

Comparison of shear bond strength of silorane and methacrylate-based composites to IPS empres 2 ceramic with various surface treatments

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Objectives Treatment of chipped or fractured porcelain with composite resin is considered as an economic treatment for minor fractures in ceramics. The aim of this study was to evaluate the effect of different ceramic surface treatments on bond strength of methacrylate-based and silorane-based composite resin to IPS Empress 2.

Methods Sixty IPS Empress 2 ceramic discs were fabricated and after etching with 9.6% hydrofluoric acid, they were divided into six groups: (1) P90 primer and bonding agent + Filtek P90 composite resin; (2) Single Bond + Filtek Z250 composite resin; (3) similar to the first group + silane pretreatment; (4) similar to the second group + silane pretreatment; (5) silane pretreatment + Filtek P90 composite resin; (6) silane pretreatment + Filtek Z250 composite resin. Each specimen was subjected to shear load until fracture occurred. Statistical analysis was performed using one-way ANOVA, Tukey's test and *t*-test.

Results Regardless of the type of surface treatment, Z250 composite demonstrated significantly higher shear bond strength than P90 composite ($P < 0.05$). Group 4 showed the highest shear bond strength values with statistically significant difference with other groups while the fifth group showed the least values ($P < 0.05$).

Conclusion Silane coating along with the application of adhesive system and etching in methacrylate-based composite was the most efficient surface treatment in terms of bond strength.

Keywords composite resins, dental porcelain, shear strength

Introduction

Dental ceramics are extensively used in esthetic dentistry due to their improved mechanical and physical properties.¹ New high-crystalline content ceramic systems including lithium disilicate ceramics (IPS Empress 2, Ivoclar), glass infiltrated alumina and zirconia (In-Ceram, Vita) and high-density alumina or zirconia ceramic systems (Procera, Nobel Biocare, Cercon, Dentsply Ceramo; Lava, 3M-ESPE) have higher strength and esthetic properties for supporting tooth structure in metal-free restorations.²

Despite the improvements in strength of ceramics, some of them undergo fracture as a result of occlusal overloads, fatigue or trauma.³ Direct repair of ceramic restorations without removal of the entire restoration is preferable because of less trauma to both restoration and tooth structure. Composite resins are recommended to repair dental ceramics, due to their low cost and good physical properties.⁴

Achieving a strong chemical and micromechanical bond between composite resins and dental porcelain is important for a durable repair.⁵ A durable resin bond could be achieved by pre-treatment. Various pretreatment techniques have been suggested in order to improve bond strength such as abrasion with diamond burs, silica coating, sandblasting, airborne particle abrasion with aluminum oxide, chemical etching with hydrofluoric acid, laser treatment or combinations of these techniques.⁶ It has been reported that etching with hydrofluoric acid followed by the application of silane coupling agent is the most preferred surface pretreatment technique to achieve high bond strength for silica-based all-ceramic restorations.⁷

Silane coupling agents are adhesion promoters that enable chemical bonding with organic surfaces such as resin materials and inorganic surfaces such as indirect glass ceramic

restorations; hence, they are considered an important factor for proper silica-based ceramic repair.⁸

Application of adhesive as an intermediate layer is claimed to improve bond strength of porcelain to methacrylate-based composites via increasing the surface wettability.^{9,10} But Hamano et al.¹¹ showed that using bonding agent might not increase the wettability of silorane-based composites as a result of high hydrophobicity. Thus, in this study, P90 primer was used to increase silorane-based composite's surface energy and bonding due to its self-etching property. Moreover, it has been shown that the type of composite resin also affects the bond strength to porcelain.¹² Silorane-based composite is the first commercially available composite resin containing a new silorane-based monomer, which is the result of a reaction between oxirane and siloxane molecules. The ring opening oxirane monomer provides a low volumetric polymerization shrinkage (<1%), which might generate less stress at the adhesive interface and consequently provide more efficient bond.¹¹

Despite many studies that investigated the bond strength of methacrylate-based composites and feldspathic ceramics with various surface treatments, there are limited studies on the bond strength of silorane-based composites to different ceramics. Therefore, the aim of this study was to evaluate the shear bond strength of silorane and methacrylate-based composite to IPS Empress 2, employing several methods for surface treatment associated with two adhesive systems and silane application.

Methods

Table 1 show the materials used in this study. The wax patterns of 60 discs (5 mm in diameter and 1 mm in height) were fabricated using a plastic mold. The wax patterns were invested and

Table 1. **Materials used in the study**

| Material | Composition |
|--------------------------------|---|
| Filtek P90 Bonding Agent | Copolymer, BisGMA, HEMA, water, ethanol, silane-treated silica filler initiator Bond: Hydrophobic Dimethacrylate, phosphorylated methacrylate, TEGDMA, silane-treated silica filler, initiators, stabilizers |
| Single Bond Universal Adhesive | MDP phosphate monomer, Dimethacrylate resins, HEMA, Vitrebond copolymer filler, ethanol, water, initiators, silane |
| Filtek P90 Composite | 3,4 epoxy cyclohexyl ethyl cyclo polydimethylsiloxane, silanized quartz, yttrium fluoride, camphorquinone, bis3,4 epoxy cyclohexyl Ethyl Phenyl Methyl Silane |
| Filtek Z250 Composite | Bisphenol A, polyethylene glycol diether, dimethacrylate, diurethane dimethacrylate, bisphenol diglycidyl ether dimethacrylate, zirconia/silica, TEGDMA |
| IPS Empress | Barium glass filler, mixed oxide: Ba-AL-fluorosilicate, dimethacrylate, prepolymer, ytterbium trifluoride, highly dispersed silicon oxide, catalysts, stabilizers, pigments |
| Porcelain Etch and Silane | 9.6% hydrofluoric acid, silane |

pressed into lithium disilicate-based core ceramic discs (IPS Empress 2; Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions. The discs were embedded in acrylic resin molds (3 mm in height). The disc surfaces were sandblasted with 50 µm aluminum oxide particles at 2.5 bar pressure for 13 seconds at a distance of 10 mm. All specimens were then ultrasonically cleaned in 96% isopropanol for three minutes. Then, the specimens were treated with 600-grit silicon carbide paper. In all groups, specimens were etched with 9.6% hydrofluoric acid for two minutes. Then, the samples were rinsed thoroughly with water (10 dL/minute for 30 seconds) and subsequently dried for 15 seconds.

A plastic tube was used to serve as the composite mold on the ceramic surface. Composite was applied incrementally into the rubber mold and was gradually built up to create a cylinder on disc specimen in all groups. The IPS Empress 2 specimens were randomly assigned to the following six groups ($n = 10$):

Group 1: One layer of P90 primer and then P90 bond were applied with a micro-brush and light cured for 20 seconds with LED light curing unit with 1100 mw/cm² light intensity (Demi plus, Kerr, Orange, CA, USA). Filtek P90 composite (A2 shade; 3M ESPE, St. Paul, MN, USA) was placed on the ceramic disc and then light cured for 40 seconds.

Group 2: One layer of Single Bond was applied with a micro-brush and light-cured for 20 seconds. Filtek Z250 (A2 shade; 3M ESPE, St. Paul, MN, USA) composite was placed on the ceramic disc and then light cured for 40 seconds.

Group 3: Silane coupling agent was applied on the ceramic surface with a micro-brush, allowed one minute and was then gently blow dried. The remaining steps were the same as those in group 1.

Group 4: The samples were treated with silane as mentioned above. Then the same procedures as in group 2 were performed.

Group 5: Silane coupling agent was applied as in group 3 and then Filtek P90 composite was placed on the ceramic disc and cured for 40 seconds.

Table 2. **Shear bond strength in the groups (MPa)**

| Group | P90 composite | Z250 composite | P ^t value |
|----------------------------|---------------------------|---------------------------|----------------------|
| Adhesive | 10.32 ± 0.37 ^a | 17.47 ± 0.44 ^a | <0.001 |
| Adhesive + silane | 9.95 ± 0.62 ^a | 23.94 ± 0.47 ^b | <0.001 |
| Silane coupling agent | 5.41 ± 0.40 ^b | 17.14 ± 0.52 ^a | <0.001 |
| P^t value | <0.001 | <0.001 | |

t: Student's *t*-test, *: One-way ANOVA. In each column, the mean shear bond strength values with different lowercase letters indicate statistically significant differences (Tukey's post hoc test).

Group 6: The samples were treated with silane as mentioned above. Filtek Z250 composite was placed on the ceramic disc and then cured for 40 seconds.

Specimens in each group were stored in distilled water at 37°C for 24 hours and then all specimens were placed in a thermocycler to undergo 1000 cycles between 5 and 55°C with a dwell time of 30 seconds.

The shear bond strength was measured using a universal testing machine (Zwick Roell, Ulm, Germany) at a crosshead speed of 0.5 mm/minute. During this procedure, the samples were held in the device until failure occurred.

All data were analyzed by SPSS 17 (SPSS Inc., Chicago, IL, USA). One-way ANOVA was applied and pairwise comparison of the means for shear bond strength was carried out using Tukey's HSD test ($P < 0.05$). The Student *t*-test was used to evaluate the correlation among the variables and outcomes.

Results

Table 2 shows the mean and standard deviation of shear bond strength data for different surface treatments. The highest bond strength was found for group 4 (silane + adhesive + Z250). Surface treatment with silane coupling agent and P90 composite (group 5) showed the lowest bond strength.

Using one-way ANOVA in each composite group, the three surface treatments were compared. Tukey's HSD test showed that differences between the first (adhesive + P90) and third (adhesive + silane + P90) groups were insignificant ($P > 0.05$), but bond strength values of these groups were significantly higher than that in group 5. In addition, no statistically significant differences were observed between groups 2 (adhesive + Z250) and 6 (silane + Z250); although group 4 showed significantly higher bond strength than other groups. The Student's *t*-test showed a statistically significant difference between the two composites. Regardless of the type of treatment, Z250 composite demonstrated significantly higher shear bond strength than P90 composite.

Discussion

The clinical success of porcelain repair systems is dependent on the integrity of the chemical or micromechanical bond between porcelain and composite resin. Ceramic surface preparation is an important step in direct repair procedures.¹³ Etching with hydrofluoric acid is effective to improve bond strength between the porcelain and resin, which can cause porosities and enable more resin penetration into ceramic that leads to retentive bond.¹⁴

The results of our study showed that significantly different bond strength values of composite to IPS Empress 2 can be

achieved by different surface treatment methods. The fourth group, which included treatment of IPS Empress 2 with silane coating of etched surfaces, indicated the highest shear bond strength values. These results are in agreement with those of Della Bona et al.,¹⁵ and Filho et al.¹⁶ However, in those studies, the test method was different (microtensile vs. shear test). According to our study, regardless of the type of treatment, shear bond strength of Z250 was higher than that of P90 composite.

Search of the literature yielded no studies about the bond strength of silorane-based composite to ceramic. But Lien et al.¹⁷ showed that silorane-based composite had lower bond strength to the enamel and dentin compared to methacrylate-based composite.

Although silorane based composite has low volumetric polymerization shrinkage, it seems that the difference in bond strength of composite resins is the result of structural characteristics of composites. It is known that methacrylate-based composites have more filler content compared to silorane-based composites which could cause higher bond strength.¹⁸ Less rigidity of methacrylate composites than packable silorane composites reduces contraction stresses at the porcelain-resin interface during polymerization.¹⁹ Moreover, there is a greater degree of subsurface polymerization and depth of curing in methacrylate-based composites than silorane-based composites.²⁰ It might cause less marginal fractures or cuspal deflection;²¹ these factors may explain higher shear bond strength of methacrylate-based composites in comparison to silorane-based composites.

Previous studies showed that silane coupling agent significantly enhanced bond strength of feldspathic ceramics to methacrylate-based composite. Silane coupling agent seems to be a crucial factor for porcelain repair procedures by facilitating chemical adhesion in both inorganic/porcelain and organic/composite surface and increasing the union of dissimilar materials.^{8,22}

Lacy et al.,²³ observed that when silane was not applied the composite bond strength to porcelain was relatively weak. Besides, the values were higher with etching and application of silane and adhesive system than etching and use of adhesive only.²³ The results of the present study corroborate this.

The shear bond strength values of the fourth group were significantly higher than those of the second group. Panah et al.,²⁴ reported that use of silane coupling agent prior to the application of the bonding agent enhanced the repair bond strength. This shows that the bonding agent increases the wettability of the surfaces and furthermore, silane increases the

wettability of the bonding agent enabling it to infiltrate more easily into porosities of ceramic and composite.

In this study, silane coupling agent could not increase the shear bond strength of silorane-based composite to IPS Empress 2 ceramic compared with methacrylate-based composite. Perhaps the reason is that silicon element of silane has a great affinity for compounds that contain available oxygen such as methacrylate-based composite. Reversely, silorane-based composite has less oxygen.²⁵

In addition, the scanning electron microscopic images of silorane-based composite in the study by Hamano et al.,¹¹ revealed only a few areas of uncovered fillers, while almost all of the smaller fillers were covered by matrix. These results indicate that it is unlikely that silane has a significant effect on silorane-based composite bond.

Our study had some limitations to simulate the clinical situations; thus, future studies better simulating the oral environment and clinical loading conditions are required to further confirm the results of this study.

Conclusion

1. Methacrylate-based composite had higher shear bond strength to IPS Empress 2 than silorane-based composite.
2. The low shrinkage property of silorane-based composite does not improve its shear bond strength to IPS Empress 2.
3. Silane coupling agents promote adhesion to methacrylate-based composites and IPS Empress 2 ceramic. However, silane could not effectively increase the shear bond strength of silorane-based composite to IPS Empress 2 ceramic.
4. Among the assessed methods, silane coating with application of adhesive system and etching in methacrylate-based composite was the most efficient surface treatment in terms of bond strength.

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Conflict of Interest

“None Declared.” ■

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