

The Effect of Different Coating Methods on Resin Band Strength to Zirconia

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Abstract

Objective: A clinical challenge of using zirconia frameworks is to achieve adequate bond with different substrates. This study aimed to evaluate the effect of bioglass and silica coating of zirconia substrates on microshear bond strength of resin cement to tetragonal zirconia.

Methods: This laboratory experimental study was conducted on zirconia discs. A total of 120 YTZP zirconia (Zirkonzahn) discs were fabricated and based on surface treatments were categorized into 8 groups of 15 including 1. Control, 2. Sandblast, 3. Etch + bioglass powder coating, 4. Silane + etch + bioglass powder coating, 5. Etch + bioglass slurry coating, 6. Silane + etch+ bioglass slurry coating, 7. Silane + colloidal silica coating, and 8. Silane + etch + colloidal silica coating. Samples were subjected to microshear bond strength testing. In coated groups, thickness of the coating was measured as well. Kruskal Wallis test and ANOVA were applied for intragroup statistical analysis and Dunnett's test and Mann Whitney U test were used for pairwise comparisons.

Results: The mean bond strength of silica-coated samples was significantly lower than the sandblasted specimens ($p < 0.001$). No significant difference was detected in the mean bond strength between specimens with different glass coatings and sandblasted samples. In other words, bond strength of sandblasted and different glass-coated samples was not significantly different. The thickness of coating in the slurry group was significantly less than in other groups.

Conclusion: Bioglass coating could effectively increase the bond strength of resin cement to zirconia in short-term.

Key words: Microshear bond strength, Resin cement, Tetragonal zirconia.

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Introduction:

Yttria-stabilized zirconia or 3Y-TZP has gained the spotlight in dentistry during the recent years. It has been suggested as a substitute for metal and metal-ceramic restorations due to its excellent mechanical properties, high esthetics and biocompatibility (1-3). Advances made in CAD/CAM systems have made it possible to use this high strength ceramic for fabrication of indirect esthetic restorations (1, 3).

Zirconia has a polymorphic nature and exists in three crystalline forms: monoclinic at low temperatures, tetragonal at temperatures higher than 1170°C and cubic at temperatures higher

than 2370°C. Yttria is added to stabilize the tetragonal phase at room temperature (4). This stabilized ceramic undergoes phase transformation; which is responsible for its superior mechanical properties (5). However, the conventional cementing/bonding techniques used for zirconia restorations cannot provide adequate bond strength in many cases (6, 7). Due to its neutral nature, chemical agents such as acids, bases and solvents have no effect on this material (6) and due to its non-silicate composition, silane coupling agent is not effective either. Improvement of zirconia bonding is especially important to increase retention, prevent microleakage and enhance

resistance to fracture and fatigue (4). Several techniques have been used to improve bonding to zirconia such as sandblasting (8-10), tribochemical silica coating (11-15), silicoating (16, 17), selective infiltration etching (18, 19), hot etching (19) and use of phosphate ester monomers (20). In some studies, sandblasting and tribochemical coating of silica had the potential to cause mechanical damage to zirconia structure making it susceptible to radial cracks in service (21). Silicoating, selective infiltration etching and hot etching are complex despite being effective (4). Selective infiltration etching and hot etching have not been evaluated in terms of their mechanical effects (22). Considering the complexity and problems associated with the majority of techniques used to improve resin cement bond strength to zirconia, the present study was designed aiming at using several zirconia surface coating techniques to create an etchable intermediary

layer that enables us to use silane coupling agent. Their effect on increasing the bond strength of resin cement to zirconia was investigated as well. The null hypothesis was that different coating techniques have no effect on bond strength of resin cement to zirconia.

Methods:

This laboratory experimental study was conducted on zirconia discs. A total of 120 zirconia discs measuring 10x7x2 mm were used. These discs were cut from YTZP zirconia blocks (Zirkonzahn) and sintered according to the manufacturer’s instructions. Before any surface treatment, all discs were cleaned in an ultrasonic bath containing 98% ethanol (Bidestan, Iran) for 10 min. Specimens were divided into 8 groups of 15. Study groups are demonstrated in Figure 1.

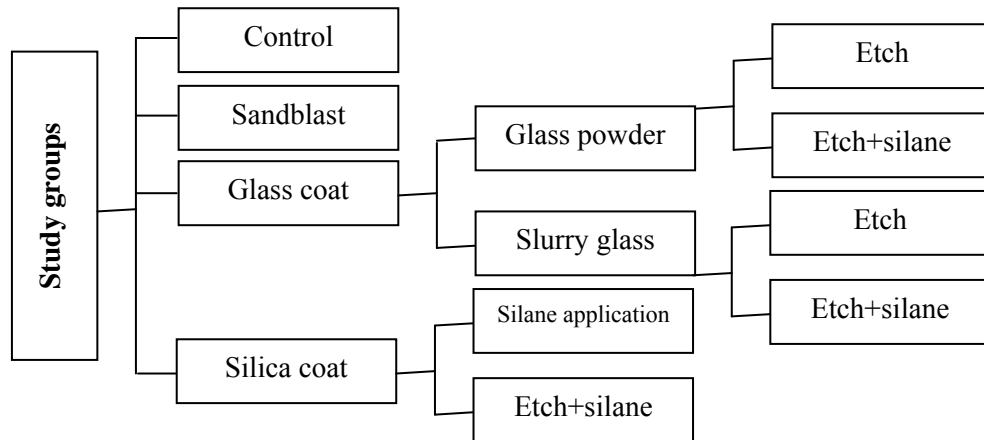


Figure 1- Under study groups

Control group samples received no surface treatment. In the sandblasting group, discs were sandblasted with 50 μ aluminum oxide particles (True Etch, Ortho Technology, Florida) with 4kg/cm² pressure at 10 mm distance for 15 s using micro-etcher machine (Danville Engineering, USA). Then, specimens were cleaned in an ultrasonic bath containing 96%

ethanol for 10 min to eliminate any loose particles resulted from sandblasting from the zirconia surface. Four groups received bioglass coating. Bioglass coating was applied by two methods (30 specimens in each group). In the first group, a layer of PVA (polyvinyl alcohol) binder (Sigma, Germany) was applied to the substrate followed by bioglass powder. The

powder bottle was placed on a flat surface and the powder surface was smoothed using a spatula. After the application of binder, disc surface was placed upside down on the powder surface. In group 2, (second technique), A slurry was prepared from bioglass, distilled water and PVA binder with 500 µg, 1 cc and 1 cc ratios, respectively and applied to the discs using a micro-brush (Fine TPC, Advanced Technology). Specimens were then heated in a furnace with a heating rate of 100°C/hour up to 1200°C and remained at this temperature for 2 hours. Then, they were cooled off at a cooling rate of 200°C/hour. The composition of used bioglass powder was 45S5 bioglass powder with 45 wt.% SiO₂, 24.5 wt.% Na₂O, 24.5 wt.% CaO and 6 wt.% P₂O₅ produced in the Materials and Energy Research Center. Each glass-coated group was divided into two subgroups. In subgroup 1, disc surfaces were etched with hydrofluoric acid (Ultradent Porcelain Etch, Ultradent Products, USA) for 60s, washed with water and air spray for 90s and air-dried. In subgroup 2, disc surfaces were etched with HF acid, silane agent (Ultradent silane, Ultradent Products, USA) was applied to the surfaces according to the manufacturer's instructions and allowed to dry. Two groups received silica coating. Zirconia discs were immersed into colloidal silica, removed, dried and heated in a furnace according to the firing regime described earlier for the glass group specimens. Afterwards, one subgroup was etched with HF acid for 30s, washed for 60s, air dried and received silane application. The second subgroup only received silane.

After preparation of samples, Tygon tubes with 0.7 mm diameter (Tygon, Norton Performance Plastic, Cleveland, OH, USA) were used for the application of Panavia F2.0 cement (Kuraray Medical Inc.) to the disc surfaces. The cement was then light cured for 40s using a diode light-curing unit (Radiolus, SDI). Specimens were immersed in distilled water, stored in an

incubator (Model PL-455G PecoPooya Electronic Co.) at 37°C for 24 hours and then transferred to a microtensile tester (Bisco Inc., USA) for the measurement of micro-shear bond strength. By vertically soldering the cast molds to the jig, the applied tensile load was converted to shear load. The amount of load at failure with a crosshead speed of 0.5 mm/min was recorded by the machine and the micro-shear bond strength values were calculated using the equation below:

$$S=F(N)/A(\text{mm}).$$

Determination of the mode of failure: After the measurement of bond strength, the fractured surfaces were evaluated under a light microscope (Carl, Zeiss, Germany) with an external light source (LED radiation, BO913 Jansjo, China) to determine the mode of failure. The mode of failure fell into one of the following groups:

1. Adhesive failure: Fracture at the cement-zirconia, cement-coating or zirconia-coating interface
2. Cohesive failure: Fracture within one substrate including the cement layer, coating or zirconia
3. Mixed failure: A combination of adhesive and cohesive fractures

Determination of the thickness of coating: In coated groups, thickness of discs before and after coating was measured with a digital micrometer (Mitutoyo, Mitutoyo Corporation, Japan) with 1µ readability. The two values were subtracted to calculate the coating thickness.

The mean, standard deviation, minimum and maximum values of micro-shear bond strength in different study groups were calculated. Data distribution was assessed using Kolmogorov-Smirnov test. Due to the relatively normal distribution (histogram with a normal distribution curve), ANOVA and Dunnett's test were used for statistical analysis. Descriptive values for the thickness of coating were presented in respective tables and Kruskal

Wallis and Mann Whitney U test were used for the comparison of the coating thickness between the coated groups.

Results:

Descriptive bond strength values of the understudy groups are presented in Table 1. Evaluation of bond strength in different groups by ANOVA revealed that study groups were different in terms of bond strength values ($p<0.001$). Dunnett’s test showed that the mean bond strength was significantly different

between all test and control groups. Sandblasted and bioglass coated groups had bond strengths higher than the control group while colloidal silica coated groups had lower bond strength than the control group ($p<0.001$). Thus, the null hypothesis of the study was rejected.

Different groups were compared with the sandblasted group (positive control) in terms of bond strength and a significant difference was found in mean bond strength between silica-coated groups (silica+ silane+ and silica+ etch+ silane) and sandblasted group (-31.28, -31.58 MPa, $p<0.001$).

Table 1- Bond strength descriptive values

Groups	Micro-shear bond strength in MPa				
	Mean	Median	SD	Minimum	Maximum
Control	24.42	23.66	4.148	17.41	30.16
Sandblast	44.84	44.20	7.236	34.58	58.75
Glass powder+ etch	39.50	40.56	7.675	26.52	52.52
Glass powder+ etch+ silane	42.69	41.08	6.952	32.00	59.54
Slurry glass+ etch	38.38	37.44	4.719	32.86	46.28
Slurry glass + etch+ silane	42.41	40.82	7.716	30.42	55.38
Silica + silane	13.55	11.18	5.525	9.09	27.56
Silica+ etch+ silane	13.25	11.18	4.719	8.31	27.04

In contrast, no significant difference was noted in mean bond strength between glass-coated groups and sandblasted group. In other words, the sandblasted group had no significant difference with glass-coated groups in terms of bond strength. The only exception was the slurry glass+ etch group that had a bond strength lower than 6.45 MPa compared to the sandblasted group and this difference was borderline significant ($p=0.05$).

In glass powder coated groups, all mixed fractures had occurred at the interface of cement to glass coat. In the slurry glass + etch group, 91.66% of mixed failures were at the cement-glass interface while 8.33% occurred at the glass-zirconia interface. In the slurry glass+ etch+ silane group, 80% of the mixed failures had occurred at the cement-glass interface while 20% had occurred at the glass-zirconia interface.

In this group, adhesive failures had occurred at the interface of glass-zirconia. In silica-coated groups, zirconia surface was bare in all adhesive failures.

Thickness assessment:

Descriptive values for the thickness of coating in different groups are presented in Table 2. Considering the sample size, Kruskal Wallis test was used for the analysis of thickness variable and a significant difference was noted in this respect between groups. In pair wise comparison by Mann Whitney U test, a significant difference was found between glass powder and slurry glass ($p=0.004$) and glass powder and silica ($p=0.004$) groups. Thickness of coating in the glass powder group was significantly higher than in the other two groups. The difference between slurry glass and silica groups was borderline significant ($p=0.078$).

Table 2- Coating thickness descriptive values

Groups	Coating thickness in micron				
	Mean	Median	SD	Minimum	Maximum
Glass powder	270.83	283.50	58.595	191	352
Slurry glass	46.67	37.00	23.594	29	91
Silica	76.50	79.00	21.815	40	108

Discussion:

In this study, we evaluated the effect of two types of bioglass coating and one type of silica coating on the zirconia surface to increase the bond strength of zirconia to resin cement. The micro-shear bond strength values were significantly different between test and control groups. Thus, null hypothesis of the study was rejected. In our study, sandblasting method was considered as the positive control group and different surface treatments were compared with sandblasting. Air abrasion in some studies is considered as the most effective zirconia surface treatment technique. It increases the surface roughness and causes micromechanical interlocking of the luting agent (6, 20, 23). In the present study, bond strength values in experimental groups significantly increased compared to the control group; which is in agreement with the results of previous studies.

In our study, in one group a type of bioglass was used to form a coating on zirconia substrate using firing technique to create an intermediary etchable layer on zirconia surface. In several studies, this technique has been employed to benefit from the bioactive glass characteristics and mechanical performance of zirconia in zirconia implants (24-26). One primary requirement for a coating material is to have a coefficient of thermal expansion (CTE) close to that of substrate (24, 25). Almost similar CTEs of bioglass and zirconia prevent crack formation in glass or at the glass-substrate interface due to residual thermal stresses when cooling off to room temperature (24).

Bond strength values of different glass-coated groups were comparable to that of sandblasted

group (positive control). The only exception was the slurry glass+ etch group that had a lower bond strength than the sandblasted group and this difference was borderline significant. In the slurry group, a slurry was prepared by mixing glass powder, water and binder and applied to the zirconia surface. In this technique, smaller amounts of glass particles are applied to the surface compared to the glass powder group and thus the coating might be incomplete in some areas. Nonetheless, in this group, when silane was applied after etching, the bond strength value increased to the level of sandblasted group. Due to the dissimilarity of the used materials and preparation methods of samples in different studies, precise comparison of results is not feasible. In a study by Valentino *et al.* (2012) use of glaze (Cercon Liner, Degudent) yielded higher bond strength than sandblasting with 50 μ alumina particles (27); which is different from our obtained result. Considering the similar sandblasting conditions in the two studies, this difference may be explained by the different composition of the coating layer and duration of acid etching since the etching time was shorter (20s) in Valentino's study (2012) (27). In a study by Cura *et al.* in 2012, acid etching of zirconia surface after applying glaze and silane increased the shear bond strength of resin cement but in cases where MDP-containing primer was used instead of silane, etched glaze layer could not efficiently increase the bond strength (28). In a study by Usumez *et al.* in 2013, MDP-containing primers in glazed and etched groups were not as effective as in the sandblasted group (29). MDP enhances the bond between resin cement and zirconia ceramic (8,

20). It seems that the glaze coating of the zirconia surface neutralizes the effect of MDP-containing primers (29). We used Panavia resin cement in our study' which contains MDP monomer. However, glass-coated groups after etching showed bond strength values as high as that of the sandblasted group. In comparison with previous studies, this increase may be attributed to the different composition of glass coat and higher surface roughness and micromechanical retention of etched groups. In the mentioned study, a glaze coat with a low melting point was used forming an amorphous layer. But, in our study, crystallization of glass coat occurred during firing regime and thus, after etching with HF acid, a different etched pattern was obtained.

Use of silane in glass-coated groups could not significantly increase the bond strength but this small increase in the slurry glass group raised the bond strength to the level of sandblasted group. Use of silane in silica-based ceramics causes the formation of Siloxane network on the ceramic surface and increases the bond strength of ceramic to resin cement (6). In the study by Valentino *et al.* in 2012 glaze (Cercon Ceram Liner, DeguDent) was used as coating but could not significantly increase the bond strength after etching and application of silane; this finding is in agreement with our results. They explained the reason to be the loss of a significant part of glaze layer due to etching and sandblasting (27). Kitayama *et al.* in 2009 showed that use of silane on coated porcelain (Cercon Ceram Kiss, DeguDent) significantly increased the bond strength to resin cement; which is in contrast to our obtained results (30). In our study, due to the silica content of glass coat, use of silane increased the bond strength; but due to having small silica content (45%) in comparison to the feldspathic porcelains, this increase was insignificant.

In silica-coated groups, bond strength values were significantly less than the control and

sandblasted groups. It seems that this method of coating has not been successful which might be due to the inadequate wetting of the zirconia surface by silica, not using a binder or in coordination between the CTEs of silica and zirconia.

Lower bond strength values than the control group in experimental groups are attributed to the presence of silica with a weak bond at the cement-zirconia interface interfering with the bond.

Among the glass-coated groups, most fractures were mixed (83.33%) in the glass powder+ etch group. By using silane coupling agent, the frequency of mixed failure decreased and added to the frequency of cohesive failure of cement. This finding shows the effect of silane on improving the resin cement bond to glass. Additionally, in these groups, no failure was observed at the interface of glass layer and zirconia substrate and thus, we may state that the bond between the glass layer and zirconia in glass powder coated specimens was stronger than the bond between the resin cement and glass. In the slurry glass+ etch group, all failures were of mixed type. In one specimen, the mixed failure had occurred at the interface of glass-zirconia; which shows the weaker bond of glass in this coating technique or incomplete coating of surface that is in agreement with the results of bond strength testing. In this method of coating, after the use of silane, cohesive failure occurred in resin cement in 8.33% of cases that indicates increased bond strength to resin cement after the application of silane. Furthermore, in the mentioned group, one adhesive failure and two mixed failures occurred at the interface of glass-zirconia. This finding shows weaker bond of glass in this coating method or incomplete surface coating.

In the study by Kitayama *et al.* in 2009 no adhesive failure occurred at the zirconia-veneer interface in the porcelain-coated group. No cohesive failure occurred in veneering porcelain

either showing that the bond between the veneering porcelain and zirconia was stronger than the bond between the cement and porcelain (30). In a study by Everson *et al.* in 2012, SEM analysis demonstrated that the majority of failures in the glazed group were of mixed type (31); which is in accord with our obtained results.

In silica-coated groups, all failures were of adhesive type and observed at the interface of coating and zirconia; which further confirms inadequate bonding between the silica layer and underlying zirconia.

Thickness assessment:

Among different groups in this study, the slurry glass group had the thinnest coating thickness. In a study by Ferraris *et al.* in 2000 zirconia samples were directly coated with bioglass powder yielding a coating thickness of 100-300 microns (24). This finding is in agreement with our results. Krajewski *et al.* in 1998 used glass suspension and the thickness of the obtained layer based on the fluidity of suspension used varied between 40 to 100 microns (25). Everson *et al.* in 2012 used a glaze-on technique and reported a 120 micron thickness. CAD/CAM technology has the ability to consider the thickness of the internal coating to preserve the fit and seating of the restoration. However, adhesive bridges with retaining wings have simpler geometry and subsequently less

problems in terms of fit (31).

Conclusion:

Within the study limitations, the following conclusions were drawn:

1. Bioglass powder/slurry coatings can be applied to zirconia surface using firing method
2. Bioglass powder coating is effective for short-term increase in bond strength of resin cement to zirconia
3. Bioglass slurry coating is effective for short-term increase in bond strength of resin cement to zirconia
4. Bioglass coating of zirconia surface is as effective as the sandblasting technique in increasing the bond strength to resin cement in short-term
5. Colloidal silica coating of zirconia surface as applied in this study was not successful in increasing bond strength to resin cement
6. Colloidal silica coating reduced the bond strength of zirconia to resin cement

Bioglass slurry coating yielded the thinnest thickness of coating compared to other methods

Conflict of Interest: “None Declared”

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