Research Paper: Evaluation of the effect of three different post etching surface treatments of feldespathic ceramic on the shear bond strength to composite resin

Seyedeh Maryam Tavangar[1], **Reza Tayefeh Davalloo**[1], **Farideh Darabi**[1] **Forough Vaez Ghasemi**[2*]

[1]Dental Sceinces Research Center,Department of Operative Dentistry, School of Dentistry, Guilan University of Medical Sciences, Rasht, Iran
[2]Dentist

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ABSTRACT

Introduction: The clinical porcelain repair system is almost entirely dependent on the integrity of the bond between porcelain and composite resins. The preferred manner of conditioning the fitting surface of the ceramic restoration is by etching with hydrofluoric acid followed by the application of a silane coupling agent and bonding resin to achieve a high bond strength. Hydrofluoric acid etching of silica-based ceramics produces insoluble silica-fluoride salts, which can interfere with the bond strength to the resins.

Materials and Methods: 66 porcelain disks (Super Porcelain EX3, Noritake) of 8 mm diameter and 3 mm thickness were fabricated and stored in distilled water for 10 days. Porcelain surfaces were abraded with number 023 football shaped bur, etched with 9.5% Hydrofluoric acid and rinsed with water. The disks were randomly divided into 3 groups: Group 1: without any additional treatment Group 2: Etching with 35% phosphoric acid for 30 seconds followed by water spray rinse. Group 3: Ultrasonic cleaning in distilled water for 5 minutes.

Silane and porcelain bonding resin (Bisco Inc.) was applied on the bonding surface of porcelain disks, according to manufacturer's instructions. The composite samples (AELITE All Purpose Body) of 4 mm diameter and 3 mm thickness were bonded to the porcelain disks following fixation of a plastic mold on the center of disks.

Study samples were stored in distilled water in room temperature for 1 week. Shear bond strength of each specimen was determined using universal testing machine following thermocycling protocol. (1000 cycles between 5°C and 55°C) The fracture modes (adhesive, cohesive, mixed) were examined under scanning electron microscopy at ×25 magnification. Data were analyzed by one-way ANOVA, post hoc Tukey's test and post-hoc Dunnett's t-test.

Results: For both group 2 (P=0.015) and 3 (P<0.0001) the mean bond strength were significantly different from group 1. The bond strength values were significantly higher in group3 compared with group 2. (P=0.011) The highest and lowest bond strength was achieved in group 3 and 1, respectively.

Conclusion: According to increased bond strength between composite resin and ceramic following application of phosphoric acid and ultrasonic cleaning, they are both effective methods for post etching ceramic treatment, whereas regarding to the highest shear bond strength in group 3, ultrasonic cleaning is more recommended than phosphoric acid application.

*** Corresponding Author:**
Forough Vaez Ghasemi.
Address: Department of Operative Dentistry, School of Dentistry, Guilan University of Medical Sciences, Rasht, Iran
Tel: +98 (13) 33330862
E-mail: vaez.forough@gmail.com

Introduction

Despite the advances made in the field of dental composites and glass ionomers, porcelains still have better aesthetics results(1).

In addition to aesthetics, it is highly acceptable for its biocompatibility, long-term color stability, chemical stability, wear resistance, low thermal conductivity(2), and the ability to turn into precise shapes(3).

The increased use of porcelains has led to an increased need for a reliable method for repairing broken porcelains intraorally,(4)which is relatively easier and more cost and time effective to the patient and the dentist in comparison to removal and replacement of the entire restoration. On the other hand it eliminates the risk of damaging the prepared abutment while attempting to remove the restoration(5).

In the ceramic repair process, surface treatment should be performed, which involves mechanical or chemical treatment to provide roughness on the ceramic surface(2)

When composite materials are used to repair broken ceramic restorations, the use of HF or hydrofluoric acid and silane and bonding resin on the ceramic surface is the most efficient method for increasing bond strength(6,7). The use of acid on the surface of the ceramic selectively removes the glass matrix and the crystalline structure becomes exposed, which leads to increase the roughness on the surface of the ceramic that is expected for micromechanical retention(8). It has been reported that etching the silica-base ceramic with hydrofluoric acid produces silica-fluoride insoluble salts. These salts remain as a side-effect on the surface of the ceramic, and if they are not removed completely, they can interfere with the bond strength between resin and the ceramic surface(6,7). Some studies have suggested cleaning methods for ceramic surfaces to remove these residual particles after HF etching(9). These methods include ultrasonic baths(9,10,11)phosphoric acid 37%, washing under running water, or a combination of 37% phosphoric acid and ultrasonic baths(9).

Despite all of the above, there is still no standardized method for removing crystalline particles remained after etching porcelain with HF. The use of phosphoric acid has not yet been identified as a standard method in texts and references, but in a series of cements brochures, it is referred to as an effective method for the proper bonding of composite to porcelain.

Meanwhile, in the studies conducted to investigate the effect of phosphoric acid, contradictory results have been reported(9,11).

In this regard, the purpose of this study is to investigate the bond strength of composite to porcelain after its surface treatment with HF and the use of phosphoric acid compared to ultrasonic bath for removal of residual crystalline particles and finally comparing these two groups with regular washing treatment after etching with HF.

Methods and Materials

66 wax discs were made in 8 mm diameter and 3 mm height in the plexiglass molds. The samples were invested in refractory plaster (Nori-Vest, Noritake, Japan) and then entered the wax removal furnace. (700 °C for 20 min)

After wax removal, porcelain discs were made with porcelain feldspathic powder. (Super Porcelain EX3, C4B, Noritake Dental Supply Co., Aichi, Japan). The firing process was carried out at 950 ° C for 7 minutes and 600 ° C for 10 minutes. (Heat rate = 45 °C/min)

Porcelain firing was conducted 3 increments for each sample in order to control porcelain shrinkage and preventing crack formation in refractory plaster. (Fig1)

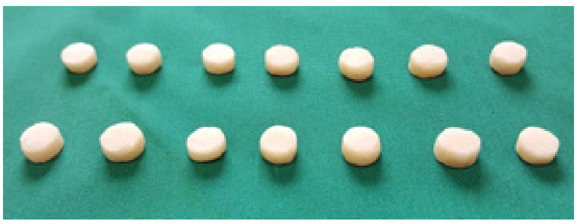

Figure 1:porcelain discs

Porcelain Disc were stored in distilled water for 10 days after finishing the firing steps. The bonding surface of each disc was grounded under running water with 240-, 320-, and 600- grit abrasive papers. (Buehler,Ltd.,Lake Bluff, IL)

The roughed surfaces of all disc were etched with 9.5% hydrofluoric acid (Bisco Inc, Schaumburg, IL, USA) for 90 seconds, washed for 30 seconds under running water and dried, and then the porcelain disc were randomly divided into 3 groups, n=22:

Group 1: No further treatment was performed on the disc surface.

Group 2: 35% phosphoric acid (Bisco Inc, Schaumburg, IL, USA) was applied to the porcelain surface with a gentle micro brush rubbing motion for 30 seconds and then washed under running water for 30 seconds(9,11).

Group 3: Samples were placed in distilled water for 5 minutes in a Digital Ultrasonic Cleaner(9). (CD-4820 Ultrasonic Cleaner)

The surfaces of all specimens were dried up by air spray. Then 1 to 2 thin layer of BIS-SILANE (Bisco, Schaumburg, IL, USA) was applied on the ceramic surfaces of each of the 3 groups. After 30 seconds, the surfaces of the samples were dried by applying a gentle air spray.

A layer of Porcelain Bonding Resin (unfilled resin made by Bisco, Schaumburg, IL, USA) was applied on the silane according to the manufacturer's instructions and then turned into a thin layer with a gentle air spray and light-cured for 30 seconds using LED light. (Bluedent LED Smart, Bulgaria) The light-curing device was calibrated with a radiometer (DigiRate – Radiometer, LM-100, Monitex, Taiwan) to 1200 mw/cm2 previously.

In the next step, a plastic mold with a diameter of 4 mm and a height of 3 mm was fixed by sticky wax in the middle of the porcelains without compromising isolation. The AELITE All-Purpose Body Composite Resin A2 shade (Bisco Inc, Schaumburg, IL, USA) was placed on the surface of the porcelain inside the mold through the incremental technique. (In two 1.5mm increments)

The first increment was polymerized for 20 seconds. The last increment was cured for 30 seconds according to the manufacturer's instruction. (Fig 2) It should be noted that one operator performed all the procedures and a calibration of the LED light intensity was performed after every 2 times using of the light-curing device.

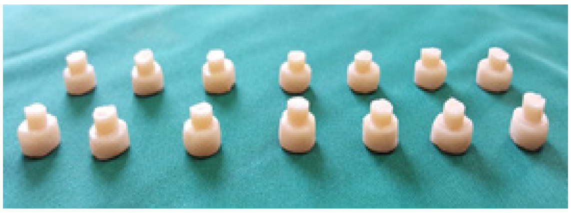

Figure 2 porcelain specimen after bonding to composite

All the specimens were stored in distilled water in room temperature for one week subsequently were thermocycled (DORSA device, Iran) between 5 ° C and 55 ° C (± 2 ° C) for 1000 cycles with a 15 seconds transfer time and 30 seconds dwell time in each bath(2).

Shear strength evaluation test

The shear bond strength test in this study was performed by STM-20, Universal testing machine, SANTAM series.

To measure the porcelain-composite bond strength, an acrylic supporting box was made for porcelain specimens that was fitted to the clamps of the STM device.

The shear force was applied by a 0.25 mm thick stainless steel blade at a crosshead speed of 0.5 mm / min along the composite and porcelain bond interface until fracture(11)

The amount of force applied in the STM Controller software was measured. The force recorded at the fracture point (Newton) was divided into the surface area of the bond between the composite and the porcelain (mm2) to calculate the bond strength in MPa.

Fractured Surface Analysis

The porcelain surface was evaluated under a stereomicroscope (Echo Lab, SM B12, Italy) at 25× magnification.

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The types of failure were divided into three modes: cohesive, adhesive and mixed:

Cohesive failure was defined as a fracture in the body of ceramic or composite resin; the adhesive failure when the separation occurred between ceramic and composite bonding surface and mixed when there was adhesive and cohesive failure, simultaneously. (Fig 3)

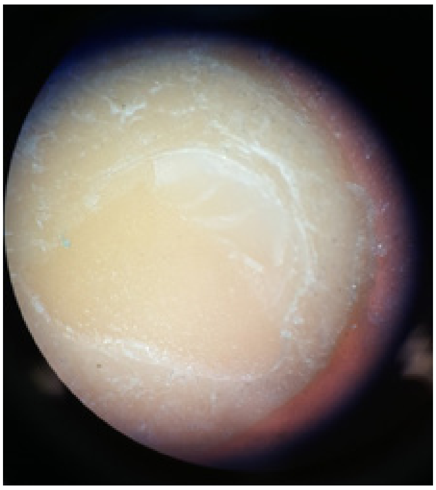

Figure 3 mixed failure

Statistical Analysis

After collecting data, the information was analyzed with SPSS version 21 software. In order to determine the shear bond strength in three groups, the statistical indicators such as mean, standard deviation, minimum, maximum and confidence interval of 95% were used.

Shapiro-Wilk test was used to test the normal distribution of bond strength. The results indicated that the bond strength have followed the normality distribution.

Therefore, in order to compare the bond strength in three groups, one way ANOVA test was used and to compare the bond strength two by two, Tukey HSD and Dunnett t tracking tests were used. The significance level of the tests was considered with $P < 0.05$.

Results

According to table 1, the average and standard deviation of bond strength is 14.99 ± 3.7 MPa in group 1, 17.80 ± 3.88 MPa in group 2 and 20.85 ± 2.42 MPa in group 3.

Comparison of these averages based on one-way ANOVA test showed that the three groups had a significant difference in the bond strength. ($P < 0.0001$)

Table 1 Mean shear bond strength, standard deviation, minimum and maximum values in each group

		Groups			
		without post Etching Treatmen	Cleaned With H3PO4	cleaned Ultrasonically	P
bond strength (MPa)	count	22	22	22	>0.0001
	mean	14.99	17.80	20.85	
	standard deviation	3.70	3.88	2.42	
	minimum	7.99	11.20	17.51	
	maximum	21.28	28.20	27.11	
	95.0% Lower CL for mean	13.35	16.08	19.78	
	95.0% Upper CL for mean	16.63	19.53	21.92	

*ANOVA

Table 2 data which shows bond strength of binary groups based on POST HOC test by Tukey HSD and Dunnett t methods indicates that the bond strength of group 2 ($P = 0.015$) and group 3 ($P < 0.0001$) both had statistically significant difference compared to that of group 1, also group 3 had more average bond strength compared to 2 and this is statistically significant.

Based on Fig. 4 group 3 and group 1 have the highest and lowest bond strength respectively.

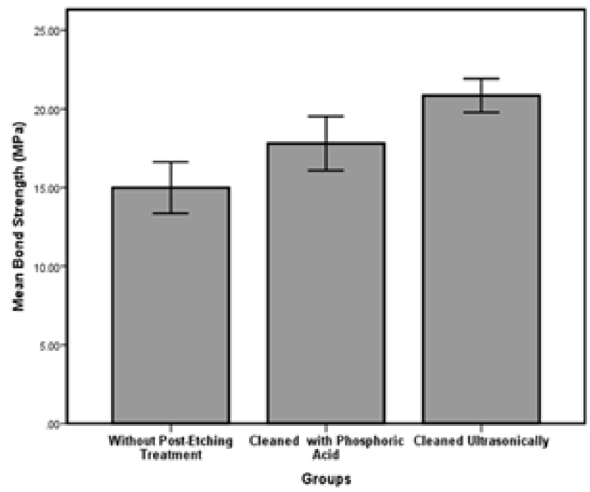

Figure 4 Average bond strength

Table 2 Dual comparison of shear bond strength

POST HOC Test	(I)Groups	(J)Grops	Mean Difference(L-J)	Std.Error	p	95%confidence Interval	
						Lower Bound	Upper Bound
Dunnett t	Etched with H_3PO_4	without post Etching treatment	2.815	1.024	.015	.498	5.132
	Cleaned Ultrasonically	without post Etching treatment	5.860	1.024	.001	3.543	8.177
Tukey HSD	Cleaned Ultrasonically	Etched with H_3PO_4	3.045	1.024	.011	.587	5.503

Dependent Variable:Bond Strength(MPa)

Discussion

Dental ceramics have been used for decades for a variety of reasons such as esthetics, masticatory function, color stability, radiopacity, coefficient of thermal expansion similar to dentin, wear resistance, durability and biocompatibility(2,12). The attempt to repair the fractured ceramic intraorally with the resin composite is more cost effective, has faster outcome and helps preserving the supporting structures compared to removing prosthesis for indirect restorations(12). Therefore, a reliable method for repairing broken porcelains is necessary.

One of the factors that improve bonding between ceramic and composite resin is the surface preparation method(13).

Since etching silica-base ceramics with hydrofluoric acid leads to the production of insoluble salts that interferes in bond strength(6,7) some studies have suggested surface treatment methods, including the use of phosphoric acid and ultrasonic techniques to clean the ceramic surface, which aims to remove these remaining particles after HF etching(9).

In the studies conducted to investigate the effect of phosphoric acid, contradictory results have been reported. Steinhauser et al 9 study eliminates any effect of phosphoric acid on the bond strength between the HF etched ceramic an adhesive system and some other studies11, 14 claimed that surface treatment of the ceramic with phosphoric acid after applying HF would significantly increase the bond strength between the ceramic and the adhesive system. Some of the methods for assessing bond strength are shear, tensile, micro shear and micro tensile testing. A commonly used method is shear bond test which was used in this investigation.

In order to consider the effect of temperature changes on the bond strength, thermocycling was performed on the samples to accommodate to intraoral environment.

The hypothesis in this study was that the ceramic-composite bond strength won't be affected whether the phosphoric acid and ultrasonic method were used or not after etching the ceramic with HF. The hypothesis was rejected by this study.

Based on the results of this investigation, the application of phosphoric acid on feldspathic ceramics after etching with HF leads to increased shear bond strength than the control group. The increase of bond strength in ultrasonic cleaning group was also observed, and in both of these groups, the increase was statistically significant. The strength of the composite bond to porcelain in the ultrasonic group was significantly higher than that of the specimens treated with phosphoric acid.

Similarly, several other studies(11,14) showed that the bond strength of the treated group with phosphoric acid after HF was significantly higher than the control group in which phosphoric acid was not applied.

As previously mentioned, HF leads to the production of water insoluble side-effects on the surface of silica-based ceramics, which, according to the results of this investigation, destruction or removal of these sediments by phosphoric acid, increases the strength of the ceramic bond to the composite, as well as phosphoric acid can reduce ceramic's PH levels and increase the H+ concentration and activate the silane coupling agent(14).

According to the study of Magne and Cascione10, similar results to present investigation were obtained and it was observed that these sediments were partially removed with phosphoric acid application. In the previous study, the only surface treatment method was phosphoric acid followed by an ultrasonic bath, which both methods proved to be necessary to be used together for removing the remaining crystalline particles and increasing the bond strength.

Steinhauser et al(9) found that the bond strength of the feldspathic ceramic and the adhesive system was not influenced by different techniques of ceramic cleansing such as phosphoric acid and ultrasonic methods after HF etching, which the mentioned result, contradicted the findings of this study. The contradiction between Steinhauser's and the present study could be because of the difference between the types of composite, silane and adhesive that was used, the lack of thermocycling and the difference in the type of bond strength test (micro shear).

Several other studies(15,16) proved that the bond strength between ceramic and composite resin significantly increases following ultrasonic cleaning as shown in the present investigation. In fact, ultrasonic treatment allows removal of residues remained from HF etching and produces a clean surface, which facilitates the reaction between the silane coupling agent and the hydroxyl groups on the etched ceramic surface(17).

It should be noted, however, Sato et al(15) showed that apart from increasing the bond strength, no significant progress was made in ceramic mechanical properties (such as flexural strength) after purification protocols, including ultrasonic treatment. In the mentioned study, lithium disilicate ceramics were used, and the mechanical properties of this ceramic are based on the percentage of glass in the structure, so that the HF etching cannot undermine the ceramic structure. On the other hand, the formation of post-etching sediments in this type of ceramics may be less than other types, including the feldspathic types(15).

According to Aida et al. 18, bond strength was not dependent on porcelain preparation methods such as ultrasonic cleaning, and there was no significant difference in the bond strength of composite to porcelain between ultrasonic cleaning samples with other specimens. This difference can be due to the difference in the type of silane used, the lack of bonding porcelain, the different type of composite used in 2 studies, the storage of samples in water at 37 ° C for a short time (1 day) and no thermocycling.

It should be noted that according to Matsumura et al.19 bond strength in all mechanical or chemical adhesion systems should be at least 10 MPa to be acceptable for clinical situations which has been provided in phosphoric acid and ultrasonic methods in present study.

Conclusion

Despite the limitations of laboratory studies, it can be concluded that phosphoric acid and ultrasonic methods are both effective methods for purifying the feldspathic ceramic after HF etching in comparison to no additional treatments, which leads to a significant increase in the strength of the composite bond to porcelain, but the ultrasonic method, due to further increase in the shear bond strength is more effective in cleaning the porcelain surface and is preferable to phosphoric acid application.

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Conflicts of interest

There are no conflicts of interest

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