Original Article

Saliva Contamination Effect on Shear Bond Strength of Self-etching Primer with Different Debond Times

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ABSTRACT

Objective: To evaluate shear bond strengths (SBSs) of a self-etching primer (SEP) following saliva contamination at different stages of bonding at debond times of 5, 15, and 30 minutes and 24 hours.

Materials and Methods: Two-hundred forty human premolars were divided into four groups: group 1, uncontaminated; group 2, saliva contamination after priming; group 3, saliva contamination before priming; and group 4, saliva contamination before and after priming. Four subgroups according to debond times of 5, 15, 30 minutes and 24 hours were composed. Metal brackets were bonded with an SEP (Transbond Plus) and light-cure adhesives paste (Transbond XT). SBS values and the adhesive remnants were determined.

Results: The highest SBS was obtained at a debond time of 24 hours for the control group. This was significantly different from the other groups. SBSs at 5, 15, and 30 minutes showed no significant difference from each other in the control group ($P > .05$). Lowest SBSs were obtained at a debond time of 5 minutes for groups 1, 2, 3, and 4 (8.38, 7.10, 7.06, and 6.26 MPa, respectively) and were not significantly different from each other ($P > .05$). SBSs at 24 hours were not significantly different from each other for groups 2, 3, and 4 ($P > .05$). Significant differences were found in the adhesive remnant ($P < .001$).

Conclusions: SEP (Transbond Plus) may produce clinically acceptable bracket bonding after 5, 15, and 30 minutes from time of placement on the teeth, even with light and heavy saliva contamination.

KEY WORDS: Self-etching primer; Saliva contamination; Debond time; Bond strength

INTRODUCTION

The conventional method for bonding orthodontic brackets to enamel surfaces necessitates three different agents: an enamel conditioner, a primer solution, and an adhesive resin. It is paramount to keep the tooth surface dry for clinically adequate bond strengths. Clinically adequate tensile bond strengths for metal orthodontic brackets to enamel should range from 6 to 8 MPa.[1]

Enamel surface contamination can occur at two critical stages of the bonding procedure: after the tooth surface has been etched and after the primer has been applied.[2] Saliva is reported to be the most frequently encountered contamination in the clinic.[3] Following saliva contamination, a biofilm forms over the etched enamel.[4] Thus, most of the pores become blocked, and the penetration of resin is altered, resulting in resin tags insufficient in number and length.[5]

The introduction of acid-etch primers, such as Transbond Plus Self Etching Primer (3M Unitek, Monrovia, Calif), has attracted considerable interest since they combine the etching and priming steps into one, eliminating the need for separate etching, rinsing, and drying. The active ingredient of the self-etching primer is a methacrylated phosphoric acid ester. Phosphoric acid and a methacrylate group are combined into a molecule that etches and primes simultaneously.[6] The manufacturer states that Transbond Plus Self Etching

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Primer (SEP) performs well in either a moist or dry environment.

SEP (Transbond Plus) has been tested on saliva-contaminated enamel, and adequate bond strength was reported.[7–9] When dry and saliva-contaminated enamel were compared, Zeppieri et al[7] did not observe any significant difference with contamination at different stages of bonding. However, Cacciafesta et al[8] and Rajagopal et al[9] found significant differences between dry and saliva-contaminated enamel when contamination occurred after priming. In these studies,[7–9] shear bond strengths (SBSs) were evaluated 24 hours after the bonding procedure.

Testing at 24 hours is usually preferred because it has been widely reported and permits comparison with other in vitro bond strength studies.[10] However, this time period of 24 hours does not reflect clinical orthodontics, in which the archwire is generally placed after bracket bonding.[11] The initial bond strength of brackets is highly important since most orthodontists place the archwire into the slot from 10 to 15 minutes after bonding.[12] In clinical orthodontic practice, there is no consensus about the minimum time required before loading the bracket.[13] Suitable time intervals ranging from 5 minutes to 30 minutes and to 24 hours have been suggested.[14–16]

The SBS of SEP (Transbond Plus) during the first 30 minutes was evaluated in a limited number of studies.[17–20] In these studies, SEP (Transbond Plus) was tested on uncontaminated enamel surfaces.

The aim of this study was to evaluate the effect of saliva contamination at different stages of the bonding procedure on the SBS of an SEP (Transbond Plus) at different debond times of 5, 15, and 30 minutes and 24 hours.

MATERIALS AND METHODS

Two-hundred forty human premolar teeth were stored in distilled water. The water was changed weekly to avoid bacterial growth. Selection criteria of the teeth were as follows: intact buccal enamel, the absence of pretreatment with chemical agents (such as hydrogen peroxide), and the absence of cracks and caries. Each tooth was embedded into a cold-cure acrylic resin (Orthocryl; Dentaurum, Ispringen, Germany) cylindrical block. A jig was used to align the buccal surface of each tooth parallel to the cylinder's base. The teeth were cleansed and polished with pumice and rubber prophylactic cups for 10 seconds. The sample was randomly segregated into four groups of 60 teeth each.

Brackets

Stainless steel premolar brackets (Gemini bracket; 3M Unitek) were used. The mean area of each bracket base was 10.61 mm^2 according to the manufacturer.

Bonding Procedure

Group 1 (control, dry). SEP 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 adhesive resin (Transbond XT Light Cure Adhesive Paste) was placed onto the bracket base, and the bracket was positioned on the enamel surface. Excess adhesive resin was removed with an explorer. Polymerization for a total of 20 seconds from two directions using a visible light-curing unit having an output power of 600 mW/cm^2 was performed.

Group 2 (saliva contamination after priming). SEP was applied as in group 1. The buccal surface was contaminated with one coat of fresh human saliva via a brush. The bracket was bonded as in group 1.

Group 3 (saliva contamination before priming). The enamel surface was contaminated with saliva as in group 2. SEP was applied, and the bracket was bonded as in group 1.

Group 4 (saliva contamination before and after priming). The enamel surface was contaminated with saliva as in group 2. SEP was applied as in group 1. Saliva contamination was repeated once more. The bracket was bonded as in group 1.

Saliva was collected from one of the researchers, who was instructed to brush her teeth and not to eat for 1 hour before saliva collection.

Debonding Procedure

Each group was divided into four subgroups (15 teeth) according to the debond time of 5, 15, 30 minutes and 24 hours. Two minutes after bonding, the specimens were stored in distilled water (37°C) to prevent dehydration.

For shear testing, each specimen was secured in the lower part of the machine so that the bracket base paralleled the direction of the shear force. The specimens were stressed in an occluso-gingival direction with a crosshead speed of 1 mm/min. The shear bond test was performed with a universal testing device (Lloyd LRX; Lloyd Instruments Ltd, Fareham, UK). 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).[21] The ARI scale has a range from 0 to 3, with 0 indicating no composite remaining on the enamel; 1, less than half of the composite remaining; 2, more than half of the composite

Table 1. Two-Way Analysis of Variance of Force (MPa) Required to Debond Metal Brackets From Teeth

Source of Variation	Sum of Squares	*df*	Mean Square	*F* Ratio	Significance
Debond time	787.538	3	262.513	82.125	.000
Saliva contamination	501.736	3	167.245	52.321	.000
Debond time × saliva contamination	265.683	9	29.520	9.235	.000
Error	716.016	224	3.197		
Corrected total	2270.973	239			

remaining; and 3, all composite remaining on the tooth surface.

Scanning Electron Microscope Evaluation

To evaluate the effect of saliva contamination on the enamel surface etched with SEP, four premolar teeth were prepared: dry, contamination before priming, contamination after priming, and contamination before and after priming. These enamel surfaces were examined under a field emission scanning electron microscope **(**SEM; JSM-6335F; Jeol, Tokyo, Japan) at 15.0 kV. The SEM photomicrographs were taken with 5000× magnification.

Statistical Analysis

Two-way analysis of variance was used to determine the significant differences among saliva contamination, debond times, and their interactions. All treatment combination means for bond strength values were compared using the Tukey multiple comparison test ($\alpha = .05$). The χ^2 test was also used to determine significant differences for the ARI scores among the groups ($P < .05$).

Table 2. Mean Shear Bond Strengths, Standard Deviations, and Minimum and Maximum Values for Each Group (n = 15)[a]

Debond Time	$\bar{x}$	SD	Min	Max	Homogeneous Subsets*
Group 1, control					
24 h	17.61	4.04	10.54	24.72	G
30 min	10.40	1.98	7.07	13.82	EF
15 min	9.91	1.22	8.33	12.21	CDEF
5 min	8.38	1.59	6.11	10.94	ABCDE
Group 2, saliva contamination after priming					
24 h	10.94	2.05	7.99	14.96	F
30 min	8.70	1.39	6.27	10.68	BCDEF
15 min	8.62	1.71	6.26	11.82	BCDE
5 min	7.10	1.53	4.37	9.89	AB
Group 3, saliva contamination before priming					
24 h	10.05	1.96	5.42	12.70	DEF
30 min	8.29	1.23	6.36	11.38	ABCDE
15 min	7.89	1.32	5.75	9.64	ABC
5 min	7.06	1.39	3.71	8.82	AB
Group 4, saliva contamination before and after priming					
24 h	9.79	1.60	6.81	11.98	CDEF
30 min	7.79	0.75	6.15	9.00	ABC
15 min	7.33	1.15	5.29	8.97	AB
5 min	6.26	1.31	3.27	9.30	A

[a] Means for groups having the same letters show homogeneous subsets.

* Significance level $P < .05$.

RESULTS

The two-way analysis of variance showed a significant difference for saliva contamination, debond time, and interaction between saliva contamination and debond time on bond strength values ($P < .001$; Table 1). Mean SBS, minimum and maximum values, and standard deviations for each group are given in Table 2. For each group, the mean SBSs are shown in Figure 1. The analysis of the results of the Tukey multiple comparison test for the mean SBS values are given in Table 2.

In the control group, the highest SBS was at a debond time of 24 hours (17.61 MPa), and this value was significantly different from the values obtained at 30, 15, and 5 minutes (10.40, 9.91, and 8.38 MPa, respectively). In this group, SBSs at 30, 15, and 5 minutes showed no significant difference from each other.

For group 2 (saliva contamination after priming), group 3 (saliva contamination before priming), and group 4 (saliva contamination before and after priming), the SBSs at 24 hours (10.94, 10.05, and 9.79

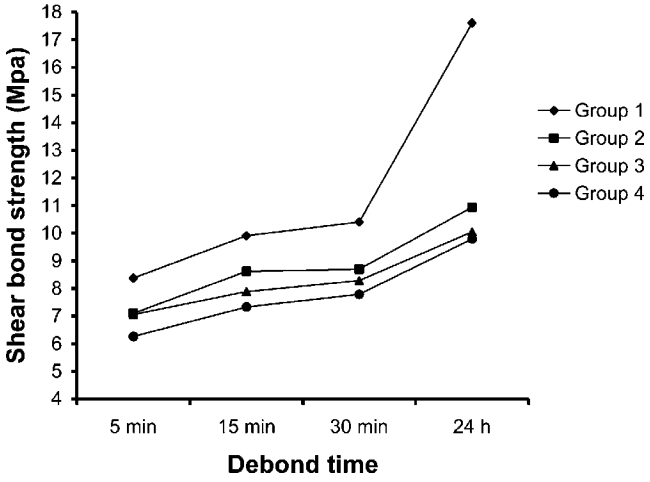

Figure 1. Mean shear bond strength (MPa) at 5, 15, 30 minutes and 24 hours for each group.

Table 3. Frequency Distribution and the Result of the χ^2 Analysis of the Adhesive Remnant Index (ARI)[a]

Debond Time	ARI Scores[b]			
	0	1	2	3
Group 1, control				
24 h	—	5	5	5
30 min	—	3	5	7
15 min	—	7	6	2
5 min	—	3	6	6
Group 2, saliva contamination after priming				
24 h	—	10	3	2
30 min	—	7	7	1
15 min	—	10	4	1
5 min	—	8	4	3
Group 3, saliva contamination before priming				
24 h	2	11	1	1
30 min	3	8	1	3
15 min	2	8	4	1
5 min	4	9	2	0
Group 4, saliva contamination before and after priming				
24 h	4	8	2	1
30 min	3	8	2	2
15 min	4	7	2	2
5 min	6	6	1	2

[a] $\chi^2 = 84.901$, $P = .000$.

[b] ARI scores: 0 indicates 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.

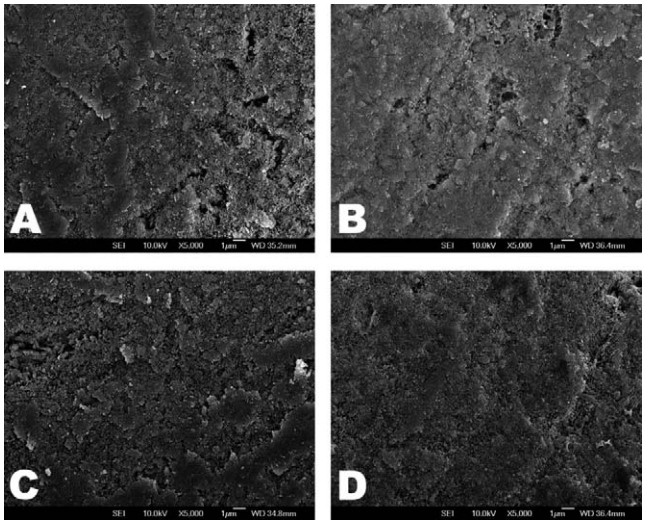

Figure 2. Scanning electron microscope photomicrographs of enamel surface. (A) Dry condition. (B) Saliva contamination after priming. (C) Saliva contamination before priming. (D) Saliva contamination before and after priming. Original magnification 5000×.

MPa, respectively) were significantly higher than those at 15 and 5 minutes. In each of these three groups, SBSs at 30, 15, and 5 minutes showed no significant difference from each other.

When comparing the debond time (5, 15, and 30 minutes and 24 hours), the SBS did not show a significant difference among groups 2, 3, and 4 at 24 hours. The highest SBS was obtained for group 1 at 24 hours, and this value was significantly different from the SBS values of the other groups.

The SBSs at 30 minutes for groups 1, 2, and 3 did not show any significant difference from each other. A significant difference was observed between groups 1 and 4. Moreover, the same findings were found for the SBSs at 15 minutes. However, the SBSs at 5 minutes for groups 1, 2, 3, and 4 did not show a significant difference from each other.

Frequency distribution and the result of the χ^2 analysis of the ARI scores are given in Table 3. The result of the χ^2 comparisons indicated that there was a significant difference ($P < .001$) for the groups. In group 1, a comparable distribution of the ARI scores of 1, 2, and 3 was observed for all debond times. An ARI score of 0 was not observed for group 1. Under saliva contamination, a higher frequency of the ARI scores of 1 was noted. In group 2, ARI scores of 0 were present, whereas in groups 3 and 4, ARI scores of 0 were recorded.

The SEM photomicrographs of the enamel surfaces are presented in Figure 2. Saliva contamination, before and after priming (D), presents a homogeneous appearance.

DISCUSSION

In the control group, the mean SBS at 24 hours (17.61 MPa) was higher than the values of the other debond times (5, 15, and 30 minutes). This outcome seemed inevitable since 24 hours has been stated to be the time when the composite reaches its maximum strength.[22] Our results corroborate this statement. SBS values during the first 30 minutes (5, 15, and 30 minutes) were not significantly different from each other. The mean SBSs at 5 (8.38 MPa) and at 15 (9.91 MPa) minutes are in agreement with the results (8.8 MPa and 11.0 MPa, respectively) of Movahhed et al[19] and Turk et al[20] (8.97 and 10.61 MPa, respectively). The SBS at 30 minutes (10.40 MPa) is comparable with the result (9.4 MPa) of Bishara et al[18] and Turk et al[20] (10.15 MPa).

In the present study, a gradual increase in the SBSs was observed from 5 minutes to 24 hours for all groups. Movahhed et al[19] observed an increase in SBS with light-cured adhesive (Transbond XT in combination with Transbond Plus SEP) at 5 and 15 minutes. With the same bonding system, Turk et al[20] reported an increase of SBS values from 5 minutes to 24 hours. Although an increase of SBSs was observed with an increase of debonding time for all groups, significant differences were not observed among the SBS

values obtained at 30, 15, and 5 minutes for each group.

During the bonding procedure, surface contamination can occur at two critical stages: after the tooth surface has been etched and after the primer has been applied. Therefore, bonding can be compromised at these stages.[2] Since these two different stages of contamination seem relevant in a clinical setting, saliva contamination before or after SEP application (ie, light contamination) and saliva contamination before and after primer application (ie, heavy saliva contamination) were simulated in the present study.

Under saliva contamination (groups 2, 3, and 4), the highest SBSs, demonstrating no significant difference from each other, were observed at 24 hours. Nevertheless, these SBS values were significantly different from the SBS value obtained with a dry enamel surface (group 1) at 24 hours. Cacciafesta et al[8] reported significant differences between dry (12.29 MPa) and saliva-contaminated enamel, that is, saliva contamination after priming (7.25 MPa) and saliva contamination before and after priming (7.70 MPa). Rajagopal et al[9] observed a significant difference between the median bond strength of dry (11.10 MPa) and saliva-contaminated enamel (10.79 MPa) with contamination after priming. These findings are in agreement with our results.

When the dry condition and light saliva contamination (ie, before or after priming) were compared with each other, no significant differences were observed for SBSs at 30 minutes. SBSs at 30 minutes with heavy saliva contamination (ie, before and after priming) were significantly different from the dry condition. The same findings were received for the SBSs at 15 minutes. The difference between SBSs under the dry condition and heavy saliva contamination might be attributed to the effect of saliva contamination on the etching pattern. With heavy saliva contamination, the primer becomes diluted, and the penetration of the primer might be decreased. SEM photomicrographs revealed a homogeneous appearance with heavy saliva contamination. It was reported that SEP (Transbond Plus) generates a more conservative etch pattern and a lower adhesive penetration than 37% phosphoric acid even under a dry condition.[23] However, SBSs at 5 minutes did not show any significant difference among the dry condition and all saliva contamination groups. In all groups, the lowest SBS values were perceived at 5 minutes. This might be explained by the early debond time rather than the effects of saliva contamination.

A comparable distribution of the ARI scores of 1, 2, and 3 was observed for the control group during the first 30 minutes (5, 15, and 30 minutes). Thus, bond failure was within the adhesive or at the adhesive-bracket interface. With saliva contamination, a higher frequency of the ARI scores of 1 was recorded. In group 2, ARI scores of 0 were not observed, whereas groups 3 and 4 showed ARI scores of 0. This indicated a bond failure at the enamel-adhesive interface. This type of bond failure demonstrated that the bond strength between the adhesive and the bracket and the cohesive strength of the adhesive were stronger than the bond strength between the adhesive and the enamel. In the studies[7–9] investigating the effect of saliva contamination on the bond strength of SEP, significant differences between the ARI scores of dry and saliva-contaminated enamel were not observed.

Brackets are subject to either tensile, shear, or torsion forces or a combination of these forces, which are difficult to measure.[19] It was reported that clinically adequate tensile bond strengths for metal orthodontic brackets to enamel should range from 6 to 8 MPa.[1] 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 during the first 30 minutes (5, 15, and 30 minutes) were above these optimal values for all groups. Since most orthodontists place the archwire into the slot from 10 to 15 minutes after bonding, the initial bond strength of brackets is highly important.[12] The current study indicates that using an SEP (Transbond Plus) with a light-cure adhesive (Transbond XT) may produce clinically acceptable bracket bonding, even after 5 minutes from time of placement on the teeth, with saliva contamination. However, as with any in vitro study, discretion should be exercised when attempting to extrapolate laboratory findings to the clinical setting.

CONCLUSION

- SEP (Transbond Plus) may produce clinically acceptable bracket bonding, after 5, 15, and 30 minutes from time of placement on the teeth, even with light and heavy saliva contamination.

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