

## Thermocycling Effects on Shear Bond Strength of a Self-Etching Primer

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### ABSTRACT

**Objective:** To determine the effects of thermocycling on shear bond strengths (SBSs) of a self-etching primer (SEP) after 0, 2000, and 5000 thermal cycles.

**Materials and Methods:** Brackets were bonded to bovine incisors with two etching protocols. In the control group (conventional method) teeth were etched with 37% phosphoric acid. In the experimental group, an SEP (Transbond Plus) was applied as recommended by the manufacturer. Brackets were bonded with light-cure adhesive paste (Transbond XT) and light cured for 20 seconds in both groups. The SBSs were measured after water storage at 37°C for 24 hours, after 2000 and 5000 cycles of thermocycling between 5°C and 55°C. Bond failure location was determined with the Adhesive Remnant Index (ARI).

**Results:** In the control group, SBSs did not show any significant differences among 0, 2000, and 5000 thermal cycles. However, in group SEP, SBSs decreased with 2000 and 5000 thermal cycles, and these decreases were significantly different from no thermocycling ( $P < .001$ ). A significant difference was observed between ARI scores of the control group with 5000 thermal cycles and group SEP with no thermal cycles ( $P < .003$ ). In addition, a significant difference was found between group SEP with no thermocycling and with 5000 thermal cycles ( $P < .003$ ).

**Conclusion:** The results of this study indicate that the SEP (Transbond Plus) provides clinically acceptable bond strength values compared with the conventional method after thermocycling.

**KEY WORDS:** Self-etching primer; Thermocycling; Bond strength

### INTRODUCTION

In orthodontic practice, it is essential to obtain reliable adhesive bonds between orthodontic brackets and tooth enamel.<sup>1</sup> The conventional method for bonding orthodontic brackets to enamel necessitates three different agents: an enamel conditioner, a primer solution, and an adhesive resin. Phosphoric acid solution is the most widely applied enamel conditioner. It was reported that a phosphoric acid concentration of 30% to 40% results in the most retentive etching pattern.<sup>2</sup> Nevertheless, phosphoric acid etching has been

claimed to cause iatrogenic damage to the enamel surface.<sup>3,4</sup>

To simplify orthodontic bonding, save chair time, and minimize the amount of enamel loss, manufacturers have introduced self-etching primers (SEPs).<sup>1,4,5</sup> Cal-Neto and Miguel<sup>6</sup> stated that SEP (Transbond Plus Self Etching Primer, 3M Unitek, Monrovia, CA) produced a more conservative etch pattern and a lower adhesive penetration than 37% phosphoric acid. Hosein et al<sup>7</sup> observed that the median enamel loss was significantly lower with SEP (0.27  $\mu\text{m}$ ) than with 37% phosphoric acid (2.76  $\mu\text{m}$ ). In spite of the conservative etch and the lower adhesive penetration patterns, the effectiveness of the SEP has been proven with numerous in vitro studies.<sup>8-11</sup>

Because of the fact that orthodontic adhesives are routinely exposed to thermal changes in the oral cavity, it is paramount to establish whether these changes introduce stress in the adhesive that might affect bond strength. Thus, any new adhesive should be tested both at 24 hours of storage in water and after thermal cycling.<sup>12</sup> Thermal cycling is the in vitro process through which the adhesive resin and the tooth are subjected to temperature extremes compatible with the oral cavity.<sup>13</sup>

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Gale and Darvell<sup>14</sup> pointed to the absence of agreement and standardization between the various thermocycling studies. Different thermocycling regimens were used in in vitro studies investigating the effectiveness of SEP (Transbond Plus).<sup>1,15-17</sup> The main difference among these studies was in the number of thermal cycles (500, 750, 1500, and 2500). At the same time, the temperature extremes were different. The low-temperature points were 5°C or 10°C, and the high-temperature points were 45°C, 50°C, or 55°C. Nevertheless, in these studies the thermocycled samples were not compared with nonthermocycled samples as recommended by Bishara et al.<sup>12</sup>

Saito et al<sup>3</sup> evaluated the bonding durability of metal brackets bonded with Megabond self-etching primer (Kuraray Medical Inc, Tokyo, Japan) with the thermocycling test. In the absence of thermocycling, no significant difference in shear bond strength (SBS) was reported between phosphoric acid and Megabond self-etching primer. After 2000 and 5000 thermal cycles, significant decreases in SBS were observed with phosphoric acid etching. However, with the Megabond self-etching primer, there were no significant differences among SBS values for 0, 2000, and 5000 thermal cycles.

The aim of this study was to evaluate the effects of thermocycling on the SBS values of an SEP (Transbond Plus) after 0, 2000, and 5000 thermal cycles.

## MATERIALS AND METHODS

### Teeth

One-hundred-fifty bovine incisors free of any defects were obtained from a local slaughterhouse. The teeth were cleaned of debris and soft tissue remnants and then polished with pumice and rubber prophylactic cups for 10 seconds. Roots were sectioned 2 mm apical to the enamel cemental junction, and the teeth were stored in 0.1% thymol solution.

### Brackets Used

Stainless steel lower incisor brackets (Gemini bracket; 3M Unitek, Monrovia, CA) were used. The mean area of each bracket base was 10.62 mm<sup>2</sup>.

### Bonding Procedure

The teeth were randomly divided into two groups of 75 teeth each. The brackets were bonded according to one of the following two protocols:

*Control group (conventional method).* The teeth were etched with 37% phosphoric acid for 30 seconds, washed for 20 seconds, and dried for 10 seconds. After etching, a thin uniform coat of primer (Transbond XT Primer; 3M Unitek) was applied. The adhesive res-

in (Transbond XT Light Cure Adhesive Paste; 3M Unitek) was placed onto the bracket base and the bracket was positioned on the enamel surface. Excess adhesive resin was removed with an explorer. Adhesive resin was polymerized for a total of 20 seconds from two directions using a visible light-curing unit with an output power of 600 mW/cm<sup>2</sup>.

*SEP or experimental group.* Transbond Plus Self Etching Primer (3M Unitek) was applied to the enamel surface and rubbed for 3 seconds. Then, a gentle burst of dry air was delivered to thin the primer. The bonding procedure with Transbond XT adhesive resin was performed as for the control group.

### Debonding Procedure

After bracket bonding, all samples in the control group and group SEP were placed in distilled water at 37°C for 24 hours. After water storage, each group was randomly divided into three groups of 25 samples each according to the thermocycling test for 0, 2000, and 5000 cycles.

Twenty-five samples of each group were immediately debonded at room temperature. The remaining samples for the control group and group SEP were subjected to the thermocycling tests of 2000 and 5000 cycles and subsequently debonded. Thermocycling was performed between 5°C and 55°C with a dwelling time of 30 seconds as recommended by the International Organization for Standardization.<sup>18</sup>

The samples were embedded into a cold-cure acrylic resin (Orthocryl; Dentaureum, Ispringen, Germany) cylindrical blocks before the shear bond test.<sup>1</sup> A jig was used to align the buccal surface of each tooth parallel to the cylinder's base.

The shear bond test was performed using a universal testing device (Lloyd LRX; Lloyd Instruments Ltd, Fareham, UK) at a crosshead speed of 1 mm/minute. The bond strengths were calculated in megapascals (MPa).

### Residual Adhesive

The enamel surfaces were examined with a stereomicroscope (Stemi 2000-C; Carl Zeiss, Göttingen, Germany) at a magnification of 10× to determine the amount of composite resin remaining according to the adhesive remnant index (ARI).<sup>19</sup>

### Statistical Analysis

Two-way analysis of variance was used to obtain the significant differences among etching protocols, thermocycling, and their interactions. All treatment combination means for bond strength values were compared by using the Tukey multiple comparison test ( $\alpha = .05$ ).

**Table 1.** Two-way analysis of variance of force (MPa) required to debond metal brackets from bovine teeth

Source of Variation	Sum of Squares	df	Mean Square	F	P value
Adhesive	88.398	1	88.398	11.039	.001
Thermocycling	185.500	2	92.750	11.582	.000
Adhesive × Thermocycling	50.442	2	25.221	3.150	.046
Error	1153.131	144	8.008		
Corrected total	1477.471	149			

A Weibull analysis was performed, and the Weibull modulus, characteristic bond strength, correlation coefficient, and the stress levels at 5% and 10% probability of failure were calculated. Kruskal-Wallis and Mann-Whitney U nonparametric tests were used to determine whether there were any significant differences in the ordinal ARI values ( $P < .003$ ).

**RESULTS**

The two-way analysis of variance showed a significant difference for etching protocols ( $P \leq .001$ ), thermocycling ( $P < .001$ ), and interaction between etching protocols and thermocycling ( $P < .05$ ; Table 1). Mean SBS and standard deviations for each group as well as analysis of the results of the Tukey multiple comparison test are presented in Table 2. For each group, the mean SBSs are shown in Figure 1.

The parameters of the Weibull analysis (modulus, correlation coefficient, characteristic bond strength, and stress levels at 5% and 10% probability of failure) for each group are given in Table 2. The Weibull distribution plots of the probability of failure at a certain shear stress level for the control group and group SEP are depicted in Figures 2 and 3, respectively.

A significant difference was not observed between the control group (18.08 MPa) and group SEP (18.15 MPa) in the absence of thermal cycling ( $P > .05$ ). After 2000 thermal cycles, the mean SBS obtained for the control group (17.14 MPa) was significantly higher than for group SEP (14.50 MPa) ( $P < .05$ ). After 5000

thermal cycles, there was no significant difference between the control group (16.70 MPa) and group SEP (14.68 MPa) ( $P > .05$ ).

In the control group, SBS values did not show any significant differences among 0, 2000, and 5000 thermal cycles. However, in group SEP, SBS values decreased with 2000 and 5000 thermal cycles. These decreased SBS values were significantly different from the SBS value with no thermocycling ( $P < .001$ ).

Median, mean, standard deviation, and range of the ARI scores are given in Table 3. The Kruskal-Wallis test indicated that there were significant differences among the groups ( $\chi^2 = 22.820, P = .000$ ). The Mann-Whitney U test showed a significant difference between ARI scores of the control group with 5000 thermal cycles and group SEP with no thermal cycles ( $P < .003$ ). Also, a significant difference was found between group SEP with no thermal cycles and with 5000 thermal cycles ( $P < .003$ ). ARI scores for all other group comparisons were not significantly different from each other.

**DISCUSSION**

The highest SBS values were obtained for the conventional method and SEP after 24 hours of water storage. These SBSs did not show a significant difference from each other. In previous studies, significant differences were not observed between the conventional method and SEP application.<sup>8-11</sup>

Laboratory tests are often used to evaluate the per-

**Table 2.** Mean shear bond strengths, standard deviations (SD), minimum (min) and maximum (max) values and Weibull parameters for each group (n = 25)<sup>a</sup>

Groups	Thermo-cycling	Mean	SD	Homogeneous Subsets*	Weibull Modulus	Correlation Coefficient	Weibull Analysis		
							Characteristic Bond Strengths (MPa)	Shear Stress at 5% Probability of Failure (MPa)	Shear Stress at 10% Probability of Failure (MPa)
Conventional method	0	18.08	1.57	A	13.37	0.975	18.77	15.03	15.87
	2000	17.14	3.17	A	5.65	0.982	18.54	10.96	12.45
	5000	16.70	3.97	AB	4.03	0.968	18.50	8.86	10.59
Self-etching primer	0	18.15	2.81	A	7.43	0.982	19.31	12.95	14.27
	2000	14.50	2.61	B	6.28	0.886	15.60	9.72	10.90
	5000	14.68	2.25	B	7.01	0.964	15.69	10.27	11.38

<sup>a</sup> Means for groups having the same letters show homogeneous subsets.

\*  $P < .05$ .

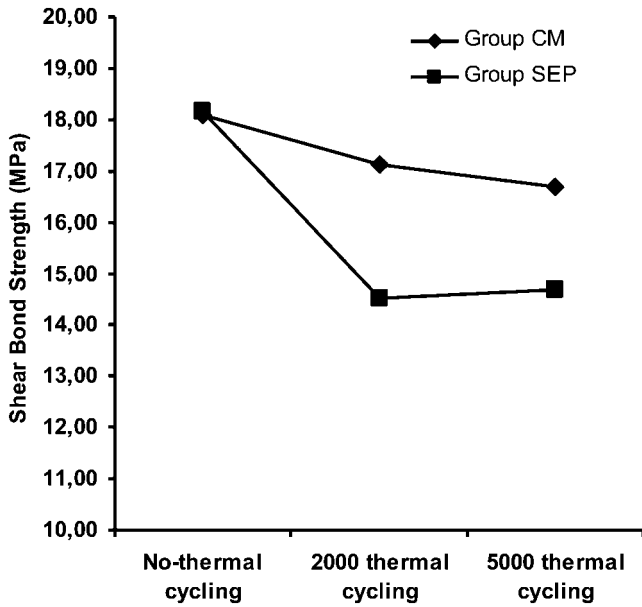


Figure 1. Mean shear bond strength (MPa) at 0, 2000, and 5000 thermal cycles for the control group and group SEP.

formance of bonding systems before proceeding with long-term clinical trials to determine the clinical efficacy of the bonding systems.<sup>20</sup> The thermocycling test is the in vitro process through which samples are subjected to temperature extremes that simulate conditions in the oral cavity.<sup>13</sup> There is no agreement and standardization between the various thermocycling studies.<sup>14</sup> Ozcan et al<sup>21</sup> stated that when no or limited thermocycling was performed, high bond strengths

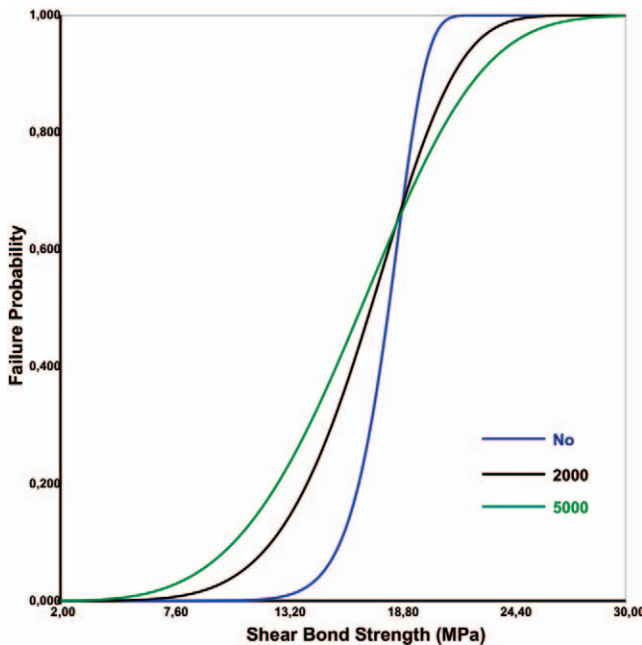


Figure 2. Cumulative failure probabilities versus shear bond strength for the control group at 0, 2000, and 5000 thermal cycles.

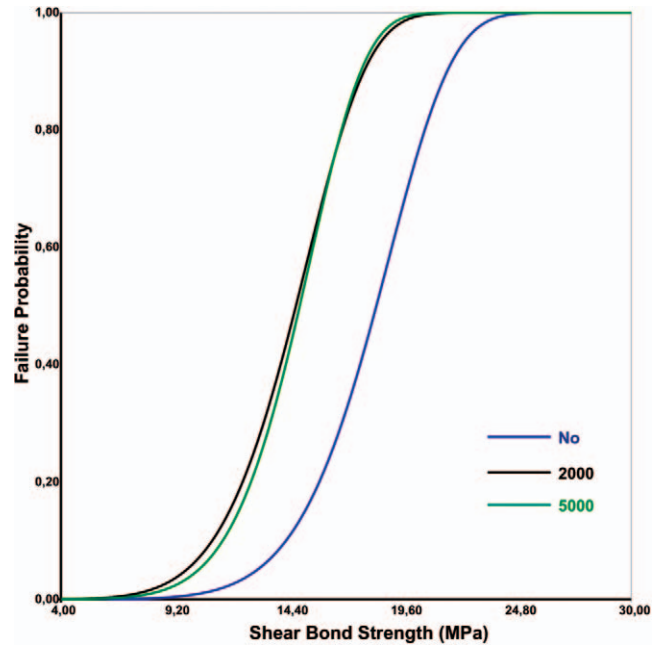


Figure 3. Cumulative failure probabilities versus shear bond strength for group SEP at 0, 2000, and 5000 thermal cycles.

can be found that do not correspond to chairside experiences. In the present study, 2000 and 5000 thermal cycles were chosen to evaluate the bonding durability of an SEP.

Bovine teeth were used because bovine teeth possess physical properties similar to human teeth and provide a more uniform sample.<sup>22,23</sup> The current assumption is that orthodontic brackets bonded to bovine enamel will perform in the same way as they do to human enamel.<sup>24</sup> Bond strength measurements for bovine teeth have been found to be lower than for human teeth.<sup>22,23</sup>

In the current study, the mean SBS values slightly decreased at 2000 and 5000 thermal cycles with the conventional method. These decreases were not statistically significant. However, Daub et al<sup>25</sup> reported that the mean SBS of metal brackets bonded with phosphoric acid and Transbond XT adhesive to human premolars decreased significantly by 16.7% after 500 thermal cycles. Saito et al<sup>3</sup> observed significant decreases in SBSs with phosphoric acid etching after 2000 and 5000 thermal cycles. The comparison of bond strength measurements of different studies is complicated because of the 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.<sup>24</sup> Fritz et al<sup>26</sup> suggested a separate control for each study because the SBS can differ significantly depending on the method applied.

After 2000 thermal cycles, a significant decrease of



**Table 3.** Frequency distribution of the Adhesive Remnant Index (ARI)<sup>a</sup>

Groups	Thermocycling	ARI Scores <sup>b</sup>							
		0	1	2	3	Median	Mean	SD	Range
Conventional method	0	5	9	10	1	1	1.28	0.84	0–3
	2000	10	4	9	2	1	1.12	1.05	0–3
	5000 <sup>c</sup>	17	1	5	2	0	0.68	1.07	0–3
Self-etching primer	0 <sup>c,d</sup>	1	5	19	—	2	1.72	0.54	0–2
	2000	3	12	9	1	1	1.32	0.75	0–3
	5000 <sup>d</sup>	15	2	8	—	0	0.72	0.94	0–2

<sup>a</sup>  $\chi^2 = 22.820$ ,  $P = .000$ .

<sup>b</sup> ARI scores: 0, no composite left on enamel surface; 1, less than half of composite left; 2, more than half of composite left; and 3, all composite left.

<sup>c</sup> A significant difference was observed between groups ( $P < .003$ ).

<sup>d</sup> A significant difference was observed between groups ( $P < .003$ ).

the mean SBS value was observed for group SEP. This mean SBS value remained almost unchanged after 5000 thermal cycles. A significant difference was not observed between these two values. Nevertheless, SBSs for 2000 and 5000 thermal cycles presented a significant difference from SBS obtained for 0 thermal cycles. Saito et al<sup>3</sup> reported that Megabond self-etching primer maintained the initial bond strength after thermocycling.

During the thermocycling test the samples are subjected to thermal changes and additional water exposure. Because of the differences in the coefficient of thermal expansion among the metal bracket, adhesive and tooth, repetitive contraction/expansion stresses are generated. These stresses may affect the adhesion of the resin to the bracket and the tooth and may result in bond failure.<sup>13,14,27</sup> The decrease in bond strength after thermocycling can also be attributed to increased water absorption or solubility of the composite, or both.<sup>25</sup>

The simultaneous etching and priming facilitates the penetration of the primer for the entire depth of the etched enamel.<sup>28</sup> This penetration of acidic primer into etched enamel creates resin tags. Scanning electron microscope evaluations revealed a more conservative etch pattern and a lower adhesive penetration with SEP (Transbond Plus) than with 37% phosphoric acid.<sup>6</sup> With SEP a less pronounced etching of the surface enamel was obtained, and bonding resulted in smaller and fewer resin tags.<sup>29</sup> Although thermocycling affects the mechanical properties of the adhesive for both groups, the significant decrease of SBSs for 2000 and 5000 thermal cycles with the SEP can be explained with the conservative etch pattern and a lower adhesive penetration.

Brackets are subjected to either tensile, shear, or torsion forces or a combination of these forces, which are difficult to measure.<sup>30</sup> It was reported that clinically adequate tensile bond strengths for metal orthodontic brackets to enamel should range from 6 to 8 MPa.<sup>31</sup>

Although these values were suggested as adequate bond strength values for most clinical orthodontic needs, the minimum clinically acceptable SBS is not known. In the present study, the SBSs were above these optimal values for all groups.

The Weibull analysis gives information about the probability of bracket failure and gives the clinician an indication of how the material is likely to perform in a clinical situation.<sup>24</sup> Even for materials with a high mean bond strength value, there is a finite measurable probability of failure occurring at relatively low stress levels.<sup>32</sup> Littlewood et al<sup>33</sup> suggested using the 5% chance of failure as a more appropriate level to assess bond strength. According to Littlewood et al<sup>33</sup> the bond strength of a material with a 5% chance of failure should be at least 5.4 MPa. In the present study, SBSs showed shear stress levels higher than 5.4 MPa at a 5% probability of failure for all groups. This indicates that using an SEP (Transbond Plus) with a light-cure adhesive (Transbond XT) may produce clinically acceptable SBSs after 2000 and 5000 thermal cycles.

For the control group and group SEP with 5000 thermal cycles, ARI scores were significantly lower than for group SEP with no thermal cycles, indicating that 5000 thermocycling resulted in an adhesive failure between the adhesive and the enamel surface. When brackets fail at the enamel/adhesive interface the enamel surface can be damaged.<sup>15</sup> Saito et al<sup>3</sup> observed enamel fracture with both phosphoric acid etching and self-etching primer treatment on thermocycled and nonthermocycled samples. In the present study enamel fracture was not observed although the SBSs observed in all groups were higher than clinically adequate tensile bond strengths (6–8 MPa).

## CONCLUSIONS

- Thermal stresses significantly reduced the mean bond strength of the self-etching primer (Transbond Plus).

- The self-etching primer (Transbond Plus) provided clinically acceptable bond strength values compared with the conventional method after 2000 and 5000 thermal cycles.

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