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Effect of Surface Treatment on the Shear Bond Strength of a Resin-Based Cement to Porcelain

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The purpose of this *in vitro* study was to evaluate the effect of different surface treatments on the shear bond strength of a resin-based cement to porcelain. Sixty pairs of 50% aluminous porcelain discs were fabricated. In each pair, one disc measured 6 mm in diameter X 3 mm thickness (A) and the other measured 3 mm in diameter X 3mm thickness (B). The specimens were randomly assigned to 6 groups (n=10 pairs of discs), according to the surface treatment: etching with 10% hydrofluoric acid for 2 or 4min (G1 and G2); 50-µm particle aluminum oxide sandblasting for 5 s (G3); sandblasting followed by etching for 2 or 4min (G4 and G5) and control - no treatment (G6). A silane agent was applied to the treated surface of both discs of each pair. Bistite II DC dual-cure resin cement was applied and the B discs were bonded to their respective A discs. Specimens were stored in distilled water at 37ºC for 24 h and were tested in shear strength at a crosshead speed of 2 mm/min. Means in MPa were: G1: 14.21 ± 4.68; G2: 8.92 ± 3.02; G3: 10.04 ± 2.37; G4: 12.74 ± 5.15; G5: 10.99 ± 3.35; G6: 6.09 ± 1.84. Data were compared by one-way ANOVA and Tukey's test at 5% significance level. Bond strength recorded after 2-min acid etching was significantly higher than 4-min etching ($p<0.05$) and control ($p<0.05$), but did not differ significantlyfrom sandblasting alone ($p>0.05$) or followed by etching for 2 or 4 min ($p>0.05$). Within the limitations of an *in vitro* study, it may be concluded that 2-min hydrofluoric acid etching produced a favorable micromechanical retention that enhanced resin cement bond strength to porcelain.

Key Words: porcelain, resin-based cement, surface treatments, cementation.

INTRODUCTION

When cavities exceed the limits recommended for direct composite fillings, indirect restorations employing tooth-colored materials have been indicated in order to minimize the effects of polymerization shrinkage and provide and adequate proximal contouring. The indication of indirect procedures is based on the possibility of reinforcing the remaining dental structure by means of adhesive techniques.

The use of ceramic restorations became widespread due to the possibility of bonding these restorations to enamel and dentin with an adhesive system. Aluminous porcelain presents 40-50% more aluminum oxide crystals than feldspathic porcelain, increasing the hardness and reducing the thermal expansion coefficient. This kind of material is indicated for laminate veneers, inlays/onlays and as covering material for porcelain crowns.

The increasing use of ceramic restorations has led to the development of a wide array of resin cements in order to provide improved esthetics and optimal bond strength. The clinical performance of ceramic restorations, however, is directly related to the properties and type of luting agent employed.

A high-quality adhesion of the resin cement to tooth structure and restoration surface is primordial for the success of ceramic bonding. Different surface treatments on ceramic surface have been recommended to enhance this adhesion, such as sandblasting, etching with different acids and grinding with diamond burs. All of these procedures are intended to improve the bond

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strength by producing micromechanical retention and thus modifying the porcelain surface texture (1).

In addition to this mechanically retentive surface, the use of silane provides a chemical interaction, which is attributed to its bifunctional characteristic. A high proportion of porcelain's allows reaction of the silane agent both to the crystal portion of the treated porcelain and to the organic portion of the luting agent (2).

The application of a silane between ceramic and resin composite provides an effective chemical bonding (3). Nevertheless, several authors consider etching of the ceramic surface as the critical point on the bond strength of resin cement to porcelain (4-7).

The purpose of this study was to evaluate the effect of sandblasting and/or two etching cycles with 10% hydrofluoric acid on the shear bond strength of a resin-based cement to porcelain.

MATERIAL AND METHODS

A total of 120 fifty-percent aluminous porcelain discs (Vitadur Alpha; Vita Zahnfabrik, Bad Säckingen, Germany) were fabricated: 60 discs had 6 mm in diameter X 3 mm thickness (A) and the other 60 discs had 3 mm in diameter X 3 mm thickness (B). The discs with smaller diameter (B) were bonded to the discs with larger diameter (A) for the shear bond strength testing. In addition, 6 discs (A) were selected for a complementary analysis under scanning electron microscopy (SEM).

Ceramic was prepared and packed in an aluminum matrix and excess liquid on the surface was removed with an absorbent paper. Condensers were used to provide a uniform packing of the ceramic and to remove the discs from the matrix. These specimens were positioned in an automatic vacuum furnace (Wizard; Jelrus Technical Products Corp., New Hyde Park, NY, USA) for porcelain firing. Due to ceramic contraction, the discs were repositioned in the matrix for adjustment of their dimensions. Second and third firings were performed subsequently.

All 120 ceramic discs were ground on wet 240, 400- and 600- grit silicon carbide papers to obtain a smooth flat surface, and were then ultrasonicated in distilled water for 5 min. The 60 discs with larger dimensions (A) were embedded in chemically cured acrylic resin (Jet; Clássico Artigos Odontológicos Ltda., São Paulo, SP, Brazil) using a silicon mould in order to obtain a plane base for the shear testing.

Thereafter, the discs with larger (A) and smaller (B) diameters were randomly assigned to 6 groups, being 10 discs of each size *per* group. The bonding surface of the discs received different surface treatments, as follows: G1: Etching with 10% hydrofluoric acid for 2 min; G2: Etching with 10% hydrofluoric acid for 4 min; G3: Sandblasting with aluminum oxide (50 µm) for 5 s; G4: Sandblasting + 10% hydrofluoric acid etching for 2 min; G5: Sandblasting + 10% hydrofluoric acid etching for 4 min; G6: No treatment (control group). After surface treatment, the specimens were washed in distilled water for 40 s, ultrasonicated in distilled water for 1 min and air-dried. A silane agent (Tokuso Ceramic Primer; Tokuyama Dental Corp., Tokyo, Japan) was then applied to the bonding surfaces of the discs. The silane agent was mixed thoroughly in equal parts of Primer A and Primer B. After 3 min, a thin layer of silane was applied and after 10 s a gentle air spray was applied following a second layer application.

A modified paralelometer (Bio Art Equipamentos Odontológicos Ltda., São Carlos, SP, Brazil) was used to standardize the cementation procedure. A fixed horizontal arm was adapted to the paralelometer, the spring of the vertical movable portion was removed, and a plane tip was coupled to the vertical arm to maintain the disc to be cemented in position. The weight of the vertical arm produced a static, constant load of 300 g applied for 1 min to standardize resin cement thickness.

Pastes A and B of Bistite II DC dual-cure resin cement (Tokuyama Dental Corp., Tokyo, Japan) were mixed in equal parts and a thin layer of cement was applied to the bonding surface of A and B discs. Disc B was positioned and stabilized on the disc A and cement excess was removed with a brush. Light curing was performed for 40 s in four different directions, according to the sides of the acrylic resin block, totalizing 160 s. For complete polymerization, Air Barrier (Tokuyama Dental Corp., Tokyo, Japan) was applied to the margins and the discs were maintained in position for 5 min.

The specimens were stored in distilled water at 37°C for 24 h. Shear bond strength was tested in a universal testing machine (DL2000; Emic Equipamentos e Sistemas de Ensaios Ltda., São José dos Pinhais, PR, Brazil) at crosshead speed of 2 mm/min. Data were analyzed by one-way ANOVA and Tukey's test at 5% significance level.

After shear testing, the debonded surfaces were examined under a stereomicroscope (Stemi 2000C; Carl

Zeiss Inc., Thornwood, NY, USA) at X16 magnification. Failure mode at resin cement/porcelain interface in each sample was determined. Failures were classified as follows: a) *adhesive failure*: all cement dislocate from the ceramic; b) *cohesive failure of the cement*: fracture inside the cement layer, with a thin layer of cement adhered to the ceramic; c) *cohesive failure of the ceramic*: fractured ceramic adhered to the cement; d) *mixed failure*: combination of adhesive and cohesive fractures and part of the cement adhered to the ceramic.

Table 2. Failure modes for the 6 surface treatments.

Surface treatment	Failure mode			
	Adhesive	Cohesive cement	Cohesive ceramic	Mixed
HF (2 min) (G1)			9	1
HF (4 min) (G2)			6	4
Sandblating (G3)			7	3
Sandblasting + HF (2 min) (G4)			8	2
Sandblasting + HF (4 min) (G5)			6	4
Control (no treatment) (G6)	2		5	3

HF = Hydrofluoric acid.

In order to perform a qualitative micromorphologic examination of porcelain surface, one disc (6 mm x 3 mm) from each group was sputter-coated with gold and analyzed using a scanning electron microscope (JSM-5310; JEOL, Peabody, MA, USA) at 15 kV. Photomicrographs of representative areas were taken at X500 magnification.

RESULTS

Data were first examined by one-way ANOVA test (F 7.90; p=0.001) and statistically significant differences were found. Multiple comparisons by Tukey's test set up 3 homogeneous groups with statistically similar shear bond strength (Table 1).

The failure modes are given on Table 2. Figure 1 shows a set of SEM micrographs representative of the 6 groups after surface treatment.

Table 1. Shear bond strength means in MPa (±SD) for the 6 surface treatments.

Surface treatment	Means ± SD
HF (2 min) (G1)	14.21 ± 4.68a
Sandblasting + HF (2 min) (G4)	12.74 ± 5.15ab
Sandblasting + HF (4 min) (G5)	10.99 ± 3.35 ab
Sandblasting (G3)	10.04 ± 2.37 ab
HF (4 min) (G2)	8.92 ± 3.02bc
Control (no treatment) (G6)	6.09 ± 1.84c

Different letters indicate statistically significant difference at 5%. HF = Hydrofluoric acid.

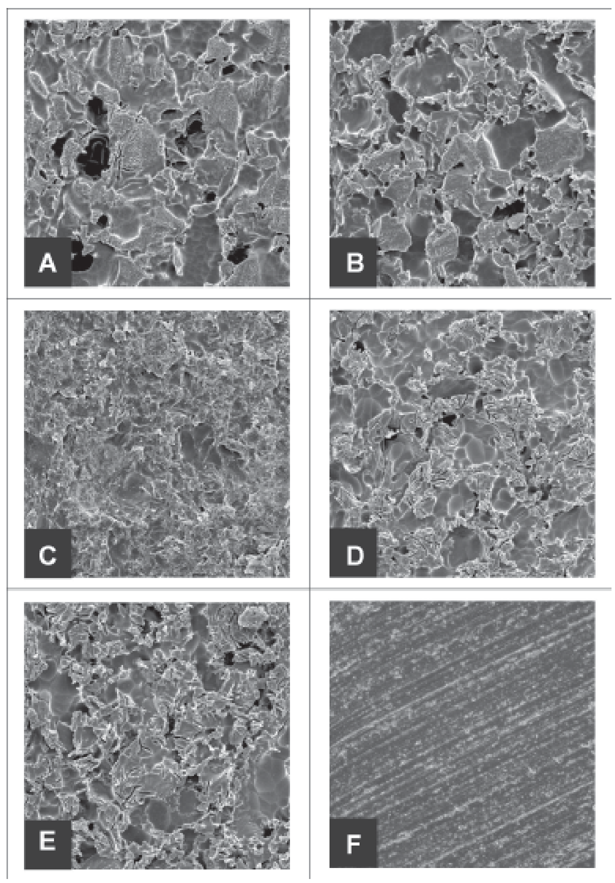

Figure 1. Comparative pannel showing a set of SEM micrographs representative of porcelain appearance after the surface treatment performed in each experimental group. (A) Hydrofluoric acid etching (2 min); (B) Hydrofluoric acid (4 min); (C) Sandblasting; (D) Sandblasting + Hydrofluoric acid (2 min); (E) Sandblasting + Hydrofluoric acid (4 min); (F) Control (no treatment). Original magnification X500.

DISCUSSION

For a reliable and satisfactory union between ceramic and resin cement, a combination of chemical and mechanical retention must occur. Porcelain surface treatments modify its texture, increasing the micromechanical retention of the resin cement. Chemical retention is achieved with the use of silane agents that reacts with the vitreous compounds of the ceramic and with the composite organic matrix (2).

Since the concept of etching porcelain surface was introduced and adhesive cementation of porcelain laminate veneers was reported, several authors have demonstrated that the concentrations and etching periods must be adjusted to each specific type of ceramic in order to optimize the bond strength (1,4,5,8,9).

An alternative for creation of micromechanical retention is aluminum oxide sandblasting. This technique was included in the present study for being a commonly employed procedure in prosthodontic laboratories and due to its adaptation to dental offices by means of the use of miniaturized devices, which facilitates the use of this technique (10,11).

The mechanical retention provided by surface treatment is of paramount importance for proper adhesion. However, the association with a chemical procedure (silanization) is required for better results (5,7-9,12-14). For this reason, a silane agent was used in all specimens before bonding in the present study.

The high percentage of silica in porcelain allows the chemical bonding of silane and resin cement to the ceramic. This occurs due to the hydrolysis and adsorption of silane in the ceramic surface, and the covalent bond with the resin cement. Moreover, silanization increases the wettability of ceramic surface, facilitating the spreading of resin cement that fulfils the superficial irregularities (2).

The ceramic primer employed in this study (Tokuso Ceramic Primer) is composed by two solutions that contain a γ-MPTS based silane and an acidic phosphate monomer that promotes the catalysis of silane reaction. The acidification of ceramic surface leads to the formation of hydroxyl groups (OH^-), which bond to the silane, thus enhancing the creation of siloxane bonds and facilitating the composite-ceramic adhesion (9,15,16).

In the present study, etching with hydrofluoric acid (Figs. 1A and 1B) produced remarkable morphological alterations on ceramic surface, which presented a porous and dendritic appearance, sufficient for creating micromechanical retention. This is in accordance with the results of previous studies (4,16-19).

The highest shear bond strength was obtained by etching for 2 min (G1), this group being statistically different only from G2 (etching for 4 min) and G6 (control). Although it has been reported that it is hard to etch aluminous porcelain due to alumina resistance to chemical treatment (20), the findings of the present study showed that a 2-min etching time was sufficient to obtain high bond strength values.

The increase of the etching time in G2 (Fig. 1B) produced a more irregular surface than that observed in G1 (Fig. 1A), which is consistent with findings of previous works (1,4,6). Sandblasting (G3) produced an irregular surface with angular appearance, leaving easily identifiable aluminum oxide fragments on ceramic surface (Fig. 1C). This appearance is due to microfractures resulting form the impact of alumina particles onto ceramic (11,19).

According to Della Bona and Van Noort (17), the application of hydrofluoric acid removes the surface morphology created by the treatments prior to etching, due to its aggressive potential. However, in the SEM micrographs of G4 and G5 (Figs. 1D and 1E), the morphology created by sandblasting is clearly observed, even after acid etching. Similar findings have been reported by other authors (1,19).

The bond strength recorded in the sandblasted group (G3) did not differ significantly those obtained with the association of sandblasting and etching for 2 or 4 min (G4 and G5, respectively). These results are in agreement with those of other studies (10,11,14).

In the present study, no significant difference was observed when different hydrofluoric acid etching times associated with sandblasting were compared to each other. On the other hand, Estafan et al. (13) found a better performance when a shorter etching time was used plus sandblasting.

The SEM micrograph of G6 (Fig. 1F) shows a flat surface. Grooves due to polishing and surface defects resultant of the laboratorial procedures are evident. Bond strength obtained with silane application exclusively (G6) was significantly lower than those obtained in the other groups, except for 4-min etching time (G2).

Aida et al. (3) evaluated bond strength after application of Tokuso Ceramic Primer and did not find significant differences between polished ceramics and those etched with hydrofluoric acid. According to these authors, the formation of siloxane bond was more important than the micromechanical retention produced by etching. Shimada et al. (9) and Foxton et al. (15), using a ceramic primer with acid monomer, also obtained an adequate bonding, independently of the previous surface treatment. On the other hand, other authors (5,7), using a similar silane, observed a significant increase in the bond strength after hydrofluoric acid etching previously to the silanization, which is consistent with our results.

Additionally, the findings of the present study showed that a 4-min etching time was significantly less efficient than the 2-min etching time, which suggests that an over-etching may lead to stress concentration in the adhesive interface and weaken the ceramic surface (6). These results revealed that a large number of irregularities on ceramic surface was not sufficient to yield higher bond strength means. These results also suggest that etching with hydrofluoric acid for 2 min produced the most favorable micromechanical retention among the tested surface treatments. Even when associated with sandblasting, 2-min etching led to better results than the longer etching time. The findings of this study emphasizes the importance of hydrofluoric acid-induced micromechanical retention for resin cement-ceramic bonding (1,5,7,11-13,20).

There was a predominance of cohesive failures of porcelain in all the experimental conditions (Table 2). This result is in accordance with those of several studies that evaluated the bond strength at ceramic/composite interface after acid etching and/or silanization (1-3,8,10,11,13). Therefore, it would be expected to have similar bond strength means for all surface treatments, which was not observed. This could be explained by the effects of the different treatments on porcelain fracture strength, modifying its superficial energy, which is an important factor on fracture the spreading (6,18).

Within the limitations of an *in vitro* study, it may be concluded that 2-min hydrofluoric acid etching produced a favorable micromechanical retention that enhanced resin cement bond strength to porcelain.

RESUMO

O objetivo deste estudo *in vitro* foi avaliar o efeito de diferentes tratamentos de superfície na resistência ao cisalhamento da união entre porcelana e cimento resinoso. Foram confeccionados sessenta pares de discos de porcelana aluminizada a 50%, de 6 mm de diâmetro por 3 mm de espessura (A) e 3 mm de diâmetro por 3 mm de espessura (B). Os espécimes foram divididos aleatoriamente em 6 grupos (n=10 pares), de acordo com os tratamentos de superfície: condicionamento com ácido fluorídrico por 2 e 4 min (G1 e G2), jateamento com óxido de alumínio (50 μm) por 5 s (G3), jateamento seguido de ácido fluorídrico por 2 e 4 min (G4 e G5) e controle (G6). Em seguida, foi aplicado silano na superfície tratada de ambos os discos, e os discos B foram cimentados sobre os respectivos discos A, utilizando o cimento resinoso dual Bistite II DC. Os espécimes foram armazenados em água destilada a 37ºC por 24 h, para posterior teste de cisalhamento em máquina de ensaio universal. As médias em MPa foram: G1: 14,21 ± 4,68; G2: 8,92 ± 3,02; G3: 10,04 ± 2.37; G4: 12,74 ± 5,15; G5: 10,99 ± 3,35; G6: 6,09 ± 1,84. Os dados foram submetidos à análise de variância a um critério e teste de Tukey ($p<0,05$). O condicionamento ácido por 2 min apresentou resultados significantemente superiores ao condicionamento por 4 min ($p<0,05$) e ao grupo controle ($p<0,05$), porém não diferiu estatisticamente do jateamento, associado ou não ao ácido fluorídrico por 2 ou 4 min ($p>0,05$). Dentro das limitações de um estudo *in vitro*, conclui-se que o condicionamento com ácido fluorídrico por 2 min produziu uma retenção micromecânica favorável, que aumentou a resistência de união entre o cimento resinoso e a porcelana.

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