Effect of silanization on shear bond strength between high translucency zirconia and resin cement - An in-vitro study

Dr. Tanya
Final Year Post Graduate Student, Yenepoya Dental College, Prosthodontics, Mangalore, Karnataka, India
Email: tanyatulika9@gmail.com

Dr. Savitha Dandekeri
Professor, Yenepoya Dental College, Prosthodontics, Mangalore, Karnataka, India
Email: savitadandekeri3117@gmail.com

Dr. Sanath Shetty
Professor and HOD, Yenepoya Dental College, Prosthodontics, Mangalore, Karnataka, India
Email: drshetty@rediffmail.com

Dr. Zahid Mohammed
Reader, Yenepoya Dental College, Prosthodontics, Mangalore, Karnataka, India
Email: dr.zahid02@gmail.com

Dr. Fahad Mohammed
Reader, Yenepoya Dental College, Prosthodontics, Mangalore, Karnataka, India
Email: fahad11ma@gmail.com

Dr. Mallikarjuna Ragher
Reader, Yenepoya Dental College, Prosthodontics, Mangalore, Karnataka, India
Email: drmallis@gmail.com

Dr. Shreshta Hegde
Second Year Post Graduate Student, Yenepoya Dental College, Prosthodontics, Mangalore, Karnataka, India
Email: hegdeshreshta@gmail.com

Abstract—Aim: This in-viro study aimed to evaluate and compare the shear bond strength of resin cement with high translucency zirconia with silane coupling agents and bonding agent separately (Group 3), bonding agent containing silane (Group 2) and bonding agent which does not have silane (Group 1). Materials and Methods: The study was...
conducted by using the high translucency zirconia blanks along with the CAD-CAM equipments. After sintering the specimens according to manufacturer’s instructions surface modifications, bonding procedures and evaluation of bond strength was performed. Bond strength of the samples was tested using universal testing machine. The results were analyzed statistically using descriptive analysis. Results: The mean shear bond strength obtained for group 1, group 2 and group 3 was 17.97, 22.01, 28.87MPA respectively. The SBS of group 3 was significantly higher than the other two groups as the surface of zirconia was treated with both Silane and Silane-containing universal adhesive. Conclusion: The null hypothesis was rejected. Within the limitations of the study, it was concluded that the shear bond strength between resin cement and high translucency zirconia was highest when treated with bonding agent (universal adhesive) and silane coupling agent separately after air particle abrasion.

**Keywords**---Silanization, Universal Adhesives, High Translucency Zirconia, Shear Bond Strength, Air Particle Abrasion.

**Introduction**

Digitization in the field of dentistry via Computer-aided design and computer-aided manufacturing (CAD-CAM) technology has progressed considerably wherein durable dental materials are transformed into restorations that match the appearance of natural dentition while at the same time lowering the time of fabrication and preventing technician-related errors.\(^1,2\) Because of its superior aesthetic appearance and metal-free structure, all ceramic zirconia restorations have a great capacity to replicate the appearance of natural teeth among the many varieties of Fixed Partial Denture Restorations.\(^3-6\)

The longevity and clinical efficiency of a restoration is determined by the adhesive bonding of dental restorative materials to adhesive cement.\(^5,7\) However, one difficult challenge that a prosthodontist poses, is getting adequate adhesion between abutment teeth and restorative material. The microstructural characteristics of Yttria-Stabilized Tetragonal Zirconia Polycrystalline(Y-TZP) as well as its chemical and biological inertness, present intrinsic barriers to resin-mediated bonding. Poor adhesive bonding has long been linked to a number of mechanical and biological difficulties, in literature.\(^8-15\)

A plethora of methods have been proposed for surface conditioning (SC) that result in a stronger bond between the resin cement and the restorative component. These methods make changes onto the repair surface thus, increasing its porosity to promote chemical and micromechanical retention.\(^16,17\) Sandblasting the surface with alumina oxide particles or Tribochemical silica coating, are the most commonly used surface treatment procedures to improve the bond strength between zirconia substrate and resin-based cements.\(^5-7, 18-21\) APA (air particle abrasion) improves the surface energy and wettability of zirconia surfaces. Furthermore, the roughened micron-scale surface created by the resin cement can improve bonding area and construct a micromechanical
interlocking structure. Sandblasting, on the other hand, may cause the surface or lower-layer crystals to shift from the tetragonal to the monoclinic phase, lowering the materials’ mechanical properties. As a result, some manufacturers advocate sandblasting the zirconia surface with 50-micron alumina particles to reduce crystal phase transition.\textsuperscript{18-21}

The wettability of zirconia ceramic surfaces can be improved by adding a silane-coupling agent. Typically, a single liquid kind of commercial silane-coupling agent, an aqueous solution of 3-methacryloxypropyl trimethoxysilane and acidic organic monomer, is used.\textsuperscript{22-24} The silane molecule contains an organic functional group as well as three hydrolyzable methoxyl groups. Silane should be hydrolyzed in an acidic ethanol–water solution to speed up the production of siloxane. Finally, hydrolyzed silane can be chemically or physically adsorbed on the porcelain’s surface. The three methacrylate groups can copolymerize with the unreacted C=C of the resin cement monomer using the light-curing process.\textsuperscript{25-28}

Various adhesives have been developed to improve the bonding strength of zirconia ceramic. Universal adhesives can be used instead of silane primers to produce a chemically based binding to Zirconia ceramics.\textsuperscript{29} The bonding and durability is improved by use of adhesives containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP). The multiple advantages that MDP-based adhesives provide is, they are simple to use and apply, as well as cost-effective and do not require any special equipment. Hence they are good adhesion enhancers.\textsuperscript{30}

The use of a silane coupling agent in universal adhesives may help to improve adhesion between resin cement and Zirconia Ceramics.\textsuperscript{31-45} The efficiency of the integrated silane, on the other hand, is currently being investigated. According to certain makers of new universal adhesives, a separate silane priming step is not required for successful bonding.\textsuperscript{46,47} Regardless of whether silane-containing or silane-free universal adhesives are used, certain studies have demonstrated a positive effect on bond strength after inclusion of a separate silane step during bonding procedure.\textsuperscript{48}

There is no consensus on the best Surface Conditioning approach. There is insufficient information on the influence of silanization as an additional step on the shear bond strength (SBS) between resin cement and zirconia, to the best of the authors’ knowledge.\textsuperscript{49,50} As a result, the goal of this research was to see how silanization affected the SBS between resin cement and high translucency-zirconia. This study evaluated the Shear Bond Strength (SBS) of Resin Cement with High Translucency Zirconia using different Surface Conditioning (SC) approaches. The null hypothesis was that, adding a silane coupling agent as an extra step would have no effect on the SBS between the high translucency zirconia and the resin cement.

**Materials and Methods**

A total of 42 Zirconia specimens were CAD milled from commercially available high translucency zirconia blank (AmannGirbach Ceramill® CAD/CAM Material –
Zolid HT+ 14mm. Zirconia blocks of 10x6x2mm dimension were designed using Ceramill mind software and nested onto 14mm Ceramill zirconia blanks. (Figure 1). Dry milling of this block was done using a milling machine (AmannGirbach Ceramill® motion2). The collected zirconia blocks were dedusted and connectors were retained on one side of each block to improve the retention of the zirconia block in acrylic resin. (Figure 1a-d).

The samples were then placed on to the zirconia holding tray and kept inside the sintering furnace (AmannGirbach Ceramill® therm). A sintering cycle of 12 hours was used in total, with an average rise in temperature of 8°C/min and peak temperature of 1450°C and holding time of 2 hours. Same process was repeated to achieve 42 zirconia blocks in this group. (Figure 2a, 2b).
A customized L Shaped metallic mold of dimension 27 mm x 10 mm x 10 mm was fabricated to make acrylic resin blocks. This metallic mold was filled with self-cure acrylic resin and the zirconia samples were then embedded in the centre of the acrylic resin. Finishing and polishing of the acrylic blocks were done using finishing and polishing burs and emery paper. The samples were sandblasted with 110-µm aluminum oxide particles at 0.25 MPa pressure and placed in an ultrasonic bath with deionized water for 5 minutes. A standardized and customized metallic mold composed of cylindrical split metallic halves of 4 mm height with 5 mm base diameter and upper diameter was used for filling the resin cement. These split metallic compartments were stabilized together by means of two external screws to facilitate the insertion and removal from the sample. (Figure 3a-3f)
The samples, divided into three groups of 14 samples each underwent surface conditioning treatments as follows:

**For group 1:**
- **Luting Procedure:** Silane free universal adhesive (Dentsply Prime and Bond Universal™, Dentsply, Caulk, Milford, DE USA)

**Sample Size:** 14

Prime and Bond Universal adhesive applied in one coat with microbrush to the bonding surface and kept slightly agitated for 20 seconds and gently air-dried for 5 seconds. Light cured for 10 seconds in Light cure unit (GC Labolight LVII, Europe)

**For group 2:**
- **Luting Procedure:** Silane-containing universal adhesive (3M™ Scotchbond™ Universal Adhesive, 3M Oral Care, St. Paul, MN, USA)

**Sample Size:** 14

One coat of Scotchbond Universal was applied over the bonding surface with the using a micro brush, followed by air drying for 5 seconds and Light curing for additional 10 seconds in Light cure unit (GC Labolight LVII, Europe).

**For group 3:**
- **Luting Procedure:** Silane (3M™ ESPE™ SIL 68300, 3M ESPE, Seefeld, Germany) And Silane-containing universal adhesive (3M™ Scotchbond™ Universal Adhesive, 3M Oral Care, St. Paul, MN, USA)

**Sample Size:** 14

Passive application of 3M™ ESPE™ SIL on the bonding surface was done with the help of micro brush. After waiting for 60 seconds, compressed air was used for 5 seconds and the excess silane was gently removed. This was followed by, application of one coat of Scotchbond Universal with a micro brush, for 20 seconds and then gently air drying for 5 seconds. Afterwards, Light curing of the sample was done in Light cure unit (GC Labolight LVII, Europe).

For the bonding experiments, resin cement (RelyX U200) cylinders were built carefully on the surface of each zirconia slab by positioning and stabilizing the customized split metallic mould, and then filling it with Resin cement (3M™ RelyX™ U200 Self-Adhesive Resin Cement Refill, 3M Oral Care, St. Paul, MN, USA). Finally, the veneered specimen was polymerized using the light cure unit (GC Labolight LVII, Europe) for 180 seconds and placed in distilled water for 24 hours at 37°C. Next, the samples were mounted on the universal testing machine (TSI- Tecsol – Chennai; India). The shear bond strength was measured by using a loading head at a speed of 0.5mm/min. Shear load was applied till the resin cement debonded. (Figure 4a-i)
Results

All 42 samples were secured to the holding device of universal testing machine (TSI-Tecsol; India) to perform shear bond tests under static condition. The samples were submitted to shear load with a crosshead speed of 0.5 mm/min. The load required to de-bond each specimen were recorded in kilogram force. This load was divided by the surface area of the layered composite block to get the shear bond strength which was in the unit of
kgf/mm², it was then converted into MPa and mean values of each group were determined. One way ANOVA, multiple comparisons, and t-test were used for statistical analysis. Table 1 shows the mean values of the shear bond strength recorded for each group.

Table 1:

<table>
<thead>
<tr>
<th>SBS (MPA)</th>
<th>Group 1</th>
<th>Group 2</th>
<th>Group 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.47</td>
<td>20.71</td>
<td>27.55</td>
</tr>
<tr>
<td>2</td>
<td>18.36</td>
<td>23.17</td>
<td>29.24</td>
</tr>
<tr>
<td>3</td>
<td>19.83</td>
<td>22.64</td>
<td>28.21</td>
</tr>
<tr>
<td>4</td>
<td>16.02</td>
<td>21.46</td>
<td>27.4</td>
</tr>
<tr>
<td>5</td>
<td>17.9</td>
<td>23.4</td>
<td>28.7</td>
</tr>
<tr>
<td>6</td>
<td>17.49</td>
<td>20.17</td>
<td>29.6</td>
</tr>
<tr>
<td>7</td>
<td>16.5</td>
<td>22.8</td>
<td>30</td>
</tr>
<tr>
<td>8</td>
<td>18.5</td>
<td>20.18</td>
<td>30.21</td>
</tr>
<tr>
<td>9</td>
<td>17.49</td>
<td>21.45</td>
<td>29.9</td>
</tr>
<tr>
<td>10</td>
<td>19</td>
<td>22.25</td>
<td>28.7</td>
</tr>
<tr>
<td>11</td>
<td>17.12</td>
<td>20.5</td>
<td>28.25</td>
</tr>
<tr>
<td>12</td>
<td>17.53</td>
<td>22.42</td>
<td>29.14</td>
</tr>
<tr>
<td>13</td>
<td>18.63</td>
<td>24.44</td>
<td>30.2</td>
</tr>
<tr>
<td>14</td>
<td>18.74</td>
<td>22.6</td>
<td>27.15</td>
</tr>
<tr>
<td>Mean</td>
<td>17.97</td>
<td>22.01</td>
<td>28.87</td>
</tr>
</tbody>
</table>

The mean shear bond strength obtained for group 1, group 2 and group 3 was 17.97, 22.01, 28.87MPA respectively. The SBS of group 3 was significantly higher than the other two groups as the surface of zirconia was treated with both Silane and Silane-containing universal adhesive. For group 2, where the surface treatment was done with Silane-containing universal adhesive, the SBS was also found to be significantly higher than the group 1 in which the surface treatment was done with Silane-free universal adhesive (22.01MPA and 17.97MPA). The intergroup comparison of Shear Bond Strength has been listed in Table 2 and graph 1.

Table 2

<table>
<thead>
<tr>
<th>GROUPS</th>
<th>Mean</th>
<th>Std Deviation</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>GROUP 1</td>
<td>17.97</td>
<td>1.02</td>
<td>331.48</td>
<td>0.00*</td>
</tr>
<tr>
<td>GROUP 2</td>
<td>22.01</td>
<td>1.30</td>
<td></td>
<td></td>
</tr>
<tr>
<td>GROUP 3</td>
<td>28.87</td>
<td>1.05</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

F=ANOVA

| statistically significant |

TABLE 2: Shows the descriptive statistics for all the 3 groups derived using the students t test. The p value for the groups was statistically significant; p>0.05.
Graph 1:
Intergroup Comparision Of Shear Bond Strength

![Graph showing mean values (MPA) of shear bond strength of resin cement to zirconia, following various surface treatments.](image)

Graph 1: Bar graph shows the mean values (MPA) of shear bond strength of resin cement to zirconia, following various surface treatments.

One way ANOVA was used to compare the shear bond strength of zirconia to composite resin with different surface treatments. Difference in mean bond strength among the groups was found to be statistically significant.

Discussion

In this study, the effects of silane coupling agent and two different universal adhesives on the shear bond strength between the high translucency zirconia and resin cement was evaluated. The three surface conditioning agents used in this study were the **Silane-free universal adhesive, Silane containing universal adhesive and Silane along with Silane-containing universal adhesive.** The first group involved the application of a **Silane-free universal adhesive** which is a MDP Based primer between the high translucency zirconia and resin cement. The second group used **Silane containing universal adhesive** and the third group involved the application of **Silane coupling agent along with Silane-containing universal adhesive.**

There was a statistically significant effect of Surface Conditioning (SC) method on the mean Shear Bond Strength (SBS) values as per the results attained through two-way ANOVA. Therefore, both null hypotheses were rejected. As per manufacturers’ recommendation, high translucency zirconia was sandblasted with 50 µm Al2O3 particles under pressure conditions in the range of 1 -2 bar. Increasing the area of bonding surface, eliminating the surface contamination, enhancing the micromechanical bond, and removing the smear layer resulting from the milling process are some of the major benefits of sandblasting. However,
sandblasting if done with particles exceeding appropriate size or pressure can cause surface deterioration and thus reducing the bond strength.

The **Silane-free universal adhesive of group 1** used in this study is a primer containing 10-MDP and carboxylic acid. Mechanism of action employs the development of a chemical bond (Zr-O-P) between zirconia ceramic and **Silane-free universal adhesive**. The phosphate group reacts with zirconium, forming zirconium phosphate, with each phosphate group bound to three zirconium atoms (tridentate bridging mode) or to one zirconia atom (tridentate chelating mode), resulting in a thermally and hydrolytically stable interface. The secondary ingredient contained in the primer, such as the carboxylic acid monomer, favour the bonding process. The results obtained with this primer are almost similar with the findings in the study of Lopes et al., wherein Silane-free universal adhesive had significantly lower bond strength than silane containing universal adhesive.

However, according to a study done by Pereira et al., there was no difference in bond strength for the groups where the zirconia was sandblasted with aluminum oxide followed by surface treatment with universal adhesive with or without silane. The method of testing used to assess the bond strength and the sample preparation are the primary cause for discrepancies in the results.

The present study also evaluated the application of universal bonding agent (SBU; Group 2) that has silane, 10-MDP monomer, HEMA and Bis-GMA in its composition. Metal oxides such as Zirconium Dioxide (ZrO2) bond harmoniously with 10-MDP monomer. This monomer targets and creates bond between the acidic groups (phosphoric acid) present in MDP and the oxide layer of the zirconia, thus resulting in an increase in the bond strength between resinous substrate and zirconia. Therefore, the bond between SBU and zirconia ceramics favored by the presence of the 10-MDP monomer. The results for the SBU are in congruence with those of another study.

Scotchbond 3M (universal adhesive containing silane) has reinforced adhesion to the silica-based inorganic fillers due to the presence of silane as its constituent ingredient. The productivity of this silane was highly effective via light activated polymerization, considerably improving the bond strength. As per the evidence provided in the literature, photo-polymerized adhesives and silane coupling agents when used together can increase the bonding ability.

The concentration of 10 MDP between the two universal adhesives has a significant role to play when it comes to bonding. There is a positive association of 10 MDP (% by weight) with the bond strength to zirconia. However, there is scarcity of information in the available literature regarding the exact concentration of 10 MDP in the universal adhesive; it can be assumed that **Silane-free universal adhesive of group 1** used in this study does not contain the required concentration of 10 MDP to facilitate chemical reaction with the zirconia ceramics.

The present study also evaluated the application of silane coupling agent prior to the application of Universal Bonding Agent. Separate step of silanization yielded
higher shear bond strength value when compared to the other groups. Higher bond strength values achieved would be due to the physical effect of silane, that ameliorates the wettability of the zirconia ceramics, promoting higher degree of contact between the universal adhesive and the resin cement applied onto the zirconia ceramics. The physical property of surface wettability of silane coupling agent is thus responsible for the higher bond strength values. Additional step of surface conditioning with silane improves the bond strength between the resin cement and high translucency zirconia also due to the active reaction of the silane that expands equally with the exposed inorganic area. Significant difference was found amongst all the three groups in this study, which is consistent with the previous studies cited in the literature.

Several factors are responsible for higher values of μSBS; first, the initial chemical reaction that is nurtured by low degree of pH. Second, the hydrophobic nature of methacryloxydecyl dihydrogen phosphate (10-MDP) monomer that prevents hydrolytic degeneration and thus protects the adhesive interface. It also enhances the reaction stability. The adhesion to the passive hydroxyl groups is provided by the phosphate ester group. Third, there is an activation of the resin on the zirconia surface due to the dissolution of modified methyl-methacrylate (MMA) with polycrylic acid monomer. This results in the formation of a favourable medium for polymerization of free carbon double bonds with carbon compounds present in the universal adhesive. Also, the bond strength improves due to the incorporation of the resin primer as a result of relatively low molecular weight of MMA into the roughened resin matrix of the Zirconia. Besides the above stated factor, the presence of water in universal adhesives aids in the activation process by the acidic monomers ionization.

Study limitations include the fact that clinically, bonded restorations are subjected to a combination of thermal and biomechanical stresses rather than only the thermal stress used in this in-vitro study. Non-uniform stress distribution observed with the SBS test can be a possible reason for the inconsistencies in the data of shear bond strength. Adhesive bonding between zirconia and the resin cement plays a crucial role in long-term durability of the prosthesis. Hence, long-term clinical trials need to be conducted in order to know the importance, surface conditioning methods have in enhancing the bond strength, to substantiate the results of this in vitro study.

**Conclusion**

The application of Silane Coupling Agent prior to the application of Universal Bonding Agent yielded highest Shear Bond Strength values between high translucency zirconia and the resin cement. This research may have a clinical implication in dental prosthodontic research, indicating that the application of MDP primers along with silane treatment yielded highest bond strength.

In the present study, a combination of air abrasion, treatment with MDP-based product and silane treatment resulted in high bond strength values and chemical affinity, due to enhanced surface wettability via air abrasion and increased bond strength via the treatment with MDP-based primers and silane coupling agent.
Acknowledgements

- This work is supported by Prof. Dr. Riyaz HOD., Department of Oral Pathology and Microbiology, Yenepoya Dental College, Mangalore.
- The testing procedures conducted in the study were done under the guidance of Dr. Jayaprakash, Department of Dental Materials, Yenepoya Dental College, Mangalore.

References


30. Colares RC, Neri JR, Souza AM, Pontes KM, Mendonça JS, Santiago SL. Effect of surface pretreatments on the microtensile bond strength of


