

Effect of etchant type and the use of silane on the shear bond strength of composite resin to porcelain.

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ABSTRACT

Background: This study was formulated to compare the effect of 5%hydrofluoric acid in comparison to 37%phosphoric acid with and without the application of silane on bond strength of composite to porcelain.

Materials and Methods: Specimen preparation was divided in to two phases, metal-disks fabrication (8mm-diameter and 4mm-thickness) and ceramic veneering. Thirty two specimens were prepared, sandblasted with 50 µm aluminum oxide, and divided into four groups of eight samples. Groups I and III were etched with 37%phosphoric acid while groups II and IV were etched with 5%hydrofluoric acid; and groups I and II were silanated while groups III and IV were not. Heliobond, and resin composite were applied to each specimen using a plastic transparent split mold (4mm-diameter and 4mm-height). Specimens were stored in distilled water for 24 hours then thermocycled for 100 cycles. Shear bond strength was determined by a Zwick universal machine at a cross-head speed 5 mm/minute.

Results: Mean shear bond strength values were 21.02(group I), 23.31(group II), 4.00(group III), and 18.60(group IV). Paired-t revealed that specimens treated with 5%hydrofluoric acid were significantly higher than those treated with 37%phosphoric acid regardless the use of silane. The use of silane dramatically enhanced the bond strength of composite resin to porcelain for both acids. All specimens treated with 37%phosphoric acid without silane showed adhesive failure, while those treated with 5%hydrofluoric acid showed mostly cohesive failure within ceramic.

Conclusion: Silane improved the shear bond strength of composite to ceramic, and hydrofluoric acid performed greater bond strength compared to phosphoric acid.

Key words: Silane, Hydrofluoric acid, Ceramic repair, Shear bond strength. (J Bagh Coll Dentistry 2011;23(4):1-6).

INTRODUCTION

Failure of an all-ceramic restoration due to localized chipping or fracture presents a challenging clinical situation because replacement may involve discomfort for the patient and increased cost. Chairside repair using composite may be a simple alternative method to total replacement of the prosthesis. ⁽¹⁾

For the procedure to be clinically acceptable, interfacial bond between the ceramic substrate and composite should be strong and durable enough to withstand loading stresses. ⁽²⁾

To achieve this goal, specific treatments of the substrate must be performed. These may be treatments that promote mechanical retention or treatments that promote chemical adhesion or combination of them. ⁽³⁾

Mechanical retention includes airborne-particle abrasion with aluminum oxide, ⁽⁴⁾ roughening with a diamond rotary cutting instrument, ⁽⁵⁾ and etching with hydrofluoric acid, ⁽⁶⁾ phosphoric acid, ⁽⁷⁾ or with acidulated phosphate fluoride. ⁽⁸⁾

Hydrofluoric acid works by creating surface pits via preferential dissolution of the glassy phase from the ceramic matrix. ⁽⁹⁾

In addition, treatments may be used that promote chemical adhesion, such as silanization and the application of adhesive primers

Finally, treatments that promote mechanical retention as well as chemical adhesion may also be considered, such as the deposition of silica by conventional airborne-particle abrasion or by the use of specific equipment and tin electroplating. ⁽¹⁰⁾

This study evaluated the effect of 37% phosphoric acid in comparison to 5% hydrofluoric acid (HF) on shear bond strength of the composite resin to porcelain with or without the use of silane coupling agent.

MATERIALS AND METHODS

Specimen Fabrication

Thirty two specimens composed of metal discs veneered by ceramics were fabricated. The metal specimens were obtained using the lost wax technique. Using copper rings, wax patterns were cut from sheets of wax as flat disks of 8 mm diameter and 4mm thickness. Each wax pattern was sprued at 45° angle with a 3mm thick and 6mm long round wax sprue with the reservoir. Each 8 wax patterns were sprued together and were painted with a surface tension reducing agent. Investing was done using a gypsum free phosphate bonded investment material (Gilvest MG, 0064759660, GmbH, Germany) which was mixed with a powder\liquid ratio as recommended by the manufacturer using vacuum mixing machine (Motova, Bego, Germany) and poured in to the casting ring. Size 10X iron casting ring was used for investing the wax patterns. After one

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hour, the casting ring was placed in an electric furnace (MANFREDI ,L7C, ITALY), and heated up to the temperature 300°C and held at that temperature for 30 minutes for burning out procedure, then the heat increased slowly to the temperature 850°C and held at that temperature for another 60 minutes. A multiple orifice oxygen gas torch was used to melt the alloy and the casting procedure was done using a compact manual-driven centrifugal casting machine using nickel-chromium alloy (Megaplug Compact, Dentarum, Liechtenstein). The composition of the alloy and the other materials used in the study are listed in Table 1.

After casting, the ring was bench cooled for two hours (according to manufacturer's instructions), then divested and the sprues were separated. The castings were cleaned and smoothed with stone bur and rubber bur in a slow speed straight handpiece. The specimens were treated according to the manufacturer's recommendation for the conventional feldspathic porcelain application (Ivoclar, Liechtenstein) opaque (Batch no.247043) and body (Batch no.147092). The alloy surface was blasted with aluminum oxide (50µm particle size, and 3-4 bar pressure) and the surface was cleaned by water and brush thoroughly. The specimens subjected to an oxidation cycle in the porcelain furnace (Ivoclar programat X1, Ivoclar, Liechtenstein) at 970°C for 7 minutes. Opaquer layers and dentine porcelain were applied to the samples in a modified syringe tube with 8mm diameter and 6 mm height. A thin wash layer of opaquer was applied, followed by a second opaquer layer and two dentine body layers, each of them fired separately according to the manufacturer's instruction. Thickness of porcelain was 2mm (±0.1mm) and the total sample thickness was 6mm (metal, opaquer, and porcelain).

Ceramic Surface Preparation Methods

Air Abrasion

All specimens were embedded in acrylic resin blocks except their examined surfaces, then they receive air abrasion treatment using Air flow hand-piece (EMS, Elctromedical system, SA, CH-1260-Nyon, Switzerland), a 5mm distance was maintained between the nozzle of air flow device and the sample surface in all specimens. With 50µm particle size of aluminum oxide⁽¹¹⁾, at air pressure 3.5 bar and water pressure of 0.7 bar. The air flow was applied to each specimen for 20 seconds⁽¹²⁾, then thoroughly rinsed for 15 seconds using air water spray and dried for 5 seconds by oil free air.

Sample Grouping (according to the type of acid and the use of silane)

Restorative Dentistry

The samples were equally divided in to 4 groups of 8 samples to receive different treatment:
Group I: Etching with 37% phosphoric acid gel for 15 seconds with silane.

Group II: Etching with 5% hydrofluoric acid solution for 2 minutes with silane.

Group III: Etching with 37% phosphoric acid gel for 15 seconds without silane.

Group IV: Etching with 5% hydrofluoric acid solution for 2 minutes without silane.

The specimens were thoroughly rinsed with water spray for 15 seconds and dried with air-oil free spray for 5 seconds. Following the manufacturer's directions one drop of Monobond-s bottle was dispensed on a plastic dish, with the aid of disposable brush, the solution was applied to the ceramic surface, and allowed to set for 60 seconds (for groups I and II only). Subsequently a thin layer of Heliobond was applied homogenously with the aid of disposable brush, it serves as bonding agent between the silane and the resin composite, the excess material was dispersed with oil free air and light cured for 20 seconds. Resin composite (Tetric Evo ceram, Ceramic repair, Vivadent /Ivoclar , Schaan /Liechtenstein) was applied to each specimen according to manufacturer's instruction with the use of a plastic transparent split mold (4mm diameter and 4mm height) specially designed for this purpose. The 1st increment of the composite was introduced with plastic instrument and adapted to avoid air entrapment, the material was light cured using light curing unit (Astralis 5, Vivadent, Austria) for 40 seconds, then the second layer was added and light cured for another 40 seconds. The tip of light curing unit was in intimate contact with the plastic cylinder mold. All specimens were stored in 37 °C distilled water for 24 hours before being thermocycled between 5°C and 55 °C for 100 cycles⁽¹³⁾ with a 30 seconds dwell time. After thermocycling, specimens were stored in distilled water for additional 8 days before being subjected to a shear load⁽¹⁴⁾. A universal testing machine (Zwick 1454 ,Germany) with 100kg load cell, and a 5mm/min cross head speed, and a chisel apparatus was used to direct a parallel shearing force as close as possible to the resin/ceramic interface (Figure 1).The shear load in Newton at the point of failure was noted, and calculated in MegaPascal's. Fracture sites were examined using stereomicroscope to determined the location and type of failures during debonding⁽¹⁵⁾. Mode of failure was recorded as adhesive (failure at the substrate-resin interface), cohesive (failure within the substrate), or combination (areas of adhesive)

Table 1: Characteristics, composition, and manufacturers of the materials used in the study

Materials	Characteristics and Composition	Manufacturer
Casting alloy	Ni 61.4 ,Cr 25.7 ,Mo 11.0 ,Si 1.5 ,Mn<1 ,Al<1.0 , C<1.0	Ivoclar/Vivadent ,Schaan, Liechtenstein
HF	Hydrofluoric acid 40% solution.	Thomas Baker, India. Lot No. 90290
Total etch	Acid etch gel, phosphoric acid (37wt. %), water, thickening agents and pigments.	Ivoclar/Vivadent ,Schaan, Liechtenstein. Lot No.146373
Mono bond-s	silane (primer agent between the ceramic & composite) 3-Meth acryloxypropyl-trimthoxsilane (1.0 wt.%) in a water/ethanol solution.	Ivoclar/Vivadent ,Schaan, Liechtenstein. Lot No. M42089
Heliobond	Light curing bonding agent, it contains Bis-GMA and triethylene glycol dimethacrylate (99wt.%) catalysts and stabilizers<1% .	Ivoclar/Vivadent ,Schaan, Liechtenstein. Lot No. M45479
Tetric Evo Ceram	light curing nano hybrid composite, the total content of inorganic fillers 53-55% vol, particle size ranges from40nm-3.000nm with a mean particle size of 550nm. <u>Fillers</u> :barium glass, ytterbium tri fluoride, mixed oxide and prepolymer(82-83% wt). <u>Matrix</u> : dimethacrylates(17-18% wt). <u>Additional contents</u> :additives ,catalysts, stabilizers and pigments	Ivoclar/Vivadent ,Schaan, Liechtenstein. Lot No. M340002

and cohesive failure). A one-way analysis of variance (ANOVA) was performed to test any statistically significant difference among the tested groups. Comparisons between each pair of the groups were performed by using the least significant difference (LSD) test.

RESULTS

Mean shear bond strength values (in Mpa), standard deviation, minimum and maximum

values for each group are presented in Table 2 and Figure 2.

Table 2: Mean shear bond strength values (in Mpa), standard deviation, minimum and maximum values for each group.

Groups	N	Mean	Std. Deviation	Minimum	Maximum
Group I	8	21.0175	2.15863	18.10	24.12
Group II	8	23.3088	1.89494	19.83	25.38
Group III	8	4.0025	0.62133	2.85	5.05
Group IV	8	18.6062	2.65550	15.05	22.62

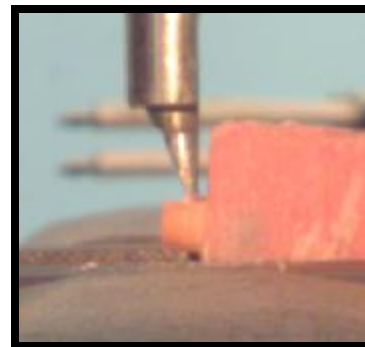


Figure 1: Shear force applied at the resin ceramic interface.

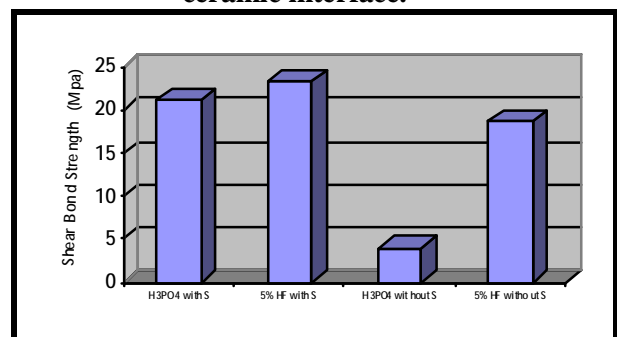


Figure 2: Bar chart shows the difference in mean shear bond strength values (Mpa) among groups.

Effect of Type of Acid Etching

Paired t-test was used and results revealed that specimens treated with 5% hydrofluoric acid with silane (group II) were statistically significantly higher than specimens treated with 37% phosphoric acid with silane (group I), and specimens treated with 5% hydrofluoric acid without silane (group IV) were statistically significantly higher than specimens treated with 37% phosphoric acid without silane (group III) as shown in Table 3.

Table 3: Paired t-test between corresponding groups to see the effect of type of acid.

Presence of Silane	Type of Etching	
	H ₃ PO ₄	5% HF
With Silane	←————→ 0.016 (S)	
Without Silane	←————→ 0.000 (HS)	

Effect of Silane

Paired t-test results revealed that group I (specimens treated with 37% phosphoric acid with silane) was statistically significantly higher than group III (specimens treated with 37% phosphoric acid without silane), and group II (specimens treated with 5% hydrofluoric acid with silane) was statistically significantly higher than group IV (specimens treated with 5% hydrofluoric acid without silane) as shown in Table 4.

Table 4: Paired t-test between corresponding groups to see the effect of silane.

Type of Etching	Presence of Silane	
	With Silane	Without Silane
H ₃ PO ₄	←————→ 0.000 (HS)	
5% HF	←————→ 0.005 (HS)	

Stereomicroscopic Examination

Stereomicroscopic examination of the fracture site revealed that when using silane, specimens treated with phosphoric acid exhibited 25% cohesive failure within ceramic and 75% adhesive/cohesive failures while those treated with hydrofluoric acid showed 100% cohesive failures within ceramic. However, when the silane was not used, specimens treated with phosphoric acid showed 100% adhesive failures and those treated with hydrofluoric acid showed 37.5% adhesive, 37.5% cohesive failures within ceramic and 25% combined adhesive/cohesive failures as shown in Table 5 and figure 3.

Table 5: Failure modes of the tested groups.

Groups	Adhesive	Cohesive	Adhesive/cohesive
Group I	----	25% *	75% **
Group II	----	100% *	----
Group III	100%	----	----
Group IV	37.5%	37.5% *	25% **

* Cohesive with in ceramic.

** Most of them Predominantly cohesive with in ceramic, partially adhesive.

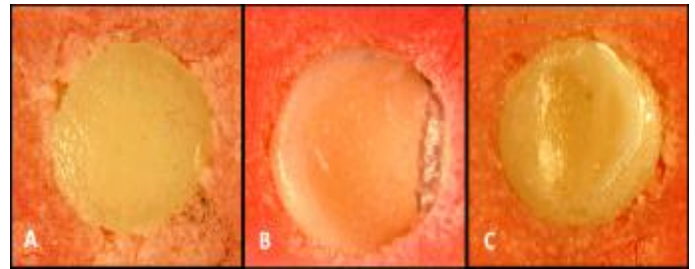


Figure 3: A. Adhesive failure between composite and ceramic, B. Cohesive failure within ceramic (extending to the underlying metal), C. Combined adhesive/cohesive failure.

DISCUSSION

Accuracy and clinical relevance of various in vitro bond strength testing methods for ceramic-composite bonding have been discussed extensively. Common tests are 3-point loading, tensile, microtensile, and shear tests⁽¹⁴⁾. The shear test was chosen for this study due to its simplicity and also due to the fact that anterior restorations are subjected primarily to shear stresses. In addition, the shear test is considered appropriate for quantifying the strength of porcelain repairs⁽⁷⁾.

Several methods of measuring in vitro resin-ceramic bond strength have been described. These include tensile and shear bond strength tests.

Type of Etchant

Etching of dental ceramics with HF acid or acidulated phosphate fluoride gel is a common procedure to achieve a clean microretentive surface before bonding or repairing of ceramic restoration⁽¹⁶⁾.

In principle, chemical etching agents dissolve the glass matrix selectively and cause physical alteration to promote adhesion of composite resin to the porous surface of fractured ceramic⁽¹⁷⁾.

The main crystalline component of dental porcelain is leucite (K₂O.Al₂O₃.4SiO₂). leucite dissolves more rapidly than the surrounding glass in HF acid, so etching produces microretentive channels in the porcelain where leucite had been. The number and size of such irregularities has been associated with increase in bond strength⁽¹⁶⁾.

In this study, groups treated with 5% HF acid showed higher retentive shear bond values compared to groups treated with phosphoric acid.

This finding of this study is in agreement with the results of Llobell et al.⁽¹⁸⁾, Aida et al⁽¹⁹⁾, and Tylka and Stewart⁽⁸⁾ who found significantly higher bond strengths with HF acid compared with phosphoric acid. This finding could be attributed to the fact that HF acid produces deeper

etch pattern with greater roughness on ceramic surface than other acid etching agents⁽¹⁷⁾.

Canay et al.⁽¹⁶⁾ studied the effect of different acids on the surface texture of feldspathic porcelain. They found that HF acid etched patterns appeared more pronounced and aggressive than those of other acids resulting in the production of small channels throughout the ceramic surface. The surface is uniformly porous with numerous distinguishable microundercuts.

Effect of Silane

Silanes function as mediators and promote adhesion between inorganic and organic matrices through dual reactivity⁽²⁰⁾.

These silane coupling agents are bifunctional, i.e. they have dual reactivity. The non-hydrolysable functional group with a carbon-carbon double bond can polymerize with resin composite monomers containing double bonds. The hydrolysable alkoxy groups react with a hydroxyl group rich ceramic surface⁽²⁰⁾.

The bond with ceramic occurs via a condensation reaction between the silanol group (Si-OH) of the ceramic surface and the silanol group of the hydrolyzed silane molecule, creating a siloxane bond (Si-O-Si) and producing a water molecule (H₂O) by-product.

Silanes enhance porcelain- resin bonds by promoting the wetting of the ceramic surface and thus making the penetration of the resin into the microscopic porosities of the acid conditioned porcelain more complete⁽²¹⁾.

In this study, groups treated with silane showed higher shear bond values compared to those treated with acids only. The effect of silane is greatly pronounced for phosphoric acid treated groups where the mean shear bond strength value jumped from less than 5MPa to more than 20 MPa after the use of silane. This finding agrees with the results of Shahverdi et al.⁽²²⁾, Filho et al.⁽²¹⁾, and Panah et al.⁽²³⁾.

Mode of Failure

The definition of adhesion zone is critical in classification of the mode of failure, which should be an integral component of all failure analysis.

A careful microscopic analysis of the fracture surface can produce a more consistent and complete description of the fracture process. Thus the quality of the bond should not be assessed based on bond strength alone. The mode of failure could provide important information about the clinical performance limit, which is the ultimate test of any adhesive system⁽²³⁾.

Stereomicroscopic examination of the fractured surfaces revealed that all specimens treated with H₃PO₄ acid alone, the adhesive layer separated from the ceramic substrate totally (100% adhesive

failure). However, specimens treated with HF alone showed the three types of failure modes (adhesive, cohesive, and mixed).

In silane-coated groups, no complete adhesive failure was noticed with the fractures occurred totally cohesive (within ceramic substrate) in HF acid group; and either cohesive (within ceramic) or mixed in phosphoric acid group.

This indicated that HF acid created deeper and more retentive surface compared to phosphoric acid.

The application of silane coupling agent to the ceramic surface provide a chemical covalent and hydrogen bond of resin systems to ceramic and is significant factor for sufficient resin bond to ceramics⁽²³⁾.

These results are consistent many previous studies^(5, 24, 25).

Despite the statistical difference, the variation between the bond strength values of the tested groups (except for group III, treated with phosphoric acid alone) was small (18.6-23.3 MPa).

This may be explained by the fact that all groups presented ceramic cohesive failures, indicating that the bond strength between the repair material and the ceramic substrate was superior to the strength of the substrate itself. This behavior was also reported in the literature^(5, 7, 8, 14, 19, 26, 27).

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