INFLUENCE OF SURFACE TREATMENT PROTOCOLS ON SHEAR BOND STRENGTH OF VENEERING COMPOSITE TO ZIRCONIA

Hassan Salim Skainhe*; Mohammad M. Rayyan** and Mostafa Fakhri Khalil***

ABSTRACT

Statement of problem: the formation of reliable bond to zirconia-based materials has proven to be difficult which is the major limitation against fabricating adhesive zirconia restorations. Materials and Methods: a total of 35 fully sintered zirconia 5 mm diameter discs (Z1) (Ceramill zi, Amann Girrbach AG) were constructed using MAD MAM technique (Ceramill Multi-x, Amann Girrbach AG). Another 35 composite resin discs (Filtek P60, 3M, U.S.A) (C1) of 5 mm diameter were constructed. The prepared zirconia discs (Z1) were divided into five groups- according to surface treatment applied- each of 7 specimens. (Gr I; control, Gr II; 50 μm Al₂O₃ Air abrasion, Gr III; 100 μm Al₂O₃ Air abrasion, Gr IV; 9% hydrofluoric acid etched, Gr V; 50 μm Al₂O₃ Air abrasion then 9% hydrofluoric acid etched). C1 discs were bonded to the Z1 discs using Resin cement (Multilink, Ivoclar Vivadent Schaan, Liechtenstein) under 500 gm. fixed load. Results: shear bond strengths of the luting cement to the treated zirconia surface were in Gr I:(16 MPa), Gr II:(16 MPa), Gr III:(6.59 MPa ), Gr IV:(11.46 MPa) and Gr V: (5.38 MPa). Conclusions: the air borne particle abrasion surface-treatment applied to the zirconia surface and the non-treated group had approximately similar results, while hydrofluoric acid decreased the bond strength with resin cement.

INTRODUCTION

Ceramics became the winning horse for esthetic dentistry. With the excessive demand on beautiful smiles, the popularity of all-ceramic restorations has increased in recent years. All-ceramic systems must meet the biomechanical and longevity requirements associated with metal ceramic restorations, while providing enhanced esthetics. Zirconia has been recently introduced in prosthetic dentistry due to its excellent mechanical properties, biocompatibility, low degree of bacterial adhesion and acceptable optical properties. The establishment of reliable and durable chemical bond between dental ceramics and composite resin is of great importance. However, formation of reliable bond to zirconia-based materials has proven to be difficult which is

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the major limitation against fabricating adhesive zirconia restorations. This is related to glass-free composition structure characterizing zirconia as acid-resistant material, also it was shown to be minimally affected by conventional roughening techniques. (5) So achieving strong and reliable bond to all-ceramic restorations is a pre-requisite for long term clinical success. Recent researches regarding bonding to zirconia ceramics focused on different approaches. Chemical bonding to zirconia by using a phosphate ester monomer, 10-methacryloyloxydeceyl dihydrogenphosphate (MDP) in combination with airborne particle abrasion showed satisfactory results. (6)

Blatz MB, et al, used MDP-containing bonding agent and found a significantly higher bond strengths before and after long-term storage and thermocycling. (7)

Airborne-particle abrasion with Al2O3 abrasive particles, in combination with the modified phosphate monomer, has been identified as a key factor in achieving a stable durable bond for alumina and zirconia-based ceramics, by Phark JH, et al, in 2009. (8)

Also it has been shown earlier in 2006 by Atsu SS, et al, that silica coating followed by silanization can be used to improve the bond strength for silica-based, glass-infiltrated alumina and zirconium ceramics. (9)

Yun JY, et al, subsequently used alumina oxide-particle abrasion on ceramic surfaces prior to acid etching and found substantial increase in the surface area and enhance the potential for micromechanical retention. (10)

Few studies have evaluated the effect of different surface treatments on the bond strength of zirconium oxide to composite resin as an important factor for repair. Can effective bond to zirconia be achieved? A question worth answering.

**MATERIALS AND METHOD**

A total of 35 fully sintered zirconia discs (ceramill zi, Amann Girrbach AG, Austria) were constructed having the diameter of 8 mm. and thickness of 5 mm using Manually Aided Design and Manufacturing (MAD MAM) technique (Ceramill Multi-x, Amann Girrbach AG, Austria). A metal disc (M1) replica (Remanium GM 380 CoCr, Dentaurum GmbH, Germany) was constructed having the same dimension of the zirconia discs and placed onto the mounting plate of the milling machine. The copying arm of the machine traced the metal pattern and zirconia discs (Z1) were milled with a size 20% greater than the metal pattern to compensate for the sintering shrinkage. After sintering of the discs (Z1) they were measured for standardization. To be able to construct perfectly identical composite discs; 35 Plexiglas rectangles (Plexiglas Egypt, Heliopolis, Egypt) having the dimension of 2 x 5 cm and 5 mm thickness were fabricated. A 5 mm hole was drilled in the center with two linear indentations were made on each side of the hole for easy cut and assembly. An Aluminum housing was fabricated to house the rectangle after being cut to secure its movement. Premasured composite resin rod (Filtek P60, 3M center Building, St. Paul, MN, U.S.A) was applied inside the hole using cement spatula and packed tightly. A second glass sheet was placed on the face of composite to flatten the surface of composite, then cured for 40 seconds each, from top and 2 sides for a total of 120 seconds. Each composite disc was cured for 5 minutes before the bonding procedure. Then the covering slide was removed and the Plexiglas was removed from the Aluminum housing and broken into two pieces for easily removal of composite resin discs (CI) (Fig.1).

The prepared zirconia discs (Z1) were divided into five groups each of 7 specimens (n=7).

Each group underwent mechanical and chemical surface treatment before bonding to the composite resin according to table I.
C1 discs were bonded to the Z1 discs using Resin cement (Multilink automix, Ivoclar Vivadent Schaan, Liechtenstein). Primer A and Primer B were mixed in a 1:1 ratio and applied as a thin layer on the surface of C1. A thin layer of Zirconia primer was applied on the treated surface of Z1 and left for 180 sec and then dispersed using a strong stream of air. Resin cement was mixed and applied directly onto the surface of Z1. C1 was seated on the resin cement under a fixed load of 500 g perpendicular to the surface of C1 using a custom made device. Excess material was immediately removed using a brush (Microbrush International, 1376 Cheyenne Ave. Grafton, United States). The bonded specimen was light polymerized for 40 sec from four sides (Fig. 2 & 3). Load was removed from the surface of C1 and the 35 bonded Zirconia and Composite cylinders (S1) were numbered and stored in distilled water at room temperature for one week (Fig. 4).

S1 were then thermocycled between 5°C and 55°C water bath with 1 minute dwell time for 1000 cycles. Samples were collected and dried using woven gauze pieces. At time for shear bond strength testing, S1 was mounted, using acrylic resin, in a metallic ring and then secured to the universal testing machine (Applied Test Systems (ATS) Inc., U.S.A). A uni-bevel chisel-shaped indenter was used to direct the shearing force to the zirconia composite interface at a crosshead speed of 1 mm/ min until fracture occurred. Data were collected and grouped for statistical analysis using SPSS package version 15 (IBM Corporation, New Orchard, N, United States).

Table (I) Surface treatment protocols applied to ceramic discs

<table>
<thead>
<tr>
<th>Group</th>
<th>Surface treatment used</th>
<th>No</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group I</td>
<td>Control, received no treatment</td>
<td>7</td>
</tr>
<tr>
<td>Group II</td>
<td>Air abrasion applied using 50 μm Al₂O₃ at a pressure of 1.5 bars and at a 2 cm distance perpendicular to the surface, followed by ultrasonic cleaning in distilled water for 10 minutes.</td>
<td>7</td>
</tr>
<tr>
<td>Group III</td>
<td>Air abrasion applied using 100 μm Al₂O₃ at a pressure of 1.5 bars and at a 2 cm distance perpendicular to the surface, followed by ultrasonic cleaning in distilled water for 10 minutes.</td>
<td>7</td>
</tr>
<tr>
<td>Group IV</td>
<td>Acid etched with 9% hydrofluoric acid for 3 minutes then washed with distilled water and air-dried.</td>
<td>7</td>
</tr>
<tr>
<td>Group V</td>
<td>Air abrasion applied using 50 μm Al₂O₃ at a pressure of 1.5 bars and at a 2 cm distance perpendicular to the surface, followed by ultrasonic cleaning in distilled water for 10 minutes. Then Z1 were etched with 9% hydrofluoric acid for 3 minutes then washed with distilled water and air-dried.</td>
<td>7</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>35</td>
</tr>
</tbody>
</table>
RESULTS

Results were summarized using descriptive statistics: mean, standard deviation, median, minimal and maximum values for quantitative variables.

Table 2 and figure 5 show shear bond strength among the studied groups with the highest mean values among groups are in groups I and II followed by group IV and the lowest values were found in group III and V. All measured in MPa unit.

Statistical differences between groups were tested using

1- Kruskal-Wallis test to compare between more than 2 groups.

2- Mann Whitney test to compare between each 2 groups

P-values less than or equal to 0.05 were considered statistically significant.

TABLE (2) Mean, standard deviation, median, minimal and maximum values among studied groups

<table>
<thead>
<tr>
<th>Groups</th>
<th>No.</th>
<th>Mean</th>
<th>St. Deviation</th>
<th>Median</th>
<th>Minimum</th>
<th>Maximum</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>7</td>
<td>16.63</td>
<td>9.44</td>
<td>12.25</td>
<td>8.28</td>
<td>30.20</td>
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<tr>
<td>II</td>
<td>7</td>
<td>16.61</td>
<td>9.66</td>
<td>12.91</td>
<td>6.96</td>
<td>32</td>
</tr>
<tr>
<td>III</td>
<td>7</td>
<td>6.59</td>
<td>4.39</td>
<td>4.96</td>
<td>1.56</td>
<td>12.54</td>
</tr>
<tr>
<td>IV</td>
<td>7</td>
<td>11.46</td>
<td>7.88</td>
<td>11.90</td>
<td>4.38</td>
<td>27.17</td>
</tr>
<tr>
<td>V</td>
<td>7</td>
<td>5.38</td>
<td>3.66</td>
<td>5.23</td>
<td>0.56</td>
<td>10.77</td>
</tr>
</tbody>
</table>

P-value = 0.015 using Kruskal Wallis Test
Table 3 shows that by doing multiple comparisons between each 2 studied groups using Mann-Whitney test the following was found:

1- Group I was statistically higher than group III (p value = 0.048) and group V (p value = 0.006) while no statistical significant difference exists between group I and group II or group IV.

2- Group II was statistically higher than group III (p value= 0.025) and V (p value= 0.018) while no statistical significant difference exist between group II and group IV.

3- No statistical significant difference exists between groups III, IV and V.

**DISCUSSION**

Assuming a minimal bond strength requirement of approximately 10–13 MPa for acceptable clinical bonding. (1,11)

The results of this study revealed the mean shear bond strengths of the luting cement to the treated zirconia surface were in Gr I control (16 MPa), Gr II 50µ Al₂O₃ (16 MPa), Gr III 50µ Al₂O₃, Hydrofluoric acid (6.59 MPa), Gr IV 100µ Al₂O₃ (11.46 MPa),and Gr V Hydrofluoric acid(5.38 MPa).

Our results are in agreement with kulunks, et al, (12) who reported that the highest bond strengths were obtained by air abrasion with 30-50 µm. and with Ramez Shahin, et al, (13) in which they had a significant higher retention strength for the surfaces treated with air abrasion 50µ and luted with resin cement resin. Also this study is in accordance with Jeong-yeon Yun, et al, (14) who used a combined surface treatment for the zirconia surface (air abrasion alone, and air abrasion with metal primer) and had a shear bond strength for the surface treated with air abrasion alone is 13.8 MPa and for the surface treated with air abrasion and metal primer is 17.1 MPa. Cavalcanti AN et al (15) also found that Air abrasion with Al₂O₃ particles and the application of metal primers increased bond strength to zirconia surfaces.

However, airborne particle abrasion has the possibility to create subcritical microcracks and phase transformation within the zirconia surface, consequently causing unfavorable changes of superior mechanical properties of zirconia ceramics (Karakoca & Yılmaz, 2009 (16); Ayad, et al., 2008 (17)).

This may be the cause of low mean bond strength for groups treated with 100µ Al₂O₃ air abrasion, but Jin-Ho Phark, et al, (8) in previous study found that Airborne-particle abrasion of the machined zirconia, Mean Shear bond strength ranged from 10.1 to 20.0 MPa after 3 days regardless of the abrasive particle size (50 or 110 µm). However our finding is not in agreement with other study by Ji-eun Moon et al, (11) in spite that they used the same type of zirconia surface treatment and the same type of resin cement. Their shear bond strength was...
4.17 MPa, this difference in shear bond strength may be due to the way they used the Air-borne particle abrasion which is circular movements instead of perpendicular as in this study and the pressure used while treating the zirconia surface which is 3 bars. No axial fixed load was applied during setting of resin cement.

Also our finding is not in agreement with other study by Carlo Monaco, et al, (18) who used different pressure during air abrasion, which was 4 bars, and different resin cement. Their mean Shear bond strength was 6 MPa. For the groups treated with hydrofluoric acid in this study the mean shear bond strength were low in comparison with the groups treated with air abrasion and the control group.(Gr III 50µ Al₂O₃, Hydrofluoric acid 6.59 MPa and Gr V Hydrofluoric acid 5.38 MPa ).

In comparison to the present study, Ozcan and Vallittu (19) found mean shear bond strength values for the zirconia treated with hydrofluoric acid (8.1 MPa), which was significantly lower than the mean values for the groups blasted with aluminum oxide particles (16.5 MPa) and the group treated with the Rocatec system (17.4 MPa). Dérand P, et al, (20) examined the surface treatments for a zirconia ceramic and also showed that HF etching produced the lowest bond strengths.

Casucci A, et al, (21) in a previous study used 9.5% HF acid etching for the zirconia surface. Scanning electron microscope (SEM) reveals relatively smooth surfaces resulted by the application HF etching.

CONCLUSIONS

Within the limitations of the study, it can be assumed:

1. The air borne particle abrasion surface-treatment applied to the zirconia surface and the non-treated group had approximately similar results, which means that the resin cement and primers played a major role in bond strength.

2. The surface treatment of zirconia surface with hydrofluoric acid decreases the bond strength with resin cement.

Further studies are needed to test new surface treatment protocols to increase bond strength with zirconia, so that one can understand more the behavior and reaction of zirconia under stressful oral environment. For better designing and construction of longer serving esthetic restorations.

REFERENCES


