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# Ceramic repairs with resins: silanization protocols


## Abstract

**Aim:** To assess the shear bond strength of a microhybrid compound resin (3M-ESPE, St. Paul, MN, USA) to a feldspar ceramic (Noritake EX-3), using different silanization protocols and qualify the failures types resulting from these different treatment protocols.

**Materials and methods:** Were manufactured 120 ceramic pellets, included in PVC pipes. Surfaces were conditioned with hydrofluoric acid at 10% for 60s, then rinsed and dried. Blocks were divided into four groups: G1 (without silane); G2 (silane air-dried for 3 minutes at room temperature); G3 (silane dried in a heating chamber at 50 °C for 5 minutes); G4 (silane cooled at 10 °C for 5 minutes). Following surface silanization, the adhesive was applied and photopolymerized for 10s. A matrix was fit in the PVC pipe, where a portion of resin was inserted, and then photopolymerized for 40 s. Test specimens were subjected to thermal cycling for 5,000 cycles at 5°C, 37.5°C and 55°C. Then, the shear mechanical test was conducted. Tabulated data were analyzed with D'Agostino's normality test, one-way ANOVA and Tukey's test.

**Results:** We found no significant differences in force and strength between experimental groups ($p>0.05$).

Conclusions: Silanization protocols showed no effect on the samples shear strength. There was also no significant association between type of failure and treatment.


**Keywords:** composite resins, dental porcelain, shear strength

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## Introduction

Dental ceramics is characterized by its exceptional esthetic performance, low thermal and electrical conductivity, and high resistance to compression and wearing. Ceramic restorations have proven clinical efficacy, with a success rate of up to 97% after 5 years and 91% after 10.5 years.[1] However, due to its hardness, they are friable and prone to fractures.[2] Considering the limitations of esthetic prostheses made of porcelain and the complexity of the buccal environment, fractures on the esthetic coating are common, exposing the metal substrate.[3] Repairs consist of the application of compound resin layers associated to adhesive systems,[3,4] with different surface treatments on the fractured ceramics.[5] Among the several surface treatments used in ceramics repair, silanization and hydrofluoric acid conditioning stand out.[6,7] In addition to that, heating of the silanized surface has been tested with the aim of eliminating water and other components, and forming a thinner silane layer, favoring a complete condensation reaction between silane and silica, increasing the number of reaction sites. Also, this heating promotes post-conditioning surface smoothness and keeps microretentions, enlarging the adhesive surface of contact.[8,9] With this context in mind, this work aims to establish a silanization protocol for ceramics protocols based on silane temperature in order to optimize its performance. We assessed shear bond strength of a compound resin (Z-100-3M ESPE) to a feldspar ceramic (Noritake EX-3), using different silanization protocols.

## Materials and methods

To obtain the porcelain samples, we added powdered feldspar porcelain Noritake EX-3 (Noritake, Japan) to distilled water and accommodated the mixture in a metal matrix with a hole of 12mm x 4mm. For the insertion, we used a plastic spatula (Clearfill, Kuraray, Cotia, SP, and Brasil). In total, 120 porcelain pellets were manufactured (12 x 4mm).[10] The pellets were removed from the matrix and taken to the porcelain furnace Vulcano Platiniun (EDG, São Carlos, Brazil) at 600°C for 6 minutes, at a heating speed of 55°C/min, under vacuum, until reaching 910°C. Once the desired temperature was reached, the pellets were kept for one more minute in the absence of a vacuum. The pellets were left to cool at room temperature.

Ceramic pellets were included using ½" PVC pipes with 15 mm of height (Tigre, Castro, Paraná, Brazil) and self-polymerized colorless acrylic resin Jet, (Artigos Odontológicos Clássico Ltda., São Paulo, Brazil). Each block was taken to the mechanical polisher (Politriz, Arotec, Cotia, SP, Brazil), that used wet-and-dry sandpaper Aquaflex (Norton, Guarulhos, Brazil) of grit size 320, 400, 600, 800, 1200, and 1500 for 30s each, at 300rpm.

The ceramic blocks were randomly distributed in four groups:

1. G1 - Without Silano (control) - Conditioned with hydrofluoric acid 10% Condac Porcelana (FGM, Joinville, SC, Brazil) for 60 seconds. Adhesive Adper Single Bond 2 (3M-ESPE, St. Paul, MN, USA) was applied and photopolymerized for 10s.
2. G2 - Silane at room temperature - Conditioned with hydrofluoric acid 10% Condac Porcelana (FGM, Joinville, SC, Brazil) for 60 seconds. Silane RelyX Ceramic Primer (3M-ESPE, St. Paul, MN, USA) was applied and air dried for 3 minutes at room temperature. Adhesive Adper Single Bond 2 (3M-ESPE, St. Paul, MN, USA) was applied and photopolymerized for 10s.
3. G3 - Heated silane - Conditioned with hydrofluoric acid 10% Condac Porcelana (FGM, Joinville, SC, Brazil) for 60 seconds. Silane RelyX Ceramic Primer (3M-ESPE, St. Paul, MN, USA)

was applied, air-dried for 3 min and taken to a heating chamber at 50ºC for 5min. Adhesive Adper Single Bond 2 (3M-ESPE, St. Paul, MN, USA) was applied and photopolymerized for 10s.

4. G4 - Cooled silane - Conditioned with hydrofluoric acid 10% Condac Porcelana (FGM, Joinville, SC, Brazil) for 60 seconds. Silane RelyX Ceramic Primer (3M-ESPE, St. Paul, MN, USA) was applied, air-dried for 3 minutes and taken to a cooling chamber at 10ºC for 5min. Adhesive Adper Single Bond 2 (3M-ESPE, St. Paul, MN, USA) was applied and photopolymerized for 10s.

Following the surface treatment, the microhybrid compound resin Z-100 (3M-ESPE, St. Paul, MN, USA) was inserted. To standardize the process, we used a bipartite metal matrix with a central hole of 5.0 mm of diameter and 3.0 mm of thickness.[11] A portion of the resin was inserted on the central hole. The portions were previously weighted on a precision scale (0.0001g) and photoactivated for 40 seconds. The obtained specimens were then thermally cycled for 5,000 cycles at 5°C, 37.5°C and 55°C. The shear mechanical test was conducted on a universal testing machine EMIC DL2000 (EMIC, São Paulo, Brazil), with a load cell of 20 KN and actuator speed of 0.5mm per minute. Shear resistance was registered in MPa. Tabulated data was subjected to D'Agostino's normality test followed by one-way ANOVA and Tukey's test, using the software Bio Estat, version 3.0. Failures were analyzed with a stereoscopic magnifying glass at 40x and categorized in adhesive, cohesive or mixed.

## Results

Table 1 shows the results of force in N and kgf and strength in Mpa for the experimental groups (silanization protocol). We notice that force and strength show no significant differences between experimental groups ($p>0.05$). Following an exploratory analysis, force and strength results were submitted to one-way variance analysis (ANOVA). Association between treatments and type of failure was assessed with Fisher's exact test. All analyses were conducted in SAS (Institute Inc., Cary, NC, USA, Release 9.2, 2010) considering 5% of significance.Three types of failures were identified after the test: cohesive, mixed, and adhesive (Figure 1) (Figure 2).

**Table 1** Average (standard deviation) of force (N and kgf) and strength (Mpa) as a function of time and experimental group.

| Group | Force (N) | Force (kgf) | Strength (Mpa) |
|---|---|---|---|
| Without silane | 289.21 (116.95)a | 29.49 (11.92)a | 14.73 (5.96)a |
| Silane at room temperature | 283.26 (128.57)a | 28.88 (13.11)a | 14.43 (6.55)a |
| Heated silane | 309.75 (107.17)a | 31.59 (10.93)a | 15.78 (5.46)a |
| Cooled silane | 319.31 (91.57)a | 32.56 (9.34)a | 16.26 (4.66)a |
| p-value | 0.4905 | 0.4905 | 0.4905 |

Caption: Averages followed by the same letters show no statistical differences ($p>0.05$).

**Figure 1** Test specimen (ceramic-resin).

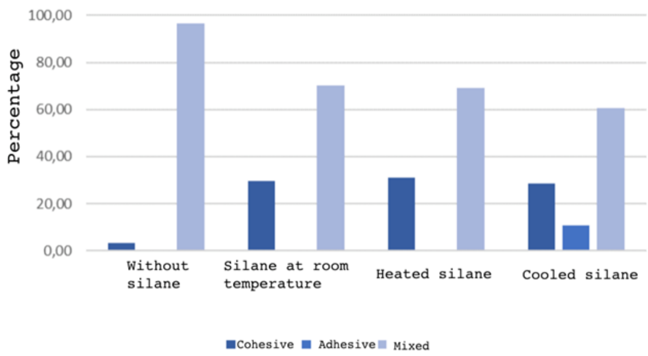


**Figure 2** Frequency distribution for the types of failure according to treatment.

## Discussion

At the interface of resinous materials and ceramics, silane provides a chemical bond considered effective.[3,12–18] Conditioning of porcelain surface with acid is an essential step for the adhesive bond strength between porcelain and resinous materials.[11,13,14] Some authors claim that its use associated with acid conditioning is the best surface treatment for porcelain.[11,12,15–19] Our results show a lack of significant effect of silane application on bond strength, corroborating results found by other authors.[14–21] On the other hand, some studies show an improvement in resin/porcelain bond strength due to silane application.[3,12,23] Some studies have shown that hydrofluoric acid conditioning was not sufficient to increase adhesion and that silanization is preferable.[9,24,25] The present study uses hydrofluoric acid in all groups analyzed. According to some authors, this step could not have been avoided given its relevance in the adhesion process.[13,26] However, some authors suggest some alternatives, such as the thermal treatment of silane,[9,27,28] which would result in the elimination of water and other components, such as alcohol and acetone, and would promote covalent binding between silane and silica, improving the chemical bond between silane and ceramics.[28–30] Temperature increase can also optimize viscosity, improving some adhesive systems[31] and resinous materials[32] wettability. There is still no consensus in the literature regarding the effect of temperature variation on silanes. Some authors have observed improvement in bond strength after thermal treatment with heated air,[8,9,28–33] high temperature furnaces[11,34–36] or hot water bath.[9,28,33] Other authors have observed a worsening in bond strength following thermal treatment with heating,[37] hot water bath of silane,[35] silane cooling,[36] or no improvements at all.[13,16,26,36] The present study showed no significant differences between the experimental groups. The type of failure is an important factor to be considered in the adhesive systems strength assessment. Cohesive failures suggest a more favorable bond between resin and porcelain.[11,29,38] However, here the most common failures found in all groups were mixed. For this reason, a relationship between silane heating and type of failure could not be drawn, in agreement with the results of other studies.[13,26] Further studies are needed for the definition of a silanization protocol based on temperature change so that its clinical use can be reliably recommended.

## Conclusion

Different silanization protocols have failed to show significant differences regarding shear strength and mixed failure ratio in all groups, showing no significant association ($p<0.05$) between treatment and type of failure.

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## Conflict of interest

The author declares that there is no conflict of interest.

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