

## **Bonding To Zirconia with A Recently Introduced Universal Adhesive. An In-Vitro Study**

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**Aim:** This in vitro study was aimed to evaluate the efficacy of a recently introduced universal adhesive in replacing traditional adhesive promoters for bonding resin to ultra translucent zirconia.

**Materials and Methods:** 8 rectangular disc-shaped ultra translucent zirconia specimens (Katana UTML Kuraray Noritake Dental Inc. Tokyo, Japan) with dimensions of (18 x 10 x 2 mm) were used as bases to attach 56 miniature cylinders of flowable conventional composite material (Polofil NHT Flow, VOCO GmbH, Germany) with a 1 mm diameter. Every zirconia disc served as a substrate for 7 composite microtubes. Zirconia plates were divided into 4 groups (2 plates each) according to surface pretreatment as follows: GPI: air abrasion+ Zr primer, GPII: air abrasion+ universal adhesive, GPIII: silica coating+silane and GPIV: silica coating+ universal adhesive. (n=14 each). Using a wire and loop technique, the micro-shear bond strength was evaluated using a universal testing equipment. Failure load (N) was determined, and the failure type was determined when the samples were studied under a stereomicroscope. Data were statistically analyzed by the two-way ANOVA test.

**Results:** GPI was significantly highest mean values for micro-shear bond strength (27.83MPa±2.29) followed by GPIII with less mean values (27.45MPa±3.16) but no significant difference, GPII and GPIV: were significantly lowest mean values of microshear bond strength being (14.77MPa±3) and (5.16MPa±1) respectively.

**Conclusion:** The bond strength decreased significantly when traditional zirconia primers with air abrasion or silica coating were replaced with the tested universal adhesive alone.

**Keywords:** Bonding, Zirconia, Universal Adhesives, Air-abrasion, Silica-coating

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## Introduction

Because of its exceptional mechanical properties—flexural strength of over 1000 MPa—, chemical stability, biocompatibility, and beneficial optical qualities over metallic-based restorations, Zirconia is a material that is becoming increasingly prevalent every day.<sup>1</sup> With the launch of its new generations, even its opaqueness and veneering needs have been addressed and enhanced over the previous 10 years.<sup>2</sup> Raising the percentage of the yttrium oxide  $Y_2O_3$  content above traditional 3%mol to stabilize more cubic phase boosted the translucency for creating aesthetically monolithic restorations.<sup>3</sup> yet the loss of transformation toughening with the increase of cubic phase on the expense of tetragonal one has led to more dependence on bonding to increase the flexural strength specially with minimal restoration thickness.<sup>4</sup> The paradigm change toward minimally invasive bonded restorations completely relies on the provision of a long-term, secure bonded restoration, significantly reducing the importance of mechanical retention.<sup>5</sup> However, zirconia is a polycrystalline, chemically inert substance that is devoid of silica, rendering bonding to it an intricate procedure that has been extensively investigated in the literature since its first generations.<sup>6</sup> Modifying zirconia surface to increase the bonding to resin was mainly enhanced through three channels that include every tested surface treatment.<sup>7</sup> Initial reliance of these channels was placed on creating micromechanical surface roughness that increases the surface wettability through multiple investigated procedures such as: sandblasting, silica coating, selective infiltration etching, hot chemical etching solutions, plasma spraying, laser irradiation, or zirconia ceramic powder coating.<sup>8,9,10</sup> Chemical adhesion to metallic oxides through adhesive promoters such as phosphate ester group or 10 methacryloxydecyl dihydrogen phosphate (10-MDP) was the second main

way to secure bonding.<sup>11</sup> The third channel complied with deposition of silica particles on the surface to compensate for the inherent absence of silica in its composition combining mechanical and chemical retention.<sup>12</sup> Recent years have witnessed the release of universal primers, the ultimate generation of adhesives for enhancing the bonding to zirconia, which has received some research attention without having enough supported data from literature.<sup>13</sup> These adhesives comprise the main adhesive promoters such as silane and phosphate-ester based monomers. Regardless of surface preparation or cement type, the producers assert that these agents can offer good bonding to zirconia.<sup>14</sup> There are a few thorough investigations comparing various zirconia primer or universal adhesive combinations with surface treatments.<sup>13, 15</sup> This study aimed to evaluate the effectiveness of a newly introduced adhesive that was claimed by the manufacturer as universal substitution to Zr primers and adhesive promoters. The null hypothesis was that no significant difference of micro-shear zirconia resin bond strength when using the newly introduced Universal Adhesive “containing adhesive promoters” to substitute traditional primers after Zr surface pretreatments.

## Materials and Methods

Materials used in this study are listed in Table (1).

### Sample size calculation:

In order to do a statistical test of the null hypothesis—which states that there is no difference in bond strength across the various tested groups—a power analysis was created with sufficient power. With an alpha ( $\alpha$ ) level of 0.05, a beta ( $\beta$ ) level of 0.85, and an effect size ( $\omega$ ) of 0.398 determined by utilizing data from earlier research, the total number of samples needed ( $n$ ) was determined to be 56, with 14 samples needed for each group. For

Windows, R statistical analysis software version 4.3.2 was used to calculate the sample size.

Table 1: The material, composition and manufacturer

Material	Composition	Manufacturer
<b>Ultra-translucent Zirconia</b>  (Katana UTML)	87–92% ZrO <sub>2</sub> + HfO <sub>2</sub> , 8–11% Y <sub>2</sub> O <sub>3</sub> , other oxides 0–2%	Kuraray Noritake Dental Inc .  (Tokyo, Japan)
<b>Flowable nano hybrid resin composite</b>  (Polofil NHT Flow)	Filled dimethacrylate based (BISGMA, TEGDMA, HEMA, UDMA).	VOCO GmbH, Germany.
<b>Universal bonding agent</b>  (Beautibond Xtreme)	Acetone, water, Bis-GMA, carboxylic acid monomer, TEGDMA, organophosphate monomer, acid resistant silane coupling agent	Shofu, Kyoto, Japan.
<b>Porcelain primer</b>  ( Z prime plus)	Priming agent, MDP monomer, BPDMA, carboxylate monomer	BISCO, Inc. Schaumburg, IL The USA.
<b>Silane coupling agent</b>  ( Silane Porcelain Primer <sup>TM</sup> )	Pre-hydrolyzed no-mix silane primer and Bis-Silane.	BISCO, Inc. Schaumburg, IL, USA.

### Specimens' preparation:

56 mini cylinders of flowable conventional composite material (name) with diameter of 1mm, attached to eight rectangular disc-shaped zirconia specimens (18x10x 2 mm). Each zirconia disc holds

seven microtubes. Zirconia discs were prepared from pre-shaded (Katana UTML; Kuraray Noritake Dental Inc. Tokyo, Japan) blank. Single block was milled after digital designing from the blank in a form of bar with dimensions of (16mm x18mm x10mm) which was then sliced in a thickness of 2mm each by a high-performance cutting micro saw (Isomet 4000 Buehler, USA) using a diamond disc under water cooling. Every plate was set up on the zirconia sintering furnace tray (Nabertherm, Germany). The manufacturer's instructions for the sintering cycle were followed. Silicon carbide was used to polish each plate. The specimen was 1.8 mm thick following sintering and polishing using abrasive sheets (P1200, P1500, and P2500). Specimens were cleaned and dried in an oven at 100°C after five minutes in an ultrasonic bath (iSonic Inc., Chicago, Illinois) containing 78% isopropyl alcohol and ten minutes in acetone. Subsequently, the samples were examined using a ×10 optical microscope to assess the consistency of the ceramic surfaces' polishing. Based on the surface treatment process, the discs were split into the following four groups:

- 1) GpI: Air-borne particle abrasion (APA) + Zr primer
- 2) GpII: Air-borne particle abrasion (APA)+ universal adhesive.
- 3) GpIII: silica coating (SC)+ silane
- 4) GpIV: silica coating (SC) + universal adhesive.

### Substrate surface treatments:

Air-borne particle abrasion (APA):

Blocks were air abraded with an intraoral air abrasion device (Air prophylaxis unit, Artspa industrial company, China) with 50 µm of aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) with 2.5 bar pressure at 10 mm distance perpendicular to the surface for 10 seconds. Following an extensive 10 seconds of air/water spray rinsing and 5 seconds of air drying, the air-abraded surfaces were ultrasonically cleaned

(Eumax ®, Hong Kong Model number: UD80SH-2.6L) in distilled water for 5 minutes and 10 seconds of air drying.

For **GP I**: air abrasion was followed by Z-Prime Plus application (BISCO, Inc, Schaumburg, U.S.A.) according to manufacturer's instructions. The zirconia ceramic surfaces received a single, even application of Z-Prime Plus to completely cover the surface. For three to five seconds, the surfaces were softly dried with air spray.

While for blocks in **GPII**, air abrasion was followed by universal adhesive application (BeutiBond Universal, Shofu, Japan) according to manufacturer's instructions. Using a micro-brush, it was applied to the ceramic surfaces for 20 seconds. The solvent was then evaporated by air drying for 5 seconds with an air sprayer. Finally, an LED light curing unit (Radii plus, SDI, Australia) was used to light cure it for 20 seconds.

**Silica Coating (SC):**

Regarding zirconia blocks in **GP III** and **GP IV**, Cojet Sand (3M, Blast Coating Agent, USA) was used to provide a silica coating to every block. The blocks underwent 10 seconds of air abrasion at a distance of 10 mm and a pressure of 2.5 bar perpendicular to the surface. After the silica coating process, the surfaces were completely cleaned with air/water spray for 10 seconds and allowed to air dry for 5 seconds. Then, for five minutes, the Eumax ®, Hong Kong model number: UD80SH-2.6L was ultrasonically cleaned in distilled water, and it was allowed to air dry for ten seconds. This was followed by:

For **GP III**, The tribochemical silica surface was coated with a layer of Silane coupling agent (Silane Porcelain Primer™), allowed to react for 60 seconds, and then allowed to air dry.

While for **GPIV**, universal adhesive application (BeutiBond Universal, Shofu, Japan), with the same procedure discussed earlier.

**Application of the resin material:**

After surface treatment in all groups, matrices in a form of transparent tygon tubes (Tygon Medical Tubing, Saint-Gobain; Akron, OH, USA) with a height of 0.7 mm and an interior diameter of 1 mm. Seven tygon tubes were placed on each zirconia disc, giving rise to seven specimens per zirconia disc, with a total of 14 specimens per each group and 56 specimens total for all groups. A2 shade of flowable resin composite (Polofil NHT Flow, Voco, GMBH, Germany) used in each group and handled according to the manufacturer's instructions, and injected into the tygon tube positioned on the pretreated ceramic blocks, under magnification of 4.3x using magnifying loupes (Carl ZEISS Meditec, Germany) under LED light to facilitate the injection step. The excesses were removed with a spatula, then covered with a Mylar strip (Stripmat, Polydentia, Mezzovico, Switzerland). Then a glass slide was placed on top over the tygon tube. The flowable resin composite was light-cured for 20 seconds using a light-curing unit (Radii plus, SDI, Australia) with an output of 1600 mW/cm<sup>2</sup>, the intensity of the LED light curing unit was periodically checked after each 10 specimens using the device building radiometer according to manufacturer's instructions. The tygon tubes were carefully removed by a surgical blade #15, yielding resin composite cylinders that adhered to the ceramic surface. For a week, all the samples were kept at 37 °C in an incubator (Titandx, Italy) filled with distilled water.

**Micro-Shear Bond Strength Testing:**

Cyanoacrylate glue (UHU, Germany) was used to attach each zirconia disc to chemically cured resin blocks (Acrostone, Egypt) so they could be installed on the Universal Testing Machine (LR5K series, LLOYD Instruments, Ltd., UK). The wire and loop method was used to measure the micro-shear bond strength. Each composite cylinder had a small, 0.2 mm-diameter wire



attached to it that contacted the cylinder's lower half. The wire was fastened to bars intended for this use on the other side. To provide appropriate shear stress distribution at the interface, the connections between the cylinder, wire, and rods were placed in the same line. Wire and loop should be as near to the interface as feasible before loading the program. Then, until failure, shear force was applied at a crosshead speed of 1 mm/min. The load at failure was measured and shown on the monitor in Newtons. By dividing the load in N by the composite cylinder's surface area (the contact surface area with a 0.7 mm diameter), the micro-shear bond strength in MPa was computed.

#### Determination of the Mode of failure:

By observing fractures on surfaces under a stereomicroscope (SMZ 745T, Nikon, Japan) at 40x magnification, the mode of failure was identified. The failure mode was identified as cohesive (inside the composite), adhesive (at the interface between the resin composite and zirconia), or mixed (a mixture of both cohesive and adhesive). Every failed bonded region was photographed using the microscope's (WAT-221S, Japan) camera.

#### Statistical analysis:

Values for the mean and standard deviation (SD) were used to show numerical data. Using the Shapiro-Wilk and Levene tests, respectively, and observing the distribution, they were examined for normality and variance homogeneity. The data were normally distributed with homogenous variances across different variables. They were analyzed using a two-way ANOVA test. The comparisons of simple effects were made using the error term of the two-way model with p-value adjustment using the False Discovery Rate (FDR) method. The significance level was set at  $p < 0.05$  within all tests. Statistical analysis was performed with R statistical analysis software version 4.4.1 for Windows.

## Results

Results of the Two-way ANOVA presented in Table (2) showed that there was a significant interaction between the type of surface treatment and adhesion primer on micro-shear bond strength ( $p < 0.001$ ). The comparisons of simple effects presented in Table (3) showed that when using traditional primers, there was no significant difference between samples treated using either method ( $p = 0.717$ ). However, while using alternative primers, air-abraded samples had significantly higher bonding strength than silica-coated samples ( $p < 0.001$ ). Regardless of the surface treatment utilized, significantly higher bond strengths were achieved with traditional priming methods ( $p < 0.001$ ). Mean and standard deviation values for bond strength values are presented in Figures (1) and (2).

Table 2: Two-way ANOVA test

Source	Sum of squares (II)	df	Mean square	f-value	p-value
Surface treatment	349.11	1	349.11	46.61	<0.001*
Primer	4370.24	1	4370.24	583.54	<0.001*
Treatment * primer	298.45	1	298.45	39.85	<0.001*

df degree of freedom, \* significant ( $p < 0.05$ ).

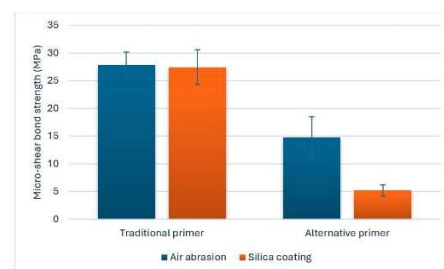


Figure1 : Bar chart showing mean and standard deviation values of micro-shear bond strength (MPa).

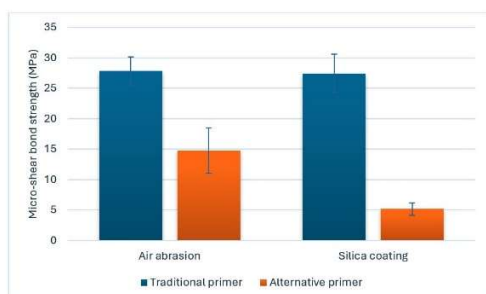


Figure2 : Bar chart showing mean and standard deviation values of micro-shear bond strength (MPa).

## Discussion

With zirconia becoming more and more important in dental practice, optimizing adhesion to it is a challenging yet crucial procedure. Literature addressed this topic throughout the years of using zirconia in dental practice.<sup>6,16,17</sup> The first most straightforward and most efficient methods for improving zirconia bonding have been mechanical roughening by air blasting and chemical conditioning by reacting with adhesion boosters such as phosphate-ester based monomers.<sup>18,19</sup> APC approach<sup>20</sup> was recommended by Blatz M and coworkers as the basic routine for treating zirconia surface. The second most suggested method in bonding to zirconia literature was the deposition of silica on the surface of zirconia that permits roughening and silica deposition, initiating chemical bonding through the conditioning of surface with silane coupling agent via what is known as tribochemical silica coating TBS<sup>21</sup>. Manufacturers are attempting to streamline processes and reduce the number of clinical stages by offering universal adhesives that come with the necessary adhesion promoters and have been demonstrated to be an effective replacement for zirconia primers. To the best of the authors' knowledge, there aren't many studies in dental literature examining how well these universal adhesives work to replace the chemical bond formed by adhesive promoters that are given individually specially regarding the new generations of zirconia.<sup>22</sup> This study was

aimed to examine the effectiveness of one of the universal adhesives to substitute the chemical conditioning step -including 10-MDP monomer after air blasting or Silane coupling agent after silica deposition-producing a durable zirconia resin bond strength. conventional flowable nanohybrid composite was used in this investigation to standardize the samples and guard against any potential issues that may arise from using tooth structure,<sup>23</sup> avoid the contribution of any functional monomers in the resin part to exclude its effect in evaluating the used adhesive promoters,<sup>24</sup> and this helped partly to simulate a repair scenario besides being used to replace resin cement whenever higher filler content is needed to secure mechanical properties.

The null hypothesis was rejected as significant difference between groups that utilize the universal adhesive and the groups that utilize the traditional recommended surface treatments and its compatible adhesive promoters as follows; the significantly highest bond strength was recorded for GpI that represented the mechanical roughening with air blasting followed by conditioning with the Zr primer containing 10-MDP, the selection of the size of alumina particles of 50µm may have attributed to the high bond strength values. these results were coinciding with the literature supported the benefits of using smaller sizes of alumina particle abrasion for conventional 3-YTZP.<sup>12,25</sup> Zhang et al<sup>26</sup> recommended the same particle size optimized by other parameters of pressure and distance like the used in this study to obtain the best surface treatment for 5-YTZP. While other authors like Zhao et al<sup>27</sup> suggested larger size and found the smaller ones insufficient for desired surface roughness revealing substantial improvement in bond strength with particle sizes up to 110 µm and air pressure up to 3 bar. They asserted that there was no reduction in flexural

strength and no tetragonal monoclinic transition which was supported with McLaren and coworkers.<sup>28</sup> Modifying blasting parameters might give different bonding results.<sup>29</sup> Z-Prime Plus conditioning undoubtedly contributed to the high records of bond strength. As at the resin-zirconia contact, carboxylic and MDP adhesive functional monomer interact chemically with the zirconium oxide layer.<sup>30</sup> MDP monomer-based primers outperformed other functional monomer-based primers by a large margin, according to research by De Angelis and colleagues.<sup>31</sup> Using conventional composite as the cement component eliminates the contribution of any functional monomers in the cement, making Zr primer functional monomers 10-MDP the only component that is evident. As reported, the hydroxyl group of zirconia's oxide layer forms a connection with the phosphate group of MDP.<sup>32</sup> Methylacrylate's carbon double bond is utilized to polymerize and bind with composite resin that can withstand aging.<sup>33</sup><sup>34</sup> These interfacial pressures increased zirconia's surface wettability and chemical affinity, which increased resin interlocking.<sup>35,36</sup> The combination of acidic 10-MDP and carboxylate monomers gives this primer the higher bond strength records.<sup>37</sup> The mode of failure was mainly cohesive that represented a durable resin-zirconia bond strength. In second place, GPIII recorded a significantly higher bond strength with lower mean microshear bond strength values than GPI but with insignificant difference. as it was well proved that SC efficiently serves for both surface roughening and adding more silica to the zirconia surface, which makes the surface more receptive to silanization and increases its chemical affinity for resin.<sup>38</sup> Failure modes were predominately mixed supporting the weaker bond strength than the first group of APA. One possible explanation is that the surface roughness resulting from silica coated particles might not be as suitable for bonding

as surface roughness induced by APA, this have been referred to in literature through SEM analysis and surface roughness analysis.<sup>39</sup> Moreover, the particles covered with silica might lack the ability to securely attach themselves into the zirconia surface, especially after aging and hydrolysis of the silanized layer<sup>40</sup> added to the use of silane that is prehydrolyzed.<sup>41</sup> Using a two-bottle silane system might have given different results. Absence of MDP molecule may also be a cause for this technique to give lower bond values as some literature conversely reported higher bond strength values with TBS while using a primer or a cement that having MDP functional monomer.<sup>42</sup> literature revealed that bond strength is directly related to the concentration of 10-MDP component as zirconia needed a minimum of 1-ppb MDP, according to research by Nagaoka and colleagues<sup>21</sup>, while primers with higher 10-MDP concentrations exhibited larger shear bond strengths with a concentration dependency. According to this study, the used Zr primer used in GPI possesses 1–5% 10-MDP.

GP II and GP IV had the third and last, respectively, statistically significant lowest bond strengths values with the adhesive mode of failure was predominating. This pointed out that using this universal adhesive in place of the original functional primers drastically reduced the values of the zirconia bond rather than increasing it. However, GPII with the preceded air abrasion significantly recorded higher mean micro-shear bond strength values than GPIV which was preceded by silica coating. This might be explained by the effectiveness of alumina air-abrasion to treat the surface<sup>43</sup> and at the same time the low efficiency of silane to attach well to the silica coated surface. The low silane effectiveness may be referred to the form it is supplied with and the concentration.<sup>44</sup> The Beautybond Xtreme adhesive monomers are primarily composed of organophosphate molecules and

an acid-resistant silane coupling agent, according to the manufacturer.<sup>45</sup> The adhesive is also offered in a HEMA-free version that facilitates water evaporation and application.<sup>45</sup> Meanwhile, when it was used with any of the surface pretreatments, reduced mean micro shear bond values were observed. Even with the efficient air-drying of such universal adhesive, the production of a silane layer applied onto the zirconia surface appeared to be less plausible.<sup>46</sup> This helps partially explain why universal adhesive did not prime as well as conventional primers. Similar results were assessed by Hafez AK and coworkers.<sup>47</sup> The universal adhesive's acidic nature, on the other hand, may have had an impact on the silane molecules' chemical stability inside the universal adhesive formulations, reducing their capacity to prime, therefore producing a weak resin-zirconia connection.<sup>48</sup> Although this kind of universal bond is provided as a Quadro-functional adhesive agent, its lower zirconia-resin mean bond values appeared to be related to the lack of 10-MDP, the well-proven adhesive promoter due to its forementioned properties.<sup>49,50</sup> More investigations with other types of universal adhesives are needed for further evaluation of their efficacy for bonding of such zirconia material.

### Conclusions

1. Air-borne particle abrasion combined with 10-MDP conditioning is the most reliable surface treatment for zirconia restorations.
2. Tribochemical silica coating is a reliable method for zirconia conditioning, but stability of the bond is questionable.
3. The tested universal adhesive failed to replace the conventional Zr primers with significantly lower bond strength values.

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### Data availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

### Ethics approval

“Research Ethics Committee of Faculty of Dentistry Ain Shams University FDASU-REC” waived ethical approval for this study due to being an In-vitro study with no patients, animal experiments or living tissues were included. Exemption no: FDASU-Rec ER032413

### Conflict of interest

The authors declare that they have no conflicts of interest.

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