

# Effect of Self-etchant pH on Shear Bond Strength of Orthodontic Brackets: An *in vitro* Study

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## ABSTRACT

**Aims:** To determine the effect of self-etchant pH on shear bond strength of orthodontic brackets; to compare the shear bond strengths of brackets bonded with three SEPs and brackets bonded with conventional etch, rinse, bond method and to find the brackets/adhesive failure mode.

**Materials and methods:** One hundred and twenty premolar teeth were cleaned, mounted, and randomly divided into four groups of 30 samples each- Transbond XT conventional etch and bond system (control), Adper SE Plus SEP (3M ESPE) with a pH of 0.9 to 1.0, Transbond Plus SEP (3M Unitek) with a pH of about 1.0 and Clearfil SE Bond SEP (Kuraray America) with a pH of around 2.0. All teeth were bonded with Transbond XT paste (3M Unitek). The teeth were debonded within half an hour after initial bonding by using a universal testing machine. The residual adhesive on each tooth was evaluated. ANOVA was used to compare the shear bond strength (SBS) of the three groups, and the Chi-square test was used to compare the adhesive remnant index (ARI) scores for the three groups.

**Results:** ANOVA indicated significant differences between the groups. Clearfil attained the SBS ( $6.5 \pm 0.6689$  MPa) closest to the control group, whereas Adper in spite of being the most aggressive recorded the lowest SBS ( $5.7 \pm 0.5695$  MPa). Transbond self-etching primer achieved a mean SBS of  $6.1 \pm 0.6211$  MPa. However, all the three SEPs recorded SBS which was significantly less than that of Transbond conventional etch, rinse and bond system ( $11.8027 \pm 0.8059$  MPa). The comparisons of the ARI scores between the three groups indicated that bracket failure mode was significantly different between the three groups ( $p < 0.05$ ).

**Conclusion:** These findings show that factors other than pH, such as the ability of the bonding adhesive to form a chemical bond to enamel and the strength of the bonding adhesive itself, significantly influence the SBS of orthodontic brackets.

**Keywords:** Self-etching primers, pH, Shear bond strength, Adhesive remnant index.

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## INTRODUCTION

Bis-GMA or Bowen's resin, a self-cure resin, was introduced in late 1960s and was the only choice available to orthodontist for many years.<sup>1-4</sup> Though it provided good

bond strength, it had a few inherent flaws like being extremely technique sensitive, having a short setting time which effects bracket positioning accuracy and a low initial bond strength.

The clinical significance of utilizing microscopic interlocks for bonding followed the introduction of the enamel acid-etch technique by Buonocore in 1955.<sup>5</sup> By the 1970s, bonding of orthodontic brackets had become an accepted clinical technique.<sup>6,7</sup> 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>7,8</sup>

The established procedure for bonding brackets to enamel with composite resin requires a series of preparatory steps-pumicing, etching, priming and bonding. This traditional 3-step etch/prime/adhesive procedure has been used for years to successfully bond orthodontic brackets to teeth. Even though the acid-etching technique is useful in orthodontics, improved techniques are needed to maintain clinically useful bond strengths while minimizing enamel loss and to simplify the technique by reducing the number of steps. SEPs have become popular in orthodontic bonding because they combine the conditioning and priming agents into a single acidic primer solution. This eliminates the washing and drying stages, which are necessary in the conventional method, saving clinical time, reducing procedural errors and minimizing technique sensitivity.

Research on the ability of self-etching primers to adequately bond orthodontic brackets has provided mixed results. Bishara and Gordon (1999) concluded that the shear bond strength obtained with an acidic primer might not be reliable.<sup>9</sup> However, in a more recent study, Bishara et al (2001) suggested that although the mean shear bond strength of a self-etching primer was significantly lower than that of the control group (37% phosphoric acid and sealant), it was clinically acceptable.<sup>10</sup>

However, there seems to be little information in the literature on how the pH and aggressiveness of SEPs affect the enamel surface and bracket bonding.

So, the present study has been designed to evaluate the shear bond strength of orthodontic brackets bonded with three self-etching primer systems having different pH values and compare them with normal etch, rinse and prime method.

## MATERIALS AND METHODS

This study was designed to compare the shear bond strength of orthodontic brackets bonded with three different self-etching primers having different pH values and also to compare these shear bond strength values to that of brackets bonded by conventional etch, rinse and bond method.

For the purpose of the study 120 freshly extracted premolars were collected and stored in a solution of 0.2% (weight/volume) thymol.

To meet the criteria for this study, the teeth were selected only if:

- They had intact buccal enamel
- They had not been pretreated with chemical agents (e.g. hydrogen peroxide)
- They had no surface cracks from extraction forceps
- They had no caries.

The teeth were embedded in acrylic resin placed in cuboidal aluminum blocks. A mounting jig (Figs 1A to C) 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. After mounting, the teeth were cleaned and polished with pumice and rubber prophylactic cup for 10 seconds.

Metallic first premolar brackets (Dentaurum equilibrium 2) were used (Fig. 5) the three SEPs used in this study were as follows:

- Adper SE Plus (3M ESPE) (Fig. 4B)
- Clearfil SE Bond (Kuraray America) (Fig. 4D)
- Transbond Plus (3M Unitek) (Fig. 4C).

In the conventional group, 3M Unitek etchant and 3M Unitek Transbond XT light cure adhesive primer (Fig. 4A) were used for etching and priming the tooth surface respectively.

These SEPs were applied to the mounted teeth according to the protocols suggested by the manufacturers.

The same light-cured composite adhesive resin, Transbond XT paste (3M Unitek) (Fig. 2), and the same light source, Monitex Bluelex LD-105 LED light curing unit (Fig. 3), were used to bond brackets in all the groups.

Samples were divided into four different groups each containing 30 teeth (Figs 6A to D):

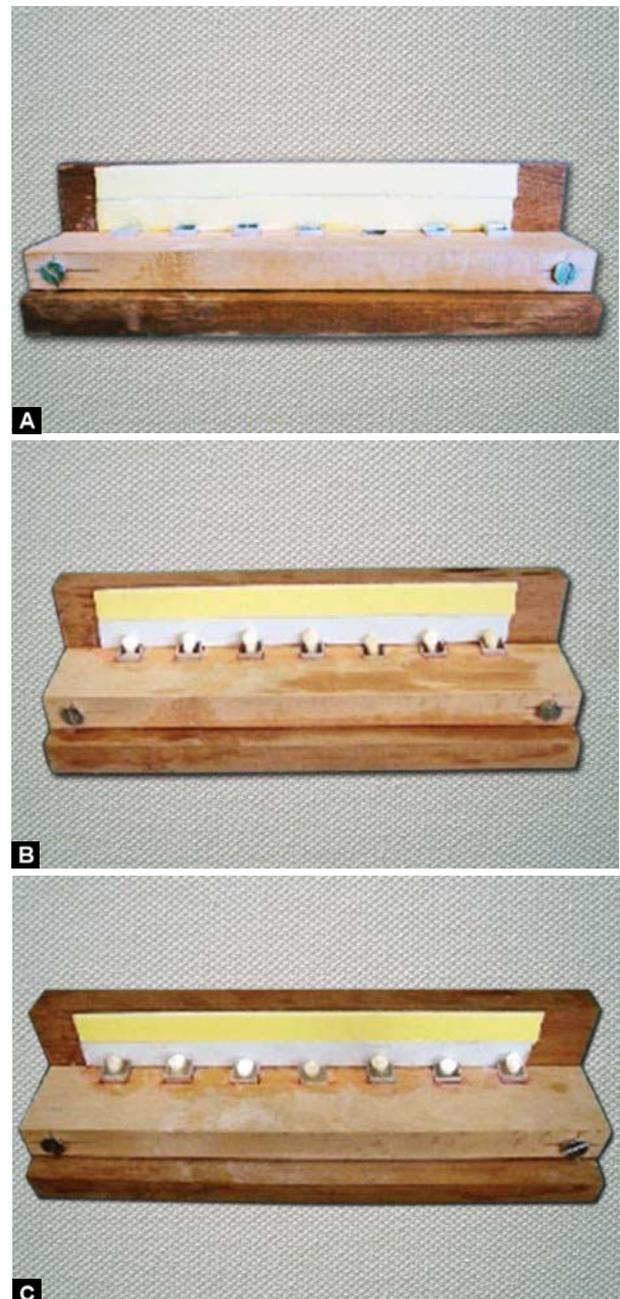
*Group 1:* Control group—bonding with conventional acid etching and priming

*Group 2:* Experimental group A—bonding with Adper SE Plus SEP

*Group 3:* Experimental group B—bonding with Transbond XT SEP

*Group 4:* Experimental group C—bonding with Clearfil SE Bond SEP

The pH of Adper is marketed as 0.9 to 1.0, but it might be as low as 0.4.<sup>11</sup> The pH of Transbond Plus is about 1.0.<sup>12</sup> The pH of Clearfil SE Bond is 2.0.<sup>13</sup>



**Figs 1A to C:** Wooden jig for mounting extracted teeth into aluminum cuboids with acrylic resin: (A) Before mounting, (B) after pasting teeth to the tape, (C) after putting acrylic resin



**Fig. 2:** Composite used for bonding brackets



**Fig. 3:** LED light curing unit



**Fig. 4D:** Clearfil SE Bond SEP (group 4)



**A**



**Fig. 5:** Brackets used in the study  
Dentaurum equilibrium 2 bracket kit



**B**



**C**

**Figs 4A to C:** Materials used for bonding brackets to extracted premolars: (A) Transbond XT conventional etch and bond (control group—group 1), (B) Adper SE Plus SEP liquids A and B (group 2), (C) Transbond XP SEP (group 3)

Shear bond strength test was done with a JJ Lloyd Universal testing machine (Fig. 7) having a capacity of 20000 N (2040 kg) at Brakes India Private Limited, Nanjungud. The machine was set and calibrated according to manufacturer's instructions. The aluminum block with mounted premolar and its bonded bracket were positioned in the jig (Fig. 8), so that the labial surface is parallel to the force during the shear strength test. A steel rod with a flattened end was used to apply load at the bracket. The brackets were shear tested to failure using a load cell of 12N (346.5 Kg) and a crosshead speed of 1.0 mm/min. The force producing failure was recorded in Newton by a computer attached to the machine and converted into force per unit area (MPa) by dividing the measured force values by the surface area of the bracket. The surface area of the mesh was approximately 9.608 mm<sup>2</sup> as measured with digital vernier calipers.

#### Adhesive Remnant Index

The percentage of the surface of the bracket base covered by adhesive was determined using stereomicroscope (Fig. 9) with an eye piece magnification of 16 $\times$ .

The percentage of the area still occupied by adhesive remaining on the tooth after debonding was obtained by



**Fig. 6A:** Ready samples with color coding done: Yellow—group 1



**Fig. 6B:** Ready samples with color coding done: Blue—group 2



**Fig. 6C:** Ready samples with color coding done: Black—group 3



**Fig. 6D:** Ready samples with color coding done: Green—group 4



**Fig. 7:** Universal testing machine



**Fig. 8:** Jig for mounting the specimen to the clamp of the universal testing machine

subtracting the area of adhesive covering the bracket base from 100%. Later each tooth was assigned an adhesive remnant index (ARI) value according to Artun and Bergland.<sup>14</sup>

*Score 0:* No adhesive left on the tooth (Fig. 10A)

*Score 1:* Less than 1/2 of adhesive left on the tooth (Fig. 10B)

*Score 2:* More than 1/2 of adhesive left on the tooth (Fig. 10C)

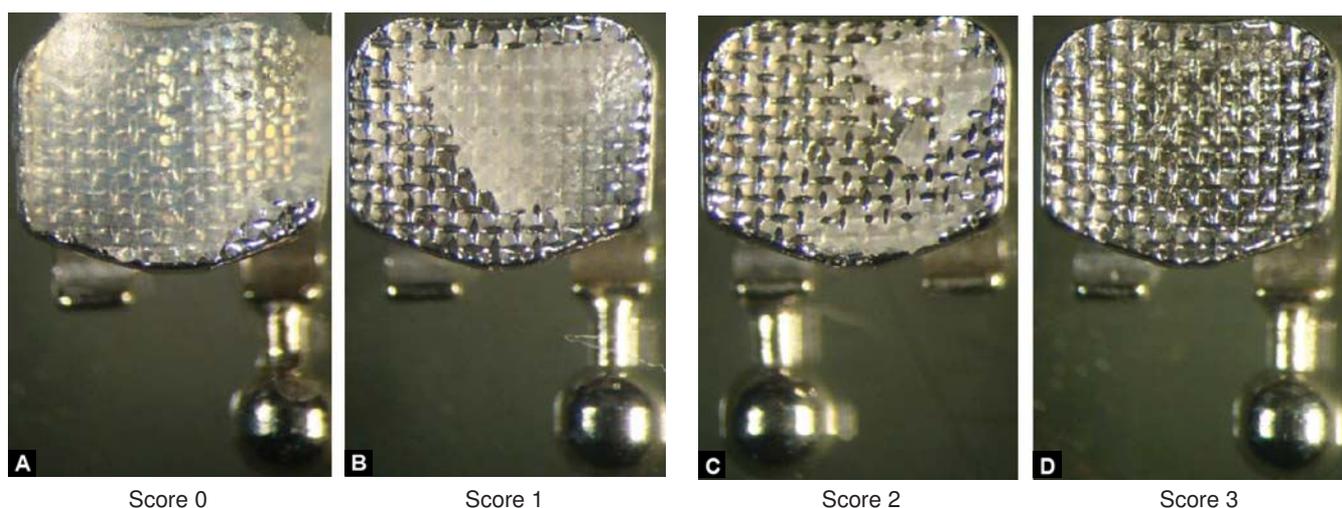
*Score 3:* All adhesive left on the tooth (Fig. 10D).

### Statistical Analysis

ANOVA was used to determine whether there was a significant difference in SBS between the four groups, and the Duncan multiple range test was used to determine which groups were significantly different from each other. The Chi-square test was used to compare the bond failure mode (ARI scores) among the four groups. For the statistical analysis, ARI scores of 0 and 1 and of 2 and 3 were combined. Significance for all statistical tests was predetermined at  $p \leq 0.05$ .



Fig. 9: Stereomicroscope used in the study



Score 0

Score 1

Score 2

Score 3

Figs 10A to D: Undersurface of debonded brackets seen under 16× magnification in a stereomicroscope

## RESULTS

### Shear Bond Strength

Mean SBS for all the four groups are shown in Table 1. In the control group (group 1-Transbond XT) the mean shear bond strength was  $11.8027 \pm 0.8056$  MPa; in case of group 2 (Adper SEP) it is  $5.7053 \pm 0.5695$  MPa; in case of group 3 (Transbond SEP) it is  $6.1000 \pm 0.6211$  MPa and in case of group 4 (Clearfil SEP) it is  $6.5000 \pm 0.6689$  MPa (as depicted in Graph 1). The p-value (Table 1) showed that the shear bond strength of the control group was significantly increased compared to experimental groups (groups 2, 3 and 4). Duncan's post-hoc test was done from which it became evident that there was a significant difference between the mean shear bond strengths among all the four groups.

### Adhesive Remnant Index

Comparison between groups was done using Chi-square test (Table 2). p-value was higher than 0.05 which showed a significant difference between the four groups. In self-etching primer group ARI scores (Graphs 3 to 5) show that more than half of the adhesive comes off with the bracket during debonding (score 0 and 1). In control group (Transbond XT) ARI scores (as depicted in Graph 2) show that the entire or more than 50% of adhesive was left over the tooth surface (score 2 and 3). This signifies that most of the failures in the control group were between bracket mesh and adhesive or within the adhesive. In case of experimental groups (2, 3 and 4) it occurred between the resin and the tooth leaving less than half of the adhesive on the tooth surface (score 0 and 1).

**Table 1:** Descriptive statistics in megapascals (MPa) and the results of ANOVA for the comparisons between the SBS of the four groups

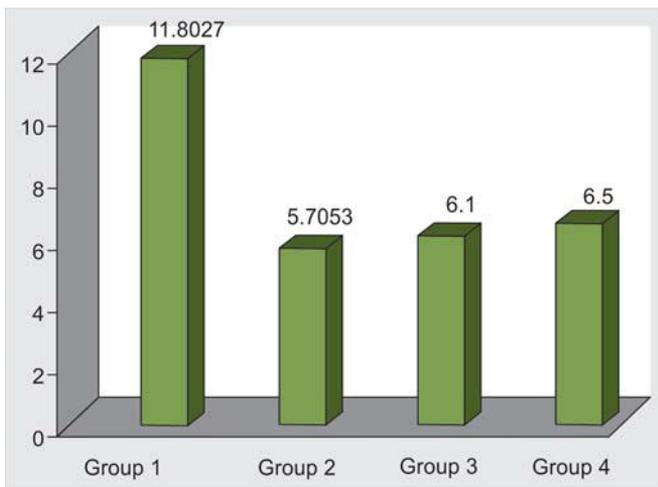
SEP	n	Total SBS	Mean SBS	SD	Range	*
Transbond conventional	30	354.0800	11.8027	0.8056	10.5300-13.0200	A
Adper SE Plus SEP	30	171.1600	5.7053	0.5695	4.5600-7.0200	B
Transbond Plus SEP	30	183.0000	6.1000	0.6211	4.3600-7.0700	C
Clearfil SE Bond SEP	30	195.0000	6.5000	0.6689	5.2300-7.7100	D

F ratio = 546.7256; p = 0.0000; \*groups—with the same letters are not significantly different from each other

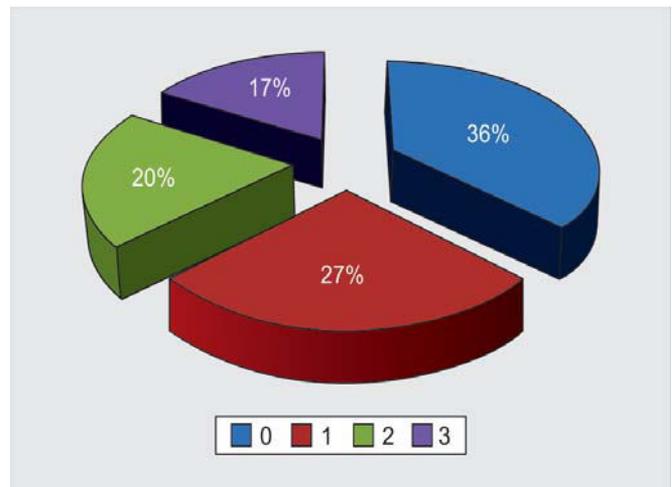
**Table 2:** Frequency distribution of the ARI scores and the result of the Chi-square comparisons

Groups	ARI scores				
	n	0	1	2	3
Transbond conventional	30	7	3	9	11
Adper SEP	30	11	8	6	5
Transbond Plus SEP	30	9	8	6	7
Clearfil SE Bond SEP	30	10	8	7	5

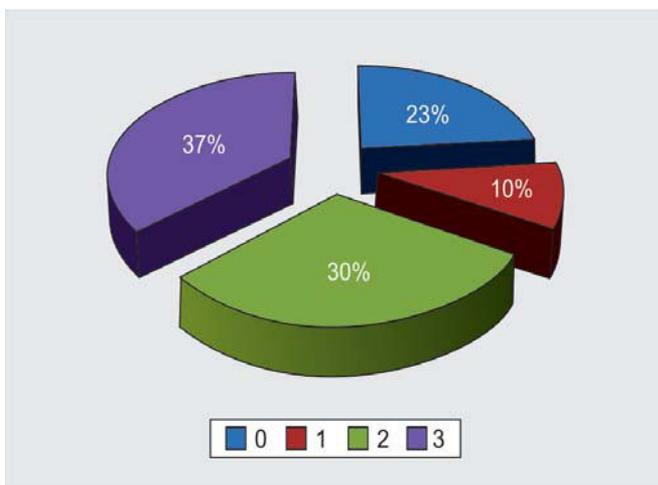
Chi-square = 8.0094; Degree of freedom = 9; p = 0.5332



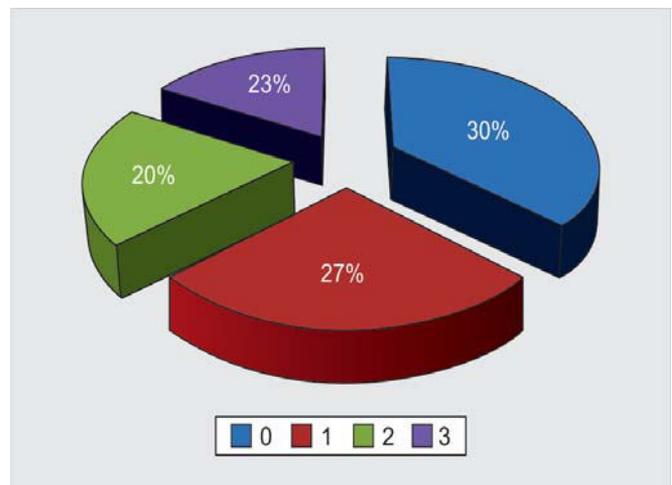
**Graph 1:** Comparison of the SBS of the four groups



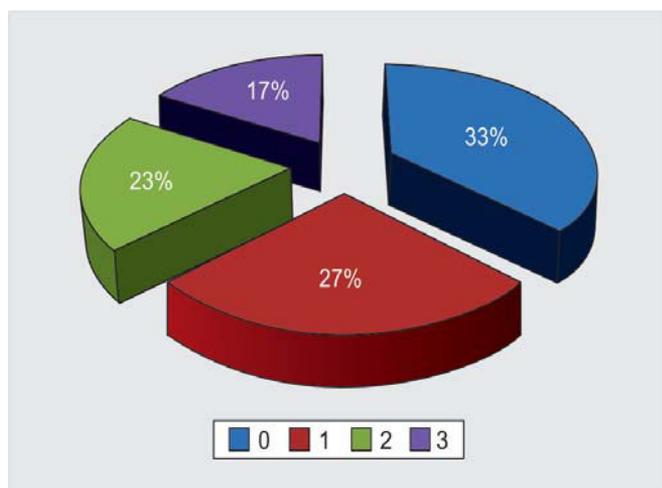
**Graph 3:** Pie diagram depicting distribution of ARI scores in group 2



**Graph 2:** Pie diagram depicting distribution of ARI scores in group 1



**Graph 4:** Pie diagram depicting distribution of ARI scores in group 3



**Graph 5:** Pie diagram depicting distribution of ARI scores in group 4

## DISCUSSION

Various SEPs have been developed with unique formulations, pH values and etching characteristics. Although all SEPs have a low pH, an attempt was made to group these products based on their relative acidity. Van Meerbeek et al classified self-etch adhesives as ‘mild’ (pH about 2, submicron hybrid layer formation) and ‘strong’ (pH <1.0, interfacial morphology similar to etch and rinse adhesives).<sup>15</sup> SEPs with a lower pH are typically more aggressive than those that are not as acidic; they have consistently shown greater enamel-dentin dissolution, a deeper etching pattern, and the formation of thicker hybrid layers.<sup>16-20</sup> This correlation has been observed on both ground and unground enamel, as well as on dentin. However, the effect of 10 contemporary self-etching systems on the dentin interface morphology was evaluated, and different etching patterns among SEPs of similar pH were observed. It was concluded that the pH of the SEP was not the only determining factor in the self-etchant's aggressiveness. The authors suggested that other factors, including the acid dissociation constant, compound structure, application time, and the solubility of the salts formed by the interactions, also influence the action of SEPs.<sup>21</sup>

Historically, it was assumed that the primary mechanism of retention during bonding was the mechanical interlocking between the enamel and the adhesive. As a result, it was thought that a deeper etching pattern would provide a greater surface area for bonding and thus greater bonding strengths. However, in a test of three SEPs on unground enamel, no correlation was seen between etching aggressiveness and tensile strength.<sup>20</sup> More specifically, the most aggressive self-etchant was shown to produce a similar etching pattern as a 37% phosphoric acid control, but it produced a tensile bond strength ( $\mu$ TBS) that was significantly lower (13.9

MPa) than the control (27.0 MPa). Conversely, the most aggressive self-etchant had a similar  $\mu$ TBS to the two self-etchants (11.6 and 10.3 MPa) that produced much shallower and uneven etching patterns.<sup>20</sup> Similar findings were observed by De Munck et al who demonstrated that the most acidic of five self-etchants produced the deepest hybrid layer but had the weakest bond strengths to both enamel and dentin.<sup>18</sup> Other studies showed that SEPs that produce shallow, less-defined etch patterns on ground enamel achieve similar SBS as those with a deeper etching pattern.<sup>22</sup> However, it was suggested that the bond strength of less aggressive SEPs might produce lower bond strength when used on intact enamel.<sup>23</sup> Moreover, Pashley et al used a modeling approach to show that theoretically, the strength of the adhesive-hard-tissue bond is influenced not only by the hybrid layer and surface adhesion, but also by the strength of the resin.<sup>24</sup> SEPs have different pHs, penetration abilities and SBS values. This range of pH-penetration ability and how it influences the SBS of orthodontic brackets have not been tested.

Although Adper SE Plus is the most acidic among all the self-etching systems used, this system did not provide the highest resin-enamel bond strength value. As Adper SE Plus is very acidic, the adhesive layer after solvent evaporation can be quite thin, meaning polymerization may be inadequate due to oxygen inhibition. This may also explain the observation that increasing the number of coats of aggressive self-etching primers enhances the resin-dentin bond strengths.

A second hypothesis to explain the low resin-enamel bond strength observed for Adper SE Plus is related to the adhesive placed over the self-priming solution. The effect of the residual solvent that remains within the adhesive interface affects the resin-enamel bond strengths. It was demonstrated that lower acetone concentration—which can be anticipated from solvent evaporation during clinical use of the bonding agent—did not seem to lower the resin-enamel bond strengths, but rather improved the integrity of the adhesive interface. Besides, the lower the remaining solvent content, the higher the mechanical properties of the polymer formed, which is also relevant for the integrity of the adhesive interface.

A lower pH produces a deeper etch pattern, but an aggressive etch does not always correlate to greater bond strength. The integrity of the enamel surface after debonding is of primary importance to the clinician; therefore, products with sufficient SBS while minimizing the etching effects on the enamel surface would be more advantageous.

Bond failure sites were characterized using the adhesive remnant index. Analysis of teeth tested showed that there was significant difference in pattern of bond failure sites

among the two groups. There was maximum frequency of score 0 and 1 in self-etch priming group and score of 2 and 3 in conventional group, i.e. majority of the samples in self-etch primer failed at the tooth-adhesive interface, whereas in conventional group failed at the bracket adhesive interface. Previous studies have provided inconsistent findings about the amount of residual adhesive on the teeth with use of self-etching primer. Some studies found less residual adhesive on the enamel with self-etching primer than with conventional phosphoric acid etching, whereas other reported significantly more.

## CONCLUSION

The following conclusions can be drawn from the study:

- The SBS of the most acidic SEP in the study Adper is less than the other groups and is also outside the ideal range of 6.0 to 8.0 MPa for bonding orthodontic brackets to teeth.
  - The SBS of the other two SEPs, Transbond SEP and Clearfil SE, is less than the control group but is within the ideal range for bonding orthodontic brackets.
  - The SEP with the highest pH (least acidic), Clearfil SE Bond, bonded brackets successfully and with the strongest SBS among the three SEPs; this suggests that the pH of the SEP is not the primary determinant of the SBS.
- These findings show that factors other than pH, such as the ability of the bonding adhesive to form a chemical bond to enamel and the strength of the bonding adhesive itself, significantly influence the SBS of orthodontic brackets.
- The ARI scores signify that the bond failure occurs mostly between the bracket and adhesive or within the adhesive itself while bonding with a conventional etch-rinse-bond system; and it occurs between the tooth and the adhesive while bonding with a SEP. So there is an increased chance of enamel removal while using an airtor to remove the remnant composite resin from the tooth surface in case of a conventional etch-rinse-bond method.

So along with reduced chairside time, reduced enamel removal can be another advantage of SEPs.

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