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Research Article

Influence of Surface Treatment on Adhesion to Zirconia

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Abstract

Objectives: To evaluate the influence of different treatments of surface on the bond strength to Zirconia.

Materials and Methods: Thirty cylinders of Y-TZP (4 mm X 5 mm) were made by CAD/CAM system and thirty resin cylinders were prepared in the same dimensions and cemented to Zirconia according the groups (n = 10): G1 (Blasting+Cement), G2 (Blasting+Primer+Cement), G3 (Blasting+adhesive+cement). The samples were stored in distilled water (37ºC, 60 days) and thermocycled (5000 cycles, 5ºC-55ºC), where they were submitted on shearing test (SBS).

Results: The data were analysed by Exact test of Fisher and Kruskal-Wallis. There were significantly statistical difference between the groups, G3 presented the highest bond strength. Failure mode analysis were performed by the binocular stereomicroscopy (40X) and observed in SEM, where adhesive failure was predominant.

Conclusion: The use of universal adhesive with MDP associated to silane potentiated the adhesive strength to Y-TZP zirconia.

Keywords: *10-MDP cpd; Dental Air Abrasion; Resin Cements; Shear Strength; Y-TZP Ceramic*

Abbreviations

10-MDP: 10-methacryloyloxydecyl Dihydrogen Phosphate; Bis-GMA: Bisphenol A-diglycidyl Ether Dimethacrylate; HEMA: Hydroxyethyl Methacrylate; 3-MPS: 3-Trimethoxysilylpropyl Methacrylate; TEGMA: Triethylene Glycol Dimethacrylate; UDMA: Dimethacrylate Urethane; Bis-EMA: Bisphenol A-Polyethylene Glycol Diethyl Methacrylate

Introduction

Clinical studies showes a good performance from treng to a long term of the restaurations-based-zirconia [1-5] for their upper mechanical properties [6-8] and na strengths biocompability [1-5,9]. The polycrystalline ceramics of tetragonal zirconia stabilized with yttrium (Y-TZP) may be used how na alternative to conventional metallic structures, but it has a restriction due the strengh and a strengh phase, interfering on its adhesion to cementing strengh and limmiting the efficacy of the adhesive cement processing [9,10], turning into strength clinical problem [3,11].

Considering that the oxides-zirconias needs to have the bonding surface optimized, when bonded to resin cements [12], several clinical protocols have been proposing to have na effective and a stable bond between Y-TZP and resin cements, but there is no consensus about the better method of surperficial treatment to reach na great bond [13,14].

Several techniques of superficialis pretreatment are practiced to improve the bond strength of the resin cement bonding to the intern surface of zirconia [15-18], for example, lasers irradiation of Er:YAG, Nd:YAG or of CO_2, conditioning technique of selective infiltration, blasting of alluminium oxide of strength particle sizes before and after the sintering and blasting with alumina-silica before silanization of surface [11,13,17,19-27]. All of these methods pretend to improve the mechanical bond through the increase of surface roughness [10], it is not just to become rougher, but also to activate chemically the zirconia, thus becoming, more receptive to the chemical bond through silanization [18]. However, some of those treatments were ineffective, and in so many cases, they may cause damage to the surface [28]. In chemical pretreatment, the exclusive application of traditional ceramic primers are not effective in zirconia [18,29,30], while the application of primers which contains MDP (10-methacryloxydecyl dihydrogen phosphate) have been documented obtaining chemical bonding, specially when it is applicated in Y-TZP previously blasted [14,31-33]. Through the combination of this functional monomer MDP with a bifunctional monomer of silan, a specie of "universal" primer could be apllicable to the strengths types of ceramics (with or without strengh phases) [34], promoting a chemical bond and pottentially creating a long-lasting link of bond to zirconia [16,35-38].

Aim of the Study

The aim of the study was avaluating the influence of strength protocols of surface treatment in bonding strength to Y-TZP, at efficacy of bonding and analysis of fracture mode. The hipothesis of nullity is that the use of na universal adhesive containing 10-MDP associated to the silan, does not potentiate the bond to Y-TZP.

Materials and Methods

Specimen preparation

The materials used in the present study are described into the table 1. Thirty cylinders of presintered (Ceramill Zi, Amann Girrbach AG, Koblach, Austria) were confectioned by CAD/CAM (Ceramill Zi), measuring 4 mm of diameter and 5 mm in height and sintered at a temperature of 1450ºC, where have been embedded into PVC cylinders (25 mm of diameter X 20 mm of lumen diameter and 5 cm of height) using colorless uncolor resin (Redelease, São Paulo, SP, Brasil). After the inclusion, cylinders were abrasioned with aluminum oxide strips (Norton Saint-Gobain, Guarulhos, SP, Brazil) in granulations #150 and #280, on politrix PLR II (Risitec, São Paulo, SP, Brazil), to simulate the superficial roughness caused by strength process in the confection of zirconia restorations and, to debris removal, were placed in a strength washer (Odontobrás 1440 DA, Ribeirão Preto, SP, Brasil).

Material Product Name	Lot	Composition	Manufacturer
Zirconia blocks eramill ZI(CZ)	00S304315B	Stabilized zirconium oxide with 3 mol% of yttrium (ZrO_2-3%Y_2O_3)	Amann Girrbach AG Koblach, Austria
Resin cement Panavia F 2.0 PNV)	Pasta A 9V0114 Pasta B 9R0026	Pasta A: Hydrophobic and hydrophilic dimethacrylates, 10-MDP, Colloidal, sílica, Silanized sílica, Camphorquinone, benzoyl peroxide. Pasta B: Hydrophobic and hydrophilic dimethacrylates, Sinalized barium glass, Silanized titanium oxide, Sodium fluoride, Colloidal sílica, Sodium sulfinate benzene salt 2, 4, 6 Triisopropyl, n, n-diethanol p-toluidine and pigments.	Kuraray Noritake Dental Kurashiki, Okayama, Japan
Adhesive Scotchbond ™ Universal (SBU)	582957	Bis-GMA, Organophospate monomer (MDP), Vitrebond™ Copolymer, HEMA, Ethanol, Water, Charge particles, Silane, Initiators.	3M ESPE Dental Products, St Paul, MN, EUA
Particle abrasive Aluminum Oxide (50µ- Al_2O_3)	42883	Al_2O_3 particles (50µm)	Bio-art Equipamentos Odontológios, São Carlos, SP, Brazil
Primer ceramic Clearfil Ceramic Primer (CCP)	240009	3-MPS, 10-MDP, Ethanol	Kuraray Noritake Dental Kurashiki, Okayama, Japan
Composite resin Filtek™ Z350 XT (Z350)	455134 564691	Organic part: Bis-GMA, TEGDMA, UDMA and Bis-EMA. Inorganic part: charge particles (Zirconia/Silica 60% - 0.01 to 3.5 micrometers)	3M ESPE Dental Products, St Paul, MN, EUA

***Table 1:** Description, composition, manufacturer and lot of materials used in this study.*

The specimens were randomly dividing in 03 groups (n = 10), according to the study design (Figure 1). The blasting with particles of 50, µm-Al_2O_3 (Bio-art Equipamentos Odontológicos, São Carlos, SP. Brazil), was realized in a distance of 10 mm for 15 seconds to a pression of 3,92-5,39 bars.

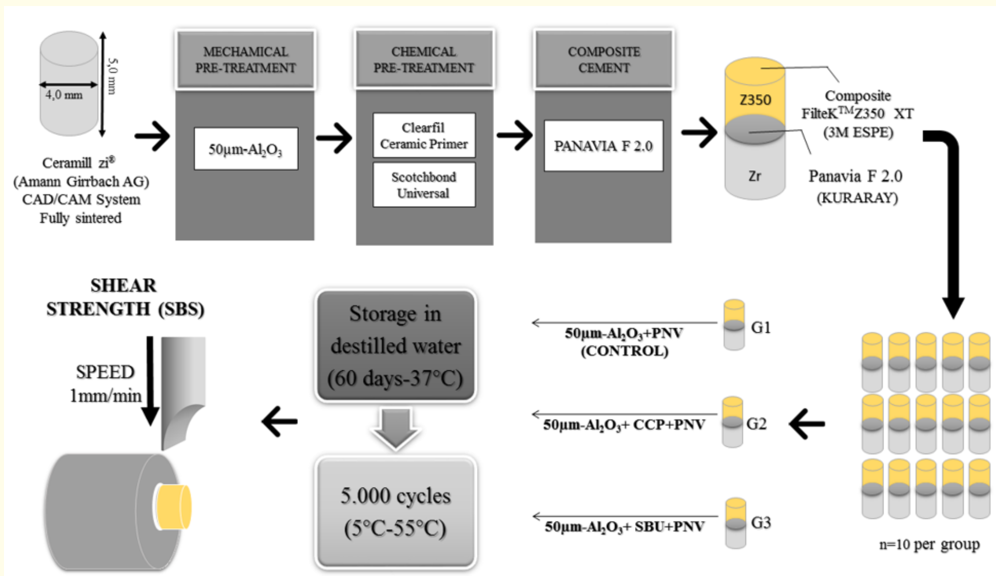

Figure 1: *Study design.*

Cementing of the resin cylinders

Cylinders of resin were confectioned in the same dimensions of Y-TZP cylinders, they have been cemented under a pression of 500 grams for 20 seconds, to avoid the bubble formation and the excesses of cement were carefully removed. Before the light curing, a water-soluble Oxyguard II was applied to avoid the no light curing of the superficial cement layer, by the exposure to the air. After this strength on, the set was light cured for 40 seconds each side in a light intensity of 1200 mW/cm^2 with the light curing unity Radii-Cal (SDI, Bayswater, Victoria, Australia).

Shear Bond strength

The experimental groups were stored in distilled water at 37ºC for 60 days and thermocycled for 5000 cycles (5ºC - 55ºC with 30 seconds of immersion time). The specimens were fixed in an appropriate device and taken to the universal test machine (EMIC, São José dos Pinhais, PR, Brazil), at a speed of 1,0 mm/min until to occur the fracture.

Analysis of fracture mode

The fracture mode was evaluated by a stereomicroscopy (Q744S, QUIMIS, Diadema, SP, Brazil) with an increase of 40X, where it has been classificated in: adhesive failure (Cement/zirconia interface); Cohesive failure (Failure in Zirconia or resin) and mixing failure (combination of adhesive and cohesive failure). A representative sample of each mode of fracture was observed by a SEM (JOEL, JSM-6460-Japan) (Figures 2-4).

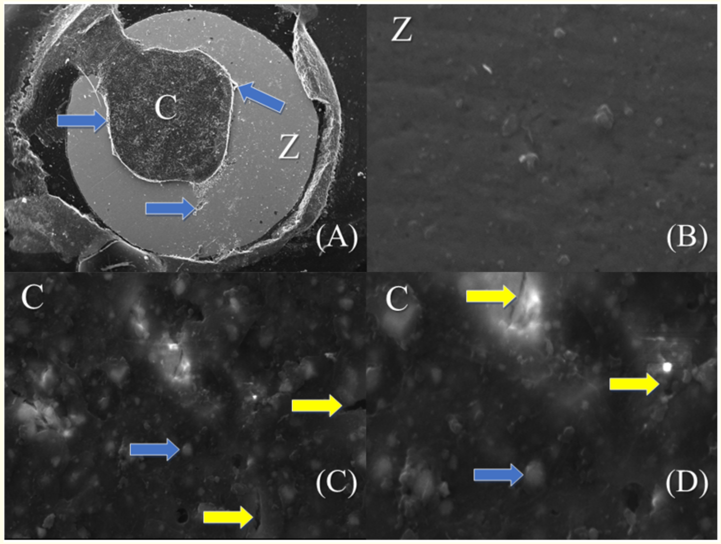

Figure 2: *(A) Photomicrograph of a test specimen showing an adhesive fracture, Z - zirconia (lighter area); C - resin cement (Darker area), the blue arrows shows the areas of cement fracture (Increasing of 22x); (B) Photomicrograph showing the zirconia area (Z); (C) e (D) Area of PNV cement with an increasing of 1000X and 2000X, respectively. Yellow arrows stand out areas of fracture into own cement and blue arrows reveal charge particles of the cement.*

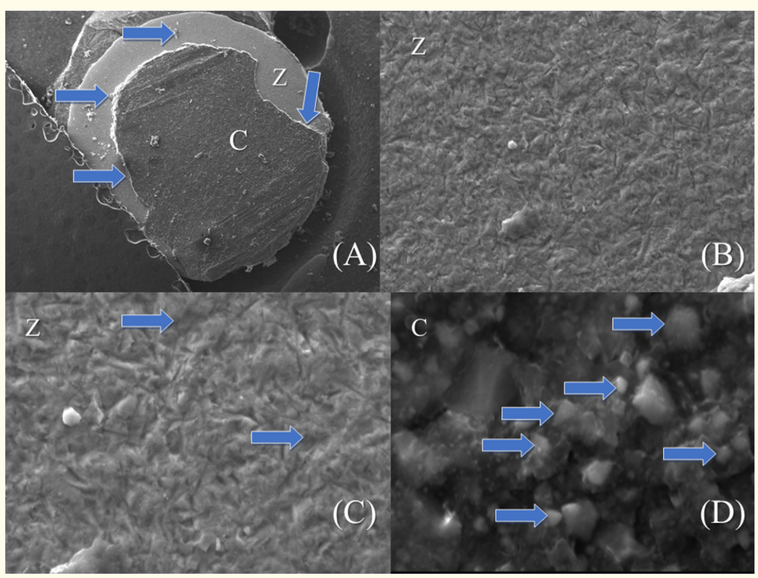

Figure 3: *(A) Photomicrograph of a test specimen showing cohesive fracture, Z - zirconia (lighter area); C - resin cement (darker area), blue arrows shows the areas of cement fracture (increasing of 22x); In the images, (B) and (C) we can verify the surface irregularity of zirconia caused by particles blasting of 50μ- Al2O3 with an increase of 500X e 1000X, respectively; (D) showing the area of cement C. The blue arrow shows charge particle of several sizes of cement PNV.*

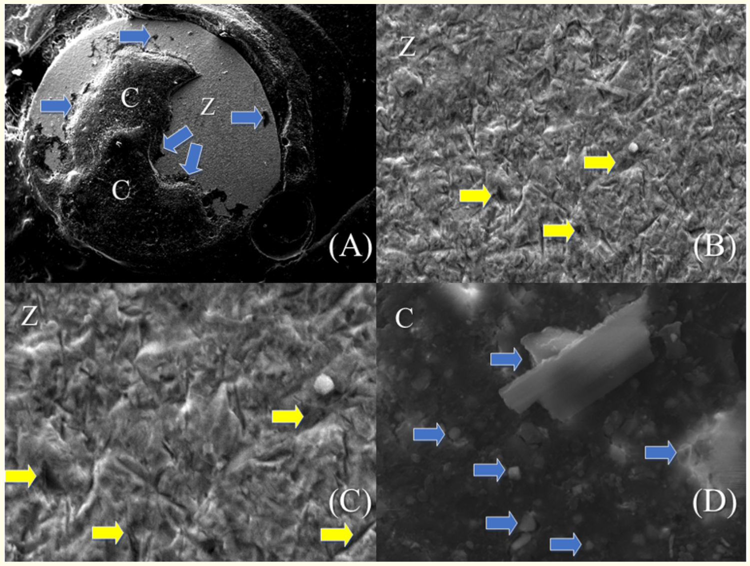

Figure 4: *(A) Photomicrograph of the SEM detailing a mix of fracture, Z - Zirconia (lighter area); C - resin cement (darker area), blue arrows shows areas or cement fracture (increasing of 22x); (B) and (C) shows the zirconia area (Z), with irregularities formation (yellow surface) caused by the blasting with aluminum oxide, with an increase of 500X and 1000X, respectively; (D) resin cement area. Blue arrows shows the charge particles of several sizes and forms of cement PNV (increasing of 1000x).*

Statistical analysis

The groups were analysed by Fisher and Kruskal-Wallis Exact Test for the strength variable. The statistical calculus were realized by the program Statistical Package for the Social Sciences (IBM SPSS Statistics 22, IBM Corp, Armonk, NY, EUA).

Results

Results to the bond strength by shearing

The results to the bond strength by shearing have been showed on table 2. It was verified significative difference between groups ($p < 0,001$).

Fracture mode classification

The predominant failure type was the adhesive one.

Discussion

Referring to this study, the hypothesis of nullity was rejected, the results showed that those different superficial treatment protocols influenced in bond strength. The use of a universal adhesive system associated with a silane significantly increased bond strength. This upper behaviour may be explained due the use of 10-MDP (10-meth-cryloyloxydecyl dihydrogen phosphate) in its composition. This bifunctional monomer bonds to the extremity of ceramic metal oxides and also to the cement resin matrix, as a coupler of silane to create ties with SiO_2 with bases in ceramic increasing significantly the bond strength. These results corroborate with the findings of Koizumi40, where it was observed that the apply of cement agents, primers and adhesive systems containing MDP provided better bond strength to the zirconia than others systems, although, it was observed on this study that the universal adhesive is upper than the ceramic primer when it is about the promotion of a safer adhesion between Y-TZP and resin cement.

The improvement of bond strength using the adhesive is referring to the better surface wetting, making possible its flow and in that way, it has a great nearness with zirconia, generating a better mechanical locking [17]. The superiority of bonding strength was observed on this study in the group G3, it was not just referring the improvement in the surface wetting produced by the adhesive, but also, by the capacity to bond the 10-MDP to metal oxides creating, thus, a physico-chemical adhesion for the set.

At adhesive evolution, a new universal system containing an acid monomer (MDP) was incorporated in the treatment of both substrates, ceramic and dentin, then forming a zirconia-resin-dentin complex. Independently of the reached improvement with the initiators MDP, the adhesions are still susceptible to hydrolytic degradation. MDP is many times provided how a ceramic starter which contains a polymerizable monomer, a suitable solve and silane. Primers containing MDP and silane shows enhanced adhesion in resin-zirconia, but there is no consensus about their good work without silane agents. In particular, some researchers defended the coating of silica and applying of silane on their use in the surface treatment [14,16,31-33,35-38].

However, traditional silane agents such as γ-methacryloxypropyltrimethoxysilane (γ-MPS) are not truly effective with zirconium-based ceramics such as zirconium dioxide, which it has a relatively non-polar surface and is chemically more stable than ceramic based on silica [17].

Due to the lack of silica in the Y-TZP, effective chemical bonds may not be generated with the resin. A meta-analysis reported that silanization for silica-coated Y-TZP promoted a hydrolysis resistant bond. However, the effects of silane, when used in zirconia need to be further evaluated [17]. There must be a well-established clinical protocol. The literature does not yet provide a consistent basis for effective procedures, allowing safety to use Y-TZP as a cosmetic restorative material for dental crowns.

Currently, several vitreous or non-vitreous ceramic initiators are composed of MPS and MDP. In the presence of an acid initiator or MDP-containing resin cement, the silane can hydrolyse, improving chemical adhesion with the silica-based ceramic, increasing water strength. However, it is questioned the synergistic effect of mixing these two primers in the surface treatment of zirconia, requiring more laboratorial and clinical studies of bond stability in degrading means.

It is important to abrasion with aluminum oxide particles to increase the adhesive strength and maintain the bond between the zirconia and the cementing agent durable. The surface roughness increases, besides removing contaminants, favoring the chemical adhesion on the ceramic surface [41]. In this study, the influence of ceramic surface abrasion has not been proven, but there is evidence that it is essential to increase the adhesive strength between the cementing agent and Y-TZP. Although the use of different surface treatments, the analysis of the fracture mode shows the predominance of the adhesive fracture [42-45].

One of the limitations of this study is that the specimens suffered only thermal aging, however other factors are related to the stress dynamics that these materials would suffer in the oral cavity, among these factors the mechanical stresses and chemical corrosion occurring in the oral cavity.

When we try to compare this study with others we need attention because there is no standardization in the size of the specimens, type of mechanical test and the number of cycles to be performed during thermocycling. The clinical relevance of this study shows that among the three protocols used, the best performance for cementing Y-TZP ceramics is blasting with a universal adhesive containing MDP associated with silane.

Conclusion

By the obtained results about the study, it may conclude that the use of universal adhesive system potentiated the bonding strength to Y-TZP.

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Conflict of Interest

The authors declare no conflict of interest.

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