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Artificial Saliva Contamination Effects on Bond Strength of Self-etching Primers

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ABSTRACT

Objective: To test the hypothesis that there is no difference in the bond strength with or without contamination with artificial saliva when using two different self-etching primers (Transbond Plus and iBond) in comparison with a conventional acid-etching method (37% phosphoric acid and Transbond XT) for bonding of orthodontic brackets.

Materials and Methods: One hundred fifty extracted human premolars were randomly allocated to six different groups, with 25 teeth in each group. Orthodontic metal brackets (APC II, Victory Twin 22 UNIV) were used. For contamination, a saliva replacement (Ptyalin) was applied. After contamination the surface was air-dried for 5 seconds and the bonding procedure continued. The bonded teeth were stored in deionized water at 37°C for 30 days and then thermocycled for 24 hours before debonding with a universal testing machine. The load was recorded at bond failure. The location of adhesive failure was determined under magnification using the adhesive remnant index (ARI).

Results: Clinically acceptable bond strengths were found for all primers used in this study. The contamination by saliva significantly decreased the bond strength when using the conventional acid-etching method (t = 0.0001). Self-etching primers were less influenced by saliva contamination. There was no significant difference in the ARI score among the groups (P > .05).

Conclusions: Saliva contamination significantly decreased the bond strength when the conventional acid-etching method was used. The self-etching primers were influenced the least. The bond strengths achieved for the self-etching primers and the conventional etching method after saliva contamination were not significantly different.

KEY WORDS: Self-etching primer, Saliva contamination, Bond strength.

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INTRODUCTION Return to TOC

The use of self-etching primers has increased for bonding of orthodontic brackets. Their quick and simplified technique has become very popular. Furthermore, the reduced enamel dissolution and therefore the reduced enamel loss, as a result of the shallower etch pattern, ^{1,2} indicates an essential benefit of this procedure.

The efficacy of using a self-etching primer on behalf of the bond strengths has been shown in various studies.^{2–16} Although some of these investigations were carried out using bovine teeth, ^{2,7,11,12} they can, due to their morphologic similarity be extrapolated to human teeth.¹⁷ The required clinically acceptable bond strength of 6–8 MPa¹⁸ was achieved in the majority of the available studies. The mean bond strength was, however, sometimes significantly less than that of the conventional acid-etching method.^{2,4,6,7,15,16}

Clinical conditions during bonding procedure include a risk of contamination of the etched surface by saliva. Saliva contamination of the enamel surface is regarded as the most common reason for bond failure.¹⁹ It has been reported that when using phosphoric acid etching, contamination with saliva causes a noticeable decrease in bond strengths.^{6.14} Self-etching primers are considered bicomponent hydrophilic adhesives²⁰ and are known to be the least influenced by the presence of moisture. Recent investigations comparing bond strengths of self-etching primers with and without saliva contamination showed no significant decrease in bond strength.^{6.7.12}

Nevertheless, the effect of saliva contamination on bond strength when using self-etching primers has been described controversially. The shear-peel bond strengths of self-etching primers have also been mentioned to be significantly decreased by saliva contamination.¹⁴

The effect of self-etching primers on the tooth surface after debonding is also of great interest. After bracket removal the enamel surface should remain unaffected with as little as possible residual adhesive on it. The removal of residual adhesive will lead to enamel loss, the degree of which is dependent on the type of clean-up, and to an increase of treatment time. ^{21.22} However, when the locus of bond failure is located macroscopically at the adhesive-enamel interface, some enamel loss has been shown. ²³ Regardless of this it has been concluded that most surface loss occurs during enamel clean-up.¹

Recently, Vicente et al²⁴ and other authors^{1.9} reported that after using the conventional acid-etching technique (etch and rinse) more adhesive remained on the enamel surface after debonding than after the use of a self-etching primer. A low frequency of adhesive failure between the bracket and adhesive has been shown, whereas more adhesive failures at the adhesive-enamel interface were reported in studies using self-etching primers. ^{4,20,25} No significant differences in debond location were found when comparing saliva contaminated teeth and noncontaminated teeth, which had been bonded with a self-etching primer. This was independent of whether the contamination was carried out before or after priming.⁷

Even if it has been maintained that bond failure at the bracket-adhesive interface or within the adhesive is safer than failure in the adhesive-enamel interface due to enamel cracking,²⁶ phosphoric acid techniques are reported to be associated with a risk of enamel cracks during debonding.⁶ It has been concluded that phosphoric acid etching produces more enamel fractures than self-etching primer treatment.⁶ This might be a possible result of the reduced depth of demineralization of self-etching primers.

The aim of this in vitro study was to determine the influence of saliva contamination on bond strength and to determine the location of adhesive failure when using two different selfetching primers compared with a conventional acid-etching method for bonding of orthodontic brackets.

MATERIALS AND METHODS Return to TOC

Freshly extracted human premolars, stored according to international standards (ISO 11405/2003) for testing of adhesion to tooth structure, were used. Teeth with microscopically detected cracked surfaces, restorations and caries were excluded. One hundred fifty teeth satisfied the inclusion criteria and were randomly allocated to six different groups, with 25 teeth in each group. Two self-etching primers (Transbond Plus and iBond) and one conventional etch and rinse system (37% phosphoric acid liquid and Transbond XT Primer) were tested with and without saliva contamination. The roots of the teeth were cut off with a water-cooled low-speed diamond saw. All teeth were cleaned with fluoride- and oil-free pumice for 10 seconds, rinsed and dried with oil- and moisture-free compressed air, each for 10 seconds further. Orthodontic precoated stainless steel brackets (Victory Twin 22 UNIV UBI, APC II, 3M Unitek, Monrovia, Calif) were bonded (average determined surface area = 13.96 mm²). Thus, a light-polymerized composite resin applied by the manufacturer at the bracket base (APC II) was used.

Conventional etching was performed by using 37% phosphoric acid liquid for 30 seconds followed by rinsing with water for 20 seconds and drying with oil- and moisture-free compressed air for 10 seconds. Afterwards, a thin uniform coating of Transbond XT Primer was applied to the etched enamel. Transbond Plus was rubbed for 5 seconds on the enamel and then evaporated gently for 2 seconds. iBond, which belongs to the seventh generation of self-etching bonding primarily used in conservative dentistry, was applied as recommended by the manufacturer.

For contamination, a saliva replacement spray (Ptyalin neutral, TMP Tüshaus, Velen-Ramsdorf, Germany) was used. After contamination for 10 seconds the surface was air-dried for 5 seconds and the bonding procedure was continued. The contaminated teeth were not re-treated with the self-etching primer. If the conventional etching method was used, the primer was applied afterwards. Each precoated bracket was bonded by one experienced operator using 300 g of force applied with a Correx gauge (Haag-Streit, Bern, Switzerland) for 3 seconds in order to achieve a comparable resin layer thickness. Excessive resin was carefully removed, and light curing followed for 20 seconds (10 seconds mesially and 10 seconds distally on each bracket) with a light-emitting diode (LED) curing light unit (Ortholux LED Curing Light, 3M Unitek) at a distance of 3 mm and an angle of 45° to the surface. The required irradiance was controlled by measuring with a radiometer (Model 100, Demetron, Dansbury, Conn) before each curing.

Rectangular stainless steel wires (0.017 × 0.022 inch, 3M Unitek) were inserted and ligated to the brackets prior to embedding the crown with acrylic resin (Technovit 4004, Heraeus Kulzer, Wehrheim, Germany) in fabricated metal rings. This allowed the same horizontal orientation of the nonembedded buccal surface where the brackets were bonded. The bonded teeth were stored for 30 days in deionized water at 37°C and thermocycled (Willytec, Dental Research Division, Munich, Germany) at 5°C and 55°C (dwell time: 30 seconds; transfer time: 5 seconds) for 1300 cycles before debonding. The archwire segments acted as a guide for placing the brackets parallel to the shear force direction and were useful to minimize deformation of the brackets during debonding.

The brackets were debonded using a shear-peel load (nonvarying distance of 1.5 mm from the bracket base) on a universal testing machine (MCE 2000ST, quickTest, Langenfeld, Germany) at a crosshead speed of 0.5 mm/min (ISO 11405). The load was recorded at bond failure and used to calculate the bond strength (1 MPa = 1 N/mm²). The location of adhesive failure was determined under magnification using the modified adhesive remnant index (ARI), which includes scores from 0 to 3. In addition, it was possible to include enamel fractures by scoring them with a 4.²⁷

The data were analyzed with the statistical software program SPSS 12.0 (SPSS Inc, Chicago, III). First, all data were tested for normal distribution (Kolmogorov-Smirnov test) and variance homogeneity (Levene test). Since the shear-peel bond strength fulfilled the criteria, statistical analyses were conducted by one-way and two-way analysis of variance (ANOVA) with post hoc Tukey's test and Student's *t*-test. Additionally, a Weibull analysis was performed. The Kruskal-Wallis one-way ANOVA was used to determine significant differences in the ARI scores between the groups, since these data were not normally distributed. Significance for all statistical tests was predetermined at *P* < .05.

RESULTS <u>Return to TOC</u>

The results of the shear-peel bond strength measurements are shown in Table 1 \bigcirc . ANOVA indicated significant differences between the groups (P < .001). The post hoc test revealed significant differences (P < .05) for nearly all test groups when compared with the values achieved with uncontaminated Transbond XT as the gold standard. However, Transbond Plus was an exception when saliva contamination was present. No significant differences were found by its comparison with the values of all other groups (P > .05). The contaminated Transbond Plus group was therefore characterized by two superscript letters in Table 1 \bigcirc , which means that it belongs to both statistically generated homogenous subgroups (a and b). The bond strengths achieved for the self-etching primers and the conventional etching method after saliva contamination were not significantly different (P > .05).

By using the Student's *t*-test, Transbond Plus showed significantly lower mean shear-peel bond strength (t = .006) without saliva contamination. For Transbond XT, contamination decreased the shear-peel bond strength significantly (t = .0001). For the self-etching primer i-Bond, no significant differences were found according to the shear-peel bond strengths with or without saliva contamination (t = .783).

Within the Weibull statistics, higher values for characteristic strength (σ_0) and especially for Weibull modulus (*m*), which characterizes the scatter in strength, are preferred. The σ_0 corresponds to a probability of failure *F* = 63.2%. The higher the *m* is, the more reliable the tested adhesive system. However, the shear-peel bond strengths of Transbond Plus under both conditions (contaminated and noncontaminated by saliva) as well as that of Transbond XT under dry conditions were the most reliable. By using the two-way ANOVA the saliva contamination showed no significant effect (*P* = .254) on bond strength. The material used for enamel treatment showed a minor significant effect (*P* = .047), whereas the interaction of these variables was highly significant (*P* = .001).

There was no significant difference in the ARI score among the groups (Kruskal-Wallis test, *P* >.05). The distribution (<u>Table 2</u> **O=**) showed that most of the samples (81.3%) had an ARI score of 1, which means that less than 50% of the adhesive usually remained on the enamel. There was no indication for unequal distribution of the observed enamel fractures (N = 11) with or without saliva contamination. A low frequency of adhesive failures (adhesive-enamel interface) (4.7%) was found within all groups without evidence of a distinct distribution.

DISCUSSION Return to TOC

Moisture contamination is still a problem during direct bonding of orthodontic brackets, especially while bonding posterior teeth as well as surgically exposed teeth.¹⁴ In agreement with previous studies, ^{2,4,6,7,15,16} the mean shear-peel bond strength values achieved without saliva contamination were significantly lower for the self-etching primers (iBond: mean 11.3 MPa; Transbond Plus: mean 10.6 MPa) in comparison to the conventional acid-etching method (mean 13.2 MPa). The achieved bond strengths of the self-etching primers used in this study were within the range of the values that have been described in the literature.^{16,20} Even after saliva contamination, the mean bond strengths remained within the upper range of these values (iBond: mean 11.1 MPa; Transbond Plus: mean 11.8 MPa).

Saliva contamination leads to a noticeable bond strength reduction when using phosphoric acid etching.^{6,14} In accordance, a significant decrease of the values was found for the conventional etch and rinse group. Saliva contamination did not cause a statistical significant decrease of bond strength when the self-etching primer iBond was tested. This is in agreement with the literature.^{6,7,12} Recently Cacciafesta et al^Z showed that after saliva contamination the bond strengths of a self-etching primer were, in comparison to those of a conventional primer, significantly higher. The authors concluded that the self-etching primer was the least influenced in terms of bond strength values. In agreement, we found that the

self-etching primers were less affected by artificial saliva contamination, and that the bond strengths for Transbond Plus were even higher. However, there was no statistical difference between the contaminated groups, including that of Transbond XT.

Factors that may influence the results of in vitro studies dealing with bond strength of orthodontic brackets, have been well described in the literature.²⁸ In order to standardize our methods, precoated brackets with the equal amount of composite resin (APC II) were used. Additionally, the brackets were bonded by one experienced operator, using the same force for an equal duration of time in order to achieve the exact same adhesive layer thickness as mentioned by many authors.^{8,29} With regard to the different loading rates, the international standards (ISO 11405/2003) for testing of the adhesion to tooth structures were used. High-velocity debonding forces that represent the velocity of tooth occlusion during mastication³⁰ are not in accordance with the complexity of clinical bracket failure, particularly with regard to the undesired contact of brackets during occlusion. However, loading of the bracket, not close to the base, has been claimed to be more representative for in vivo loading and ensures a more consistent application of debonding force.²⁵

The storage time and a supplementary interpolated thermocycling before bond-strength testing have been suggested as potential critical factors in evaluating the effectiveness of an orthodontic bonding adhesive. ^{20.31,32} In this study a 30-day storage period, which in prior published studies^{20.25} showed the lowest shear-peel bond strength values, was used. Since temperature change simulation is essential in order to achieve clinical relevance of in vitro investigations, an additional thermocycling for 24 hours was carried out.

The ARI scores, as already mentioned by other authors, 4.20.25 were not significantly different. No significant differences were also found in debond location with the self-etching primers on saliva contaminated and noncontaminated teeth. This is in accordance with a recent investigation.⁷ We found that no benefit exists regarding the residual adhesive on enamel after debonding by using self-etching primers. Despite their reduced depth of demineralization, nearly the same amount of enamel loss will occur due to the required clean-up, which in turn has been described to cause the most surface loss.¹ Another reason for losing enamel is the enamel cracking which is commonly described with the use of phosphoric acid techniques.⁶ In this study, 11 fractures were registered despite the strict inclusion criteria. There were no differences regarding their allocation in the groups tested.

Nevertheless, most samples presented in this study had ARI scores 0 and 1 (together 86%). The ARI score of 3 never appeared, which indicates a minimum amount of adhesive remaining on teeth. Clinically, this would imply a minimal clean-up time after debonding.

CONCLUSIONS Return to TOC

- Clinically acceptable bond strengths were found for all primers used in this study.
- Saliva contamination within the conventional acid-etching method significantly decreased the bond strength.
- Compared with the acid-etching method self-etching primers are less influenced by saliva contamination.
- The bond strengths achieved for the self-etching primers and the conventional etching method after saliva contamination were not significantly different.
- There was no significant difference in ARI scores among the groups.

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TABLES Return to TOC

Table 1. Descriptive Statistics and Results of the One-Way Analysis of Variance (ANOVA) With Post Hoc Tukey's Test, Student's t-test, and Weibull Analysis Comparing the Six Groups Tested

Artificial Saliva	Transbond Plus		Transbond XT		iBond	
	No	Yes	No	Yes	No	Yes
Ν	25	25	25	25	25	25
Bond strength (MPa)						
Mean	10.6ª	11.8 ^{ab}	1 3.2 [⊾]	11.1ª	11.3ª	11.1ª
SD	1.6	1.2	1.6	2.3	2.7	3.0
Median	10.6	11.4	13.3	11.6	11.5	10.6
P (ANOVA)	.001					
Weibull parameters						
σ_{o}	11.2	12.3	13.9	12.0	12.7	12.2
m	8.0	11.2	9.4	5.4	3.8	4.2
t (t-test)	.006		.0001		.783	

* Homogenous subgroups are identified by the same superscript letter. The increase in shear-peel bond strength for Transbond Plus after artificial saliva contamination is reflected in an increase of reliability which is expressed by the Weibull modulus (m). N indicates sample size; SD, standard deviation; σ_0 , characteristic strength; m, Weibull modulus.

Table 2. Distribution of the Modified Adhesive Remnant Index (ARI)^{32,a}

			ARI Score					
	Saliva		0	1	2	3	4	
Transbond Plus	No	Ν	2	19	3	0	1	
		%	1.3	12.7	2.0	0.0	0.7	
	Yes	Ν	0	21	1	0	3	
		%	0.0	14.0	0.7	0.0	2.0	
Transbond XT	No	Ν	0	22	3	0	0	
		%	0.0	14.7	2.0	0.0	0.0	
	Yes	Ν	3	19	1	0	2	
		%	2.0	12.7	0.7	0.0	1.3	
iBond	No	Ν	0	22	1	0	2	
		%	0.0	14.7	0.7	0.0	1.3	
	Yes	Ν	2	19	1	0	3	
		%	1.3	12.7	0.7	0.0	2.0	
Total		Ν	7	122	10	0	11	
		%	4.7	81.3	6.7	0.0	7.3	

^a N indicates sample size. ARI score key: 0, no adhesive on tooth;

1, <50% adhesive on tooth; 2, >50% adhesive on tooth; 3, 100%

adhesive on tooth; 4, enamel fracture.

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