Effect of Silane on Shear Bond Strength of Two Porcelain Repair Systems

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Abstract
Objectives: Ceramics have advantages such as optimal esthetics and biocompatibility. However, in the oral environment, they are subjected to high levels of stress due to masticatory forces, saliva, thermal changes and alterations of pH, which increase their risk of fracture. Since replacement of these restorations is costly and time-consuming, composite resin is often used for intraoral repair of these restorations. This study aimed to assess the shear bond strength of two porcelain repair systems by Pulpdent and Ultradent and evaluate the effect of number of silane layers on the shear bond strength.

Methods: This invitro experimental study was conducted on 66 porcelain blocks measuring 3x5x8mm. In each kit, samples were randomly divided into three groups of 11. Silane was not used for group one. Groups two and three received one coat and two coats of silane, respectively. After surface preparation, composite was bonded to ceramic surfaces. Data were analyzed using two-way ANOVA.

Results: The LSD test showed that application of Ultradent silane significantly affected the shear bond strength (P<0.05) while Pulpdent silane had no such effect (P=0.89). Application of one layer and two layers of silane was not significantly different (P=0.94).

Conclusion: Ultradent ceramic repair kit yields higher shear bond strength at the ceramic-composite interface compared to Pulpdent ceramic repair kit. Use of one or two layers of silane does not make any significant difference with regard to the shear bond strength of ceramic to composite.

Key words: Ceramics, Composite Resins, Dental Bonding, Dental Porcelain, Dental Restoration Repair, Shear Strength

How to Cite:
Fracture of these restorations is divided into three groups of fracture of porcelain, fracture of both porcelain and metal and fracture of metal (13,14).

Repair techniques are divided into two types of direct and indirect. Indirect repair refers to restoration retrieval and its subsequent repair in a laboratory. Direct repair includes techniques of directly applying composite on the broken restoration. The latter is faster, more affordable and easier to perform compared to the indirect technique (15,16).

Silane is a coupling agent made of organic silicon, which enables a bond between organic and inorganic phases of dental composites. Manufacturers treat the filler surface with this coupling agent before mixing the mineral fillers with organic oligomers (i.e. Bis-GMA and UDMA) in order to obtain a strong bond between fillers and organic oligomers during setting (17). These agents are also used to obtain a bond between porcelain and composite. Silane forms a covalence chemical bond between the silica of the ceramic surface and composite, which enhances micromechanical interlocking. Theoretically, use of silane yields a stable and durable bond between the composite and broken porcelain (18).

Methacryloxypropyl-trimethoxysilane (or 3-trimethoxysilylpropyl methacrylate) is a mono-functional silane most commonly used in the laboratory and clinical settings. It is often diluted to less than 2wt% in water-ethanol solution. Its pH is adjusted to 4-5 with acetic acid prior to hydrolysis. Silanization is critical to obtain an adequate bond. A strong and durable bond can only be obtained via a combination of increasing the surface roughness (by air abrasion and etching) and silanization. However, some authors have reported an acceptably high bond following silanization alone (19). Silanization may increase the composite bond to porcelain by 25% (20). Berry et al. in 1999 showed that the shear bond strength of porcelain to composite during long-term water storage increased by silanization. Also, the bond strength yielded by the use of different silanes such as Fusion (two-mix), Mirage (two-mix and one-mix) and Cerinate Prime (one-mix) is variable (21). Barghi et al. (22) in 2000 assessed the effects of silanization intervals (using one-mix and two-mix silane) and heat on the shear bond strength of porcelain to composite. They showed that heat increased the bond strength at all time points (three minutes, 24 hours and one week).

Matinlinna et al. (23) compared the bond strength of five dental silanes and concluded that different silanes yield different bond strength values; they were also different with regard to concentration, pH and type of solvent. In their study, silanes with lower pH values yielded a stronger bond than silanes with a higher pH. Menga et al. (24) evaluated five dental silanes and concluded that ceramic bond strength was influenced by the type of silane and conduction of thermocycling. Previous studies have reported increase in composite-porcelain bond strength following the use of silane (compared to not using it) (1,5) However, no previous study has evaluated the effect of higher number of silane coats on the bond strength. Moreover, factors such as high cost, risk of trauma to the restored teeth, shortage of time and difficult retrieval of
restoration delay the replacement of a metal-ceramic restoration (7,14,25,26). Thus, when a broken restoration compromises periodontal health and its replacement is not feasible (due to the abovementioned reasons), it needs to be directly repaired (27). This study sought to compare the shear bond strength of composite to porcelain following the application of two commonly used porcelain repair systems. The effect of increasing the silane coats on the shear bond strength was also evaluated. It should be noted that these systems have different compositions.

**Methods**

In this *in vitro*, experimental study, a two-piece steel mold (MO40) with two interlocking male and female parts was fabricated. For further strength, it was plated after fabrication. Each piece measured 8×5×3 mm and was hollow (Figure 1).

One piece (half) of the mold was considered for porcelain matrix for all samples (25). To fabricate porcelain blocks, inside of the metal mold was waxed up using inlay wax (Dentaurum, Ispringen, Germany) and after flasking, porcelain (Emax, Ivoclar, Schaan, Liechtenstein) was injected into the mold in an Ivoclar furnace using heat pressed technique. After the fabrication of porcelain block, a thin layer of A2 shade Emax porcelain (Ivoclar, Liechtenstein, Germany) was applied over it (20g of powder with 6mL of distilled water) and heated in a furnace at 750°C (Figure 2).

The same type of porcelain was used for all samples (all groups). Sample size was calculated to be 10 samples in each group according to previous studies (22-24). To compensate for possible dropouts due to errors, 11 samples were fabricated for each group (six groups of 11 samples each). All samples were roughened by 018 cylindrical diamond burs (Dentsply, Philadelphia, USA) (Figure 3). Each bur was used for five surfaces and then discarded.

This process was followed by further roughening by sandblasting with 50μm aluminum oxide particles at 10mm distance.
from the surface and a 90° angle for 20 seconds at 2.5 bar pressure. Porcelain samples were then washed for 10 seconds and air-dried (5). A total of 66 porcelain samples were randomly divided into six groups of 11. In groups one to three, Ultradent porcelain repair kit containing 9.5% hydrofluoric(HF) acid and silane was used while in groups four to six, Pulpdent porcelain repair kit was used containing 9.6% HF acid and silane (Table 1).

| Table 1-Ultradent and Pulpdentsilanes |  |
|---------------------------------------|  |
| Trade name, manufacturer              | Effective silane                  | Solution                   |
| Pulpdentsilane bond Enhancer.         | A silane (%Not applicable)        | Ethanol 92.6%, Aceton 7.4% |
| Pulpdent                              | 3-thimethoxyxilpropyl methacrylate(5%-15%) | Ethanol-87%               |
| Ultradent.Ultradent                    |                                       |                           |

*Information in this table is based on the manufacturers’ brochures*

In all six groups, the bonding surface was etched with HF acid available in the respective kit for three minutes and was then washed with water spray for 30 seconds and dried with air spray. Silane was not applied to the surface of 11 samples in groups one and four. One layer of silane was applied to the surface of samples in groups two and five using a microbrush; after 60 seconds, it was gently dried with air spray for five seconds. Two layers of silane were applied on the surface of samples in groups three and six. The first coat was painted on the ceramic surface by a microbrush. After 60 seconds, the surface was gently air dried for five seconds using air spray and then the second coat was applied as the first one. Bonding resin (Multi-Purpose Adhesive, 3M ESPE, St. Paul, MN, USA) was then applied on the surface of all samples and thinned by air spray. It was light cured for 10 seconds according to the manufacturer’s instructions using Coltolux 2.5 (Coltene AG, Feldwiesentiasse ALT statten, Switzerland) light curing unit with a light intensity of 480mW/cm² at 0.5mm distance from the surface. Each surface was light cured for 40 seconds. After fabrication of samples, they were all stored in distilled water at 37°C for one week and were then subjected to 1000 thermal cycles between 5-55°C with 30 seconds of dwell time and 12 seconds of transfer time (20). The temperature of baths (5 and 55°C) was constantly monitored by a thermometer and adjusted using ice and boiling water. All specimens were then stored in distilled water at 37°C for 48 hours. Afterwards, the samples were mounted in autopolymerizing acrylic resin (Taklon, Rodent s.r.l, Milan, Italy) in metal rectangular molds measuring 33x24x12mm. A surveyor was used in order to mount all samples perpendicular to the acrylic surface. Mounted samples were then immersed in distilled water at 37°C for 24 hours and were then subjected to shear load in a universal testing machine (Z050 ZwickRoell, Ulm, Germany) at a crosshead.
speed of one millimeter/minute. The load was applied to the ceramic-composite interface (Figure 4).

![Figure 4- Sample mounted in acrylic resin; (a) Blade of Instron machine applying load to the composite-ceramic interface (b) acrylic (c) composite](image)

Load application was continued until fracture occurred. Two-way ANOVA was used to assess the effect of type of porcelain repair kit (Ultradent and Pulpdent) and number of silane layers (zero, one layer and two layers) as well as the interaction effect of both. Mode of failure was evaluated under a stereomicroscope (SM800 C-DS, Nikon, Melville, USA) at×10 magnification. Mode of failure was divided into three categories of adhesive (at the resin-substrate interface), cohesive (fracture within the substrate or restorative material) and adhesive-cohesive (mixed). To compare the frequency of each type of failure among zero, one and two layers of silane in each porcelain repair kit, chi square test and Fisher’s exact test were applied. Data were analyzed analytically and descriptively using SPSS version 16 software (Microsoft, IL, USA). Level of significance was set at \( P<0.05 \).

### Results

Two-way ANOVA showed that type of porcelain repair kit significantly affected the shear bond strength \( (P<0.01, \text{ mean difference of 3.7}) \); however, the interaction effect of type of kit and silane was not significant \( (P=0.17) \). Ultradent porcelain repair kit yielded higher shear bond strength than Pulpdent. The LSD test showed that silanization significantly affected the bond strength compared to not applying silane \( (P<0.05, \text{ mean difference of 3.09}) \). Also, the LSD test showed that use of Ultradentsilane significantly affected the shear bond strength \( (P<0.05, \text{ mean difference of 10.2}) \). However, Pulpdentsilane had no significant effect on shear bond strength \( (P=0.89, \text{ mean difference of 0.8}) \). Application of one and two layers of Ultradent (mean difference of 1.06) and Pulpdent (mean difference of 0.14) silanes did not cause a statistically significant difference in results \( (P=0.94 \text{ for Pulpdent and } P=0.60 \text{ for Ultradent, Table 2 and Diagram 1}) \).

### Table 2- The mean and standard deviation of shear bond strength based on the type of kit and number of silane coats

<table>
<thead>
<tr>
<th>Porcelain repair kit</th>
<th>Silane</th>
<th>Mean shear bond strength</th>
<th>Standard deviation</th>
<th>Number of samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultradent</td>
<td>Two layers</td>
<td>16.12</td>
<td>3.43</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>One layer</td>
<td>15.06</td>
<td>6.30</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>No silane</td>
<td>10.21</td>
<td>3.89</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td>13.80</td>
<td>5.26</td>
<td>33</td>
</tr>
<tr>
<td>Pulpdent</td>
<td>Two layers</td>
<td>10.36</td>
<td>5.99</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>One layer</td>
<td>10.50</td>
<td>4.90</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>No silane</td>
<td>9.63</td>
<td>2.75</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td>Total</td>
<td>10.16</td>
<td>4.61</td>
<td>33</td>
</tr>
</tbody>
</table>

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Effect of Silane on Shear Bond Strength

<table>
<thead>
<tr>
<th></th>
<th>Two layers</th>
<th>One layer</th>
<th>No silane</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>13.24</td>
<td>12.78</td>
<td>9.92</td>
<td>11.9852</td>
</tr>
<tr>
<td></td>
<td>5.60</td>
<td>5.98</td>
<td>3.30</td>
<td>66</td>
</tr>
<tr>
<td></td>
<td>22</td>
<td>22</td>
<td>22</td>
<td></td>
</tr>
</tbody>
</table>

Diagram 1 - The mean and standard error of the shear bond strength values (MPa) based on the type of kit and number of silane layers

Discussion

Intraoral repair of broken porcelain restorations with composite is challenging for dentists. The new generations of multipurpose adhesive systems offer several solutions for composite repair of fractured porcelain. Silane is commonly used for composite repair of porcelain. However, no consensus has been reached about the long-term effect of silane on bond strength (28,29). This study aimed to assess the efficacy of two porcelain repair kits and the effect of number of silane coats on the shear bond strength of porcelain to composite. The results showed that type of porcelain repair kit and silane significantly affected the shear bond strength but number of coats had no effect on shear bond strength. No difference was noted in the results of application of one and two coats of silane. Ultradent kit yielded higher shear bond strength than Pulpdent. This result confirms the findings of previous studies recommending the use of silane to enhance bond strength (28). No study was found on the effects of number of coats of silane applied but the manufacturers of both Ultradent and Pulpdent kits recommend application of one layer of silane.

Accuracy and clinical relevance of different methods for in vitro assessment of ceramic-composite bond strength have been extensively studied. In the current study, shear bond strength was measured due to simplicity, extensive use in several studies and the fact that anterior teeth are primarily subjected to shear stresses (18,21).

Several factors affect the ceramic-composite bond strength such as microstructure of ceramic and composite (30), type of composite (29,30), type of silane (30), method of surface preparation (5,30) and technique of etching (type of acid, its concentration and time) (31-33). In our study, different silanes were used. In 2005, Kermanshah et al. compared the effects of three porcelain etchants on shear bond strength of composite to porcelain. In their study, three types of acids namely 9.5% HFacid (Ultradent) for one minute, 1.23% APF gel (Kimia) for 10 minutes and 37% PHA (Kimia) for one minute were used. A total of 120 samples were evaluated in three groups of 40. It was shown that PHA and APF did not create micro-undercuts for porcelain retention; but HFacid created micro-undercuts and it was shown that Ultradent silane had no effect on porcelain-composite bond strength (33). In the current study, 9.5% (Ultradent) and 9.6% (Pulpdent) HF acids were used to etch porcelain. It was
shown that Ultradentsilane significantly affected the shear bond strength but Pulp dentsilane had no such effect. Such a difference between the effects of these two silanes may be due to their different compositions. Ultra dentsilane contains isopropanol and meth acryloxypropyltrimethoxy silane, which can affect the bond strength testing results. Ultradent silane is inactive because it does not contain acids in its formulation; thus, it does not play a role in composite-porcelain bond strength alone and its action depends on micromechanical retention caused by porcelain etching by use of HF acid. Pulpdent silane contains ethylalcohol in organic solvents. In addition to the type of acid, its variable concentrations also cause significant differences in porcelain microstructure; this indicates selective dissolution of a porcelain phase. For instance, 52% HF selectively dissolves the glass phase of porcelain while its 20% concentration selectively dissolves the crystalline phase. The most suitable microstructure for micromechanical bonding is achieved by use of 10% HF (27). Therefore, 9.5% and 9.6% HF acids were used in the current study.

Difference in porcelain repair kits is attributed to different chemical reactions of silane in forming a bond between the substrate and resin. Evidence shows that silanes with different chemical compositions and concentrations of solvents have variable adhesions. This is related to silane hydrolysis and poly-condensation of polysiloxane network on the substrate. Factors such as substrate surface acidity also affect the poly-condensation rate. Poly-condensation of silane is variable in different kits and further studies are required to better elucidate this topic.

Shahverdi, et al. in 1998 evaluated the effect of different surface treatments on bond strength of composite to porcelain and showed that substrate surface preparation by special burs (like K1 bur) probably fills the porosities on the substrate surface by silica particles. Although it may seem that surface roughness would be higher with the use of K1 bur, the bond strength was lower compared to the group etched with phosphoric acid. This is probably attributed to the adverse effects of rotary burs (due to high speed and pressure) on the ceramic surface properties (5). In the current study, the shear bond strength of Pulpdent porcelain repair kit was lower than that of Ultradent kit, which may be attributed to the single or interaction effect of burs, silane, adhesive or composite. The effects of these factors on bond strength of composite to ceramic must be investigated in future studies.

Clinical success of porcelain repair kits depends on the quality of bond between porcelain and composite. This bond can be achieved by mechanical or chemical methods or a combination of both. Etching causes porosities on the porcelain surface, which result in stronger bond. Combination of sandblasting and acid etching of porcelain along with silanization results in the highest quality of bond achievable (34).

Air abrasion with alumina particles is another method of substrate surface preparation prior to ceramic-composite bonding. It should be noted that if alumina particles are embedded in the substrate =Al-
O-Si≡ bonds are formed, which are unstable and weaker than Si-O-Si≡ bonds (23). In the current study, 50μm alumina particles at 10mm distance and 90° angle with 2.5 bar pressure were used for 20 seconds, and possible formation of weak =Al-O-Si≡ bonds is among the limitations of our study. Some studies have recommended the application of two or more layers of silane instead of just one coat for bonding of composite to porcelain in order to enhance the bond strength and decrease microleakage (35). However, our findings showed no significant difference in results between the application of one and two layers of silane. Thus, both techniques can be successfully used. Application of two layers of silane did not cause a significant improvement in clinical service. Also, increase in number of silane layers may negatively affect the bond strength due to increased thickness. Further studies on application of different numbers of silane coats are recommended to assess the accuracy of this theory.

**Conclusion**

Ultradent porcelain repair kit provides higher shear bond strength of composite to porcelain than Pulpdent kit. Using Ultradent repair kit, application of silane yielded higher shear bond strength compared to no application of silane. Using Pulpdent repair kit, no significant difference was noted with regard to shear bond strength in use or no use of silane. In both kits, use of one or two layers of silane made no difference with regard to the shear bond strength of ceramic to composite. Considering the limitations of this study, further investigations on the effect of number of silane coats on shear bond strength are required.

**Conflict of Interest:** “None Declared”

**References:**