

# Evaluation of Tensile Retention of Y-TZP Crowns Cemented on Resin Composite Cores: Effect of the Cement and Y-TZP Surface Conditioning

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## Clinical Relevance

Adhesive cementation using adhesive resin cements promotes higher crown retention when compared with self-adhesive, resin-modified ionomer, and zinc phosphate cements. Conditioning with tribosilicization and application of a thin low-fusing glass porcelain layer plus silanization on the intaglio surface of an yttria-stabilized polycrystalline tetragonal zirconia crown seems to improve crown retention.

## SUMMARY

**This study evaluated the effect of the cement type (adhesive resin, self-adhesive, glass ion-**

**omer, and zinc phosphate) on the retention of crowns made of yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP). Therefore, 108 freshly extracted molars were embedded in acrylic resin, perpendicular to their long**

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axis, and prepared for full crowns: the crown preparations were removed and reconstructed using composite resin plus fiber posts with dimensions identical to the prepared dentin. The preparations were impressed using addition silicone, and Y-TZP copings were produced, which presented a special setup for the tensile testing. Cementation was performed with two adhesive resin cements (Multilink Automix, Ivoclar-Vivadent; RelyX ARC, 3M ESPE, St Paul, MN, USA), one self-adhesive resin cement (RelyX U100, 3M ESPE), one glass ionomer based cement (RelyX Luting, 3M ESPE), and one zinc phosphate cement (Cimento de Zinco, SS White, Rio de Janeiro, Brazil). For the resin cement groups, the inner surfaces of the crowns were subjected to three surface treatments: cleaning with isopropyl alcohol, tribochemical silica coating, or application of a thin low-fusing glass porcelain layer plus silanization. After 24 hours, all groups were subjected to thermocycling (6000 cycles) and included in a special device for tensile testing in a universal testing machine to test the retention of the infrastructure. After testing, the failure modes of all samples were analyzed under a stereomicroscope. The Kruskal-Wallis test showed that the surface treatment and cement type ( $\alpha=0.05$ ) affected the tensile retention results. The Multilink cement presented the highest tensile retention values, but that result was not statistically different from RelyX ARC. The surface treatment was statistically relevant only for the Multilink cement. The cement choice was shown to be more important than the crown surface treatment for cementation of a Y-TZP crown to a composite resin substrate.

## INTRODUCTION

Zirconia crowns have superior flexural strength ( $> 1000$  MPa)<sup>1</sup> and fracture toughness ( $> 9-10$  MPam<sup>1/2</sup>)<sup>2</sup> than those of other ceramics due to the presence of tetragonal crystals stabilized by yttrium oxide in the material microstructure. By means of thermal, mechanical, or chemical stimulation, these tetragonal grains transform into a monoclinic arrangement, leading to a significant increase in grain volume, generating localized compressive stresses that hinder both slow and fast crack propagation.<sup>3</sup> The outstanding mechanical behavior of yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP) makes it an excellent candidate

material to be used as the infrastructure of both all-ceramic crowns veneered with porcelain and monolithic prostheses.<sup>4</sup>

However, the physical and chemical characteristics of Y-TZP do not favor bonding to current resin cements.<sup>5,6</sup> Although it has been reported by Casucci and others<sup>7</sup> and Blatz and others<sup>8</sup> that resin-based luting agents are the most appropriate materials for the purposes of marginal seal, retention, and fracture resistance, the use of surface pretreatments to improve bonding to the luting agent is mandatory in this case. Different surface treatments have been proposed to modify the zirconia surface and promote mechanical and/or chemical adhesion.<sup>9-13</sup> According to Inokoshi and others,<sup>14</sup> the combination of mechanical and chemical pretreatments can be recommended to promote a stable bond to zirconia.

Sandblasting with aluminum oxide particles coated by silica, followed by the application of silane (silanization),<sup>15,16</sup> has been widely used to increase surface roughness (micromechanical retention) and to establish chemical bonds with resin cements.<sup>17,18</sup> An alternative method is the application of a thin layer of glassy porcelain (rich in silica), followed by etching with hydrofluoric acid and silanization.<sup>12,19-23</sup> According to Ntala and others,<sup>21</sup> that surface treatment increases the capacity of the Y-TZP surface to establish both chemical and micromechanical interactions with resin composites.

Another important factor to be considered when using all-ceramic restorations is the substrate on which the crown will be cemented. When the dentin remaining is insufficient, it is recommended to use an intraradicular post and a composite core buildup to support the crown. In this clinical situation, the use of prefabricated fiber posts associated with a composite core will have a positive impact on the final esthetic result of a cemented zirconia crown.<sup>24</sup> However, it is important to keep in mind that the adhesion process of resin cements to dentin is significantly different than that observed for composite resins. A resin composite core has very few unreacted methacrylate groups at the surface when the cementation procedure takes place. This fact reduces the potential for bonding to a new resinous material, such as the resin cement.<sup>25</sup> In order to improve the adhesion forces, it is necessary to modify the composite core surface to improve chemical bonding to resin cements. An example of a surface treatment for the resin core is the use of self-etching adhesives, which have been proven to promote high bond strength values between aged and new resin

due to an efficient wettability provided by the self-etching systems.<sup>26</sup>

Therefore, it can be concluded that it is necessary to assess not only the bond strength of the resin cements to dentin but also the interface between the resin cement and the composite resin core. Thus, the objective of this study was to compare different treatments of the inner surface of frameworks made of Y-TZP and different types of cements in terms of crown retention (tensile forces) in preparations made mostly of resin composite. The hypotheses were that 1) the different adhesive cements would lead to similar crown retention values; 2) adhesive resin cements would promote higher retention values than the self-adhesive, glass ionomer, and zinc phosphate cements; and 3) the Y-TZP intaglio surface treatment would improve retention regardless of the cement used.

## METHODS AND MATERIALS

In order to determine the sample size, a sample calculation was made based on two other articles.<sup>27,28</sup> Considering a statistical power of 80%, mean standard deviation of 1.7, and a detectable difference of 2.3 MPa, the sample size was established as  $n = 12$ , for a total of 108 teeth.

The teeth were donated by the Human Teeth Bank of the Federal University of Santa Maria and stored in distilled water (4°C) until needed. The 108 teeth were numbered and assigned randomly into nine testing groups (Table 1), using a computer program ([www.randomizer.org](http://www.randomizer.org)).

### Embedding the Teeth

The coronal part of the tooth was glued to an adapted surveyor to keep the long axis of the tooth perpendicular to the ground (horizontal plane) when embedding each root into the acrylic resin. Self-cured acrylic resin (Dencrilay, Dencril, Caieiras, SP, Brazil) was prepared and poured into the matrix. Then the tooth was inserted into the resin, up to 3 mm below the cemento-enamel junction.

### Tooth Preparation

The occlusal surfaces of all teeth were cut with a diamond blade mounted on a cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA), 4 mm above the cemento-enamel junction. Conical trunk diamond burs (KG 3139 and KG 3139FF, KG Sorensen, Cotia, Brazil) were mounted in a high-speed hand piece and fixed to a modified optic microscope to obtain reductions as parallel as

possible to the long axis of the tooth. Thus, the axial walls were reduced at depths of 1.5 mm (similar to the bur diameter), and a standard angle of convergence was created. The height of the preparation was 4 mm.

### Composite Core

- 1) A vinyl polysiloxane impression (Elite Light Body-normal set, Zhermack, Badia Polesine, Italy) of each dental preparation was performed. Then master dies were produced, and a silicone matrix was fabricated on each master die. Thus, the future composite core reconstruction had the same characteristics as the full crown preparation for each tooth.
- 2) All of the dental preparations were removed by cutting with a diamond blade (Isomet 1000).
- 3) For the post cementation, the greatest root canal of each molar received intracanal preparation with a custom #2 drill of the glass fiber post system (White Post DC, FGM, Joinville, Brazil). Afterward, the coronal and root dentin received an etch-and-rinse single-bottle adhesive system (Ambar, FGM). The dentin was etched with 37% phosphoric acid, rinsed with water for 20 seconds, and dried with absorbent papers. The adhesive agent was then applied according to the manufacturer's instructions and light-cured for 20 seconds (Radii-Cal, SDI, Australia). The posts received silane application (Prosil, FGM) and were cemented with a dual-cure resin cement (Allcem, FGM). The core was built up with a composite resin (Opallis, Joinville, Brazil), using the silicone matrix previously made. All preparations were finished with a fine conical trunk diamond bur (3139FF, KG Sorensen) under low rotation. The specimens were stored in water (37°C) for 24 hours.

### Crown Manufacture

The preparations of the specimens were molded with polyvinylsiloxane (Elite Light Body-normal set, Zhermack). Afterward, master dies (CAM-BASE type 4, Dentona, Dortmund, Germany) were obtained and taken to the CEREC MC XL IN LAB for the crown manufacture using Software Inlab 3.60. The copings were designed with retentive features on the occlusal surface for subsequent tensile testing, and milling was performed using VITA In-Ceram 2000 YZ CUBES (VITA Zahnfabrik, Bad Säckingen, Germany) and sintered in a Zircomat

Table 1: *Experimental Design*

Groups (n=12)	Cement Type	Composition	Ceramic Surface Treatment Type
MultC	Self-cured, Hema based resin cement (Multilink) <sup>a</sup>	Dimethacrylate, HEMA, phosphonic acid, methacrylate monomers and barium glass, ytterbium trifluoride, spheroid mixed oxide fillers	Sinalization
MultS			Silicatization <sup>b</sup> + silanization
MultV			Vitrification <sup>c</sup> + silanization
RelC	Dual-cured, Bis-GMA based resin cement (RelyX ARC) <sup>d</sup>	Bisphenol-A-diglycidylether, dimethacrylate (BisGMA) and triethylene glycol dimethacrylate (TEGDMA) polymer, zirconia/silica filler	Silanization
RelS			Silicatization + silanization
RelV			Vitrification + silanization
Ion	Resin-modified glass ionomer cement (RelyX Luting) <sup>e</sup>	Fluoroaluminosilicate (FAS) glass, methacrylated polycarboxylic acid, proprietary reducing agent, bisGMA, HEMA, opacifying agent, potassium persulfate, zirconia silica filler	—
Self	Self adhesive cement (RelyX U100) <sup>f</sup>	Glass powder, methacrylated phosphoric acid esters, triethylene glycol, dimethacrylate (TEG-DMA), silane, treated silica, sodium persulfate	—
Zinc	Zinc phosphate cement (Cimento de Zinco) <sup>g</sup>	Zinc oxide powder, magnesium oxide, dyes, phosphoric acid, aluminum hydroxide	—

<sup>a</sup> The tooth and core surfaces were pretreated with the self-etching and self-curing primers system of the cement. The two primer liquids Multilink Primer A and B were mixed and scrubbed on the core for 30 seconds. Dispersed excess with blown air until the mobile liquid film was no longer visible. Pastes A and B of the Multilink Automix cement were squeezed from the dispenser syringe and the mix applied into the crown. The material excess was removed immediately with a microbrush.

<sup>b</sup> The inner surfaces were air-abraded with 30 μm aluminum oxide particles coated by silica oxide (Cojet Sand, 3M ESPE, St. Paul, MN, USA). The sandblasting was performed with the aid of a suitable device<sup>22</sup> and a constant pressure of 2.8 bars at a distance of 15 mm from the occlusal infrastructure region, with circular movements, for 20 seconds.

<sup>c</sup> Application of a thin layer of low-fusing porcelain glaze (VITA AKZENT, Vita Zahnfabrik, Bad Säckingen, Germany) with the aid of a brush. The infrastructure was subjected to a sintering cycle as recommended by the manufacturer. Afterward, the surface was etched with 10% hydrofluoric acid for 1 minute, washed with water, and air-dried. Subsequently, the copings were cleaned again by an ultrasound device (5 minutes in distilled H<sub>2</sub>O).

<sup>d</sup> The tooth and composite core surfaces were etched with 37% phosphoric acid, rinsed with water for 5 seconds, and dried with absorbent papers. The Single Bond total-etch single bottle adhesive system (Single Bond, 3M ESPE, St. Paul, MN, USA) was applied according to manufacturer's instructions and photocured for 20 seconds (Radii-cal). The two pastes of the RelyX ARC cement were squeezed from the dispenser syringe and mixed and applied into the crown. The material excess was removed immediately with a microbrush and photocured for 20 seconds from each side.

<sup>e</sup> The two pastes of the RelyX Luting cement were squeezed from the dispenser syringe and mixed and applied into the crown. The material excess was removed immediately with a microbrush and photocured for 20 seconds from each side.

<sup>f</sup> The two pastes of the RelyX U100 cement (3M ESPE, St. Paul, MN, USA) were squeezed from the dispenser syringe and mixed and applied into the crown. The material excess was removed immediately with a microbrush and photocured for 20 seconds from each side.

<sup>g</sup> The powder and the liquid of the zinc phosphate cement (Cimento de Zinco - SS White, Rio de Janeiro, Brazil) were mixed and applied into the crown. Material excess was removed immediately with a microbrush.

furnace (VITA Zahnfabrik) as recommended by the manufacturer.

### Framework Cementation

Before Y-TZP framework cementation, the inner surface of Y-TZP copings of the groups Multilink and RelyX ARC were treated using three different methods, according to Table 1.

After the Y-TZP surfaces were conditioned, the surfaces were silanized with a silane agent (ESPE-Sil, 3M ESPE, St. Paul, MN, USA) that was applied for 60 seconds and then thoroughly air-dried. All of the infrastructures were cemented using a device that exercised a force of 750g over the assembled tooth/Y-TZP infrastructure.

### Thermocycling

After cementation, all specimens were stored in distilled water at 37°C for 24 hours and then submitted to thermocycling (6000 cycles) between

5°C and 55°C, according to Ernst and others<sup>27</sup> and Palacios and others.<sup>28</sup>

### Tensile Test

Before tensile testing, part of the cemented infrastructure was embedded in acrylic resin (Dencrilay, Dencril, SP, Brazil) until the resin covered the retention form on the crown. This procedure was performed following the same axis as used with root embedding with the aid of an adapted surveyor.

For testing, the lower base of the assembly was fixed on a universal testing machine (DL-2000, Emic, Pinhais, PR, Brazil), and the upper base was connected to a mobile device that contained a load cell of 1000 N, and the tensile strength test was performed until fracture with a speed of 0.5 mm/min (Figure 1).

### Failure Analysis

The fractured interfacial surfaces of the tested specimens were analyzed under a stereomicroscope

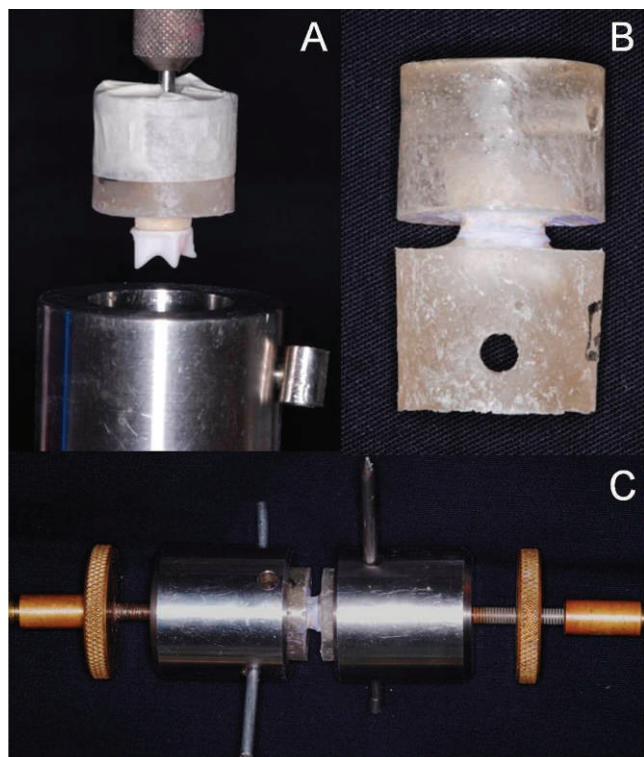


Figure 1. Construction of the specimen for tensile testing. (A): The embedding of the crown into the specific matrix for tensile test. (B): The root and crown embedding with a hole in the acrylic resin. (C): The tensile test device with the specimen inside.

(SteREO Discovery V12, Carl Zeiss, Gottingen, Germany). The failure mode was classified as: over 50% of cement in crown; over 50% of cement in substrate; and catastrophic failure (post debonding, root fracture and removal of the root from the acrylic resin).

### Micromorphological Analysis

Three Y-TZP discs (10 mm in diameter, 3 mm in thickness) were treated with tribochemical silica coating, vitrification, and no treatment, exactly as if conditioning of the inner surfaces of the framework. The micromorphological changes were inspected using scanning electron microscopy (SEM).

### Statistical Analysis

The tensile retention data were submitted to Kruskal-Wallis and Dunn multiple comparison tests ( $\alpha=0.05$ ). A statistical analysis was performed among the groups that were cemented with Multilink and RelyX ARC in order to compare only adhesive resin cements.

The data of the groups MultC and RelC were subject to the Dunn multiple comparison test to

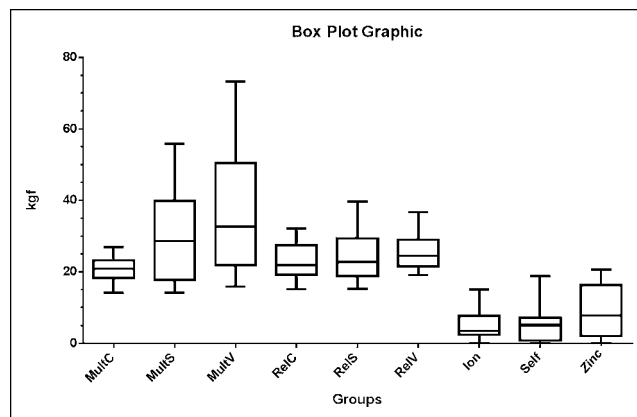


Figure 2. Box-plot graph of all groups, where the upper and lower vertical lines represent the highest and lowest retention values, respectively. The upper and lower lines of the box represent 75th and 25th percentiles, respectively. The horizontal line represents the median.

verify the effect of the cement on the crown without surface treatment. The same was performed for the other surface treatments. The data of the groups MultC, MultS, and MultV were subject to the Dunn multiple comparison test to verify the effect of the surface treatment on the crown cemented with Multilink cement. The same was performed for the RelyX ARC cement.

Another statistical analysis was performed among all groups without the zirconia surface treatment. The data of the groups MultC, RelC, Ion, Self, and Zinc were subject to the Dunn multiple comparison test to compare the cement types without the zirconia surface treatment.

## RESULTS

The median, maximum, minimum, and first and third quartiles (Q1 and Q3) of all groups are shown in the box plot (Figure 2). The data are shown as mean and standard deviation for better comparison with others studies. The comparisons of the groups cemented with the adhesive resin cements are shown in Table 2. The vitrification method showed significantly higher retention values when compared to the control group (no treatment) only for the groups cemented with Multilink. However, the vitrification and silicatization surface treatments were statistically similar for the Multilink cement. The factor "cement type" was not statistically significant.

Table 3 shows the comparisons between cement groups without conditioning of the framework inner surfaces. Bis-GMA- and HEMA-based resin cements showed the highest retention values. The other cements showed similar retention values.

Table 2: Mean ( $\pm$  Standard Deviation) of the Tensile Retention Data (kgf) for Different Resin Cement Groups<sup>a</sup>

Cements	Surface Treatment		
	Control	Silicatization	Vitrification
Multilink	20.5 $\pm$ (3.5) Aa	29.9 $\pm$ (13.1) ABa	38.8 $\pm$ (19.6) Ba
RelyX ARC	22.8 $\pm$ (5.3) Aa	25.2 $\pm$ (7.8) Aa	25.9 $\pm$ (5.3) Aa

<sup>a</sup> Different capital letters indicate a significant difference ( $p < 0.05$ ) between surface treatment types maintaining the same cement (line). Different lowercase letters indicate a significant difference ( $p < 0.05$ ) between the cement types maintaining the same surface treatment (column).

The failure analysis indicated that most of the groups had high percentages of cement remaining over the composite core preparation after the tensile testing, except for crowns cemented with Multilink (Figure 3), since most failures for those crowns were catastrophic. For the crowns cemented with a resin-modified glass ionomer (RelyX Luting), all specimens showed a higher percentage of cement on the crown side. Representative images for the failure analysis can be seen in Figure 4.

Representative SEM images of the conditioned Y-TZP surfaces are shown in Figure 5. Relevant changes were observed after the different surface treatments (Figure 5B,C) when compared to the untreated surface (Figure 5A). Pits and microretentions caused by selective etching with hydrofluoric acid can be seen on the glazed Y-TZP surface (Figure 5C).

**DISCUSSION**

The retention values obtained for both the chemically activated dual-cure resin cement (Multilink) and the BIS-GMA-based cement were not statistically different from each other. Thus, the first hypothesis of this study was accepted. Multilink Automix contains HEMA molecules, which possess a molecule that contains one hydroxyl radical, increasing the stability of the monomer under moist and acidic conditions.<sup>29</sup> Additionally, Multilink is chemically activated, which minimizes polymerization shrinkage due to the slower polymerization reaction.

Table 3: Mean ( $\pm$  Standard Deviation) of the Tensile Retention Data (kgf) for Different Resin Cement Groups<sup>a</sup>

Cements	Median
MultC	20.5 $\pm$ (3.5) A
RelC	22.8 $\pm$ (5.3) A
Ion	5.4 $\pm$ (5.1) B
Self	4.9 $\pm$ (4.2) B
Zinc	8.6 $\pm$ (7.5) B

<sup>a</sup> Different letters indicate a significant difference ( $p < 0.05$ ) among cement types.

It is important to note, though, that all three groups cemented with Multilink showed higher percentages of catastrophic failures, especially for group MultS. It may be concluded that the posts debonded from the intracanal dentin before the crown debonded from the preparation, indicating that the bond strength between the post and the dentin substrate was not as high as the one obtained between the cement and dentin. If the post had not debonded from the root canal, the retention values of these groups might have been higher. This observation is in line with the results obtained from Palacios and others,<sup>28</sup> who stated that the tensile retention values of some of the experimental groups were influenced by the cohesive failure of the zirconia coping and the teeth, thereby underestimating the real retention values. In the present investigation, specimens cemented with Multilink showed significantly more catastrophic failures than those cemented with RelyX ARC. Thus, it might be inferred that the Multilink cement could promote greater retention values if the posts had not debonded. According to Luthy and others,<sup>5</sup> RelyX ARC is a conventional dual-cure resin cement (based on BIS-GMA) that does not have any functional monomer in its composition, keeping it from developing any kind of chemical bonding to the crown or the core.

When considering the factor “surface treatment,” it was significant only for the Multilink cement, and

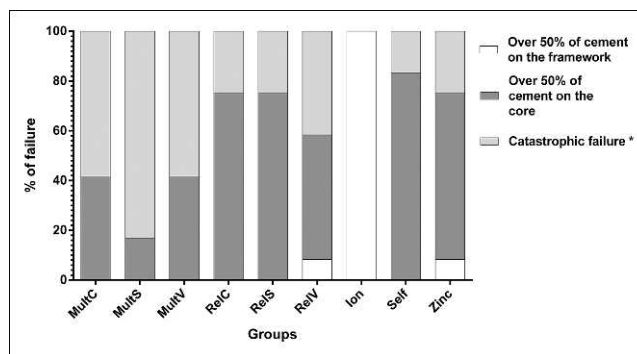


Figure 3. Percentages of types of failure for each cement. Catastrophic failure\*: post debonding.

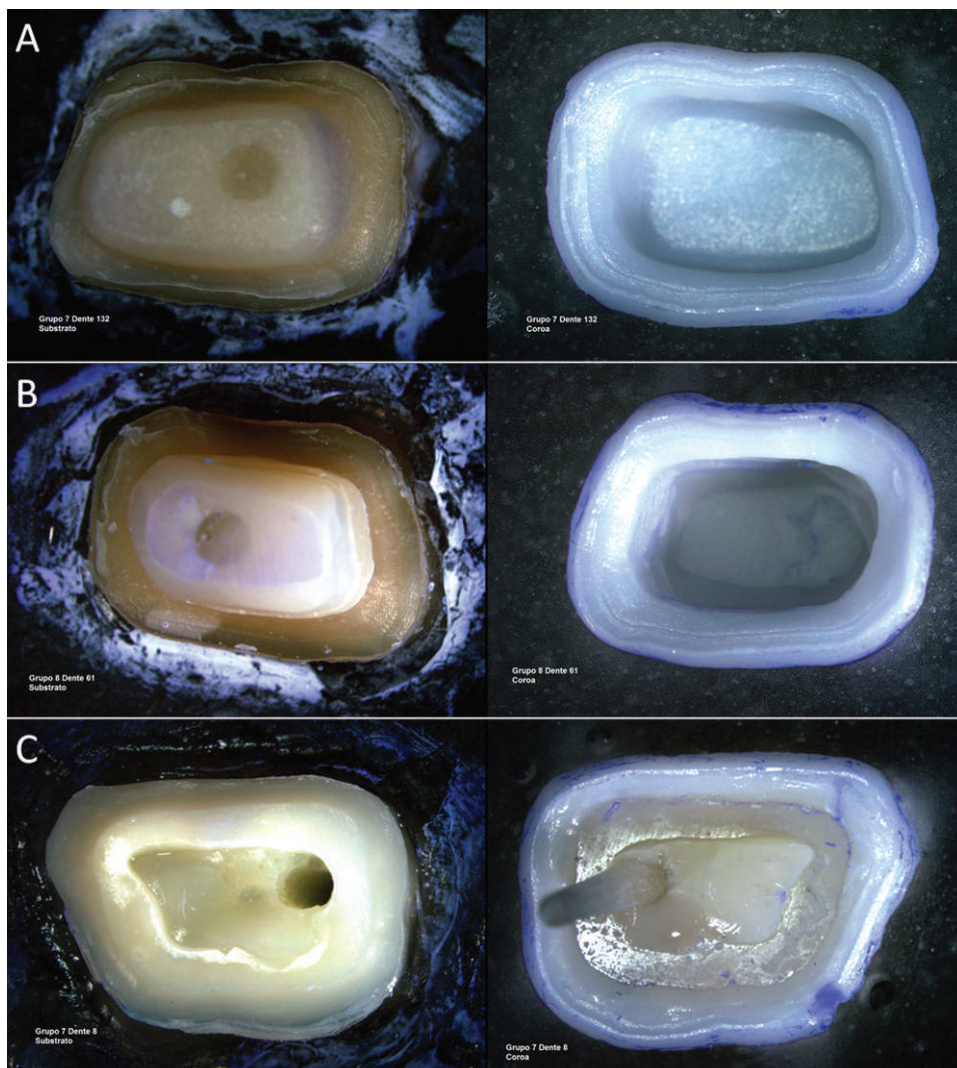


Figure 4. Photos from stereomicroscopy of the failure types. (A): Over 50% of cement on the substrate, (B): Over 50% of cement on the crown inner surface. (C): Catastrophic failure.

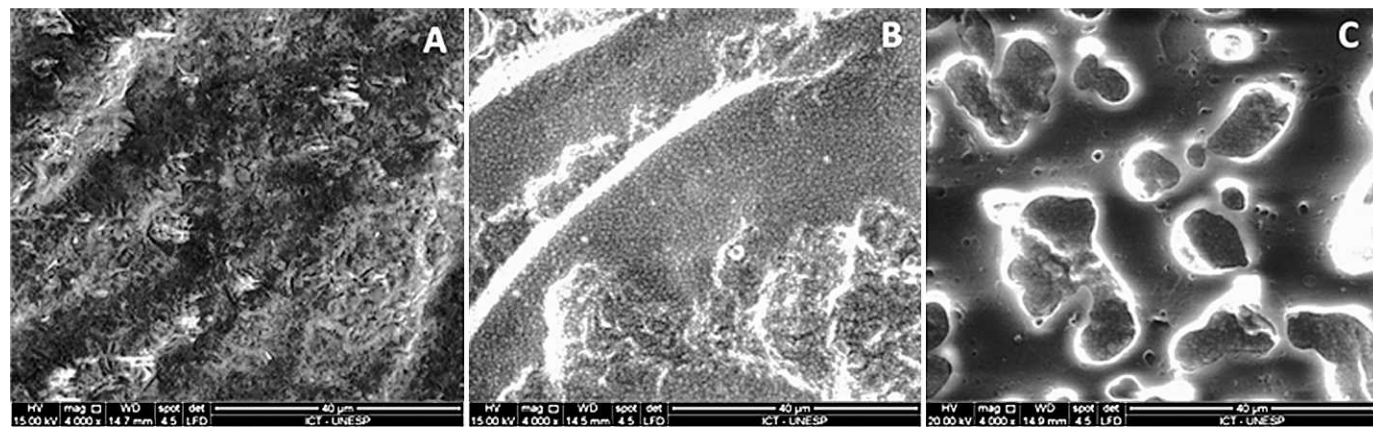


Figure 5. Representative SEM images of Y-TZP ceramic surfaces. (A): Zirconia without treatment. (B): Zirconia air abraded with 30-μm particles of aluminum oxide coated with silicon. (C): Glazed and etched zirconia surface.

therefore the second hypothesis was rejected. Silica coating did not increase the retention values of the crowns for the Multilink and RelyX ARC cements when compared to the control group. This fact may be related to the difficulty in applying the silica layer on the zirconia surface since Y-TZP has very high hardness and fracture toughness. Ntala and others<sup>21</sup> found that this surface treatment did not create the micromechanical retention required for efficient adhesive bonding, confirming the findings of the present study. Additionally, according to Borges and others<sup>30</sup> and Oyague and others,<sup>31</sup> airborne particle abrasion using 50  $\mu\text{m}$  of alumina oxide has little effect on the morphological surface features of zirconium dioxide ceramics and does not result in a deep surface modification. In the present study, smaller particles were used (30  $\mu\text{m}$ ), which might have prevented surface modification. Perhaps this is the reason why the group cemented with silica coating and Relyx ARC showed the highest amount of remaining cement on the core surface and not on the crown cementation surface. For the Multilink cement, most of the failures were catastrophic, indicating that this group (MultS) probably would show greater retention values if post debonding were avoided.

The vitrification process was efficient only for the Multilink cement, as it resulted in improved retention values when compared to the control groups. However, for the MultV group, most failures were either catastrophic or between the crown and cement with higher amounts of cement remaining on the core surface. As previously mentioned, if the specimens that fractured catastrophically could have been tested, the failures would probably have occurred between the crown and cement because no failures occurred between the core and the cement in the MultV group.

It is probable that the 10- $\mu\text{m}$ -thick<sup>12</sup> glass layer on the inner surface might have caused increased friction between the crown and the preparation walls, leading to higher retention values. In addition, Vanderlei and others<sup>12</sup> showed that one of the limitations of the vitrification technique is the difficulty in standardizing the glaze application inside the Y-TZP infrastructure since the glass layer applied on the intaglio surface of the Y-TZP infrastructure creates a layer thick enough to interfere with seating the infrastructure. Thus, technical improvements need to be tested for reducing the effect on the marginal adaptation.

When all cements without zirconia surface treatment were compared (Table 3), the groups that had

the highest retention values were the BIS-GMA- and HEMA-based cements. Thus, the third hypothesis of this study was accepted. Regarding RelyX U100, although its chemical composition containing methacrylated phosphoric esters has not been fully disclosed by the manufacturer, it has been shown that these monomers can bond to ceramic surfaces by means of the same mechanisms previously described for the monomer 10-MDP (also a methacrylated phosphoric acid ester).<sup>32</sup> Yap and others<sup>33</sup> reported that the bonding mechanism of RelyX Unicem is reminiscent of the self-adhesiveness of glass ionomer cements and that a possible improvement in bond strength may occur after cement maturation, over time. However, according to the failure analysis, which shows that the largest amount of cement remained attached in the core, it can be concluded that the bond failure occurred between the cement and the zirconia.

Manufacturers of the glass ionomer based-cement reported that, although this material has a chemical affinity for dentin hydroxyapatite, it has little affinity to the resin composites, which explains their low performance in terms of retention values and failure mode (between cement and composite resin core for all specimens).

In the present study, the manufacturers of the zinc phosphate cement (control group) still claim that this material can be used for cementation of zirconia crowns. It is interesting to note that this cement showed statistically similar retention values when compared to those obtained by the ionomer and self-adhesive resin cements. This retention behavior of the zinc-phosphate cements may be attributed only to the high friction coefficient between the crown and composite resin core walls, as this material does not bond to either the ceramic or the preparation substrate.<sup>34</sup>

One of the limitations of this study was the fact that it was not possible to measure the adhesive area of the preparation in order to calculate the nominal tensile retention stress for all of the ceramic crowns. However, it is believed that the retention force values (in kgf) used by this current investigation were reliable enough once samples were randomized and preparations were standardized. Palacios and others<sup>28</sup> compared the tensile strength (in MPa) and the load for crown pull-out (tensile retention in kg) and showed similar outcomes for both measurement units due to the homogeneity of preparation total areas. Those authors also calculated the average total preparation area for each experimental group



and showed that the adhesive areas were similar for the different teeth used.

The clinical relevance of this study is that it simulated different cementation protocols for Y-TZP crowns in an *in vitro* design, as a clinical crown cemented on a tooth reconstructed with resin composite core and a fiber post. Further studies should be conducted to investigate other factors involved in the retention of Y-TZP crowns, such as longitudinal fatigue testing, the evaluation of different cementation strategies, and other surface treatments for the inner surface of Y-TZP frameworks.

### CONCLUSION

- 1) The type of resin cement (BIS-GMA- or HEMA-based) did not affect the crown retention values.
- 2) The conditioning of the zirconia intaglio surface by the application of a thin low-fusing glass porcelain layer plus silanization was capable of improving the retention force for the HEMA-based cement.
- 3) For the untreated intaglio surfaces of Y-TZP crowns, resin cements showed significantly higher retention values when compared to those obtained for the self-adhesive resin cement, glass ionomer, and zinc phosphate cements.

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### Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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### REFERENCES

1. Piconi C, & Maccauro G (1999) Review: Zirconia as a ceramic biomaterial *Biomaterials* **20**(1) 1-25.
2. Christel P, Meunier A, Heller M, Torre JP, & Peille CN (1989) Mechanical properties and short-term in-vivo evaluation of yttrium-oxide-partially-stabilized zirconia *Journal of Biomedical Materials Research* **23**(1) 45-61.
3. De Aza AH, Chevalier J, Fantozzi G, Schehl M, & Torrecillas R (2002) Crack growth resistance of alumina, zirconia and zirconia toughened alumina ceramics for joint prostheses *Biomaterials* **23**(3) 937-945.
4. Sabrah AH, Cook NB, Luangruangrong P, Hara AT, & Bottino MC (2013) Full-contour Y-TZP ceramic surface roughness effect on synthetic hydroxyapatite wear *Dental Materials* **29**(6) 666-673.
5. Lüthy H, Loeffel O, & Hammerle CH (2006) Effect of thermocycling on bond strength of luting cements to zirconia ceramic *Dental Materials* **22**(2) 195-200.
6. Özcan M, Kerckdijk S, & Valandro LF (2008) Comparison of resin cement adhesion to Y TZP ceramic following manufacturers' instructions of the cements only *Clinical Oral Investigations* **12**(3) 279-282.
7. Casucci A, Monticelli F, Goracci C, Mazzitelli C, Cantoro A, Papacchini F, & Ferrari M (2011) Effect of surface pre-treatments on the zirconia ceramic-resin cement micro-tensile bond strength *Dental Materials* **27**(10) 1024-1030.
8. Blatz MB, Sadan A, Arch GH Jr, & Lang BR (2003) In vitro evaluation of long-term bonding of Procera AllCeram alumina restorations with a modified resin luting agent *Journal of Prosthetic Dentistry* **89**(4) 381-387.
9. Aboushelib MN, Matinlinna JP, Salameh Z, & Ounsi H (2008) Innovations in bonding to zirconia-based materials: Part I *Dental Materials* **24**(9) 1268-1272.
10. Piascik JR, Swift EJ, Thompson JY, Grego S, & Stoner BR (2009) Surface modification for enhanced silanation of zirconia ceramics *Dental Materials* **25**(9) 1116-1121.
11. May LG, Kelly JR, Bottino MA, & Hill T (2012) Effects of cement thickness and bonding on the failure loads of CAD/CAM ceramic crowns: Multi-physics FEA modeling and monotonic testing *Dental Materials* **28**(8) e99-e109.
12. Vanderlei A, Bottino M, & Valandro L (2013) Evaluation of resin bond strength to yttria-stabilized tetragonal zirconia and framework marginal fit: Comparison of different surface conditionings *Operative Dentistry* **39**(1) 50-63.
13. Aboushelib MN, Kleverlaan CJ, & Feilzer AJ (2007) Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials *Journal of Prosthetic Dentistry* **98**(5) 379-388.
14. Inokoshi M, Kameyama A, De Munck J, Minakuchi S, & Van Meerbeek B (2013) Durable bonding to mechanically and/or chemically pre-treated dental zirconia *Journal of Dentistry* **41**(2) 170-179.
15. Bottino MA, Valandro LF, Scotti R, & Buso L (2005) Effect of surface treatments on the resin bond to zirconium-based ceramic *Journal of Prosthetic Dentistry* **18**(1) 60-65.
16. Passos SP, May LG, Barca DC, Ozcan M, Bottino MA, & Valandro LF (2010) Adhesive quality of self-adhesive and conventional adhesive resin cement to Y-TZP ceramic before and after aging conditions *Operative Dentistry* **35**(6) 689-696.
17. Wegner SM, Gerdes W, & Kern M (2002) Effect of different artificial aging conditions on ceramic-composite bond strength *International Journal of Prosthodontics* **15**(3) 267-272.
18. Valandro LF, Ozcan M, Amaral R, Leite FP, & Bottino MA (2007) Microtensile bond strength of a resin cement to silica-coated and silanized In-Ceram Zirconia before and

- after aging *International Journal of Prosthodontics* **20(1)** 70-72.
19. Cattell MJ, Chadwick TC, Knowlesb JC, & Clarkec RL (2009) The development and testing of glaze materials for application to the fit surface of dental ceramic restorations *Dental Materials* **25(4)** 431-441.
  20. Kitayama S, Nikaido T, Maruoka R, Zhu L, Ikeda M, Watanabe A, Foxton RM, Miura H, & Tagami J (2009) Effect of an internal coating technique on tensile bond strengths of resin cements to zirconia ceramics *Dental Materials Journal* **28(4)** 446-453.
  21. Ntala P, Chen X, Niggli, & Cattell M (2010) Development and testing of multi-phase glazes for adhesive bonding to zirconia substrates *Journal of Dentistry* **38(10)** 773-781.
  22. Valentino TA, Borges GA, Borges LH, Platt JA, & Correr-Sobrinho L (2012) Influence of glazed zirconia on dual-cure luting agent bond strength *Operative Dentistry* **37(2)** 181-187.
  23. Bottino MA, Bergoli C, Guerra E, Salazar-Marrocho SM, Souza RO, & Valandro LF (2014) Bonding of y-tzp to dentin: Effects of surface conditioning, resin cement type, and aging *Operative Dentistry* **39(3)** 291-300.
  24. Bottino MA, Baldissara P, Valandro LF, Galhano GA, & Scotti R (2007) Effects of mechanical cycling on the bonding of zirconia and fiber posts to human root dentin *Journal of Adhesive Dentistry* **9(3)** 327-331.
  25. Swift EJ Jr, LeValley BD, & Boyer DB (1992) Evaluation of new methods for composite repair *Dental Materials* **8(6)** 362-365.
  26. Teixeira EC, Bayne SC, Thompson JY, Ritter AV, & Swift EJ (2005) Shear bond strength of self-etching bonding systems in combination with various composites used for repairing aged composites *Journal of Adhesive Dentistry* **7(2)** 159-164.
  27. Ernst CP, Cohnen U, Sternder E, & Willershausen B (2005) In vitro retentive strenght of zirconium oxide ceramic crowns using different luting agents *Journal of Prosthetic Dentistry* **93(6)** 551-558.
  28. Palacios RP, Johnson GH, Phillips KM, & Raigrodski AJ (2006) Retention of zirconium oxide ceramic crowns with three types of cement *Journal of Prosthetic Dentistry* **96(2)** 104-114.
  29. Mirmohammadi H, Aboushelib MNM, Salameh Z, Feilzera AJ, & Kleverlaan CJ (2010) Innovations in bonding to zirconia based ceramics: Part III. Phosphate monomer resin cements *Dental Materials* **26(8)** 786-792.
  30. Borges GA, Sophr AM, de Goes MF, Sobrinho LC, & Chan DC (2003) Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics *Journal of Prosthetic Dentistry* **89(5)** 479-488.
  31. Oyagüe RC, Monticelli F, Toledano M, Osorio E, Ferrari M, & Osorio R (2009) Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic *Dental Materials* **25(2)** 172-179.
  32. Miragaya L, Maia LC, Sabrosa CE, de Goes MF, & da Silva EM (2011) Evaluation of self-adhesive resin cement bond strength to yttria-stabilized zirconia ceramic (Y-TZP) using four surface treatments *Journal of Adhesive Dentistry* **13(5)** 473-480.
  33. Yap AUJ, Cheang PHN, & Chay PL (2002) Mechanical properties of two restorative reinforced glass-ionomer cements *Journal of Oral Rehabilitation* **29(7)** 682-688.
  34. Hill EE, & Lott J (2011) A clinically focused discussion of luting materials *Australian Dental Journal* **56(1)** 67-76.