

# Shear Bond Strength of Saliva Contaminated and Re-etched All-in-One Adhesive to Enamel

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## Abstract:

**Objective:** The aim of this study was to investigate the effect of phosphoric acid re-etching of an enamel surface treated via a one-bottle adhesive system on shear bond strength between resin composite and the enamel surface in different stages of adhesive application.

**Materials and Methods:** Extracted intact premolars (n=84) were divided into seven groups (n=12). In the control group 1, the adhesive i-Bond was used according to the manufacturer's instructions, with no contamination. In groups 2 to 4, the conditioned and saliva, contaminated enamel was blot dried only, rinsed, and blot dried, rinsed blot dried and re-etched, respectively. In groups 5, 6 and 7 cured adhesive was contaminated with saliva and then rinsed and blot-dried, blot dried only and rinsed, blot-dried and re-etched respectively. In groups 3, 4, 6 and 7 the adhesive was reapplied. Afterward, Z100 composite cylinders were bonded to the enamel surfaces. The samples were thermocycled (5°C and 55°C, 30 s, dwelling time: 10 s, 500 cycles). Finally, the samples were sheared using Dartec testing machine and shear bond strength data were subjected to one-way ANOVA analysis and Tukey's HSD test.

**Results:** There were statistically significant differences among groups 1 and 5-7. The samples in groups 1 and 4 demonstrated higher bond strengths than those in the other groups.

**Conclusion:** Using phosphoric acid etching may be effective, only where contamination occurs prior to curing of the adhesive. After curing of the adhesive, none of the methods in this study would be preferred.

**Key Words:** Adhesives; Dental Bonding; Dental Enamel; Dental Etching; Saliva

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## INTRODUCTION

Protecting the restoration against contamination by oral fluids is still a necessity in most of the practiced clinical treatment methods [1].

All-in-one adhesives provide increased user reliability due to their faster application, reduced number of components and application steps. However, in order to achieve the best

results possible in bond strength, manufacturers recommend two, three, or more time application of these materials. Previously published studies on how saliva contamination affects shear bond strength (SBS) of different generations of bonding agents have resulted different conclusion [2-11]. There are studies proposing saliva contamination of etched enamel to cause

a significant decrease in bond strength between resin and the enamel surface [2,12]. Fritz et al [5] reported a 50% reduction in mean bond strength value when the composite resin was bonded directly to saliva contaminated enamel and dentin. Other studies reported the significantly lower bond strength of the composite resin bonded directly to contaminated enamel [12-14]. On the other hand, there are previously published data indicating that the use of dentin bonding agents tends to reduce tooth sensitivity to saliva contamination [2,9]. A reduction rate of 40-50% in bond strength to contaminated tooth when using self-etch adhesives have also been cited in the literature [15,16]. In addition, conclusion have been made that re-etching of the saliva-contaminated dentin with phosphoric acid is the best method to overcome the negative effects of saliva on bonding strength [8,12,16]. The aim of this study was to investigate the effect of re-etching the saliva contaminated, adhesive treated enamel surface by phosphoric acid on SBS of composite resin to the surface during different steps of the application of the one-bottle adhesive system of i-Bond.

## MATERIALS AND METHODS

Eighty-four extracted intact premolars were stored in thymol solution 0.2% for a maximum of 4 months until further processing in the laboratory. The teeth were mounted in cylindrical moulds with self-cure acrylic resin up to their cervical region. Buccal surfaces of the specimens were reduced with a 600-grit silicon carbide paper under water spray to create flat and fresh enamel surfaces. Afterward, the samples were randomly divided into seven

groups of 12 and 0.05 cc of fresh human saliva was applied on all of them by Hamilton syringe for 30 seconds.

The adhesive system used under several conditions was i-Bond (Heraeus-Kulzer Co., Germany, Lot No#010062). Four procedures (not contaminated, contaminated, contaminated and rinsed, contaminated, rinsed and re-etched) using i-Bond adhesive system before and after light curing were followed and evaluated.

The study groups were prepared as follows:

Group 1: In this group, application of i-Bond with a small-saturated brush in three consecutive coats was followed by 5 seconds of gentle air-drying, to remove the solvent and water, and 20 second light activation (Coltolux 2.5, C7906, Coltene, USA).

Group 2: After applying the adhesive and before curing it, fresh saliva was applied on the samples and left undisturbed for 30 seconds. After 5 seconds of gentle air blow, the adhesive was re-applied and cured.

Group 3: The procedure was similar to the one in group 2; however, after half a minute, the contaminating saliva was thoroughly rinsed for 15 seconds prior to blot drying and application of the adhesive.

Group 4: The procedure was similar to that in group 3, and after blot drying, H<sub>3</sub>PO<sub>4</sub> (37%, lot no. 80114, 3M ESPE, USA) was applied to the surface and rinsed, both for 15 seconds and blot dried before the adhesive was applied as described previously.

Groups 5-7: In these groups, saliva contamination occurred after curing of the adhesive.

In group 5, saliva was rinsed for 15 seconds prior to blot drying and resin composite application.

**Table 1.** Shear bond strength (MPa) on enamel by application technique.

SBS	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7
Minimum	15.004	10.757	10.191	16.277	10.333	6.228	8.634
Maximum	22.647	16.227	26.185	23.071	17.127	13.730	16.419
Mean	17.409	12.514	15.333	18.459	13.069	10.368	12.267
SD	2.122	1.728	5.668	2.039	2.153	2.832	3.019

SBS=Shear Bond Strength, SD=Standard Deviation

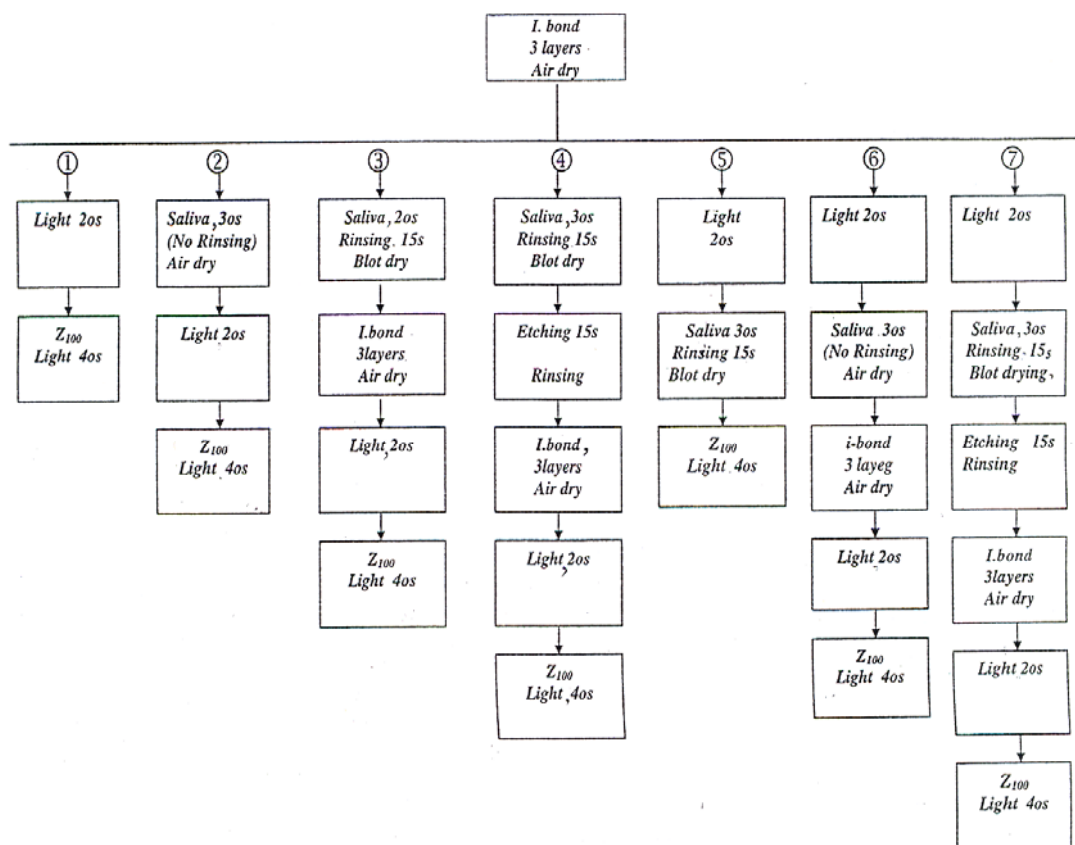


Fig 1. Application techniques used in the study.

In group 6, without rinsing the saliva, the samples were blot dried for five seconds and the composite resin was applied. In group 7, the treatment was similar to that in group 5, except that after blot drying, etching with 37% phosphoric acid was carried out and rinsed thoroughly for 15 seconds followed by i-Bond adhesive being reapplied and cured. In groups 5-7 the adhesive was reapplied before composite bonding. (Fig 1).

Teflon molds (3 mm width and 4 mm height) slightly overfilled with resin composite (Z100, lot No.8004 A3, 3M ESPE, USA), were firmly and carefully placed on enamel surfaces of all the specimens and light cured for 40 seconds from three sides of the cylinders.

The specimens were then stored in deionized water at 37°C for 24 hours and thermocycled for 500 rounds before being subjected to shear force in a universal testing machine (Dartec,

model HC10, UK) with the cross-head speed of 1 mm/min until occurrence of failure. SBS data were analyzed using one-way ANOVA followed by Tukey's HSD test to identify the differences at a significance level of 0.95.

## RESULTS

One-way ANOVA showed that surface salivary contamination causes statistically significant reduction in bond strength (Table 1).

The results revealed that there were significant differences among the groups ( $P < 0.0005$ ). According to Tukey's HSD test significant differences between groups 1 and 2, 1 and 5-7, 2 and 4, and 4 and 5-7 were found at a significance level of 0.05 (Table 2).

## DISCUSSION

The results of our study depicted statistically significant differences among groups 1 and 5-7

and the samples in groups 1 (control group) and 4 demonstrated higher bond strengths than those in the other groups.

Protein contamination of the operating field from accidental contact with either blood or saliva is a frequent problem in dentistry. In addition, the application of rubber dam is sometimes difficult or even impossible, e.g., when deep cervical lesions are restored or indirect restorations are seated. Thus, resin adhesives bonding effectively to dental substrates in spite of protein contamination, would be highly desirable [2-6]. Although in all-in-one adhesive systems all three basic steps of etching, priming, and applying the adhesive occur simultaneously, they should mostly be applied in two or more layers to obtain enough thickness and adequate bond [2,3].

In addition, in these all-in-one systems, the primer does not need to be rinsed off after application. What happens to the acid is still not completely understood, but it is thought to be neutralized at some point by hydroxyl ions released from the hydroxyapatite during demineralization [13,14].

The effect of salivary contamination on bonding efficacy of adhesives, with respect to their hydrophilic nature, is yet greatly controversial [2-11,16]. Some studies have reported saliva contaminated and unwashed enamel to provide significantly lower bond strengths of resin composite to them. In addition, others have reported the use of bonding agents under fissure sealants reducing their sensitivity to saliva contamination and resulting in high bond strengths [3,5,9,11,12].

It seems that the hydrophilic nature of newer adhesive systems may bring about better behaviors in the presence of saliva contamination [2,5,9].

Measuring SBS values is a common method to evaluate the efficiency of bonding systems [17]. It has been previously depicted that if SBS values of composite to dentin and enamel range between 15-35 MPa, the system will be clinically acceptable [13]. In this regard, groups 1, 3, and 4 in the present study showed acceptable values of SBS. In addition, it has been stated that SBS values of 17-20 MPa can compensate the polymerization shrinkage, thus, in our study only the treatment in group 4, has resulted into a reliable bond. According to some researches, other procedures such as resurfacing with burs, especially after curing of the adhesive, might be valuable [5].

In the present study, we used i-Bond being an all-in-one adhesive system. Its main components are 4-META and diurethandimethacrylate, HEMA and Glutaraldehyde. This system includes acetone and water as solvents. The recommended time for applying and curing of the three layers of the mentioned system, as instructed by the manufacturer, is about 75 seconds; therefore, contamination during application periods is highly probable.

In the present study, comparison of the contaminated samples after application of the adhesive and before curing it, with the control group showed that SBS values in group 2 was significantly lower than the those in the other three groups. When contamination occurred before curing of the adhesive, no significant

**Table 2.** Two by two comparison of study groups.

	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7
Group 1	S	NS	NS	S	S	S
Group 2		NS	S	NS	NS	NS
Group 3			NS	NS	NS	NS
Group 4				S	S	S
Group 5					NS	NS
Group 6						NS

S=statistically significant, NS=Not statistically significant

difference was seen either when the contaminated enamel was rinsed or both rinsed and etched (groups 3 and 4). This shows that, when using i-Bond system, it is not necessary to etch the enamel again, thus, rinsing and blot drying of the contaminated enamel seems to be enough.

The specimens contaminated with saliva in group 2 had mean bond strength of 12.514 MPa, while the amount for the control group was 17.409 MPa, which shows a reduction of about 35%. Several researches have reported a 50% reduction in SBS value when the enamel is contaminated with saliva and not rinsed afterwards. On the other hand, it has been advocated that when saliva is applied on the one-bottle adhesive and air-dried either prior to or after curing, SBS will be reduced [5,7,9]. Taskonak and Sertgoz [4] have reached similar results; however, in the study by Fritz et al [5] the statement goes for dentin substrates only.

In our study, SBS values in group 4, where saliva contamination was rinsed off and phosphoric acid etching was applied, were more than other groups (mean: 18.459 MPa). There are previously published data confirming the mentioned result [12]. This shows that using H<sub>3</sub>PO<sub>4</sub> before curing the adhesive as a decontaminating agent can be effective.

The present study has shown that after curing the adhesive, salivary contamination cannot be completely removed by rinsing and drying (group 5). Comparison of the results of groups 5 and 6 shows the effect of rinsing as a saliva decontaminating procedure; though, it seems that after curing the adhesive, rinsing and drying does not fully recover the SBS.

Glycoproteins are reported to act as barriers. While rinsing and drying, the oxygen-inhibited layer is removed and as a result, its copolymerization with the next layer is impaired. In our study, we did not air dry the surface, but blot-dried it. That is why relatively good results were obtained in group 5, confirming that salivary proteins are rinsed off the contami-

nated surface. Yet, it should also be noticed that bonding systems are believed to be sensitive to excess moisture, artificial saliva, and plasma [3]. May be that is why we did not obtain better results for group 5.

In addition, saliva contamination of enamel after curing the adhesive, without additional rinsing (group 6), caused a dramatic reduction in SBS values. It is not surprising that adsorption of salivary glycoproteins generates a film on the hydrophobic surface of enamel after curing the adhesive [2-6]. Fritz et al [5] and el-Kalla and García-Godoy [9] have reported similar results on this matter.

Applying H<sub>3</sub>PO<sub>4</sub> and rinsing prior to re-application of the adhesive made a significant fall in SBS amounts in group 7. The mentioned procedure has not been used in previous studies. It seems that neither rinsing nor using H<sub>3</sub>PO<sub>4</sub> could recover the SBS after curing. Townsend and Dunn have also depicted similar results [2]. Rinsing and drying saliva contamination is proposed as an accepted treatment to recover SBS when the contamination occurs after curing the adhesive [3,5,7]. In addition, Fritz et al [5] suggested that the contaminated area must be removed by resurfacing the tooth with a bur, as it cannot be wetted properly by the composite material. The mentioned treatment was not performed in this study.

According to our findings the best way to decontaminate the surfaces before curing the adhesive (comparison of groups 2-4) was rinsing and blot drying followed by etching with phosphoric acid and reapplying the bonding agent on the basis of the manufacturer's instructions, as practiced in group 4. It appears that the use of phosphoric acid removes salivary contamination properly and makes the surface ready for a more reliable bond.

Nevertheless, when saliva contamination occurred after curing the adhesive, using phosphoric acid could not be suggested; probably, resurfacing the substrate with a bur would be

preferred then. The authors would suggest more studies in the field including those involving stereo electron microscope evaluations.

## CONCLUSION

Saliva contamination of uncured i-Bond adhesive system can simply be removed by the application of phosphoric acid, rinsing and reapplying of the system.

After curing the adhesive, none of the procedures practiced in this study would be preferred.

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