Effect of adhesive resin flexibility on enamel fracture during metal bracket debonding: an ex vivo study

Young Kyung Kim*, Hyo-Sang Park**, Kyo-Han Kim*** and Tae-Yub Kwon***

Departments of *Conservative Dentistry, **Orthodontics, and ***Dental Biomaterials, School of Dentistry, Kyungpook National University, Daegu, Korea

Correspondence to: Tae-Yub Kwon, Department of Dental Biomaterials, School of Dentistry, Kyungpook National University, 2-188-1 Samduk-dong, Jung-gu, Daegu 700-412, Korea. E-mail: tykwon@knu.ac.kr

Summary

Objective: To test the null hypothesis that neither the flexural properties of orthodontic adhesive resins nor the enamel pre-treatment methods would affect metal bracket debonding behaviours, including enamel fracture.

Materials and methods: A dimethacrylate-based resin (Transbond XT, TX) and two methyl methacrylate (MMA)-based resins (Super-Bond C&B, SB; an experimental light-cured resin, EXP) were tested. Flexural strength and flexural modulus for each resin were measured by a three-point-bending test. Metal brackets were bonded to human enamel pretreated with total-etch (TE) or self-etch adhesive using one of the three resins (a total of six groups, \(n=15\)). After 24 hours of storage in water at 37°C, a shear bond strength (SBS) test was performed using the wire loop method. After debonding, remaining resin on the enamel surfaces and occurrence of enamel fracture were assessed. Statistical significance was set at \(P<0.05\).

Results: The two MMA resins exhibited substantially lower flexural strength and modulus values than the TX resin. The mean SBS values of all groups (10.15–11.09 MPa) were statistically equivalent to one another (\(P>0.05\)), except for the TE-TX group (13.51 MPa, \(P<0.05\)). The two EXP groups showed less resin remnant. Only in the two TX groups were enamel fractures observed (three cases for each group).

Limitations: The results were drawn only from ex vivo experiments.

Conclusions: The hypothesis is rejected. This study suggests that a more flexible MMA resin is favourable for avoiding enamel fracture during metal bracket debonding.

Introduction

In orthodontic bracket bonding, the bond strength should be sufficient to withstand the forces of mastication and stresses exerted by the archwires (1). For orthodontists, however, maintaining a sound enamel surface after debonding orthodontic brackets is also a primary concern (2). Retief (3) demonstrated enamel fractures on specimens with bond strengths as low as 9.7 MPa.

Bond failure at the bracket–adhesive interface or within the adhesive is more desirable than failure at the adhesive–enamel interface in terms of conservation of enamel structure (1, 2). Self-etching primers (SEPs) may produce a more conservative etching pattern than phosphoric acid etchant, consequently minimizing enamel loss (4). Bishara et al. (2) reported that a SEP used for metal bracket bonding resulted in more residual adhesive on the enamel surfaces than a conventional adhesive system, while maintaining clinically acceptable shear bond strength (SBS). In a randomized controlled trial (5), there was no difference between the clinical bond failure rates of brackets bonded with a SEP and with a conventional adhesive system.

In terms of bond strength, the use of SEPs with methyl methacrylate (MMA)-based resins has also been found to be effective.
for bracket bonding (6, 7). Adhesive monomer contained in SEP can improve the wettability and penetration ability of MMA resin (7). However, relatively little effort has been paid to the merit of flexibility of MMA resins used for orthodontic bracket bonding (8). Unfilled MMA-based resins, such as Super-Bond C&B (Sun Medical Co., Ltd, Shiga, Japan), are considerably elastic and flexible compared to filled dimethacrylate-based resins (composite resins) such as Transbond XT (3M Unitek, Monrovia, California, USA) owing to their linear structure after polymerization (9, 10). When debonding force is applied to a bracket, various stresses may be generated within the adhesive resin layer under the bracket (8, 11). It can be assumed that more flexible MMA resin reduces the stress delivered to the enamel surface during debonding and, as a result, the possibility of enamel damage such as fracture (8, 12).

The present ex vivo study investigated the relationship between the flexural properties of unfilled MMA resin and filled dimethacrylate resin and the debonding behaviour of metal brackets bonded to the enamel surface using the resins. For comparison, an experimental light-cured MMA resin was also designed. The resins were applied to enamel surfaces etched with phosphoric acid [total etch (TE)] or SEP [self-etch (SE)]. The null hypothesis tested was that neither the flexural properties of the resins nor the enamel pre-treatment (etching) methods would affect the bracket debonding behaviours including enamel fracture.

### Materials and methods

#### Bracket bonding systems

For orthodontic adhesive resins, two commercial resins (Transbond XT, TX; Super-Bond C&B, SB) were used. In addition, one experimental light-cured MMA resin (EXP) was prepared and tested (Table 1). Unitek Etching Liquid/Transbond XT Primer (TXP), Red Activator, or Transbond Plus SEP (TPSEP) was used for the enamel pre-treatment prior to the application of the resins. Their manufacturers, compositions, and lot numbers are summarized in Table 1.

#### Flexural test

To compare the flexural properties of the orthodontic adhesive resins, 10 sticks (2.5 × 2 × 2 mm) were made for each resin using a stainless steel mold, which was placed on a polyester film over a glass slide (13). TX, SB, and EXP were filled into the mold (for the latter two, using the brush-dip method) (8), covered with another polyester film and glass slide, gently pressed to expel the excess material, and either light-cured (TX and EXP) or self-cured (SB). The light-curing was performed from the centre towards the edge in five overlapping sections on both the top and bottom surfaces. The SB specimens were transferred into a 37°C water bath for 1 hour before removal from the mold. All the specimens were stored in water for 24 hours at 37°C before testing.

A three-point-bending test was performed using a universal testing machine (4200, Instron Inc., Canton, Massachusetts, USA) with a crosshead speed of 1.0 mm/minute. Dimensions of the specimens were measured with a digital caliper. The flexural strength (σ) in MPa

---

**Table 1.** Phosphoric acid etchants, primers, resins used.

<table>
<thead>
<tr>
<th>Type</th>
<th>Material (code)</th>
<th>Manufacturer</th>
<th>Composition (manufacturer supplied)</th>
<th>Lot number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phosphoric acid etchant</td>
<td>Unitek Etching Liquid</td>
<td>3M Unitek, Monrovia, California, USA</td>
<td>37 wt% phosphoric acid</td>
<td>9BU</td>
</tr>
<tr>
<td></td>
<td>Red Activator</td>
<td>Sun Medical Co., Ltd, Shiga, Japan</td>
<td>65 wt% phosphoric acid</td>
<td>VG3</td>
</tr>
<tr>
<td>Primer</td>
<td>Transbond XT Primer (TXP)</td>
<td>3M Unitek, Monrovia, California, USA</td>
<td>Bisphenol A diglycidyl methacrylate, triethylene glycol dimethacrylate, camphorquinone, 2-[4-(dimethylamino)phenyl]ethanol, hydroquinone</td>
<td>N185882</td>
</tr>
<tr>
<td></td>
<td>Transbond Plus Self-Etching Primer (TPSEP)</td>
<td>3M Unitek, Monrovia, California, USA</td>
<td>Bis(2-methacryloxy)ethyl phosphate, ethylene glycol methacrylate phosphate, 2-(methacryloyloxy)ethyl phosphate, water, camphorquinone, ethyl 4-dimethylaminobenzoate, potassium hexafluorotitanate</td>
<td>517094A</td>
</tr>
<tr>
<td>Adhesive resin</td>
<td>Transbond XT (TX)</td>
<td>3M Unitek, Monrovia, California, USA</td>
<td>Bisphenol A diglycidyl methacrylate, bisphenol A ethoxylate dimethacrylate, quartz, silica, diphenylidoxynium hexafluorophosphate</td>
<td>N356322</td>
</tr>
<tr>
<td></td>
<td>Super-Bond C&amp;B (SB)</td>
<td>Sun Medical Co., Ltd, Shiga, Japan</td>
<td>Polymer powder (clear): polymethyl methacrylate, Monomer liquid: methyl methacrylate, 4-acryloyloxyethyl trimel-litate anhydride, Catalyst V: tri-n-butylborane, acetone, ES1</td>
<td>FL11, FX1, ES1</td>
</tr>
<tr>
<td></td>
<td>Experimental light-cured MMA resin (EXP)</td>
<td>Sun Medical Co., Ltd, Shiga, Japan</td>
<td>Monomer liquid: methyl methacrylate 86 wt%, ethylene glycol dimethacrylate 10 wt%, camphorquinone 1 wt%, 2-(dimethylamino)ethyl methacrylate 3 wt%</td>
<td>2012J4042, 05604BJ, 619-25-01, BCB-J3899V, respectively</td>
</tr>
</tbody>
</table>
was calculated using the formula (13): \( \sigma = \frac{3FL}{2bd^2} \), where \( F \) is the load at fracture (N), \( L \) is the support span (20 mm), \( b \) is the width (mm), and \( d \) is the depth (mm) of the specimen. The flexural modulus (\( E \)) in GPa was calculated using the formula (13): \( E = \frac{\text{modulus of elasticity (GPa)}}{\text{flexural modulus (GPa)}} \times 10^3 \), where \( F/d \) is the slope of the force–displacement curve (N/mm). For the SB resin, the loads at the yield point were used to calculate the flexural strengths because the specimens were too elastic to fracture (Figure 1) (9).

Bonding specimen preparation

Non-caries human premolars, extracted for orthodontic treatment, were collected after obtaining the patients’ informed consent under a protocol approved by the Ethics Committee. The teeth were disinfected in 0.5 per cent chloramines, stored in distilled water, and used within 6 months after extraction (14). Ninety teeth without enamel damage or craze lines were selected by careful examination under a stereomicroscope (SZ61; Olympus, Tokyo, Japan) at ×20 magnification (15). Each tooth was mounted in an acrylic block and the buccal crown surface then rinsed and dried after polishing for 15 seconds with fluoride-free pumice.

The tooth specimens were randomly divided into six groups. In group 1 (TE-TX), the enamel surface was etched with Unitek Etching Liquid (37 per cent phosphoric acid) for 15 seconds, rinsed, and air-dried. TXP was applied to the acid-etched enamel surface in a thin film. Upper premolar stainless steel brackets (Gemini 0.022 inch slot Twin; 3M Unitek) were then bonded to the acid-etched/primed enamel surface using TX adhesive paste. The average surface of the orthodontic bracket base was 12.4 mm². The excess material was removed from around the bracket with a scaler, and light-curing was performed from the mesial and distal aspects for 5 seconds each using a light-emitting diode curing unit (bluephase G2; Ivoclar Vivadent AG, Schaan, Liechtenstein) with an output intensity of 1050 mW/cm² (high power mode) as measured with a radiometer. In group 2 (SE-TX), the enamel surface was treated with TPSEP for 5 seconds, and the brackets were then bonded to the enamel surface using TX. In groups 3 and 4 (TE-SB and SE-SB), the enamel surface was treated with Red Activator (65 per cent phosphoric acid, for 30 seconds) and TPSEP, respectively. The brackets were then bonded to the enamel surface by applying SB to them using the brush-dip technique as per the manufacturer’s instruction (8). The SB specimens were left undisturbed for 30 minutes in air at room temperature (16). In groups 5 and 6 (TE-EXP and SE-EXP), the enamel surface was treated with Red Activator and TPSEP, respectively. The brackets were then bonded to the enamel surface with EXP, also applied using the brush-dip technique. Light-curing was performed from the mesial and distal aspects for 40 seconds each using the curing light. All the bracket-bonded specimens were then stored in water at 37°C (8).

The enamel surfaces treated with the three different etchants were observed under a field emission-scanning electron microscope (FE-SEM, JSM-6700F; Jeol, Tokyo, Japan). A 0.018 × 0.025-inch steel wire was engaged under tie wings. The shear load (pull of the steel wire) was applied at a crosshead speed of 1.0 mm/minute until failure (18). The results were finally calculated in MPa.

Bond failure assessment

Once the brackets had been debonded, the enamel surface of each tooth was examined under the SZ61 stereomicroscope at ×20 magnification to determine the amount of residual adhesive resin on each tooth. The adhesive remnant index (ARI) scores were recorded as originally described, on the following scale: 0 = no resin remaining; 1 = less than 50 per cent of resin remaining; 2 = more than 50 per cent of resin remaining; and 3 = all resin remaining, with a distinct impression of the bracket base. In addition, the number of teeth with enamel fracture was counted (20). When necessary, enamel surfaces after bracket debonding were observed under SEM.

Statistical analysis

The Shapiro–Wilks normality test and Levene’s variance homogeneity test were applied to the SBS and flexural test data. The former, having met both the normality and variance homogeneity assumptions, was analysed using one-way analysis of variance and the Tukey post hoc test. Both the flexural strength and modulus values were log\(_{10}\) transformed to meet homogeneity of variance prior to analysis. The ARI scores were analysed using Fisher’s exact test. The statistical analyses were carried out using SPSS 17.0 for Windows (SPSS Inc., Chicago, Illinois, USA) at a significance level of 0.05. For SBS values, a post hoc power analysis was carried out to examine the power of the adhesion data using G*Power 3 software. A power of 0.80 was regarded as acceptable (21).

Results

Figure 1 and Table 2 show the flexural test results. The TX resin exhibited the highest flexural strength (130.7 ± 9.7 MPa) among the three resins, the differences being significant (\( P < 0.001 \)). The resin also showed a significantly higher flexural modulus (11.4 ± 1.3 GPa) than the other two resins (\( P < 0.001 \)). The SB resin showed a significantly higher flexural strength than the EXP resin (\( P < 0.001 \)), whereas the EXP resin showed a significantly higher flexural modulus than the SB resin (\( P < 0.001 \)).
Figure 2 shows the SEM images of the enamel surfaces treated with the three etchants, showing that a higher concentration of phosphoric acid did not necessarily result in aggressive enamel etching and that TPSEP produced a less aggressive etching pattern than the phosphoric acid etchants.

The SBS value, ARI scores, and number of teeth that showed enamel fracture for each group are summarized in Table 3. According to the post hoc power analysis, the power values for the SBS were higher than 0.80 (0.99). The Tukey post hoc comparisons revealed that group 1 achieved the highest bond strength (13.51 ± 1.68 MPa, P < 0.05), while the bond strengths for the other five groups ranged between 10.15 ± 1.13 and 11.09 ± 2.08 MPa, with no significant differences among the groups (P > 0.05). Fisher’s exact test indicated significant differences in ARI scores among the groups (P < 0.001). Groups 1 and 2 showed predominantly the ARI scores of 1 and 2, respectively. For groups 3 and 4, the predominant score was 2, whereas 1 was the predominant score for groups 5 and 6. Enamel fractures were observed only in groups 1 and 2 (three cases for each group) (Figure 3A and 3B). The SEM observations also clearly showed enamel damage in the form of cracks or tear-outs near the impression of the bracket base (Figure 3C).

Discussion

In the present ex vivo study, although no direct comparison between the image of each tooth specimen before bracket bonding and that after debonding was made (15), teeth without any visible enamel damage were carefully selected and used. The bond strength test results showed no significant differences in SBS among the groups tested, except for group 1 (Table 3). All the SBS values obtained for the three adhesive resins used with the phosphoric acid etchants or TPSEP were above the minimum values suggested to achieve clinically effective adhesion in orthodontics (1, 20, 22). However, groups 1 and 2 were accompanied by enamel fracture (Figure 3). Groups 5 and 6 showed less resin remnant on the enamel surface after debonding than groups 3 and 4, all without enamel fractures. These results may be partly attributed to significant differences in flexural properties among the three resins (Figure 1 and Table 2). The enamel pretreatment methods also affected the ARI scores. Therefore, the null hypothesis tested was rejected.

The SB resin has a limited working time, particularly when the powder and activated liquid are bulk-mixed. Thus, an experimental light-cured MMA resin was designed to make ‘command setting’ possible. The photopolymerization of MMA is known to be weak

Table 2. Flexural strength and modulus of the three resins studied (n = 10).

<table>
<thead>
<tr>
<th>Resin</th>
<th>Flexural strength* (MPa)**</th>
<th>Flexural modulus* (GPa)**</th>
</tr>
</thead>
<tbody>
<tr>
<td>TX (Transbond XT)</td>
<td>130.7 (9.7)A</td>
<td>11.4 (1.3)A</td>
</tr>
<tr>
<td>SB (Super-Bond C&amp;B)</td>
<td>62.3 (6.4)B</td>
<td>1.7 (0.1)B</td>
</tr>
<tr>
<td>EXP (Experimental light-</td>
<td>40.1 (3.0)C</td>
<td>2.2 (0.2)C</td>
</tr>
<tr>
<td>cured MMA resin)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*For each parameter, values with the same capital superscript letter indicate no statistically significant difference based on the Tukey post hoc test at α = 0.05. Means were log transformed prior to analysis.

**Mean values (standard deviation).

Figure 2. Scanning electron microscope images of enamel surfaces treated with Unitek Etching Liquid (37 per cent phosphoric acid) (A), Red Activator (65 per cent phosphoric acid) (B), and Transbond Plus Self-Etching Primer (C) (magnification x1500, bar = 10 μm).

Table 3. Shear bond strength (SBS), adhesive remnant index (ARI) scores, and enamel fracture (EF) for each group (n = 15).

<table>
<thead>
<tr>
<th>Group</th>
<th>SBS in MPa***</th>
<th>ARI scores*</th>
<th>Median</th>
<th>EF**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group 1 (TE-TX)</td>
<td>13.51 (1.68)A</td>
<td>0</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>Group 2 (SE-TX)</td>
<td>10.96 (1.57)A</td>
<td>0</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>Group 3 (TE-SB)</td>
<td>10.78 (1.45)A</td>
<td>2</td>
<td>4</td>
<td>2</td>
</tr>
<tr>
<td>Group 4 (SE-SB)</td>
<td>11.09 (2.08)A</td>
<td>3</td>
<td>6</td>
<td>2</td>
</tr>
<tr>
<td>Group 5 (TE-EXP)</td>
<td>10.15 (1.13)A</td>
<td>0</td>
<td>11</td>
<td>1</td>
</tr>
<tr>
<td>Group 6 (SE-EXP)</td>
<td>10.57 (1.78)A</td>
<td>5</td>
<td>7</td>
<td>1</td>
</tr>
</tbody>
</table>

*0 = no resin remaining; 1 = less than 50% of resin remaining; 2 = more than 50% of resin remaining; and 3 = all resin remaining, with a distinct impression of the bracket base.

**The number of teeth.

***Mean values (standard deviation). For each parameter, values with the same capital superscript letter indicate no statistically significant difference based on the Tukey post hoc test at α = 0.05.
because oxygen diffuses more easily into MMA due to its low viscosity and the radicals have higher affinity to react with oxygen than with monomers (23). The formulations of the light-cured MMA resin (Table 1) were determined by several preliminary bonding tests, which were repeated until acceptable SBS values were obtained (24). Using the high-intensity curing light, nevertheless, a total curing time of 80 seconds was required for one metal bracket to bond to the enamel surface with an acceptable SBS. As shown in Table 1, TPSEP contains various acidic monomers. Thus, a Lewis acid–base reaction may have occurred between the acidic monomer in TPSEP left uncured and the tertiary amine in the overlying EXP resin, resulting in poor polymerization at the interface and poor bond strength of the resin (25, 26). However, the SBS value for group 6 (SE-EXP) indicates that such an adverse chemical interaction did not occur extensively due to the fast photopolymerization rate of the light-cured MMA resin under the metal bracket (25).

The filled dimethacrylate-based resin TX was substantially more rigid, strong, and brittle than the other two MMA-based resins (Figure 1). Thus, it can be assumed that most of the stress developed within the resin during bracket shear debonding was delivered directly to the enamel surface, thereby causing damage such as enamel fracture (Figure 3) (8, 12), notwithstanding the difference in the ARI scores.

On the contrary, the two MMA resins (groups 3–6) did not produce such enamel fractures (Table 3), probably due to their substantially flexible and weak characteristics (12). Of the MMA resins, EXP showed slightly different mechanical properties from SB (Figure 1 and Table 2). The incorporation of 10 per cent ethylene glycol dimethacrylate, a common crosslinking agent in dental MMA resins (Table 1) (9), made the EXP resin slightly brittle. This may have allowed less resin remnant during debonding (12), as seen in the lower ARI scores in groups 5 and 6 relative to groups 3 and 4. Moreover, the ARI scores were lower in group 6 than in group 5. Although bond failure at the enamel–adhesive interface may cause enamel damage (2), less resin remnant on the enamel surface may be advantageous to the clinician because it will reduce cleaning time after debonding (22). In addition, post-debonding cleanup procedures may cause enamel loss and change in enamel texture (27, 28). Thus, the EXP resin, particularly when used with a SEP, shows outstanding debonding behaviour in this study.

Since various materials and methods are used in bond strength studies, direct comparisons of different data are difficult and often impossible (29). Moreover, clinical conditions may significantly differ from an ex vivo setting (30). Thus, the findings of this ex vivo study should be interpreted carefully. Although ceramic brackets may cause enamel fractures and fractures more easily than metal brackets (15, 29, 31), only metal ones were used in the present SBS study. The wire loop method was used for bracket debonding based on its similarity to clinical loads (18, 19). However, different techniques may produce different bracket debonding behaviours.

An ongoing challenge in orthodontics is the development of a bracket–enamel bond strong enough to accomplish treatment but which can be broken for debonding without damage to the enamel surface (31). Although the EXP resin requires a long light-curing time (total 80 seconds), a SBS value higher than that suggested for routine clinical treatment and favourable debonding behaviour indicates the resin’s potential as an adhesive resin for bracket bonding. The EXP resin may cure better under a translucent ceramic bracket, thus potentially reducing the total light-curing time and the possibility of enamel damage when used with such a bracket. Nonetheless, the formulation of light-cured MMA resin for improved light-curing efficacy needs further study. Although the present study did not assess the degree of cure, monomer leaching, or cytotoxicity of the EXP resin, these also clearly require further investigation (32).

Conclusions
Within the limitations of the present investigation, the following conclusion was established.

When considering the occurrence of enamel fracture during bracket debonding, flexible adhesive resins may be preferable to rigid ones, as long as a clinically acceptable bond strength is guaranteed. In addition, the light-cured MMA (EXP) resin has potential as a new adhesive resin for bracket bonding.

Funding
Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education (2013R1A1A2012382).

References


