brackets such as Dyna-lock are reportedly lower than those for other brackets.30 Moreover, ceramic brackets seem to produce stronger bonds than do metallic orthodontic brackets.3 Therefore, these conditioners could be indicated for bonding ceramic brackets; however, further studies are warrant to evaluate the results.

A bracket bonded to a tooth surface is temporary; the bracket is removed after the active treatment period. Therefore, greater consideration has been given to
debonding techniques and the amount of adhesive remaining, in addition to the effect of these procedures on the enamel surfaces. Damage to the enamel can be attributed not only to the acid-etching procedure but also to bracket removal and cleaning of the teeth after debonding.\textsuperscript{31} Although we found significant differences in the ARI scores, there were no scores of 3 and only 1 score of 2 in all groups. These findings suggest that the union between Transbond XT and the bracket was stronger than that between the enamel and the adhesive.

The use of phosphoric acid complicates the removal of residual adhesive on the enamel after debonding and can also lead to surface scratches, cracking, and loss of sound enamel.\textsuperscript{13,31-33} Moreover, it has been demonstrated that the amount of adhesive remaining tends to be greater with a high SBS.\textsuperscript{34,35} In this connection, group I showed significantly higher SBS values, and also the amount of residual adhesive was greater than that in the other groups. In contrast, less residual adhesive has been found when SEPs are used.\textsuperscript{4,6,13,15,36-38} This could be clinically advantageous, since tooth cleanup is likely to be easier and faster with less residual adhesive.\textsuperscript{15,16} Consequently, no time loss can be expected with SEPs. Additionally, formation of white spot lesions on enamel or decalcification might be reduced by eliminating the acid-etching procedure.\textsuperscript{31,36}

On the other hand, there was no enamel fracture in this study; however, fracturing might occur when the adhesion force exceeds 14 MPa.\textsuperscript{39} and orthodontists should give extra attention to certain areas of enamel after debonding.\textsuperscript{40} Enamel fractures might be prevented by using special techniques and gentler clinical debonding.\textsuperscript{13,41}

Direct bracket bonding to the etched enamel surface has many advantages but also many disadvantages. The main problems are surface enamel loss and demineralization near the bracket. A strong acid conditioner or a longer etch time can cause surface enamel loss and weakening of the subsurface enamel, leading to enamel surface detachment or fracture during debonding.\textsuperscript{31} It has been extensively reported that the use of SEPs produce a gentler etch pattern than 37% phosphoric acid.\textsuperscript{13-15,27,35,42-44} Although the enamel surface treatment with the conventional acid-etching technique leads to more enamel loss that does the use of SEPs,\textsuperscript{35} the etch pattern observed with 37% phosphoric acid for 15 seconds (Fig 2, A) seems more conservative than the typical honeycomb etched pattern observed when the enamel surface was etched for 30 seconds.\textsuperscript{4,13,42,44} In any case, SEPs seemed to produce a gentler etch pattern than the phosphoric acid. Figure 2, B and D, shows images of enamel surfaces conditioned with Transbond Plus SEP and Primer A and B, respectively, indicating that the action of both conditioners was less aggressive than that of phosphoric acid. The images of the enamel surfaces conditioned with AdheSE, Clearfil Mega Bond FA, Peak SE and Peak LC Bond, and Bond Force (Fig 2, C, E, F, and G) illustrate the appearance of the enamel after the SEPs were light-cured. There was an interesting diversity of
CONCLUSIONS

Under the conditions of this in-vitro study, we found the following.

1. The SBS values of all groups might be clinically acceptable, and orthodontic stainless steel brackets can be successfully bonded with Transbond XT after the enamel has been conditioned with any of the SEPs tested.

2. The SBS of the control group (37% phosphoric acid for 15 seconds) was significantly higher than that of the other groups except group II (Transbond Plus SEP).

3. Although the SBS for group II (Transbond Plus SEP) was slightly higher than that for groups V (Clearfil Mega Bond FA), VI (Peak SE and Peak LC Bond), and VII (Bond Force), there was no significant difference between it and those of the other SEPs. Thus, the latter can be a suitable alternative for bonding orthodontic brackets.

4. The SEPs used in groups III (AdheSE) and IV (Primers A and B) significantly lowered SBSs. Although the values might still be clinically acceptable, further studies are warranted to evaluate their effectiveness for bonding other kinds of stainless steel or ceramic brackets that produce stronger bonds.

5. The use of SEPs produces a gentler etch pattern on the enamel surfaces; however, there was great diversity of etch patterns, and further studies are required to find the gentlest SEP.

6. The application of a SEP reduces the amount of adhesive after debonding, and less invasive procedures might thus be needed to clean up the enamel surface.

7. Understanding the advantages and disadvantages of each SEP can be helpful for obtaining better results when a gentler bonding procedure is used.

We thank Nobukazu Wakamatsu for his valuable help with the Weibull analysis.

REFERENCES


