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Original Article

## Bond Strenght of Zirconia Submitted to Different Surface Treatments

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### Abstract

**Objective:** To evaluate the shear bond strength of a recent adhesive system used in the cementation of zirconia. **Material and Methods:** Overall, 72 zirconia specimens (Zirconzhan) were divided and randomized into 3 groups according to the type of surface treatment: G1 no treatment + adhesive system; G2 blasted with aluminum oxide + adhesive system and G3 tribochemical treatment (Rocatec Plus) + adhesive system. Half of each group ( $n = 12$ ) had bond strength evaluated on two occasions: 24 hours after cementation, kept stored in distilled water at 37 °C without thermal cycling, and after thermal cycling (5000 cycles, 5°C-55°C). Data were analyzed by ANOVA with Tukey's post-test ( $\alpha \leq 0.05$ ). **Results:** At first moment, G1 and G3 showed higher bond strength ( $8.64 \pm 3.43$  MPa and  $6.55 \pm 2.27$  MPa) compared to G2, with no statistically significant difference between them. After thermal cycling, G3 showed higher bond strength ( $7.70 \pm 1.82$  MPa). **Conclusion:** Initially, only the adhesive system promoted higher bond strength, but after thermal cycling, bond strength decreased. The best treatment to promote high bond strength to zirconia is to associate tribochemical treatment with the adhesive system; most failures observed after thermal cycling were mixed and cohesive, showing a mechanical imbrication of the adhesive system, suggesting that there is no chemical bond; and the surface of the group with greater bond strength after thermal cycling showed more surface irregularities compared to the other groups.

**Keywords:** Ceramics; Air Abrasion; Materials Testing; Dentin-Bonding Agents.

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## Introduction

There are many materials in dentistry to restore teeth function and aesthetics in the oral cavity. For the rehabilitation of total crowns and anterior and posterior fixed partial dentures, there is the option of using zirconia, which is the common name given to zirconium dioxide ( $ZrO_2$ ). Zirconia crown has advantages over other types of materials used as infrastructure for these types of fillings, as it presents properties such as biocompatibility, high flexural strength (about 1,000 MPa), chemical stability and favorable optical characteristics [1-3].

One of the advantages of zirconia crowns compared to metal-ceramic crowns is esthetic, as the latter can, over long periods, show a dark halo in dental contouring, while zirconia is metal-free. Another advantage of zirconia compared to alumina and lithium disilicate crowns is its high hardness and greater strength to fracture [4].

In dentistry, zirconia used is that in the tetragonal crystalline phase, partially stabilized by yttria (Y-TZP), because pure zirconia is unstable for dental use [2]. One of the disadvantages of zirconia is degradation by aging at low temperatures and degradation in aqueous medium. Therefore, there is need for a binding agent strongly adhered to it when this material is cemented in the oral cavity [5]. In addition, due to its zirconia polycrystalline tetragonal structure stabilized by yttria, it becomes resistant to some acids used for cementation [6,7].

Several manufacturers and researchers have developed adhesives and ceramic surface conditioning techniques for cementation of zirconia restorations to provide cementation with high bond strength. It has been reported that the best bond strength provided in cementation between zirconia and cementing agents is obtained by cements with phosphate monomers in their composition [8].

The adhesive system used for zirconia cementation containing phosphate monomer has a monomeric bifunctional phosphate molecule, or a polymerizable organic chain that reacts with restorative materials; and a phosphate hydrophilic group that reacts with the zirconia surface. The most potent cements for zirconia cementation are based on Bis-GMA, 4-META methyl methacrylate or phosphate monomers [7].

Blasting with aluminum oxide and silica coating followed by silanization can be used to increase the bond strength of alumina ceramic infiltrated with glass and zirconia. This treatment with silica coated alumina particles creates a tribochemical effect on the ceramic surface [6,9,10].

These approaches of modifications on the zirconia surface using air abrasive particle such as tribochemical treatment followed by silanization or aluminum oxide particles followed by application of ceramic primer with a chemical functional group of phosphate monomer (MDP) increase cementing adhesive strength [7]. An adhesive system suitable for use in zirconia classified as a conventional 3-step containing MDP in its chemical composition has been recently launched on the market.

However, since there is still no consensus in literature about the best and most effective adhesive system to promote better bond strength when in contact with zirconia, the aim of this study

was to use this new adhesive system and modify the clinical steps recommended by the manufacturer, analyzing the results.

It is hoped that using only the adhesive system alone without the association with different surface treatments indicated for zirconia provide greater bond strength. It is still necessary to provide an efficient clinical protocol with sufficient bond strength for zirconia cementation which is safe for use.

This study aimed to evaluate the shear bond strength of an adhesive system with MDP with zirconia-based ceramic with different surface treatments; classify the fracture mode of cemented materials as adhesive, cohesive or mixed and assess the fracture surface morphology by scanning electron microscopy (SEM).

## **Material and Methods**

Specimens were manufactured according to ISO 10477 standards for ceramic testing.

### **Zirconia Specimen Preparation**

A model was prepared in Duralay acrylic resin (Reliance, Co Worth, IL, USA) from a PVC plastic matrix (polyvinyl chloride) with dimensions of 3 mm in diameter x 3 mm in height, which was reproduced in special Duroneplaster (Dentsply, Petrópolis, RJ, Brazil) for subsequent fabrication of zirconia discs.

Overall, 72 zirconia discs were made from the plaster model (3 mm in diameter x 3 mm in height) stabilized by yttria (Zirconzhan, Bousano, Italy) by the CAD-CAM system and sintered according to manufacturer's recommendations.

These zirconia discs were embedded in epoxy resin (Redelease, São Paulo, SP, Brazil), leaving only one free surface, which received finishing with abrasive sandpaper with granulation of 280 µm (3M Espe, São Paulo, Brazil), refrigerated with water in Politriz (Teclago, São Paulo, Brazil). After polishing, specimens were cleaned with distilled water in ultrasonic tank (Cristófoli, Paraná, Brazil) for 5 minutes at 70°C [9].

### **Preparation of the Composite Resin Disc**

Zirconia discs were molded with condensation silicone (Zetaplus, Labordental, São Paulo, Brazil), which served as a template for the preparation of composite resin discs. Overall, 72 composite resin discs with 3 mm in diameter x 3 mm in height, color A2 dentin (Charisma, Heraeus Kulzer, Henau, Germany) were incrementally prepared and light polymerized (Radii-cal, SDI, Victoria, Australia) for 40 seconds each increment at intensity of 1200 mW / cm<sup>2</sup>. These resin discs were cemented on the zirconia ceramic surface for subsequent shear test.

### **Zirconia Surface Treatments**

Specimens were randomly divided into 3 groups according to the surface treatment: G1 - without surface treatment; G2 - blasting with  $\text{Al}_2\text{O}_3$  aluminum oxide with abrasive particles of 110  $\mu\text{m}$  of size (Polidental, São Paulo, Brazil); G3 - tribochemical treatment, blasting with  $\text{Al}_2\text{O}_3$  aluminum oxide with abrasive particles of 110  $\mu\text{m}$  of size (Polidental, São Paulo) +  $\text{SiO}_2$  silica coating (Rocatec Plus, 3M Espe, MA, United States). In the blasting treatment of G2 and G3, specimens were fixed in a device of the blasting machine made at the University of São Paulo (USP), which launched  $\text{Al}_2\text{O}_3$  particles at a distance of 10 mm with pressure of 2.8 bars for 10 seconds and inclination of  $45^\circ$ , making horizontal and vertical movements. G3, in addition to blasting with  $\text{Al}_2\text{O}_3$ , also received silica coating.

Each group was divided into 2 subgroups ( $n = 12$ ) and half underwent shear after adhesive cementation after immersed in distilled water at  $37^\circ\text{C}$  for 24 hours. The other half was stored for 15 days also in distilled water at  $37^\circ\text{C}$  and then thermocycled for 5000 cycles in thermal baths at temperature from  $5^\circ\text{C}$  to  $55^\circ\text{C}$  ( $\pm 3$ ) with immersion time of 30 seconds and transfer time of 2 seconds (Table 1).

**Table 1. Distribution of groups according to surface treatment.**

Groups (n=12)		Surface treatment
G1	No treatment	No treatment
G2	Blasting with abrasive $\text{Al}_2\text{O}_3$ particles (110 $\mu\text{m}$ )	Blasting with abrasive $\text{Al}_2\text{O}_3$ particles (110 $\mu\text{m}$ )
G3	Blasting with abrasive $\text{Al}_2\text{O}_3$ particles + $\text{SiO}_2$ (Rocatec Plus) (110 $\mu\text{m}$ )	Blasting with abrasive $\text{Al}_2\text{O}_3$ particles + $\text{SiO}_2$ (Rocatec Plus) (110 $\mu\text{m}$ )

#### Cementing of the Composite Resin Disc on Zirconia Ceramics

After surface treatment, zirconia ceramic specimens received application of the Signum Zirconia Bond I + II adhesive system (Heraeus Kulzer, Hanau, Germany). Initially, the adhesive from flask Bond I was applied with microbrush and dried with a brief blast of air, then, Bond II was applied and light-polymerized for 40 seconds with the Radii-cal device.

Conditioning was conducted with 37% Condac phosphoric acid (FGM, SC, Brazil) for 30 seconds on resin discs, being washed at the same time and dried in paper towel. Then, Ed primer (liquid A) was mixed with (liquid B) of Panavia F 2.0 cement (Kuraray, Okayama, Japan) and applied to the composite resin surface. Then, the base paste was mixed with the catalyst paste of the Panavia F 2.0 cement (Kuraray, Okayama, Japan) with a paper pad and spatula for a time of 20 seconds, and placed on the zirconia surface. The disc that had the surface prepared with the adhesive system was placed on the cement and a 500 gram weight was placed on the zirconia / cement / resin set for 20 seconds, and the excess cement resin was removed with exploitative probe and microbrush. Light-polymerization was performed for 20 seconds on each side of the specimens immediately after the application of Oxyguard II from the Panavia F 2.0 kit (Kuraray, Okayama, Japan) for 3 minutes and then washed in running water.

After cementation of specimens, half were stored in distilled water at 37°C for 24 hours the other half was stored for 15 days and then submitted to thermal cycling.

### Shear Bond Strength Test

Samples were mounted in a universal tester machine (Instron, MA, USA). The constant loading force was applied at the crosshead at speed of 1.0 mm / min until fracture occurs. The load cell provided a force of up to 500 N until failure. The shear bond strength was calculated by dividing the maximum fracture load by the circular cementation area. Results were expressed in MPa.

### Analysis of Fracture Mode through Stereomicroscope and Scanning Electron Microscopy (SEM)

After shearing, the morphology of specimens was observed in stereomicroscope (Coleman) to count the fracture mode in adhesive, cohesive or mixed. The classification in the fracture mode was assigned as follows: adhesive (when there was less than 1/3 of the remaining cementing material), cohesive (when there was more than 2/3 of the remaining cementing material), mixed (there was more than 1/3 of the remaining cementing material and less than 2/3) [11].

After quantifying the failure of the union joint on the stereomicroscope, a sample from each randomly chosen group was submitted to scanning electron microscopy (SEM) (JOEL, JSM-638OLV, Japan). For SEM analysis, all fracture surfaces received gold sputtering by the Sputtering technique with the aid of the Denton Vacuum device (United States). The energy-dispersive X-ray spectroscopy (EDX) was used to determine the elemental composition of each surface of fractured specimens.

### Statistical Analysis

In this study, parametric statistical tests were performed, since data were submitted to the Shapiro-Wilk normality test. The shear bond strength was evaluated by using two-way ANOVA statistical test with Tukey's post-test and one-way ANOVA and Student's t test. Confidence interval of 95% was adopted ( $p = 0.05$ ).

## Results

**Table 2. Average shear values (MPa) and standard deviation ( $\pm$ ) of specimens.**

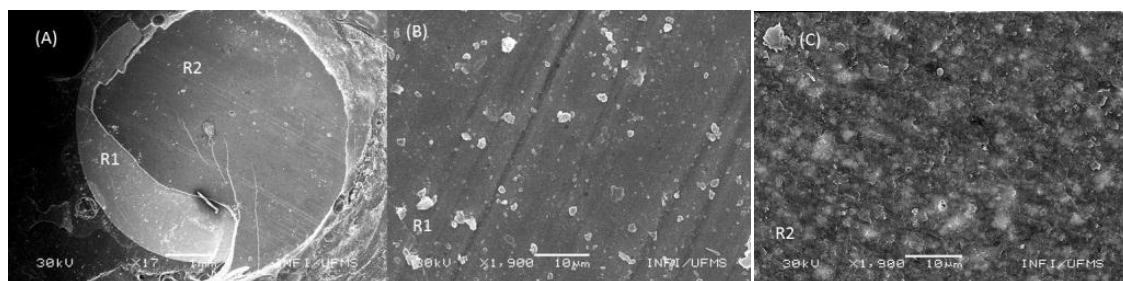
Time	Shear For Groups (N=12)		
	G1	G2	G3
No thermal cycling	8.64 $\pm$ 3.43 Aa	5.74 $\pm$ 1.32 Ba	6.55 $\pm$ 2.27 Aba
Thermocycled	5.22 $\pm$ 1.70 Bb	5.61 $\pm$ 1.89 Ba	7.70 $\pm$ 1.82 Aa

Different capital letters in the line indicate significant differences between groups in each moment (Tukey post-test). Lowercase letters in column indicate significant differences between moments in each group (Student's t test)  $p \leq 0.05$ .

SEM without thermocycling showed in the topographical image of sample from G1 a type of cohesive failure where the clear region shows zirconia (R1) and the dark region shows resin cement (R2). In EDX, the light region revealed that 90.53% of weight corresponds to zirconium; 5.69% to

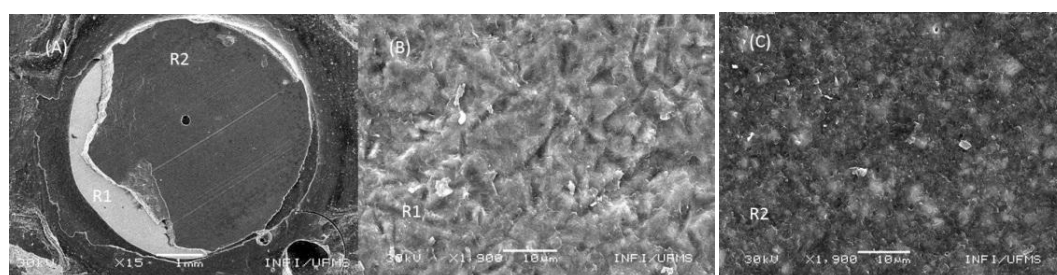


yttrium; 3.31% to gold and 0.37% to aluminum and in the dark region, 66.09% of weight corresponds to silicon; 23.95% to barium; 4.23% to gold; 2.34 to aluminum; 1.61% to zirconia and 2.34 to aluminum (Figure 1).



**Figure 1.** (A) Photomicrograph of specimen from G1 without thermal cycling showing cohesive failure; R1 zirconia; R2 resin cement (Magnification 17 x); (B) Photomicrograph of specimen from G1 showing the region of zirconia R1 (Magnification of 1900 x) without surface irregularities; (C) Photomicrograph of specimen from G1 Group showing the resin cement region R2 (Magnification 1900 x).

In G3, after thermocycling, a type of cohesive failure where the light region shows zirconia and the dark region shows resin cement was observed. On EDX, the light region revealed that 93.22% of weight corresponds to zirconium; 5.32% to yttrium and in the dark region, 63.02% corresponds to silicon and 25.15% to barium (Figure 2).



**Figure 2.** (A) Photomicrograph of thermocycled specimen from G3 Group showing cohesive failure; R1 zirconia; R2 resin cement (Magnification 15x); (B) Photomicrograph of specimen from group G3 showing the region of zirconia (R1), with depressions caused by the tribochemical treatment (Magnification 1,900 x); (C) Photomicrograph of specimen from G3 showing the region of the resin cement (R2), (Magnification 1,900 x).

The evaluation in percentage through stereomicroscope of all specimens from all groups and at all time intervals is shown below (Table 3).

**Table 3. Relative Frequency (%) of the type of fracture of specimens submitted to different surface treatments.**

Groups (n=12)	No thermal cycling			Thermocycled		
	Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed
G1	16.5	25	58.5	33.5	8	58.5
G2	0	33.5	66.5	0	0	100
G3	0	58.5	41.5	0	92	8

## Discussion

It is known that zirconia ceramic has high hardness and fracture resistance. However, it has limitations when used in dental prosthetic reconstructions for not having good adhesion [12]. For this reason, acid resistant ceramics such as zirconia need to optimize the union surface when combined to resin cements [13]. However, technique with air abrasive particles with alumina has been reported to increase imbrication when associated with silica coating in laboratory, which has recently been criticized for causing cracks inside the zirconia [14].

Several studies have confirmed that the use of MDP in the resin cement composition increases the bond adhesive strength [3,8,13,15]. However, this is still controversy in literature, as in 2012, some authors [9] reported that the use of MDP is not effective. Although primer with MDP increases the initial bond strength between zirconia and resin cement, there is information that the effect on the bond strength is limited [14].

However, this study evaluated the shear bond strength of an adhesive system containing MDP (Signum Zirconia Bond I + II) recently launched on the market, associated or not with different surface treatments. Surfaces that were blasted with the tribochemical system had higher resistance values after aging ( $7.70 \pm 1.82$  MPa). Both G1 (untreated surface) as G3 (blasting with  $\text{Al}_2\text{O}_3$  and silica), before aging, obtained the highest bond strength results ( $8.64 \pm 3.43$  MPa and  $6.55 \pm 2.27$  MPa), but with no significant difference between them.

From the beginning, the highest value shown by G1 is justified by having MDP in the chemical composition of the adhesive system and in the resin cement. MDP is responsible for promoting higher initial bond strength for cementing agents, although with no statistical difference in G3. However, after aging, groups that had blasted surface had become more stable and did not decrease the bond strength, which did not occur with the group that did not have blasted surface, i.e., irregularities on the surface caused by blasting increases the contact area of the adhesive system and promoted mechanical retention. This also occurs because under immersion in water, water diffuses into the interfacial layer of the composite resin and zirconia and causes hydrolytic degradation of the adhesive system. The combined effect of thermal cycling and hydrolytic degradation can promote a linear difference of the thermal expansion coefficient between the composite resin and zirconia, inducing a thermal stress in the interfacial layer, which causes adhesive breakage [13,16].

There is much controversy in literature regarding bond strength. There are reports that after thermal cycling, there is an increased bond strength between cementing agent and zirconia [17,18]. Moreover, it has been reported that the adhesion values remained unchanged after thermal cycling [19]. Adhesives that had no MDP in their composition showed decreased values after thermal cycling [20]. It was observed that specimens became loose with thermal cycling and that Panavia F 2.0 showed higher bond strength value under dry conditions [21]. The metal / zirconia primer improves bond strength only in initial condition and that after thermal cycling, all specimens became loose [22]. In a survey, the group without surface treatment, several samples became loose after thermal cycling, but tribochemical treatment enhances the bond strength [6]. Specimens



without surface treatment became loose in storage in water after 90 days; however, silica coating after immersion in water increased its bond strength [23]. In tensile testing, it is also reported that tribochemical treatments provide durable bond strength [15], however, adhesive systems cannot provide clinically acceptable bond strength [24].

There are studies in literature in which researchers used the same adhesive system of the present study (Signum Bond Zirconia). It was observed that Signum has higher shear bond strength value ( $54.9 \text{ MPa} \pm 9.4 \text{ MPa}$ ) compared to other adhesive systems [25]. However, the specimen size was different from that of this study, where specimen had diameter of 6 mm while in this work, specimen had diameter of 3 mm. Then, the adhered area was very different; in their study thermal cycling was not performed, causing distrust in the durability of the adhesive retention values. The authors also claimed that Signum Zirconia Bond has and acetone, MDP and methyl methacrylate in its composition and that acetone improves air contamination on the ceramic surface and can increase the resistance between zirconia and cement. The adhesiveness of Signum Zirconia Bond I + II investigated and it was concluded that Signum promotes similar bond strength (8.98 MPa), when compared to treatments of surfaces prepared with tribochemical treatment (CoJet System) (7.80 MPa) [26]. However, the authors did not carry out thermal cycling and the specimen size was different from that of this study. It is necessary to know the bond strength over time, for immediately, adhesion values are always higher.

The comparison of this work with others becomes impossible because there is no standardization of studies in relation to the size of specimens, type of mechanical trial performed and number of cycles performed during thermal cycling. However, this study followed the ISO (International Standard Organization) 10477. This standard establishes that the minimum bond strength value to avoid being disapproved should be 5.0 MPa. The adhesion values obtained in this study in various groups were close to this value. However, these are still low to keep zirconia cemented in the oral cavity for a long period. It was also observed in this research that for bond strength values of 5.0 MPa when zirconia is cemented in the oral cavity, it remains adhered on average 2-3 years because there is an adhesive degradation and prosthetic piece became loose [17]. Literature is extremely conflicting regarding the bond strength between zirconia and cementing agents.

The manufacturer of the Signum Zirconia Bond I + II adhesive system recommends that one should only clean and keep clean the zirconium dioxide surface for application of the adhesive system. In this study, group without surface treatment had its ceramic surface prepared according to these instructions. The other groups had their surfaces blasted.

The highest bond strength obtained in this work was the group that had surface blasted with aluminum oxide and silica coating, and after aging, it remained stable, with a statistically significant difference with respect to other groups. This is due to the fact that air abrasion carried out with aluminum oxide particles and silica coating (tribochemical system) on the zirconia surface promoted chemical bonding between the ceramic surface and the resin compound [10].

The adhesive system and resin cement used in this study contain phosphate monomer in their composition. The phosphate ester functional group in the MDP molecule promotes adhesion on the zirconia surface.

The structural formula contained in the MDP monomer is composed of two functional groups: a di-valent phosphoryl group and a methacrylate group. The di-valent phosphoryl group is absorbed within the zirconia, while the methacrylate group can copolymerize with other monomer groups in the adhesive system [27].

Some reactions may occur between zirconium dioxide and phosphate ester monomer. Primers or resin cements containing MDP can increase the bond strength in zirconia ceramics. It is believed that the monomer has adhesiveness capacity in the chemical form with metallic oxides through the Van der Waals force or hydrogen bonds in the resin zirconia interface. This interfacial force can improve wettability on the zirconia surface and increase bond strength with the resin component with low percentage of adhesive failure on the ceramic surface. Perhaps this may be the explanation for the chemical reaction of the zirconia surface with the MDP component, with this reaction site, and these sites are limited, which means that the chemical effect has an upper limit that cannot be overcome [15,28]. Also in this work, the group without surface treatment dramatically decreased the bond values when submitted to thermal cycling probably because the sorption of water must have caused the degradation of adhesive interface during aging [6].

There are claims that after thermal cycling, there is increased bond strength due to accelerated post-polymerization by the permanence of specimens at temperature of 55°C [17].

Typical silane bonding agents, more specifically trialkoxysilanes, are inorganic and organic bifunctional molecules and play a remarkable role as an adhesion agent. Typical silane bonding agent contains an organo functional portion and three hydrolysable alkoxy groups. Before being activated, trialkoxysilanes must undergo a slightly acidic water hydrolysis reaction and ethanol solvent to form silanols of trialkoxy group. The organo functional part, most often a methacrylate group, can then be polymerized with composite resin monomers. The silane bonding agents decrease the surface tension of a substrate improves wettability and increases the surface energy, making adhesion more effective. Thus, a hydrophobic matrix (composite resin) can adhere to hydrophilic surfaces, such as silica, glass and glass ceramic. The monomeric ends react with methacrylates of adhesive resins by a free radical process (conventional). Silanes are not as effective with high bond strength in ceramics like alumina and zirconia. These ceramics are chemically more stable than silica contained in glass ceramics is not easily hydrolyzed [25].

It has been reported [29] that specimens blasted with aluminum oxide particles in various pressures increase the adhesive strength, which is kept durable between the zirconia and an indirect composite. They also reported that the surface roughness increases and chemically activates the adhesive surface, removing organic contaminants from the ceramic surface.

Through SEM, it was found that the group that received no surface treatment was more leveled and free from irregularities; the surface blasted with aluminum oxide had little irregularity,

but the surface that received tribochemical treatment has become quite irregular. These analyses show that mechanically, blasted surfaces have more retentive areas for bonding between resin cement and zirconia.

After thermal cycling, higher amount of mixed and cohesive failure was observed in blasted groups and lower amount of adhesive fracture compared to the group that had no surface treatment. This occurred because G1 was not submitted to mechanical retention by the blasting process. All groups analyzed through scanning electron microscopy showed resin cement on the surface of specimens, except for G2.

Analysis of fracture mode shows that regardless of moment (before and after thermal cycling), blasting improved the stability of the bond strength, i.e., groups submitted to blasting showed no significant difference with regard to adhesion when thermocycled or not; while groups not submitted to blasting showed a significant difference.

Zirconia is an extremely aesthetic material; however, there are no surveys reporting its stability in the oral cavity for a long period of time and results indicating the best technique and method of fixation. However, the dentist must be careful when choosing the best restorative material.

The hypothesis of this research was to evaluate the bond strength of the adhesive system applied on the zirconia surface that received no surface treatment when compared to groups receiving different types of surface treatments; but the hypothesis has been rejected.

The clinical relevance of this study showed that after thermal cycling, the best technique for cementation was tribochemical treatment in zirconia (blasting with aluminum oxide and silica coating associated with the adhesive system).

An ideal situation would be if only the application of an adhesive system would result in stable bond strength without damage to the surface without the need of air abrasion and surface treatments that do not cause cracks to the zirconia surface, causing a subsequent fracture to the work piece. Techniques of spraying zirconium dioxide to increase the mechanical retention appear to be interesting [30], in addition to techniques of glass impaction on the surface to promote higher bond strength [12].

There should be a clinical protocol that enables security for professional to use zirconia as an aesthetic material for rehabilitation of dental crowns. While literature does not yet provide a consistent basis for implementation of effective and safe procedures, zirconia has limited indication or is even contraindicated for use in dentistry.

Future studies are needed to determine whether the actual chemical effect of the silica coating on the retentive effect on the rough surface produced, and other bonding agents such as silane, adhesives with primer metal must be investigated in chemical interaction with zirconia.

## Conclusion

Initially, only the adhesive system promoted greater bond strength, but after thermal cycling, bond strength decreased. The best treatment to promote greater bond strength to zirconia is to associate tribochemical treatment with the adhesive system; most failures observed after thermal cycling were mixed and cohesive, suggesting the presence of mechanical imbrication of the adhesive system, indicating that there is no chemical bond, and the surface of the group with greater bond strength, after thermal cycling, showed more surface irregularities compared to the other groups.

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