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ANA CAROLINE LIMA COLOMBINO

O EFEITO DO TRATAMENTO DE SUPERFÍCIE E DA TERMOCICLAGEM NA RESISTÊNCIA DE UNIÃO DE UMA CERÂMICA DE ZIRCÔNIA ULTRA-TRANSLÚCIDA E CIMENTO RESINOSO

THE EFFECT OF SURFACE TREATMENT AND THERMOCYCLING ON THE BOND STRENGTH OF ULTRA-TRANSLUCENT ZIRCONIA CERAMIC AND RESIN CEMENT

UBERABA - MG
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Dissertação apresentada ao Programa de Pós-Graduação – Mestrado Acadêmico da Universidade de Uberaba como parte dos requisitos para obtenção do título de Mestre em Odontologia, na área de concentração em Clínica Odontológica Integrada.

Orientador: Prof. Dr. Gilberto Antônio Borges.

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O EFEITO DO TRATAMENTO DE SUPERFÍCIE E DA TERMOCICLAGEM NA RESISTÊNCIA DE UNIÃO DE UMA CERÂMICA DE ZIRCÔNIA ULTRA-TRANSLÚCIDA E UM CIMENTO RESINOSO

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RESUMO

O objetivo desse estudo foi avaliar o efeito do tratamento de superfície e da termociclagem na resistência de união de uma cerâmica de zircônia ultra-translúcida e um cimento resinoso. Cento e vinte amostras de zircônia ultra-translúcida foram confeccionadas para receber um tratamento de superfície específico: Jateamento com óxido de alumínio (OA) e condicionamento de dióxido de zircônia (DZ). Seções de zircônia (n = 10) foram distribuídas em 12 grupos de acordo com dois fatores: 1) tratamento e 2) termociclagem, sendo 1) controle (C), silano (S) e primer (P) e 2) sem termociclagem (TC_0), e com termociclagem 10.000 termociclos (TC). Para a aplicação do cimento resinoso, moldes de polivinilsiloxano foram colocados na superfície da cerâmica para determinar a área de união. Removido o excesso de cimento, em seguida foi fotoativado por 20 s, com uma fonte de luz LED. Após a remoção da matriz de silicone as amostras foram testadas em microcisalhamento (μSBS) 0,5 mm / min após 24h e após a TC. Os dados foram analisados usando o teste de Kruskal-Wallis e teste post hoc de student Newman-Keuls ($\alpha = 0{,}05$). Para a condição de TC_0, a média dos grupos (OA + S), (OA + P) e (DZ + S) resultou em maior resistência de união do que os outros grupos (OA + C), (DZ + C) e (DZ + P), sendo p 0.005. Para a condição TC, todos os grupos mostraram uma diminuição significativa na média em comparação com aqueles de 24h. No entanto, os grupos (OA + C) e (DZ + C) não resistiram aos ciclos, não sendo realizado o teste microcisalhamento. Os resultados do presente estudo mostraram a eficácia do jateamento de óxido de alumínio, juntamente com outros agentes de união e com uma resistência de união mais estável após a termociclagem e mais estudos serão necessários para o uso da pasta de dióxido de zircônia e sua concentração ideal.

Palavras chave: Cerâmica, zircônia, tratamento de superfície.

Abstract

The aim of this study was to evaluate the effect of surface treatment and thermocycling on the union strength of an ultra-translucent zirconia ceramic and a resin cement. One hundred and twenty samples of ultra-translucent zirconia were made to receive a specific surface treatment: Aluminum oxide blasting (OA) and zirconia dioxide (DZ) conditioning. Zirconia sections (n = 10) were distributed in 12 groups according to two factors: 1) treatment and 2) thermocycling, where 1) control (C), silane (S) and primer (P) and 2) without thermocycling (TC0), and with thermocycling 10,000 thermocycles (TC). For the application of resin cement, polyvinylsiloxane molds were placed on the ceramic surface to determine the joining area. Removed the excess cement, then was photoactivated for 20 seconds, with an LED light source. After removal of the silicone matrix, the samples were tested in microshear (*μ*SBS) 0.5 mm / min after 24h and after CT. The data were analyzed using the Kruskal-Wallis test and post hoc student Newman-Keuls test (α = 0.05). For the condition of TC0, the media of the groups (OA + S), (OA + P) and (DZ + S) resulted in higher union resistance than the other groups (OA + C), (DZ + C) and (DZ + P), being p 0.005. For the CT condition, all groups showed a significant decrease in media compared to those of 24h. However, the groups (OA + C) and (DZ + C) did not resist the cycles, and the microshear test was not performed. The results of the present study showed the efficiency of aluminum oxide blasting, together with other joining agents and with a more stable union resistance after thermocycling and further studies will be necessary for the use of zirconia dioxide paste and its ideal concentration.

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Key words: Ceramics, zirconia, surface treatment.

LISTA DE ABREVIATURAS

OA	Óxido de alumínio
DZ	Dióxido de zircônia
S	Silano
C	Controle
P	Primer
TC	Termociclagem
CAD/CAM	Computer Aid Design and Computer Aid Manufacturing
MPa	MegaPascal
***µ*SBS**	Microcisalhamento
h	Horas

LISTA DE TABELAS

Tabela 1. Materials used in the study 33

Tabela 2. Mean and standard deviation of the tested groups....................................... 33

LISTA DE FIGURAS

Imagem 1. Cimentação das amostras. A – Amostra da zircônia no momento da cimentação. B – Fotoativação do cimento resinoso. C – Corpos de prova após cimentação na superfície da zircônia. D – Armazenamento das amostras na estufa em temperatura de 37º **46**

Imagem 2. Teste de microcisalhamento. A – Máquina de teste. B – Cinzel de carga onde a amostra da cerâmica de zircônia foi presa a célula. C – Célula de carga presa ao corpo de prova até que ocorra falha. D – Amostra de zircônia após a falha adesiva **47**

Imagem 3. Análise após os testes. A – Imagem de magnitude 400x da amostra de zircônia após os testes. B – Lupa de aumento **48**

SUMÁRIO

1. INTRODUÇÃO 17

2. OBJETIVOS 22

3. CAPÍTULO 1: THE EFFECT OF SURFACE TREATMENT AND THERMOCYCLING ON THE BOND STRENGTH OF ULTRA-TRANSLUCENT ZIRCONIA CERAMIC AND RESIN CEMENT 24

Summary 26

1. Introduction 27

2. Materials e methods 28

2.1. Materials selection 28

2.2. Surface treatment procedures 28

2.3. Specimens cementation 29

2.4. Microshear test (µTBS) and aging 30

2.5 Statistical analysis 30

3. Results 30

4. Dicussion 31

5. Conclusion 32

Conflict of interest statement 33

References 35

4. CONCLUSÃO..38

5. REFERÊNCIAS...40

APÊNDICES..44

ANEXOS...48

1. Introdução

Uma ampla variedade de materiais em odontologia tem sido explorada para substituir dentes perdidos, com o propósito de restaurar função e estética. Entre os materiais dentários, a cerâmica tem se destacado devido sua diversidade de fabricação, e propriedades desejáveis (GALI; K, 2019). Para Venâncio *et al.,* em 2013, a cerâmica é o material que reproduz melhor as características ópticas do esmalte e da dentina, como fluorescência, opalescência e translucidez, bem como biocompatibilidade, alta resistência à compressão e à abrasão e estabilidade da cor.

O termo cerâmica vem do grego *κέραμος*, que significa matéria-prima queimada, ou seja, para que esse material obtenha propriedades desejáveis, é necessário que ocorra sua queima (BORGES *et al.,2015*). As cerâmicas odontológicas podem se apresentar de diversos tipos, exibindo propriedades químicas, mecânicas, físicas e térmicas que se diferenciam dos outros materiais, além de serem mais resistentes à corrosão e não reagem de imediato com a maioria de líquidos, gases, bases ou ácidos fracos (ANUSAVICE *et al*., 2013).

No fim do século XX, vários sistemas inovadores foram introduzidos para a fabricação de restaurações dentárias de cerâmica pura, que possibilitam um preparo na mesma altura da margem gengival livre, o que leva a um melhor resultado estético sem invadir o espaço biológico (LODI *et al*., 2017), além de que possui uma melhor adaptação em relação aos materiais metalocerâmicos (BONFANTE *et al.,* 2010). De início foi usado um sistema cerâmico-vítreo passível de fundição, no qual a cerâmica era fundida utilizando a técnica de cera perdida e depois recebia tratamento térmico para promover a sua transformação em cerâmica com estrutura vítrea. Com as dificuldades do processamento e alta incidência de fraturas devido a friabilidade do material, esse sistema foi abandonado, permitindo que outros processos fossem mais utilizados para as confecções (POWER; SAGAKUCHI, 2012).

As cerâmicas podem ser processadas de diferentes maneiras, sendo tradicionalmente aplicadas em camadas ou sobre uma estrutura metálica ou troquel refratário e posteriormente sinterizadas, melhorando a qualidade final do revestimento (SILVA *et al.,2017)*. Com a evolução do processamento surgiram os métodos de prensagem isostática e CAD/CAM.

Montazerian e Zanotto, em 2016, afirmaram que as cerâmicas podem ser classificadas de acordo com a presença ou ausência de uma fase da matriz de vidro ou de acordo que o material contém uma matriz orgânica altamente preenchida com partículas de cerâmica. Segundo Gracis *et al.,* em 2015, o sistema de classificação das cerâmicas frequentemente usado por Kelly e Benetti, em 2011, pode gerar confusão, uma vez que a cerâmica com predominância de vidro foi proposta de forma que correlaciona a quantidade de vidro e as características estéticas e mecânicas do material. Na fase vítrea, encontra-se propriedades de transmissão de luz, o que possibilita reproduzir as características da translucidez em vários níveis. Já na fase cristalina, o cristal é responsável pela resistência da propagação de trincas devido a formação de uma barreira protetora (MALHEIROS *et al*., 2013). A fabricação desses materiais foi sendo diferenciada, não apenas com componentes naturais como o feldspato, mas também o uso da cerâmica sintética. Isso resultou em uma melhoria na padronização e controle de qualidade desses materiais.

Uma das principais cerâmicas odontológicas é a zircônia que tem evoluído consideravelmente, uma vez que esse material tem resistência mecânica alta com força flexural entre 750 a 1200 Mpa. Tem sido estudada a possibilidade de uso da cerâmica de zircônia em vários tipos de restaurações, inclusive na confecção de facetas devido ao aumento do conteúdo na estabilização por óxido de ítrio (RANGANATHAN *et al., 2017)*. A translucidez das zircônias ultra-translúcidas aumentou suas frações de volume de fase cúbica opticamente isotrópica (> 50%) (YAN *et al*., 2018), aumentando de 3% mol para 4% mol (4Y-PSZ) ou 5% mol (5Y-PSZ), o que minimiza sua opacidade (LAWSON et al., 2020), tornando-a apropriada para restaurações anteriores monolíticas (MONTEIRO e*t al.,* 2020).

Todavia, há controvérsia em relação à resistência de união da zircônia, uma vez é inerte, não podendo ser condicionada com ácido fluorídrico (4% - 10%) (JABBAR; DULAMI, 2020). Diante disso, segundo Tholey *et al.,* em 2011, a demanda da cerâmica de zircônia diminuiu para menos de 25%, devido grande preocupação clínica sobre as falhas adesivas, estabilidade a longo prazo, e ocorrência de lascamento. Apesar da falta de informação sobre a maioria dos procedimentos de ligação com eficiência e durabilidade, tem se estudado diversos tratamentos como forma de auxiliar a resistência de união, como deposição de nano-filme de óxidos de silício, a técnica de glaze-on, silanos de aquecimento, condicionamento químico usado para melhor ligação à zircônia (MELO *et al*., 2015), jateamento com óxido de alumínio, cimento resinoso contendo 10-metacriloxidecil di-hidrogênio monômero de fosfato (10-MDP), primers universais também contendo monômeros de metacrilato, processamento por plasma, infiltração de sílica pelo método sol-gel, infiltração de vidro feldspático, entre outros (SOUZA *et al., 2018)*.

Segundo Aung *et al.,* em 2019, o 10-MDP tem apresentado eficácia para promover união à zircônia, um monômero adesivo até então elaborado para se ligar à estrutura dental e ao metal. Tanis e Akcaboy, em 2015, demonstraram que o uso de cimento resinoso contendo 10-MDP tem aumentado significativamente a resistência de união em zircônias que também receberam jateamento com óxido de alumínio.

O uso de jateamento de óxido de alumínio é um assunto muito discutido devido suas controversas na literatura (LIMA, 2018). De acordo com Inokoshi *et al.,* em 2014, o jateamento do óxido de alumínio com de partículas de 30 µm na superfície da cerâmica de zircônia com associação de primers/cimentos à base de MDP, pode associar em uma união mais durável à estrutura.

Seguindo os estudos de Jo *et al*., em 2020, o tratamento da superfície da zircônia com a pasta de dióxido de zircônia (ZrO_2) e nanopartículas de carbono, pode aumentar a resistência de união entre a zircônia e a estrutura dental, sendo realizado sobre a superfície da zircônia antes do processo de sinterização. As partículas de ZrO_2 que possui tamanho nanométrico, podem se conectar à superfície da zircônia por ligações de hidrogênio, o que permite que a partícula de carbono produza um material mais poroso após sofrer sinterização.

Além do tratamento de superfície da zircônia, outro fator de grande relevância clínica é o envelhecimento por meio das condições térmicas intraorais (ELIASSON; DAHL, 2019). Gale e Darvell, em 1998, já haviam avaliado que as mudanças de temperatura no ambiente bucal poderiam ser alteradas através dos alimentos, bebidas e até mesmo pela respiração, que podem provocar tensões mecânicas e comprometerem a retenção e resistência.

Os principais pontos de interesse foram amplamente discutidos, a fim de traçar recomendações clínicas com base em evidências científicas (STAPPERT *et al.*, 2005*)* e, quando ausentes, na prática clínica, juntamente com comunidade científica (ZARONE *et al*., 2019).

Diante disso, objetivo desse trabalho foi avaliar o efeito do tratamento de superfície e da termociclagem na resistência de união de uma cerâmica de zircônia ultra-translúcida. A hipótese nula investigada mostra se ambos materiais possuem resistência iguais ou parecidas para uma resistência de união favorável.

2. Objetivos

O objetivo desse estudo foi avaliar o efeito do tratamento de superfície e da termociclagem na resistência de união de uma cerâmica de zircônia ultra-translúcida e cimento resinoso.

Identificar qual tratamento de superfície será mais eficiente, sendo eles o condicionamento com dióxido de zircônia e jateamento com óxido de alumínio.

Avaliar o efeito da ciclagem térmica após a cerâmica ser tratada e cimentada, e qual tratamento ofereceu melhor resistência de união por meio do teste de microcisalhamento.

Capítulo 1: Artigo

TITLE: The effect of surface treatment and thermocycling on the bond strength of ultra-translucent zirconia ceramic and resin cement

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RUNNING TITLE: Bond strength of zirconia ceramics before and after thermal fatigue

CLINICAL RELEVANCE: When zirconia receives a surface treatment, it improves its bond strength to the tooth structure.

SUMMARY

Objective: To evaluate the bond strength between an ultra-translucent zirconia ceramic and the cementing agent under different surface treatments after 24 hours of water storage (24h) and after 10,000 thermocycles (TC), to evaluate the bonding interface. Materials and methods: One hundred and twenty zirconia samples were prepared and divided into two groups to receive a surface treatment: sandblasting with aluminum oxide (AO) and etching with zirconia dioxide (ZD). Zirconia sections (n = 10) were assigned to 12 groups according to two factors: 1) treatment (control [C], silane [S] and primer [P]) 2) thermocycling (no thermocycling [TC_0], 10,000 thermocycles [TC]). For the application of resin cement, polyvinylsiloxane molds were placed on the ceramic surface to determine the adhesion area. Excess cement was removed, then photoactivated for 20 s with a LED light source. After removal of the silicone matrix, the samples were tested in microshear (*μ*SBS) 1.0 mm/min cross speed after 24h or TC. Data were analyzed using Kruskal-Wallis and Student-Newman-Keuls post hoc test (α = 0.05). **Results:** For the TC_0 condition, the mean of the groups (AO + S), (AO + P) and (DZ + S) resulted in higher μTBS than the other groups (AO + C), (ZD + C) and (ZD + P), where p 0.005. For the TC condition, all groups showed a significant decrease in the mean compared to those at 24h p 005. However, the groups (OA + C + TC) and (DZ + C + TC) did not resist the cycles, and the μTBS could not be performed. **Conclusions:** The results of the present study showed the effectiveness of aluminum oxide blasting, together with other bonding agents and with a more stable bond strength after thermocycling. However, more studies are needed on zirconia surface treatment.

Keywords: Ceramics, zirconia, surface treatment.

1 INTRODUCTION

Dental ceramics have stood out due to their manufacturing diversity and desirable properties so that they can replace lost teeth, restoring function and aesthetics.[1,2] It is the material that best reproduces optical characteristics of enamel and dentin, such as fluorescence, opalescence and translucency, as well as biocompatibility, high compressive and abrasion strength and color stability. [2]

For this material to obtain desirable properties, it is necessary to burn it,[3] and with this, it is possible to present itself in various ways, exhibiting chemical, mechanical, physical and thermal properties that differ from other materials. [4] Several systems were introduced at the end of the 20th century for the manufacture of pure ceramics, which offer a better adaptation in relation to metalloceramic materials. [5.6]

The ceramic-vitreous system initially used was abandoned due to processing difficulties and high incidence to fractures, which allowed new forms of ceramic making. [7] Therefore, today ceramics can be processed in different ways, improving the final quality of the coating, especially with the use of CAD/CAM. [7,8] In addition, they can be classified according to the presence or absence of the glass matrix or according to which the material contains an organic matrix highly filled with ceramic particles, since the classification system can generate confusion, and ceramics with predominance of glass correlate the amount of glass and the aesthetic and mechanical characteristics of the material[9,10,11,12] The manufacture of these materials was differentiated, not only with natural components such as feldspar, but also the use of synthetic ceramics. This resulted in an improvement in the standardization and quality control of these materials.

Zirconia is one of the ceramics that has evolved considerably, since it has high mechanical resistance with flexural strength between 750 to 1200 Mpa. With greater translucency through the increase of the content in the stabilization by yttrium, the possibility of using this ceramic for aesthetic purposes has been researched.[13] This translucency, especially in ultra-translucent zirconia, increased its volume fraction to optically isotropic cubic phase (> 50%),[13,14] increasing from 3% mol to 4% mol (4Y-PSZ) or 5% mol (5Y-PSZ), which minimizes its opacity making it suitable for monolithic previous restorations. [15.16]

However, there is controversy regarding the bond strength of zirconia, once it is inert and cannot be conditioned with hydrofluoric acid (4% - 10%)[17,18] In view of this, there is a decrease in the demand of zirconia ceramics due to great clinical concern about adhesive failures, long-term stability, and occurrence of chipping. [19] Despite the lack of information on most connection procedures with efficiency and durability, several treatments have been studied as a way to aid the bond strength, one of them is aluminum oxide blasting and zirconia dioxide conditioning, the object of this study. [19,20] The use of aluminum oxide blasting is a subject much discussed due to its controversies in the literature [23].

In addition to the surface treatment of zirconia, another factor of great clinical relevance is aging through intraoral thermal conditions.[26] Gale and Darvell, in 1998, had already evaluated that temperature changes in the oral environment could be altered through food, beverages and even breathing, which can cause mechanical tensions and compromise retention and resistance. [27]

The main points of interest were widely discussed in order to outline clinical recommendations based on scientific evidence and clinical practice. [29,30] Therefore, the objective of this work was to evaluate the effect of surface treatment and thermocycling on the union strength of an ultra-translucent zirconia ceramic and a resin cement. The null hypothesis investigated shows whether both materials have equal or similar resistance to a favorable union resistance.

2 MATERIALS AND METHODS

2.1 Materials selection

The table 1 shows the materials and their respective manufacturers that were used in the study.

2.2 Surface treatment procedures

A total of 120 samples of high translucent zirconia ceramics (ZirkOM SHT, Odontomega, German) with dimensions 12 mm in width, x 12 mm in depth and 1 mm in thickness were fabricated, following the manufacturer's instructions. After preparation and sintering, the samples were divided into two groups of sixty each, where one group received a surface treatment with blasting of 50µm aluminum oxide (AO) particles (BioArt, São Carlos, Brazil) at a pressure of 5 bar at a distance of 1cm and a sandblasting angle of 45° in relation to the ceramic surface for one minute. The other pre-sintered group will receive, as a surface treatment, application of a zirconia dioxide paste [ZD] (ZirADD, PNUADD, Busan, Korea) at a concentration of 3.0 g per 100 g of distilled water. The disks with ZrO_2 paste were taken to the furnace for sintering of the paste in a specific guide (Zirkonzahn, Bolzano, Italy) at a temperature of 1530ºC for 12 hours. Then, the two groups were subdivided into three subgroups of twenty samples each. The first subgroup, being the control group (C) received no specific treatment. The second subgroup was the silane (S) (Monobond N, Ivoclar Vivadent, AG Schaan, Liechtenstein) which has a bifunctional molecule that acts as a binding agent. With a fine tip microbrush (KG Sorensen, Cotia, São Paulo, Brazil) it was applied over the ceramic surface, removing the excess with a triple syringe jet for 10 seconds, and letting the silanic agent act for 5 minutes. The third group used a ceramic primer (P) with MDP monomer (Clearfil Ceramic Primer, Kuraray, Okayama, Japan). It was passed over the ceramic surface with a microbrush, removing the excess with a triple syringe jet for 10 seconds.

2.3 Specimens cementation

For the application of resin cement (Variolink Esthetic LC, Ivoclar Vivadent AG Schaan, Liechtenstein), polyvinylsiloxane molds (Virtual, Ivoclar Vivadent AG Schaan, Liechtenstein), 0.5 mm thick, with four cylindrical holes (0, 8 mm in diameter) were fabricated and placed on the ceramic surface to determine the bonding area. The resin cement was prepared according to the manufacturer's instructions and inserted into the mold hole with a dentin spoon (Duflex – Juiz de Fora, Minas Gerais, Brazil). Excess cement was removed with a spatula #24 (Duflex, Juiz de Fora, Minas Gerais, Brazil). The holes were filled with resin cement and a polyester transparent strip was placed over the filled holes. A constant and uniform load of 454 gF was applied for 1 min using a custom-made device. The resin cement was photoactivated for 20 s in continuous mode with a LED light source (Bluephase, Ivoclar Vivadent AG Schaan, Liechtenstein) with an irradiance of 800 mW / cm^2, verified with a radiometer (Kerr Sybron, Anaheim, CA, USA). After 10 minutes, the silicone matrix was removed, and the cement cylinders were carefully evaluated under an optical microscope to observe the bonding area. Then, they were stored for 24 hours at 37ºC, 100% relative humidity until the bond strength test.

*2.4 Microshear test (µ*SBS*) and aging*

After 24 hours, the groups that underwent thermal cycling (MSCT-3, Marcelo Nucci ME, São Carlos, São Paulo, Brazil) received 10,000 cycles (TC), 10 s in hot water (55ºC) and 10 s in cold water (5ºC) and then to the microshear test (µ SBS) (figure 1). The test was performed in a test machine (EMIC DL 3000® - EMIC - Equipment and Testing Systems Ltda., São José dos Pinhais, Brazil) with a load cell of 50 kgf. A stainless-steel chisel was attached to the load cell and the test was performed at 0.5 mm/min of cross-velocity until failure occurred. The remaining groups without thermal cycling were submitted only to the microshear test. The average of each resin cement cylinder in the ceramic samples was calculated to obtain the average value of the strength of the title of each block. The test machine software has been configured to provide results in MPa.

2.5 Statistical analysis

Statistical analysis was performed using SPSS. Statistical analysis for bond strength was performed using zirconia as an experimental. The average µTBS obtained from the cylinders of each zirconia sample was used to represent the bond strength. The Shapiro Wilk test was applied to verify normality, where the non-normal distribution can be affirmed for values of $p < 0.05$. The Kruskal-Wallis test was performed to compare the groups available at $p = 0.000$. To compare the average ranks, the post-hoc student-Newman-Keuls was used, with a significance level α of 5%.

3 RESULTS

For the TC_0 condition, it can be said that the AO + S, AO + P and ZD + S groups obtained greater bond strength with statistically equal values ($p > 0.05$). For the TC condition, all groups showed a significant decrease in the mean compared to those at 24h, with the ZD + S + TC and ZD + P + TC groups having the lowest results ($p<0.005$). The AO + C, AO + S + TC, AO + P + TC and ZD + S groups did not show statistical differences. The AO + C + TC and ZD + C + TC groups did not resist the thermocycling test, losing all the specimens (Table 2).

4 DISCUSSION

The present study evaluated the effect of surface treatment and thermocycling on the union strength of ultra-translucent zirconia ceramics and resin cement. After cementing the samples and their respective assays, it was found that the groups that received blasting as treatment resulted in greater union resistance between their certain agents. The present results are in accordance with Le et. al (2019), which showed that aluminum oxide blasting is a micromechanical procedure that aims to increase roughness, allowing greater increase in surface energy and cleaning, and when combined with an adhesive agent result in greater strength.[26] However, despite increasing the union resistance, blasting can influence the creation of microcracks that, when propagated, can cause cracks and fractures.[27] It is noteworthy that the size of the aluminum oxide grain is extremely important, together with the pressure applied at the time of blasting so that it does not become harmful. Particles larger than 100 μm have been shown to result in degradation and reduction of ceramic strength. Al-Harbi et al. (2016), reported that it is possible to strengthen ceramics with the use of self-adhesive cements for penetration of cracks. Therefore, aluminum oxide blasting should be used with caution, always following the manufacturer's recommendations.[28]

Compared to the study by Jo et al, the treatment of the zirconia surface with zirconia paste ($ZrO2$) and carbon nanoparticles is recommended its use before the sintering process. [29] and these $ZrO2$ particles have a nanometric size, which can connect to the surface of zirconia by hydrogen bonds, which allows the carbon particle to produce a more porous material after being sintered. The use of zirconia dioxide paste resulted in low union resistance among the other agents used after treatment, and the same study did not report having performed the aging test for long-term analysis. The paste used in the study at the concentration of 3.0 g per 100 g of distilled water, which was not as effective. It needs further studies on the product and which concentrations would be ideal to achieve a favorable union resistance. In this sense, the present results would not recommend the use of zirconia dioxide paste.

Among the surface treatments, we subdivided the groups for the use of a joining agent (silane and ceramic primer). The use of a ceramic primer with 10-MDP associated with aluminum oxide blasting is characterized by high union strength.

Primers containing 10-MDP are widely used in dentistry, as they increase the wettability of the structure and create a stability that takes place through its chemical structure formed by an extensive hydrophobic chain carbon spacer, connecting the phosphate group to metal oxides,[30,31,32] and thus promotes strong connections between dental substrate and material. In addition to its lower water absorption due to its hydrophobic characteristic.[33.34] The use of Silane Monobond N (Ivoclar Vivadent AG Schaan, Liechtenstein) has brought significant results for both types of zirconia surface treatment, even though some studies report that it is more efficient in ceramics with vitreous structures. [35] It has been shown that silane molecules can be activated by the hydrolysis process to effect silanization of the ceramic surface. In this sense as monobond n composition, it is a metallic primer composed of three different functional monomers, namely silane methacrylast, phosphoric methacrytoand sulfide methacrylet, can say that it is capable of providing a chemical connection with the zirconium oxide layer present on the surface of 3Y-TZP (zirconia). [36] It can also be said that the pH of the solution used influences the union strength of the zirconia structure, since a process of hydrolysis of the component occurs in the middle of the acid solution. [37]

In this perspective, the results found for ultra-translucency zirconia ceramics do not differ from other zirconia. In fact, zirconia ceramics were considerably altered in terms of aesthetic result, however, the characteristics of the internal surface for cementation were not altered, which the results of the present study confirm. In view of this, the results of this study cannot simply be applied directly to clinical practice, which does not preclude its use. Zirconia ceramics to be used for example with laminated facets need to be cemented adhesively, but the results of the analysis done here with this translucent ceramic made it clear that the clinical application of this type of ceramic for restorations without good retention should not be recommended even with primers that promote better support.

5 CONCLUSION

The results of the present study showed the efficacy of aluminum oxide blasting, together with other joining agents and with a more stable bond strength after thermocycling. Further studies on the conditioning of zirconia dioxide and its concentration may be more effective for better bond strength are needed.

Conflict of interest statement

All authors declare no potential conflicts of interest with respect to the authorship and/or puclication of this article.

Materials	Manufacturer	Composition
Zirconia Ceramic	ZirkOM SHT, Odontomega, German	Zirconia, Itrium.
Zirconia oxide slurry	ZirADD PNUADD, Busan, Korea	zirconia and nanopartcles of carbon.
Ceramic Primer	Clearfil Ceramic Primer, Kuraray Okayama, Japan	3-Metacriloxipropiltrim etoxissilano, 10-Metacriloiloxidecil di-hidrogephosphate (MDP), etanol.
Silane	Monobond N ®, Ivoclar Vivadent, AG Schaan, Liechtenstein	Alcoholic solution of silane methacrylet, phosphoric acid methacryletand sulfide methacrylet.
Aluminum oxide particle	BioArt ® São Carlos, Brasil	Aluminum oxide
Polivilsiloxane	Virtual, Ivoclar Vivadent, AG Schaan, Liechtenstein	Polivinilsiloxane, silicone dioxide, aluminum inorganic filler (CAS 1318-02-1), metil hidrogen siloxane.
Resin cement	Variolink Esthetic LC ®, IvoclarVivadent, AG Schaan, Liechtenstein	Uretane Dimethacrylate and other methacrylayte monomers, trifluorete of iterbium and mixed espheroid oxides, initiators, stabilizers, and pigments.

Table 1. Materials used in the study.

Condition	**Surface treatment**					
	Aluminum oxide			**Zirconia dioxide**		
	Control	**Silane**	**Primer**	**Control**	**Silane**	**Primer**
24 hours	2,27(0,67-4,33)^{C}	6,61(3,78-16,4)^{A}	7,82(1,75-23,3)^{A}	1,12(0,17-3,42)^{C}	6,21(1,82-21,5)^{AB}	4,16(0,52 9,46)^{B}
Thermocycling	x	2,55(1,19–4,09)^{C}	2,01(0,41-5,06)^{C}	x	0,46(0,24-0,96)^{D}	,58(0,22-1,74$^{)D}$

Table 2. Median, maximum and minimum of the tested groups. Capital letters equal, denote there is no significant difference between comparisons (p>0.05).

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Conclusão

Portanto, de acordo com a metodologia empregada, e com as limitações do presente estudo, concluímos que o tratamento com óxido de alumínio resultou em uma maior resistência de união associado a outros agentes e melhores resultados aos testes de envelhecimento. Serão necessários mais estudos sobre o condicionamento com dióxido de zircônia e sua concentração ideal para a avaliação de resistência de união sobre a estrutura da zircônia.

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Apêndice

Apêndice A – Cimentação das amostras

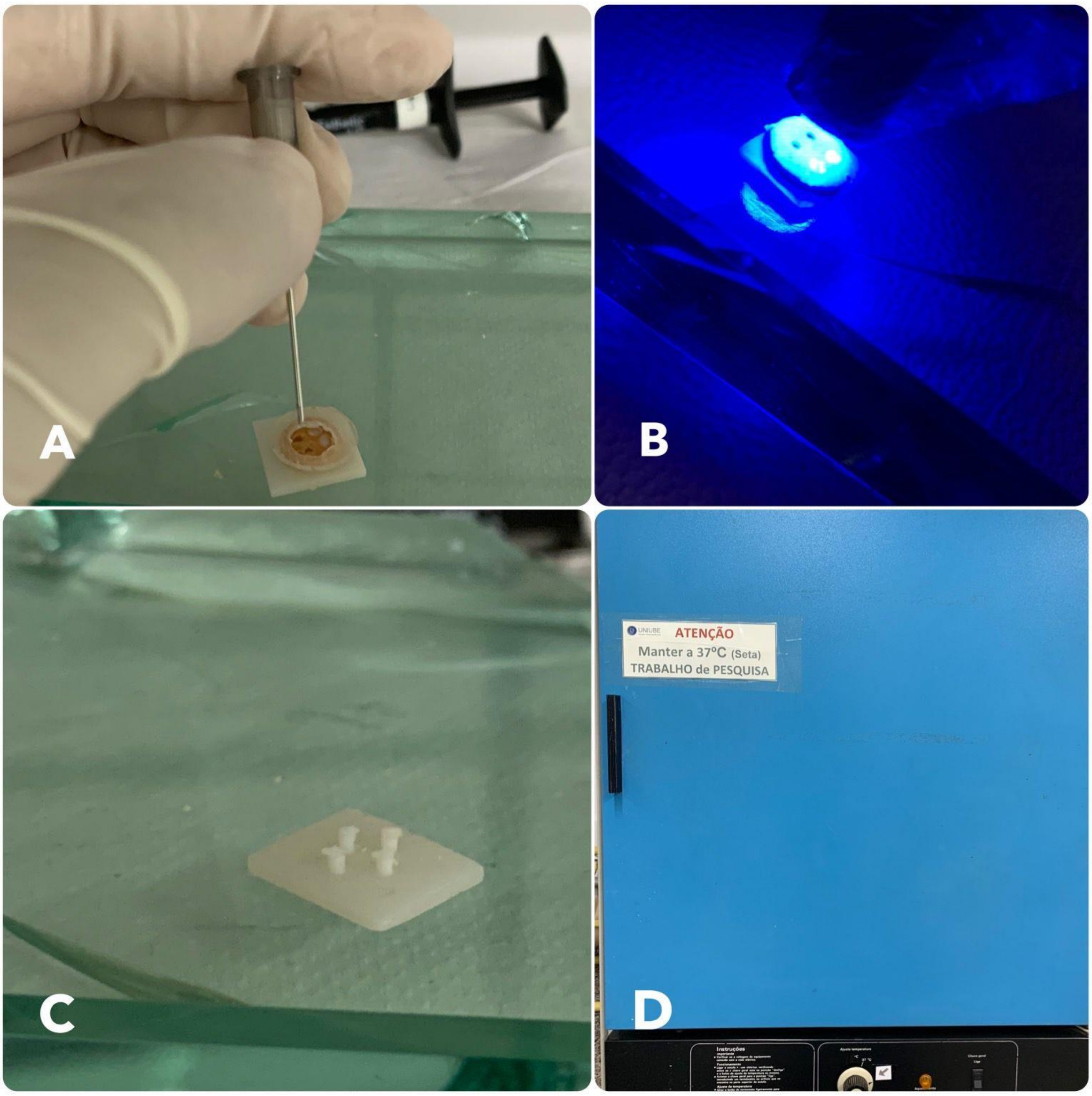

Imagem 1. Cimentação das amostras. A – Amostra da zircônia no momento da cimentação. B – Fotoativação do cimento resinoso. C – Corpos de prova após cimentação na superfície da zircônia. D – Armazenamento das amostras na estufa em temperatura de 37º.

APÊNDICE B – Teste de microcisalhamento

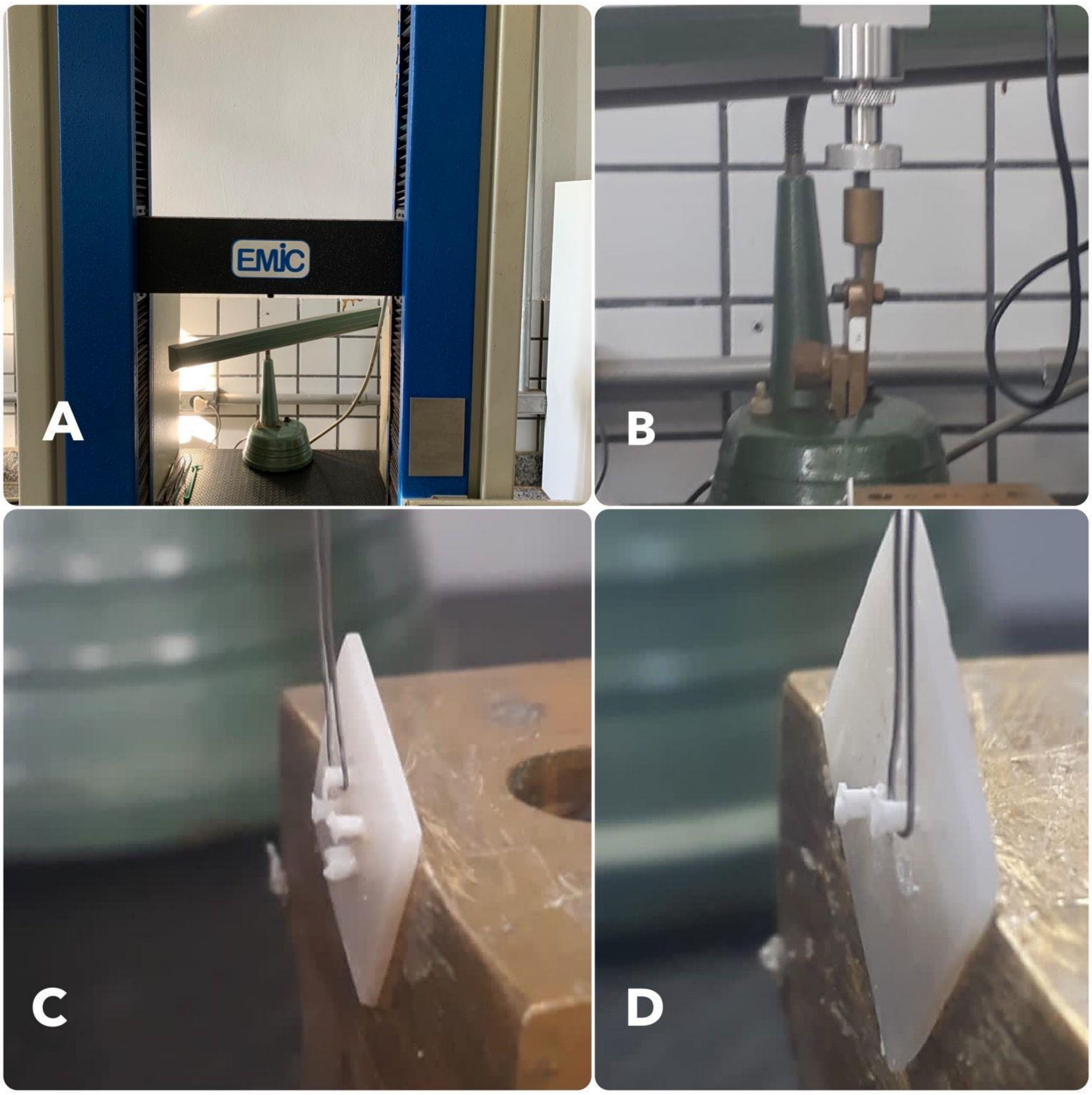

Imagem 2. Teste de microcisalhamento. A – Máquina de teste. B – Cinzel de carga onde a amostra da cerâmica de zircônia foi presa a célula. C – Célula de carga presa ao corpo de prova até que ocorra falha. D – Amostra de zircônia após a falha.

APÊNDICE C – Análise após os testes

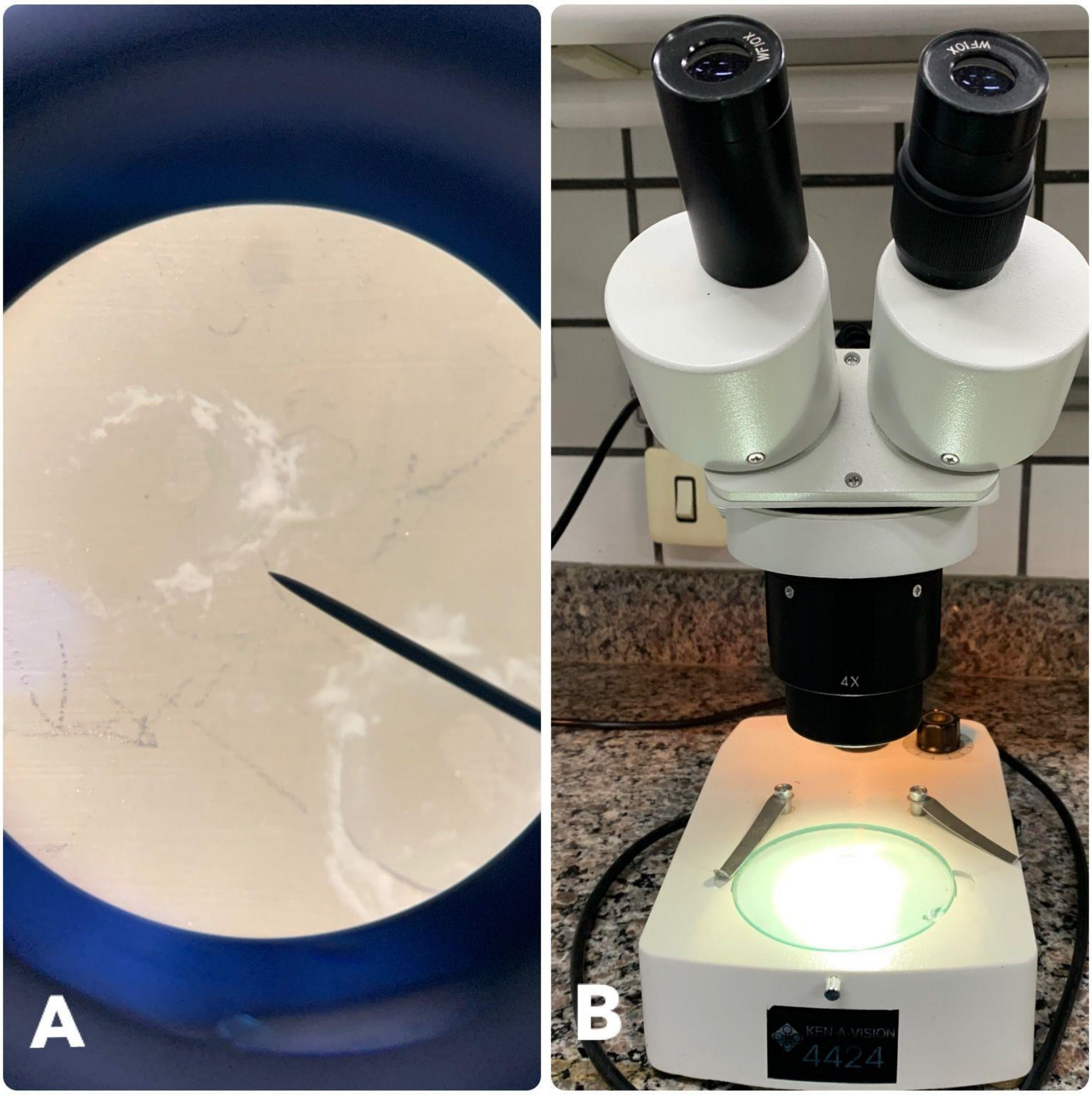

Imagem 3. Análise após os testes. A – Imagem de 400x da amostra de zircônia após os testes. B – Lupa de aumento.

Anexos

Instructions to Authors

This instruction page has been streamlined to present the minimum guidelines for a submitted article to be considered for review and possible publication.

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- a **conclusion**
- **references**