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Influence of surface treatment on the shear strength of Ultra Translucent Multi Layered Zirconia Katana

Influência do tratamento de superfície na resistência ao cisalhamento da Zircônia Katana Ultra traslúcida multi camadas

Influencia del tratamiento superficial en la resistencia al corte de Katana Ultra Zirconia translúcida multicapa

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Abstract
The aim of this study is to assess how shear strength of Ultra Translucent Multi Layered Zirconia Katana (UTML) is affected by the conditioning with 10% hydrofluoric acid combined with the current surface treatment protocol: air, primer containing 10-MDP, and cement. Samples of UTML Zirconia Katana were machined to measure 5x5x10 units of measurement, attached, and divided into two groups. In the control group, samples were treated with 27 um aluminum oxide blasting at 4 bars, application of Zr primer containing 10-MDP and, finally, cement Panávia V5. In the experimental group, samples were also treated with 10% hydrofluoric acid following blasting and prior to the primer application. The test specimens were submitted to shear mechanical strength test on a universal testing machine EMIC

DL2000 (EMIC, São Paulo, Brazil), surface analysis with Scanning Electron Microscope (SEM) (Sputter Coaster Emitech, K450) and surface roughness. Results showed that the bond strength of specimens conditioned with acid was 41% higher (p = 0.031). Student's t test showed that the samples conditioned with acid were rougher than those without conditioning (p = 0.045). Microscopic analysis showed surface alterations on samples conditioned with acid. It is possible to conclude that the group conditioned with hydrofluoric acid showed better adhesive behavior.
Keywords: CAD-CAM; Ceramics; Crowns.

Resumo
O objetivo desse trabalho foi avaliar a resistência ao cisalhamento das amostras da Zircônia Katana Ultra translúcida e multicamadas (UTML) sob influência da ação do ácido fluorídrico a 10% combinada ao protocolo de tratamento superficial vigente: ar, primer contendo a molécula 10-MDP e por fim o cimento propriamente dito. Amostras de Zr Katana do tipo UTML foram usinadas em tamanhos de 5x5x10 unidade de medida, fixadas e divididas em dois grupos. Em G1 (controle), as amostras foram preparadas superficialmente com a utilização do jateamento de Oxido de alumínio 27um a pressão de 4 bars, aplicação de primer de Zr contendo 10-MDP e por fim o cimento Panávia V5. Em G2, foi adicionado a utilização do ácido fluorídrico a 10% posteriormente o jateamento e previamente à aplicação do primer. Os corpos de prova foram submetidos ao ensaio mecânico de resistência ao cisalhamento (RC) em uma máquina de ensaio universal EMIC DL2000 (EMIC, São Paulo, Brasil), análise superficial com microscopia eletrônica de varredura (MEV) (Sputter Coaster Emitech, K450) e rugosidade superficial (RS). Os dados obtidos foram analisados estatisticamente e monstrou que os espécimes de Zr que sofreram o condicionamento ácido, foram 41% superiores em relação a resistência de união (0.031). O teste T de Student demonstrou que as amostras que sofreram o condicionamento ácido, apresentaram significantemente mais rugosas quando comparados as amostras sem o condicionamento (0.045). A análise microscopica demonstrou que as amostras submetidas a ação do acido tiveram superfície alterada. Conclui-se que o grupo que sofreu a ação do ácido fluorídrico apresentou melhor comportamento adesivo.
Palavras-chave: CAD-CAM; Cerâmicas; Coroas.

Resumen
El objetivo de este trabajo fue evaluar la resistencia al corte de muestras translúcidas y multicapas de Katana Ultra Zirconia (UTML) bajo la influencia de ácido fluorhídrico al 10% combinado con el protocolo de tratamiento superficial actual: aire, primer que contiene la molécula 10-MDP y finalmente el cemento mismo. Las muestras de Zr Katana tipo UTML se mecanizaron en tamaños de unidad de medida de 5x5x10, se sujetaron y se dividieron en dos grupos. En G1 (control), las muestras fueron preparadas superficialmente mediante chorreado de óxido de aluminio de 27um a una presión de 4 bares, aplicación de una imprimación de Zr conteniendo 10-MDP y finalmente el cemento Panávia V5. En G2 se añadió el uso de ácido fluorhídrico al 10% después del granallado y antes de aplicar la imprimación. Los especímenes fueron sometidos a ensayos mecánicos de resistencia al corte (RC) en una máquina de ensayo universal EMIC DL2000 (EMIC, São Paulo, Brasil), análisis superficial con microscopía electrónica de barrido (SEM) (Sputter Coaster Emitech, K450) y rugosidad superficial (RS). Los datos obtenidos fueron analizados estadísticamente y mostraron que las probetas de Zr que se sometieron a grabado ácido fueron un 41% más altas en relación a la fuerza de unión (0,031). La prueba t de Student mostró que las muestras que se sometieron a grabado ácido eran significativamente más ásperas en comparación con las muestras sin grabado (0,045). El análisis microscópico mostró que las muestras sometidas a la acción del ácido presentaban alteración superficial. Se concluye que el grupo que sufrió la acción del ácido fluorhídrico presentó mejor comportamiento adhesivo.
Palavras clave: CAD-CAM; Cerámicas; Coronas.

1. Introduction

For many years, dental restorations used metal-based materials coated with feldspar-based ceramics. However, despite their good mechanical properties, metal-based infrastructures face aesthetical constraints (Oliveira et al., 2017). Light transmission and the greyish color, in addition to the metal exposure in certain spots, motivated the search for new ceramic materials for this type of application (Oliveira et al., 2017). With the wide availability of digital dentistry and CAD/CAM systems, new ceramic-based materials have become available, among which is Zirconia (Alghazzawi et al., 2012).

Stabilized tetragonal Zirconia polycrystal (3Y-TZP) presents crystalline structure and is highly opaque, which makes it unfit for restorations in certain aesthetic zones (Shahmiri et al., 2018). Aiming to circumvent its opacity and trying to emulate natural teeth color shades, systems based on zirconia with larger proportions of yttrium and with several layers of color gradient have become available in the market. These systems are capable of producing crowns where the incisal portion is more translucent, increasing in terms of chroma and saturation towards the cervical third portion (Almeida et al., 2020).

The first multilayered zirconia system introduced in the market was Katana (Kuraray Noritake, Japan) that included three levels of translucency: ultra translucent multilayered (UTML), super translucent multilayered (STML) and multilayered (ML), that cover all monolithic restoration applications (Daneshpooy et al., 2019).

Another factor worth considering in regards to zirconia-based ceramics is their low adhesive capacity in comparison with other vitreous ceramics (Malheiros et al., 2013). However, the literature describes some methods to improve this feature, such as the surface treatment developed by Markus Blatz - APC Thecnique (Air, Primer, Composite), and silicatization (Blatz et al., 2016). Notwithstanding, new alternatives of surface treatment methods for zirconia are still needed.

The present study aims to assess the effects of the pre-treatment of the surface of ultra translucent multilayered Zirconia Katana (Kuraray Noritake Dental Inc.) on the shear strength to resinous dual cement Panavia V5. The assessment was performed using the APC method (Air, primer, composite), with and without the use of hydrofluoric acid at 10%. The resulting surface roughness of the specimens are also analyzed with scanning electron microscope. The working hypothesis is that 10% hydrofluoric acid increases bond strength of ultra translucent zirconia to resinous dual cement.

2. Methods

Sample calculation was run on G*Power 3.1.9.4, indicating the need for 17 specimens per group, totaling 34 samples of Katana UTML (Kuraray Noritake Dental Inc., Aichi, Japan).

A pilot test was conducted to determine the time of application of hydrofluoric acid Condac Porcelana 10% (FGM, Joinville, Santa Catarina, Brazil) to Zirconia. Sixteen blocks of Zirconia Katana UTML were machined and divided into 4 groups (n=4). All groups received surface treatment with 27um aluminum oxide blasting (Denville Materials, Cosmedent) followed by the application of a primer for Zirconia that contains a silanol group and the functional molecule 10-MDP (Ceramic Primer, Kuraray Noritake) and cementation with Panavia V5 (Kuraray Noritake Dental Inc., Aichi, Japan). The groups differed in time of hydrofluoric acid application: no application, 20 seconds, 60 seconds, and 120 seconds. The shear stress test showed that 20 and 60 seconds of application rendered the most relevant results. Considering the similarity of this material and lithium disilicate and that most studies use 20 seconds for the conditioning of this ceramic, the present study defined 20 seconds as the conditioning time (Kwon *et al*., 2018).

Thirty-four previously sintered 18 mm blocks of ultra translucent multilayered Katana (Kuraray Noritake, Tokyo, Japan) yttrium-stabilized zirconia color A1 were milled to measure 5x10x10mm using a CAD/CAM system Zenotec/Wielandpastilhas. The milled samples were placed in a ½” PVC pipe of 15mm height, (Tigre, Castro, Brazil) in a standardized way.

After the stabilization of the pipes and the samples with utility wax on glass plates, the assembly was filled with self-polymerizable acrylic resin with aid of a funnel. All pipes were completely filled and left to rest for the polymerization time, according to the manufacturer’s recommendation. Once the process was concluded, the PVC pipe was removed and the samples were stabilized by the acrylic and submitted to finish and polish in a polishing machine (Politriz, Arotec, Campinas-SP, Brazil) with sandpaper (Aquaflex GR150 Norton, Sertão) of grit sizes 600, 800, 1000 and 1200 for 30 seconds at 300 rotations per minute to remove any residues of glue or acrylic remaining on the Zirconia surface.

The test specimens were divided into two groups (n=17) to receive the surface treatment. The control group received only mechanical surface treatment with the APC method, as described by Markus Blatz (Blatz et al., 2016; 2018, consisting in the blasting with 27 µm aluminum oxide at a distance of 1 cm and 4 bars, active application with micro-brush Points (SDI) of Ceramic Primer (Kuraray Noritake) containing the functional monomer 10-MDP and a silanol group along the entire specimen, and the cementation with Panavia V5 cement with a mixer tip provided by the manufacturer and polymerization. In the experimental group, the specimens were submitted to the same protocol plus the application of 10% hydrofluoric acid for 20 seconds (Figure 1) before the application of Ceramic Primer (Kuraray Noritake).

Figure 1 – Application of 10% hydrofluoric acid and accommodation with microbrush.

Source: Own authorship.

With all the blocks properly prepared, cementation with Panavia V5 was performed on a metal bipartite matrix with a central orifice of 5.0 mm in diameter and 3.0 mm of thickness with mixer tips provided by the manufacturer; cements were polymerized with photopolymerizer Valo (Ultradent, South Jordan, USA) photoactivated for 40 seconds. All samples were manufactured and tests were conducted by the same operator.

Samples were then submitted to the shear mechanical stress test in a universal testing machine EMIC DL2000 (EMIC, São Paulo, Brazil) at the Laboratory of material testing of the University São Leopoldo Mandic, Campinas (Figure 2).

Figure 2 – Positioning of the sample under shear stress.

Source: Own authorship.

In order to strengthen the analyses, six samples were milled and divided into 2 groups (n=3). One group was milled and left intact; the other group was treated with 10% hydrofluoric acid for 20 seconds. The samples were analyzed under scanning electron microscope a the LRAC Unicamp. The specimens were attached with a double-sided carbon tape, procedure for metal coating, and taken to be analyzed on Sputter Coaster Emitech, model K450 (Kent, United Kingdom) under 15kV and 50pA and magnifications of 1500, 5000 and 15000 times to check the surface patterns with and without conditioning. Roughness was also analyzed in a rugosimeter (Sj-410, Mitutoyo, Japan).

The effect of hydrofluoric acid conditioning on the Zirconia Katana surface roughness was assessed through Student's t test given that normal distribution and variance homogeneity were met. Comparison between groups – with and without conditioning – regarding shear bond strength to resinous cement Panavia V5 was given by Mann-Whitney's non-parametric test, given the non-normal distribution of data. Type of fracture in each group was compared using Fisher's exact test with Freeman-Halton's extension. All statistical calculations were run on SPSS 23 (SPSS INC., Chicago, IL, USA) and on the website https://www.danielsoper.com/statcalc/default.aspx, considering 5% of significance.

3. Results

Student's t test showed that conditioning with hydrofluoric acid increased (44.5%) roughness of Zirconia Katana samples (p = 0.045; Table 1).

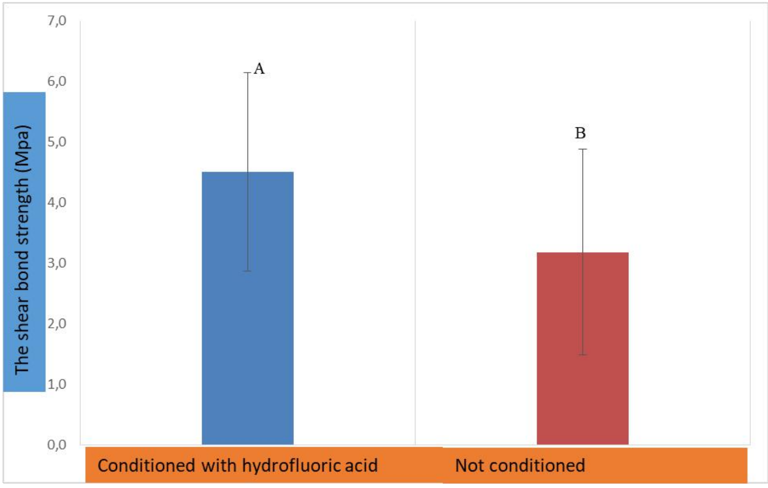
Table 1 – Averages and standard deviations of surface roughness (in µm) of Zirconia Katana, with and without conditioning with hydrofluoric acid, and shear bond strength (in MPa) to resinous cement Panavia V5.

Conditioning	Surface roughness	Bond strength
With hydrofluoric acid	0.544^{A} (0.092)	4.50^{A} (1.64)
Absent	0.376^{B} (0.130)	3.18^{B} (1.70)

Caption: Standard deviation in brackets. Within the same column, averages followed by different capital letters are significantly different (α = 0.05). Source: Own authorship.

Conditioning with hydrofluoric acid significantly increases (41.5%) shear bond strength between Zirconia Katana and resinous cement Panavia V5, according to Mann-Whitney's test (p = 0.031; Table 1; Graph 1).

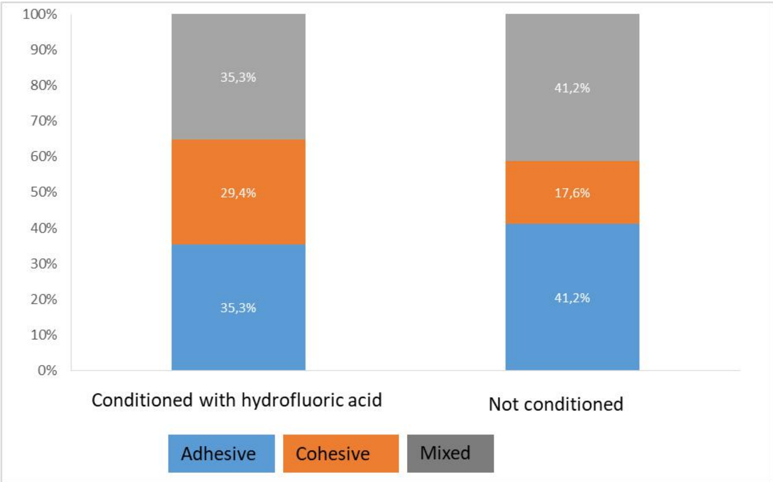
Graph 1 – Bar chart of average shear bond strength between Zirconia Katana and resinous cement Panavia V5, with and without conditioning with hydrofluoric acid.



Caption: Groups identified by different letters are significantly different. Source: Own authorship.

Graph 2 illustrates the types of fracture observed after the shear bond strength test. There is no statistical difference between groups in terms of type of fracture ($p = 0.823$), according to Fisher's exact test with Freeman-Halton's extension. In both groups, the ratio between adhesive and mixed failures were identical. Cohesive failures were less frequent in both groups.

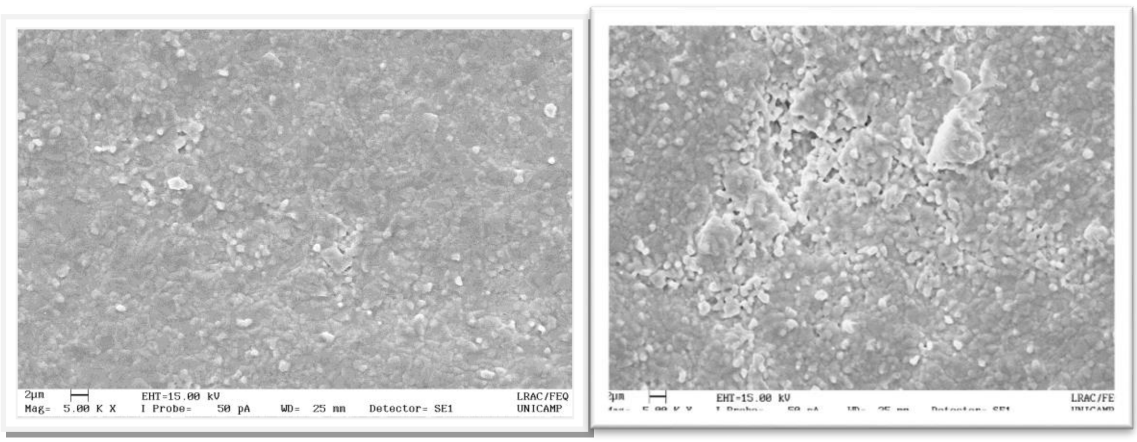
Graph 2 – Stacked column chart of types of fracture observed after the shear bond strength test for groups with and without conditioning with hydrofluoric acid.



Source: Own authorship.

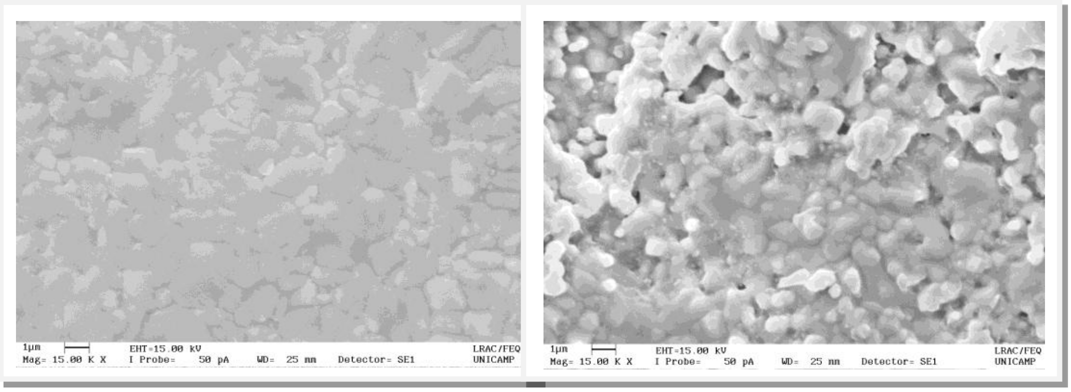
SEM images show topographical changes on the surface of Zirconia treated with 10% hydrofluoric acid in comparison with the control group, with deeper retentions and more prominent peaks observed in the treated group. Non-uniform micro-retentions were observed on the conditioned group, with regions with the presence of smooth structures with grooves identified as elongated pores (Figures 3 and 4).

Figure 3 – Scanning Electron Microscopy of conditioned and not conditioned surfaces under magnification of 5000x.



Source: Own authorship.

Figure 4 – Scanning Electron Microscopy of zirconia surface without conditioning (A) and with conditioning (B) under magnification of 15000x.



Source: Own authorship.

4. Discussion

The longevity of ceramic restorations is directly affected by the cementation protocol chosen. Currently, the protocol for vitreous ceramics consists of the dissolution of the vitreous structure with 5-10% hydrofluoric acid and the creation of a net between the ceramic's silica and a silanol group of a silane agent, and a mechanical interlock through surface roughness (Abu et al., 2012; Carvalho et al., 2021). However, due to the limited vitreous phase of Zirconia and the lack of silica in its structure (Della Bonna et al., 2014), different treatments have been suggested and studied, including blasting with particles of different sizes (Smielak et al., 2018; Czepulkowska et al., 2018), the use of functional monomers for metals and Zirconia (Magne et al., 2010), application of acid and alkaline substances (Ansari et al., 2018), incorporation of silanol groups (silicatization), and laser irradiation (Sayin et al., 2019).

The use of blasting has the objective of creating pores on the Zirconia surface to increase mechanical interlock and, as a consequence, increase retention of resinous cements. Different types and sizes of particles have been tested (Allahkarami et al., 2012; Okada et al., 2019; Inokoshi et al., 2021). However, some studies report that thicker particles blasted with higher pressure can induce a phase transition and change the material's physical and mechanical properties (Blatz et al., 2016; Zhang et al., 2019; Inokoshi et al., 2021). In the present study, blasting protocol used 27um aluminum oxide particles to avoid changing the material's properties. Other studies show that this protocol provide better adhesion quality and prevent changes to the material's structure and composition (Ozcan et al., 2015; Blatz et al., 2016).

The blasting time is also very relevant. In the present study, blasting was performed after the sintering of YTZP Zirconia blocks to avoid surface changes and harming the flow of cement and interfering with adhesion forces, as shown in previous studies (Sciasci et al., 2015; Abi-Rached et al., 2015).

Translucent Zirconias are different from past-generation Zirconias in morphology and composition and, depending on the blasting method, can undergo phase transition, which affect their flexural strength (Kaizer et al., 2020; Inokoshi et al., 2021). Studies show better results when Zirconia is submitted to blasting with aluminum oxide and a primer containing a functional monomer such as 10-MDP (Lim et al., 2020; Rodrigues et al., 2020; Valente et al., 2020). The cement used here incorporates an adhesive system with a silanol group and the 10-MDP (Tuloglu et al., 2020; Silva-Carvalho, 2020; Munoz et al., 2020). On the other hand, some studies that incorporates silica with tribo-chemical blast found better results in the first years that dropped with time. Also, the procedure is more expensive and has more steps in comparison with the Air Primer Composite (APC) treatment prescribed by Markus Blatz (Blatz et al., 2016; Colombo et al., 2020), and used in the present study.

Given that surface treatments with acids for past-generation Zirconias were poorly effective, it is important to investigate their effectiveness in the adhesion of translucent or ultra translucent Zirconias (Agren et al. 2019; Lenz et al. 2021). Also, translucent and ultra translucent Zirconias are easy to mill, and present interesting morphological, optical and mechanical properties, making them good subjects for further studies (Kolakarnprasert et al., 2019; Holman et al., 2020). Considering that the characteristics of this material are closer to lithium disilicate, the present study worked with the hypothesis that hydrofluoric acid could enhance the shear strength of Zirconia Katana UTML (Le et al., 2019; Sadid-Zadeh et al., 2021).

Here, conditioning with 10% hydrofluoric acid increased the bond strength between resinous cement and Zirconia UTML in 41%. Some studies show contrasting results, where conditioning with hydrofluoric acid has not affected bond strength, regardless of the type of Zirconia (Ansari et al., 2018; Casucci et al., 2008), or whether they are used alone or in combination with blasting (Sadid-Zadeh et al., 2021). On the other hand, some studies showed that conditioning with hydrofluoric acid for 30 seconds increased the bond strength of ultra translucent Zirconia (Le et al., 2019; Altan et al., 2019).

One study found no difference between translucent Zirconias and lithium disilicate in terms of bond strength (Agren et al., 2019). Another study showed a drop in the bond strength of cemented Zirconia blocks kept in distilled water for two years (Lenz et al., 2021). Another study showed that hydrofluoric acid promotes surface roughness on YTZP Zirconia, but this effect was of little relevance compared to conventional treatments such as abrasion with aluminum oxide and silicatization (Thammajaruk et al., 2018; Casucci et al, 2008).

SEM and the rugosimeter are key to comparing surface features such as roughness and assess the effectiveness of treatment on the adhesive quality of the material. In the present study, conditioning of Katana UTML with 10% hydrofluoric acid for 20 seconds increased roughness in 44% in comparison with the control group (without conditioning). However, another study showed that conditioning with 40% hydrofluoric acid for 20 seconds did not change the surface roughness pattern of 3-YTZP Zirconia in comparison with other groups (Souza et al., 2020). Thammajaruk et al. (2018) concluded that the chemical treatment of Zirconia with acid solutions has little effect on its surface roughness and bond strength. Here, the samples analyzed with SEM showed alterations on the surface, contrasting with the findings of Casucci et al., 2009, that used concentrations close to 10%.

The present study found 3 types of fracture – adhesive, cohesive and mixed. Good bond strengths are frequently related to mixed or cohesive fracture patterns. Cohesive fractures were not found in other studies testing Zirconia's bond strength to resinous cement (Foxton et al., 2011; Gallina, 2017).

5. Conclusion

Based on the methods used here and results obtained, it is possible to conclude that the group submitted to 10% hydrofluoric acid for 20 seconds showed higher shear bond strength, higher roughness and greater surface irregularity under Scanning Electron Microscope.

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