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# Effect of Sandblasting and Zirconia Primer Application on The Zirconia-Cement Shear Bond Strength (An in-vitro Study)

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## *ABSTRACT*

**Aim:** This study was carried out to evaluate the effect of two types of surface treatments; Sandblasting ($Al_2O_3$ 110 m) and Zirconia primer (Z-Prime Plus) applicaition on zirconia-resin cement shear bond strength. **Materials and Methods:** 28 zirconia discs were specially constructed for this study by CAD-CAM machine of dimensions 10 mm diameter and 5 mm thickness. The discs were divided into two groups according to sandblasting surface treatment as the following: Group (I):14 zirconia discs without sandblasting and Group (II):14 zirconia discs after being subjected to sandblasting. These groups are subdivided into two subgroups each according to Z-Primer Plus application. Subgroup A: 7 zirconia discs without primer application and Subgroup B: 7 zirconia discs with primer application. Sandblasting was carried out to fourteen zirconia discs using 110 $\mu$m($Al_2O_3$). Zirconia Primer (Z Primer Plus) was applied to 14 zirconia discs.28 composite discs were fabricated according to manufacturer's instructions with dimensions 5mm diameter and 2.5mm thickness. Cyclic loading was done by 10,000 Cycle, frequency 1.6 Hz, Torque; 2.4 Nm and Weight 3 kg (30 N) and also thermocycling was done, at5$^o$C and 55$^o$C and number of cycles is 500 cycles. Testing shear bond strength was done for all samples using universal testing machine. **Results:** It was found that the highest shear bond strength mean value recorded for group (IIB) followed by group (IB) then group (IIA) while the lowest mean value recorded for group (IA) The difference between different surface treatment groups was statistically **significant** as revealed with one way ANOVA test ($p<0.05$). Pair-wise Tukey's post-hoc test showed non-significant ($p>0.05$) differences between {(IIB) and (IB)}, {(IB) and (IIA)} and {(IIA) and (IA) }.It was noted that during thermo-mechanical aging a total loss of two samples in group (IIA) and only one sample in group (IA) and their results recorded as zero so not indicated in mean value calculation. **Conclusions:** The bonding to zirconia is affected by mechanical and chemical surface treatments which are sandblasting and primer application.

***KEYWORDS***

*zirconia primer,*
*shear bond strength.*

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## INTRODUCTION

All-ceramic restorations are a potentially more effective method compared with metal ceramic restorations in clinical situations requiring highly demanding esthetic restorations.

Due to its high fracture toughness and chemical durability, yttria tetragonal zirconia polycrystal (Y-TZP) is one of the most frequently used materials today.However, the bond with Y-TZP is difficult to establish due to its acid-resistant and silica-free surface [(1)].

To reproduce highly esthetic tooth-colored fixed dental prostheses, computer aided design- computer aided manufacturing (CAD-CAM) techniques are becoming frequently used. CAD-CAM makes it possible to achieve superior esthetic results in easy steps [(2)].

Zirconia ceramic posses a unique biocompatibility, as well as favorable physical and mechanical properties.Transformation toughening is an important characteristic of yttria- stabilized tetragonal zirconia polycrystalline (YTZP) ceramics, whereby crack propagation can be resisted when the crystalline structure transforms from the tetragonal to the monoclinic phase. Nevertheless a drawback to Y-TZP ceramics is its limited capability to adhere to resin cements because of its glass free, polycrystalline microstructure. Several surface treatment methods, both chemical and mechanical, have been used to overcome this problem. Improvement in the bond between resin cement and Y-TZP ceramics was reported when the airborne-particle abrasion technique was used. Airborne-particle abrasion produced a micro roughened zirconia surface with irregularities. This surface topography enhanced the bonding by increasing surface energy and wettability to promote micromechanical interlocking with luting agents. Nevertheless, it has been suggested that use of resin luting agents can heal minor cracks produced by airborne- particle abrasion, leading to strengthening of the ceramic materials. Therefore, innovative adhesive strategies combining surface treatment with chemical bonding have been developed [(3)].

Several recent resin cements, known as self adhesive cements, contain phosphorylated methacrylate monomers. These materials may interact chemically with the zirconia surface. With two phosphate groups and at least two double-bonded carbon atoms, self-adhesive cements are expected to bond to zirconia similarly to other phosphate-based adhesive materials. In addition, new phosphate monomer–based primers are now available to promote chemical adhesion between resin materials and hydroxyl groups present on the zirconia surface. One of these phosphate-based materials, Z-Prime Plus (Bisco Inc, Schaumburg, IL, USA), is a primer that includes a mixture of phosphate and carboxylate monomers to enhance the bonding to zirconia and metals, according to the respective manufacturer. This primer has been shown to enhance the adhesion of conventional and self-adhesive resin cements to air-abraded zirconia[(4)].

This study, therefore, was to evaluate the effect of mechanical (i.e sandblasting) and chemical (i.e zirconia primer) surface treatments to zirconia on shear bond strength by $Al_2O_3$ sandblasting and Z-Prime Plus application to zirconia surface then exposing all samples to both thermocycling and cyclic loading to simulate the oral cavity conditions for more reliable results. The research hypothesis was that surface treatment of zirconia by sandblasting and primer application would result in increased bond strength regardless of the luting cement type [(5)].

## MATERIALS AND METHODS

### Zirconia discs construction:

#### *I. Master model construction:*

A specially designed teflon mold is constructed for this study. It consists of a split disc shape divided into two equal halves. The central hole of the split disc was 10 mm in diameter and 5 mm thickness.

The two halves of the Teflon mold were assembled with a hollow plastic ring, then composite resin layers were incrementally condensed into the mold to fill up the mold and were light polymerized

***II.* Preparation of model for scanning:**

To obtain a three dimensional (3D) image for the model on the computer screen of the Roland CAD-CAM machine, the composite discs formed were used as a template during zirconia discs milling procedure. The composite disc is inserted in the scanner. Then click next to begin scanning. The scanning process of the composite disc was completed after 14 minutes and a digital impression was captured for the composite disc. After creating a new restoration by selecting the "new restoration" icon, by clicking "ok" the cursor jumped automatically to the "Acquire preparation" icon. Designing of the restoration begins by the preparation of the margins of the disc by moving the cursor along the preparation margin and clicking "Next" icon. "Ok" icon was clicked and by using "Design preparation" the restoration design was created. Any unwanted regions or sharp edges of the 3D image of the virtual model were cut away by correcting optical impression tool in the software. Then "next" icon was clicked so that the finished restoration was displayed in the milling situation. The scanned image was stored on the computer hard disk and the machine is ready for milling procedure.

***III. Milling process:***

To start milling procedure using Roland milling machine, the selected zirconia block of the required size was inserted in the spindle of the milling chamber of the Roland system and fastened with the set screw. In this study the disc is milled with an oversize of approximately 20- 25% to accommodate for further shrinkage during sintering process. The type and size of the disk was selected as "CG Zirconia" in the "Manufacturer" window box.DWX-30 5 axis/ auto tool changer was then activated. Then "mill" icon was clicked. The milling process ran fully automated without any interference with the two diamond stones acting together simultaneously in the shaping process. The same process was repeated 28 times to end up with 28 milled discs used in our study. After completion of the milling process the discs were separated &trimmed from the block holder with a diamond cutting abrasive instrument at a very low speed.

***IV. Sintering process of the zirconia discs***:

The milled zirconia discs were then placed inside the ceramic sintering tray, which was filled with sintering beads and then sintered in the HTC furance ( High-temprature Furnace with program control unit) according to manufacturer instructions. As the following: Starting temperature is room temperature and final temperature is 1500° C and time is 8 hours while holding time is 2 hours. The discs were divided into two groups according to sandblasting surface treatment as the following:

Group (I): Including 14 zirconia discs without sandblasting.

Group (II): Including 14 zirconia discs after being subjected to sandblasting.

These groups are subdivided into two subgroups each according to Z-Primer Plus application: Subgroup A: Including 7 zirconia discs without primer application.

Subgroup B: Including 7 zirconia discs with primer application

**Surface treatment of zirconia discs**

***Mechanical treatment by Sandblasting:***

Sandblasting was carried out to fourteen zirconia discs using 110 $\mu$m Aluminum oxide particles ($Al_2O_3$) applied perpendicular to the surface, using an airborne particle-abrasive device Renfert, Germany. The specimens were mounted in a metallic holder at a distance of 10 mm between the surface of the sample and the blasting tip. Air-abrasion was performed for 15 seconds, with 3 bar pressure **(5).**

**Chemical treatment by Z-Prime Plus application:**

It is applied in two layers to each sample. The primer is applied to the surface of the samples using clean disposable micro brush in one direction (from right to left). the application of the second layer and then dried with air syringe for 5 seconds according to manufacturer's instructions.

**Ultrasonic Cleaning:**

Ultrasonic cleaning was done using an ultrasonic cleaner by immersing the zirconia discs in distilled water for 5 minutes to eliminate blasting particles from the sample surfaces and then air dried with oil free compressed air [(6)].

**Composite discs construction:**

A specially designed teflon mold is constructed for this study, similar to that used in construction of zirconia discs with 5 mm diameter and 2.5 mm thickness. The composite used is Bisco AELITE Composite. The composite resin layers were incrementally condensed into the mold to fill up the mold and were light polymerized. Twenty-eight composite discs were constructed according to manufacturer's instructions.

**Cementation of the samples:**

Composite discs were treated with universal bond before cementation**.** The cementation of the composite discs to the zirconia discs is done using dual cured adhesive resin cement (Duo-link Universal cement). Working Time: Minimum 2 minutes at 22˚C (71.6˚F) Setting Time: Maximum 3 minutes, 30 seconds at 37˚C. Pressing the plunger will mix and dispense the DUO- LINK UNIVERSAL.

**Cementation Procedures:** Apply cement to the surfaces of the composite discs then fully seat the zirconia discs and gently remove the excess cement with a brush prior to spot curing. A specially designed cementing device was constructed, this device is composed of metallic base that contains a metallic holder to place the zirconia-composite complex inside it and ensure fixation of the complex. Two fixed vertical arms connected to the base on each side. One horizontal arm fixed to the two vertical arms. A central movable vertical arms, that has a rounded table in its upper end to accommodate for the load and its lower end is 5mm diameter. A load of 3 kilo gram was applied for 12 minutes, resulting in standardized uniform cement film thickness. LED polymerization light for 2-3 seconds per quarter surface (mesio-facial, disto-facial, disto- ligual, mesio-lingual), at a distance of approximately 0-10 mm. after excess cement has been removed, each surface of the restoration may be cured for up to 40 seconds.

**Cyclic loading:**

All specimens are subjected to cyclic loading through cyclic fatigue loading, Mechanical aging via cyclic loading was performed using a programmable logic controlled equipment; the newly developed four stations multimodal ROBOTA chewing simulator integrated with thermo-cyclic protocol operated on servo-motor.

ROBOTA chewing simulator, which has four chambers simulating the vertical and horizontal movements simultaneously in the thermo-dynamic condition. Each of the chambers consists of an upper Jackob's chuckas hardened steel stylus antagonist holder that can be tightened with a screw and the lower part was Teflon housing as sample holder. A weight of 3 kg, which is comparable to 30 N of chewing force, was exerted. The test was repeated 10,000 times of chewing condition [(7)].

**Thermal cycling procedures for laboratory testing of dental restorations:**

In this study the number of cycles used was 500 cycle equivalent to approximately six months. Dwell times were 25 seconds. In each water bath with a lag time 10 seconds. The low- temperature point was 5˚C. The high temperature point was 55˚C [(8)] .

**Shear Bond Strength test:**

These tests were performed using Bluehill Lite Software from Instron(R) Shear test was designed to evaluate the bond strength. All samples were individually and horizontally mounted on a computer controlled materials testing machine (Model 3345; Instron Industrial products, Norwood, USA) with a loadcell of 5 KN and data were recorded using computer software (Bluehill Lite; Instron Instruments). Shearing test was done by compressive mode of load applied at ceramic-resin interface using a mono-bevelled chisel shaped metallic rod attached to the upper movable compartment of testing machine traveling at cross-head speed of 0.5mm/min. The load required to debonding was recorded in Newton.

***Shear bond strength calculation:***

The load at failure was divided bonding area to express the bond strength in Mpa :

$\tau = P / \pi r^2$;

where: $\tau$ = shear bond strength (MPa, P = load at failure(N) $\pi$ = 3.14 and r = radius 0f resin disc (mm).

**Evaluation of mode of failure:**

Failed samples were examined using USB Digital microscope with a built-in camera connected with IBM compatible personal computer using a fixed magnification 45x and the type of failure was classified according to the following types:

1. Adhesive failure: failure between the zirconia and resin cement (at the interface).
2. Cohesive failure: failure within the cement layer.
3. Mixed failure: where area of zirconia is covered by cement and other area with no cement covering the zirconia surface.

## RESULTS

It was found that the highest shear bond strength mean value recorded for (**Sandblast+ Primer)** group (IIB) (10.007 MPa) followed by (**No Sandblast+ Primer)** group (IB) (7.6636MPa) then (**Sandblast + No Primer)** group (IIA) (5.1232 MPa) while the lowest mean value recorded for (**No Sandblast No Primer)** group (IA) (4.1536MPa). The difference between different surface treatment groups was statistically **significant** as revealed with one way ANOVA test (P<0.05). Pair-wise Tukey's post-hoc test showed non-significant (P>0.05) differences between {(**Sandblast+ Primer)** and (**No Sandblast+ Primer**)}, {(**No Sandblast+ Primer)** and (**Sandblast + No Primer**)} and {(**Sandblast + No Primer** and **No Sandblast No Primer**)}.It was noted that during thermo-mechanical aging a total loss of two samples in group (IIA) and only one sample in group (IA) and their results recorded as zero so not indicated in mean value calculation. Table(1)

**Table (1)** *Comparison of shear bond strength (Mean±SD) between all groups as function of surface treatment*

| Variable | | Mean | ± SD | 95% confidence intervals | | Statistics | |
|---|---|---|---|---|---|---|---|
| | | | | Lower | Upper | F | P value |
| Surface treatment | Sandblast+ Primer Group (IIB) | 10.007$^{A}$ | 2.9 | 6.954 | 13.060 | | |
| | No Sandblast+ Primer Group (IB) | 7.6636 $^{AB}$ | 1.6 | 5.970 | 9.358 | 14.48 | <.0001* |
| | Sandblast + No Primer Group (IIA) | 5.1232$^{BC}$ | 0.435 | 4.667 | 5.579 | | |
| | No Sandblast No Primer Group(IA) | 4.1536$^{C}$ | 0.499 | 3.629 | 4.678 | | |

*Different letters indicating significant (P<0.05); significant (P<0.05)ns; non-significant (P>0.05)*

**Effect of sand-blasting on shear bond strength;** Regardless to other variables; totally it was found that sand-blasted groups recorded statistically significant higher bond strength mean values than non-sand-blasted groups as indicated by paired t-test (P 0.05)

**Effect of primer on shear bond strength;** Irrespective of other variables; totally it was found that primed groups recorded statistically significant higher bond strength mean values than non- primed groups as indicated by paired t-test (p 0.05)

**Results of failure mode analysis:** Table (2), Fig.(1)

| | **Failure Mode Percentage** | | |
|---|---|---|---|
| Group | **Adhesive** | **Cohesive** | **Mixed** |
| Sandblasting & primer | — | 100% | |
| Sandblasting & no primer | | 42.9% | 57.1% |
| No Sandblasting & primer | 60% | 40% | |
| No Sandblasting & no primer | 100% | | |

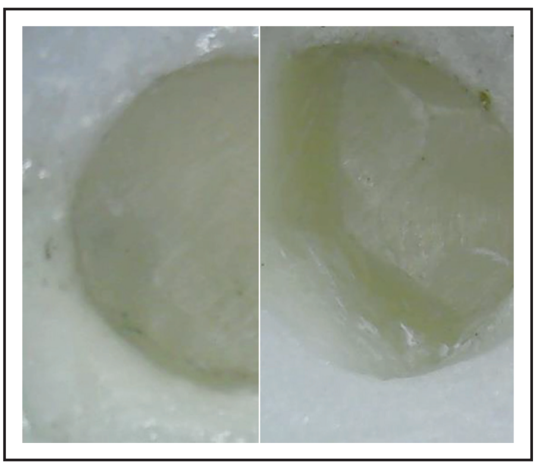

Fig. (1) Cohesive failure of group (IIB)-Mixed failure of group (IIA)

## DISCUSSION

The use of zirconia materials in this study is because, zirconia material differ from other high strength dental ceramics because of their distinct mechanism of stress-induced transformation toughening, which means that the material undergo micro-structural changes when submitted to stress. . The flexural strength of Y-TZP ceramics can reach values from 700 to 1200 MPa. These values exceed the maximum occlusal loads during normal chewing. Y-TZP materials might also exhibit fracture resistance higher than 2000N, which is almost twice the value obtained for alumina-based materials and at least three times the value demonstrated by lithium disilicate-based ceramics [9].

In the present study, for standardization of zirconia discs (10mm diameter and 5mm thickness) Roland CAD-CAM milling machine was used and also for standardization of composite discs a specially constructed Teflon mold of dimensions 5mm diameter and 2.5mm thickness. Because the purpose of the investigation was to analyze the effect of surface treatments on bonding to zirconia-cement interface, so we used in this study composite discs instead of tooth structure, where the micro-structural variations of natural tooth tissues, which could result in ambiguous results, were avoided [10].

Although conventional techniques for cementation with zinc phosphate and resin modified glass ionomer cement can be applied to zirconia surface, adhesive cementation has been recommended as more appropriate to obtain marginal sealing and adequate retention[102]

.However, to achieve a stable bond with adhesive cementation, it is necessary to prepare the ceramic surface with chemical-mechanical or mechanical treatments. Chemical and/or mechanical surface treatment provides a reliable adhesive bonding to resin cements and ceramic on metal-free prosthetic restorations [11,12]. Dual-cured resin luting agent have been recommended for luting ceramic and resin composite restorations to compensate for attenuation of the curing light and to allow complete polymerization of the resin luting agent even at areas not accessible by the curing light [13,14].

The shear bond strength test is one of the most commonly used bond strength tests, because of being fast, easy to perform and also reflecting the clinical situation, and it was used in our study to evaluate the bond strength of resin cement to the ceramic. In the present study the shear force was applied in a direction parallel to the bonding plane [12,15-19].

Thermocycling can simulate the oral cavity environment, and be used as a reference for the artificial aging process. There is a large variation in the number of cycles and in the temperature extremes between studies. This large variation led the International Organization for Standardization (ISO) to make standard protocol for themocycling tests to enable investigators and industry to interpret and compare results. According to this protocol, a thermocycling regimen comprising 500 cycles in water between 5 and 55°C is an appropriate artificial ageing test and thus our study was carried out following the ISO standard [20]. All of the samples in this study were subjected to thermocycling [8].

Cyclic loading test was done for all samples where a weight of 3 kg, which is comparable to 30 N of chewing force, was exerted on all samples. The test was repeated 10,000 times of chewing condition [7]. These cyclic loading cycles simulated occlusal loading during mastication.

In this study, the sample classified into four groups according the type of surface treatment. The samples received no treatment (no sandblasting, no primer showed the least shear bond strength and this may be explained to that unmodified ceramic surface produced a smooth and shallow porous surface, that was unsuitable for bonding (some specimens was lost through cyclic loading and thermocycling). This weak bond strength was reflected in the pattern of failure of this group which showed adhesive failure between zirconia and the resin cement.)

In the group received sandblasting, grit-blasting with alumina was found to give good results with resin cements, Grit-blasting is intended to remove contamination and increase the roughness and hence the specific surface area, thereby decreasing the effective contact angle and increase the wettability of luting material [21].

In a previous study they evaluated the effect of air abrasion $Al_2O_3$ of zirconia. It was found that air abrasion of zirconia promoted better bond performance of tested primers [22]. Also in another study it was concluded that sandblasting modifies the surface of the zirconium-oxide ceramic, creating a rougher more retentive surface, and providing a better mechanical interlocking with the ceramic and cement [23]. And the same results reported in another study where they evaluated the effect of sandblasting of zirconia on bonding to resin cement; the highest mean shear bond strength was recorded for the airborne-particle abrasion group [7].

In a previous study they disagreed with our results, where it has been demonstrated that the presence of carboxylic acid monomer Z-Prime Plus can weaken the connection between this primer methacrylate groups found in self-adhesive resin cements, which could explain the low shear bond strength obtained for the association of Z-Prime Plus, where the Z-Prime Plus showed better results in comparison to the control group only for Panivia F and NX3 [24].

## CONCLUSION

Under the conditions of the present study, the following conclusions could be delivered:

1. The bonding to zirconia is affected by mechanical and chemical surface treatments which are sandblasting and primer application.
2. Under cyclic loading and thermocycling the shear bond strength was highly affected by combination of chemical and mechanical surface treatments.

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