Shear bond strength of stainless steel brackets bonded to porcelain surface treated with 1.23% acidulated phosphate fluoride gel compared to hydro fluoric acid with silane coupling agent (In vitro comparative study)

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ABSTRACT

Background: With the increasing demands for adult orthodontics, a growing need arises to bond attachments to porcelain surfaces. Optimal adhesion to porcelain surface should allow orthodontic treatment without bond failure but not jeopardize porcelain integrity after debonding.The present study was carried out to compare the shear bond strength of metal bracket bonded to porcelain surface prepared by two mechanical treatments and by using different etching systems (Hydrofluoric acid 9% and acidulated phosphate fluoride 1.23%).
Materials and Methods: The samples were comprised of 60 models (*28mm *15mm*28mm*) of metal fused to porcelain (feldspathic porcelain). They were divided as the following: group I (control): the porcelain surface left untreated and glazed, group II (Diamond bur group): the porcelain surface was treated with fine diamond bur at speed of 350000 rpm for 20 seconds, group III (Red stone bur): the porcelain surface was treated with coarse red stone bur at speed of 8500 rpm for 20 seconds. Each group consists of 20 samples, then each group subdivided into two subgroups; one treated with acidulated phosphate fluoride 1.23% and the other subgroup treated with Hydrofluoric acid 9 % with silane coupling agent.
Results: The result of this study revealed that there was very high significant difference among all tested groups and the highest shear bond strength was for diamond bur group with HFA and Silane (8.67 MPa), the 2nd highest strength was for control group with HFA and Silane (7.52 MPa), the 3rd was (7.38 MPa) in red stone bur with HFA and Silane, the least shear bond strength values were obtained for subgroups treated with acidulated phosphate fluoride gel 1.23%.
Conclusions: The most reliable procedure for bonding orthodontic brackets to the porcelain surfaces is through the surface treatment combinations of mechanical roughening by using diamond bur, 9% Hydrofluoric acid and Silane coupling agent application.
Key words: Acidulated Phosphate Fluoride, Hydrofluoric Acid, Silane coupling agent, Feldspathic porcelain. (J Bagh Coll Dentistry 2013; 25(Special Issue 1):167-173).

INTRODUCTION

Dental porcelain is a popular restorative material, especially for adult patients, where it is used for restorations such as veneer, crown, and bridge. As the demand for adult orthodontic treatment increases, orthodontists are more likely to deal with the problem of placing brackets on teeth restored with porcelain. Conventional acid-etch technique is not effective in preparation of non-enamel surface for mechanical retention of orthodontic attachment (1).

Silane was used as a coupling agent to increase the bond strength to either glazed or roughened porcelains in many studies, but there is a tendency for cohesive failure of porcelain during the debonding process. Additionally, the limited shelf-life time of Silane causes a problem for orthodontists when finding it expired without other spare bottles (2-5).

Mechanical roughening with the fine or coarse diamond burs and sandblasting were reported to provoke crack initiation and propagation within the porcelain (6).

Since the restorations usually remain in the mouth after debonding the brackets, porcelain damage due to extreme roughening of the surfaces during pretreatment or debonding must be avoided (7).

Hydrofluoric (HF) acid and acidulated phosphate fluoride gel (APF gel) was reported to facilitate micromechanical retention (8, 9). Both acids can etch glass or porcelain and thus create a mechanically retentive surface (10). **Nelson and Barghi** found that an APF gel etching produced bond strength comparable to an HF acid etched control. In their study, a 10-minute etch produced the highest bond strength for APF gel whereas the control was etched 1-minute with 10% HF acid. Despite its effectiveness, the hazards of HF acid are well recognized. Mucosal contact with HF can cause erythema and burning associated with loss of tissue, along with intense pain for several days (11,12).

To our knowledge, there is no published study available in the searched data base which directly investigates the bond strength of metal bracket to porcelain surface prepared by 1.23% APF gel as compared to silane coupling agent using feldspathic porcelain which is different

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form the porcelain used in denture teeth that were used by most previous studies. We intend to calibrate our samples according to the surface micro hardness not by just their physical properties [13,14].

MATERIALS AND METHODS

Seventy two molds were fabricated from metal fused to ceramic (Vita 3D Master), each sample **(Figure 1)** was checked under magnification (10 X) for any roughness, bubbles or irregularities, then 12 samples were taken after porcelain firing to test for micro-hardness in order to make sure that all the samples have similar mechanical properties in macro- and micro- level [15,16].

The selection for the hardness testing group was performed randomly by taking one sample from each firing group (6 samples), so the total number was 12 samples from 72 samples [16].

The remaining sixty models were divided into 3 groups, 20 molds each, the first group is the control group the 2nd and 3rd group were treated mechanically by high speed turbine diamond bur and low speed red stone bur, respectively. Then the groups subdivided into two subgroups (APF 1.23% and Silane), 10 mold each. Subgroup APF 1.23 % was treated by acidulated phosphate fluoride 1.23 %, while subgroup Silane was treated by Hydrofluoric acid and Silane coupling agent.

Figure 1: Ceramic Fused to metal model used in the study.

Construction of the metal models

Seventy two wax molds were fabricated by one dental technician in rectangular shape **(28 mm * 15mm*28mm)** by using base plate sheet wax ,then each three models sprued together in order to make the investment mold pattern. After that the sprued wax models were placed inside rubber ring and molded to the cover of the ring, the investing procedure was performed on the vibrator and layer by layer to avoid the bubble formation according to the manufacturers' instructions.

The ring would be then left for 10 minutes for setting according the manufacturer's instructions then it would be placed inside wax oven and at temperature of 280 C° for 30-40 minutes to remove the wax completely from the investment mold (wax burn-out). Then the ring was placed in the electric centrifuge machine (Deguzza, Germany) for metal casting procedure and the mold would be then left for cooling.

Then the metal models removed from the investing ring and grinded from the main sprue to be separated from each other and sandblasted by using alumina oxide particles (250 µm) to remove the investment remnant particles, then finished by using special laboratory carbide and diamond burs using micro motor at a speed of 500000 rpm (**figure 2**).

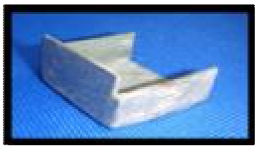

Figure 2: Finished Metal Mold

Building and Firing of porcelain

After the metal models were prepared and finished ,the process of porcelain building was started by painting the metal with Opaquer layer and firing for 15 minutes at a temperature of 950 C°, after that building the ceramic layer 2mm in thickness and firing for 18 minutes at temperature of 930C°. The firing process was made by using Ivoclar ceramic oven, model P300, Germany. The model then should be grinded and finished by special burs, after that painted with glaze layer and fired at 920 C° for 15 minutes [16].

Vickers hardness Test

One model was taken from each firing group (6 models), so twelve models were chose for the Vickers hardness test machine in the University of Technology in Baghdad, **(Figure 3)**. Six micro-indentation were made on each sample and VHN (Vickers Hardness Number) was measured. This is to ensure that all the samples used in the study have nearly the same mechanical properties.

Figure 3: Vickers Micro-Hardness test machine

Sample Grouping

The specimens were divided according to porcelain surface treatment into APF 1.23% and Silane. Group APF 1.23 %: the specimens were treated by using the APF 1.23 % for 10 minutes [17].Group Silane: The specimens were treated by using Hydrofluoric acid and silane coupling agent.

Group (I) control group*:*

The porcelain surfaces were left as they came from the Dental lab; glazed porcelain**.**

Group (II):

The porcelain surfaces were treated mechanically by using tapered diamond bur at speed of 320000 rpm, for 20 seconds with water spray, and then dried for 20 seconds with oil free air.

Group (III):

The porcelain surfaces were treated mechanically by using stone bur with low speed straight hand piece for 20 seconds at speed of 85000rpm, and then the specimens were dried with oil free air for 20 seconds.

Bonding Procedure

The bonding was done by applying a thin layer of bonding agent on the labial porcelain surface using a disposable brush, and then an equal amount of the light cure composite was applied on the bracket base according to the manufacturer instructions, which was then positioned in the center of the model using a clamping tweezers. Then a constant load of 300 grams was applied by pressure tension gauge(after a modification done in its end in order to have a flat surface to prevent bracket dislodgement during loading) which placed on the bracket at 90°for 10 sec.[17,18], to ensure that each bracket was seated under equal force. Any excess bonding material was carefully removed from around the bracket base with a sharp hand scaler without disturbing the seated bracket.

Shear Bond Strength Test

Shear test was accomplished using Tinius Olsen universal testing machine **(Figure 4)** with loading cell 50 kilogram and a crosshead speed of 0.5 mm/min [19, 20].

Each sample was seated in the mounting metal vice and placed on the base of the testing machine (which was parallel with the horizontal plane). The chisel end rod was fitted inside the upper arm of the testing machine with its chisel end downward parallel to the bonded porcelain labial surface to apply a force in a gingivo-incisal direction of the bracket that produce a shear force at the bracket base/ porcelain surface interface, until debonding occurs. When the bracket was debonded from the porcelain labial surface by the force applied from the testing machine, the ultimate magnitude of the reading was taken; this force was measured in kilograms and converted into Newton according to the following equation:

Force (N) = Load (kg) X Ground acceleration (9.8 m/sec.).

Then the force was divided by bracket base surface area (10.9 mm²), which was taken from the manufacturer to get the strength value in Mega Pascal (MPa) units. Each debonded bracket was kept with its corresponding porcelain surface to estimate the adhesive remnant index.

Figure 4: Tinius Olsen Universal Testing Machine.

RESULTS

Vickers hardness test

Hardness test was performed to the ceramic surfaces of the samples used in this study in order to confirm that all the groups had the same mechanical property although that these samples were baked in different production time.

The load used was 900 grams (0.9 Kg) for 15 seconds on the top of each sample surface make the indentation, this indentation then measured under the microscope of the micro tester, the VHN (Vickers Hardness Number) was calculated by special equation:

$$\text{VHN} = 1.8544 * \frac{p}{(D\ average)^2} \quad (16)$$

Where p is the load value, D average is the mean of the six indentations made and (1.8544) is a constant value. This equation was used for each sample to get the Vickers hardness number and it was performed by a program incorporated inside the digital micro tester.

Descriptive statistics of the Surface hardness

Descriptive statistics were performed including mean, standard deviation and error for all tested samples, and then Kruskal Wallis test was used. There was statistically no significant difference among all the groups (p= 0.44). All the samples have similar mechanical property of the porcelain surface although they were baked in different production times**, (Table 1).**

Table 1: Descriptive statistics of the VHN

	Descriptive Statistics			Kruskal Wallis Test		
	Mean	S.D.	S.E.	χ^2	d.f.	p-value
VHN	221.18	10.003	2.89	11	11	0.44

Table 2: Descriptive statistics and groups differences

Variables	Descriptive statistics									Group difference		
	Control			Diamond bur			Red stone bur			ANOVA test		
	Mean	S.D.	S.E.	Mean	S.D.	S.E.	Mean	S.D.	S.E.	F-test	p-value	Sig.
APF	0.97	0.09	0.03	4.94	0.45	0.14	6.18	0.51	0.16	470.04	0.000	HS
Silane	7.52	0.75	0.24	8.67	0.56	0.18	7.38	0.64	0.2	11.57	0.000	HS
t-test	-27.39			-16.37			-4.65			d.f.=29		
d.f.	18			18			18					
p-value	0.000			0.000			0.000					
Sig.	HS			HS			HS					

Descriptive Statistics of Shear Bond Strength (Figure 5)

The descriptive statistics were performed for all variables, which include two types of etching systems (Acidulated phosphate fluoride 1.23 % and Hydrofluoric acid) on two types of porcelain surfaces (glazed and mechanical treated surface). These statistics included, Mean, Standard deviation, Standard error. The shear bond strength values for all tested samples were expressed in Mega Pascal (MPa) and are displaced in **(Table 2).**

In the *control Group* (Glazed porcelain surface), the (APF 1.23 % and Silane coupling agent) samples showed Very High Significant difference (P-value = 0.000), APF 1.23% had mean shear bond strength 0.97±0.09 MPa, While Silane group had Shear bond strength 7.52±0.75 MPa**.**

In the *other groups* (two types of mechanical treatment), there was high significant difference with both etching systems (P-value = 0.000), the Diamond bur group showed Mean shear bond strength = 4.94±0.45 MPa with APF 1.23 %, while with silane and Hf it showed 8.67 ± 0.56 MPa shear bond strength. The Red stone bur group showed also high significant difference (P-value = 0.000) between the APF1.23% and Silane etching systems, for the 1st it was 6.18 ±0.51 MPa and for the 2nd it was 7.38±0.64 MPa, respectively.

Mode of Failure Site

The sites of bond failure of all tested groups are shown in **(Figure 6).**

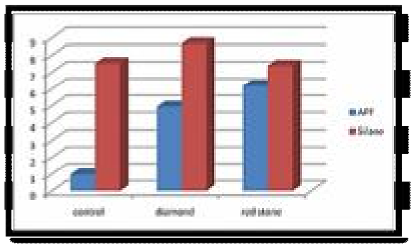

Figure 5: Mean Shear bond Strength of all tested groups

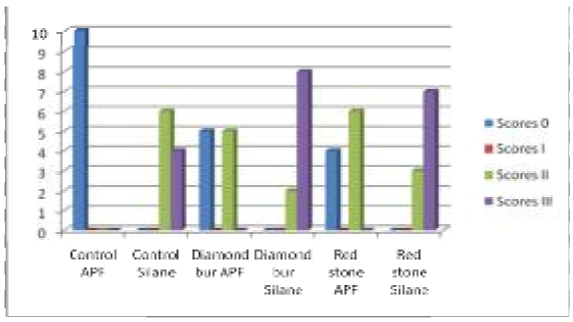

Figure 6: Adhesive Remnant Index of total samples.

Adhesive remnant Index according to Ärtun and Bergland (1984) was as following:

Score (0) in 100 % of the control group (glazed porcelain) treated with APF 1.23%, in 50% of the 2nd group (diamond bur group) and 40 % in 3rd group **(red stone bur).**

Score (I) was not noticed in any of the tested groups

Score (II) in 60 % of the control group **(glazed porcelain)** treated with Hf + Saline, and 3rd group (red stone bur) treated with APF 1.23 %, in 50 % of the 2nd group **(diamond bur)** treated with APF 1.23 %, in 30 % of 3rd group (red stone bur) treated with HF+ Silane, and only 20 % of the 2nd group **(diamond bur)** treated with HF + Silane.

Score(III) in 40 % of the control group (glazed porcelain) , in 80 % in 2nd group **(diamond bur)**

,and in 70 % of the 3rd group(red stone bur) ,all treated with HF+ Silane.

Chi Square test was used for such non parametric data and the Statistical analysis showed very high significant difference among all tested groups (glazed porcelain, diamond bur, and red stone bur) with P-value = 0.000.

DISCUSSION

Shear bond strength

There are few scientifically based recommendations in the literature for minimum orthodontic bracket shear bond strength. *Whitlock et al.* [22] suggested that 6-8 MPa was adequate for orthodontic attachments to endure the course of treatment and sufficiently weak to preserve the porcelain restoration following bracket removal [22].

One of the aims of this study is to evaluate the effectiveness of different surface conditioning methods on the shear bond strength of stainless steel brackets bonded to porcelain as follow:

1. <u>Glazed porcelain (Control group)</u>

The samples of the control group were divided into two subgroups; the 1st was etched by using APF 1.23% for 10 minutes and these showed bond failure at 0.97MPa ± 0.09 S.D**.(Table 2)** , Similar results with the same methodology used in the present study were recorded by other previous studies [9,23,24].

The premature loss of the brackets was occurred due to the fact that the APF 1.23% could not do etching to the glazed porcelain. It simply cleans the surface and hydrolyzes the silica of porcelain in the same time preserving the intact smooth surface of the porcelain.

The 2nd subgroup was etched by using HFA 9% for 2 minutes according to the manufacturer's instructions, and then porcelain primer (silane coupling agent) was used. It showed 7.52 MPa ± 0.75 S.D. and this is considered the 2nd highest value of mean shear strength among all tested group in the present study.

These results agree with other researchers [17,25] who found that porcelain preparation with HFA etching followed by silane application, resulted in high shear bond strength. The significant increase in bond strength is due to the effect of HFA by facilitating micro-retention and the silane coupling agent which provide a chemical link between organic resin compound and inorganic porcelain compound.

2. <u>Mechanical surface treatment of porcelain:</u>

A. Roughening by diamond turbine bur.

Samples of this group also divided into two subgroups; 1st subgroup etched by APF and the other by HFA 9%. The 1st subgroup was etched for 10 minutes, this subgroup showed shear bond strength of (4.94 ± 0.45 S.D) with very high significant difference from other subgroups of same surface treatment. The increase in shear bond strength value may be due to the surface roughness more than that of APF micro etching. Depending on previous studies in the literature [26]; scanning electron microscope showed that diamond bur removed the glaze completely leaving islands and tunnels of rough porcelain that of course increases bond strength.

The 2nd subgroup was etched by HFA 9% for 2 minutes according to the manufacturer´s instructions then a porcelain primer added (Silane coupling agent). The shear bond strength was 8.67±0.56 S.D. which is the highest value among all the tested groups in the present study. This high value may be due to the fact that roughening the porcelain causing islands and tunnels as shown in SEM [26], also the HFA removes the glassy and crystalline layer from porcelain which depends on its concentration and 9 % was found to be so efficient. The high shear bond strength of this subgroup comes from mechanical retention and chemical bonding.

These results coincides with other studies [25,27],while disagrees with others [1,8] who found that there is no significant difference between the HFA and other agents and suggested the main bond strength comes chemically from silane **.**

B. Roughening by using Red Stone bur.

Samples of this group also divided into two subgroups; one etched by APF1.23 % and the other by HFA 9%.

The 1st subgroup (APF 1.23 %) showed remarkably higher shear bond strength which was 6.18±0.51 S.D. when compared with the pervious subgroups of the same etching system. This may be due to the micro and macro etching done by rough stone bur. Roughening the porcelain surface with coarse bur produces random peeling appearance, thus enlarging the porcelain surface with only shallow mechanical retention as revealed by SEM, although macroscopic appearance of the rough porcelain gives the impression of high retention surface. Besides ,the APF 1.23% could help in the chemical bonding to porcelain.

This result agrees with some studies [9] while, disagrees with others [28,29], who found that etching with APF, might be enough for bonding on porcelain. The difference may be due to varieties in etching times, concentration, type of porcelain used, storage media, and adhesive agent used.

The findings of the present study agrees with the results in the literature [26] who found that there was no significant difference between using HFA and roughening at the same time especially with red stone bur, while disagrees with others [1] who stated that the roughening showed the highest shear bond strength among all other conditioning methods.

Clinically, the method of choice to improve the bond strength to porcelain surface will be probably the one that provides sufficient shear bond strength to porcelain surface however, this choice will also depend a lot on the patient´s oral function and Para functional habits and the orthodontist´s mechanics in tooth movements. The continuously increasing load applied in vitro is not the same type of stimulus that occurs clinically. Bonded brackets are subjected to shear, tensile, torsion, and combination of these forces. Except for traumatic incidents, brackets coming loose in the mouth as a result of repeated stresses that produce micro cracks that propagate until bond failure occurs.

Type of debonding force in the machine is not the same as force applied in careful clinical debonding so the risk of damaging the porcelain surface need not to be a great problem with gentle, still effective manual technique.

From the results of the present study, we advise the use of mechanical surface treatment with diamond turbine bur and using Hydrofluoric acid 9 % with silane coupling agent as an effective method for bonding the metal brackets to porcelain especially when heavily tooth movement and/or long treatment duration or patient presents with Para functional habits. As an alternative method we advise to use low speed red stone bur with APF 1.23 % or HFA + silane coupling agent or to do direct etching with HFA to the glazed porcelain with the use of silane coupling agent ,this is applicable for short duration treatment or light tooth movements required.

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