

## Influence of Self-etchant Application Time on Bracket Shear Bond Strength

Adam Wade Ostby<sup>a</sup>; Samir E. Bishara<sup>b</sup>; John Laffoon<sup>c</sup>; John J. Warren<sup>d</sup>

### ABSTRACT

**Objective:** To determine the influence of self-etching primer (SEP) application time on the shear bond strength of orthodontic brackets.

**Materials and Methods:** Forty human molars were cleaned, mounted, and randomly divided into two groups. The same SEP, adhesive, and brackets were used in both groups. Twenty teeth were conditioned following the manufacturers' recommendations by rubbing the SEP on the enamel surface for 3 to 5 seconds. The remaining 20 teeth were conditioned using the same SEP, but the application time was increased to 15 seconds. The teeth were debonded within half an hour following initial bonding using a universal testing machine. After debonding, the amount of residual adhesive remaining on the tooth was determined. Student's *t*-test was used to compare the shear bond strength (SBS) of the two groups, and the  $\chi^2$  test was used to compare the Adhesive Remnant Index (ARI) scores for the two adhesive systems.

**Results:** The mean SBS of the brackets bonded to the teeth subjected to the SEP for 3 to 5 seconds was  $8.0 \pm 4.6$  MPa and was not significantly different ( $t = -0.69$ ,  $P = .494$ ) from the SBS of the brackets bonded using a 15-second SEP application time ( $\bar{x} = 8.9 \pm 3.4$  MPa). The comparisons of the ARI scores between the two groups ( $\chi^2 = 2.16$ ) indicated that bracket failure mode was not significantly different ( $P = .340$ ) for both groups, and most failures were at the bracket-adhesive interface.

**Conclusion:** The present findings indicate that increasing the SEP application from 3 to 5 seconds to 15 seconds does not result in a significant increase in SBS.

**KEY WORDS:** Self-etch duration; Shear Bond; Brackets

### INTRODUCTION

Over the past 50 years, the bonding of various adhesives to enamel and dentin has developed a niche in nearly all areas of dentistry, including orthodontics. Adhesion is currently described as a combination of mechanical, adsorption, diffusion, and electrostatic phenomena.<sup>1</sup> Mechanical theories propose that ad-

hesion occurs primarily through microscopic interlocks between the adherend and adhesive. The increase in the contacting surface area between the two results in a greater number of interlocks and thus greater adhesive forces.

The clinical significance of using these microscopic interlocks for bonding followed the introduction of the enamel acid-etch technique by Buonocore in 1955.<sup>2</sup> By demonstrating a 100-fold increase in retention of small polymethylmethacrylate buttons to teeth that had been etched with 85% phosphoric acid for 30 seconds, Buonocore opened the door to modern adhesive dentistry techniques. Further studies determined that microporosities created during the acid-etching process allowed for the incorporation of small resin tags into the enamel surface, thereby creating microscopic mechanical interlocks between the enamel and resin.<sup>3-5</sup>

While manufacturers of bonding systems typically recommend specific conditioning protocols, the effect of changing the conditioning time on the etching pattern has been investigated previously, yielding differ-

<sup>a</sup> Predoctoral Dental Student, College of Dentistry, University of Iowa, Iowa City.

<sup>b</sup> Professor, Orthodontic Department, College of Dentistry, University of Iowa, Iowa City.

<sup>c</sup> Research Assistant, College of Dentistry, University of Iowa, Iowa City.

<sup>d</sup> Associate Professor, Department of Preventive and Community Dentistry, University of Iowa, Iowa City.

Corresponding author: Dr. Samir E. Bishara, Orthodontic Department, College of Dentistry, S219 DSB, University of Iowa, Iowa City, IA 52242

(e-mail: karla-starckovich@uiowa.edu)

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ing results. Some studies have shown that a reduced etching time does not result in lower bond strengths<sup>6-8</sup>; other authors have reported significant reductions in bond strengths when decreasing etching time.<sup>9,10</sup> In addition, Olsen et al<sup>11</sup> specifically investigated the effect of enamel etching time on the shear bond strength (SBS) of orthodontic brackets. Their study concluded that while 0- and 5-second etch times were insufficient to successfully bond brackets, no significant difference in SBS was seen when the etch time was reduced from the recommended 30 seconds to only 10 seconds.<sup>11</sup> Thus, it was determined that this shorter etching time provided clinically acceptable SBSs when used to bond orthodontic brackets.

The traditional three-step etch-prime-adhesive procedure has been used for years to successfully bond orthodontic brackets to teeth. Since the depth of enamel dissolution during the etching process is of importance, the potential use of alternative enamel conditioners has been studied to improve the bonding procedure by minimizing enamel loss and reducing chair time while still maintaining sufficient bond strengths between the brackets and enamel.<sup>12-14</sup> While these conditioners were initially developed for use on dentin, researchers have determined that adhesive systems combining conditioning and priming can be successfully used to bond orthodontic brackets to enamel.<sup>15-17</sup> It has been demonstrated that SBSs of brackets bonded using different self-etch primers (SEPs) were not significantly different from brackets bonded with the conventional acid-etch technique.<sup>15,17</sup>

As each SEP is unique in formulation and conditioning aggressiveness, the manufacturers of these products typically recommend using a specific conditioning time and method to achieve optimal results. While the recommended protocols usually provide satisfactory results, it was reported that significantly less leakage was found in class V restorations when an SEP was used for twice the manufacturer's recommended application time.<sup>18</sup> Additional results from a recent study indicated that for certain self-etching adhesives, doubling the recommended application time resulted in significantly stronger tensile bonding strengths to enamel.<sup>19</sup> The method of applying the SEP has also been shown to have an influence on bond strength; Miyazaki et al<sup>20</sup> found that active application (rubbing) of SEPs to ground enamel improves the resulting bond strength and produces a more pronounced enamel etch pattern.<sup>20</sup>

While studies have shown that conditioning time influences the marginal integrity of restorations as well as microtensile bond strengths, little data are available regarding the effect of SEP application time on the SBS of orthodontic brackets. Thus, the purpose of this study was to determine the effect of increasing the rec-

ommended SEP application time on the SBS of orthodontic brackets.

## MATERIALS AND METHODS

### Teeth

Forty freshly extracted human molars were collected and stored in a solution of 0.2% (weight/volume) thymol. To meet the criteria for use in the study, the teeth were selected only if they had intact buccal enamel, had not been pretreated with chemical agents (eg, H<sub>2</sub>O<sub>2</sub>), had no surface cracks from extraction forceps, and were free of caries. The teeth were embedded in dental stone placed in phenolic rings (Buehler Ltd, Lake Bluff, Ill). A mounting jig was used to align the facial surfaces of the teeth perpendicular with the bottom of the mold. This kept the buccal surface of the tooth parallel to the applied force during the shear test. Following mounting, the teeth were cleaned and polished with pumice and rubber prophylactic cups for 10 seconds.

### Brackets

Orthodontic lateral incisor metal brackets (APC Plus Victory series; 3M Unitek, Monrovia, Calif) were used in the study. These brackets are precoated with a light-cured composite adhesive. Before bonding, the average surface area of the bracket base was determined to be 10.3 mm<sup>2</sup>.

### Groups Tested

*Group 1.* Twenty teeth were bonded using the manufacturer's recommended protocol. Transbond Plus SEP (3M Unitek) uses a loli-pop system that has two compartments: one contains methacrylated phosphoric acid esters, initiators, and stabilizers, whereas the other contains water, fluoride complex, and stabilizers. To activate the product, the two compartments are squeezed so that the contents of each compartment are allowed to mix. The resulting mix is then applied by continuously rubbing the SEP on the enamel surface for 3 to 5 seconds. The SEP is then lightly dried using compressed air for 1 to 2 seconds. Each precoated bracket was placed on the tooth, and a 300-g force was applied (Correx force gauge, Bern, Switzerland) for 10 seconds. The force gauge is used to help ensure a uniform adhesive thickness between the bracket and enamel. The bracket was then light cured using a Halogen curing light (Ortholux XT Curing Light; 3M Unitek) for 20 seconds (10 seconds from each proximal side).

*Group 2.* Twenty teeth were bonded following the same procedure as described for group 1, but with one exception. In group 2, the SEP was applied by contin-

**Table 1.** Descriptive Statistics in Megapascals (MPa) and the Result of the *t*-Test Comparison of the Two Groups Tested<sup>a</sup>

Group	SEP Application		$\bar{x}$	SD	Range
	Time	n			
1	3–5 s	20	8.0	4.6	3.5–17.4
2	15 s	20	8.9	3.4	2.9–14.4

<sup>a</sup>  $X@Xt$ -test =  $-0.69$ ;  $P = .494$ . SEP indicates self-etching primer; n, sample size;  $\bar{x}$ , mean; SD, standard deviation; and P, probability.

uously rubbing the enamel surface for 15 seconds instead of the recommended 3 to 5 seconds. The SEP was dried, brackets placed, and bonding adhesive cured in the same manner as described for group 1.

### Debonding Procedure

The SBS of each group was determined within half an hour from the time of bonding to simulate the clinical conditions wherein archwires are first tied to newly bonded teeth. A steel rod with a flattened end was attached to the crosshead of a Zwick testing machine (Zwick GmbH, Ulm, Germany). The rod applied an occlusogingival load to the bracket, producing a shear force at the bracket-tooth interface. The results of each test were recorded by a computer that is electronically connected to the testing machine. The Zwick machine (cell capacity = 50 kN) recorded the results from each test in megapascals (MPa) at a crosshead speed of 5.0 mm per minute.

### Adhesive Remnant Index

Once the brackets were debonded, the enamel surface of each tooth was examined under  $10\times$  magnification to determine the amounts of residual adhesive remaining on each tooth. A modified adhesive Remnant Index (ARI) was used to quantify the amount of remaining adhesive using the following scale: 1 = all the adhesive remained on the tooth, 2 = more than 90% of the adhesive remained on the tooth, 3 = between 10% and 90% of the adhesive remained on the tooth, 4 = less than 10% of the adhesive remained on the tooth, and 5 = no adhesive remained on the tooth.

### Statistical Analysis

Student's *t*-test was used to determine whether there was a significant difference in SBSs between the two test groups, and the  $\chi^2$  test was used to compare the bond failure mode (ARI scores) between the two groups. For the purpose of statistical analysis, the ARI scores 1 and 2 as well as 4 and 5 were combined. Significance for all statistical tests was predetermined at  $P \leq .05$ .

**Table 2.** Frequency Distribution of the Modified Adhesive Remnant Index (ARI) Scores and  $\chi^2$  Comparisons of the Two Groups<sup>a</sup>

Group	SEP Application		Modified ARI Scores				
	Time	n	1	2	3	4	5
1	3–5 s	20	7	4	5	3	1
2	15 s	20	8	6	5	1	—

<sup>a</sup>  $X@X\chi^2 = 2.16$ ;  $P = .340$ . SEP indicates self-etching primer; 1, all adhesive remained on the tooth; 2, more than 90% of the adhesive remained on the tooth; 3, 10% to 90% of the adhesive remained on the tooth; 4, less than 10% of the adhesive remained on the tooth; and 5, no adhesive remained on the tooth.

### Scanning Electron Microscopy

To assess the effects of the two SEP application protocols on the enamel surface, scanning electron microscopy (SEM) was performed following their application on a molar with an intact enamel surface. One molar, which met the same criteria as those molars used for strength testing, was selected and sectioned in half mesiodistally. On one half, the buccal surface was conditioned using the SEP application described for group 1 (3–5 seconds with rubbing). On the other half, the buccal surface was subjected to the SEP application protocol described for group 2 (15 seconds with rubbing). The purpose of using the same buccal surface for the two application methods is to ensure some standardization in the enamel surface tested. After conditioning, the two halves were washed with acetone to dissolve the layer of SEP on the enamel surface and then dried with compressed air. The teeth were not light cured. These halves were then affixed to aluminum SEM stubs and sputter coated with gold at a current of 20 mA for 2 minutes. The enamel surfaces of representative examples of each tooth half were observed under SEM, and photographs were taken at  $500\times$  and  $2000\times$ .

## RESULTS

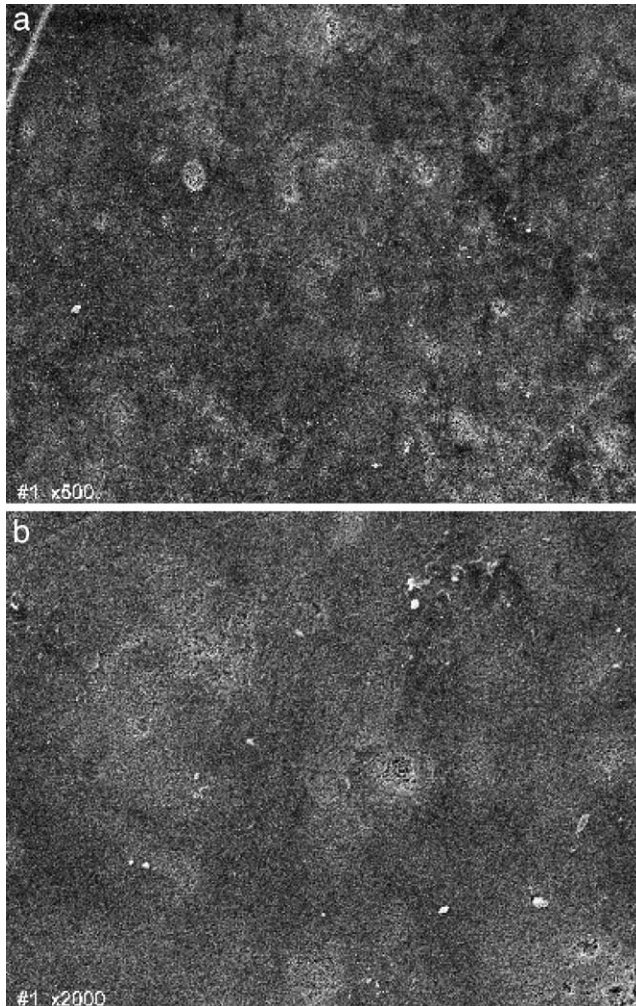
### Shear Bond Strength

The descriptive statistics including the mean, standard deviation, minimum values, and maximum values for the two bonding protocols are presented in Table 1. The mean SBS for the brackets bonded using the recommended 3- to 5-second SEP application time was  $8.0 \pm 4.6$  MPa and for the brackets bonded using an SEP application time of 15 seconds,  $8.9 \pm 3.4$  MPa. The *t*-test comparisons ( $t = -0.69$ ) indicated that these values were not significantly different from each other ( $P = .494$ ).

### Adhesive Remnant Index

The failure modes of the two groups are presented in Table 2. The  $\chi^2$  comparisons of the ARI scores be-



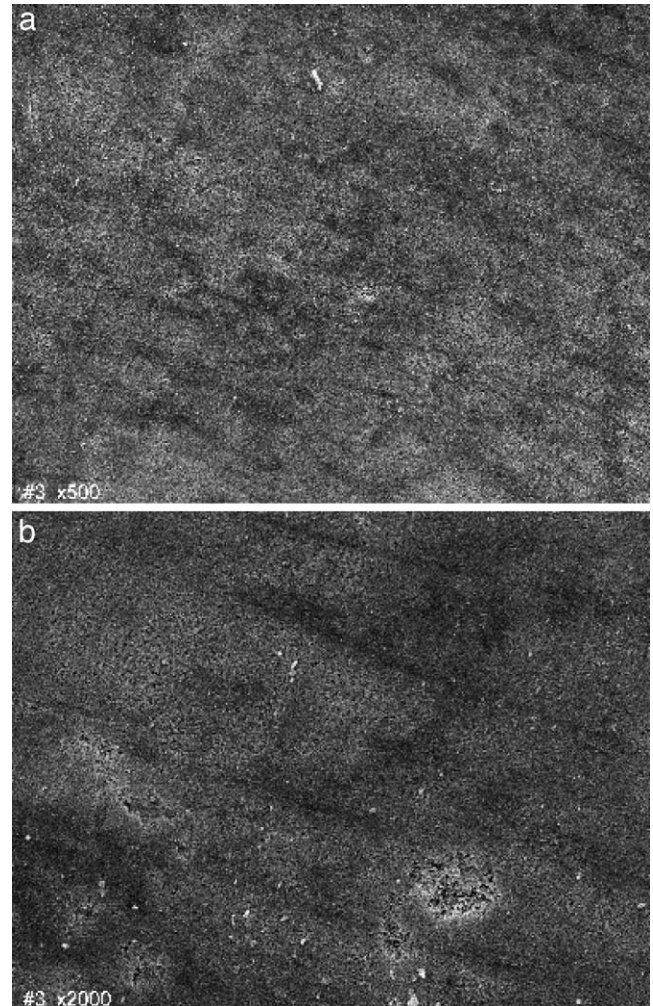


**Figure 1.** (A) Scanning electron microscopy (SEM) view of enamel surface after application of Transbond Plus SEP for 3 to 5 seconds with rubbing (500 $\times$ ). (B) SEM view of enamel surface after application of Transbond Plus SEP for 3 to 5 seconds with rubbing (2000 $\times$ ).

tween the two groups ( $\chi^2 = 2.16$ ) indicated that the two methods of SEP application had similar ( $P = .340$ ) bracket failure modes. For both groups, most adhesive remained on the tooth after debonding (groups 1 and 2), indicating failure at the bracket-adhesive interface.

### Scanning Electron Microscopy

SEM micrographs were obtained after the enamel surfaces were subjected to the different SEP application times and washed with acetone. These micrographs are presented in Figures 1 and 2. The enamel etch patterns seen in the two groups were very similar; both protocols produced sporadic shallow fossae on the enamel surface (Figures 1a and 2a). When viewed at higher magnification, the presence of microscopic porosities in the enamel surface was also apparent in both groups (Figures 1b and 2b).



**Figure 2.** (A) Scanning electron microscopy (SEM) view of enamel surface after application of Transbond Plus SEP for 15 seconds with rubbing (500 $\times$ ). (B) SEM view of enamel surface after application of Transbond Plus SEP for 15 seconds with rubbing (2000 $\times$ ).

### DISCUSSION

Traditional methods of bonding orthodontic brackets to teeth have relied on the use of the acid-etch technique to achieve adequate retention. However, of primary concern to the clinician is the maintenance of a sound, unblemished enamel surface after the removal of brackets. SEPs were introduced in an effort to minimize enamel loss and also to reduce the bonding procedure from three to two steps, effectively decreasing technique sensitivity and reducing chair time. While typically designed for use in operative bonding procedures, SEPs have been used to successfully bond orthodontic brackets with SBS values similar to the conventional acid-etch technique.<sup>15</sup>

Although many researchers have studied the effect of etching time on bond strengths, most of these studies were conducted using phosphoric acid as the enamel etchant.<sup>6-11</sup> More recently, the effect of SEP

application time on bonding strength has also been tested. It was determined that increasing SEP application time resulted in a more pronounced etch pattern on ground enamel as well as greater tensile bonding strengths.<sup>19,20</sup> While the effects of SEP application time on etching pattern and bond strengths have been investigated for operative dentistry needs, little research has specifically focused on whether SEP application time influences bracket bond strengths. Thus, this study was conducted to determine whether increasing the SEP application time affects the SBS of orthodontic brackets to intact enamel.

The present findings indicated that for the SEP tested, increasing the application time from the recommended 3 to 5 seconds up to 15 seconds does not significantly increase the SBS. The mean SBS of the brackets bonded using a 3- to 5-second conditioning time was  $8.0 \pm 4.6$  MPa, and for the brackets bonded using an SEP application time of 15 seconds, it was  $8.9 \pm 3.4$  MPa. In addition, both application methods have a similar effect on the enamel surface (Figures 1 and 2) and result in comparable debonding modes.

It has been suggested that a minimum SBS of 6.0 to 8.0 MPa is adequate for bonding orthodontic brackets to teeth.<sup>21,22</sup> In this study, the mean SBS of both groups was within this range in the first half hour. While both bonding protocols provide acceptable SBS, increasing the SEP application time provides little benefit to the clinician and may increase chair time.

## CONCLUSIONS

- Increasing the SEP application time from the recommended 3 to 5 seconds to 15 seconds did not significantly increase the bracket bond strength.
- From a clinical standpoint, increasing the SEP application time results in a minimal benefit and would likely increase bonding time.

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