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Original Article

Microshear bond strength evaluation of surface pretreated zirconia ceramics bonded to dentin

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ABSTRACT

Objectives: To comparatively assess the micro shear bond strength (MSBS) of dentin bonded surface pre-treated zirconia ceramics. **Materials and Methods:** Zirconia blocks were sectioned into 50 cubical blocks. The blocks were further categorized into five groups (n = 10 each). Group I: No treatment was performed on zirconia samples; Group II: The zirconia samples were sand-blasted; Group III: Group II + etched with 9.8% of hydrofluoric (HF) acid for 60 s; Group IV: The sandblasted zirconia samples were selectively infiltrated with low fusing porcelain; and Group V: Group IV + etched using 9.8% HF acid gel. The zirconia specimens were then bonded to dentin samples, and the samples were tested for MSBS evaluation using universal testing machine. **Results:** The MSBS of all the four experimental groups shows greater value than group I. Among the experimental groups, group V and group IV do not show any statistical significant difference, whereas the mean MSBS of groups IV and V were statistically greater than group III and group II. However, groups I, II, and III do not show any statistical significant difference in mean MSBS values between them. **Conclusion:** Selective infiltration etching of zirconia ceramics provides the highest bond strength with resin cement.

Key words: Hydrofluoric acid etching, sandblasting, selective infiltration etching, Zirconia blocks

INTRODUCTION

Zirconia (ZrO_2) has been developing as a reliable material for fabrication of dental prosthesis thanks due to the tremendous advances in ceramic materials. The crystalline dioxide of zirconium results in ZrO_2, which possesses the strength of metals in addition to being tooth color. By virtue of these properties, ZrO_2 is also known as ceramic steel.[1]

With ZrO_2 ceramics, the conventional techniques of bonding (i.e. mechanical and chemical bonding) on silica-based ceramics are not viable. Acid etching and traditional silanes, which are essential for mechanical bonding,[2] cannot be employed in ZrO_2 microstructure due to lack of silica or any glassy phase.[3] ZrO_2 cannot be dissolved by acids, alkalis, or any dissolving agents by virtue of their chemical inertness.[4] Air abrasion technique, by means of alumina and rotary abrasion method, with diamond burs have been unsuccessfully tried to improve mechanical bonding of ZrO_2 with adhesive cements.[5,6] Selective infiltration etching (SIE), where glassy phase is preamble on the surface, is also being analyzed to improve the bonding ability of ZrO_2.

According to Tsuo *et al*. (2006),[7] air abrasion of ZrO_2 ceramics and application of ZrO_2 primers has

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considerably increased the bond strength of ZrO_2 to the resin cement whereas Qeblawi *et al*. (2010)[8] stated that silica coated ZrO_2 surface after silanation showed improved bond strength compared to air abrasion. Similarly, Aboushelib *et al*. (2007)[9] stated that SIE with low fusing porcelain showed improved bond strength than air abrasion using aluminum oxide. However, there are no studies to comparatively evaluate the various surface pre-treatment techniques on ZrO_2 ceramics.

Hence, present study was undertaken to comparatively assess the microshear bond strength of various surface pre-treatments such as, sandblasting, etching with hydrofluoric (HF) acid, SIE, and combinations of these pre-treatments on ZrO_2 ceramics bonded to dentin using resin cement.

MATERIALS AND METHODS

Zirconia blocks (Lot No. 0905150/2, Amann Girrbach, Germany) were sectioned into 50 cubical blocks of dimensions 8 mm × 2 mm × 2 mm using prosthetic die cutting jigsaw. The blocks were then sintered at 1400°C for 12 h in ZrO_2 sintering furnace. The blocks were then ultrasonically vibrated in deionized water for 5 min and consigned to five equally sized experimental groups (n = 10 each) as follows:

- Group I: (Control) - No treatment was performed for 10 ZrO_2 specimens
- Group II: The specimens were sandblasted using 150 μm grain-sized aluminum oxide particle (Tirupathi Industries, Palanpur, India) for 20 s at 3.8-bar pressure. The tip distance from the surface of the ZrO_2 blocks was maintained at 10 mm. The tip head was positioned at right angle to the ZrO_2 surface
- Group III: The specimens were treated as per group II, and then etched using 9.6% HF acid gel (Azure Research Lab, Kochi, India) for 60 s
- Group IV: The specimens were treated as per group II and then subjected to a heat-induced maturation/SIE (HIM/SIE) method. The bonding surface of the ZrO_2 blocks was covered with a thin film of an infiltration agent. The infiltration agent to control its viscosity and thermal expansion coefficient ($10.1 \times 10 - {}^{6}/°C$) has been composed of a low temperature melting glass loaded with different additives. The infiltration agent mainly consists of silica and oxygen with traces of titanium, alumina, potassium, rubidium, and magnesium. The blocks were heated to 750°C (60°C/min) in an electrical induction furnace (Programat CS, Ivoclar Vivadent, USA), cooled to 650°C for 1 min (60°C/min), reheated to 750°C for 1 min (60°C/min), and finally, cooled to room temperature. The residue of the remaining infiltration agent was then vibrated in an ultrasonic bath for 15 min, followed by washing under demineralized water for 5 min
- Group V: In addition to group IV treatment, the blocks were etched using 9.6% HF acid gel.

Of the five groups, the ZrO_2 blocks of groups I, II, and III were silanated using metal/ZrO_2 primer for 1 min, while the blocks of groups IV and V were silanated using Monobond-S (Batch No. 480842, Ivoclar vivadent, USA).

Fifty dentin blocks were prepared 2 mm × 2 mm × 4 mm in dimensions from caries-free posterior teeth. The prepared dentin samples were then sanitized in an ultrasonic bath for 15 min to render it free from surface debris accumulated during sectioning. These samples were then etched using 37% phosphoric acid and rinsed under running tap water. The samples were then dried and primed using Excite DSC and light-cured for 20 s. The ZrO_2 blocks using resin cement Variolink II (Ivoclar vivadent, USA) were then bonded to these dentin samples and light-cured for 60 s.

Acrylic jigs were prepared using self-cure acrylic and these bonded samples were mounted on the jigs. They were then subjected to testing for microshear bond strength using universal testing machine at a crosshead speed of 1 mm/min. The data obtained

Table 1: Mean (±SD) microshear bond strength of various surface pretreated zirconia blocks bonded to dentin using resin cement

Groups	Surface pretreatment	Mean	SD
1	No treatment	8.88[a]	1.94
2	Sandblasting	10.12[a]	1.97
3	Sandblasting+HF acid etching	11.04[a]	1.28
4	Sandblasting+selective infiltration etching	29.24[b]	1.60
5	Sandblasting+selective infiltration etching+HF acid etching	31.23[b]	1.92

Different alphabetical superscript indicates a significant difference among the groups at 5% interval (P<0.05). Same alphabetical superscript indicates no statistical difference among the groups. SD: Standard deviation, HF: Hydrofluoric, [a,b] value is 0.5

were then tabulated and statistically evaluated using one-way ANOVA followed by Tukey HSD *post-hoc* test.

RESULTS

According to the results obtained [Table 1], Group I (Control) shows micro shear bond strength (MSBS) value (8.88 MPa) less than the experimental groups (II to V). Among the experimental groups tested, no statistical significant difference exists between the mean MSBS of groups II (10.12 MPa) and III (11.04 MPa). Among the mean MSBS of groups IV (29.24MPa) and V (31.23 MPa), there exists no statistically significant difference between them.

The mean MSBS of groups IV and V were statistically greater than groups I, II, and III.

DISCUSSION

Zirconia frameworks are insoluble to strong acids, alkalis, and organic/inorganic dissolving agents by virtue of their chemical inertness.[4] The bond strength of different cements on zirconium oxide ceramic surface prior to various pre-treatments has been evaluated by many authors, whereas the combination of these surface pre-treatment has not been studied at large.[5,6] Hence, the aim of this research was to comparatively evaluate the microshear bond strength of different "surface pre-treated" ZrO_2 ceramics bonded to dentin using resin cement.

Tetragonal ZrO_2 polycrystals (TZP ZrO_2 [Amann Girbac, Germany]) also known as Yttrium-stabilized ZrO_2 is currently the most considered ZrO_2 for ceramic restorations and hence was used in this study.[10,11] Dentin blocks were used in this study as a bonding substrate rather than composite blocks in order to simulate the clinical situation.

In Group I (control), no surface pre-treatment was done on the ZrO_2 samples. The ZrO_2 blocks were coated with metal/ZrO_2 primer and then bonded to dentin blocks using resin cement. The mean micro-shear bond strength of untreated ZrO_2 samples was 8.88 MPa. This baseline value was used to compare the efficacy of other surface pre-treatment methods followed in this study. The methodology and the results obtained were similar to that of Qeblawi *et al*. (2010).[8]

In groups II to V, the ZrO_2 samples were sandblasted using 150 μm aluminum oxide particles for 20 s at 3.8 bar pressure and coated with metal/ZrO_2 primer. This methodology was similar to that of Tsuo *et al*. (2006)[7] In group III, after sandblasting etching with 9.6% of HF acid for 60 s was done to the ZrO_2 samples as suggested by Piascik *et al*. (2011).[3] In group IV and V, the sandblasted ZrO_2 samples were selectively infiltrated with low fusing porcelain. This was done by coating low fusing porcelain on the ZrO_2 surface and subjecting it to HIM at 750°C as recommended by Aboushelib *et al*. (2007)[9] and Aboushelib *et al*. (2008).[12] After the SIE treatment, the ZrO_2 samples of group V was etched using 9.6% HF acid gel similar to group III. All the ZrO_2 ceramics were bonded to dentin using resin cement, and the MSBS evaluation was done by placing the bonded ZrO_2 ceramics in the universal testing machine.

Among the experimental groups, no statistically significant difference exists between groups II and III when compared with the group I (control). For ceramics containing silica particles, the micro-porosities/roughness can be created on the surface for adequate bonding, whereas the surface of ZrO_2 ceramics is free of silica particles; hence, the bond strength of the ZrO_2 ceramics treated with HF acid does not show any significant increase. The mean MSBS of group III obtained in this study were similar with the studies done by Casucci *et al*. (2011)[13] and Chaiyabutr *et al*. (2008).[14] However, when compared to Groups I (control), II and III, groups IV and V showed statistically significant increase in bond strength values. The mean shear bond strength value obtained in group IV was similar to the study done by Aboushelib *et al*. (2007).[9]

A significant amount of silica in the bonding surface is essential for adequate ceramic bonding which are sustained by chemical bonds.[2] Resin cement and ceramic are chemically adhered by means of silanes, which are bifunctional compounds that encourage chemical bonding between dissimilar materials. Organo-silanes copolymerize with the organic matrix of resin cement by means of a degradable functional group create the chemical bonds necessary for the successful bonding of resin cement to dental ceramics. The surface energy and the wettability of ceramic surfaces are also increased by the silanes which ensure better mechanical and chemical bonding.[15]

To achieve this, SIE was examined to produce a retentive surface architecture on the ZrO_2 ceramics. The main difference between SIE and other common surface roughening methods like airborne particle abrasion is three-fold: Primarily, without any applied external mechanical stresses it is self-introduced by the

material, secondarily, it happens on the ultra-structural grain level without any structural defects or loss of material, and finally, it generates three-dimensional retentive features where the adhesive resin can infiltrate. In this method, the low fusing glass particles which were coated over the bonding surface and fired at 750°C, infiltrates into the grain boundaries of the ZrO_2 surface and forms a glassy mesh which is responsible for increasing the bond strength of the ZrO_2 ceramics significantly.[9]

The marginal increase in the mean MSBS value of group V (31.23 MPa) was not statistically significant when compared to the Group IV. This could be because the infiltration of the glass phase into the ZrO_2 ceramics at the grain boundaries by SIE was limited to only 8–10 μm, which may not be sufficient for the action of HF acid etching to improve the bond strength significantly.

To summarize this study, the MSBS of all the experimental groups is in the following order: Group V ≥ Group IV > Group III ≥ Group II ≥ Group I.

CONCLUSION

Selective infiltration etching of ZrO_2 ceramics provides the highest bond strength with resin cement. Further HF acid etching on SIE-treated ZrO_2 did not improve the bond strength significantly. Conventional methods such as sandblasting and HF acid etching alone did not achieve clinically satisfactory bond strength values.

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