Comparison of enamel-bracket bond strength using direct- and indirect-bonding techniques with a self-etching ion releasing S-PRG filler

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This in vitro study compared the shear bond strength (SBS) and adhesive remnant index (ARI) of two systems for bonding orthodontic brackets to enamel. The first system involved a self-etching primer (Beauty Ortho Bond, BO) containing surface-pre-reacted glass filler. The second involved a primer applied with phosphoric acid etching (Transbond XT, TX). Ninety-six extracted human premolars were divided into eight groups: Group I (TX/direct bonding), Group II (TX/indirect bonding), Group III (BO/direct bonding), and Group IV (BO/indirect bonding). Groups V–VIII were identical to Groups I–IV, respectively, but were also subjected to 1,500 thermal cycles between 5 and 55°C. ARI was scored by binocular microscopy. SBS was analyzed by three-way ANOVA and the Bonferroni test. ARI was analyzed by the chi-squared test. The BO groups showed lower SBS and ARI results than the TX groups. SBS was significantly influenced by the primer material, bonding technique, and thermal cycling.

Keywords: Shear bond strength, Self-etching, Direct bonding, Indirect bonding, Enamel loss

INTRODUCTION

The acid-etching bonding technique, introduced by Buonocore1 in 1955, has revolutionized the bonding of orthodontic brackets2,3. Newman described the first use of acid-etching in bonding orthodontic brackets with an epoxy-derived resin4,5. In 1972, Silverman and Cohen5 introduced the first indirect bonding process, in which the brackets before bonding were positioned on a cast model of the patient, and then successively transferred to the patient’s mouth via customized trays. Development of this technique reduced the chair time and operator stress, increased the precision and accuracy of bracket placement, reduced the need for re-bonding, and, consequently, reduced enamel loss6,7. Enamel loss due to demineralization around the brackets can also result from plaque accumulation, independent of the bonding technique8.

When a direct technique of bracket bonding is compared with the indirect bonding technique, it is shown that the bonding protocol of indirect bonding is less technically sensible, and the bonding is performed in a one-step procedure, in other hand, the direct technique requires more skills and experience, also the position of the bracket it is not always accurate6,7,9, when a self-etch adhesive is used with the indirect bracket bonding technique, this method could be more simplified and bonding performance would be comparable with the acid-etching technique10.

Over the years, several studies have compared direct and indirect bonding in vitro7–9 and in vivo11,12. Most of these have compared light-cured and chemically-cured sealants7,8,10. These sealant materials require three different agents: an enamel conditioner, a primer solution, and an adhesive resin. Although most clinicians accept 37% phosphoric acid as the standard enamel conditioner, some authors argue that acid etching produces an iatrogenic effect that results in enamel surface loss, estimated at between 10 and 30 μm13–15. Furthermore, the resin tag can penetrate up to 50 μm into the enamel. Cleanup of the adhesive after bracket removal produces a total enamel loss of between 50 and 55.6 μm13–17.

To reduce enamel loss and simplify bonding, self-etching primers have been introduced in orthodontics18. A new self-etching primer, which contains surface pre-reacted glass-ionomer (S-PRG) filler, has been shown to inhibit demineralization. The S-PRG filler has been shown to inhibit demineralization by releasing of Al, Si, and Sr19,20. Although several studies have compared direct and indirect bonding in vitro and in vivo, there has not been comparison between the two bonding methods when the brackets were cemented by means of a self-etching primer.

The aim of this in vitro study was to evaluate the shear bond strength (SBS) of two bonding methods with a direct and indirect bonding a self-etching primer containing S-PRG filler, and compared SBS the same methods with phosphoric acid etching. This study evaluated how thermal cycling influences the SBS of the two systems and assessed the adhesive remnant index (ARI)21.

MATERIALS AND METHODS

Specimen preparation

Ninety-six human premolars extracted for orthodontic reasons were collected and stored in distilled water at
37°C. This *in vitro* study received approval from an ethics committee at the Universitat Internacional de Catalunya (Sant Cugat, Catalunya, Spain). All teeth were cleaned and polished with pumice using rubber cups and fluoride-free paste (Detartrine, Septodont, Saint-Maur-Des-Fossés, France) applied with a low-speed handpiece (10 s). Teeth were washed with water (30 s) and dried with an oil-free air source before bonding. All samples were mounted with a custom-made jig (Fig. 1) to standardize the position of the teeth, with the buccal surface of the teeth parallel to the direction of the force during the SBS test. Teeth were attached with wax to the jig and placed in a container filled with cold-cured acrylic.

For indirect bonding, samples were mounted in blocks, each holding four teeth. Samples for direct bonding were mounted individually (Fig. 1). A working model was manufactured for the samples used for indirect bonding. A silicone impression (Hydorise Putty Fast; Zhermack, Marl, Germany) of the mounted teeth was made, and hard stone (Elite Model, Zhermack) was poured into the impression. The working model was set overnight, covered with a layer of separating medium (Prothyl Isolator, Zhermack), and left to dry for 20 min.

**Materials used in this study** are listed in Table 1. Teeth were randomly divided into eight groups (*n* = 12), according to the adhesive system and bonding method used. Premolar stainless steel brackets with a 0.022-inch slot (Victory series; 3M/Unitek Corp., Monrovia, CA, USA) were used. The average bracket base area was 9.75 mm². The bonding procedure was performed according to the manufacturer’s instructions, as described below.

**Experimental groups**

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![Fig. 1](image)

**Fig. 1** Representative images of the custom-made jig for standardize the position of the teeth.

(a) Direct bonding; (b) Indirect bonding views. Representatives images of a bonded tooth set in an acrylic block and positioned in the testing machine. (c) Direct bonding; (d) Indirect bonding views.

**Table 1 Composition and handling procedure of the adhesive systems tested**

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Components (Lot No.)</th>
<th>pH</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transbond XT</td>
<td>3M/Unitek, Monrovia, California, USA</td>
<td>Etching gel: (DF5JW9) primer: (DF5JW); paste: (DF4JW)</td>
<td>1.39</td>
<td>35% phosphoric acid, tetraethyleneglycol dimethacrylate (TEGDMA), bisphenol-A-diglycidel methacrylate (Bis-GMA); Bis-GMA, TEGDMA, silane-treatedquartz, amorphoussilica, camphorquinone</td>
</tr>
<tr>
<td>Beauty Ortho Bond</td>
<td>Shofu, Kyoto, Japan</td>
<td>Primer A: (1010); primer B: (1010); paste: (1010)</td>
<td>2.20</td>
<td>Water, acetone, others, phosphoric acid monomer, ethanol, TEGDMA, surface pre-reacted glass-ionomer, filler, Bis-GMA, camphorquinone</td>
</tr>
</tbody>
</table>
Table 2 Descriptive and comparative statistics of shear bond strength for two adhesive systems

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean (MPa)</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>14.99</td>
<td>2.64</td>
</tr>
<tr>
<td>II</td>
<td>13.40</td>
<td>2.69</td>
</tr>
<tr>
<td>III</td>
<td>11.38</td>
<td>3.66</td>
</tr>
<tr>
<td>IV</td>
<td>9.06</td>
<td>3.30</td>
</tr>
<tr>
<td>V</td>
<td>14.54</td>
<td>2.67</td>
</tr>
<tr>
<td>VI</td>
<td>13.20</td>
<td>2.01</td>
</tr>
<tr>
<td>VII</td>
<td>7.23</td>
<td>2.85</td>
</tr>
<tr>
<td>VIII</td>
<td>6.46</td>
<td>2.40</td>
</tr>
</tbody>
</table>

1. Group I: TX (Transbond XT, 3M/Unitek Co., California, USA)/direct bonding
Teeth were etched with acid (35% H₃PO₄) for 30 s, washed for 20 s with an air-water spray, and dried to a chalky white appearance. The TX primer was applied to the etched surface. Brackets were bonded with TX and light-cured by LED curing light (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) for a total of 30 s, divided into three, 10-s intervals on the mesial, distal, and occlusal sides, respectively.

2. Group II: TX/indirect bonding
Brackets were bonded on the working model with TX and light-cured by the same protocol as for Group I. Transfer trays were made from clear addition silicone which can be light-cured through (Elite Glass; Zhermack, Marl, Germany), and had a working time of about 40 s. After the transfer trays had set, they were soaked in warm water for 30 min and removed from the models. Composite adhesive on the custom bracket base was cleaned by sandblasting (Dento-prep; Ronvig, Daugaard, Denmark) for 3 s and 2–10 mm distance, aluminium oxide 50 μm and 2 bar were used to remove stone and separating medium. Enamel was prepared by the same direct-bonding procedure as for Group I. The transfer tray was placed over the mounted teeth. The adhesive was light-cured by the same protocol as for Group I.

3. Group III: BO (Beauty Ortho Bond, Shofu, Kyoto, Japan)/direct bonding
Teeth were conditioned with self-etching primer after mixing the two components (Primer A and Primer B). The brackets were bonded with BO and light-cured by the same protocol as for Group I.

4. Group IV: BO/indirect bonding
Brackets were bonded on the working model with BO and light-cured by the same protocol as for Group I. Transfer trays were made by the same process as for Group II. Enamel was prepared with the same direct-bonding procedure as for Group III. The transfer tray was placed over the mounted teeth, and the adhesive was light-cured by the protocol as for Group I.

5. Groups V, VI, VII and VIII
These groups were identical to Groups I, II, III, and IV, respectively, but were also subjected to thermal cycling according to the ISO11405 recommendation. Each specimen underwent 1,500 complete cycles between 5 and 55°C in distilled water, with a dwell time of 1 min.

Debonding procedure
All teeth were stored in distilled water at 37°C for 24 h. The SBS was tested on a universal testing machine (Quasar 5; Galdabini, Cardano al Campo, Italy) at a crosshead speed of 0.1 mm/min. The buccal surface of each tooth was placed parallel to the direction of the force of the machine (Fig. 1). An occlusogingival load was applied to each bracket, producing a shear force at the bracket-tooth interface. This step was accomplished by using the flattened end of a steel rod, which was attached to the crosshead of the testing machine. The SBS was calculated by dividing the load by the base area of the bracket (9.75 mm²).

Adhesive remnant index evaluation
The ARI on the enamel surface was examined under a SZ40-PT binocular microscope (Olympus; Tokyo, Japan) using 4× magnification. The ARI of each sample was scored according to the method of Årtun. The ARI score measures bond failure by assessing the amount of adhesive left on the tooth as follows: 0, no adhesive remaining; 1, less than half of the adhesive remaining; 2, more than half of the adhesive remaining; 3, all adhesive remaining.

Statistical analysis
Three-way analysis of variance (ANOVA) was used to determine the significant differences among the samples consisting of different materials and subjected to various bonding methods, thermal cycling, and combinations thereof. The Bonferroni test was used to analyze multiple comparisons. Chi-squared analysis was used to test the significance of differences in the distributions of the ARI scores. Results were analyzed with a statistical software program (Statgraphics; Warrenton, VA, USA). The level of statistical significance was set at p<0.05.

RESULTS

Shear bond strength
Table 2 presents the descriptive statistics of SBS. The highest mean SBS value (14.99±2.64 MPa) was observed for Group I, and the lowest mean value (6.46±2.14 MPa) was observed for Group VIII. Three-way ANOVA (Table 3) showed that the SBS was significantly influenced by the materials, bonding techniques, and exposure to thermal cycling (p<0.05). The three-way interaction between materials and thermal cycling showed that the...
Table 3  Three-way ANOVA test of factors influencing SBS (*p<0.05): materials (MAT), bonding techniques (BT) and thermal cycling (T)

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>Freedom of Motion</th>
<th>Mean Square</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAT</td>
<td>725.72</td>
<td>1</td>
<td>725.72</td>
<td>93.45</td>
<td>0.00*</td>
</tr>
<tr>
<td>BT</td>
<td>54.13</td>
<td>1</td>
<td>54.13</td>
<td>6.97</td>
<td>0.00*</td>
</tr>
<tr>
<td>T</td>
<td>82.83</td>
<td>1</td>
<td>82.83</td>
<td>10.6</td>
<td>0.00*</td>
</tr>
<tr>
<td>MAT × BT</td>
<td>0.03</td>
<td>1</td>
<td>0.03</td>
<td>0.00</td>
<td>0.94</td>
</tr>
<tr>
<td>MAT × T</td>
<td>55.95</td>
<td>1</td>
<td>55.95</td>
<td>7.2</td>
<td>0.00*</td>
</tr>
<tr>
<td>BT × T</td>
<td>4.88</td>
<td>1</td>
<td>4.88</td>
<td>0.63</td>
<td>0.42</td>
</tr>
<tr>
<td>MAT × BT × T</td>
<td>2.57</td>
<td>1</td>
<td>2.57</td>
<td>0.33</td>
<td>0.56</td>
</tr>
<tr>
<td>Error</td>
<td>691.16</td>
<td>89</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Total</td>
<td>1614.2</td>
<td>95</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

Table 4  Results of Bonferroni test for comparing the influence of materials (MAT), bonding techniques (BT) and thermal cycling (T)

<table>
<thead>
<tr>
<th>Groups</th>
<th>Contrast</th>
<th>Sign.</th>
<th>Difference</th>
<th>Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAT</td>
<td>BO-TX</td>
<td>p&lt;0.05</td>
<td>−5.49</td>
<td>1.30</td>
</tr>
<tr>
<td>BT</td>
<td>DB-IB</td>
<td>p&lt;0.05</td>
<td>1.50</td>
<td>1.30</td>
</tr>
<tr>
<td>T</td>
<td>NT-T</td>
<td>p&lt;0.05</td>
<td>1.85</td>
<td>1.30</td>
</tr>
</tbody>
</table>

* TX (TransBond XT), BO (Beauty Ortho Bond), DB (direct bonding), IB (indirect bonding), NT (Not Thermally cycled), T (Thermally cycled)

Table 5  Distribution frequency and percentages of adhesive remnant index (ARI). The level of statistical significance was set at p<0.05

<table>
<thead>
<tr>
<th>Groups</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>2 (16.67)</td>
<td>5 (41.67)</td>
<td>3 (25.00)</td>
<td>2 (16.67)</td>
<td>12</td>
</tr>
<tr>
<td>II</td>
<td>4 (33.33)</td>
<td>3 (25.00)</td>
<td>2 (16.67)</td>
<td>3 (25.00)</td>
<td>12</td>
</tr>
<tr>
<td>III</td>
<td>6 (50.00)</td>
<td>4 (33.33)</td>
<td>1 (8.33)</td>
<td>1 (8.33)</td>
<td>12</td>
</tr>
<tr>
<td>IV</td>
<td>6 (50.00)</td>
<td>4 (33.33)</td>
<td>2 (16.67)</td>
<td>0 (0.00)</td>
<td>12</td>
</tr>
<tr>
<td>V</td>
<td>2 (16.67)</td>
<td>7 (58.33)</td>
<td>2 (16.67)</td>
<td>1 (8.33)</td>
<td>12</td>
</tr>
<tr>
<td>VI</td>
<td>3 (25.00)</td>
<td>5 (41.67)</td>
<td>4 (33.33)</td>
<td>0 (0.00)</td>
<td>12</td>
</tr>
<tr>
<td>VII</td>
<td>9 (75.00)</td>
<td>1 (8.33)</td>
<td>2 (16.67)</td>
<td>0 (0.00)</td>
<td>12</td>
</tr>
<tr>
<td>VIII</td>
<td>8 (66.67)</td>
<td>4 (33.33)</td>
<td>0 (0.00)</td>
<td>0 (0.00)</td>
<td>12</td>
</tr>
</tbody>
</table>

χ²=31.93, p value=0.05

TX samples were influenced by thermal cycling to a lesser extent than the BO samples (p<0.05; Table 3). Thermal cycling decreased the SBS values for both bonding techniques (indirect and direct bonding, p<0.05; Table 3). Indirect bonding caused a statistically significant decrease in the SBS values of the two materials (TX and BO; Table 3). The Bonferroni test showed statistically significant differences for multiple comparisons between different groups (Table 4).

Adhesive remnant index
Table 5 shows the ARI scores after debonding. Chi-
squared analysis (level of statistical significance was set at $p<0.05$) comparing the ARI scores among all groups ($\chi^2=31.93$) indicated that the groups were significantly different ($p=0.05$). TX had a greater frequency of ARI values of 2 and 3 (Fig. 2), whereas BO showed a greater frequency of ARI values of 0 and 1 for all groups (Fig. 2). Group I exhibited the highest ARI score, corresponding to the most composite remaining on the enamel surface. The lowest score (least composite remaining) was observed in Group VIII.

**DISCUSSION**

Numerous studies have investigated the strength of self-etching primer systems. Most of these studies demonstrated that self-etching systems have significantly lower SBS values than conventional acid-etching systems. However, the greater SBS of conventional phosphoric acid-etching adhesive systems is offset by the greater extent to which these systems erode the enamel. Plaque accumulation around fixed orthodontic appliance is another major cause of enamel demineralization.

The present study aimed to analyze whether the SBS of an indirectly bonded system, using a self-etching primer containing S-PRG filler as a fluoride-releasing source, could be considered clinically acceptable. This self-etching primers is less abrasive and inhibit the demineralization of adjacent enamel; indirect bonding reduces enamel loss by helping to avoid unnecessary rebonding. For both direct and indirect bonding, the BO self-etching primer provided a lower SBS value than TX with conventional etching. However, the SBS values for all groups were greater than the 6 to 8 MPa estimated by Reynolds to be necessary in orthodontic treatment.

Although self-etching is clinically acceptable, it produces weaker bonds compared to phosphoric acid etching. The findings of the present study are further corroborated by the results of Elekdag-Turk et al. Those authors compared the clinical performances of self-etching and acid-etching systems over a 6-month period, and reported that the two adhesive systems did not show significant variations in failure and survival rates. Thus, the self-etching system can be effectively used for the bonding of orthodontic brackets.

The higher SBS value of conventional acid-etching can be attributed to the etching action of phosphoric acid, which offers the advantage of increased bond strength. However, phosphoric acid etching causes a greater degree of enamel loss. Hashimoto et al. evaluated the erosion of BO and TX on enamel by atomic force microscopy, observed that TX increased the porosity of the enamel surface and significantly increased the enamel loss. Scougall-Vilchis et al. reported that in scanning electron microscopy (SEM) images of the enamel surface morphology, BO induced more conservative effects than did the acid-etching system.

The BO used in this experiment contained S-PRG filler as a fluoride-releasing source, which contributed to inhibiting enamel demineralization. Fluoride release also promotes tooth mineralization and modulates the acidic conditions produced by oral cariogenic microorganisms. Phosphoric acid-etching adhesive systems for the enamel result in higher SBS values than self-etching systems, but can increase the occurrence of caries, visible as white and brown spots around or under the brackets after debonding.

One other self-etching primer, Transbond Plus [TP] (3M/Unitek Corp.), has a significantly higher mean SBS than that of BO and produces results similar to those of conventional etching with 35% phosphoric acid. BO displays a lower pH value and milder self-etching ability compared to TP. The lower SBS of BO, compared to TP and TX, may arise from the reduced penetration of the adhesive resin into the enamel surface. Indeed, lower porosity in BO systems has been observed via SEM micrographs of untreated enamel surfaces, phosphoric acid-etched surfaces, and self-etched surfaces (TP and BO).

Numerous authors have discussed the advantages and disadvantages of indirect bonding compared to direct bonding, and all agree that the indirect system is more accurate. The indirect procedure was found to provide optimal visibility of the bracket placement as the
bond develops, and is less sensitive to timing. Bracket positioning on the patient model is straightforward and precise, and brackets can be changed easily if necessary. Most studies report significant differences in the SBS of directly and indirectly bonded systems. Some exceptions include Thiyagarajah et al., who compared the clinical performance of the direct and indirect techniques, as well as Linn et al. and Daub et al., who observed no significant differences in the SBS of the two techniques. Comparisons should be interpreted carefully and with discernment by the reader, as most of the previous studies related to indirect bonding have investigated different techniques of bonding procedures with two different types of materials, such as light-cured and chemically cured, but these materials require the application of a conditioner agent such 37% phosphoric acid, which have showed higher mean values of SBS than the self-etching system that has been analyzed in the present study.

During thermal cycling tests, samples are subjected to thermal changes and exposed to water, to simulate the conditions of the oral cavity. In the current study, thermal cycling reduced the mean SBS in all groups. Significant differences were observed in the SBS values before and after thermal cycling. Thermal cycling had a significantly different effect on each material, producing a larger difference in the SBS of BO compared to TX. This difference may explain why the penetration of the self-etching primer into the enamel was lower than with 37% phosphoric acid. Elekdag-Turk et al. noted significant decrease in the mean SBS of systems that had been prepared with phosphoric acid etching after 2,000 and 5,000 thermal cycles, whereas systems that had been prepared with self-etching primer showed a significant decrease of the mean SBS value after 2,000 thermal cycles. However, Daub et al. reported a significant decrease in the mean SBS of metal brackets, directly and indirectly bonded with phosphoric acid and TX adhesive, after only 500 thermal cycles. The comparison of bond strength measurements of different studies is complicated because of variety of materials and methods, including variations in tooth type, storage conditions, method of debonding, analysis of the results, and the selection of products for comparison.

The differences in the ARI scores between the two materials investigated were statistically significant. In the BO groups, less adhesive remained on the enamel, which corresponded to lower SBS scores. In orthodontics, a lower ARI score is favourable because the clinician must remove any remaining adhesive from the tooth after debonding. Significant differences in the distribution of the ARI scores between BO, TX, and TP indicated that the use of BO resulted in more frequent bond failure at the enamel-adhesive interface. Hence, the advantage of the self-etching BO in facilitating clean-up of the enamel surface would be mitigated by the corresponding decrease in SBS between the enamel and bonding resin. Lower ARI was observed in the indirect techniques to compare with direct techniques. The findings of the present study are further corroborated by the results of Klocke et al., whose reported similar ARI result of indirect techniques for TX groups after sandblasting with aluminium oxide 50 μm to remove stone and separating medium. Shinha et al. observed higher ARI in the indirect techniques when the bracket were bonded on the working model with water soluble glue instead of resin where sandblasting were unnecessary.

CONCLUSIONS

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

1. The mean values of SBS yielded by a fluoride-releasing orthodontic adhesive (BO) were significantly lower than those of TX with 35% phosphoric acid etching.
2. Indirect bonding resulted in significantly lower SBS values than direct bonding.
3. Thermal cycling decreased the SBS values of all tested groups. BO exhibited significantly lower SBS values than TX before and after thermal cycling.
4. Samples prepared with TX primer and phosphoric acid etching showed higher ARI values than samples prepared with BO, indicating that more adhesive remained on the teeth. Use of the BO self-etching primer with the indirect-bonding technique provides sufficient SBS for clinical applications, and a reduced amount of remnant adhesive.

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