

Efficiency of treatment and use of a MDP-primer on the bond strength of zirconia-cement interface

Eficiência do tratamento e da utilização de um primer a base de MDP na resistência de união da interface zircônia-cimento

Eficacia del tratamiento y de la utilización de un primer a base de MDP en la resistencia de unión en la interfaz circonio-cemento

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Abstract

In order to improve the adhesion between cement and zirconia, aluminum oxide sandblasting (JAT) and Rocatec, sandblasting with silica coated aluminum oxide (ROC), also the primer Signum Zirconia Bond (SZB) were used. The objective of the study was to verify the influence of the surface treatment and the presence of SZB primer in relation to the bond strength between cement and zirconia, using 60 zirconia specimens measuring 7 mm in length, 7 mm in width and 3 mm in length. thickness. Three surface treatments were evaluated: sandblasting with pre-sintering aluminum oxide (OPre), Rocatec Soft before (RPre) and zirconia final sintering (RPos), and also, regarding the presence or not of SZB. All specimens were cemented with Panavia F. The bond strength (RU) was evaluated through a shear test with a standardization device from SDI (Southern Dental Industrie) on a universal testing machine (Kratos), with a speed of 0,5 mm / min. The data were evaluated using Kruskal-Wallis / Mann-Whitney ($\alpha = 0.05$). Average (SD) of UK without the use of SZB: OPre: 5.5 (0.9) Ba; RPre: 4.0 (1.4) Bb; RPos: 5.0 (0.5) Bab. Average (SD) of UK using SZB: OPre: 7.9 (1.5) Aab; RPre: 6.7 (2.3) Ab; RPos: 9.0 (1.2) Aa. The use of primer increased the cement / zirconia bond strength regardless of the surface treatment performed, in addition, the Rocatec group after sintering with primer, obtained higher bond strength values compared to the other study groups.

Keywords: Dentin bonding; Ceramics; Zirconium.

Resumo

Para melhorar a adesão entre cimento e zircônia, foram utilizados jateamento com óxido de alumínio (JAT) e o Rocatec, jateamento com óxido de alumínio revestido por sílica (ROC), também o primer Signum Zircônia Bond (SZB). O objetivo do estudo foi verificar a influência do tratamento de superfície e da presença de primer SZB em relação a resistência de união entre cimento e zircônia, utilizando 60 corpos de prova de zircônia medindo 7 mm de comprimento, 7 mm de largura e 3 mm de espessura. Foram avaliados três tratamentos de superfície: jateamento com óxido de alumínio pré-sinterização (OPre), Rocatec Soft antes (RPre) e após-sinterização final da zircônia (RPos), e

também, quanto a presença ou não de SZB. Todos os corpos de prova foram cimentados com Panavia F. A resistência de união (RU) foi avaliada através de teste de cisalhamento com dispositivo de padronização da SDI (Southern Dental Industrie) em uma máquina de ensaios universal (Kratos), com velocidade de 0,5 mm/min. Os dados foram avaliados utilizando Kruskal-Wallis/Mann-Whitney ($\alpha=0,05$). Médias (DP) de RU sem a utilização de SZB: OPre: 5,5 (0,9) Ba; RPre: 4,0 (1,4) Bb; RPos: 5,0 (0,5) Bab. Médias (DP) de RU com a utilização de SZB: OPre: 7,9 (1,5) Aab; RPre: 6,7 (2,3) Ab; RPos: 9,0 (1,2) Aa. A utilização do primer aumentou a resistência de união cimento/zircônia independentemente do tratamento de superfície realizado, além disso, o grupo Rocatec pós-sinterização com primer, obteve maiores valores de resistência de união comparados aos demais grupos do estudo.

Palavras-chave: Adesivos dentinários; Cerâmica; Zircônio.

Resumen

Para mejorar la adhesión entre el cemento y el zirconio, fue utilizado el chorro con óxido de aluminio (JAT) y el Rocatec, chorro con óxido de aluminio revestido por sílica (ROC). también el primer Signum Zirconia Bond (SZB). El objetivo de este estudio fue verificar la influencia del tratamiento de superficie y de la presencia del primer SZB en relación a la resistencia de unión entre el cemento y el zirconio, utilizando 60 cuerpos de prueba en zirconio midiendo 7mm de altura, 7mm de ancho y 3 mm de espesor. Fueron evaluados tres tratamientos de superficie : chorro con óxido de aluminio pre-sinterizado (OPre), Rocatec Soft antes (RPre) y después de la sinterización final del zirconio (RPos) y también cuando hay presencia o no del SZB. Todos los cuerpos de prueba fueron cementados con Panavia F. La resistencia de unión (RU) fue evaluada por el teste de cizallamiento con el dispositivo de padronización de la SDI (Southern Dental Industrie) en una maquina de ensayo universal (Kratos), a una velocidad de 0,5 mm/min. Los datos fueron evaluados utilizando Kruskal-Wallis/Mann-Whitney ($\alpha=0,05$). Medias (DP) de RU sin utilización del SZB: OPre: 5,5 (0,9) Ba; RPre: 4,0 (1,4) Bb; RPos: 5,0 (0,5) Bab. Medias (DP) de RU con la utilización de SZB: OPre: 7,9 (1,5) Aab; RPre: 6,7 (2,3) Ab; RPos: 9,0 (1,2) Aa. El uso del primer aumento la resistencia de unión cemento/zirconio independentemente del tratamiento de superficie realizado, además, el grupo tratado con Rocatec pos-sinterización con primer obtuvo mayores valores de resistencia de unión comparados a los demás grupos de estudio.

Palabras clave: Adhesivos dentinarios; Cerámica; Zirconio.

1. Introduction

The improvement of adherence to zirconia is a constant search in dental research. Over the years, different methodologies have been tested in order to compensate for the lack of chemical reaction associated with the absence of silica in these materials (Palacios et al., 2006), what in vitreous ceramics is responsible for the union with resin cements (Thompson et al., 2011). Among these methods, we can mention aluminum oxide blasting (Denry et al., 2008)(Ebeid et al., 2018), tribochemical abrasion treatment with silica-coated aluminum oxide particles (Akio Maeda et al., 2014)(Ruales-Carrera et al., 2019), experimental hot pickling solution (Elsaka, 2013) selective infiltration (Casucci et al., 2009), silane (Egilmez et al., 2013) and even ND:Yag (Neodymium - Itrio-Aluminum-Granada) and ER:yag lasers (Erbium - Itrio-Aluminum-Granada) have been suggested as alternative methods for increasing the roughness of polycrystalline ceramics (Akio Maeda et al., 2014).

Despite the cementation of zirconia restorations with traditional cements (zinc phosphate or ionomeric) guarantee adequate clinical fixation, adhesive cementation is preferable for ensuring greater retention and marginal adaptation (Andreiuolo et al., 2011). Some studies suggest the use of chemical bonding by means of primers with phosphate monomers such as 10-MDP (10 – methacryloyloxydecyl dihydrogen phosphate) (Ruales-Carrera et al, 2019;Ali et al., 2019;Turp et al., 2016). These monomers, like silanes, are bifunctional molecules that join at one end to the metal oxides of ceramics and at the other end, present groups that co-polymerize with the resin matrix of cements.(Andreiuolo et al., 2011) Achieving reliable adhesion to zirconia-based ceramics requires surface treatments based on physical retention through sandblasting and chemical bonding using silanes and / or primers. In the specific case of zirconia, since it is a highly crystalline ceramic, free from the vitreous phase, the application of hydrofluoric acid is not effective for creating roughness on the surface of the zirconia, making it difficult to adhere to the cement. (Thompson et al., 2011; Amaral et al., 2008;Lüthy et al., 2006).

Studies demonstrate the effectiveness of surface treatment through blasting with aluminum oxide. This technique would increase the roughness of the zirconia, increasing the surface area of the ceramic and thus facilitating its mechanical wrapping (Atsu et al., 2006). Another option is silication, which consists of sandblasting with aluminum oxide particles

covered by silica (Rocatec -3M ESPE). The impact of these particles generates a local increase in temperature, causing the silica to partially melt with the ceramic surface, which allows for a relative chemical bond, through silanes, between cement and zirconia. (Andreuolo et al., 2011;Atsu et al., 2006;Pozzobon et al., 2017), improving this reaction (Akio Maeda et al., 2014). Surface treatment by tribochemical abrasion associated with the use of universal adhesives containing 10-MDP can provide a durable bond to conventional and highly translucent zirconia. (Ruales-Carrera et al, 2019;Yue et al., 2019;Ali et al., 2019).

The objective of the study was to evaluate the bond strength between zirconia stabilized by Itrium and cement depending on the factors: surface treatment (blasting with aluminum oxide before final sintering, Rocatec Soft before and after final zirconia sintering) and presence of a specific zirconia primer with 10-MDP (Signun Zircônia Bond - Heraus).

2. Methodology

Specimen preparation

Zirconia blocks were used (Lava, 3M-ESPE St Paul,MN EUA) and cutted with a diamond disc mounted on a high precision cutter under water refrigeration (Isomet 1000, Buehler, Germany), obtaining uniform blocks with approximate dimensions of 7x7x3 mm. During sintering these specimens presented a volumetric reduction of about 25%.

All specimens had their surfaces polished with abrasive aluminum oxide sandpaper with granulation up to 1200.

Experimental groups

The specimens were divided into six experimental groups with 10 specimens each group, according to the factors analyzed: Surface treatment (3 levels) and presence of primer (2 levels).

AO - Sandblasting with aluminum oxide before final sintering.

AOp - Sandblasting with aluminum oxide before its final sintering followed by application of the primer.

S - Sandblasting with ROC before its final sintering.

Sp - Sandblasting with ROC before final sintering followed by primer application.

S * - Sandblasting with ROC after final sintering

Sp * - Sandblasting with ROC after final sintering followed by application of primer.

Sandblasting

The specimens of groups AO and AOp were blasted with aluminum oxide particles of 110 µm (N. Martins e Teixeira Ltda, Colombo, PR. Brasil) (JAT), at a distance of 40 mm, angulation of 45 ° and a pressure of 2.8 bar for 10 seconds.

The specimens of groups S, Sp, S * and Sp * were first sandblasted with aluminum oxide as performed for AO and AOp and then received tribochemical treatment with aluminum oxide particles coated with silica (Rocatec Soft – 3M-ESPE St Paul, MN EUA) 30 µm, shot blasted at a distance of 40 mm, angulation of 45 ° and a pressure of 2.8 bar for 10 seconds. For the standardization of distance and angulation during blasting with aluminum oxide and Rocatec.

Sintering

The specimens were sintered in the oven (Zyrcomat Vita, Zahnfabrik, Germany), with a sintering cycle of 1530°C for 2 hours, a heating rate of 25°C/min and a cooling time of 7.5 hours.

Inclusion of specimens

The specimens were included in PVC tubes with 25 mm in diameter (3/4 of an inch), filled with chemically activated acrylic resin (Jet - Artigos Odontológicos Clássico Ltda. Brazil), leaving the treated face free for the adhesive procedure.

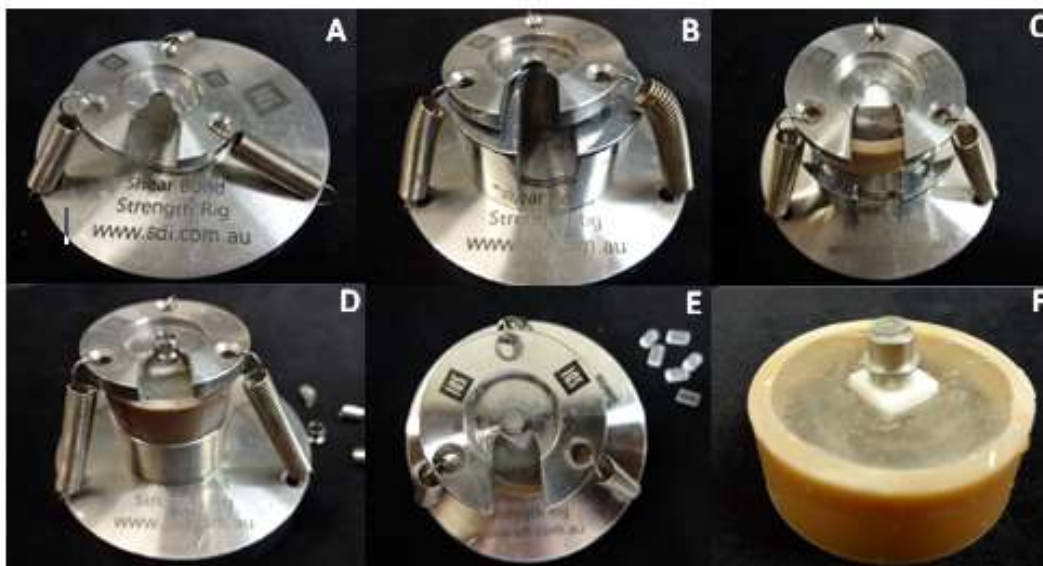
Application of the specific primer for Zirconia

The groups, AOp, S * and Sp * received the application of Signun Zirconia Bond I (Heraeus Kulzer) primer over the entire surface of the zirconia with the help of disposable applicators KG Brush (Medical Burs industry and commerce - Cotia Brazil), followed by application of a light air jet for three seconds, application of the Signun Zirconia Bond 2 primer, 40-second light curing (according to the manufacturer's recommendations) using a VIP Jr halogen lamp light curing with 500 mW/cm² irradiance.

Preparation of the shear bond strength device

After surface treatments, final sintering and inclusion of resin specimens in PVC tubes, they were assembled in the shear bond strength device (5), which consists of a hollow metal cylinder, with an external diameter of 4 mm and an internal diameter of 3, 5 mm, with a metallic outer ring that divides at the height of 1/3 of the cylinder, transparent acrylic pin with 3 mm in diameter, 3.8 mm high, metal shear device.

Figure 1. Sequence of assembly of the specimens in the SDI device, A) - Empty device; B) - Device with compression system; C) - Specimen added to the device; D) - Addition of the hollow metal cylinder; E) - Acrylic pin inserted in the device; F) - Final specimen ready for shear test.



Source: Authors.

The specimens were mounted on the shear device, followed by the placement of the metallic cylinder, which was under compression next to the specimens, due to the outer ring of the cylinder fitting juxtaposed to the upper plate of the device.

Cementation

After mixing the resin cement Panavia F (Kuraray, Osaka, Japan), according to the manufacturer's instructions, it was placed inside the hollow cylinder.

The acrylic pin was placed inside the cylinder, making it very close to the height of the cylinder. The excess cement was removed, and the set was photo-polymerized for 30 s, with irradiance of 500 mW/cm². The protection of the specimens was then carried out with oxiguard (Kuraray, Osaka, Japan) for 3 minutes, the specimens were then washed in running water for 10 seconds and stored in distilled water at 37°C for 24 h.

Shear test

The shear bond strength tests were performed using a universal testing machine (Kratos, LKC3 – USB, Brasil). The specimens were adapted to a metallic matrix for vertical positioning on the machine and parallel to the straight-edge knife. The shear force was applied until the fracture between cement and Y-TZP occurred, at a constant speed of 0,5 mm/min.

The bond strength values were obtained by measuring the load required to cause fracture of the top and expressed in kilogram-force (Kgf). To calculate the bond strength the following formula was used: $BS = L/A$, where: BS is the bond strength (MPa); L (Kgf), load; A, bonding area (mm²).

Failure mode analysis

The failure mode of each specimen was determined using a stereo-microscope under 40x magnification (Olimpus SZ61) equipped with a CCD camera (Q-Color 3, Olympus), and classified as: (1) predominantly adhesive failure between cement / zirconia; (2) cohesive failure in cement; (3) mixed failure, with adhesive and cohesive failures on the same fracture surface.

Scanning electron microscopy (SEM)

Representative specimens of each type of failure were evaluated using a scanning electron microscope (JSM-6610LV, JEOL Ltda, Tokyo, Japan), for more detailed analysis of the failure mode under 50x magnification.

Statistical analysis.

Due to the absence of normality and homoscedasticity, data were analyzed using the Kruskal-Wallis and Mann-Whitney non-parametric test. For both tests, the global level of significance was 5%.

Results

Table 1 shows the bond strength values obtained in the present study. The lowest value obtained was for the pre-sintering ROC group without the use of primer (4.0 MPa), and the highest value obtained for the post-sintering ROC group with the use of primer (9.0 MPa).

Table 1. Means (SD) values of bond strength (MPa).

Surface treatment	MDP Primer	
	NO	YES
Oxide before sintering (OA, OAp)	5,5 (0,9) Ba	7,9 (1,5) Aab
ROC before sintering (S, Sp)	4,0 (1,4) Bb	6,7 (2,3) Ab
ROC after sintering (S*, Sp*)	5,0 (0,5) Bab	9,0 (1,2) Aa

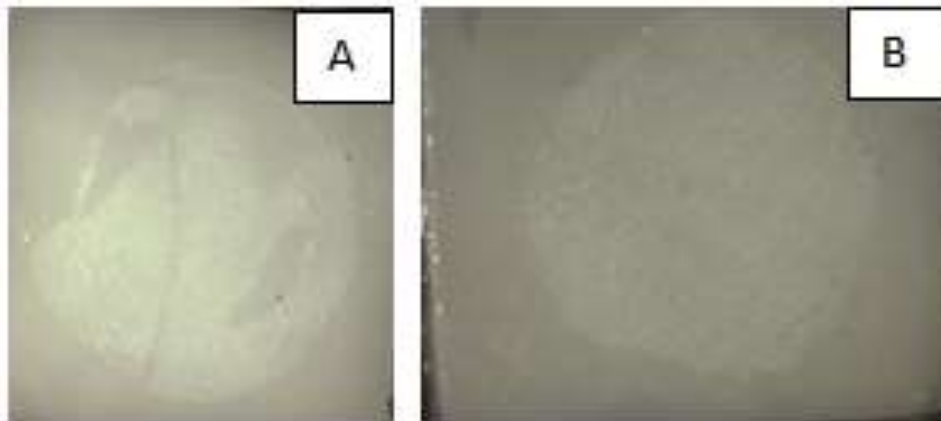
Source: Authors.

Table 2. Failure mode analysis.

Failure	Frequency	
	Count	Percentage
Predominantly adhesive failure between cement / zirconia	58	97%
Cohesive failure in cement	0	0%
Mixed failure	2	3%

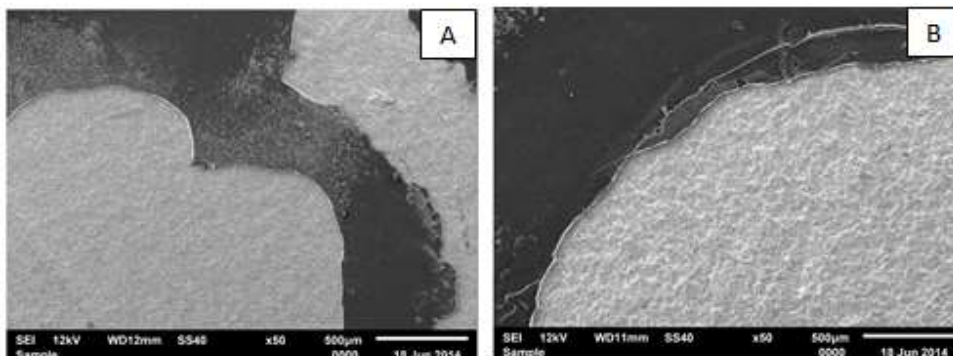
Source: Authors.

Figure 2. Representative examples of the failures found A) Mixed failure; B) adhesive failure between cement / zirconia.



Source: Authors.

Figure 3. Scanning electron microscopy. A) Mixed failure; B) Adhesive failure



Source: Authors.

3. Results and Discussion

Zirconia has a high resistance to hydrofluoric acid, so much so that its use is not enough to create retentions on its surface which would increase the mechanical embryosation of cement (Thompson et al., 2011; Turp et al., 2016).

To achieve a reliable bond between cement and zirconia, it is necessary to treat the surface by physical or chemical means, or even a combination of both media (Thompson et al., 2011; Andreiuolo et al., 2011; Amaral et al., 2008; Lüthy et al., 2006). Some authors (Thompson et al., 2011; Andreiuolo et al., 2011; Atsu et al., 2006) argue that physical treatments such as blasting with aluminum oxide are effective for increasing the zirconia's roughness. However, the technique is associated with premature aging of the material, by accelerating the reverse phase transformation, which can affect fracture resistance, also favoring the formation of cracks (Denry et al., 2008; Atsu et al., 2006).

In an attempt to achieve the benefits of blasting but without the problems caused by it, in this work we evaluated this technique in pre-sintered zirconia, as the sintering process is capable of reversing the transformation of phases caused by the sandblasting process (Atsu et al., 2006). We found that the group treated prior to sintering showed a greater amount of the tetragonal phase, which guarantees a better clinical performance of zirconia.

To further increase the adhesion between cement and zirconia, we combine chemical techniques with sandblasting, including the use of 10-MDP primer, which according to (Andriuolo et al., 2011; Turp et al., 2016); would act as bifunctional molecules, joining the metal oxides of ceramics on one side and copolymerizing the resin matrix of the cement on the other.

Another option of chemical treatment used was silicate, where a thin layer of silica fuses the surface already blasted with aluminum oxide, and this layer would allow a relative chemical adhesion through silanes. (Thompson et al., 2011; Akio Maeda et al., 2014; Andreiuolo et al., 2011; Atsu et al., 2006).

Sandblasting prior to sintering was more effective than the treatment of tribochemical abrasion, increasing the bond strength of resin cements, corroborating with results of (Ebeid et al., 2018). Sandblasting with aluminum oxide corresponds to a physical method of surface treatment, while primer and Rocatec, by forming a thin layer of silica on the grooves caused by blasting with aluminum oxide, are chemical ways of treating the surface.

The enhancement of adhesion between cement and zirconia in this study was the combination of sandblasting of pre-sintered ceramic to silicate and the use of primer containing 10-MDP with results similar to those mentioned by (Ali et al., 2019; Yue et al., 2019); where he obtained superior RU results associating sandblasting with adhesives containing MDP.

The comparison of the time of sandblasting, pre or post sintering, whether with aluminum oxide or silization, was also another objective of the present study. The results of coating with silica by tribochemical abrasion is the main surface conditioning Y-TZP for enhancement of resin bonds.

When comparing only surface treatments without primer, it can be seen that treatment with Rocatec prior to sintering (S) resulted in less bond strength compared to AO. In the presence of the primer, Sp was inferior but similar to AOp, demonstrating that this thin coating probably ceased to exist after sintering.

(Akio Maeda 2014) Evaluated the bond strength between resin cement and zirconia at three levels: a control group without treatment (CO), sandblasting with aluminum oxide and Rocatec plus (ROC) plus application of the bond zirconia bond (JAT + SZB). As a result, obtained higher RU values for the JAT + SZB group, followed by ROC and last CO, regardless of the device used to register the RU, when comparing in this study, blasting with pre-sintering aluminum oxide, plus the application of primer (OPre + SZB); Rocatec soft, pre and post-final sintering without the presence of the primer (RPre; RPos), also obtained greater results in relation to RU in the OPre + SZB group.

The reduced adhesion of zirconia to dental cements is one of the critical points in its choice as a restorative material. In order to compensate for the lack of sensitivity to hydrofluoric acid it is necessary to use an alternative capable of increasing

the roughness on the zirconia surface, thus making it more retentive in relation to cement. According to the findings of the present study, the primer containing 10 MDP resulted in higher bond strength values, regardless of the type of surface treatment used, which reinforces its chemical adhesion effect. However, longevity studies of the zirconia-cement interface are necessary to prove the permanence of this bond strength after aging.

4. Conclusion

Within the limitations of the present study, it can be concluded that:

In the absence of primer, pre-sintering treatment with Rocatec resulted in lower bond strength than blasting with aluminum oxide. With the addition of primer, Rocatec post-sintering has greater resistance compared to pre-sintering.

The use of the Signum Zirconia Bond primer increased the bond strength of zirconia to cement regardless of the chosen surface treatment.

For future research we recommend performing tests using universal adhesives compared to the specific adhesives for zirconia and also comparing the resistance between two or more resin cements.

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