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

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

Abstract

Statement of the problem: Due to their high crystal content and lack of glass, zirconia ceramics resist acid etching and chemical bonding with silane. More durable zirconia-resin cement bonding techniques require advancement. **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, Sintering to all plates was carried out according to manufacturer's regulations. All the plates were sandblasted using 50 µm alumina particles. Plates were allocated 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 a 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. The shear bond strength test was carried out using a 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 value (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 value with both resin cements.

Conclusions: Application of MDP-containing primer had 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.

Introduction

In recent decades the increasing demand for esthetic restorations made all ceramic material an excellent choice for dentists. Zirconia ceramics are utilized in conventional and resin-bonded fixed partial dentures because they offer better mechanical qualities than lithium disilicate ceramics. [1] 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 has lower mechanical properties. [2] High-strength Zirconia restoration can be cemented to the prepared tooth using conventional or self-adhesive resin cement. However, resin cement is more favored in some situations where retention is highly required. [3] Resin bonding to Zirconia ceramic is challenging. For zirconia-resin bonding, hydrofluoric acid etching and silanization end up failing, unlike glass ceramics. Zirconia is a polycrystalline structure devoid of any glass component and therefore, they are categorized as nonetchable ceramic. [4] Sandblasting is the most preferred surface roughening method for zirconia ceramics. This technique raises its surface energy and wettability. [5] However mechanical adhesion through surface roughening is not sufficient to provide a durable bond between zirconia and resin cement. [6] Therefore, a combination of mechanical and chemical adhesion is mandatory. The chemical bonding is accomplished through the innovation of primers and resin cement. [7]

To improve resin cement-dental zirconia chemical bonding, various primers have been invented. They are simple to use and require no special equipment. [8] After sandblasting, primers or resin cement with 10-methacryloyloxy-decyldihydrogen phosphate (MDP) monomer enhance zirconia-resin cement bonds. [8,9] Although originally designed to bond to metal oxides, MDP is currently employed with zirconia. It allows passive zirconia surface hydroxyl groups and MDP phosphate ester groups to interact chemically. Therefore, it can strengthen zirconia-resin cement bonds. [10]

Multipurpose 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 utilized in selfetch, etch and rinse, and selective-etch modes. These primers can provide adhesive strength to zirconia ceramic and precious metal adherents through a few steps. Their composition includes phosphate monomers. The functional phosphate ester group in MDP molecules bonds directly to zirconia, strengthening resin cement-zirconia attachment. [11] The purpose of this study was to explore the influence 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 ultratranslucent zirconia to resin cement. The null hypothesis of this study postulated that neither the adhesive promoter type nor the resin cement type would affect the bond strength of ultra translucent zirconia to resin cement.

Materials and Methods:

The materials used in this study regarding their product names, manufacturers, and chemical composition were presented in Table 1. Table1: Materials used in this study.

Material Description, product name and manufacturer.	Chemical composition (wt. %)
Ultra-translucent zirconia	ZrO ₂ >99.0%,Y ₂ O ₃ 5.15%,HFO ₂ <3%, AL ₂ O ₃ <0.01%,SiO ₂ , 0.02,Fe ₂ O ₃ <0.01%and
(BruxZir Anterior White)	Na ₂ O<0.04%
Zirconia primer	Ethanol 75-85%, Bisphenol ADiglycidyl - methacrylate 5-10%, 2-Hydroxyethyl
(Z-Prime plus, BISCO Schaumburg, USA)	Methacrylate 5-105-%, Proprietaryl-5%, MDP1- 5%
Universal bonding agent(All- Bond Universal, BISCO Schaumburg, USA)	10-MDP,Dimethacrylate resins, HEMA, Ethanol, Water, Initiators
Conventional composite resin cement (Duo_Link cement, BISCO Schaumburg,USA)	Base composition: BisGMA 10-13%,Urethan Dimethacrylate 10-13%,Tetrahydrofurfuryl Methacrylate 1-5%. and Trimethylolpropane Trimethacrylate 1-5%. Catalyst composition: Bisphenol A Diglycidylmethacrylate 10-30% and Dibenzoyl peroxide< 1%
Phosphate containing resin cement (TheraCem, BISCO,Inc,Schaumburg,USA)	Base composition: Ytterbium w/Barium Glass 30- 50%, Portland cement 20-50%, proprietary 1- 10%, Ytterbium Fluoride 1-5%, BisGMA 1-5% and proprietary <1%. Catalyst composition: 10 Methacryloyloxydecyl Dihydrogen phosphate 10- 30%, 2Hydroxyethyl Methacrylate 1-5% and Tert- butyl Perbenzonate 1-5%

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 400grit 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 specially 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 90° 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, followed by 10 seconds of light curing. 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 cement (n=21). Subgroup 2: plates were bonded to adhesive resin cement (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 °c for 1 week.



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 a 5 KN load, and data were collected 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 samples were examined using a 15X Scope capture digital microscope (Guangdong, China) to determine the mode of failure. Adhesive, cohesive, or combined adhesive/cohesive failures were encountered.

The Shapiro-Wilk test examined the numerical data distribution for normality. The distribution was parametric. A two-way ANOVA and Tukey's post hoc test were used to examine factors' effects and interactions. The main and simple effects were compared using multiple t-tests with a Bonferroni adjustment.

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 (07.73 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(15.91wt%) and lower peak of phosphate (2.17 wt%) were detected.

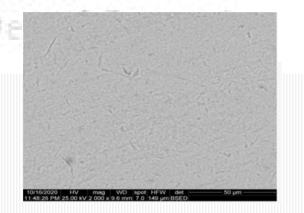


Fig.3: SEM image of untreated Zirconia

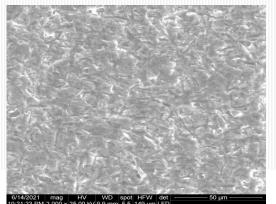


Fig.4: SEM image of sandblasted Zirconia.

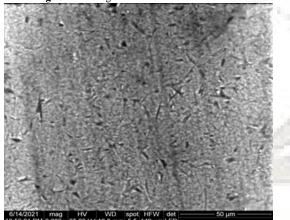


Fig. 5: SEM image of sandblasted zirconia coated with Z-PRIME

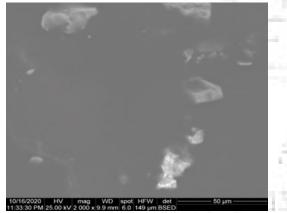


Fig.6: SEM image of sandblasted zirconia coated with universal bonding

Shear bond strength: Main effects:

Regardless of the type of resin cement, there was a significant difference between the 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 \pm standard deviation (SD) of shear bond strength (MPa) for different primers.

Shear bond strength (MPa) (mean±SD)

No primer	Zirconia primer	Universal bonding agent	p- value
15.20±3.34 AB	17.32±5.23A	12.32±3.18B	0.008*

Means with different superscript letters are statistically significantly different *; significant ($p \le 0.05$) ns; non-significant (p > 0.05)

Regardless of primer type, conventional resin cement (15.26 MPa \pm 5.04) had a higher value than phosphate containing resin cement (14.63MPa \pm 3.85) yet the difference was not statistically significant (p=0.610) (Table 3).

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

Shear bond strength	p-value		
Conventional	Phosphate containing		
15.26±5.04	14.63 ± 3.85	0.610ns	
*; significant ($p \le 0.05$) 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 (p=0.278). 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 \pm standard deviation (SD) of shear bond strength(MPa) for different primers with resin cements.

Shear bond strength (MPa) (mean±SD)				Р
Primer Resin cement	No primer	Z –PRIME	All Bond Universal	value
Conventional composite resin cement	14.53±2.75	19.28±6.13	11.98±2.73	0.005
Phosphate- containing resin cement	15.88±3.94	15.63±3.57	12.65±3.76	0.278

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 $(15.36MPa\pm3.57)$ 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)

Table5: Mean \pm standard deviation (SD) of shear bond strength (MPa) for resin cements with different primers

Shear bond strength (MPa) (mean±SD)			
			value
Resin cement	Conventional	Phosphate	
	composite resin	containing	
primer	cement	resin cement	
No primer	14.53±2.75	15.88 ± 3.94	0.531
Z-PRIME	19.28±6.13	15.63 ± 3.57	0.074
ALL BOND	11.98±2.73	12.65±3.76	0.754
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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. This in vitro study examined the effect of two adhesive promoters and two resin cement types on the bond strength of sandblasted ultratranslucent zirconia to resin cement.

In this study sandblasting was done to cubic zirconia ceramic with 50 μ m Al₂O₃[13] [14], 2.5MPa pressure and 10 mm operation distance according to Blatz MB et al.[13] best zirconia ceramic surface-The roughening process is sandblasting. This approach enhances surface energy and wettability but weakens ceramic by producing zirconia microcracks. However, resin luting agents reinforce ceramic and repair sandblasting surface imperfections. [5] Airborne-particle abrasion with aluminum oxide does not always give rise to a wellfounded resin bond to zirconia. [15] Therefore, different zirconia primers have been invented to attain chemically improved bonding between the resin cement and the zirconia. [16]

This study used Z Prime Plus to improve zirconia adhesion. Carboxylic and adhesive functional **MDP** monomers chemically interact with the zirconium oxide layer at the resin-zirconia interface. A universal bonding agent ,containing 10dimethacrylate resins, HEMA, MDP, ethanol, water, and initiators, was also chosen to improve adhesion. Some self-adhesive resin cements contain MDP monomers, which may increase zirconia-resin cement adhesion. This study also examined the bond strength of conventional and phosphatecontaining resin cements to cubic zirconia.

The results of this study showed that: The SEM image of the 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 could be attributed to MDP in phosphatecontaining resin cement. It was originally meant to bond to metal oxides; however, 10-MDP was employed with zirconia. It bonds well to metal oxides and passive oxidecovered zirconia surfaces, making it useful resin-cement-zirconia strengthening for bonds. [18] [19] Miragaya L. et al. [20] found phosphate-containing resin cement that bonded to zirconia ceramic better than traditional resin cement, regardless of surface treatment.

When primer was used followed by application of conventional resin cement, it was found that: The highest shear bond strength (MPa) value was reported for 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 might occur because Z-PRIME Plus has carboxylic and MDP adhesive monomers. MDP's phosphate group bonds with zirconia's oxide layer's hydroxyl group. Polymerizing and bonding methacrylate to composite resin uses its carbon double bond. These interfacial pressures increased zirconia's surface wettability and chemical affinity, which increased resin cement interlocking. This primer's greater bonding strength is due to acidic MDP and carboxylate monomers. [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 zprime 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 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 could be related to the lower phosphate content as was evident in EDX analysis (2.17 Wt%). Also, the layer deposited on the surface as evident in SEM images might act as a barrier for proper bonding with resin cement.

When the primer and phosphate containing resin cement were utilized, 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 the values of different groups. The highest shear bond strength when no primer was applied before bonding to phosphate resin cement could be referred to the phosphate group in MDP that chemically bonded with the hydroxyl group in the oxide layer found in zirconia.

These findings coincide with Stefani [23], who found that priming did not affect the bond strengths of phosphate-containing resin cement to zirconia ceramics. Increased MDP content in primer and phosphate-containing resin cement did not boost bond strength.

Nagaoka [24] and others found that primers with greater 10-MDP concentrations had stronger shear bond strengths with a concentration dependency and that zirconia minimum MDP. required 1-ppb а Commercial primers and phosphatecontaining resin cements include greater than 1wt% 10-MDP. In this investigation, z-prime 1-5wt% 10-MDP and phosphatehas containing resin cement has 10 -30wt%.Primers with 1-5wt% 10-MDP produced high bond strengths.

When bonding to phosphatecontaining resin cement, z prime had inferior bond strength than no primer. But the difference was insignificant (p=0.074). Other investigations have found that using a 10-MDP primer increases adhesion, especially with phosphate-containing resin cement.[25][26] A different study found the opposite in phosphate-containing resin cement due to molecular saturation. For non-MDP resin cement, an MDP-containing primer increased bond strength, but not for 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 rather than universal bond after bonding to phosphate-containing resin cement could be related to the phosphate content revealed in EDX analysis (7.73Wt% & 2.17Wt % respectively).

This was aligned 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 failure mode 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 Zprime and conventional resin cement (19.28 MPa) to obtain a high shear bond strength value and mixed mode of failure that denotes more about better bonding quality.

According to the results of this study, the null hypothesis was rejected with both primers and was accepted with both resin cements.

Based on the findings of the present study, the following conclusions could be drawn:

1- Both types of resin cement reacted almost equally when used directly on zirconia without primer application.

2- The application of MDP containing zirconia primer prior to bonding with conventional resin cement provided the highest bond strength to zirconia.

3- The application of the Universal bonding agent exhibited the lowest bond strength for both resin cement types tested in this study.

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