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ALUMINUM OXIDE BLASTING AS SURFACE TREATMENT OF TITANIUM AND ZIRCONIUM OXIDE

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ABSTRACT

Aim: In this study we assess the shear bond strength of two resin cements to Grade 5 Titanium (Ti) surface and yttrium-stabilized zirconium oxide ceramic (Zr) surface, with and without surface treatment. Cements tested are RelyX U200 (3M ESPE, Seefeld, Germany) and MultilinkSistem Pack (IvoclarVivadent, Schann, Lichenstein).

Materials and methods: We tested eight groups (n = 10): G 1= Zr treated U200; G 2= Zr untreated U200; G 3= Zr treated Multilink; G 4= Zr untreated Multilink; G 5= Ti treated U200; G 6= Ti untreated U200; G 7= Ti treated Multilink; G 8= Ti untreated Multilink. Samples were either untreated or primed with aluminum oxide blasting and cemented. They were subjected to 5000 cycles of thermal cycling and to shear testing. Bond strength values were analyzed with three criteria ANOVA and Tukey's test.

Results: Titanium samples with surface treatment showed significantly higher bond strength in comparison with zirconia ceramics, regardless of cement type.

Conclusion: Surface treatment with aluminum oxide blasting was efficient to increase cement bond strengthto titanium, and ineffective on zirconia surface.

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INTRODUCTION

Yttrium-stabilized zirconium oxide ceramic has been widely used in dentistry due to its desirable optical properties, biocompatibility, low thermal conductivity, chemical stability, as well as its high resistance to fracture and good mechanical performance in comparison to other dental ceramics (Kern, 1994; Wolfart *et al*. 2007 and Mirmohammadi *et al*., 2010). However, it degrades in the presence of humidity, which represents a great disadvantage (Belo, 2013; El-Ghany, 2016 and Tzanakakis, 2016). Titanium has always been one of the materials of choice for prosthetic restorations, and along with Co-Cr and Ni-Cr alloys, are commonly used as crowns frameworks (Al Jabbari, 2014). Clinical success of metal-free ceramics is still highly dependent on cementation (El-Ghany, 2016; Inokoshi, 2014; Papia, 2014 and Hallmann, 2016). Composition and microstructure of ceramics associated to physical and chemical properties of cementing agents significantly affect its adhesion mechanism and durability (Özcan, 2003; Mosele, 2014 and Pozzobon, 2017). Adhesion of resin cement to zirconia is difficult in comparison with

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feldspar ceramics due to its higher content of vitreous phase (Tzanakakis, 2016; Hallmann, 2016; Thompson, 2011). Adhesion of resin cement to the inner surface of zirconia requires a specific preparation to promote a retentive surface and reliable chemical binding (Tzanakakis, 2016; Mosele, 2014 and Pozzobon, 2017). Several techniques promote adhesion of the substrate to the zirconia surface, such as mechanical and chemical treatment, laser treatment, silicatization, silanes, and cement initiators (Tzanakakis, 2016; Pozzobon, 2017; Ozcan, 2015). Studies show a poor adhesion of dental cement to the inner surface of Zirconium Oxide. Therefore, one should adequately associate the cement system with the correct mechanical surface treatment, since there are several factors influencing the results obtained in these studies, such as aging methodology, type of tests conducted, surface topography and possible damages to the Zirconium surface arising from mechanical treatment (Hallmann, 2016; Mosele, 2014). Given the diversity of surface treatments and types of bonding agents, one needs to base the cementation technique on reliable evidence. To this moment, there is no consensus about the best surface treatment for a good and durable bond between the resin cement and zirconium oxide ceramics (Hallmann, 2016; Mosele, 2014; Pozzobon, 2017). The aim of this work is to study the shear bond strength of two different resin cement systems: self-polymerizable cement MultilinkSistem Pack (IvoclarVivadent, Schann, Lichenstein), and self-adhesive cement RelyX U200(3M ESPE, Seefeld, Germany), to grade 5 titanium and to a yttrium-stabilized zirconium oxide ceramic, either treated or untreated with aluminum oxide blasting.

MATERIALS AND METHODS

A pre-sintered yttrium-stabilized zirconium oxide block Ceramill (AmannGirrbach, Curitiba, Brazil), measuring 100.0x8.0mm, was machined to produce 20 square pads of 7.0x7.0x8.0mm. The pads were split in half and taken to sintering oven Zirkonzahn (Zirkonzahn, Gais, Italy) for 8h, at 1500ºC. The result of this process was 40 pads of 7.0x7.0x3.0mm. Another set of 40 grade 5 titanium alloy Singular (Singular Dalton, Parnamirim, Brazil) cylindrical pads measuring 5.5 x 3.0 mm were supplied by the manufacturer. Both ceramic and titanium pads were prepared using ½ inch PVC pipes 15 mm height (Tigre, Castro, Brazil) and self-polymerizable colorless resin (Artigos Odontológicos Clássico Ltda., São Paulo, Brazil). Titanium blocks were taken to a mechanical polishing machine PolitrizArotec (Arotec, Cotia, Brazil), for polishing using water sandpaper Aquaflex (Norton, Guarulhos, Brazil) with grit sizes of 320, 400, 600, 800, 1200 for 30s each, at 600 rpm. The 80 samples were randomized in eight groups (n = 10).

- G1 – Zirconia, with surface treatment and cementation using RelyX U200.
- G2 – Zirconia, without surface treatment and cementation using RelyX U200.
- G3 – Zirconia, with surface treatment and cementation using MultilinkSistem Pack.
- G4 – Zirconia, surface treatment and cementation using MultilinkSistem Pack.
- G5 – Titanium, with surface treatment and cementation using RelyX U200.
- G6 – Titanium, without surface treatment and cementation using RelyX U200.
- G7 – Titanium, with surface treatment and cementation using MultilinkSistem Pack.
- G8 – Titanium, with surface treatment and cementation using MultilinkSistem Pack.

Forty titanium and zirconia specimens were blasted with 50µm aluminum oxide granules andpressure of 0.25 MPa, at a distance of 10.0 mm for 10 seconds. All samples were blasted with air/water for 30 seconds and soaked in distilled water for 10 minutes in an ultrasonic bath.We then proceed with cementation. The specimens cemented with Multilink were first applied with metal/zirconia primer (IvoclarVivadent, Schann, Lichenstein) according to the manufacturer's recommendations. This procedure was not performed with RelyX U200 cement due to its self-conditioning and self-adhesive properties. For standardization of the pieces during cement insertion, we used a bipartite metal matrix with a central hole of 5.0 mm of diameter and 3.0 mm of thickness. A portion of cement was inserted over the central hole, weighted in a precision scale (0.0001g), model BL 210S (Sartorius, Gottingen, Germany), and photopolymerized for 40 seconds using a photopolymerizer Ultra lux (Dabi Atlante, Ribeirão Preto, São Paulo). Test specimens were then thermally cycled in a cycling simulatorMSCT-3 PLUS (Marcelo Nucci-ME, São Carlos, Brazil), for 5,000 cycles at 5 °C, 37,5 °Cand 55 °C.
For the shear testing we used a universal testing machine EMIC DL2000 (EMIC, São Paulo, Brazil), with a load cell of 20 KN and actuator speed of 0.5 mm per minute (Figure 1). Shear strength values were recorded in MPa. Shapiro-Wilk normality test, followed by three criteria ANOVA and Tukey's test were used to analyze the data. The statistical tests were run in SPSS 20 (SPSS Inc., Chicago, IL, USA), and significance level was defined as 5%.Failures were analyzed using a stereoscopic magnifying glass EK3ST (Eikonal Equipamentos Ópticos e Analíticos, São Paulo, Brazil) with magnification of 40x and classified in adhesive, cohesive or mixedfailures.

RESULTS

Three criteria ANOVA showed no significant triple interaction between the following variables: Material, Surface Treatment, and Resin Cement ($p = 0.066$). Double interaction *Material*x *Surface* was significant ($p < 0.001$). Tukey's test showed no significant difference between zirconium oxide ceramic and titanium regarding bond strength in the absence of surface treatment, regardless of resin cement type. On the other hand, in the presence of surface treatment, titanium led to higher bond strength values (Figure 2). Intragroup comparison using Tukey's test, however, showed no effect of surface treatment on bond strength of zirconium oxide ceramics, regardless of resin cement type. Contrarily, surface treatmenthas significantly increased titanium bond strength (Figure 2). Three criteria ANOVA also showed no significant double interaction between variables *Material* and *Resin Cement* ($p = 0.935$), nor between *Surface Treatment* and *Resin Cement* ($p = 0.418$). Also, there was no significant difference in bond strength between resin cements Rely X U200 and Multilink ($p = 0.444$), regardless of material (zirconium oxide ceramic or titanium) or surface treatment. Concerning failure mode, the ruptures were exclusively adhesive on zirconium oxide ceramic, regardless of surface treatment or cementing agent. On titanium, adhesive failures accounted for 60 to 90% of rupture modes in the groups with surface treatment (regardless of resin cement type) and in the group with no surface treatment and RelyX U200 cement. Cohesive ruptures were

Figure 1. Shear testing

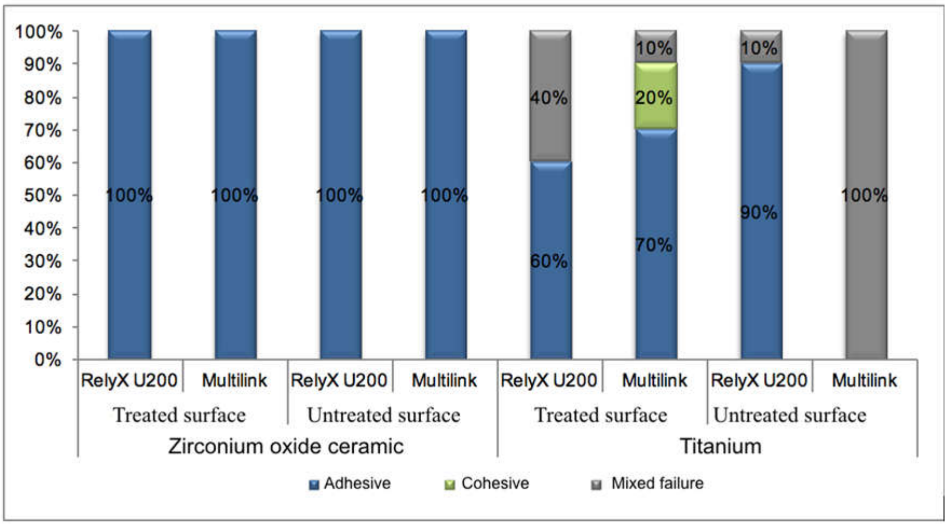

Figure 2. Bar graph of average bond strength according to material used, surface treatment, and resin cement type (vertical bars indicate standard deviation)

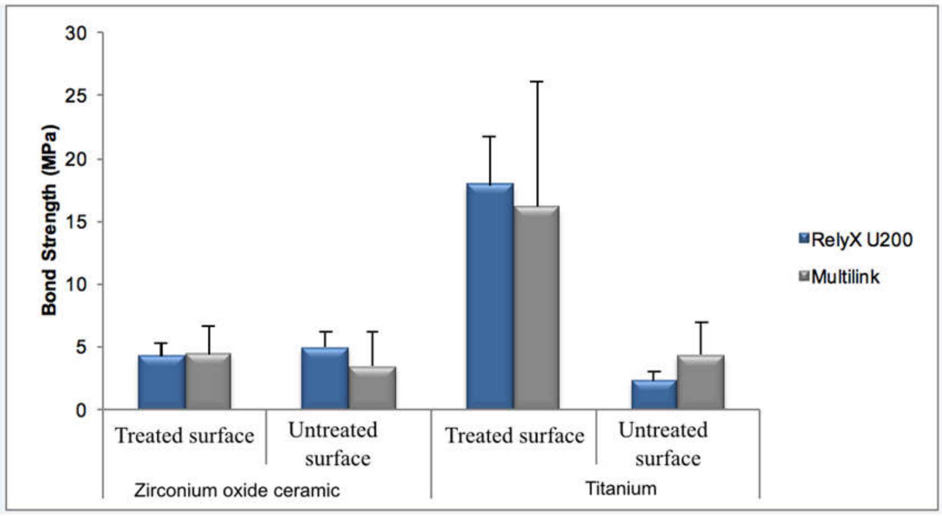

Figure 3. Bar graph of failure modes proportions according to material, surface treatment, and resin cement type

observed only in titanium treated and cemented with Multilink, accounting for 20% of the samples. Exclusively mixed failures were observed in the group of untreated titanium pieces cemented with Multilink (Figure 3).

DISCUSSION

The success rate of ceramic crown cementation in natural teeth depends on strength and durability of the bond between the tooth surface, the cementing agent, and ceramics. Given the variety of cementing systems and techniques used to improve efficacy, there is still no consensus on the best combination for an efficient and durable bond (Belo, 2013; Tzanakakis, 2016; Papia, 2014; Mosele, 2014; Thompson, 2011; Ozcan, 2015; Souza, 2014 and Luthra, 2016). A material surface treatment aims to prime it, creating micro-retentions and favoring mechanical imbrications between substrate and cement. Depending on the technique chosen, surface rugosity can vary. A rougher surface can affect wettability of the bonding agents to the material, allowing cement leakage through the micro-retentions, improving bonding between cement and surface (Mirmohammadi, 2010; Tzanakakis, 2016; Inokoshi, 2014; Papia, 2014; Hallmann, 2016; Özcan, 2003; Mosele, 2014; Pozzobon, 2017; Shahin, 2010; Amaral, 2014). The surface treatment applied in this study was the blasting with 50µm aluminum oxide particles (Almeida, 2010), at 0.25 MPa, with exposure time of 10s, at 10mm of distance. With surface treatment, titanium showed significantly higher strength compared to zirconia ceramics. This result was supplemented by the failure mode results which were 100% adhesive for zirconia regardless of type of cement or presence of surface treatment. Within the zirconia group, surface treatment had no effect on bond strength, contrasting with Shahim eKern (Thompson, 2011), study that showed an increase in strength with the same surface treatment. However, we observed a difference on this property depending on blasting time, specifically for 15 seconds. Yang *et al*. (Yang, 2010), obtained good results with the same blasting method used here.

Studies that used a combination of mechanical and chemical methods in zirconium oxide ceramics showed higher bond strength values than those using either one of the methods alone (Luthra, 2016). Regarding chemical retention, different cementing system have been proposed for use with zirconium oxide ceramics attempting to reach reliable adhesion. Under adequate conditions, resin cements provide stronger bond and better physical properties than conventional ones (Tzanakakis, 2016; Inokoshi, 2014; Papia, 2014; Hallmann, 2016; Özcan, 2003; Mosele, 2014; Pozzobon, 2017; Luthra, 2016; Amaral, 2014). Bonding between resin and zirconia are still not completely reliable and may not withstand occlusal load conditions (Behr, 2011). Here, surface treatment showed no significant effect on the zirconia samples, regardless of resin cement applied. Some studies have claimed that, in addition to the surface treatment, a stronger and durable bond between substrate and cement requires the application of silanes (Matinlinna, 2006; Matinlinna, 2006; Matinlinna, 2006 and Piascik, 2009). Nonetheless, surface treatment has significantly increased bond strength values of titanium samples, regardless of cement type, which is corroborated by failure mode results that indicate adhesive, cohesive and mixed failures when Multilink cement is associated to surface treatment.

MDP-based resin cement tend to show better results in comparison with other types of cement. MDP-based silanes or self-adhesive cement are considered good alternatives for association with blasting (Ozcan, 2015). Here, we chose two cementing systems, Multilink (Ivoclair/Vivadent) self-polymerizable, and RelyX U200 (3M-ESPE) self-adhesive with dual polymerization. Because the samples cemented with RelyXU200 are self-adhesive, they were not silanized. Materials used here showed no influence of the type of cement applied, in agreement with Mirmohammadi *et al*. (Mirmohammadi, 2010) and Aleisa *et al*. (Aleisa, 2013), that showed that cementation is not influenced by the type of resin cement.

Conclusion

Based on the methods used here and results showed, we conclude that surface treatment using aluminum oxide blasting is effective to increase bond strength of cements MultilinkSistem Pack and RelyX U200 to titanium samples. On the other hand, surface treatment failed to affect cements' bond strength to zirconia samples.

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