International Journal of Dentistry and Oral Science (IJDOS)
ISSN: 2377-8075

# Evaluation Of The Internal Surface Treatment Type Of Zirconia On The Shear Bond Strength Of Different Patternsofresin Cements (In Vitro Study)

Research Article

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## Abstract

**Aim:** to evaluate the shear bond strength of zirconia bonded with two dual cure adhesive resin cements PANAVIA F 2.0 and PERMAFLO DC using two surface treatment techniques: sand blasting and silica coating using Cojet system.
**Materials and Methods:** 60 cubes samples with dimensions (10×10×10mm) and 60 cylinders samples with dimensions (3mm diameter, 2mm height ) were cut from three zirconia ceramic blocks. Sandblasting was done using air blasting machine with 110 µm AL2O3 particles and Silica coating surface treatment was done using Cojet system with 30 µm $SiO_2$ particles. Half of the cylinders sandblasted zirconia samples were cemented to half the sand blasted cubic samples using PANAVIA F 2.0 adhesive resin cement and the rest were cemented using PERMAFLO DC adhesive resin cement. Also, half of the small silica coated zirconia samples were cemented to half the silica coated large samples using both cements. Thermocycling were done for 5000 thermal cycles. The shear bond strength was tested using a computerized universal testing machine.
**Results:** Zirconia specimens cemented with PANAVIA F 2.0 showed higher shear bond strength than specimens cemented with PERMAFLO DC with silica-treated zirconia surfaces and sandblasted surface treatments.
**Conclusion:** PANAVIA F 2.0 resin cement and silica coating surface treatment could be the best cement and surface treatment for zirconia and sand blasting could be a promising alternative surface treatment.

## Introduction

A need for non-metallic restorative materials with optimal esthetics and characteristics such as biocompatibility, color stability, high wear resistance and low thermal conductivity is often stated as a reason for the use of ceramics in dentistry. Various materials can be used as all-ceramic core materials such as leucite-reinforced ceramics, glass-infiltrated ceramics, lithium disilicate, alumina and zirconia [1].

The use of zirconia in restorative dentistry has grow exponentially over the past decade [2, 3].

In clinical dentistry, zirconia is used for construction of orthodontic brackets, posts and cores, implants and implants abutments, crown substructure and frameworks for fixed partial dental prostheses [4].

In addition to its favorable mechanical properties and chemical and dimensional stability, zirconia substructure exhibits good radio-opacity, enhancing radiographic evaluation of marginal integrity and detection of recurrent caries. Zirconia offers several advantages including high flexural strength and metal free structure. It also demonstrates excellent optical properties, biocompatibility and low heat conductivity making it one of the most efficient material both for anterior and posterior restoration [5].

Establishing a strong bond with zirconia is only one part of the problem. A more crucial aspect would be maintaining this bond under the influence of fatigue conditions, in presence of saliva and temperature changes for a clinically acceptable time.

To enhance the bond strength of luting cement to the ceramic surface, a number of techniques have been reported which mechanically facilitate resin ceramic bonding. Etching the inner surface of a restoration with hydrofluoric acid followed by the application of a silane coupling agent is a well-known and recom-

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**Received:** September 10, 2021
**Accepted:** October 01, 2021
**Published:** October 13, 2021

**Citation:** Ibrahem Abu shanab, Eyad Swed, Muaaz Alkhouli. Evaluation Of The Internal Surface Treatment Type Of Zirconia On The Shear Bond Strength Of Different Patterns of resin Cements (In Vitro Study). *Int J Dentistry Oral Sci.* 2021;8(10):4746-4752. doi: http://dx.doi.org/10.19070/2377-8075-21000963

mended method to increase bond strength. Although hydrofluoric acid is efficient in roughening feldspathic ceramic for bonding composite resin [6-8], neither etching with these solutions nor adding silane resulted in an adequate resin bond to some new ceramics [9-12]. Particularly high-alumina [13, 14] or zirconia ceramics [15-17] cannot be roughened by hydrofluoric acid etching since such ceramics do not contain a silicon dioxide (silica) phase. For this reason, special conditioning systems are indicated for these types of ceramics.

Advances in adhesive dentistry have resulted in the recent introduction of modern surface conditioning methods that require airborne particle abrasion of the surface before bonding in order to achieve high bond strength. One such system is silica coating. In this technique, the surfaces are air abraded with aluminium trioxide particles modified with silica [18, 19]. The blasting pressure results in the embedding of these silica coated alumina particles on the ceramic surface, rendering the silica-modified surface chemically more reactive to the resin.

There has been no concensus in the literature regarding the best surface conditioning method for optimum bond strength depending on the luting cements or ceramics used [20]. Therefore, the objectives of this study were to evaluate the effect of current surface conditioning methods on the bond strength of a resin luting cement bonded to ceramic surfaces and to identify the optimum method to be used for conditioning the ceramics prior to cementation.

## Materials And Methods

60 cubes samples with dimensions (10×10×10mm) and 60 cylinders samples with dimensions (3mm diameter, 2mm height), picture (1.2) were cut from three zirconia ceramic blocks using low speed precision saw machine. Sandblasting was done using air blasting machine with 110 μm AL2O3 particles for 30 samples of each (cubes & cylinders). The abrasive was applied to the zirconia surface samples at a distance of 10 mm between the surface of the samples and the blasting tip at 2.5 bar pressure for 10 seconds. Silica coating surface treatment was done using an extra oral blaster with 30 μm SiO2 particles for the remaining 30 samples of each (cubics & cylinders). The abrasive was applied perpendicular to the surface of the zirconia surface samples at 3 bar pressure for 15 seconds at a 10 mm distance.

**Cementation of the samples:**

Zirconia specimens were divided into 2 groups according to surface treatment: Group 1: The specimens were sandblasted with (110μm) $Al_2O_3$ (30 specimens cubes & cylinders).

Group 2: The specimens were silica coated with high purity (30μm) SiO2 using COJET system(3m ESPE AG. ESPE Platz. Seefield. Germany) (30 specimens cubes & cylinders).

The zirconia samples were sanded and coated with silica using a dimension adjuster that was designed so that the distance between the sanding grip head and the sanded surface was 10 mm.picture (3)

Each group was subdivided into 3 subgroups (10 specimens each) according to the type of adhesive resin cements (n = 10).

Subgroup A1: The specimens were sandblasted and cemented with PANAVIA F2.0 (PANAVIA F2 F2:0 Kuraray, Okayama, Japan) adhesive resin cement (10 Specimens).

Subgroup A2: The specimens were sandblasted and cemented with PERMAFLO DC (PERMAFLO DC ultradent,salt lak city,UT,USA) adhesive resin cement (10 Specimens).

Subgroup A3: The specimens were sandblasted and cemented with PERMAFLO DC adhesive resin cement after conditioning the zirconia surfaces with CLEARFIL(CLEARFIL Ceramic Primer, Kuraray, Okayama, Japan) ceramic primer (containing MDP) (10 Specimens).

Subgroup C1: The specimens were silica coated with and cemented with PANAVIA F2.0 adhesive resin cement (10 Specimens).

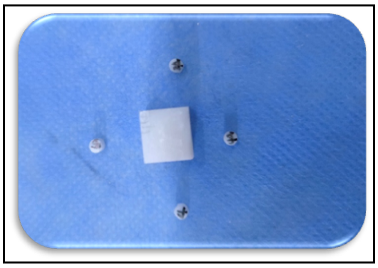
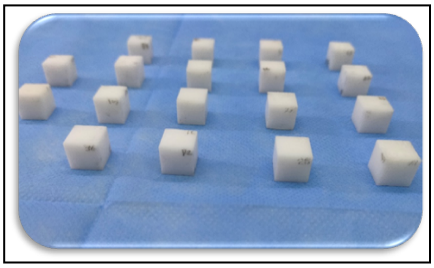
Subgroup C2: the specimens were silica coated with and cemented with PERMAFLO DC adhesive resin cement (10 Specimens).

Subgroup C3: the specimens were silica coated with and cemented with PERMAFLO DC adhesive resin cement after conditioning the zirconia surfaces with CLEARFIL ceramic primer (containing MDP) (10 Specimens).

The cylinders zirconia samples were seated on the center of surface of the cubes zirconia samples and a standardized load (3kg) was applied using a Using the force application device located in the Department of Fixed Prosthodontics, Damascus University picture (2), after which the curing was done first for 5 seconds and then the appendages were removed with a small brush to complete the final curing for 20 seconds on each side of the cube and then the application of Oxyguard glycerine gel; (Kuraray Co Ltd) for ten minutes. The samples were then washed with a stream of air and water and thermocycled at 37°C in a water bath for 24 hours before being tested.

**Evaluation of shear bond strength of the cemented specimens:**

**Figure 1. The Samples (cubes & cylinders).**

The shear strength test (it is the ISO approved test in international scientific research to test the bonding strength between two materials) was carried out by the General Mechanical Testing Device at the Industrial Research Center in Damascuspicture 4, where a cubic zirconia was installed between the jaws of the device so that the device head was as close as possible to The interface (cylinder - cube) at speed of 0.5 mm/min and the forces were applied to the samples, then the applied forces were calculated by the computer of the device, where the forces continued to be applied until the zirconia cylinders were debonded from the cubic zirconia, then the values of the shear forces in Newtons were recorded.

The load at failure was divided by interfacial bonding area to express the bond strength in Mpa.

### Statistical analysis

The collected data were tabulated and statistically analyzed to evaluate shear bond strength of different adhesives to zirconia. Data were collected and recorded on Excel from Microsoft. Then, statistical tests were conducted using SPSS v.25 (IBM, USA) program with a significance level of 0.05.

Two-way ANOVA was used to study the effect of sanding and luting material on the shear strength resistance of zirconia cylinders with cubic zirconia. One-Way ANOVA with Games-Howell test was used to study the simple main effect of adhesive. The independent samples t-test was used to study the simple main effect of surface treatment and their interaction on shear bond strength for all groups.

## Results

### Shear bond strength

The mean shear bond strength of silica-coated zirconia specimens cemented with PANAVIA F2.0 cement (C1) (21.8) was higher than that of sandblasted zirconia specimens cemented with PANAVIA F2.0 cement (A1) (20.5) and the difference was NOT significant (p=0.669) as shown in Table (1). The mean shear bond strength of sand blasted zirconia specimens cemented with PERMAFLO DC cement (A2) (6) was higherthan that of silica-coated zirconia specimens cemented with PERMAFLO DC cement (C2) (2.4) and the difference was significant (p= 0.009) as shown in Table (2). The mean shear bond strength of silica-coated zirconia specimens cemented with PERMAFLO DC cement after conditioning with CLEARFIL primer (C3) (16.8) was higherthan that

**Figure 2. The force application device.**

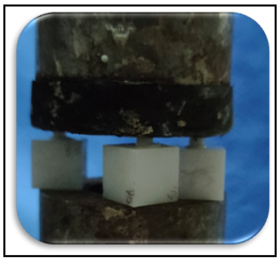


**Figure 3. Dimension Adjustment Tool.**

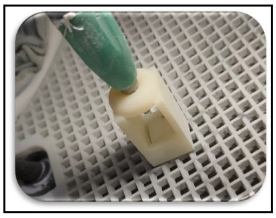


**Figure 4. Universal mechanical Testing Device.**

**Figure 5. Cementation of cylinder on a cube.**

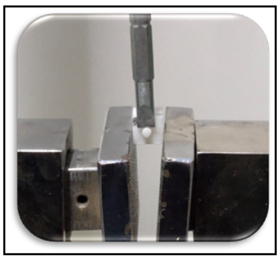


**Figure 6. Electron microscope image showing the effect of coating on the surface of zirconia (CoJet system).**

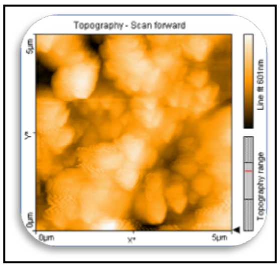



**Figure 7. An electron microscope image showing sandblasting on the surface of zirconia with 110µm aluminum oxide particles.**

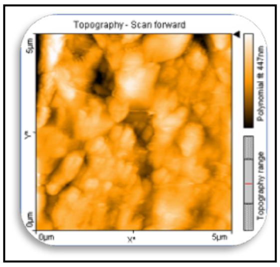
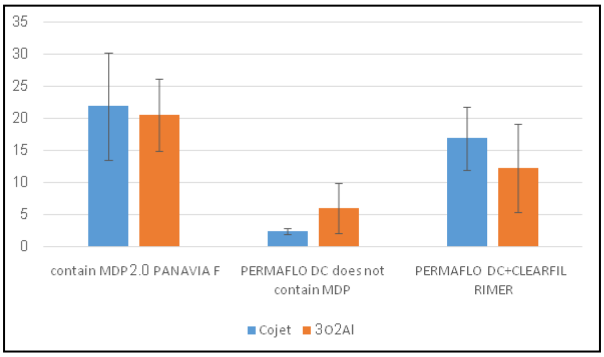



**Table 1.**

| adhesive resin cements | Sand blasting AL2O3 (A) | silica-coating CoJet(C) | P |
|---|---|---|---|
| PANAVIA F2.0 (1) | 20.5 ± 5.6 | 21.8± 8.4 | 0.669 |
| PERMAFLO DC (2) | 6 ± 3.9 | 2.4± 0.5 | 0.009 |
| PERMAFLO DC+CLEARFIL RIMER(3) | 12.2± 6.9 | 16.8± 4.9 | 0.103 |

**Table 2.**

| Treatment type | Adhesion cements | SMA | Standard Deviation | Minimum Value | Maximum Value | The Confidence Interval is 95% for the Mean | |
|---|---|---|---|---|---|---|---|
| | | | | | | Minimum | Maximum |
| CoJet 50% group (C) | PANAVIA F2.0 (C1) | 21.8 | 8.4 | 13.2 | 35.4 | 15.8 | 27.9 |
| | PERMAFLO DC (C2) | 2.4 | 0.5 | 1.7 | 3.5 | 2 | 2.7 |
| | PERMAFLO DC+CLEARFIL RIMER( MDP (C3) | 16.8 | 4.9 | 10.5 | 27.3 | 13.3 | 20.3 |
| Al2O3 50% group(A) | PANAVIA F2.0 (A1)) | 20.5 | 5.6 | 14.3 | 29.7 | 16.4 | 24.5 |
| | PERMAFLO DC (A2) | 6 | 3.9 | 1.5 | 13.1 | 3.2 | 8.8 |
| | PERMAFLO DC+CLEARFIL RIMER( MDP) ( A3) | 12.2 | 6.9 | 4.5 | 21.3 | 7.2 | 17.1 |




of sandblasted zirconia specimens cemented with PERMAFLO DC cement after conditioning with CLEARFIL primer (A3) (12.2) and the difference was not significant (p=0.103).

## Discussion

A durable and stable bond between dental tissue, luting cements and ceramics is fundamental for the long-term performance of all ceramic restoration.

In this study it was done using (twin sample technique) as bonding cylinderszirconia samples with dimensions (3mm diameter, 2mm height) to cubes zirconia samples with dimensions (10×10×10 mm) The dimensions of the sample were chosen in this way in order to match the shape and size of the holders of the General Mechanical Testing Device (Testometric (M350-10)) located in the Center for Industrial Research and Tests in Damascus, also which is in the range of sample dimensions of most previous studies to give same change after surface treatment and thermocycling procedure [21, 22].

As zirconia is resistant to traditional ways of surface treatments like acid etching due to its silica and glass-free polycrystalline structure, the current study used sandblasting to increase surface roughness and allow formation of zirconium oxide layer to improve bond strength between zirconia and adhesive resin cement by chemical bond.

Zirconia samples received airborne particle abrasion with 110µm aluminum oxide ($Al_2O_3$) particles at 2.5 bar pressure using an air abrasion device for 10 sec from a distance of 10mm perpendicularly to the surface [23, 24].

The current study selected 110µm as a sample size because big size powder produce higher surface roughness than small size particles (50 µm) thereby produce higher micromechanical retention [25, 26].

Duration of sandblasting was selected in this study for 10 sec because sandblasting for long period of time causes sharp margins in surface topography that acts as stress points lead to formation and propagations of cracks that can adversely affect the fracture resistance of zirconia [7].

Due to lack of silica in zirconia, silica coating techniques have been explored to utilize the chemical bonding provided by MDP monomer. The use of tribochemical silica coating is a common practice for coating zirconia-based dental ceramics with silica [27]. Using the CoJet system being the most heavily favored commercial products utilized for applying the coating. The tribochemical technique air-abrades particles is appropriate to produce surface treatment with nano- particles than do with large particles size [28].

The bonding was done using dual-curing resin cements (PANAVIA F2 F2:0, PERMAFLO ) because the curing begins with photoactivation in the light-reached places and the curing process continues chemically in the far-unreached places [29].

Also, PERMAFLO DC dual-curing resin cement that does not contain functional phosphate monomers (MDP) was used to compare its effectiveness with PANAVIA F2:0 cement containing (MDP) and study the effect of surface treatment and thermal cycles on the bond strength of each of them [30].

Methacryloyloxyde Dihydrogen Phosphateext monomers in adhesive cements have been proven to be effective for adhering to the non-silica-based polycrystalline materials of zirconia. Numerous studies have shown that phosphate monomers are promising chemical agents for improving zirconia bonding [10]. The possible mechanism is the ability of phosphate monomers to form chemical bonds with zirconia oxide layer on the surface, and have polymerizable resin terminal end groups (eg, methacrylate), which enable cohesive bonding to appropriate resin cements [11, 31].

Cementation of the samples was done using the force application device located in the Department of Fixed Prosthodontics, Damascus Universitythat allowed application of standardized load of 3 kg for all samples. This load was chosen in many studies to avoid the risk of damaging the zirconia samples [32].

Aging by thermocycling was undertaken in this study to examine the effect of simulated in vivo temperature variations on the strength of the bond at the resin/ceramic interface [33].

Shear bond strength not only evaluate the bond strength of adhesive/substrate combination, but also the effectiveness of the surface treatment of the substrate on the bond. It also provides a means of comparing different bonding material [16].

The result of the current study showed that shear bond strength of silica-coated zirconia specimens cemented with PANAVIA F2.0 cement (21.8 Mpa) was higher than that of sandblasted zirconia specimens (20.5 Mpa) and the difference was not significant. The higher bond strength values for the silica coated zirconia specimens may be explained as silica coating leave a physically and chemically active outer surface layer (oxide layer) that produced by silica coating more than sand-blasting surface treatment, this silica coating promotes a chemical bonding with phosphate monomer (MDP) which is an ingredient of a composition of PANAVIA F2.0 [17]. This result in agreement with previuos studies that reported silica coating treatment with PANAVIA F2.0 provides a strong and long lasting resin zirconia bond [13].

Also the result of this study recorded that the shear bond strength of silica-coated zirconia specimens cemented with PERMAFLO DC cement after conditioning the zirconia surface with CLEARFIL primer (16.8 Mpa) higher than that of sand blasted zirconia specimens cemented with PERMAFLO DC cement after conditioning the zirconia surface with CLEARFIL primer (12.2 Mpa) and the difference was not significantThis result can be explained in the same way as the previous one, except that the difference between this group and the previous one is that the MDP is separate from the cement structure within the CLEARFIL ceramic primer group [17].

finally the result of this study recorded that the shear bond strength of sand blasted zirconia speci¬mens cemented with PERMAFLO DC cement (6 Mpa) was higher than that of silica-coated zirconia specimens cemented with PERMAFLO DC cement (2.4 Mpa) and the difference was significant. The reason may be due to the Cojet system (30µm Sio2) lead to Si deposition that might tend to produce a surface with lower roughnessand consequently lowers the possibility of mechanical interlocking with PERMAFLO DC adhesive resin cement [34].

Sand blasting surface treatment with (110µm $AL_2O_3$) particles produces more roughness on zirconia surface than silica coatingpicture (6.7) and obtains micromechanical retention on the zirconia surface more than silica coating with PERMAFLO DC adhesive resin cement [35].

Likewise, high viscosity of the PERMAFLO DC causes poor penetration of the cement to the small pores caused by silica coating and good penetration to the large pores caused by sand blasting [36].

This result is in agreement with the finding of several studies which reported that bond strength to zirconia was not improved after silica coating compared to airborne paricle abrasion. However, this result was contradicting to the results of other studies which reported that silica coating improved bond strength to zirconia ceramics compared to sand blasting [37].

According to the results of this study the shear bond strength of sand blasting zirconia specimens cemented with PANAVIA F2.0 cement (20.5 Mpa) was higher than that of sand blasting zirconia specimens cemented with PERMAFLO DC cement after conditioning the zirconia surface with CLEARFIL primer (12.2 Mpa) which was higher than that of sand blasting zirconia specimens cemented with PERMAFLO DC cement (6 Mpa) with significant difference. This high bond of PANAVIA F2.0 group &CLEARFIL primer group can explained here by the (MDP) monomer present either within the structure of (PANAVIA F2.0) or in the ceramic primer (CLEARFIL) separated from the risen structure of PERMAFLO DC, which forms a chemical bond with the surface of zirconia, MDP is a functional group with a long hydrophobic chain molecule with two ends. One end has a vinyl group that reacts with the monomer of the resin cement when polymerized. At the other end, hydrophilic phosphate ester groups bond strongly with zirconia oxide layer [38].

It was also noticed that also the shear bond strength of silica coated zirconia specimens cemented with PANAVIA F2.0 cement (21.8 MPa) was higher than that of silica-coated zirconia specimens cemented with PERMAFLO DC cement after conditioning the zirconia surface with CLEARFIL primer (16.8 MPa) which was higher than that of silica-coated zirconia specimens cemented with PERMAFLO DC cement (2.4 MPa) and the difference was significant.

This high bond of (PANAVIA F2.0) can be explaine by its content of MDP that produces chemical bond with oxide layer created by silica coating in zirconia surface in addition to mechanical bond created by the roughness of silica coating while PERMAFLO DC cement depends only on mechanical bond created by silica coating on zirconia surface [39].

Another reason may be due to low viscosity of PANAVIA F2.0 that increases surface wettability and in¬creases penetration of PANAVIA F2.0 cement to the small pores that caused by silica coating that leads to high bond strength than PERMAFLO DC which is more viscous [40].

Tribochemical silica coating is a type of surface treatment in which zirconia surface is abraded with aluminium-oxide particles modified by silica and the blasting pressure results in embedding of silica particles on zirconia surface which results in chemical bond between zirconia surface and PANAVIA F2.0 adhesive resin cement as phosphate ester group of MDP binds directly to zirconia oxide. The efficacy of this surface treatment has been demonstraded in previous studies [41].

## Conclusion

Within the limitations and conditions of this in vitro study, it could be concluded that:

1. Silica coating surface treatment improved Shear bond strength of PANAVIA F2.0 as the CoJet system promotes surface roughness and provides micromechanical retention and also a chemical bond.
2. Silica coating surface treatment of zirconia specimens showed inferior bond strength for PERMAFLO DC in comparison with the sand blasting surface treatment.
3. Sand blasting surface treatment of zirconia specimens showed high result of shear bond strength than silica coating with PERMAFLO DC as sand blasting with 110 µm AL2O3 obtains micromechanical retention on zirconia surface more than silica coating.
4. PANAVIA F2.O produced high bond strength in the zirconia specimens treated with silica coat¬ing and sand blasting more than PERMAFLO DC.
5. zirconia/resin bond strength could be significantly improved using sandblasting & silica coating techniques in combination with MDP monomer.

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