

## Shear Bond Strength of Brackets Bonded to Enamel with a Self-Etching Primer

*Effects of Increasing Storage Time After Activation*

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### ABSTRACT

**Objective:** To evaluate bonding efficacy of activated Transbond Plus Self-Etching Primer (TPSEP) used at different time points with Transbond XT to bond metallic orthodontic brackets to bovine incisors.

**Materials and Methods:** The inferior incisors of 210 bovines were randomly divided into seven groups (n = 30). TPSEPs were mixed, activated, and kept activated for 30 (group 30), 21 (group 21), 15 (group 15), 7 (group 7), 3 (group 3), or 1 (group 1) days before bonding, and in one group (group 0) TPSEP was used immediately after mixed. At day zero, incisors in each group were bonded in exactly the same way. After applying TPSEP, brackets were bonded with Transbond XT, according to the manufacturer's instructions. After 24 hours, shear bond strength (SBS) tests were performed for all samples at a crosshead speed of 0.5 mm/min, and the Adhesive Remnant Index was scored.

**Results:** There were no significant differences between the SBS of groups 0, 1, 3, 7, and 15 ( $P > .05$ ) However, those groups had higher SBS ( $P < .05$ ) compared with groups 21 and 30. No significant difference ( $P > .05$ ) was observed between groups 21 and 30. Despite the decrease in SBS for groups 21 and 30, bond strength values were still satisfactory.

**Conclusion:** After activation, the TPSEP mix can be stored for a period of 15 days without losing its adhesive properties. (*Angle Orthod.* 2009;79:133–137.)

**KEY WORDS:** Self-etching primer; Shear bond strength; Orthodontic brackets; ARI score

### INTRODUCTION

The bonding of orthodontic brackets onto tooth surfaces has greatly improved with the advent of new

products with excellent adhesive properties.<sup>1</sup> Traditional orthodontic bracket bonding systems required the use of a three-step procedure involving three separate agents, an enamel conditioner, a priming agent, and an adhesive resin.<sup>2,3</sup> Self-etching primers (SEPs) were introduced in an effort to reduce the three-step procedure to two steps, effectively reducing chair time and increasing cost-effectiveness, which resulted in increased convenience and potentially reducing costs to the patient. Although designed for use in operative bonding procedures, SEPs/adhesives have been used to successfully bond orthodontic brackets with shear bond strength (SBS) values similar to those for the conventional acid-etch technique.<sup>4,5</sup>

Transbond Plus Self-Etching Primer (TPSEP; 3M Unitek, Monrovia, Calif) is a dental adhesive system developed for orthodontic bonding. The TPSEP system has three compartments. The first contains methacrylated phosphoric acid esters, initiators, and stabilizers; the second contains water, fluoride complex, and stabilizers; and the third is empty. For activation, the chemicals of the first two compartments are

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squeezed into the third compartment, and the resulting mix can be applied directly to the tooth surface. TPSEP allows simultaneous etching and priming of the enamel<sup>6</sup> without requiring the traditional washing after acid application.<sup>7</sup> According to the manufacturer, each TPSEP unit dose contains sufficient material for etching and priming one arch. However, when the whole arch is not bonded in the same session, it is unknown whether the opened, active, SEP can be stored and reused in another session.

The objective of this study was to evaluate shear bond strength of orthodontic brackets bonded with TPSEP that has been activated and then stored for different periods of time before bonding the brackets.

## MATERIALS AND METHODS

For the study, 210 freshly extracted permanent mandibular bovine incisors were collected, cleaned of soft tissue, and stored in 0.1% thymol solution. The criteria for tooth selection included intact buccal enamel that was examined by transillumination with a light-emitting diode (LED) and that had not been pretreated with chemical agents (eg, hydrogen peroxide), had not been cracked by the extraction forceps, and did not contain caries.

The teeth were inserted into polyvinyl chloride tubes (Tigre, Joinville, Brazil) filled with acrylic resin (Clásico, São Paulo, Brazil) leaving only their crowns exposed. The buccal surfaces of the crowns were perpendicularly positioned to the base of the shearing die with a glass square to facilitate correct alignment of the teeth and to enable proper mechanical testing.

Before the bonding procedures, the buccal surfaces of all teeth were submitted to prophylaxis using rubber cups with an extra-fine pumice and water for 15 seconds. Next, the samples were washed with an air/water jet for 15 seconds and dried with an air jet free of oil and water vapor for the same period of time. The rubber cups were replaced after every fifth prophylactic procedure to control for cup wear.<sup>8</sup>

After prophylaxis, the teeth were randomly divided into seven groups ( $n = 30$ ) and stainless steel 0.018-inch maxillary central incisor brackets with a base area of 13.8 mm<sup>2</sup> (AbzilLancer, São José do Rio Preto, Brazil) were selected for the bonding procedure. TPSEPs (methacrylated phosphoric esters, Bis-GMA, initiators based on camphorquinone stabilizers, liquid 2 [yellow blister], water, 2-hydroxyethyl methacrylate, and polyalkenoic acid [Lot 261899D, date of expiry May 2008]) were mixed and activated, and each opened TPSEP was individually packed in small zipped bags and vertically stored, to avoid chemical degradation, inside an 8°C refrigerator. TPSEPs were kept activated for 30 (group 30), 21 (group 21), 15 (group 15), 7 (group 7),



Figure 1. View of the bond strength test setup.

3 (group 3), or 1 (group 1) days before bonding, and in one group (group 0) TPSEP was used immediately after mixing. At day zero, each group was bonded in exactly the same way.

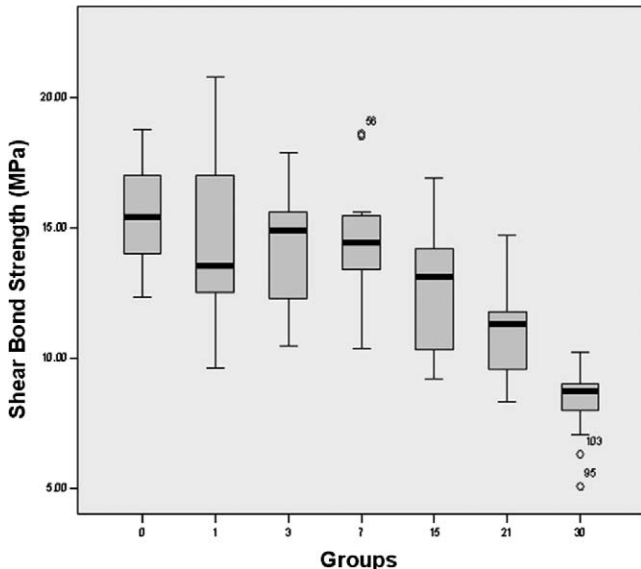
After TPSEP was applied, brackets were bonded with Transbond XT according to the manufacturer's instructions. Activated TPSEP was brushed onto the enamel surface for 3–5 seconds by rubbing with the applicator. The composite Transbond XT (3M Unitek) was applied on the bracket base, after which the bracket was placed on the enamel surface with a 300-g force using a force gauge (Correx force gauge, Bern, Switzerland) for 10 seconds. The force gauge ensured a uniform adhesive thickness between the bracket and enamel. Excess adhesive was removed from the teeth with a probe, and each bracket was then light cured with an LED (850 Mw/cm<sup>2</sup>) for 40 seconds (10 seconds on each side). After bonding, all samples were stored in artificial saliva (Apsen, São Paulo, Brazil) in a 37°C oven for 24 hours.

A custom-made stand was used to stabilize teeth for debonding tests (Figure 1). Each tooth was subjected to a shear load in a Universal Testing Machine model DL-10.000 (EMIC, São José dos Pinhais, Brazil) with a knife-edged blade at a crosshead speed of

**Table 1.** Descriptive Statistics of Shear Bond Strengths (MPa)

Group	N	Mean	SD	Minimum	Median	Maximum	Significance*
0	30	15.42	2.06	12.31	15.42	18.76	A
1	30	14.69	3.25	9.61	13.55	20.79	AC
3	30	14.41	2.42	10.46	14.88	17.9	AC
7	30	14.38	2.25	10.36	14.42	18.6	AC
15	30	12.7	2.48	9.21	13.12	16.89	AC
21	30	10.97	1.83	8.29	11.31	14.72	BC
30	30	8.35	1.35	5.07	8.7	10.24	B

\* Equal letters = absence of statistically significant difference ( $P > .05$ ).



**Figure 2.** Box plot with shear bond strength values. Error bars indicate standard deviation. The circles (o) indicate outliers.

0.5 mm/min. The force was applied parallel to the tooth surface on top of each orthodontic bracket base, and the shear load was recorded at the point of failure. The force per unit area required to dislodge the bracket was then calculated and recorded as the shear bond strength (SBS) in megapascals (MPa).

The enamel surfaces were examined with a stereomicroscope (Stemi 2000-C, Carl Zeiss, Göttingen, Germany) under 16 $\times$  magnification to determine the amount of composite remaining, and then they were

classified according to the Adhesive Remnant Index (ARI).<sup>9</sup> The ARI scores ranged from 0 to 3, with 0 indicating no composite left on the enamel; 1, less than half of the composite left; 2, more than half of the composite left; and 3, all of the composite remained on the tooth surface.

Statistical analyses were performed with the SPSS 13.0 program (SPSS Inc, Chicago, Ill). Descriptive statistics that included mean, standard deviation, median, and minimum and maximum values were calculated for all seven groups. Analysis of variance (ANOVA) was applied to determine whether significant differences existed among the groups. For the post hoc test, the Tukey's test was used. A Kruskal-Wallis and Mann-Whitney U test were used for assessing the ARI scores.

## RESULTS

The descriptive statistics for the SBSs of the seven groups are given in Table 1. No significant differences ( $P > .05$ ) were found among group 0, group 1, group 3, group 7, and group 15 regarding the SBS values (Table 1). However, those groups had higher bond strength ( $P < .05$ ) compared with groups 21 and 30 ( $P < .05$ ). No significant difference ( $P > .05$ ) was observed between groups 21 and 30 ( $P = .522$ ) (Figure 2).

The ARI scores for the seven groups are listed in Table 2. There was no significant difference among groups 0 to 15 and among groups 21 to 30. However,

**Table 2.** Distribution and Descriptive Statistics of the Adhesive Remnant Index (ARI) Scores<sup>a</sup>; Values Represent Number and Frequency (%)

Group	ARI = 0	ARI = 1	ARI = 2	ARI = 3	Median	Mean	SD	Significance*
0	0 (0.0%)	3 (20%)	7 (46.7%)	5 (33.3%)	2	2.13	0.74	A
1	1 (6.6%)	3 (20%)	8 (53.3%)	3 (20.1%)	2	1.86	0.83	AB
3	0 (0.0%)	7 (46.7%)	4 (26.6%)	4 (26.6%)	2	1.8	0.86	AB
7	1 (6.6%)	7 (46.7%)	6 (40.1%)	1 (6.6%)	1	1.46	0.74	B
15	0 (0.0%)	2 (13.2%)	9 (60.2%)	4 (26.6%)	2	2.13	0.63	A
21	8 (-0.533)	4 (26.6%)	2 (13.2%)	1 (6.6%)	0	0.73	0.96	C
30	9 (-0.602)	6 (40.1%)	0 (0.0%)	0 (0.0%)	0	0.4	0.5	C

\* Equal letters = absence of statistically significant difference ( $P > .05$ ).

<sup>a</sup> ARI scores ranged from 0 to 3, with 0 indicating no composite left on the enamel; 1, less than half of the composite left; 2, more than half of the composite left; and 3, all of the composite remained on the tooth surface.

there were significantly different values between groups 0 to 15 and groups 21 to 30 ( $P = .000$ ).

## DISCUSSION

Among SEPs, TPSEP is one of the most frequently used in orthodontics. According to the manufacturer, one TPSEP brush would be enough to bond one dental arch. However, when this material is only needed for bonding a small number of orthodontic brackets, theoretically the remaining material could be individually packed and stored for use on the same patient at another appointment without waste. After TPSEP activation, exposure of the brush to the oral environment and possible saliva contamination could limit reusing the same TPSEP, as it might compromise the final bonding result. Although the complexity of the oral environment could not be reproduced in this study, with proper care, bonding could be easily done in the oral cavity without any contamination. Furthermore, TPSEP has a hydrophilic nature, and the surface of the teeth should not be completely dried to proceed with bonding. In this experiment, cross contamination was also not a concern, as activated TPSEP would never be reused in different patients.

All SBS values of this study were corroborated by other authors<sup>10-14</sup> who similarly compared brackets bonded with TPSEP that was mixed and activated immediately before application. A gradual decrease in those values can be observed in our study, but there was no statistical difference. Even after the components are mixed, activated, and properly stored at 8°C for a prolonged period of time, this material can keep the adhesive properties, probably because storage in low temperatures can reduce the rate of chemical reaction and degradation mechanism.<sup>15</sup>

In the present study no significant differences were found regarding the SBS values among groups 0 to 15. Storage TPSEP could be used within 15 days, and the bond strength was not significantly affected. The SBS significantly decreased in the 21- and 30-day groups, which had an average of 10.96 MPa and 8.33 MPa, respectively. No statistical difference was observed between the 21- and 30-day groups.

Despite the statistical differences between the first five groups and groups 21 and 30, the SBSs for all groups were still satisfactory, as 6 to 8 MPa is adequate for most clinical orthodontic needs.<sup>16</sup> These bond strengths are considered enough to withstand normal masticatory and orthodontic forces, although the low scores (using 21- and 30-day mixed TPSEP) revealed a tendency toward inadequate adhesion to enamel. It might be inferred from this experiment that with longer time points (more than 30 days) the adhesive properties of TPSEP may be compromised.

The ARI analysis revealed that the adhesive for most specimens from groups 0, 1, 3, 7, and 15 remained on the tooth surfaces that demonstrated a better enamel adhesiveness. These results are clinically interesting because bonding resin with high ARI scores can exert a protective effect on the enamel during the process of removing the orthodontic accessories, thereby avoiding enamel fracture.

Studies have demonstrated that when SEPs are used, the degree of penetration by the adhesive to the etched enamel is less compared with the conventional acid-etching technique.<sup>1</sup> The more deeply the enamel surface is penetrated by the adhesive, the greater the risk of damage to the enamel.<sup>4,17</sup>

To avoid premature polymerization after SEP activation, important recommendations include mixing the SEP in a dark ambient to avoid sunlight coming through windows or overhead room light and making the package airtight to prevent water evaporation. It is necessary to mix the storage SEP again before using to allow a proper mix between the camphorquinone and contents of the middle blister. More studies need to be done to test color stability using stored SEP.

An important clinical application of this experiment occurs in the initial phase of the orthodontic treatments because accidental debonding is more frequent.<sup>18</sup> And if a new microbrush is used every time, saliva contamination might be prevented. Additionally, if extreme care were taken to avoid cross-contamination, then the activated TPSEP might be used to bond brackets in other patients.

## CONCLUSIONS

- Storing activated TPSEP up to 15 days does not significantly affect the SBS of orthodontic brackets.
- The low ARI scores observed on days 21 and 30 confirm the time-dependent decrease in the adhesive properties of activated TPSEP.

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