Bond Strength Comparison and Scanning Electron Microscopic Evaluation of Three Orthodontic Bonding Systems

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This study sought to assess the efficacy of two selfetching primer systems (Transbond Plus and Beauty Ortho Bond) on orthodontic brackets. Therefore, shear bond strengths and bracket-adhesive failure modes (ARI scores) were determined and compared against an etch-and-rinse adhesive system (Transbond XT) under two experimental conditions (dry and saliva application). Shear bond strength test was performed at a crosshead speed of 0.5 mm/min, while enamel surfaces and enamel-adhesive interfaces were examined with SEM. There were no significant differences between Transbond XT (9.15 MPa) and Transbond Plus (9.74 MPa) under the dry condition, whereas that of Beauty Ortho Bond (6.47 MPa) was significantly lower than these two systems. Under SEM examination, both selfetching primers showed a milder etching effect and decreased depth of resin penetration into intact enamel than Transbond XT. In conclusion, results of this study showed that both selfetching systems seemed to offer more merits than conventional acid etching because of fewer irreversible changes to enamel.

Key words: Self-etching primer, Shear bond strength, Orthodontic brackets

INTRODUCTION

At present, the bonding of attachments, such as brackets and tubes, on the enamel surface is a routine clinical procedure, typically utilizing a resin composite adhesive. Since Buonocore6 first described the use of phosphoric acid for creating irregularities in the enamel surface to enhance mechanical locking, the effects of etching time and phosphoric acid concentration have been investigated to seek the most suitable method of enamel preparation.

Although the highest possible bond strength to tooth structure is desirable in restorative dentistry, orthodontic bond strength must satisfy two-pronged requirements: it must be sufficient to retain the brackets but low enough to allow easy clean-up of adhesives when the brackets are removed. It has been suggested that bond strength values approximately in the range of 6.0 and 8.0 MPa are sufficient for a clinically effective orthodontic bonding otherwise, greater bond strength may increase the risk of enamel fracture. Although acid etching of enamel may remove about 10–20 μm of enamel, most clinicians accept acid etching of the enamel surface as a routine technique which poses a risk of iatrogenic enamel damage, such as enamel fracture, surface staining stemming from increased surface porosity, discoloration by resin tags retained in the enamel, and loss of outer enamel surface.

Recently, bonding practices based on selfetching primers, originally introduced in restorative dentistry to bond composite resin restorations to both dentin and enamel, are being used in clinical orthodontics. One obvious advantage of using a selfetching primer is to expedite the bonding procedure by combining etching and priming into a single step. In addition to saving time and reducing procedural errors, their lower etching ability, due to a relatively higher pH as compared with phosphoric acid, might minimize the potential for iatrogenic damage to enamel. In recent studies, the shear bond strength of a composite adhesive combined with selfetching primer was examined. Till then, the only available selfetching primer designed specifically for orthodontic purposes was Transbond Plus. About two years ago, a new selfetching primer system (Beauty Ortho Bond, Shofu, Kyoto, Japan) was developed.

The purpose of this study was to determine the efficacy of two different selfetching primer systems on the shear bond strength of orthodontic brackets. To this end, adhesive remnant index (ARI) scores were also evaluated and all results compared with a conventional etchandrinse adhesive system (Transbond XT).
examined by using scanning electron microscopy (SEM).

MATERIALS AND METHODS

Tooth specimens
Eighty-four healthy human maxillary premolars were used in this study. The teeth had been extracted for orthodontic reasons and with the patients’ informed consent. Seventy-two of them were allocated into six groups of 12 for shear bond strength measurement, and the rest of the maxillary premolars were used for SEM observation. Selection criteria included absence of any visible decalcification or crack of the enamel surface at $\times 10$ magnification, coupled with the availability of a macroscopically smooth, buccal surface suitable for bonding. Extracted teeth were stored in 0.5% chloramine solution at a temperature of approximately 4°C. Teeth with hypoplastic areas, cracks, or gross irregularities of the enamel structure were excluded from the study. The buccal surfaces of all teeth were cleaned using nonfluoridated pumice and water for 10 seconds. The teeth were also polished using a rubber cup, abundantly washed, and dried using an air syringe free of oil and humidity.

Preparation of bracket-bonded specimens
Group 1 (control): The enamel surfaces were treated with 35% phosphoric acid etching gel (Transbond XT Etching Gel, 3M Unitek, Monrovia, CA, USA) for 15 seconds, washed for 20 seconds, and dried with an oil-free air stream. Table 1 lists the materials employed in the present study. Transbond XT primer was applied on the etched surface, and metal brackets for upper premolars (Mini Diamond,Ormco, CA, USA) with a base area of 9.90 mm² were bonded with Transbond XT composite (3M Unitek, Monrovia, CA, USA).

Group 2: Transbond Plus Self-Etching Primer (TPSEP, 3M Unitek, Monrovia, CA, USA) was applied and rubbed on the enamel surfaces for approximately three seconds. An air jet was slightly applied to the enamel surface, and the brackets were bonded with Transbond XT composite.

Group 3: For Beauty Ortho Bond (Shofu, Kyoto, Japan), Primer A and Primer B were mixed. Then, the solution was rubbed on the enamel surfaces for approximately three seconds. An air jet was briefly applied to the enamel surface, and the brackets were bonded with Beauty Ortho Bond Paste (composite).

Each primer-composite combination was tested under two different enamel surface conditions: dry and saliva application (wet). To achieve a saliva-contaminated condition after priming (wet condition), human saliva from one donor was applied with a brush onto the buccal surfaces without air jet. Then, the brackets were bonded with composite paste. Each bonding procedure was performed by the same operator. Excess bonding material was removed with a small scaler. All samples were light-cured for 20 seconds (10 seconds from each proximal side).

Shear bond strength test
After performing the bonding procedures, the specimens were stored in artificial saliva at 37°C for 24 hours. The specimens were fixed with a custom-fabricated acrylic resin block using Model Repair II (Densply-Sankin, Tokyo, Japan), and the block was fixed in a universal testing machine (EZ Test, Shimadzu, Kyoto, Japan). Force was directly applied to the bracket-tooth interface with a knife-edged shearing blade at a crosshead speed of 0.5 mm/min, whereby the force direction was parallel to the buccal surface and bracket base.

Adhesive remnant index evaluation
After debonding, the bracket bases and enamel surfaces were examined under a stereoscopic

Table 1  Materials employed in the present study

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Components</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transbond XT</td>
<td>3M Unitek, Monrovia, CA, USA</td>
<td>Etching gel (6GN)</td>
<td>35% Phosphoric acid</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Primer: (5CL)</td>
<td>TEGDMA, Bis-GMA</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Paste: (6TG)</td>
<td>Bis-GMA, TEGDMA, silane-treated quartz, amorphous silica, camphorquinone</td>
</tr>
<tr>
<td>Transbond Plus self-etching system</td>
<td>3M Unitek, Monrovia, CA, USA</td>
<td>Self-etching primer: (237956E)</td>
<td>Water, methacrylated phosphoric acid esters, amino benzoate, camphorquinone</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Paste: (6TG)</td>
<td>Bis-GMA, TEGDMA, silane-treated quartz, amorphous silica, camphorquinone</td>
</tr>
<tr>
<td>Beauty Ortho Bond self-etching system</td>
<td>Shofu, Kyoto, Japan</td>
<td>Primer A: (030602)</td>
<td>Water, acetone, others</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Primer B: (030602)</td>
<td>Phosphoric acid monomer, ethanol</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Paste: (120503)</td>
<td>TEGDMA, S-PRG filler, Bis-GMA</td>
</tr>
</tbody>
</table>
microscope (SMZ1500, Nikon, Tokyo, Japan) at ×10 magnification. ARI scores were used to assess the amount of adhesive left on the enamel surface\textsuperscript{15,20}. These scores ranged from 1 to 5, where 1=all of the composite, with an impression of the bracket base, remained on the tooth surface; 2=more than 90% of the composite remained on the tooth surface; 3=more than 10% but less than 90% of the composite remained on the tooth surface; 4=less than 10% of composite remained on the tooth surface; 5=no composite remained on the tooth surface.

**Statistical analysis**

Statistical analysis was performed with SPSS (SPSS 14.0J for Windows). Descriptive statistics, including the mean, standard deviation, median, and minimum and maximum values of bond strength were calculated for each of the six groups. Kolmogorov–Smirnov test for normality and Levene’s test for homogeneity of variances were applied to the bond strength data and ARI scores. As the data were not normally distributed, Kruskal–Wallis test was applied to determine whether significant differences existed among the groups. Mann–Whitney test was used for two independent groups, and Bonferroni correction was applied.

**Scanning electron microscopic evaluation**

With a view to assessing the etching efficacy for intact enamel by SEM (SSX-550, Shimadzu, Kyoto, Japan), buccal enamel surfaces were conditioned with two different self-etching primers, Transbond Plus (3M Unitek, Monrovia, CA, USA) and Beauty Ortho Bond (Shofu, Kyoto, Japan), according to manufacturers’ instructions. After which, the primers were rinsed off. For the control, an enamel surface was etched with 35% phosphoric acid (Transbond XT Etching Gel) for 15 seconds and then washed for 20 seconds. After conditioning or etching, the specimens were dehydrated in increasing concentrations of ethanol and water up to 100% ethanol.

**RESULTS**

Descriptive statistics for shear bond strength are shown in Table 2. Statistically significant differences were found among the groups (P>0.0001). When comparing the three groups under the dry condition, Condition 1 (35% phosphoric acid etching + Transbond XT primer/composite) and Condition 2 (Transbond Plus self-etching primer + Transbond XT composite) showed higher average bond strength values (9.15 MPa for Condition 1 and 9.74 MPa for Condition 2) than Condition 3 (6.47 MPa) bonded with Beauty Ortho Bond self-etching system. Table 4 shows that the average bond strength of Condition

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Shear bond strengths of tested groups</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Condition</strong></td>
<td>n</td>
</tr>
<tr>
<td>1. Dry, Transbond XT</td>
<td>12</td>
</tr>
<tr>
<td>2. Dry, Transbond Plus</td>
<td>12</td>
</tr>
<tr>
<td>3. Dry, Beauty Orthobond</td>
<td>12</td>
</tr>
<tr>
<td>4. Wet, Transbond XT</td>
<td>12</td>
</tr>
<tr>
<td>5. Wet, Transbond Plus</td>
<td>12</td>
</tr>
</tbody>
</table>

1 and 4, 35% phosphoric acid etching + Transbond XT primer + Transbond XT composite; 2 and 5, Transbond Plus + Transbond XT; 3 and 6, Beauty Orthobond primer + composite
4 (1.47 MPa) was significantly lower than those for all the other conditions. No statistically significant differences were found between Condition 3 and Condition 6 (7.62 MPa; saliva application) bonded with Beauty Ortho Bond self-etching system, while Condition 5 (7.74 MPa; saliva application) showed lower bond strength compared with Condition 2 \((P=0.052)\), whereby Transbond Plus bonding system was used.

ARI scores for the six test conditions are shown in Table 3. Results obtained from Kruskal–Wallis test indicated statistically significant differences among the groups. Condition 4 showed a higher ARI score (most composite did not remain on the enamel surface) than Condition 1 using the same bonding system (Transbond XT primer+Transbond XT composite). On the other hand, Table 4 shows that there were no statically significant differences between the dry and saliva application (wet) conditions for each self-etching system \(i.e.,\) Condition 2 versus Condition 5, Condition 3 versus Condition 6. In the present study, none of the teeth showed enamel fracture after shear bond strength test.

Figure 1 shows the morphological changes in the enamel surfaces treated with 35% phosphoric acid and two different self-etching primers. The enamel surface etched with 35% phosphoric acid for 15 seconds showed a very porous surface and numerous enamel prisms could be observed (Fig. 1a), forming a typical honeycomb pattern. With Transbond Plus self-etching primer, enamel prisms could be observed in some areas but were less prominent (Fig. 1b). With Beauty Ortho Bond self-etching primer, it produced a surface less rough compared with Transbond Plus self-etching primer; many scratches and fossae were observed (Fig. 1c). In all the specimens, it was apparent that the degree of etching varied.

Figure 2 shows the adhesive interfaces between the adhesive resin and intact enamel after etching or priming and bonding. After etching with 35% phosphoric acid and bonding, many long resin tags were observed with lengths ranging from 2 to 15 \(\mu\m\) and that most tags measured about 7 to 10 \(\mu\m\). For specimens treated with self-etching primers, there was a distinct border between the adhesive resin and enamel. When treated with Transbond Plus self-etching primer, very short tags were observed between the adhesive resin and enamel, but these tags were almost invisible for the specimen treated with Beauty Ortho Bond. Accordingly, the hybrid layers\(^{21}\) of the specimens treated with self-etching

Table 3  Frequency distribution of ARI scores of tested groups

<table>
<thead>
<tr>
<th>Condition</th>
<th>ARI Scores</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Dry, Transbond XT</td>
<td>9, 3, 3, 3, 4, 4</td>
</tr>
<tr>
<td>2. Dry, Transbond Plus</td>
<td>4, 7, 2, 2, 1, 1</td>
</tr>
<tr>
<td>3. Dry, Beauty Orthobond</td>
<td>10, 2, 1, 1</td>
</tr>
<tr>
<td>4. Wet, Transbond XT</td>
<td>5, 4, 3, 3, 1</td>
</tr>
<tr>
<td>5. Wet, Transbond Plus</td>
<td>4, 4, 3, 3, 1</td>
</tr>
<tr>
<td>6. Wet, Beauty Orthobond</td>
<td>9, 3, 3, 3, 1</td>
</tr>
</tbody>
</table>

ARI Scores: 1 indicates all of the composite, with an impression of the bracket base, remained on the tooth; 2, more than 90% of the composite remained; 3, more than 10% but less than 90% of the composite remained on the tooth; 4, less than 10% of composite remained on the tooth surface; 5, no composite remained on the enamel.

Table 4  Statistical comparison of each group with bond strengths in upper diagonal portion and ARI scores in lower portion

<table>
<thead>
<tr>
<th>Condition</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Dry, Etch + XT</td>
<td>ns</td>
<td>ns</td>
<td>**</td>
<td>**</td>
<td>ns</td>
<td>ns</td>
</tr>
<tr>
<td>2. Dry, SEP (3M) + XT</td>
<td>ns</td>
<td>ns</td>
<td>**</td>
<td>**</td>
<td>ns</td>
<td>ns</td>
</tr>
<tr>
<td>3. Dry, SEP (SH) + BB</td>
<td>**</td>
<td>ns</td>
<td>**</td>
<td>**</td>
<td>ns</td>
<td>ns</td>
</tr>
<tr>
<td>4. Wet, Etch + XT</td>
<td>**</td>
<td>**</td>
<td>**</td>
<td>**</td>
<td>ns</td>
<td>ns</td>
</tr>
<tr>
<td>5. Wet, SEP (3M) + XT</td>
<td>ns</td>
<td>ns</td>
<td>ns</td>
<td>**</td>
<td>ns</td>
<td>ns</td>
</tr>
<tr>
<td>6. Wet, SEP (SH) + BB</td>
<td>**</td>
<td>ns</td>
<td>ns</td>
<td>**</td>
<td>ns</td>
<td>ns</td>
</tr>
</tbody>
</table>

*ns, not significant*
primers, consisting of porous enamel surface penetrated by primer, were very thin such that the thickness might be less than 0.5 μm (Figs. 2b and 2c).

Figure 3 shows the adhesive interfaces between the adhesive resin and enamel after shear bond strength test. A representative SEM photomicrograph of Condition 1 (Fig. 3a: Transbond XT primer + Transbond XT composite) showed that there was a thin layer of bonding resin on the enamel surface. On the other hand, a representative SEM photomicrograph of Condition 3 (Fig. 3b: Beauty
Ortho Bond self-etching primer + Beauty Ortho Bond composite) showed that there was no bonding resin on the enamel surface.

**DISCUSSION**

Previous studies\(^{22,23}\) found that individual teeth reacted differently to phosphoric acid and that the etching pattern varied across the enamel surface. In the present study, there was some variation in the region of enamel surface for specimens etched with 35% phosphoric acid and those treated with two self-etching primers. Nonetheless, the apparently much stronger etching effect shown by 35% phosphoric acid, as compared to the other two self-etching systems (Fig. 1).

On the variation of bond strength on ground versus intact enamel, a recent study\(^{15}\) concluded that self-etching primers produced bond strengths equivalent to that produced by phosphoric acid when applied to ground enamel surface. However, the bond strengths of self-etching primers were significantly lower when applied to intact enamel surface. The intact enamel surface is hypermineralized and contains more fluoride than ground enamel. It has been reported that after tooth eruption, changes occur in the outermost enamel layer\(^{24}\). Saturated calcium phosphate in the saliva might hypermineralize the enamel, allowing fluorine ions to convert hydroxyapatite into fluoroapatite\(^{25}\). Further, a thick prismless enamel layer\(^{26}\) may prevent the penetration of self-etching primers and bonding agents, thus leaving some areas partially unetched. In the present study, the three different adhesive systems produced different etching effects on intact enamel. In particular, although the self-etching products employed in the present study were designed specifically for orthodontic purposes, the depth of resin penetration into intact enamel was very shallow due to mild etching effect. Therefore, further research using TEM (transmission electron microscopy) is needed to examine the hybrid layer morphology.

On the effect of acid aggressiveness on etching, a previous study was conducted using three self-etching primers with different pH values (pH 2.0, 1.2, and 1.0). It was found that the etching patterns of aprismatic enamel were dependent on the aggressiveness of the acids, but that there was no correlation between their degree of aggressiveness and the strength of their bonds to intact enamel\(^{16}\).

In the present study, the pH values measured under room temperature with a pH meter (Model 720A, Orion Research, Boston, MA, USA) were 1.39 for 35% phosphoric acid, 1.85 for Transbond Plus, and 2.20 for Beauty Ortho Bond. Both self-etching primers with relatively less acidic pH values had a mild etching effect for intact enamel. On the other hand, 35% phosphoric acid showed the strongest etching effect for intact enamel as expected with the pH value.

Another reason for the stronger etching effect of Condition 1 (35% phosphoric acid) might be due to the longer etching time (15 seconds) than the other two conditions (three seconds). In previous studies, it was found that increase in etching time resulted in a stronger etching effect on ground enamel as well as greater tensile bonding strengths\(^{27,28}\). However, Condition 2 (Transbond Plus self-etching primer + Transbond XT composite) showed an average bond strength equivalent to that of Condition 1 (35% phosphoric acid etching + Transbond XT composite). Condition 3 (Beauty Ortho Bond self-etching system) showed a clinically acceptable bond strength\(^{2,3,19}\), although it was significantly lower than the other two bonding systems under the dry condition. At this
juncture, it is noteworthy that orthodontic brackets and tubes are intended to be bonded to teeth with an adhesive material for a limited time only. Therefore, an appropriate bond strength would serve to ease the debonding procedure and decrease the risk of enamel fracture.

For the specimen etched with 35% phosphoric acid, the resin tags ranged from 2 to 15 μm while most tags measured about 7 to 10 μm — which were slightly shorter than published values. In clinical orthodontics, total enamel loss depends not only on etching method, but also on adhesive removal (debonding) and clean-up methods. Indeed, previous studies found that both etching and non-etching mediated (glass ionomer) bonding protocols resulted in enamel color alteration after orthodontic treatment with fixed appliances. Further on enamel color alteration, it is caused not only by the residues of resin tags in enamel, but also by a host of other factors such as clean-up method (grinding and polishing) at the time of bracket removal, thickness of ground enamel, and type of adhesives resin used. Then, apart from clean-up methods, shorter resin tags also decrease the risk of color alteration. Previous work suggested that enamel loss with a self-etching primer was significantly less than conventional etching with 37% phosphoric acid.

In the present study, Conditions 4, 5, and 6 were contaminated with human saliva from one donor, via application with a brush on the buccal surface, after priming. Condition 4 (Transbond XT primer + Transbond XT composite + saliva contamination) yielded significantly lower bond strength than all the other conditions — and that this bond strength value would not be clinically acceptable. By contrast, the shear bond strengths of self-etching systems used in Conditions 5 and 6 were not adversely affected by human saliva contamination. This finding agreed with a previous study which showed that saliva had no effect on the bond strength of the Transbond self-etching system. However, another study indicated that contamination of enamel with saliva after priming decreased the bond strength although it was still clinically adequate.

With conventional etch-and-rinse adhesive systems, phosphoric acid etches the enamel by selectively dissolving calcium from the enamel structure. In so doing, irregularities are produced in the enamel surface to enhance mechanical locking. Calcium is then washed away when the tooth is rinsed. With self-etching primers, the phosphate group of methacrylated phosphoric acid ester (Transbond Plus) and the phosphoric acid monomer (Beauty Ortho Bond) modify the enamel surface to achieve chemical bonding. Consequently, the efficacy of self-etching primers might be decreased by saliva contamination, although the results of this study showed that saliva contamination had little effect on bond strength. Therefore, it is recommended to reapply the self-etching primer after drying the saliva to provide a good bond to the underlying primer, and hence to minimize the adverse effect of saliva on bond strength.

With Transbond XT, most of its specimens in the wet condition (Condition 4) had ARI score 5 (i.e., no composite remained on the enamel surface). This implied that bond strength at enamel-bonding resin interface might be weak in Condition 4. With Beauty Ortho Bond self-etching system, no specimens had ARI score 1 in both dry and wet conditions (Conditions 3 and 6). Further, there were statistically significant differences between Condition 1 and Condition 3 or 6. SEM photomicrograph of Condition 1 with ARI score 2 after bond strength test showed that there was a thin layer of bonding resin on the enamel surface (Fig. 3a). On the other hand, SEM photomicrograph of Conditions 3 and 6 with ARI score 2 or 3 after bond strength test showed that there was no bonding resin on the enamel surface (Fig. 3b). These results suggested that Beauty Ortho Bond self-etching system decreased the bond strength between enamel and bonding resin, probably due to its mild etching effect. However, the lower bond strength at enamel-bonding resin interface of the Beauty Ortho Bond self-etching system would be an advantage during clean-up of the enamel surface as it reduces the risk of enamel fracture at the time of bracket removal.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions could be drawn:

1. Both self-etching primers showed a milder etching effect than that observed for 35% phosphoric acid.
2. Both self-etching primers produced shallower depth of resin penetration into intact enamel as compared to using 35% phosphoric acid as an etchant.
3. Saliva contamination of enamel after priming had little adverse effect on bond strength when self-etching primers were used.

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