Evaluation of Surface Conditioning of Zirconia and Its Effect on Bonding to Resin-Luting Agent

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ABSTRACT

Zirconia, a recently introduced ceramic exhibits excellent esthetic qualities and demonstrates outstanding flexural strength but its extensive use, requires a reliable bond of Zirconia with resin-luting agent. Resin Zirconia bonding cannot be achieved as the material is resistant to common etching procedures used for other glass containing ceramics. With surface conditioning increased adhesion between Zirconia and resin-luting agent can be successfully achieved.

Aim: Aim of this study was to evaluate surface conditioning of Zirconia and its effect on bonding to resin-luting agent.

Materials and methods: Fifteen blocks of Zirconia (VITA Zirconia) were fabricated in the laboratory according to manufacturer’s instructions and embedded in acrylic resin to get 15 Zirconia samples. Fifteen composite resin cylinders were prepared one for each Zirconia sample. All the 15 Zirconia samples were divided into three groups of five samples each. Group A: Was kept as control with no surface conditioning done. Group B: Surface conditioning was done with 30 \( \mu \) silicon dioxide. Group C: Surface conditioning was done with 110 \( \mu \) aluminum oxide. Composite resin cylinders were cemented on the Zirconia samples using a resin-luting agent (Panavia F). The samples were subjected to universal testing machine to evaluate shear bond strength and the data was statistically analyzed by one-way ANOVA.

Results: Group A showed least shear bond strength. Shear bond strength of group C was greater than group A while group B showed highest shear bond strength.

Conclusion: Surface conditioning results in significant increase bond strength between Zirconia and resin-luting agent. Among the two methods surface conditioning with 30 \( \mu \) silicon dioxide is much better and efficient method.

Keywords: Zirconia, Surface conditioning, Microetcher, Resin-luting agent.

INTRODUCTION

Zirconia, a high strength ceramic has been recently introduced as a core material for complete coverage crowns and bridges. Way back in biblical times Zirconia was used as gemstones. Tremendous multifaceted growth in the field of dental materials and biomaterials has focused Zirconia as one of the most outstanding of all ceramic materials. 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 restorations.\(^1\)

Zirconia restorations can be cemented by conventional methods but use of resin-luting agent enhances retention and provides better marginal seal. Establishing a strong and stable bond of Zirconia with resin-luting agent has always proven to be difficult as the material is acid resistant and does not respond to common etching and silanization procedures used for other glass containing ceramics.\(^2\) Therefore, resin-luting cements could not be used for Zirconia based restorations.

With surface conditioning increased adhesion between Zirconia and resin-luting agent could be achieved.\(^1,2\) What effect the surface conditioning methods would have in enhancing the adhesion of Zirconia with resin-luting agent was to be studied and which would be a better method could be suggested by the present study.

MATERIALS AND METHODS

The study was carried out in the following order:

Preparation of Zirconia Samples

To fabricate 15 Zirconia blocks a metal block of dimensions \( 5 \times 5 \times 2 \) mm was prepared. Elastomeric impression material was used to make an impression of the metal block, which was used to condense Zirconia to form all blocks of specific dimensions. Zirconia powder and liquid (VITA Inceram Zirconia) was mixed according to manufacturer’s instructions, condensed into the mould, allowed to set and then retrieved (Figs 1 and 2). The blocks were then sintered in VITA Zirconia furnace for 60 minutes and allowed to cool (Figs 3 and 4).

Preparation of Composite Cylinders

To prepare composite resin cylinders, plastic tubes of 5 mm height and 3 mm diameter were filled with composite resin (Z-250; 3M ESPE) in increments and each increment light cured for 40 seconds. The cylinders were then retrieved and additionally light cured for 20 seconds. (Elipar excel 2500, intensity 400 mW/sqcm, 3M ESPE).
Surface Conditioning of Zirconia

All the 15 Zirconia samples were then randomly divided into three groups, group A, B and C of five samples each. They were further divided into groups A1, A2, A3, A4, A5; B1, B2, B3, B4, B5 and C1, C2, C3, C4, C5 and labeled on their undersurface respectively (Fig. 5).

**Group A:** All the samples of group A were kept as control with no surface conditioning done on the samples.

**Group B:** All the samples of group B were treated by air borne particle abrasion with 30 μ silicon dioxide (Mahavir minerals) particles applied perpendicular to the surface at 0.28 MPa pressure for 20 seconds at a distance of 10 mm using intraoral air abrasion device (Microetcher, Danville, Inc, USA).

**Group C:** All the samples of group C were treated by air-borne particle abrasion with 110 μ aluminum oxide (Korox, Bego, Germany) particles applied perpendicular to the surface at 0.28 MPa pressure for 20 seconds at a distance of 10 mm using intraoral air abrasion device (Microetcher, Danville, Inc, USA).

All the samples were then placed in ultrasonic cleaner containing distilled water for 10 minutes.

Cementation of Composite Cylinders on Zirconia Blocks

The surface of each composite cylinder was acid etched with 37% phosphoric acid rinsed and dried, then bonding agent was applied and light cured for 10 seconds. Silane coupling agent (Porcelain bond activator, Kurray, Japan) was applied to each Zirconia sample and allowed to dry for 5 minutes. A thin layer of bonding agent was applied over silane coupling agent and light cured for 20 seconds. Dual cure resin cement (Panavia F) was mixed according to manufacturer’s instruction and applied over the center of Zirconia block. Etched and bonded surface of the composite resin cylinder was placed over it and finger pressure was applied. The sample was then light cured in all directions for 40 seconds. After this oxyguard was applied at the

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**Fig. 1:** Mixing of Zirconia liquid, additive and powder

**Fig. 2:** Zirconia mix condensed into the mold

**Fig. 3:** Blocks kept in Zirconia furnace for sintering

**Fig. 4:** Fifteen sintered Zirconia blocks
adhesive interface of Zirconia and resin cement. All the 15 composite cylinders were cemented on the Zirconia blocks in the similar manner (Fig. 7).

**Evaluation of Shear Bond Strength**

All the samples were then placed in a Jig on the universal testing machine (Instron machine-crosshead speed of 0.5 N/min) to evaluate shear bond strength (Fig. 8).

**RESULTS**

The Null Hypothesis, proposed was that there is no significant difference in the shear bond strength between the unconditioned group and the groups treated by surface conditioning methods. The results obtained were statistically analyzed by one-way ANOVA test. Since p < 0.05 therefore Null Hypothesis was rejected and it was concluded that there is significant difference in the shear bond strength between the unconditioned group and the groups treated by surface conditioning methods. In the results group A showed least shear bond strength. Shear bond strength of group C was greater than group A while group B showed highest shear bond strength.

**DISCUSSION**

Zirconia restorations exhibit outstanding esthetic qualities and offer adequate strength to resist fracture loads but their extensive use as an all ceramic dental restorative material

| Mean shear bond strength of all the three groups | Group A (control group) | Group B (silicon dioxide treated) | Group C (aluminum oxide treated) |
| Mean shear bond strength in N/mm² | 7.35 N/mm² | 25.91 N/mm² | 15.84 N/mm² |

**Fig. 5:** Samples divided into groups A, B and C

**Fig. 6:** Surface conditioning with 30 μ silicon dioxide and 110 μ aluminum oxide

**Fig. 7:** Composite cylinders cemented on all the 15 Zirconia samples

**Fig. 8:** Debonded samples
requires a reliable bond between Zirconia and resin-luting agent.\textsuperscript{1} Other ceramics like alumina, glass infiltrated and lithium disilicate ceramics can be bonded to resin cements by acid etching with hydrofluoric acid. Inertness of the material and absence of silica phase in the crystalline structure makes Zirconia resistant to acid etching and therefore resin Zirconia bonding cannot be successfully achieved. Zirconia was subjected to air-borne particle abrasion with 110 μ aluminum oxide or 30 μ silicon dioxide to evaluate which method of surface conditioning brought about better adhesion between Zirconia and resin-luting agent. However, in the control group no surface conditioning was done prior to application of silane coupling agent.

Results have illustrated that shear bond strength values of surface conditioned samples treated by 110 μ aluminum oxide or 30 μ silicon dioxide were higher than the control group. Among the two methods of surface conditioning, the bond strength values of the samples treated by 30 μ silicon dioxide were much higher than those treated by 110 μ aluminum oxide.

Conditioning the surface of Zirconia produces fine rough surface which increased the total surface area and brought about better micromechanical retention. Thus, surface conditioning of Zirconia by air-borne particle abrasion enhances adhesion between composite resin and Zirconia.\textsuperscript{1,2,4} Furthermore, significantly increased bond strength values obtained by surface treatment with 30 μ silicon dioxide can be explained by the fact that air abrasion by silicon oxide leaves behind a layer of silica on the ceramic surface and produces chemical bond between silica containing ceramic surface and resin-luting agent.\textsuperscript{1,3-5,7}

The outcome of this study suggests that relatively recent surface conditioning techniques based on combination of micromechanical and chemical conditioning should be considered for improved adhesion of resin-luting agents to Zirconia-based ceramics. By employing chairside devices for air borne particle abrasion, contamination during transfer of the restoration from the laboratory to the chairside can be avoided. As long as the available conditioning methods are not optimally used, high strength ceramic are expected to continue experiencing failures in their bonding to resin-luting agent.\textsuperscript{2,4}

Taking into consideration the limitations of this study, the effect of thermal cycling and long-term storage on the durability of the resin bond strength to Zirconia could be substantiated by a clinical study.\textsuperscript{2-4,6}

**CONCLUSION**

The following conclusions were drawn from the study:
1. Unconditioned surface of Zirconia had least bond strength with resin-luting agent.
2. Zirconia surface treated with 110 μ aluminum oxide had a lesser bond strength as compared to 30 μ silicon dioxide.
3. Zirconia surface treated with 30 μ silicon dioxide showed highest bond strength with resin luting agent.
4. Zirconia surface treated with both 30 μ silicon dioxide and 110 μ aluminum oxide had a better bond strength as compared to unconditioned surface of Zirconia.

**REFERENCES**


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