

# Evaluation of Different Enamel Surface Treatment on the Bracket Bonding

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## Abstract

**Objective:** To evaluate and compare the effects of different enamel conditioning techniques on the shear bond strength of brackets, the failure sites following debonding and the surface morphology of the enamel.

**Methods:** In this study, 72 human premolars were randomly divided into six groups of 12 specimens each. The enamel surfaces of the teeth were etched with 37% phosphoric acid in group PA, deproteinized using sodium hypochlorite and 37% phosphoric acid in group NaOCl + PA, sandblasted and etched with 37% phosphoric acid in group AA + PA, conditioned by self-etching primer in group TPSEP, deproteinized using sodium hypochlorite and conditioned by self-etching primer in group NaOCl + TPSEP, sandblasted and conditioned by self-etching primer in group AA + TPSEP. After enamel conditioning procedures, brackets were bonded to the treated enamel surface, and shear bonding test was performed. After debonding, ARI scores were calculated for all groups. Two samples from each group before bonding procedure were inspected by SEM for study the surface topography of enamel following different conditioning techniques. Data were subjected to analysis of One-way variance, Scheff post hoc, Chi-square and Spearman's tests.

**Results:** The mean bond strength values for groups NaOCl and PA have a higher mean shear bond strength among other groups while self-etching primer in group TPSEP had a lower shear bond strength. A significant difference was observed in the bond strengths between groups ( $p < 0.001$ ). However, insignificant difference found in ARI in all groups ( $P = 0.118$ ). SEM observation revealed different etching patterns on the enamel surface after pretreatment.

**Conclusions:** Enamel surface pretreatments, using a combination of sodium hypochlorite or sandblasting before phosphoric acid and self-etching primer, result in increased shear bond strength of orthodontic brackets as well as the surface roughness of enamel.

**Keywords:** Sandblasting, Sodium hypochlorite, Shear Bond strength, Electron microscope.

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## Introduction

Orthodontic treatment requires the movement of teeth through the application of force. This force is transferred to the teeth through brackets that bonded to them<sup>(1)</sup>. In clinical orthodontics, it is essential to obtain a satisfactory bond between orthodontic brackets and tooth enamel<sup>(2,3)</sup>. The strength of the bond between the bracket and enamel surface depends on three factors. Namely, the retention means of the bracket base, the adhesive material, and the tooth surface preparation<sup>(4)</sup>.

When bonding orthodontic brackets to enamel surface, the current technique involves three steps, application of phosphoric acid to approximately 15-60s then thoroughly washing and drying the enamel. This etching causes dissolution of prismatic and inter-prismatic material in the enamel-producing an irregular enamel surface facilitating the retention of an orthodontic attachment via its bonding agent<sup>(5)</sup>.

In past decades, the desire to reduce the steps (etching, rinsing, drying and priming), technical sensitivity and disadvantage associated with conventional acid etching technique, led to development of self-etching primer<sup>(6)</sup>.

One of the most common problems facing clinicians is bracket detachment during active orthodontic treatment that poses a serious problem for orthodontist<sup>(7)</sup>. Different enamel surface preparation methods have been proposed for strengthening orthodontic brackets bonding. These include; application of physical abrasive methods such as air abrasion utilizes Aluminum-oxide particles and chemical conditioning methods like the application of sodium hypochlorite<sup>(8-11)</sup>. Therefore, the aim of the present study is to examine the effect of different enamel surface preparation methods on SBS and ARI.

## Patients and methods

### The teeth

One hundred freshly human maxillary premolars extracted within a two-month period in the college of dentistry and Erbil specialized polyclinic center, for orthodontic reasons in an age range of 18-25 were collected. The collected teeth were stored in distilled water at room temperature (25°C) to prevent dehydration until they were ready for experiment. The distilled water was changed every 3 days to

prevent bacterial growth as described before<sup>(12)</sup>. Among 100 collected teeth, 72 teeth were randomly selected based on the following criteria: Intact buccal surface that free of carious and restoration, unbroken buccal surface, buccal surface free from erosion, fluorosis and hypoplastic enamel deformities.

### The brackets

Orthodontic maxillary hooked premolar 0.018 inch Roth, concave base metal brackets (Gemini, 3M Unitek, Monrovia, CA, USA), which easily fitted onto the curvature of the buccal surface of the premolar, were used in the present study. The surface area of bracket base was 12.13 mm<sup>2</sup>.

### Sample preparation

All teeth were mounted vertically in cubic plastic box 3x3x1.5 mm containing a self-cure acrylic resin that only buccal crown at a level slightly below the cervical line was exposed. A dental surveyor was used to align the buccal surface of teeth in the acrylic mold. After molding of the samples, the buccal surface of each tooth cleansed by using a rubber cup with fluoride-free pumice for 10s, then thoroughly washed with water and air-dried.

### Bonding procedure

The teeth were randomly divided equally into six groups of 12 teeth each, based on the surface treatment methods proposed.

**Group PA:** teeth were etched with 37% phosphoric acid gel for 30s and then washed thoroughly for 20s till frosty white appeared<sup>(12)</sup>.

**Group NaOCl+PA:** the surface was deproteinized with 5.25%NaOCl for 1 min using a micro-brush; this was followed by rinsing, drying and acid etching with 37% phosphoric acid for 30s; the teeth were then washed and air-dried<sup>(9)</sup>.

**Group AA+PA:** the enamel surface was sandblasted with 50µm aluminum oxide in a (Renfert, Keramo Basic, Germany) at 70 psi for 3s

through a nozzle opening of 1.5 mm at distance of 5 mm at a 90° angle; after the teeth were sandblasted, their surfaces were rinsed with water and air dried to remove contaminant of operation. After that, 37% phosphoric acid etching gel was applied as previous groups<sup>(12)</sup>.

**Group TPSEP:** teeth in this group were treated with a self-etching primer (SEP; Transbond Plus; 3M Unitek, Monrovia, CA, USA), according to manufacturer instruction.

**Group NaOCl+TPSEP:** the surface was deproteinized with 5.25%NaOCl for 1 min using a micro-brush; this was followed by rinsing and drying, then TPSEP applied according to manufacturer instruction.

**Group AA+TPSEP:** enamel surface was sandblasted with 50µm aluminum oxide in a (Renfert, Keramo Basic, Germany) at 70 psi for 3s through a nozzle opening of 1.5 mm at distance of 5 mm at a 90° angle; then their surfaces were rinsed with water and air dried, thereafter TPSEP applied in accordance to manufacture instruction.

After the enamel conditioning procedures of all the groups, brackets were bonded to the teeth with an orthodontic adhesive (Transbond XT; 3M Unitek). Before curing, each bracket was subjected to a 300-g compressive force for 10s as described by Bishara et al.<sup>(13)</sup>, and excess bonding resin was removed with a small scaler. Then, the adhesive was polymerized for 40 s by a Light Emitting Diode (APOZA D-2000; 1600mW/cm<sup>2</sup>; Taiwan) placed at the mesial, distal, occlusal and gingival aspects for 10s each. All specimens were stored in distilled water at 37°C for 24 hrs.

SBS measurements were carried out at Salahadin University, College of Engineering, Department of Mechanics, Material Strength Lab. The specimens were fixed inside a holding apparatus which in turn secured at lower jaw of the testing machine so that the bracket base paralleled to shearing force. After that, shearing blade (10 mm width and tapered edge of 0.5 mm thickness) coupled to a movable upper part (crosshead) of testing machine (Figure 1). An occluso-gingival load was applied in such way that shearing blade struck against the edge of bracket base at a crosshead speed of 0.5 mm/min, producing a shear

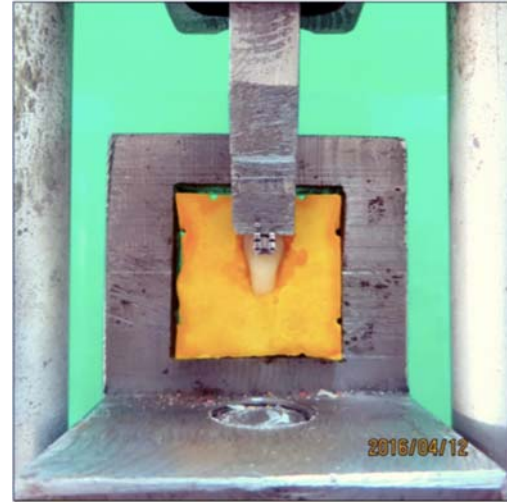


Figure 1: Specimen fixed inside holding apparatus, force at bracket-tooth interface until bracket detached. A computer, electronically connected to the testing machine, recorded the force to debond the bracket in Newton. The bond strength was calculated in Mega Pascal (MPa) by dividing the force (Newton) to the surface area of brackets in (mm<sup>2</sup>), yielding the result at MPa.

#### Adhesive remnant index

Once the brackets were debonded, the enamel surface of each tooth was examined under 40 time's magnification under a stereomicroscope (MOTIC ST-39 Series) to determine the amount of residual adhesive on each tooth. The ARI scores were recorded with the following scale: 0, no adhesive left on the tooth; 1, less than half of the adhesive left on the tooth; 2, more than half of the adhesive left on the tooth; and 3, all adhesive left on the tooth, with a distinct impression made by the bracket mesh.

#### SEM observation

After the enamel surfaces were conditioned in each group, two samples from each group before bonding procedure were inspected by SEM (Leo 1455 VP-Germany) for study the surface topography of enamel following different conditioning techniques. Samples were dehydrated in increasing concentrations of ethanol and water up to 100% ethanol and then coated with gold (approximately 10 to 15 nm) using gold coating apparatus (Nano-structured coating, Hitachi, S-4160). The enamel surface of each tooth was observed under SEM at one chamber pressure (low vacuum), 30 kV

accelerating voltage and 110-mA beam current, and photographs were taken at X1000, X5000 and X10000 magnifications. The procedure of mounting, coating, and imaging of samples carried out at the University of Tehran, College of Engineering, School of Electrical and Computer Engineering.

### Statistical analysis

Descriptive statistics including means and standard deviations were calculated for the SBS analysis. One-way ANOVA was applied to compare the SBS between the groups. A pair wise comparison within groups was analyzed with the Post Hoc Scheffé test. Chi-square test was used to compare ARI between the groups and Spearman's correlation test examined the relationship between the SBS and ARI changes. Statistical significant differences were considered at  $p < 0.05$ .

## Results

### Shear bond strength

The mean SBS; their standard deviations values are shown in Table 1. The group that treated by sodium hypochlorite and phosphoric acid etching showed the highest mean SBS values ( $17.9 \pm 3.1$  MPa), while the group conditioned by TPSEP yielded the lowest value ( $8.8 \pm 3.2$  MPa).

One-way ANOVA test showed statistically significant differences among the six surface-conditioning methods concerning the SBS ( $p < 0.001$ ). The results of the Scheffé post-hoc test showed that groups NaOCL+PA, TPSEP and AA+TPSEP were significantly different from other groups (Table 1). The intergroup differences and their levels of significance are shown in Table 1.

### Adhesive Remnant Index

The amounts of adhesive cement remain on the enamel surface after debonding of brackets are shown in Table 2. Chi-square test did not demonstrate any statistically significant association in ARI scores between the groups as P value is more than 0.05 ( $P = 0.118$ ,  $X^2 = 21.630$ ).

Table 1: Mean SBS of all experimental group.

Experimental groups	Mean (SD)
PA	14.7 (3.4) <sup>A,B</sup>
NaOCl+PA	17.9 (3.1) <sup>A</sup>
AA +PA	15.3 (2.6) <sup>A,C</sup>
TPSEP	8.8 (3.2) <sup>D</sup>
NaOCl+TPSEP	11.7 (3) <sup>B,D,C</sup>
AA+TPSEP	9.7 (3) <sup>D</sup>

The SBS values are mean with standard deviation in the parenthesis. Values exhibited similar superscript letters indicate no significant difference ( $p > 0.05$ ) as determined using Post Hoc Scheffé test.

### Correlation between ARI scores and SBS

The relationship between change in shear bond strength and ARI scores in experimental groups analyzed and presented in Table 3. Spearman's rho demonstrated a statistically significant positive, strong relationship between changes in shearing bond strengths and ARI in the all experimental groups. This correlation in groups PA, NaOCl+PA, AA +PA, TPSEP, NaOCl+TPSEP and AA+TPSEP were 0.934, 0.853, 0.934, 0.924, 0.905 and 0.909, respectively.

### Scanning electron microscope observation of enamel surface

Figure 2 shows the SEM pictures of enamel surfaces that have been treated by (1) phosphoric acid, (2) combined sodium hypochlorite and phosphoric acid, (3) combined air abrasion and phosphoric acid (4) Transbond Plus self-etching primer, (5) combined sodium hypochlorite and acid revealed uniform demineralization of enamel. TPSEP, (6) combined air abrasion and TPSEP. The enamel surface etched with 37% phosphoric acid for 30s showed a porous surface with the typical histological appearance of the enamel prisms with their rounded ends and demineralized peripheries, illustrate cobblestone appearance.

Table 2: Distributions and percentages of adhesive remaining on the teeth after debonding.

Experimental groups	N	0	1	2	3	Chi-Square
PA	10	1 (10)	2 (20)	5 (50)	2 (20)	
NaOCl+PA	10	0 (0)	0 (0)	4 (40)	6 (60)	X <sup>2</sup> =21.630
AA +PA 10	10	0 (0)	2 (20)	4 (40)	4 (40)	(P=0.118)
TPSEP	10	3 (30)	5 (50)	1 (10)	1 (10)	
NaOCl+TPSEP	10	0 (0)	3 (30)	3 (30)	4 (40)	
AA+TPSEP	10	1 (10)	3 (30)	4 (40)	2 (20)	

Table 3: Correlation between shear bond strength and adhesive remaining after debonding.

Experimental groups	Rho	P	N
PA	0.934	.000	10
NaOCl+PA	0.853	.002	10
AA +PA	0.934	.000	10
TPSEP	0.924	.000	10
NaOCl+TPSEP	0.905	.000	10
AA+TPSEP	0.909	.000	10

\*Correlation is significant at the 0.01 level

Application of sodium hypochlorite to enamel surface before phosphoric acid produce more aggressive etching pattern, create deep pits, groves and many melted enamel prisms that non-uniformly distributed throughout the surface. Furthermore, abrasion of enamel surface by aluminum oxide particles before phosphoric acid revealed uniform demineralization of enamel surface with protruding enamel prism core and dissolved prism peripheries. On the other hand, TPSEP induces mild demineralization of enamel prism peripheries, and prism cores remain unaffected. However, application of sodium hypochlorite before TPSEP, induce moderate demineralization that more pronounced in the enamel sheath covering the enamel rods.

## Discussion

Enamel bonding for orthodontic applications was introduced in 1965, which revolutionized and advanced the clinical practice of orthodontics. Adhesion of orthodontic brackets to enamel surface is mediated by adhesive cement. In restorative dentistry, adhesive materials are bonded to teeth permanently. In orthodontics, however, attachments are bonded for a limited time only.

Failures which encountered in adhesion of brackets to enamel surface reside in the two key factors which are the quantity of the etched surface as well as in the quality of the etching pattern<sup>(14)</sup>. In the present study, we evaluated the effects of different enamel conditioning techniques for bonding orthodontic brackets regarding SBS, ARI and SEM findings.

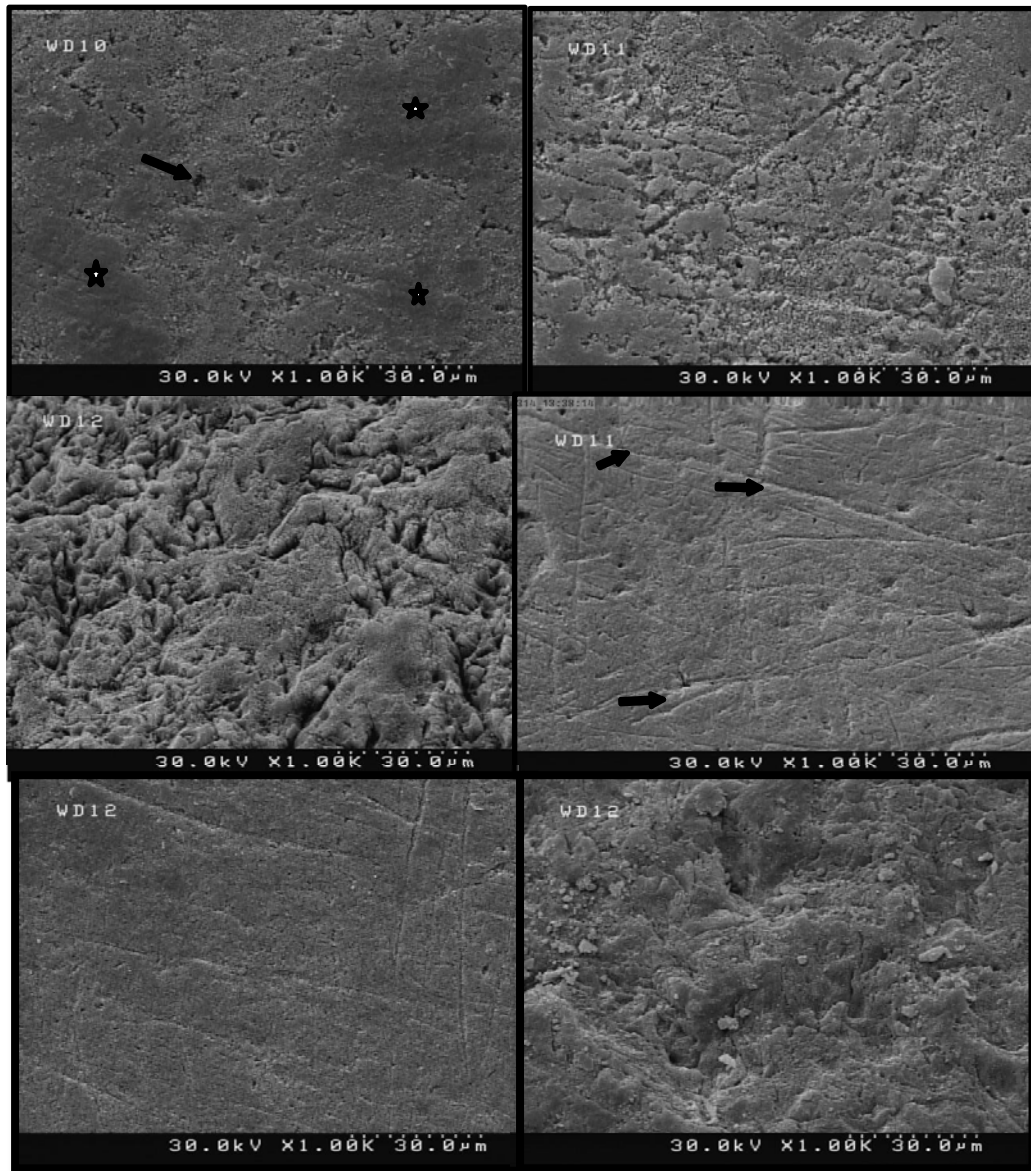


Figure 2: The SEM pictures of enamel surfaces. A: SEM image of surface etch by 37% phosphoric acid for 30s. B: SEM image of surface conditioned by NaOCl and 37% phosphoric acid. C: SEM of the sample prepared by 50 $\mu$  Al<sub>2</sub>O<sub>3</sub> follow by 37% phosphoric acid. D: SEM image of enamel surface conditioned by Transbond Plus self-etching primer. E: SEM image of enamel surface conditioned by NaOCl and TPSEP. F: SEM image of sample conditioned by 50 $\mu$  Al<sub>2</sub>O<sub>3</sub> and TPSEP.

### Shear Bond Strength

The bonding forces in groups NaOCl+PA, TPSEP and AA+TPSEP were significantly different from those in all other groups. Mean shear bond strength

values in six groups evaluated in present study ranged from 8.8 to 17.9 MPa. These values are higher than minimum bond strength values reported by Reynolds<sup>(15)</sup> as clinically acceptable bond strength (5.9 - 7.8 MPa).

Group II (NaOCl+PA) which was conditioned by application of sodium hypochlorite for the 60s and

followed by phosphoric acid etching for 30s had bond strength.

There were few possible explanations for this result. Application of sodium hypochlorite eliminates organic content on the surface of enamel which may prevent effective etching of phosphoric acid<sup>(14)</sup>. By doing this, phosphoric acid may result in more and deeper demineralization of enamel prisms as well as inter-prismatic material which in turn this allows more penetration of adhesive resin into micro-porosities and results in increasing mechanical interlock between adhesive resin and enamel surface. Furthermore, deproteinization of enamel surface doubles the retentive etched surface area (48.8% to 94.5%) as described by Espinosa et al.<sup>(14)</sup>. Additionally, application of sodium hypochlorite before phosphoric acid results in type I and type II etching patterns which described by Silverstone et al.<sup>(16)</sup>, as most retentive etching patterns. This result is consistent with those reported enamel deproteinization result in increasing the shear bond strength of bonded brackets<sup>(8,11)</sup>. Similarly, preconditioning of enamel surface by sodium hypochlorite before self-etching primer in group V results in increasing the bond strength of the self-etching adhesive system.

Air abrasion is a technique for preparing enamel and other tooth surfaces that are mechanical, unlike acid-etching, which chemically dissolves some enamel hydroxyapatite crystals<sup>(17)</sup>. Different studies evaluate the effect of sandblasting in a combination of etching agent on the bond strength of adhesive resin. Some studies found an increase in bracket bond strength<sup>(7, 18)</sup>, but others found no differences<sup>(19, 20, 21)</sup>.

In the present study, sandblasting the enamel surface by 50 $\mu$  aluminum oxide particles before phosphoric acid and self-etching primer in group AA+PA and group AA+TPSEP resulted in increasing the bond strength of orthodontic brackets. There are different reasons that might attribute to increase in bond strength. When air abrasion applied to the enamel surface results in the creation of rough and irregular surface of different height and depth. This will increase the adhesion surface area which may improve the adhesiveness of bonding agent. Another likely effect of sandblasting is that, as it applied to the enamel surface, it removes the outer highly mineralized layer of enamel which is composed of

fluoro-appetite crystal and aprismatic enamel. These enamel constitutes are highly resistant to etching agents, by removing them, it might improve the action of etching agent on core enamel<sup>(22)</sup>. Additionally, air abrasion caused permanent loss of inorganic material as well as the organic material of enamel<sup>(17)</sup>, which later on was not removed by etching agent, so its removal by air abrasion might lead to increase the performance of the conditioning agents.

The present findings concurred with the finding of studies done by others<sup>(9,23,24,25)</sup> whose demonstrated air abrasion before etching improves the bond strength of orthodontic attachments.

The use of the SEP for bonding orthodontic brackets become popular among orthodontists because it produces a gentler etch pattern compared to other methods. However, it combines etching and priming action which simplified the clinical procedure<sup>(26)</sup>. In this study, group IV, which was subjected to SEP treatment, showed lowest shear bond strength. Despite having the lowest SBS value, acceptable levels of bond strength were achieved in this group. Lowering of bond strength in the samples treated by self-etching primer as compared to phosphoric acid etching might be attributed to the quality of the etched surface area. The phosphoric acid result in the deeper dissolution of enamel prism as well as prism peripheries as compared to a self-etching primer which showed shallow and less demineralization of enamel surface, mostly prism peripheries are affected. In this turn, less adhesive resin might penetrate to enamel surface that might lead to decrease in mechanical interlock between enamel and an adhesive resin. Hence bond strength decreased.

Furthermore, calcium monophosphate and calcium sulfate by-products (smear layer) which produced by the action of methacrylate phosphoric acid ester not rinsed away and incorporate into the polymerized network. This might lead to decrease in bond strength of self-etching primers. It is also possible that the low enamel bond strengths by self-etching primers might be caused by the high amount of un-polymerized acidic monomers remaining after curing<sup>(27)</sup>.

Consistent with our results, the different SEP systems tested by others<sup>(2,5,28,29,30)</sup> afforded SBS of levels adequate for orthodontic bonding.

## Conclusions

The shear bond strength of all experimental groups were higher than clinically recommended force: Enamel surface pretreatments, using a combination of sodium hypochlorite or sandblasting before phosphoric acid and self-etching primer, result in increased shear bond strength of orthodontic brackets as well as the surface roughness of enamel. A direct and positive correlation exists between Shear bond strength and amount of adhesive remaining.

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