THE EFFECT OF TWO ADHESIVE PROMOTERS ON BOND STRENGTH OF ULTRA-TRANSLUCENT ZIRCONIA TO TWO RESIN CEMENTS.

Amira K. Hafez¹, Amina M. Hamdy², Soha O. Nabih³

Abstract

Statement of the problem: The increased amount of crystals in zirconia ceramics and the absence of glass make them resistant to acid etching and chemical bonding through traditional silane application. Alternative durable bonding techniques between zirconia and resin cement are still mandatory.

Objective: This study was conducted to investigate the effect of two adhesive promoter types and two resin cements on the shear bond strength of sandblasted ultra-translucent zirconia plates to resin cement.

Materials and Methods: BruxZir anterior Zirconia blank was sliced to obtain forty-two ultra-translucent zirconia plates, followed by their sintering. All the plates were sandblasted using 50 µm alumina particles. Plates were divided into three groups according to the type of adhesive promoter: Group A: no primer, Group B: coated with zirconia primer, and group C: coated with a universal bonding agent. One plate from each group was examined under Scanning Electron Microscope. Half of the plates in each group were bonded to conventional composite resin cement while the other half were bonded to Phosphate containing one following the manufacturers’ instructions. All plates were stored in an incubator containing distilled water at 37 °c for 1 week. Shear bond strength test was performed using universal testing machine. Data obtained were analyzed statistically.

Results: The application of Z prime and bonding to conventional resin cement resulted in the highest shear bond strength values (19.58 MPa) followed by no primer application and bonding to phosphate resin cement (15.8 MPa). Universal bonding agent showed the lowest bond strength values with both resin cements.

Conclusions: Application of MDP-containing primer showed the highest bond strength with conventional resin cement while the universal adhesive showed weak bond strength with both resin cements. Resin cements reacted almost equally when used directly on zirconia without primer application.

KEYWORDS: Adhesive promoters, Ultra-translucent zirconia, Resin cement

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Introduction

In recent decades the increasing demand for esthetic restorations made all ceramic materials an excellent choice for dentists. Zirconia ceramics have been used widely in dentistry as a core material for conventional and resin-bonded fixed partial dentures due to their superior mechanical properties in comparison to lithium disilicate ceramics. In 2015, monolithic ultra translucent cubic zirconia was introduced. The average flexural strength of cubic zirconia is 550MPa. It provides better esthetics than other types of zirconia but lower mechanical properties. High strength Zirconia restoration can be cemented to the prepared tooth using conventional or self-adhesive resin cements. However, resin cements are more favored in some situations where retention is highly required. Resin bonding to Zirconia ceramic is challenging. Unlike Glass-ceramics, hydrofluoric acid etching and silanization techniques have been ineffective for zirconia-resin bonding. Zirconia is a polycrystalline structure devoid of any glass component and therefore, they are categorized as non-etchable ceramic. Sandblasting is the most preferred surface roughening method for zirconia ceramics. This technique raises its surface energy and wettability. However mechanical adhesion through surface roughening is not sufficient to provide a durable bond between zirconia and resin cement. Therefore, a combination between mechanical and chemical adhesion is mandatory. The chemical adhesion is accomplished through the innovation of primers and resin cement.

Various zirconia primers have been innovated to enhance the chemical adhesion between the resin cement and dental zirconia. They are easy to apply and do not need costly or complicated apparatus. Application of primers or resin cements containing 10-methacryloyloxy- decyl dihydrogen phosphate (MDP) monomer after sandblasting increases the bond strength of zirconia to resin cement. MDP is an acidic phosphate monomer, which was initially intended to bond to metal oxides and nowadays has been used with zirconia. It allows for chemical interaction between the hydroxyl groups of the passive zirconia surface and the phosphate ester group of the MDP. Therefore, it can be used for increasing the bond strength between zirconia and resin cement.

Multi-purpose universal primers have been presented to improve the speed, simplify the bonding procedure to the tooth structure and lessen the chair side time in the clinical practice. They can be used in self-etch, etch and rinse, and selective-etch modes. These primers can provide adhesive strength to zirconia ceramic and precious metal adherents through a few steps. They contain phosphate monomers in their composition. The functional phosphate ester group in MDP molecules bonds directly to the zirconia surface increasing the bond strength between zirconia and resin cement.

The aim of this study was to investigate the effect of adhesive promoter type (zirconia primer and universal bonding agent) and resin cement type (conventional and phosphate-containing) on the shear bond strength of sandblasted ultra-translucent zirconia to resin cement. The null hypothesis of this study was that neither the adhesive promoter type nor the resin cement type would affect the bond strength of the zirconia to resin cement.

Materials and Methods:
The materials used in this study regarding their product names, manufacturers, lot numbers, and chemical composition were presented in table 1.
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Ultra-translucent Zirconia blank was sliced to produce 42 plates, each of dimensions: 10mm width×5mm length×2mm thickness, using a diamond disc fixed on Isomet 4000 micro saw (Buehler, USA). The plates were finished using silicon carbide 400 grit paper. All the plates were placed on the tray of zirconia sintering furnace (Nabertherm, Germany). The sintering cycle was performed according to the manufacturer’s recommendations. Acrylic resin bases were fabricated especially to hold the zirconia plates. All plates were sandblasted by sandblasting machine (Renfert, Germany) using 50 µm alumina particles with a pressure of 2.5 bar for 20 seconds. Specimens were held at 90º and 10 mm distance from the sandblasting pen using a specially designed device.

The forty-two plates were divided into 3 main groups according to the application of adhesive promoter: Group A: Sandblasted only with no primer (n=14). Group B: sandblasted and coated with zirconia primer (n=14). Z-PRIME was added in 2 coats by applying air free oil/water syringe for 10 seconds for each coat. Group C: sandblasted and coated with the universal bonding agent (n=14). The bond was also added in 2 coats, each was air-dried for 10 seconds and then light-cured for 10 seconds. One plate from each group was scanned using an environmental Scanning Electron Microscope (SEM) to explore the surface morphology at a magnification of 2000X, then elemental analysis through Energy Dispersive X-ray (EDX) was done and tabulated. Before bonding procedures, All the plates were put in an ultrasonic cleaner (MCS, China) for 3 minutes in distilled water.

Each main group was subdivided into 2 subgroups according to the resin cement type: Subgroup 1: plates were bonded to conventional resin cements (n=21). Subgroup 2: plates were bonded to adhesive resin cements (n=21). Small transparent polyvinyl tubes were cut. Each tube was 4 mm in diameter, 3mm in height, and 2mm thick. Each tube was bonded to the center of each zirconia plate using a bonding agent (BISCO Schaumburg, USA) and cured with a light-emitting diode (Woodpecker, Germany) of high intensity 1600mW/cm2 for 10 seconds. Each cement was injected into the tube by its dispensing tip till the filling of the tube. The excess cement was removed by a micro brush. Then the resin cement was light-cured for 20 seconds. A pencil was used to draw a circle around each tube to define the area of bonding during the microscopic examination after the shear bond strength test. Using a sharp scalpel blade number 15, the tubes were cut and removed to get a resin cement cylinder (Fig.1). All the samples were stored in an incubator (Titandx, Italy) containing distilled water at 37 ºc for 1 week.

### Table 1: Materials used in this study.

<table>
<thead>
<tr>
<th>Material</th>
<th>Product name</th>
<th>Chemical composition [wt %]</th>
<th>Manufacturer</th>
<th>Lot number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultra-translucent Zirconia blank</td>
<td>Dentsply/Innomax</td>
<td>25.0% BiO3, 15.0% Y2O3, 25.0% SiO2, 30.0% Al2O3, 10.0% Na2O, 5.0% La2O3</td>
<td>Dentsply/Innomax</td>
<td>DBL343</td>
</tr>
<tr>
<td>Zirconia primer</td>
<td>Z-Prime</td>
<td>38.5% Zr 39.0% Mg 19.5% Al</td>
<td>Bisco</td>
<td>10000951</td>
</tr>
<tr>
<td>Universal bonding agent</td>
<td>All Bond/Vercrem.</td>
<td>15.0% Bis-GMA, 7.0% Pentaerythritol</td>
<td>Bisco</td>
<td>10000760</td>
</tr>
<tr>
<td>Conventional resin cement</td>
<td>Z-Prime</td>
<td>15% Bis-GMA, 2.5% Amine</td>
<td>Bisco</td>
<td>10000760</td>
</tr>
<tr>
<td>Resin composite resin cement</td>
<td>Z-Prime</td>
<td>15% Bis-GMA, 2.5% Amine</td>
<td>Bisco</td>
<td>10000760</td>
</tr>
</tbody>
</table>

The forty-two plates were divided into 3 main groups according to the application of adhesive promoter: Group A: Sandblasted only with no primer (n=14). Group B: sandblasted and coated with zirconia primer (n=14). Z-PRIME was added in 2 coats by applying air free oil/water syringe for 10 seconds for each coat. Group C: sandblasted and coated with the universal bonding agent (n=14). The bond was also added in 2 coats, each was air-dried for 10 seconds and then light-cured for 10 seconds. One plate from each group was scanned using an environmental Scanning Electron Microscope (SEM) to explore the surface morphology at a magnification of 2000X, then elemental analysis through Energy Dispersive X-ray (EDX) was done and tabulated. Before bonding procedures, All the plates were put in an ultrasonic cleaner (MCS, China) for 3 minutes in distilled water.

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Figure 1: Resin cement cylinder bonded to zirconia plate after cutting the tube.

Each zirconia plate with its resin cylinder was fixed to the lower compartment of a universal testing machine (LLOYD universal testing machine, England) by screws with 5 KN load, and data were taken down by computer software. A compression mode of force at a crosshead speed of 0.5mm/min was applied to all the samples at the resin cylinder zirconia interface up to failure using a universal testing machine (Fig.2).

Figure 2: Shear bond strength test.

All the samples were investigated to detect the mode of failure using a digital microscope (Scope capture digital microscope, Guangdong, China) at a magnification of 15X. Failure modes were classified as an adhesive failure at the resin/zirconia interface, cohesive failure within the resin cement or zirconia, or mixed adhesive/cohesive failure.

Numerical data were explored for normality by checking the data distribution using the Shapiro-Wilk test. Data showed parametric distribution. Two-way ANOVA followed by Tukey’s post hoc test was used to study the effect of different tested variables and their interaction. A comparison of the main and the simple effects was done utilizing multiple t-tests with Bonferroni correction.

Results:

Morphological and elemental surface analysis:

Untreated ultra-translucent zirconia plate SEM images showed surface grooves with some debris caused by the milling process during the preparation of the samples (Fig. 3), while EDX analysis showed: the highest peak was detected for zirconia element (56.13 Wt %) also, a low peak for oxygen (23.46 Wt %) and carbon elements (20.42 Wt %) were detected.

Sandblasted ultra-translucent zirconia showed a highly rough surface with irregularities (Fig. 4) and its EDX analysis showed a noticeable peak of alumina representing 0.58Wt%.

SEM images of sandblasted zirconia coated with Z-PRIME showed micro pores and irregularities that were coated with a darker layer (Fig.5) The EDX analysis showed a peak of phosphate (0.73 wt%) and carbon (73.44 wt %). For the surface of zirconia coated with the universal bonding agent: the surface was totally covered with a grey layer and white patches were presented (Fig.6). For the EDX analysis: a peak of carbon (79.91 wt%), oxygen (15.91 wt%) and lower peak of phosphate (2.17 wt%) were detected.

Fig.3: SEM image of untreated Zirconia
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Shear bond strength:
Main effects:
Regardless of the type of resin cement, there was a significant difference between values of different groups (p=0.008). The highest shear bond strength (MPa) value was found in zirconia primer (17.32 MPa ±5.23), followed by no primer (control) (15.20 MPa ±3.34), while the lowest value was found in universal bonding agent (12.32 MPa ±3.18). Post hoc pairwise comparisons showed value of zirconia primer to be significantly higher than that of universal bonding agent (p<0.001) (Table 2).

Table 2: Mean ± standard deviation (SD) of shear bond strength (MPa) for different primers.

<table>
<thead>
<tr>
<th>Shear bond strength (MPa) (mean±SD)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>No primer</td>
<td>15.20±3.34</td>
</tr>
<tr>
<td>Zirconia primer</td>
<td>17.32±5.23</td>
</tr>
<tr>
<td>Universal bonding agent</td>
<td>12.32±3.18</td>
</tr>
</tbody>
</table>

Means with different superscript letters are statistically significantly different; significant (p ≤ 0.05) ns; non-significant (p>0.05)

Regardless of primer type, conventional resin cement (15.26 MPa ±5.04) had a higher value than phosphate containing resin cement (14.63MPa±3.85) yet the difference was not statistically significant (p=0.610) (Table3).

Table 3: Mean ± standard deviation (SD) of shear bond strength (MPa) for different types of resin cements.

<table>
<thead>
<tr>
<th>Shear bond strength (MPa) (mean±SD)</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional</td>
<td>15.26±5.04</td>
</tr>
<tr>
<td>Phosphate containing</td>
<td>14.63±3.85</td>
</tr>
<tr>
<td>p-value</td>
<td>0.610ns</td>
</tr>
</tbody>
</table>

*; significant (p ≤ 0.05) ns; non-significant (p>0.05)

Effect of primer within each resin cement:
As shown in table 4: For the conventional resin cement, there was a significant difference between the values of different groups (p=0.005). The highest shear bond strength (MPa) value was found in zirconia primer (19.28 MPa ±6.13), followed by no primer (control) (14.53 MPa ±2.75), while the lowest value was found in universal bonding agent (11.98 MPa ±2.73). Post hoc pairwise comparisons showed the value of zirconia primer to be significantly higher than that of the universal bonding agent (p<0.001).

For the phosphate-containing resin cement, there was no significant difference between the values of different groups.
The highest shear bond strength (MPa) value was found in no primer (control) (15.88 MPa ±3.94), followed by zirconia primer (15.36 MPa ±3.57), while the lowest value was found in universal bonding agent (12.65 MPa ±3.76).

Table 4: Mean ± standard deviation (SD) of shear bond strength (MPa) for different primers with resin cements.

<table>
<thead>
<tr>
<th>Primer</th>
<th>Resin cement</th>
<th>No primer</th>
<th>Z–PRIME</th>
<th>ALL BOND UNIVER S AL</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional</td>
<td>Composite resin cement</td>
<td>14.53±2.75</td>
<td>19.28±6.13</td>
<td>11.98±2.73</td>
<td>0.005</td>
</tr>
<tr>
<td>resin cement</td>
<td>Phosphate-containing resin cement</td>
<td>15.88±3.94</td>
<td>15.63±3.57</td>
<td>12.65±3.76</td>
<td>0.278</td>
</tr>
</tbody>
</table>

Effect of resin cement within each primer:

As shown in table 5: when no primer was applied, phosphate-containing resin cement (15.88 MPa ±3.94) had a higher value than conventional resin cement (14.53 MPa ±2.75) yet the difference was not statistically significant (p=0.531). For the zirconia primer: conventional resin cement (19.28MPa±6.13) had a higher value than phosphate-containing resin cement (15.36MPa±3.57) yet the difference was not statistically significant (p=0.074). While for the universal bonding agent: phosphate containing resin cement (12.65MPa±3.76) had a higher value than conventional resin cement (11.98MPa±2.73) yet the difference was not statistically significant (p=0.754).

Table 5: Mean ± standard deviation (SD) of shear bond strength (MPa) for resin cements with different primers.

<table>
<thead>
<tr>
<th>Resin cement</th>
<th>Primer</th>
<th>Conventional composite resin cement</th>
<th>Phosphate containing resin cement</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>No primer</td>
<td>14.53±2.75</td>
<td>15.88±3.94</td>
<td>0.531</td>
<td></td>
</tr>
<tr>
<td>Z–PRIME</td>
<td>19.28±6.13</td>
<td>15.63±3.57</td>
<td>0.074</td>
<td></td>
</tr>
<tr>
<td>ALL BOND UNIVERSAL</td>
<td>11.98±2.73</td>
<td>12.65±3.76</td>
<td>0.754</td>
<td></td>
</tr>
</tbody>
</table>

The mode of failure:

For the control groups, the results showed adhesive failure and all remaining groups showed mixed failure.

Discussion:

The extended durability of all ceramic dental restorations depends on mechanically and chemically stable bonds between restoration and tooth. The conventional etching-silane treatment is not indicated for polycrystalline, glass-free non-etchant zirconia. [12] Thus surface modification for zirconia is mandatory. In this in vitro study, we investigated the effect of two adhesive promoters and two resin cements on the bond strength of sandblasted ultra-translucent zirconia to resin cement. In this study sandblasting was done to cubic zirconia ceramic with 50 µm Al2O3[13] [14], 2.5MPa pressure and 10 mm operation distance according to Blatz MB et al.[13] Sandblasting is the most preferred surface-roughening method for zirconia ceramics. This method increases the surface energy and wettability but can weaken the ceramic by creating micro cracks on the zirconia surface. However, it has been shown that resin luting agents heal the minor surface flaws created by sandblasting and strengthen the ceramic. [5]

Airborne-particle abrasion with aluminum oxide does not always give rise to a well-founded resin bond to zirconia. [15] Therefore, different zirconia primers have...
been innovated to attain chemically improved adhesion between the resin cement and the zirconia. [16]

Z Prime Plus was selected as zirconia adhesion promoter in this study. It contains two adhesive functional monomers (carboxylic and MDP monomers) that interact chemically with the zirconium oxide layer at the resin-zirconia interfaces. Universal bonding agent, which is composed of 10-MDP, Dimethacrylate resins, HEMA, Ethanol, Water, and Initiators, was also selected as an adhesion promoter. MDP monomers are also available in some types of self-adhesive resin cements which might improve the bonding of zirconia to resin cement. Therefore, this study was conducted to also investigate the effect of two resin cements, conventional and phosphate-containing resin cement, on bond strength to cubic zirconia.

The results of this study showed that: The SEM image of sandblasted zirconia plate showed a highly rough surface with irregularities. This suggested the increase in bond strength to resin cement. Sandblasting increases the surface roughness and activates the surface to enhance bond strength. [17]

When no primer was used after sandblasting: the phosphate-containing resin cement showed a higher shear bond strength value (15.88±3.94) than the conventional resin cement (14.53±2.75). Yet the difference was not statistically significant. (p=0.531) This may be due to the presence of MDP in phosphate-containing resin cement. 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP) is an acidic phosphate monomer, which is originally intended to bond to metal oxides and then was used with zirconia. It has an affinity to metal oxides and zirconia surface that is covered with a passive oxide layer, so it can be used for increasing the bond strength between zirconia and resin cement. [18] [19]

These findings are in agreement with Miragaya L et al [20] who documented that irrespective of the surface treatment, the phosphate-containing resin cements performed better regarding bond strength to zirconia ceramic than a conventional resin cement.

When primer was used followed by application of conventional resin cement, it was found that: The highest shear bond strength (MPa) value was found with zirconia primer (19.28 MPa ±6.13), followed by no primer (control) (14.53 MPa ±2.75), while the lowest value was found in universal bonding agent (11.98 MPa ±2.73). This may be due to Z-PRIME Plus that has two adhesive functional monomers (carboxylic and MDP monomers. The phosphate group in MDP can bond with the hydroxyl group in the oxide layer found in zirconia. The carboxylic acid group (methacrylate) has a carbon double bond, which can be polymerized and bonded to the composite resin. These interfacial forces improved the surface wettability and chemical affinity of zirconia which caused an increase in the interlocking with the resin cement. The combined effect of the acidic MDP and carboxylate monomers is the best explanation for the higher bonding strength seen with this primer. [21][22] The high value of shear strength can also be explained by the combined effect of mechanical (sandblasting) and chemical reaction (MDP) between zirconia and resin cement. The SEM image of sandblasted zirconia coated with z-prime showed many micro pores and irregularities that served as retentive areas for resin cement. Also, the EDX analysis showed the phosphate percentage in zirconia coated with z-prime was 7.73Wt% that enhanced the chemical bonding with zirconia. The EDX analysis showed the carbon percentage was 73.44Wt%. The better aging resistance of conventional resin cement...
cement may be attributed to the presence of the Bis-GMA monomer.
The lowest shear bond strength value was found in the universal bonding agent (11.98 MPa ±2.73), this may be attributed to the lower phosphate content as was evident in EDX analysis (2.17 Wt%). Also, the layer deposited on surface as evident in SEM images might act as a barrier for proper bonding with resin cement.

When the primer was used followed by application of phosphate-containing resin cement, it was found that: The highest shear bond strength (MPa) value was found with no primer (control) (15.88 MPa ±3.94), followed by zirconia primer (15.36 MPa ±3.57), while the lowest value was found in the universal bonding agent (12.65 MPa ±3.76). There was no significant difference between values of different groups. The highest shear bond strength when no primer was applied before bonding to phosphate resin cement can be attributed to phosphate group in MDP that chemically bonded with the hydroxyl group in the oxide layer found in zirconia.

These results are in agreement with Stefani [23] who reported that bond strengths of phosphate-containing resin cement to the zirconia ceramics didn’t change by primer application. This result showed that the increased MDP concentration in both the primer and phosphate-containing resin cement did not lead to an additional enhancement in bond strength.

Nagaoka [24] and others reported that higher concentrations of 10-MDP in primers yielded higher shear bond strengths with a concentration dependency and suggested that a minimum 1-ppb MDP was needed to bond to zirconia. In fact, commercial 10-MDP-containing primers possess more than 1wt% 10-MDP, whilst phosphate-containing resin cements also contain higher concentrations of 10-MDP. In this study z-prime used has 1-5wt% 10-MDP and the phosphate-containing resin cement has 10-30 wt% 10-MDP. The results showed that the 1-5wt% 10-MDP found in primer was enough to produce a high bond strength.

On the other hand, using z prime resulted in lower bond strength than no primer application when bonding to phosphate-containing resin cement. Yet the difference was not statistically significant (p=0.074). Different studies do not agree with the results in this study as they have documented an increase in adhesion when previously applying a 10-MDP primer, especially with phosphate-containing resin cement.[25][26] On the contrary, another study stated the opposite in phosphate-containing resin cement due to the saturation of this molecule. The use of an MDP-containing primer was effective only for the non–MDP-containing resin cement but did not increase the bond strength to the phosphate-containing resin cement. [27]

In this study, the use of universal bonding agent combined with sandblasting did not improve significantly the bond strength of zirconia to resin cement. The higher shear bond strength of Z prime and bonding to phosphate-containing resin cement than using universal bond can be attributed for the phosphate content revealed in EDX analysis (7.73Wt% & 2.17Wt % respectively).

This was in agreement with Sharafeddin F et al [28] who found that the Z-Prime Plus treatment exhibited the highest bond strength than All-Bond Universal treatment. This can be explained by water absorption of this universal adhesive as a consequence of aging which resulted in decreased adhesion. [29]

The mode of failure was adhesive in control groups where no primer was used. All other groups’ mode of failure was mixed. Correlating the mode of failure and bond strength values, it is recommended to use Z-prime and conventional resin cement (19.28
To obtain high shear bond strength value and mixed mode of failure that denotes more about better bonding quality. The null hypothesis was rejected with both primers and was accepted with both resin cements.

Within the limitation of the present study, the following conclusions can be drawn:

1. Resin cements reacted almost equally when used directly on zirconia without primer application.
2. Application of MDP containing zirconia primer prior to bonding with conventional resin cement provided the highest bond strength to zirconia.
3. Application of Universal bonding agent showed the lowest bond strength to both resin cements tested in this study.

References:

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