

## Shear Bond Strength of a New Fluoride-releasing Orthodontic Adhesive

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This study evaluated the shear bond strength of stainless steel brackets bonded to enamel with a new fluoride-releasing orthodontic adhesive system. A total of 140 extracted human bicuspid teeth were randomly divided into four groups. Group I (Transbond XT) was a control group in which enamel was etched with phosphoric acid. For the remaining groups, enamel was conditioned with a self-etching primer (SEP): Group II (Transbond Plus), Group III (BeautyOrtho Bond), and Group IV (BeautyOrtho Bond + Salivatect). Stainless steel brackets were bonded to all tooth samples. After which, the samples were stored, thermocycled, tested, and statistically analyzed. Besides bond strength evaluation, the adhesive remnant index (ARI) was also evaluated. The shear bond strengths of Groups II, III, and IV were significantly lower than Group I, and Group II was significantly greater than that of Group III. Concerning ARI scores, no significant differences were found between the groups. Further, no enamel fracture was observed during shear bond test with the new SEP.

In conclusion, when enamel was conditioned with the new SEP, the mean values of shear bond strength yielded were lower than when it was etched with 37% phosphoric acid. Nonetheless, these mean values were higher than the average suggested by Reynolds as optimum for clinical treatment.

Keywords: Self-etching primer, Shear bond strength, Adhesive remnant index

### INTRODUCTION

The direct bonding of orthodontic brackets with composite resins has been considered as one of the most significant developments in orthodontics<sup>1</sup>. Nevertheless, orthodontic bonding awaits more - and much needed - clinical improvements, especially in white spot lesion reduction. In response to this problem, manufacturers have produced fluoride-releasing orthodontic adhesives to prevent this undesirable effect while maintaining adequate bond strength<sup>2</sup>.

Nowadays, the use of acid-etch bonding technique when attaching brackets to the enamel surface with conventional adhesive systems (that employ enamel conditioner, primer solution, and adhesive resin) is still widely accepted by most orthodontists as a routine technique<sup>3</sup>. However, acid etching produces iatrogenic effects on the enamel surface - and amongst which, the loss of enamel<sup>3,9</sup>.

In an effort to improve the adhesion procedure, reduce loss of enamel, prevent saliva contamination, and save chairside time, self-etching primers (SEPs) have been introduced in the market<sup>3,4,10</sup>. These self-etching systems combine conditioning and priming agents into a single acidic primer solution and are considered as bicomponent hydrophilic adhesives<sup>5</sup>. Presently, in terms of bonding stainless steel brackets to enamel, SEPs have shown to be a favorite alternative to the separate two-step etch and primer system, with a time reduction of 65%<sup>11</sup>. Although

they were introduced only recently to the dental profession, they are already enjoying routine usage by 20% of dental practitioners in the United States<sup>12</sup>.

The purpose of this study was to evaluate the shear bond strength of stainless steel brackets bonded to enamel with a new fluoride-releasing orthodontic adhesive system.

### MATERIALS AND METHODS

#### *Teeth*

A total of 140 extracted human bicuspid teeth were collected and stored in a solution of 0.1% (wt/vol) thymol. The criteria for tooth selection were similar to those described by Bishara *et al.*<sup>13</sup>. The teeth were cleansed and then pumiced with fluoride-free paste (Pressage, Shofu Inc., Kyoto, Japan) and rubber prophylactic cups (Merssage, Shofu Inc., Kyoto, Japan) in a low-speed handpiece (10 seconds). The teeth were thoroughly washed with water (30 seconds) and air-dried.

#### *Brackets*

A total of 140 stainless steel bicuspid brackets (0.018 inches; Standard Edge, Dyna-Lock, 3M Unitek, Seefeld, Germany) were used. The average surface area of the bracket base was 14.1 mm<sup>2</sup>.

#### *Bonding procedure*

Table 1 lists the adhesive materials employed in this

Table 1 Adhesive materials employed in this study.

Group	Material (Category)	Batch No.	Manufacturer
I	Transbond XT (Light-cure adhesive primer)	4BY	3M Unitek, Monrovia, Calif., USA
I	Transbond XT (Light-cure adhesive paste)	5NA	3M Unitek, Monrovia, Calif., USA
II	Transbond Plus (Self-etching primer)	200089-L5	3M Unitek, Monrovia, Calif., USA
II	Transbond XT (Light-cure adhesive paste)	5NA	3M Unitek, Monrovia, Calif., USA
III	BeautyOrtho Bond Primer A (Self-etching primer)	11031301	Shofu Inc., Kyoto, Japan
III	BeautyOrtho Bond Primer B (Self-etching primer)	03041101	Shofu Inc., Kyoto, Japan
III	BeautyOrtho Bond (Light-cure adhesive paste)	02040901	Shofu Inc., Kyoto, Japan
IV	BeautyOrtho Bond Primer A (Self-etching primer)	11031301	Shofu Inc., Kyoto, Japan
IV	BeautyOrtho Bond Primer B (Self-etching primer)	03041101	Shofu Inc., Kyoto, Japan
IV	Salivatect (Light-cure flowable adhesive)	01041401	Shofu Inc., Kyoto, Japan
IV	BeautyOrtho Bond (Light-cure adhesive paste)	02040901	Shofu Inc., Kyoto, Japan

study. The teeth were randomly divided into four groups (n=35/group).

Group I: Control; Transbond XT (3M Unitek, Monrovia, Calif., USA). The teeth were etched (37% H<sub>3</sub>PO<sub>4</sub>; 30 seconds), washed with a water spray (30 seconds), and dried to a chalky white appearance. Then, the adhesive primer (Transbond XT, adhesive primer, 3M Unitek, Monrovia, Calif., USA) was applied to the etched surface. The bracket was placed on the tooth, bonded (Transbond XT, adhesive paste, 3M Unitek, Monrovia, Calif., USA), and light-cured (Coltolux 4, Coltène/Whaledent Inc., USA) for 30 seconds. Light curing was divided into three time lapses (10 seconds each) on the mesial, distal, and occlusal sides respectively.

Group II: Transbond Plus (3M Unitek, Monrovia, Calif., USA). The teeth were conditioned with a SEP (Transbond Plus) according to the manufacturer's instructions. Brackets were bonded with Transbond XT and light-cured as in Group I.

Group III: BeautyOrtho Bond (Shofu Inc., Kyoto, Japan). The teeth were conditioned with a new SEP. After which, brackets were bonded with BeautyOrtho Bond and light-cured using the same protocol of Group I.

Group IV: BeautyOrtho Bond + Salivatect (Shofu Inc., Kyoto, Japan). Both SEP and adhesive were as per those used in Group III. However, before bonding the brackets, a thin layer of Salivatect - which was a flowable resin - was applied on the enamel surface.

#### *Storage and thermocycling*

A stainless steel wire of 0.017 × 0.025 inches was ligated into each bracket slot to help reduce deformation of the bracket during debonding process. The teeth were embedded in acrylic resin (Orthodontic

Resin, Dentsply Caulk International Inc., USA), with a label bearing the number of each sample. A mounting jig was used to align the facial surface of the tooth to be perpendicular to the bottom of the mold and its labial surface parallel to the force during the shear bond strength test. After which, the teeth were stored in distilled water at 37 °C for 15 days and thermocycled<sup>14</sup> 2000 times in 5 and 60 water baths, with a dwell time of one minute in each bath.

#### *Shear bond strength test*

An occlusogingival load was applied to each bracket, producing a shear force at the bracket-tooth interface (Fig. 1). This was accomplished by using the flattened end of a steel rod which was attached to the crosshead of a testing machine (Strograph, Toyo Seiki Seisaku-sho Ltd., Tokyo, Japan). Bond strength was measured at a crosshead speed of 5.0 mm/min, load applied at fracture was recorded, and shear bond strength was calculated by dividing the load by the bracket base area (14.1 mm<sup>2</sup>).

#### *Adhesive remnant index (ARI)*

ARI score of each specimen was evaluated according to the original description of ARI<sup>5</sup>, whereby the scores were given as follows: 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 distinct impression of the bracket mesh. In addition, similar to the modified ARI<sup>10</sup>, the presence of enamel fractures was registered.

#### *Enamel surface morphology*

The enamel surface of an untreated tooth and the enamel surfaces conditioned with the different kinds

of adhesive material employed in this study were observed under a scanning electron microscope (SEM, S-4500, Hitachi Co. Ltd., Japan). The samples were chemically prefixed with 2.5% glutaraldehyde solution buffered at pH 7.4 with 0.2 M cacodylate buffer (4 ; 2 hours), and rinsed twice with 0.1 M cacodylate buffer, pH 7.4 (4 ; 20 minutes  $\times$ 2). They were then chemically fixed with 1% OsO<sub>4</sub> solution buffered at

pH 7.4 with 0.2 M cacodylate buffer (4 ; 1 hour), and rinsed twice with 0.1 M cacodylate buffer, pH 7.4, (4 ; 20 minutes  $\times$ 2). Following which, the specimens were dehydrated in a graded series of ethanol, immersed in t-butanol (20 minutes  $\times$ 2), and freeze-dried (VFD-21S, Vacuum Device Inc., Ibaragi, Japan). Finally, the samples were placed on aluminum stabs and coated with osmium (10 seconds) (HPC-1S, Vacuum Device Inc., Ibaragi, Japan).

*Statistical analysis*

For shear bond strength test, descriptive statistics including the mean, standard deviation, and range values were calculated. Scheffé's *post hoc* multiple comparison test (one-way ANOVA) with significance predetermined at  $<0.05$  was carried out. Furthermore, chi-square test was used to analyze the ARI data.

RESULTS

*Shear bond strength*

Table 2 presents the descriptive statistics of shear bond strength. All groups showed higher bond strength than the average (5.9-7.8 MPa) suggested as optimum for clinical treatment<sup>16</sup>. Nevertheless, Groups II (12.7  $\pm$  3.3 MPa), III (8.0  $\pm$  4.2 MPa), and IV (10.0  $\pm$  4.5 MPa) were significantly lower than Group I (18.1  $\pm$  5.5 MPa), and Group II was significantly greater than Group III.

*ARI*

Table 3 shows the ARI scores after debonding. Chi-square comparisons of ARI scores among all groups ( $\chi^2=14.7$ ) indicated that the groups were not significantly different ( $p=0.096$ ). In terms of enamel fracture, it was observed only in Groups I and II.

*Enamel surface morphology*

The scanning electron micrographs of the untreated enamel surface and the enamel surfaces treated with the conditioners employed in this study are shown in Fig. 2. The smooth surface of the untreated enamel changed dramatically when it was etched with phos-

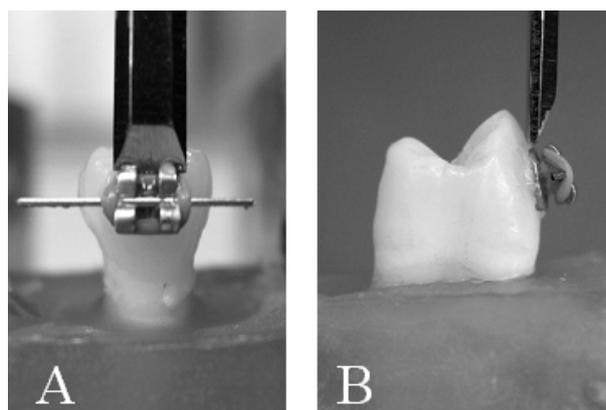


Fig. 1 Representative images of a bonded tooth set in acrylic block and positioned in the testing machine. (A) Front view; and (B) Side view.

Table 2 Descriptive statistics of shear bond strength

Group	Shear Bond Strength (MPa)		
	Mean	SD	Range
I	18.1	5.5	24.6
II	12.7	3.3	13.5
III	8.0	4.2	19.1
IV	10.0	4.5	16.6

Table 3 Distribution frequency and percentages of adhesive remnant index.

Group	ARI scores (%) <sup>15</sup>				n	EF
	0	1	2	3		
I	10 (28.5)	21 (60.0)	2 (5.7)	2 (5.7)	35	5
II	10 (28.5)	23 (65.7)	2 (5.7)	0 (0.0)	35	3
III	16 (45.7)	17 (48.5)	2 (5.7)	0 (0.0)	35	0
IV	7 (20.0)	28 (80.0)	0 (0.0)	0 (0.0)	35	0

<sup>2</sup>=14.7; df=9; p=0.0967 n: number of specimens EF: enamel fracture

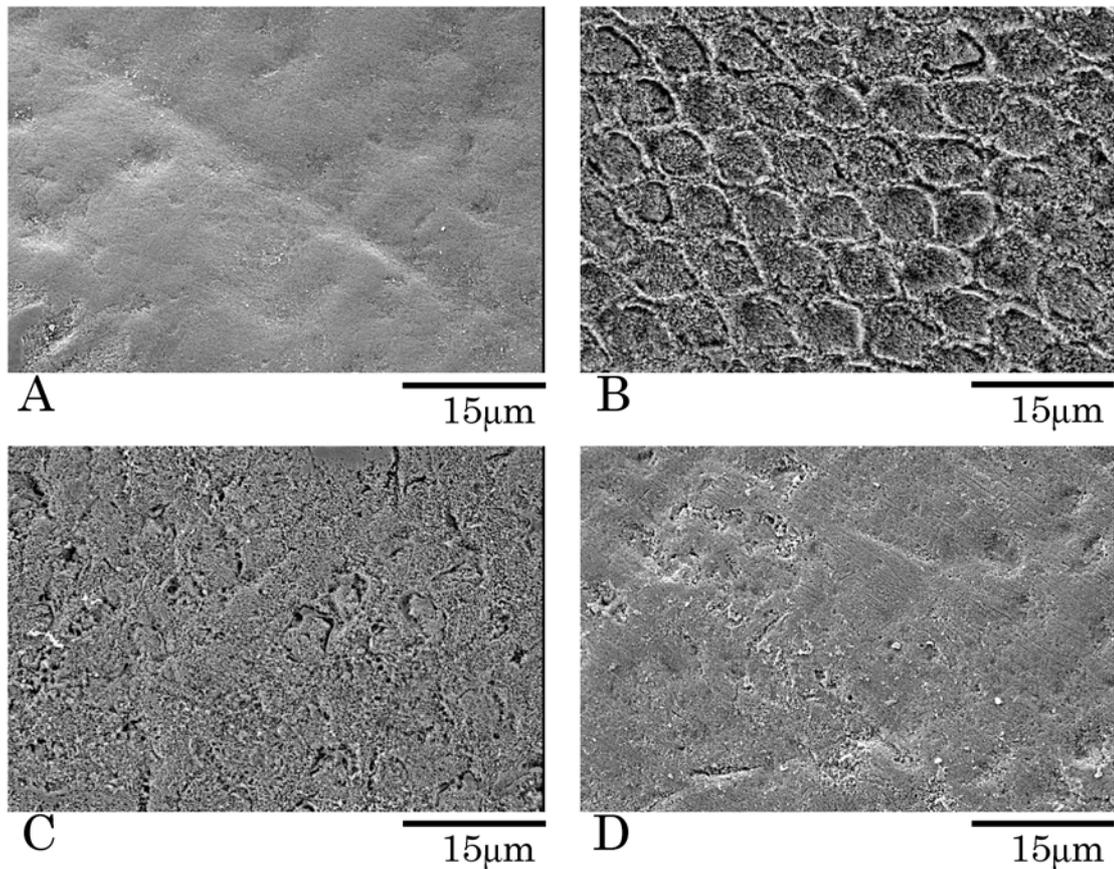


Fig. 2 Appearance of human enamel surfaces under a scanning electron microscope. (A) Untreated; (B) Etched for 30 seconds with 37%  $H_3PO_4$  (Group I); (C) Conditioned with SEP Transbond Plus (Group II); and (D) Conditioned with SEP primer A and primer B of BeautyOrtho Bond (Groups III and IV). (Accelerating voltage: 5.0kV; magnification:  $\times 2000$ ).

phoric acid. On the other hand, action of both SEPs seemed to be more conservative than the etching agent.

## DISCUSSION

### *Shear bond strength*

As per the caution sounded out by other authors<sup>2,17-19</sup>, *in vitro* studies present various limitations and their comparisons should be interpreted carefully and with much discernment by the readers. For example, variations in experimental conditions that evaluated bond strength have been shown to take a toll on the results such that it has become difficult to compare results<sup>20</sup>. In the context of shear bond strength measurement, factors that influence results include: type of teeth selected<sup>21</sup>, enamel surface morphology<sup>22</sup>, enamel conditioners<sup>9</sup>, curing devices<sup>23</sup>, light guides<sup>19,23</sup>, curing time<sup>24</sup>, bracket bases<sup>25</sup>, bracket components<sup>26</sup>, storage conditions<sup>14</sup> including storage/immersion time<sup>27</sup>, loading mode<sup>28</sup>, and debonding force direction<sup>29</sup>. Therefore, clinicians should consider these factors - and their influences - when

assessing the behavior of orthodontic adhesives in a specific situation.

Recently, Transbond XT has been widely tested and frequently selected as the control<sup>1,4,5,10,11,13,30</sup>. The bond strength of this material has shown values as low as 6.2 MPa<sup>31</sup> to as high as 23.4 MPa<sup>1</sup>; even so, majority of the studies reported that the values ranged from 9.6 to 14.8 MPa<sup>4,5,11,13,14,17,22,30,32</sup>. In agreement with the findings of this study, Transbond XT in Group I (Control) presented the highest shear bond strength at 18.1 MPa. This high value could be attributed to the etching action of phosphoric acid, which offers the advantage of increased bond strength. However, its application causes a greater degree of enamel loss<sup>9</sup>. In this study, five incidences of enamel fracture were observed when enamel was conditioned with 37% phosphoric acid. Thus, the use of etching materials should be considered only for cases where increased bond strength is a topmost priority and chief goal in the treatment plan<sup>18,20</sup>.

As for the SEPs, they demonstrated advantages such as lower extent of damage to enamel, prevent saliva contamination, and save chairside time<sup>6,11,12,20</sup>.

However, the bond strengths yielded when enamel was conditioned with SEPs varied widely, ranging from 2.8 to 11.55 MPa<sup>5</sup>. Moreover, disadvantages - such as higher bond failure rate<sup>33</sup> and poor bonding strength<sup>17</sup> - have been described and touted as reasons whenever SEPs are not recommended for clinical use. Nevertheless, some studies showed no significant differences in bond strength between SEPs and conventional etching agents<sup>10,34-36</sup>.

In this study, the bond strength of SEPs in Groups II, III, and IV were higher than the average suggested by Reynolds<sup>16</sup> as optimum for clinical orthodontic treatment. In this light, SEPs could be recommended for clinical use - which was in agreement with other studies<sup>4,30,37</sup>. Further, the shear bond strength of Group II was significantly higher than Group III - probably due to differences attributable to the action of the SEPs (Fig. 2). In this connection, Chitnis *et al.*<sup>27</sup> reported that the bond strength of Transbond XT was significantly higher than Beautifil, a fluoride-releasing dental material for restorations that contains a similar filler to BeautyOrtho Bond.

In summary, an ideal orthodontic adhesive should have adequate bond strength while maintaining an unblemished enamel<sup>5</sup>, such that the bonding is able to withstand functional forces but at the same time allow bracket debonding without causing damage to the enamel<sup>7</sup>.

#### ARI

The omission of pumice prophylaxis before orthodontic bonding with SEPs has a significant effect on clinical bonding whereby the bond failure rate is increased<sup>34</sup>. The low amount of adhesive remnant exhibited in all groups was most probably associated with the prophylaxis protocol. In any case, a low ARI score suggests that tooth clean-up after debonding is likely to be easier and faster<sup>38</sup>. Conversely, the use of acid conditioners complicates the removal of residual adhesive on the enamel after debonding and may also lead to surface scratches, cracking, and loss of sound enamel<sup>18,20,35</sup>. Furthermore, when etching agents are employed and high bond strength is presented, the amount of adhesive remaining tends to be larger<sup>6,39</sup>.

On the other hand, enamel fractures were found to occur when the force exceeded 14 MPa<sup>7</sup>. In addition, the frequency of enamel fracture increased with an increase in bond strength<sup>2,5,7,10,30</sup>. These reports agreed with our findings whereby five and three enamel fractures were observed in Group I and Group II respectively. However, *in vitro* debonding increases the frequency of enamel fractures<sup>2</sup>, and a gentle clinical debonding may prevent this undesired effect. In this study, the presence of enamel fractures correlated with a higher bond strength. Hence,

Group I presented the highest bond strength and the highest frequency of enamel fractures.

#### Enamel surface morphology

Scanning electron micrographs of the enamel surfaces in Fig. 2 show a more conservative action of both SEPs than that of the etching agent, and these observations are in agreement with previous studies<sup>3,35,40</sup>. Although both SEPs showed less damage to the enamel, the appearance of the enamel treated with the SEP in Groups III and IV was more similar to untreated enamel. In other words, action of the SEP in Groups III and IV was probably gentler than the other SEP used in Group II.

## CONCLUSIONS

When enamel was conditioned with a new self-etching primer, the mean values of shear bond strength yielded by a new fluoride-releasing orthodontic adhesive (BeautyOrtho Bond) were significantly lower than that of Transbond XT with 37% phosphoric acid etching. Nonetheless, these values were higher than the average suggested by Reynolds as optimum for clinical treatment. Moreover, no enamel fracture was observed during shear bond test when the new self-etching primer was used. In addition, SEM micrographs showed that the new self-etching primer produced less damage to enamel than the etching agent - keeping in line with the principle and concept of minimal intervention.

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