

## BOND STRENGTH OF A SELF-ADHESIVE RESIN CEMENT TO Y-TZP CERAMIC SUBJECTED TO DIFFERENT SURFACE TREATMENTS

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### ABSTRACT

**Objective:** The objective of this study was to evaluate the influence of different surface treatments on shear bond strength of a yttrium stabilized zirconia (Y-TZP) to a self-adhesive resin cement. **Methods:** Cylindrical samples (5mm diameter x 10mm height) of a Y-TZP zirconia (Lava, 3M ESPE) were divided into four groups (n = 10): C) no surface treatment (control); A) universal adhesive (Universal Bond Single, 3M ESPE); J) sandblasting with silica-coated aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) particles; and JA) sandblasting with silica-coated Al<sub>2</sub>O<sub>3</sub> + universal adhesive + silane. The self-adhesive resin cement (Rely X Ultimate, 3M ESPE) was used to cement ceramic cylinders on acrylic resin blocks. The light-curing was performed using a LED (Valo, Ultradent) with irradiance of 1000 mW/cm<sup>2</sup> for 25s. Samples were stored for 24 hours in dark at 37°C. The shear bond strength test was performed using a universal testing machine (EMIC, DL-2000). Data were analyzed statistically by ANOVA-1 way and Tukey test (5%). **Results:** The JA group showed the highest bond strength values. Both A and J groups showed the lowest bond strength values and did not differ statistically between them. The control group presented intermediate values. **Conclusions:** The surface treatment of the Y-TZP ceramic using sandblasting with silica-coated Al<sub>2</sub>O<sub>3</sub> and universal adhesive significantly improved the bond strength between this ceramic and self-adhesive resin cement.

**KEYWORDS:** Ceramic. Resin cements. Silicon dioxide.

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## INTRODUCTION

Dental ceramics have good aesthetic, biological, mechanical properties and functional requirements of a restorative material.<sup>1</sup> The fragility of feldspathic (glassy matrix) ceramic is the main disadvantage of this material. This ceramic needed to be reinforced by an increased crystalline content (synthetic ceramics) resulting in higher mechanical properties. Crystals such as leucite, lithium disilicate, alumina and zirconia were used to create the reinforced ceramics that present better mechanical properties, such as a fracture resistance two times higher than feldspathic ceramics.<sup>2</sup>

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Besides reinforced glass ceramic, there are also the polycrystalline ceramics that present a reduced (to none) volume of glass. The main characteristic of these ceramics is the fine grains content in the crystalline structure increasing their mechanical properties. However, the absence of the glassy matrix creates a limited translucency as well as does not promote etching with hydrofluoric acid in these ceramics.<sup>2</sup> Among the polycrystalline ceramics, the zirconia-based ceramics present high physicochemical proper-

ties, such as flexural strength, fracture toughness (similar to metal) and hardness, high modulus of elasticity, and excellent dimensional stability and good biocompatibility with the buccal tissues.<sup>3</sup>

Zirconia assumes three crystallographic forms according to temperature: monoclinic, tetragonal and cubic. Zirconia in its pure state has a monoclinic crystalline structure at room temperature, and is stable up to 1170°C. Between 1170°C and 2370°C the zirconia has a tetragonal structure. Above 2370°C, the zirconia crystals have a cubic shape.<sup>4</sup> During the cooling the tetragonal phase again becomes monoclinic and this transformation results in a substantial volume expansion of approximately 3-4%. But, after the cooling the yttrium oxide is added to zirconia in order to maintain in a metastable phase at temperatures below the tetragonal to monoclinic transformation temperature, creating the yttria-stabilized zirconia (Y-TZP). This procedure improves the mechanical strength of the zirconia, because when the Y-TZP is induced by tensions that initiate the propagation of a crack, the tetragonal crystals close to the crack become the stable monoclinic phase. The volume expansion occurred during this transformation causes stresses of compression that oppose the growth and propagation of the crack.<sup>5</sup> Due to this combination of factors, the Y-TZP is a viable option for manufacturing infrastructures and indirect restorations.<sup>1,2</sup>

The understanding of the cementation clinical process of ceramic restorations requires the approach of three main topics: surface treatment of the restoration, treatment of the tooth, and the luting materials used<sup>6</sup>. The polycrystalline ceramics, such as Y-TZP, have reduced glass matrix and they are considered acid-resistant ceramics. Therefore, the hydrofluoric acid (5 or 10%) does not enhances surface roughening and, consequently, surface area.<sup>1,2,6</sup> Polycrystalline ceramics require alternative adhesive techniques, such as sandblasting with different sizes of aluminum oxide ( $Al_2O_3$ ) particles. When these particles are silica-coated, the process is called a tribochemical<sup>7-9</sup>. The acceleration of the particle during the sandblasting causes an impact and the silica stays adhered on the ceramic surface. This process is also known as silicization. In this case, chemical bonding agents must be applied after silicization. The use of the silane agent is also indicated with materials containing functional monomers such as adhesive systems (primer) with phosphate monomer 10-MDP (10-methacryloxydecyl dihydrogen). As an alternative, self-adhesive resin cements with functional monomers can be used in this process.<sup>8,10</sup>

The self-adhesive capacity of resin-based materials containing functional monomers results from the presence of radicals derived from carboxylic acids, phosphoric acids or their esters, or the incorporation of organic acids as additives. These monomers etching the substrate where they are being applied. Among the functional monomers available in commercial self-adhesive materials, besides 10-MDP, there are 4-MET (4-methacryloyloxyethyl trimellitic acid), o 10-MAC (11-methacryloyloxy-1,1-undecanedicarboxylic acid) and phenyl-P (2-[methacryloyloxyethyl]phenyl hydrogen phosphat).<sup>2,10</sup>

Besides the divergence in the dental literature regarding the adhesive protocols (particle size used in sandblasting, use of primers and its association with surface treatments), several alternatives protocols have been proposed with the aim of improving the adhesion on polycrystalline ceramics<sup>7-10</sup>. Thus, the aim the present study was to evaluate and compare the effect of different surface treatments of Y-TZP on shear bond strength to self-adhesive dual resin cement.

## MATERIAL E METHODS

Forty cylindrical samples of Y-TZP (Lava, 3M ESPE) were produced by CAD/CAM system (5mm diameter x 10mm height). The dimensions were measured using a digital caliper with accuracy of 0.01mm (Mitutoyo). After polishing of the samples with silicon carbide sandpaper, the cylindrical samples were cleaned in ultrasonic bath (T14, L&R ultrasonic) for 3 minutes in deionized water.

The specimens were divided into 4 groups (n = 10) according to the surface treatment:

C (control): without surface treatment. The cementation procedure was performed following the manufacturer's instructions;

A: application of a universal adhesive system containing 10-MDP (Single Bond Universal, 3M ESPE) on Y-TZP surface;

S: sandblasting with silica-modified aluminum oxide ( $\text{Al}_2\text{O}_3$ ) particles with  $50\mu\text{m}$  during 10s, at 1.5 bar pressure, at 10 mm distance (Fig 1).

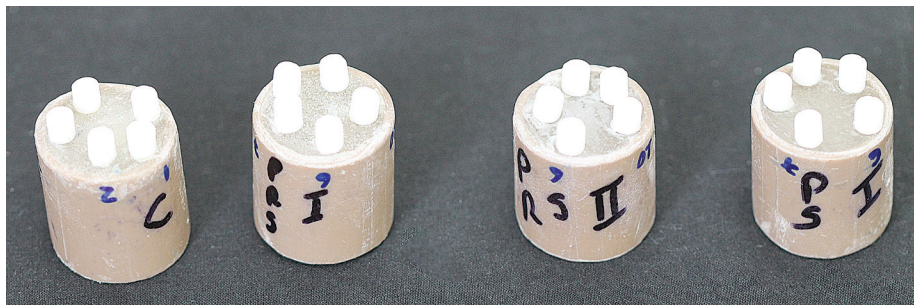


**Figure 1:**

Sandblasting of the specimens.

SA: sandblasting in the same way as in group S + universal adhesive system and silane.

The treated surfaces were marked with a permanent pen for identification during the cementation. The samples that received sandblasting were subjected to an ultrasonic bath for 3 minutes in alcohol 96%. The groups that received universal adhesive followed the protocol recommended by the manufacturer (application for 20s and gently air-dried for 5s). For the cementation of the resin cylinders, the self-adhesive dual resin cement was dispensed (RelyX Ultimate, 3M ESPE) and both base and catalyst pulps were handled for 20s. A uniform resin cement layer was applied in each sample prior the light-curing procedure. The ceramic samples were luting on acrylic resin blocks at a constant pressure (~ 1kg) (Fig 2).



**Figure 2:**

Ceramic specimens cemented on acrylic resin blocks.

Thus, the light-curing was performed using a light emitting diode (LED, Valo, Ultradent) with irradiance of 1000 mW/cm<sup>2</sup> for 25s, in order to ensure the complete polymerization of the resin cement. The specimens were stored in dark for 24h at 37°C.

The shear bond strength test was performed using a universal testing machine (EMIC, DL-2000) at a crosshead speed of 0.5 mm/min. The force required to cause fracture was recorded in Newtons (N). So, the bond strength in Megapascal (MPa) was calculated by dividing the force (N) by the area (mm<sup>2</sup>) of the adhesive interface ( $\pi r^2$ , where  $\pi = 3.14$  and  $r = 2.5\text{mm}$ ).

After the normality test (Kolmogorov-Smirnov), the data were analyzed statistically by ANOVA-one way) and Tukey test (5%).

## RESULTS

Table 1 shows that different surface treatments of the Y-TZP ceramics influenced the shear bond strength means values ( $p < 0.01$ ). The highest bond strength value was found in SA group, which did not differ statistically from the control group ( $p > 0.05$ ). Both A and S groups had the lowest bond strength values and did not differ statistically between them and with control group ( $p > 0.05$ ).

**Table 1:**

Mean (SD) of shear bond strength (MPa) of the different groups evaluated.

GROUPS	BOND STRENGTH
Control (C)	12,79 ( $\pm$ 3,89) <sup>AB</sup>
Adhesive (A)	8,18 ( $\pm$ 2,66) <sup>B</sup>
Sandblasting (S)	7,73 ( $\pm$ 5,48) <sup>B</sup>
Sandblasting + Adhesive (SA)	14,64 ( $\pm$ 6,21) <sup>A</sup>

Means followed by different letters differ from each other ( $p < 0.05$ ).

## DISCUSSION

According to the results of the present study, the tribochemical surface treatment of a Y-TZP ceramic resulted in significant increase of the shear bond strength to self-adhesive dual resin cement. The adhesion between Y-TZP ceramic and resin cement is the result of physicochemical interaction between ceramic/cement interface, and the surface treatment performed in the ceramics to promote this interaction.<sup>10,11</sup> The sandblasting (physical treatment) depends on the surface topography of the ceramic and may be characterized by surface energy, increasing the wettability of the surface.<sup>11</sup> The chemical treatment is promoted with adhesives or primers containing functional monomers, such as 10-MDP, a component present in the adhesive used in the present work.

The association of sandblasting with silica-modified  $Al_2O_3$  particles with  $50\mu m$  associated with adhesive system containing 10-MDP was significant, because SA group presented the highest values of shear bond strength. Sandblasting removes contaminated surface layers and promotes an increase in surface roughness, increasing the micromechanical retention of the resin cement on the ceramic surface and, consequently, improving the bond strength of the cement/ceramic interface.<sup>7,11</sup> It is worth mentioning that this treatment is controversial, because sandblasting could cause micro-cracks in the ceramic, reducing the longevity of the zirconia restorations in 25%.<sup>12</sup> Therefore, the dentist should be careful to choose the size of the particles used in the sandblasting as well as the pressure used during this procedure.

The adhesive and/or resin cement could obstruct the small micro-cracks,<sup>13</sup> besides the fact that sandblasting causes a transformation of the monoclinic phase in Y-TZP, creating stresses of compression that oppose the growth and propagation of the crack and increase the flexural strength of the Y-TZP ceramic.<sup>14</sup> The possible deleterious effects of sandblasting (structural defects in ceramics) were also observed in the present study, because the S group presented the lowest bond strength values. In addition, despite the negative results of sandblasting with silica-modified  $Al_2O_3$  particles on the mechanical properties of Y-TZP ceramics, only adhesives systems containing 10-MDP did not result in improvement of the adhesion values (A group). For this reason, it is necessary the surface treatment associating sandblasting and adhesive, as shown in the present work, where SA group presented the highest bond strength results.

The results in scientific literature should be carefully analyzed, since there is no standardization in the methodology (different sizes of  $Al_2O_3$  particles, pressure and time used in sandblasting, sample size/substrates and specimens' storage) and materials (different adhesives/primers/resin cements), resulting in different bond strength results.<sup>15-18</sup>

In addition to decreasing the flaws that may occur in sandblasting procedure, the adhesives containing 10-MDP is beneficial because the monomer chemically bonds with metal oxides present in Y-TZP.<sup>10,16,18</sup> The application protocol of an adhesive and/or primer after sandblasting is a simple technique and does not require the use of costly equipment. However, such technique is sensitive because require the application of a thin and uniform adhesive layer. A thick layer acts as an obstacle in the adhesion.<sup>10</sup> Moreover, the adhesive layer is susceptible to hydrolysis,<sup>7,10,12</sup> which could decrease the bond strength values (specimens were stored in water). This factor associated with surface roughness not completely removed during polishing with silicon carbide sandpaper of the Y-TZP surface could justify the intermediate bond strength values of the control group.

The clinical long-term success of indirect ceramic restorations in laboratory studies is realized with artificial aging.<sup>12</sup> Thermal and mechanical cycling and/or water storage for a long time are important parameters to be evaluated that may have effects on the shear bond strength of resin cement on Y-TZP surface.<sup>12,13,16</sup> Therefore, this can be considered a limitation of the present study. In vitro mechanical tests allow to evaluate indirectly the clinical performance of materials and restorative techniques. A previous study shows that the adhesive interface analysis of indirect restorations in ceramics can be realized by tensile and shear bond strength tests, with no differences in the results.<sup>19</sup> Considering that polycrystalline ceramics are difficult to cross-section, such as Y-TZP, the shear bond strength test was used in this study.

## CONCLUSION

Based on the obtained results, it can conclude the Y-TZP surface should be sandblasted silica-modified  $Al_2O_3$  particles and receive application of adhesive systems containing 10-MDP for cementation with self-adhesive dual resin cement.

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