Effect of Different Ceramic Surface Treatments on Resin Microtensile Bond Strength

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<u>Purpose</u>: The objective of this study was to evaluate the effect of different surface treatments on the microtensile bond strength (μ -tbs) of composite bonded to hot-pressed ceramic. The null hypothesis tested was that neither of the surface treatments (silanization or fluoric acid etching) would produce greater bond strength of composite resin to the ceramic.

<u>Materials and Methods</u>: Four $7 \times 7 \times 5$ mm hot-pressed ceramic blocks of IPS Empress 2 were fabricated and polished to 600 grit followed by sandblasting with 50 μ m alumina. The ceramic blocks were then divided into four groups and submitted to the following surface treatments: Group 1: 9.5% hydrofluoric (HF) acid for 20 seconds and silane (S) for 3 minutes; Group 2: silane for 3 minutes; Group 3: 9.5% HF acid for 20 seconds; Group 4: no treatment. Scotchbond adhesive was applied to the treated ceramic surfaces and covered with Filtek Z250 composite resin. The composite-ceramic blocks were cut with an Isomet low speed diamond saw machine producing sticks (n = 25), which were loaded to failure under tension in an Instron Universal testing machine. The mean μ -tbs was analyzed with one-way ANOVA and Bonferroni "t" test.

<u>Results</u>: All specimens of Group 4 experienced adhesive failure during the cutting of the block and were eliminated. The mean μ -tbs and standard deviations (SD) in megaPascals were: Group 1 = 56.8 (±10.4), Group 2 = 44.8 (±11.6), Group 3 = 35.1 (±7.7). Statistical analysis showed that the bond strength was significantly affected by surface treatment (p < 0.0001). Group 1 (HF + S) had the highest μ -tbs, and Group 2 (S) had higher μ -tbs than Group 3 (HF). The mode of fracture of the specimens was examined using scanning electron microscopy (SEM), and all fractures occurred within the adhesion zone.

<u>Conclusion</u>: The results show that surface treatment is important for resin adhesion to ceramic and suggest that silane treatment was the main factor responsible for resin bonding to ceramic.

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THE GROWING use of metal-ceramic restorations, beginning in the 1970s, resulted in the need for repair of some of those restorations. The

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material of choice for repair has been resin composite due to esthetics, low cost, and easy handling. Specific repair bonding systems, including silane primers, with their capacity for improving chemical bonds with organic and inorganic surfaces, were developed in the late 1970s.^{1,2}

The dentist's ability to bond composite resin to enamel, dentin, and ceramic has revolutionized the practice of restorative dentistry. The introduction of ceramic acid etch technique in the early 1980s allowed the first adhesive ceramic restorations to be made on anterior teeth.³ The use of ceramic as a restorative material in substitution for composite resin and metal-ceramic restorations has increased substantially. This trend can be attributed to the development of new compatible ceramic systems^{4,5} and strong, reliable adhesion provided by new bonding luting systems for placing

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ceramic restorations or repairing the fractured ceramic material. One of these new ceramic systems is IPS Empress 2, which allows the fabrication of all-ceramic crowns and three-unit fixed partial dentures.

Because of their brittle nature, all-ceramic restorations rely on adequate bonding. The adhesion of ceramic to dental structure with composite resin luting materials increases the fracture resistance of the tooth and the restoration itself; it also minimizes microleakage, which may be the determining factor in the success or failure of the treatment.⁶ Microleakage may cause marginal pigmentation of restorations or repairs, producing initial esthetic improvement and later unsuccessful adhesive failure.

Predicable bond between resin composite and ceramic is usually created by two mechanisms: micromechanical attachment provided by hydrofluoric (HF) acid etching and/or gritblasting and chemical bond by a silane-coupling agent. Hydrofluoric acid attacks the glass phase of ceramics, creating surface microporosity, which allows the formation of mechanical interlock with composite resin. This physical bond combines with the chemical bond obtained from the use of a silane agent to provide a high strength bond between the composite and ceramic. Hydrofluoric acid is poisonous and caustic, however, and represents a potential health hazard due to its toxicity and volatility.7 During the clinical procedures of adhesive fixation and mainly intraoral repairing of ceramic restorations, the dentist and patient may be exposed to this risk. The published etching time for HF acid or its substitutes has ranged from 60 seconds to 20 minutes.^{3,8-10} A reduction of time used to etch the ceramic surfaces or the elimination of this stage could be beneficial to dentist and patient.

The bond strength promoted by acid etching or silane-coupling agents can be influenced by ceramic microstructure.^{11,12-14} Sorensen et al¹⁵ reported that silane application had no significant effect on ceramic bond strength. Shimada, Yamaguchi, and Tagami¹⁶ reported that HFetching glass ceramic adversely affects ceramic bonding and is probably not necessary on clinical applications. The lithium disilicate based IPS Empress 2 is a new ceramic material; consequently, specific pretreatment methods may be necessary prior to adhesion. The purpose of this study was to evaluate the influence of HF acid and a silane-coupling agent on the microtensile bond (μ -tbs) strength between composite and IPS Empress 2 ceramic. The null hypothesis tested was that neither of the ceramic surface treatments (silanization or fluoric acid etching) would increase the bond strength to composite resin.

Materials and Methods

Four $7 \times 7 \times 5$ mm hot-pressed lithium disilicate based ceramic blocks (IPS Empress 2, shade 500, lot no. C07483, Ivoclar, Schaan, Liechtenstein) were fabricated using the lost wax technique and by pressure injection of ceramic ingots in the EP 500 furnace (Ivoclar). The ceramic blocks were air-abraded with aluminous oxide at 2 bar to eliminate investment.

The block's bonding surfaces were machined flat, for surface standardization, with No. 220, 360, and 600 grit silicon-carbide abrasive paper (Acqua Flex-Norton, Sao Paulo, SP, Brazil), under continuous coolant water irrigation, washed in water and dried with oil free compressed air. The flattened surface of each ceramic block was sandblasted with 50 μ m aluminum oxide particles for 15 seconds at a distance of 10 mm with a microetcher intraoral sandblasting device (Microetcher; Danville Engineering, Danville, CA), operating at 35 psi and ultrasonically cleaned in distilled water for 10 minutes to ensure a contaminant free ceramic surface. The blocks were then assigned to four groups which received the following surface treatments:

- 1. Group 1 (G1): HF acid (Ultradent Porcelain Etch 9.5% Buffered, lot no. C123, Ultradent Products, South Jordan, UT) applied for 20 seconds, rinsed for 30 seconds, dried with compressed air oil-free for 30 seconds, and followed by application of silane (RelyX Ceramic Primer, lot no. 0RH, 3M Dental Products, St. Paul, MN). The primer was applied with a minisponge, allowed to evaporate for 3 minutes and air-dried for 30 seconds.
- 2. Group 2 (G2): Silane only applied and allowed to evaporate for 3 minutes and air-dried for 30 seconds.
- 3. Group 3 (G3): Hydrofluoric acid only applied for 20 seconds, rinsed for 30 seconds, and dried for 30 seconds.
- 4. Group 4 (G4): No surface treatment.

Scotchbond adhesive (Scotchbond Multi-Purpose Plus Adhesive, lot no. 1MH, 3M Dental Products) was applied to the prepared surface of each ceramic block and light cured at 450 mW/cm² (Optilux, Demetron Research Corporation, Orange, CA) for 20 seconds. Following the adhesive application, composite resin (Filtek Z250, lot no 1KT, shade A3, 3M Dental Products) was placed on the ceramic surface in four 1-mm-thick layers and each layer light cured as above for 40 seconds. Each composite-ceramic block was attached to a cylindrical acrylic resin base with cyanoacrylate adhesive (Super Bonder, Loctite, Sao Paulo, SP, Brazil) and stored in distilled water at 37°C for 24 hours before being sectioned.

The composite-ceramic block was then mounted in a slow-speed sectioning saw (Isomet 1000, Buehler, Lake Forest, IL) regulated to cut 0.9 mm wide slices with a 0.3 mm diamond-disc under running water (Model 650, South Bay Technology, San Clemente, CA). The first cuts through the composite-ceramic block produced six slabs of approximately 0.9 mm width. A second set of cuts made perpendicular to the first cuts converted the slabs into 36 bars (bar shaped testing specimens) approximately 0.9 mm \times 0.9 mm in transverse cross section (Fig 1). The Group 4 specimens experienced adhesive failure during the first cutting procedure and were eliminated.

Twenty-five bars were randomly selected for testing from each group for microtensile strength tests. Before testing, the dimensions of each test bar were measured with a digital micrometer (Mitutoyo Corp., Kawasaki, Kanagawa, Japan) and the bonded surface area of the specimen calculated; this surface area was used to calculate the bond strength. Each bar specimen was glued to the flat grips of the Bencor Mult-T device (Danville Engineering, San Ramon, CA) with cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA) (Fig 2) and subjected to tensile forces until failure at a



Figure 1. Schematic illustration of specimen preparation for microtensile test. (A) Ceramic block; (B) composite-ceramic block; (C) the first cut slices perpendicular to the adhesive surface; (D) second cut, perpendicular (90°) to first cut; (E) bar specimens.



Figure 2. Bar specimen attached to the Bencor Mult-T device.

crosshead speed of 0.5 mm/min using a Universal testing machine (Instron model 4444, Canton, MA). Bending forces were avoided by gluing specimens in the most parallel possible form and in contact with the Bencor device. The rupture force was gauged in Newtons and then divided by the transverse section area of each sample, and expressed in MegaPascals (MPa). Some specimens debonded upon attachment to the flat grip of the Bencor Mult-T and were eliminated; therefore, the number of specimens per group is different. Microtensile bond strength data were then analyzed statistically with oneway ANOVA and Bonferroni "t" test. Once broken, the two fragments of each sample bar were mounted in acrylic bases and coated with palladium/gold (Bal-Tec SCD 005, Balzers, Liechtenstein) to be examined using scanning electron microscopy (Philips XL 30, Philips Electronic Instruments Inc., Mahwah, NJ). The failure modes were recorded as: adhesive (if one fracture site was at the composite or ceramic surface and the other site remained adhesive only); cohesive in adhesive layer (fractures extending through the adhesive); cohesive in composite (failure totally within composite) or cohesive in ceramic (failure totally in ceramic); and mixed failures (failure including at least two of these materials).

Results

The mean microtensile bond strengths (μ -tbs) and standard deviation (SD) in MPa were: G1 = 56.8 (\pm 10.4), G2 = 44.8 (\pm 11.6), G3 = 35.1 (\pm 7.7). The means, SD, number of specimens per group (n), and coefficient of variation (CV) of the μ -tbs test for groups 1 to 3 are shown in Table 1.

One-way ANOVA revealed that the bond strength was significantly affected by the surface

Groups	Group 1	Group 2	Group 3
	(HF + S)	(S)	(HF)
Means	56.8^{a}	44.8 ^b	35.1°
SD	10.4	11.6	7.7
N	18	21	22
CV	18.3	25.8	21.8

Table 1. Mean Microtensile Strength (μ -tbs) in MPa, Standard Deviation (SD), Number of Specimens Per Group (N), and Coefficient of Variation (CV)

Means with different letter are significantly different at $\rho \leq 0.05.$

treatment (F = 22.99, p < 0.0001). The Bonferroni "t" multiple comparative test showed that the G1 (HF + S) specimens had significantly higher bond strengths than G2 (S only) (p = 0.0002) and G3 (HF only) (p < 0.0001), and G2 showed significantly higher bond strengths than G3 (p =0.0012). This result suggests that silane was the main reason G1 had higher bond strength. All Group 4 specimens broke upon making the second cuts in the blocks and were eliminated.

SEM analysis did not reveal entirely cohesive failure in composite or ceramic. Groups 1 and 3 exhibited predominantly mixed fracture failures (Figs 3 and 4) whereas Group 2 exhibited predominantly adhesive failure (Fig 5).

Discussion

Several methods of measuring in vitro resinceramic bond strength have been described. These include tensile¹⁷ and shear bond strength tests.¹⁸⁻²⁰ Shear strength tests, in spite of being the most frequently used in testing bond strength, have had their efficacy questioned because they do not always represent the true stress generated in an interface.^{21,22} Della Bona and Van Noort²³ observed that the bond strength on resinceramic shear tests is governed by the cohesive strength of the base material used rather than the adhesive interface bond strength, due to the high tensile stress developed within the porcelain next to where the load is applied, causing cohesive fracture under very low forces. Tensile bond strength tests, on the other hand, provide a more representative measurement of the tensile bond strength of the bonding area and all failures occurred within adhesive interface.²³

Sano et al²⁴ developed the microtensile test with the objective of evaluating the adhesive performance of small bonded surface areas. The authors observed that tensile bond strength was inversely related to bonded surface area; in other words, smaller surface areas were associated with higher tensile bond strengths, whereas larger surface areas showed lower tensile bond strengths. In that study, all the bond failures at these small bonded areas were adhesive.

In the present study, the microtensile test effectively permitted higher fracture load values when compared to studies from other authors who used shearing tests.²⁵⁻²⁸ SEM analysis showed that all fractures occurred within the interface, classified as either adhesive or mixed failures; no entirely cohesive failure was recorded.





Figure 3. Group 1 (HF + S): (*A*) side of resin of failed bar specimen; (*B*) side of ceramic. The photomicrographs show an example of mixed fracture, typical of this group: cohesive failure of resin (CR), cohesive failure of ceramic (CC), and cohesive within adhesive (A).



Figure 4. Group 3 (HF): Mixed failure typical of this group. (A) Side of resin-adhesive failure, characterized by layer of adhesive (A) recovering the composite, and cohesive failure of resin (CR). (B) The ceramic side of the fractured specimen is a mirror image-cohesive failure of resin (CR), and remnants of adhesive (arrows) attached at etching ceramic surface (C).

Statistical analysis showed a significant difference (p < 0.001) between the three studied groups; Group 1 (G1, HF + S) had bond strengths greater than both Group 2 (G2, S only) and Group 3 (G3, HF only) and G2 had greater bond strengths than G3. This suggests that in spite of the HF acid effectively improving resin-ceramic adhesion, silane had greater influence in the bond strengths found for G1 than HF acid. Other authors testing different ceramics reached similar conclusions.²⁶⁻³³ The HF acid attacks the glass phase of ceramics, partially dissolving it and creating microporous retention by exposing areas of crystals which make up the crystalline phase of the material.³⁴ Microporosity increases the surface area and makes micromechanical interlocking of resin possible.35 Although various acid solutions can be used for this purpose, HF acid has been shown to be the most effective one.³⁶⁻³⁹

Silane-coupling agents are adhesion promoters, capable of forming chemical bonds with organic and inorganic surfaces. Bonding to the resin occurs by an addition polymerization reaction between methacrylate groups of the matrix resin and the silane molecule during curing of the composite. The bond with ceramics occurs via a condensation reaction between the silanol group (Si-OH) of the ceramic surface and the silanol group of the



Figure 5. Group 2 (S): Adhesive failure typical of this group (failure at adhesive-ceramic interface). (A) Side of composite; laver of adhesive (A) is observed, recovering the composite. (B) Side of ceramic; failure at ceramic surface (C). Remnants of adhesive are observed attached at ceramic surface (arrows).

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hydrolyzed silane molecule, creating a siloxane bond (Si–O–Si) and producing a water molecule (H₂O) byproduct.⁴⁰ Silanes also 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.^{2,31}

A recent work, using a design similar to that of the present study, showed bond strengths of composite resin to IPS Empress 2 ceramic, similar to ours, when the ceramic surface was treated with HF acid and silane.¹⁴ However, when silane and acid treatments were applied individually, a greater bond strength was registered for the acid-conditioned group. In addition to the isolated use of the HF acid, the longer etching time (2 min) employed in that work¹⁴ is probably responsible for the higher bond strengths. Nevertheless, studies exist showing that a longer etching time does not always correspond to greater bond strengths, particularly where a silane agent is applied over etched ceramics.^{32,41} Chen, Matsumura, and Atsuta,⁴¹ studying the effect of different HF acid etching times (without silane) on the resin-ceramic bond strength (using a machineable ceramic), also found a higher value for a 2 minute etch. In a subsequent work,³⁰ however, the same authors noticed that an extension of the acid etching of the ceramic surface might not be necessary, and suggested that 30 seconds would promote adequate bond strength when a silane agent is used simultaneously. Such findings uphold the present results, as in spite of having used longer periods of acid etching, Della Bona, Anusavice, and Shen could not find greater bond strength than those in our study when acid and silane (G1) were used.¹⁴ That implies that the 20-second HF acid etching time used in the present work would be sufficient to yield maximum bonding potential as long as silane is also used. Clinical time would be saved and risks to patient and clinician health, from longer exposure to HF acid, would be minimized. Silanization of porcelain, following etching with HF acid, has been indicated by several authors as a routine procedure indispensable to secure a reliable bond for the long term.^{27,31,33,42}

On intraoral repair procedures for fractured ceramic restorations, the use of silane bonding agents without previous etching with HF acid might be an interesting and desirable option, particularly where dentin exposure exists, a clinical circumstance where HF acid is to be avoided.⁴³

Such a possibility is clearly indicated in a study by Aida, Hayakawa, and Mizukawa.⁸ Bond strength values obtained in the present study for Group 2 (S only), where a pre-hydrolyzed one-bottle silane agent was used, also support this conclusion, despite not studying thermocycling and long-term water storage.

Ceramic silanization is a sensitive step in the adhesive procedure, capable of being affected by various factors; contaminants such as water or other solvents may decrease the bond strength.⁴⁴ Storage time between preparation of the test specimen and the test itself may affect the chemical bond strength instituted by silane, although not equally for different silanes.^{28,44,45} Barghi, Berry, and Chung demonstrated that different silanes vield different bond strength values.⁴⁶ These authors also found differences in bond strengths following thermal treatment of silanized porcelain using two-bottle silanes, whereas prehydrolized single-bottle silanes were not affected by this procedure. Different silanes may present in their composition different solvents which will influence their reactivity and stability in various ways.⁴⁷ The relative potential of a silane agent may be estimated by the quantity of silanol groups (active sites of hydrolyzed molecules of silane) available for bonding with the silanol group of porcelain, such that a positive correlation exists between the degree of hydrolysis of silane and the adhesive bond strength.⁴⁹ Anagnostopoulos, Eliades, and Palaghias⁴⁸ found a greater number of silanol groups available to react with the ceramic surface with prehydrolized single-bottle silanes compared with two-bottle silanes in which hydrolysis occurs with the aggregation of the two components (silane and hydrolysis-activating acid) moments before application. This form of silane activation introduces an operator variation possibility, and incomplete hydrolysis eventually can occur with decreased bond strength values.⁵⁰ Therefore, the use of a single-bottle prehydrolized silane, as in the present study, makes the procedure easier and reduces error. The compatibility of the materials used^{51,52} and sandblasting the surfaces prior to silanization may also have influenced results in a positive manner.

Since Group 4 samples in the present work had resin separated from ceramic when the blocks were sectioned to prepare test specimens, sandblasting, by itself, without other surface treatment, seems an ineffective procedure. On the other hand, ceramic silanization enhances bond strength more than any mechanical retention created by sandblasting or by HF acid etching treatment and seems to be an important and indispensable step in bonding composite to ceramic procedures. Further laboratory tests are to be conducted, using a similar methodology but with thermocycling and storage in an aqueous medium for an extended period, to investigate how this would influence the bond strength.

The present study demonstrates the importance ceramic surface treatment has for resinceramic bond strength.

Conclusion

Within the limits of this study, the following conclusions were obtained:

- *1.* Ceramic silanization was individually the most significant factor responsible for the bond strength.
- Acid etching and silanization promoted maximum bond strength in the composite resin— Empress 2 IPS glass ceramic union.
- 3. Sandblasting itself did not provide adequate bond strength.

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