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EDMAR CURTO ALBERTO JUNIOR

RESISTÊNCIA DE UNIÃO ENTRE CERÂMICA DE DISSILICATO DE LÍTIO E ZIRCÔNIA E UM CIMENTO RESINOSO ANTES E APÓS CICLAGEM TÉRMICA

BOND STRENGTH BETWEEN LITHIUM DISILICATE AND ZIRCONIA CERAMIC TO A RESIN CEMENT BEFORE AND AFTER THERMAL CYCLING

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Dissertação apresentada como parte dos requisitos para obtenção de título de mestre em Clínica Odontológica Integrada do Programa de Pós-graduação em Odontologia da Universidade de Uberaba (PPGODO/UNIUBE).
Orientador: Prof. Dr. Gilberto Antônio Borges

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Área de concentração: Clínica Odontológica Integrada

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Dedico este Trabalho de Conclusão de Curso à minha família, cuja dedicação, amor e apoio incondicional foram a base de minha jornada acadêmica. A vocês, que sempre acreditaram em mim e me incentivaram a perseguir meus sonhos, dedico este trabalho com profundo carinho e gratidão.

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RESUMO

O objetivo deste trabalho foi avaliar a resistência de união ao microcisalhamento (RUμC) entre um cimento resinoso fotoativado e duas cerâmicas: dissilicato de lítio (DL) e zircônia (Z), imediatamente após cimentação, após 24 h e termociclagem. Foram confeccionados 10 espécimes de cada cerâmica com espessura de 0,5 mm a partir de blocos CAD/CAM com auxílio de disco diamantado em máquina de corte de precisão. A superfície de DL foi condicionada com ácido hidrofluorídrico 10%, seguida de aplicação de agente de união com silano e adesivo de esmalte. A superfície de Z, foi jateada óxido de alumínio de 50 μm, seguida de aplicação de primer cerâmico à base de MDP (10- metacriloiloxidecil di-hidrogênio fosfato) e adesivo de esmalte. Os espécimes foram divididos em três grupos para cada tipo de cerâmica, conforme as seguintes condições: 1. imediato (DL_Im e Z_Im), 2. após 24 horas (DL_24h e Z_24h) e 3. após ciclagem térmica (DL_ApTerm e Z_ApTerm). Seis cilindros de cimento resinoso foram unidos à superfície cerâmica e cada dois cilindros foram testados imediatamente, enquanto os quatro restantes foram armazenados em água destilada à 37 °C. Após 24 horas, dois cilindros foram testados, e os dois cilindros remanescentes foram submetidos a 10.000 ciclos térmicos, alternando entre 5 °C e 55 °C. o teste de união foi realizado por microcisalhamento em máquina de ensaio universal. Modos de falha dos corpos de prova foram analisados e classificados. Microscopia eletrônica de varredura (MEV) da interface de união entre cerâmicas e cimento resinoso foi realizada. Médias e desvios padrão de RUμC foram analisados por ANOVA de medidas repetidas de 2 fatores, seguida de teste post hoc de Tukey ($p \leq 0,05$). DL apresentou valores de RUμC significativamente superiores à Z para todas as condições. DL_24h (38±7.48) apresentou RUμC significativamente superior ao DL_Im (31±6.62), porém DL_ApTerm (25,5±5.98) foi significativmnte menor que ambos. RUμC de Z_24h (30,3±6.52) foi significativamente superior ao Z_Im (21,6±4.61), contudo Z_ApTerm (4,78±1.11) foi significativamente inferior ao outros. Z apresentou, predominantemente, falhas adesivas, principalmente após ciclos térmicos, assim como DL após os ciclos. Imagens de MEV mostram diferentes condições das interfaces de união. Em conclusão, DL proporciona RUμC superior à Z. Ambas cerâmicas apresentaram uma diminuição na RUμC após ciclos térmicos. No entanto, a união à Z foi mais afetada pelo envelhecimento térmico do que DL.

Palavras-chave: Cerâmica. Zircônia Dentária. Cimentos de Resina. Cimentação. Resistência ao Cisalhamento. Microscopia Eletrônica de Varredura.

ABSTRACT

The aim of this study was to evaluate the microshear bond strength (μSBS) between a light-cured resin cement and two ceramics: lithium disilicate (LD) and zirconia (Z), immediately after cementation, after 24 h, and after thermocycling. Ten specimens of each ceramic (thickness = 0.5 mm) were obtained from CAD/CAM blocks using a precision cutting machine with a diamond disc. The LD surface was etched with 10% hydrofluoric acid, followed by the application of a silane coupling agent and an enamel adhesive. The Z surface was air-abraded with 50 μm aluminum oxide particles, followed by the application of an MDP-based ceramic primer (10-methacryloyloxydecyl dihydrogen phosphate) and an enamel adhesive. The specimens were divided into three groups for each ceramic according to the following conditions: 1. immediate (LD_Im and Z_Im), 2. after 24 hours (LD_24h and Z_24h), and 3. after thermocycling (LD_Therm and Z_Therm). Six resin cement cylinders were bonded to each ceramic surface; two cylinders were tested immediately, while the remaining four were stored in distilled water at 37 °C. After 24 hours, two cylinders were tested, and the remaining two were subjected to 10,000 thermal cycles between 5 °C and 55 °C. Bond strength was evaluated by microshear testing using a universal testing machine. Failure modes were analyzed and classified. Scanning electron microscopy (SEM) of the bonding interface between the ceramics and the resin cement was performed. Means and standard deviations of μSBS were analyzed using two-way repeated measures ANOVA, followed by Tukey's post hoc test ($p \leq 0.05$). LD showed significantly higher μSBS values than Z under all conditions. The DL_24h (38 ± 7.48) exhibited a significantly higher RUμC compared to DL_Im (31 ± 6.62), whereas DL_ApTerm (25.5 ± 5.98) was significantly lower than both. The RUμC of Z_24h (30.3 ± 6.52) was significantly higher than Z_Im (21.6 ± 4.61), yet Z_ApTerm (4.78 ± 1.11) was significantly lower than the others. Z predominantly exhibited adhesive failures, especially after thermocycling, as did LD after aging. SEM images revealed different conditions at the bonding interfaces. In conclusion, LD provided higher μSBS than Z. Both ceramics exhibited decreased μSBS after thermocycling, but the bond to Z was more adversely affected by thermal aging than that to LD.

Key-words: Ceramics. Dental Porcelain. Resin Cements. Cementation. Shear Strength. Scanning Electron Microscopy.

SUMÁRIO

1 INTRODUÇÃO

As cerâmicas são comumente utilizadas para confecção de restaurações indiretas estéticas e seu uso tem aumentado exponencialmente nos últimos anos. São materiais biocompatíveis e estáveis quimicamente (WARRETH; ELKAREIMI, 2020). Conforme sua composição, no geral, podem ser divididas em cerâmicas vítreas ou policristalinas, e ainda em infiltradas por polímero. Cerâmicas vítreas e policristalinas são materiais inorgânicos não metálicos. As cerâmicas vítreas possuem uma fase de vidro e conteúdo cristalino variável, conforme o tipo e aplicação clínica. Enquanto as cerâmicas policristalinas têm sua composição o predomínio absoluto de cristais (GRACIS et al., 2016; WARRETH; ELKAREIMI, 2020).

A modalidade de tratamento restaurador que utiliza facetas de cerâmica é um método conservador, oferecendo previsibilidade e desempenho clínico de longo prazo (KELLY, 1999). Com os avanços tecnológicos e a evolução dos materiais, é possível utilizar facetas ultrafinas, com espessura de 0,1 a 0,3 mm, que podem ser cimentadas adesivamente na superfície dental com mínimo ou nenhum preparo, permitindo a modificação da cor, forma e posicionamento dos dentes (LOPES; SPOHR; DE SOUZA, 2016). Entretanto, o uso dessas facetas deve ser cuidadosamente avaliado, uma vez que a confiabilidade dos materiais diminui significativamente em regiões submetidas a cargas mais altas (BENALCAZAR JALKH et al., 2024).

A crescente demanda por restaurações estéticas tem impulsionado a popularidade das cerâmicas. No entanto, seu uso em próteses parciais fixas (PPFs), tem sido limitado, pois as cerâmicas convencionais apresentam propriedades mecânicas inferiores. A introdução de cerâmicas de alta resistência, como a zircônia, tem levado a avanços significativos nas restaurações completas de cerâmica (KWON et al., 2018). Essas cerâmicas apresentam melhor resistência mecânica devido ao seu alto teor de fase cristalina de óxido de alumínio ou óxido de zircônio, em comparação com as cerâmicas vítreas como feldspáticas, enriquecida por leucita ou dissilicato de lítio (ZHANG et al., 2016).

O dissilicato de lítio ($Li_2O_5Si_2$) é uma cerâmica vítrea amplamente utilizada em odontologia devido à sua combinação única de propriedades mecânicas e estéticas (ZHAO et al., 2021). Introduzido no mercado em 2005, o sistema IPS e.max® trouxe um padrão elevado de desempenho óptico e resistência mecânica, com flexibilidade para aplicações tanto em sistemas de prensagem quanto em tecnologias CAD/CAM (STREIT; SYKES, 2022). A microestrutura do dissilicato de lítio apresenta uma distribuição bimodal de grãos com cristais grandes e alongados, além de cristais menores, conferindo maior resistência à flexão

e tenacidade à fratura. Essa estrutura favorece a deflexão e o fechamento de trincas, características essenciais para o uso clínico em regiões anteriores e posteriores (ZHAO et al., 2021). Além disso, o dissilicato de lítio possui excelente estética, translucidez favorável e elevada taxa de sobrevivência clínica, mostrando apenas 0,1% de falha anual (MARGVELASHVILI-MALAMENT et al., 2024).

A zircônia é uma cerâmica de alta resistência que tem sido largamente utilizada, oferecendo, por exemplo, resistência à flexão (acima de 1000 MPa) e características ópticas desejáveis (HAN, 2024), embora a espessura da camada de cerâmica influencie a core da restauração desejada (TABATABAIAN et al., 2018). Embora coroas e PPFs feitos de zircônia sejam suficientemente resistentes para suportar cargas oclusais, a cimentação adesiva tem sido recomendada para melhorar a retenção, adaptação marginal e resistência à fratura (CAMPOS et al., 2017). O uso de cimentos resinosos contendo monômeros de fosfato tem sido sugerido na fixação de uma restauração de zircônia. A união ocorre devido à ligação direta do grupo éster do monômero com o óxido metálico (ROHR et al., 2018), entretanto o tratamento da superfície da cerâmica é um fator crucial para garantir a retenção mecânica e união química entre o cimento resinoso e a cerâmica (WOLFART et al., 2007).

Com base nas características de alta translucidez e adesão eficaz ao cimento resinoso, as cerâmicas têm sido utilizadas na confecção de facetas ultrafinas. A zircônia translúcida, em particular, tem sido considerada um material estético adequado para coroas, próteses fixas monolíticas e facetas, tanto anteriores como posteriores. No entanto, a falta de retenção mecânica em determinadas situações tem sido um desafio, uma vez que a zircônia é quimicamente estável e não pode ser condicionada por ácido hidrofluorídrico (SOUZA et al., 2018).

Para otimizar a adesão entre a zircônia e o cimento resinoso, diversos tratamentos de superfície têm sido propostos, incluindo jateamento com óxido de alumínio, revestimento de sílica seguido de silanização, revestimento de alumina nanoestruturada, uso de cimentos resinosos contendo monômero de metacriloxidecil dihidrogenofosfato (MDP), primers universais também contendo monômeros de metacrilato, processamento de plasma, infiltração de sílica pelo método sol-gel e infiltração de vidro feldspático, entre outros (LOPES; SPOHR; DE SOUZA, 2016; STEFANI et al., 2016).

Apesar dos avanços científicos, ainda são necessários estudos clínicos sobre o uso de facetas de zircônia e facetas ultrafinas de zircônia. Esses estudos ajudarão a compreender melhor a eficácia dessas restaurações e a sua durabilidade em longo prazo (MALGAJ et al., 2023).

Facetas e restaurações complexas de cerâmica, incluindo a zircônia translúcida, têm se destacado na odontologia estética devido à sua alta estética, ausência de metal e resistência mecânica. No entanto, estudos adicionais são necessários para validar e aprimorar essas técnicas e entender melhor a durabilidade das facetas de zircônia e facetas ultrafinas de zircônia.

Diante o contexto, o objetivo deste trabalho foi avaliar a resistência de união ao microcisalhamento (RUμC) entre um cimento resinoso às cerâmicas de dissilicato de lítio (DL) e zircônia (Z) em diferentes condições: imediata, após 24 horas e após ciclagem térmica. Além disso, avaliar, por microscopia eletrônica de varredura (MEV), a interface de união entre as cerâmicas e cimento resinoso. O trabalho foi desenvolvido em formato artigo científico e tem como hipóteses nulas: 1) a RUμC das cerâmicas avaliadas não será diferente estatisticamente e; 2) as diferentes condições não afetarão a RUμC entre cerâmicas e cimento resinoso.

2 ARTIGO: Bond Strength Between Lithium Disilicate and Zirconia Ceramic to a Resin Cement Before and After Thermal Cycling

2.1 ABSTRACT

The aim of this study was to evaluate the microshear bond strength (μSBS) between a light-cured resin cement and two ceramics: lithium disilicate (LD) and zirconia (Z), immediately after cementation, after 24 h, and after thermocycling. Ten specimens of each ceramic (thickness = 0.5 mm) were obtained from CAD/CAM blocks using a precision cutting machine with a diamond disc. The LD surface was etched with 10% hydrofluoric acid, followed by the application of a silane coupling agent and an enamel adhesive. The Z surface was air-abraded with 50 μm aluminum oxide particles, followed by the application of an MDP-based ceramic primer (10-methacryloyloxydecyl dihydrogen phosphate) and an enamel adhesive. The specimens were divided into three groups for each ceramic according to the following conditions: 1. immediate (LD_Im and Z_Im), 2. after 24 hours (LD_24h and Z_24h), and 3. after thermocycling (LD_Therm and Z_Therm). Six resin cement cylinders were bonded to each ceramic surface; two cylinders were tested immediately, while the remaining four were stored in distilled water at 37 °C. After 24 hours, two cylinders were tested, and the remaining two were subjected to 10,000 thermal cycles between 5 °C and 55 °C. Bond strength was evaluated by microshear testing using a universal testing machine. Failure modes were analyzed and classified. Scanning electron microscopy (SEM) of the bonding interface between the ceramics and the resin cement was performed. Means and standard deviations of μSBS were analyzed using two-way repeated measures ANOVA, followed by Tukey's post hoc test ($p \leq 0.05$). LD showed significantly higher μSBS values than Z under all conditions. The DL_24h (38 ± 7.48) exhibited a significantly higher RUμC compared to DL_Im (31 ± 6.62), whereas DL_ApTerm (25.5 ± 5.98) was significantly lower than both. The RUμC of Z_24h (30.3 ± 6.52) was significantly higher than Z_Im (21.6 ± 4.61), yet Z_ApTerm (4.78 ± 1.11) was significantly lower than the others. Z predominantly exhibited adhesive failures, especially after thermocycling, as did LD after aging. SEM images revealed different conditions at the bonding interfaces. In conclusion, LD provided higher μSBS than Z. Both ceramics exhibited decreased μSBS after thermocycling, but the bond to Z was more adversely affected by thermal aging than that to LD.

Key-words: Ceramics. Dental Porcelain. Resin Cements. Cementation. Shear Strength. Scanning Electron Microscopy.

2.2 INTRODUCTION

With the growing popularity of all-ceramic dental restorations, achieving effective bonding between ceramic materials and resin luting cements has become a key determinant of their long-term clinical success [1]. The selection of ceramic materials for indirect dental restorations is influenced by multiple factors, including mechanical and aesthetic properties as well as material composition [2].

Lithium disilicate (LD) and zirconia (Z) ceramics are widely utilized due to their advantageous combination of strength and aesthetics [3]. LD ceramics consist of a glass matrix reinforced with disilicate crystals, offering high translucency and excellent resemblance to natural teeth, making them ideal for restorations in highly aesthetic regions [4]. Conversely, zirconia, particularly yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), is prized for its exceptional fracture resistance and durability, making it well-suited for areas subject to higher masticatory forces, such as posterior crowns [5]. Although zirconia typically exhibits lower translucency than LD, advancements in material formulations have expanded its application to include aesthetic regions [6].

The bonding mechanisms between resin luting cements and ceramics differ based on their surface composition and morphology. LD, with its glassy structure, can be etched using hydrofluoric acid (HF) to create microporosities that enhance micromechanical retention. The application of a silane coupling agent further promotes chemical bonding between the ceramic and the resin cement, ensuring a robust and durable interface [7]. This combination of acid etching and silane application is critical to achieving optimal adhesion in LD-based restorations [8–10].

Zirconia presents unique challenges due to its dense crystalline structure and lack of a glass phase, which makes it resistant to conventional acid etching [11]. Bonding to zirconia primarily relies on mechanical retention through sandblasting with aluminum oxide particles (Al_2O_3) to roughen the surface, paired with the application of phosphate monomer-containing primers, such as 10-MDP (methacryloyloxydecyl dihydrogen phosphate), which establish chemical interactions with the resin cement [12].

Several factors, including the type, thickness, translucency, and color of ceramics, influence the polymerization efficiency of resin cements and, consequently, the longevity of the restoration [13]. Effective polymerization is essential to prevent marginal leakage, early debonding, and failure of the adhesive interface. Conversely, incomplete polymerization

weakens the resin matrix, reduces cohesive polymer chain formation, and compromises the material's ability to withstand functional stresses in the oral environment [14].

Thicker ceramic restorations tend to attenuate or scatter light, reducing the intensity reaching the resin cement and negatively affecting its polymerization [13]. Studies indicate that ceramics thicker than 2 mm significantly hinder polymerization, necessitating adjustments in light-curing parameters or the use of dual-cure cements. In contrast, thinner ceramics allow sufficient light transmission for proper cement curing [15]. However, even at thinner dimensions, ceramic color can influence light transmission and, consequently, polymerization efficiency [16].

Polymerization of resin cements begins upon light activation and continues to progress over time. While the majority of polymerization occurs within the first 10–15 minutes, it extends up to 24 hours, allowing the material to achieve final mechanical and chemical stability [17]. To assess the durability and clinical performance of dental restorations, it is critical to evaluate bond strength at multiple time points. Immediate evaluations reflect the initial quality of the adhesive interface, while measurements after 24 hours provide insight into the impact of final polymerization and environmental factors such as moisture and temperature. Simulating oral conditions, such as temperature cycling, offers a realistic perspective on the long-term performance of the adhesive interface [18,19].

Mechanical and thermal stresses, as encountered during thermocycling, can degrade the bond interface, emphasizing the importance of understanding factors that affect resin cement polymerization and adhesion [20]. The type of ceramic, its surface treatment, and material characteristics play pivotal roles in ensuring the clinical success of bonded restorations.

The aim of this study was to evaluate the microshear bond strength (μSBS) between a resin luting cement and two ceramic types—lithium disilicate and zirconia—at different time points (immediate, after 24 hours, and after thermocycling). Additionally, the bond interface was analyzed using scanning electron microscopy (SEM). The null hypotheses tested were: (1) the type of ceramic does not influence bond strength with the resin cement, and (2) bond strength remains unaffected by different evaluation times.

2.3 MATERIALS AND METHODS

2.3.1 Study Design

The study groups were organized according to the type of ceramic material and further subdivided based on the evaluation time of bond strength, immediate, before (24h) and after

thermocycling, such as:

- Lithium disilicate groups (LD)
  - LD_Imm: Lithium disilicate immediate
  - LD_24h: Lithium disilicate at 24h
  - LD_Aftherm: Lithium disilicate ~~at~~ after thermocycling
- Zirconia groups (Z)
  - Z_Imm: Zirconia immediate
  - Z_24h: Zirconia at 24h
  - Z_ Aftherm: Zirconia ~~at~~ after thermocycling

The flowchart study (Figure 1) shows the general study design. Scanning electron microscopy (SEM) was used to evaluate the interface between the ceramics and light-activated resin luting cement. After the bond strength test, the failure mode of the specimens was analyzed by observation under optical microscope.

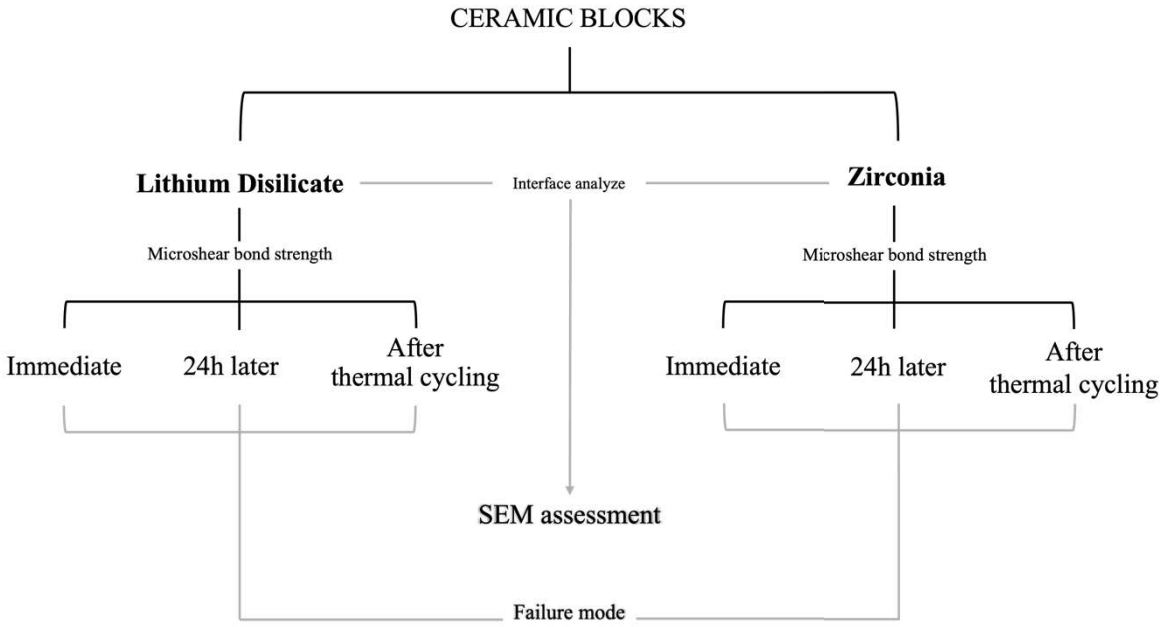

Figure 1. Flowchart study.

2.3.2 Ceramic Specimens Preparation

Specimens with 12 mm x 12 mm and 0.5 mm thickness were fabricated, being ten from lithium disilicate-based ceramic (IPS e.maxPress CAD, HT B1 / C14, Ivoclar Vivadent, Schaan,

Liechtenstein) and ten from zirconia-based ceramic (KATANA Zirconia Block, super translucent, CEREC, Kuraray, Noritake, Japan) CAD/CAM blocks. The blocks were cut using a low speed cutting machine (Isomet 1000, Buehler, Lake Bluff, IL USA) with a double-sided diamond blade (Isomet, Buehler, Lake Bluff, IL USA) measuring 3.5 inches in diameter and 0.5 mm in thickness under constant cooling. Following, both ceramics were crystallized (lithium disilicate) or sintered (zirconia) according to the manufacturer's instructions. For LD the baking was carried out in a special furnace (Programat EP300, Ivoclar Vivadent, Schaan, Liechtenstein) and for Z in a special furnace for Zinconia (Esthemat Sinta ll, Shofu, Kyoto, Japan). Next, the discs were finished in the same manner as laminate veneers, where the outer surface was polished for optimal aesthetics, while the inner surface was preserved for the cementation procedure. The outer surface of LD was finished using a diamond bur to remove irregularities, followed by polishing with ceramic abrasive rubbers (Exa-Cerapol ceramic polishers 0310UM and 0316UM). For finalization, a thin layer of glaze paste (IPS Ivocolor Glaze Paste, Ivoclar Vivadent, Schaan, Liechtenstein) was applied, and the glaze firing was performed. The final dimensions were confirmed using a digital caliper (Mitutoyo Corp, Kawasaki, Japan) (ELLAKANY et al., 2023). The outlayer of Z was finished as for LD and received an application of Cerabien ZR (Kuraray Noritake Dental Inc, Nagoya, Japan) which was covered with FL Glaze (Cerabien ZR) and a final sintering was performed in the same special furnace for Zinconia (Esthemat Sinta ll, Shofu, Kyoto, Japan).

2.3.3 Ceramic Surface Treatment

For the lithium disilicate ceramic, the internal surface (inner surface) was etched with 10% hydrofluoric acid (HF) (Condicionador de porcelana 10%, Dentsply Sirona, Pirassununga, Brazil) for 20 seconds, rinsed for the same time with an air/water spray from a triple syringe, and dried with oil- and water-free air. On the other hand, the internal surface of the zirconia ceramic was air-abraded with 50 µm aluminum oxide particles at 4 bars of pressure, approximately 10 cm away from the sandblaster nozzle.

Before the cementation procedure, a layer of MDP-based (10-methacryloyloxydecyl dihydrogen phosphate) chemical agent (Clearfil Ceramic Primer Plus, Kuraray Noritake Dental Inc., Japan) was applied to the internal surface of the zirconia ceramic with a microbrush. The lithium disilicate ceramic received a silane agent (Prosil, FGM, Joinville, SC, Brazil), which was applied for 15 seconds and allowed to react for a total of 1 minute. Then, the surfaces were dried with oil- and water-free air. To complete the surface treatment, a thin layer of adhesive (Optibond FL Adhesive, Kerr Corporation, Brea, CL, USA) was applied to the surface of both

ceramic types and light-cured for 10 s through the ceramic specimens (Figure 2) with an LED light source (Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein), with irradiance, verified by a radiometer, of 800 mW/cm².

2.3.4 Microshear Bond Strength (μSBS) Preparation Specimens

The bond strength analysis was performed using μSBS. To fabricate the specimens, a light body matrix of Poly Vynil Siloxane (Virtual, Ivoclar Vivadent, Schaan, Liechtenstein) was fabricated in a Plastic device which was printed in a 3D Printer (Flashforge Hunter, Zhejiang, China) with a 3D printing resin (Prizma 3D, Yller, Pelotas, Brasil). After polymerized, it resulted in a silicone matrix with six orifices of 1 mm in internal diameter and 1 mm in height. Then, the matrix was positioned on the previously treated ceramic surface. A light-cured resin luting cement (Variolink Esthetic LC, Ivoclar Vivadent Inc., Ontario, Canada) was applied to fill each orifice. A polyester strip and a glass slide were positioned on top of the silicone matrix, and digital pressure was applied to the set. The cement polymerization was light cured through the ceramic with the same LED light source (Bluephase N, Ivoclar Vivadent) for 40 s to ensure maximum polymerization of the material. Ten minutes after resin luting cement polymerization, the silicone matrix was carefully removed with scalpel blade and resulted a ceramic with six cylinders of resin luting cement on the ceramic surface (Figure 2).

Two cylinders from each tablet were tested immediately, while the remaining four were stored in distilled water at 37 °C. After 24 hours, two cylinders were tested, and the remaining two cylinders were subjected to 10,000 thermal cycling, alternating between 5 °C and 55 °C, before testing.

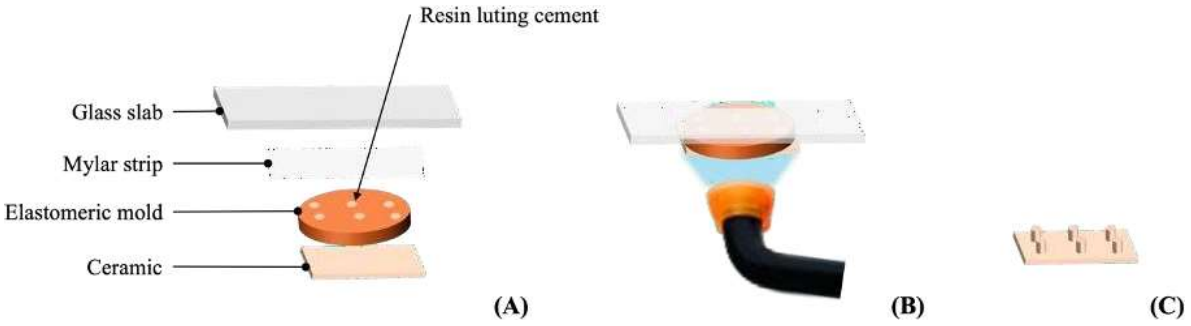

Figure 2. Procedures of microshear bond strength preparation specimens. (A) Order of positioning of the devices used, where the resin luting cement was applied into the matrix (elastomeric mold) orifices. (B) Polymerization activation of the material through the ceramic. (C) Final specimen configuration.

2.3.5 μSBS Test

The specimens were carefully bonded to the metallic device in a universal testing machine (EMIC DL3000, São José dos Pinhais, Brazil), ensuring that the bonding area between the ceramic and resin cement was perpendicular to the load applied during the test. The microshear test was performed using a 50 N load cell and a chisel-type steel blade attached to the load cell. The steel blade was positioned as close as possible to the base of the resin luting cylinder (bonding area). The test was conducted at a displacement speed of 0.5 mm per minute until failure. The test result (N) was converted to MPa using the equation:

$$MPa = N / Ab$$

where MPa is the microshear bond strength, N is the maximum force applied until the specimen fails, and Ab is the base area of the cylinder (calculated by the formula $Ab = \pi . r^2$). The diameter of each specimen (tested cylinder) was obtained through observation using an optical microscope (Axiostar Plus Microscope, Carl Zeiss MicrolmagingGmbH) at 5x magnification. The diameters of the cylinders were selected, and the equipment's software performed the measurements.

2.3.6 Modes Failure

After mechanical test, all bond areas were observed under 40x stereo microscope (Ken-a-Vision 4424, Kansas City, USA) for failure mode classification, such as: 1) Adhesive (when failure occurred at the bonding interface); 2) Cohesive in resin luting cement (when failure occurred within resin luting cement material); 3) Cohesive in ceramic (when failure occurred within the ceramic); 4) Mixed (when more than one type of failure occurred).

2.3.7 Scanning Electron Microscopy (SEM)

The analyze was made to observe the bond interface between ceramics and resin luting cement. The bond interface analyze was performed using the sandwich technique. The ceramic surfaces were treatment with the same modo to each *Ceramic Surface Treatment* and the resin luting cement was applied between ceramics. The digital pressure was applied and the excess material that overflowed was removed. The resin luting cement polymerization was made with the same LED light source for 20 s on each surface. Then, the sandwich (lithium disilicate ceramic / resin luting cement/zirconia ceramic) was embedded in self-curing resin and PVC

(polyvinyl chloride pipe) tube. After 24h, the samples were cut using a precision saw with a diamond blade, and polished using grit sandpapers (600, 1200 and 2000) to ensure a smooth and uniform surface. After polishing, all the samples were cleaned in an ultrasonic bath for 10 minutes in distilled water to remove residues and contaminants. Subsequently, they were coated with a thin layer of gold using a sputter coater (BAL-TEC MED010, Liechtenstein) to ensure proper electrical conductivity. The samples were then analyzed using scanning electron microscopy (SEM) (JEOL JSM-IT300LV, Tokyo, Japan), operating at 15 kV. An initial general observation of the interface between the ceramic and resin cement was performed, followed by image capture to best represent the observed interfacial characteristics.

2.3.8 Statistical Analysis

The mean and standard deviation were acquired and subjected to the Shapiro-Wilk test. When normality was confirmed ($p>0.05$), the data were analyzed using Two-way repeated measures ANOVA, followed by Tukey's post hoc test. A significance level of $p\leq0.05$ was adopted.

2.4 RESULTS

Table 1 presents the statistical results of microshear bond strength (μSBS) between ceramics and resin luting cement at all testing times. Regardless of the testing time - immediate, 24 hours, or post-thermocycling - lithium disilicate ceramics exhibited significantly higher μSBS values compared to zirconia ($p\leq0.05$). Both lithium disilicate and zirconia demonstrated significantly lower μSBS values after thermocycling, compared to the immediate and 24-hour tests ($p\leq0.05$). At 24 h, the μSBS was significantly higher than at the other times.

Table 1. Mean and standard deviation (□) of microshear bond strength (MPa) between ceramics and resin luting cement at immediately, 24 hours, and post-thermocycling.

Ceramics	Immediate	24h	After Thermal cycling
Lithium disilicate	31.0 ± 6.62 Ba	38.0 ± 7.48 Aa	25.5 ± 5.98 Ca
Zirconia	21.6 ± 4.61 Bb	30.3 ± 6.52Ab	4.78 ± 1.11 Cb

Note: Uppercase letters indicate statistically significant differences between values in the same row (same ceramic type) ($p\leq0.05$), and lowercase letters indicate differences between values in the same column (same testing time) ($p\leq0.05$).

Figure 3 shows the percentage of failure modes in the tested specimens at different times, where zirconia, both immediately and after thermocycling, exhibited predominantly adhesive failures (100%). Lithium disilicate at 24 hours showed half adhesive (50%) and half mixed (50%) failures, but after thermocycling, most failures were adhesive, as was the case for zirconia at 24 hours (75%). Only immediate lithium disilicate showed most mixed failures (75%). None of the cohesive failure modes in ceramic or resin cement were observed for either ceramic or at any testing time.

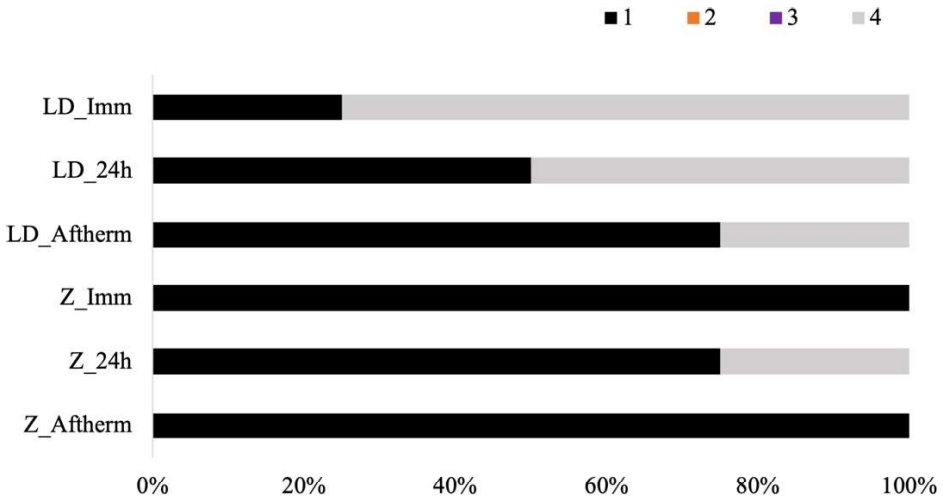

Figure 3. Failure mode across different testing times and ceramics. LD = lithium disilicate. Z = Zirconia. Imm = immediate. 24h = 24 hours. Aftherm = after thermal cycling. 1 = Adhesive (failure within the adhesive layer). 2 = Cohesive in ceramic (failure within the ceramic body). 3 = Cohesive in cement (failure within the cement material). 4 = Mixed (more than one type of failure combined).

The imagens by MEV show the difference between the types of ceramic interfaces. At lower magnification (X1500) (Figure 4 – A), the presence of discontinuities (gaps) at the zirconia and resin cement interface can be observed, in contrast to the lithium disilicate and resin cement interface. Furthermore, at higher magnification (X4000) (Figure 4 – B), these gaps between the zirconia ceramic and resin cement are more clearly visible.

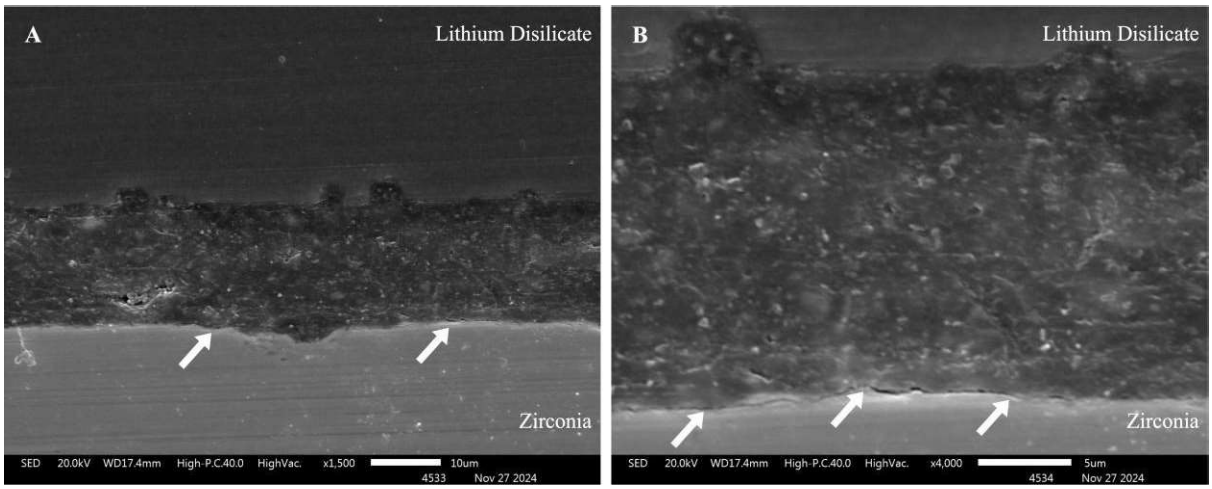

Figure 4. SEM micrographs. Representative images of the interface between ceramics (lithium disilicate and zirconia) and resin cement without thermocycling. (A) X1500 with white arrows indicating areas of discontinuity in the bond between zirconia and resin cement. (B) X4000 with white arrows highlighting areas of discontinuity in the bond between zirconia and resin cement.

2.5 DISCUSSION

Although lithium disilicate (LD) ceramics have been shown to achieve more reliable adhesion to resin cement compared to zirconia, the present study offers valuable insights into the bonding mechanisms and durability of resin luting cements applied to LD and zirconia (Z) ceramics under various conditions. The findings, particularly after thermocycling, contraindicate the use of zirconia in non-retentive dental prosthetic preparations. While zirconia manufacturers and several studies have claimed that zirconia can be used in dental prosthetic preparations without frictional retention, such as laminates, the results revealed a clear distinction in bond strength between the two types of ceramics [20–22]. Lithium disilicate (LD) consistently demonstrated higher micro-shear bond strength (μSBS) values than zirconia (Z) under all tested conditions [23].

These findings underscore the importance of material selection and surface treatment protocols to ensure long-term success in restorative dentistry and they are also in agreement with several studies [24,25]. The first null hypothesis, which stated that the type of ceramic would not influence the bond strength, was rejected. The μSBS results revealed significantly higher bond strength for LD compared to Z in all testing conditions (immediate, after 24 hours,

and post-thermocycling). This disparity can be attributed to the inherent differences in the composition and bonding mechanisms of the two ceramics. LD, being a glass-ceramic, allows for effective micromechanical retention through hydrofluoric acid (HF) etching and chemical bonding via silane coupling agents. These treatments create a strong and durable interface with the resin cement, as confirmed by the SEM analysis showing fewer gaps at the LD-resin cement interface. In contrast, Z, with its densely crystalline structure, relies primarily on mechanical retention from sandblasting and chemical bonding through MDP-based primers. Despite these measures, the SEM analysis revealed more pronounced interfacial discontinuities and gaps in Z, highlighting its challenging nature for bonding.

The second null hypothesis, which suggested that bond strength would remain unaffected by different evaluation times, was also rejected. Both ceramics exhibited significant reductions in μSBS after thermocycling. This outcome reflects the adverse impact of thermal aging on the adhesive interface, simulating the thermal and mechanical stresses experienced in the oral environment. For LD, the bond strength after 24 hours (38 ± 7.48 MPa) was the highest, while thermocycling significantly reduced it to 25.5 ± 5.98 MPa. However, the immediate bond strength (31 ± 6.62 MPa) was statistically comparable to the thermocycling condition, suggesting that the initial quality of the adhesive interface plays a critical role in its longevity. For Z, the reduction in μSBS post-thermocycling was more dramatic, dropping from 30.3 ± 6.52 MPa at 24 hours to 4.78 ± 1.11 MPa. The SEM analysis supported this observation, with Z showing extensive adhesive failures and significant interfacial gaps. These results emphasize the susceptibility of Z to thermal and mechanical degradation, despite the application of MDP-based primers and agree with other studies [23,26].

The failure mode analysis further highlighted differences between the ceramics. Adhesive failures predominated in zirconia (Z), particularly after thermocycling, indicating weak interfacial bonding - a finding consistent with studies by Ramos et al. [27] and Nadal et al. [28], which emphasize the challenges of achieving durable bonds to zirconia. In contrast, lithium disilicate (LD) exhibited a mix of adhesive and cohesive failures, reflecting a more robust bond, as also noted in studies by Kim et al. [29]. From a clinical perspective, these findings suggest that LD is suitable for restorations in both aesthetic and functional regions due to its superior bond strength and durability. However, caution must be exercised with Z, as evidenced by its significant reduction in bond strength post-thermocycling. Advanced surface treatments, such as the application of MDP primers or selective infiltration etching [30], and alternative bonding strategies may be necessary to enhance the longevity of Z-based restorations. Furthermore, the pronounced reduction in bond strength for both ceramics after

thermocycling underscores the importance of meticulous surface preparation and cementation protocols to mitigate thermal and mechanical stresses, as highlighted by the work of Alrabeah et al. [31] and Souza et al. [32].

Several types of surface treatments have been applied to zirconia; however, none has consistently demonstrated reliable bond strength [33,34]. From a clinical perspective, the indiscriminate use of zirconia for aesthetic dental laminates poses a high risk of restoration failure due to debonding. In this context, our results serve as a critical reminder for professionals to exercise caution when selecting and applying zirconia for such treatments. Proper case selection, meticulous adherence to adhesive protocols, and consideration of alternative materials, such as lithium disilicate, may significantly enhance long-term success rates in these restorations.

The results bring the conclusion that the use of zirconia in dental laminates should be avoided, as the study highlights the clear disparity in bond strength between lithium disilicate (LD) and zirconia (Z). The inherent differences in the bonding mechanisms—LD benefiting from micromechanical retention and chemical bonding via silane agents—make it more suitable for reliable and durable restorations. On the other hand, zirconia's dense crystalline structure poses challenges in achieving strong adhesion, even with surface treatments like sandblasting and MDP-based primers. The study's findings emphasize the need for advanced surface treatments and alternative bonding strategies to improve the longevity of zirconia-based restorations. Furthermore, the significant degradation of bond strength after thermocycling further underscores the necessity of proper surface preparation and cementation protocols to ensure long-term success.

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3. CONCLUSÕES

O uso de zircônia em laminados dentais deve ser evitado, uma vez que o estudo destaca

a clara disparidade na resistência de união entre dissilicato de lítio (DL) e zircônia (Z). As diferenças inerentes nos mecanismos de adesão – DL se beneficiando da retenção micromecânica e da ligação química por meio de agentes silanos – tornam-no mais adequado para restaurações confiáveis e duradouras. Por outro lado, a estrutura cristalina densa da zircônia apresenta desafios para alcançar uma adesão forte, mesmo com tratamentos de superfície como jateamento e primers à base de MDP. Os achados do estudo enfatizam a necessidade de tratamentos de superfície avançados e estratégias alternativas de adesão para melhorar a longevidade das restaurações à base de zircônia. Além disso, a degradação significativa da resistência de união após a termociclagem ressalta ainda mais a importância de uma preparação adequada da superfície e de protocolos de cimentação para garantir o sucesso a longo prazo.

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ANEXO – Metodologia

Desenho do Estudo

Os grupos do estudo foram organizados de acordo com o tipo de material cerâmico e subdivididos com base no tempo de avaliação da resistência de união: imediata, antes (24h) e após o envelhecimento térmico, como segue:

- Grupos de dissilicato de lítio (DL)
    - DL_Im: Dissilicato de lítio imediato
    - DL_24h: Dissilicato de lítio em 24h
    - DL_ApTerm: Dissilicato de lítio após termociclagem
- Grupos de zircônia (Z)
    - Z_Im: Zircônia imediata
    - Z_24h: Zircônia em 24h
    - Z_ ApTerm: Zircônia após termociclagem

O fluxograma do estudo (Figura 1) mostra o delineamento geral do estudo. A microscopia eletrônica de varredura (MEV) foi utilizada para avaliar a interface entre as cerâmicas e o cimento resinoso fotopolimerizável. Após o teste de resistência de união, o modo de falha dos espécimes foi analisado por observação em microscópio óptico.

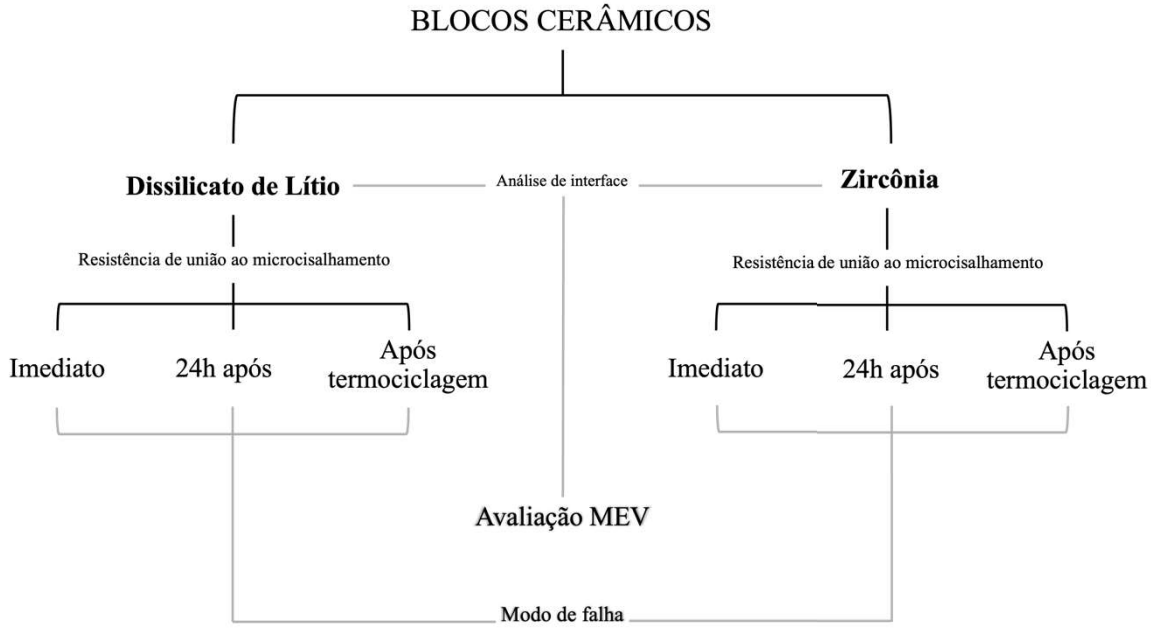

Figura 1. Fluxograma do estudo.

Preparo dos Corpos de Prova de Cerâmica

Corpos de prova cerâmicos com espessura de 0,5 mm foram fabricados a partir de blocos comerciais CAD/CAM de cerâmica à base de dissilicato de lítio (IPS e.max Press CAD, HT B1 / C14, Ivoclar Vivadent, Schaan, Liechtenstein) e cerâmica à base de zircônia (KATANA Zirconia Block, super translúcida, CEREC, Kuraray, Noritake, Japão). Os blocos foram cortados com uma máquina de corte de baixa velocidade (Isomet, Buehler, EUA) com lâmina de diamante dupla face (Isomet, Buehler, EUA) sob refrigeração constante. Ambas as cerâmicas foram cristalizadas (dissilicato de lítio) ou sinterizadas (zircônia) conforme as instruções do fabricante (Figura 2).

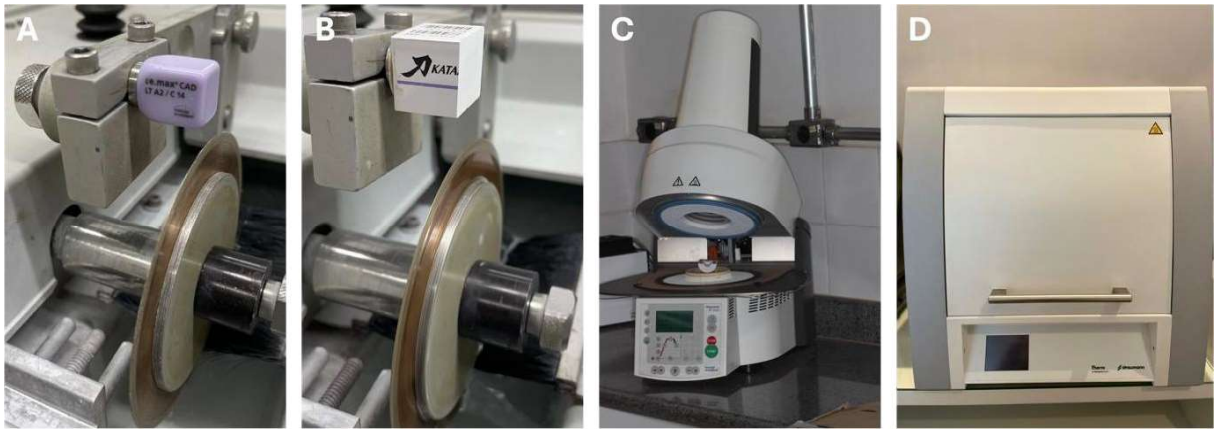

Figura 2. Preparo dos espécimes cerâmicos. (A) Corte, em cortadeira de precisão, do bloco cerâmico à base de dissilicato de lítio (IPS e.max Press CAD), (B) corte, em cortadeira de precisão, do bloco cerâmico à base de zircônia (KATANA Zirconia Block), (C) cristalização da cerâmica IPS e.max Press CAD em forno Ivoclar Vivadent e (D) sinterização da cerâmica KATANA Zirconia Block em forno Straumann.

Os discos foram finalizados como laminados cerâmicos, onde a superfície externa foi polida para estética otimizada, enquanto a superfície interna foi preservada para o procedimento de cimentação. A superfície externa foi acabada com ponta diamantada para remover irregularidades, seguida de polimento com borrachas abrasivas para cerâmica (Exa-Cerapol 0310UM e 0316UM). Para finalização, foi aplicada uma fina camada de glaze (IPS Ivocolor Glaze Paste, Ivoclar Vivadent, Schaan, Liechtenstein) e realizada queima de glaze. As dimensões finais foram confirmadas com um paquímetro digital (Mitutoyo Corp, Kawasaki, Japão) (Figura 3).

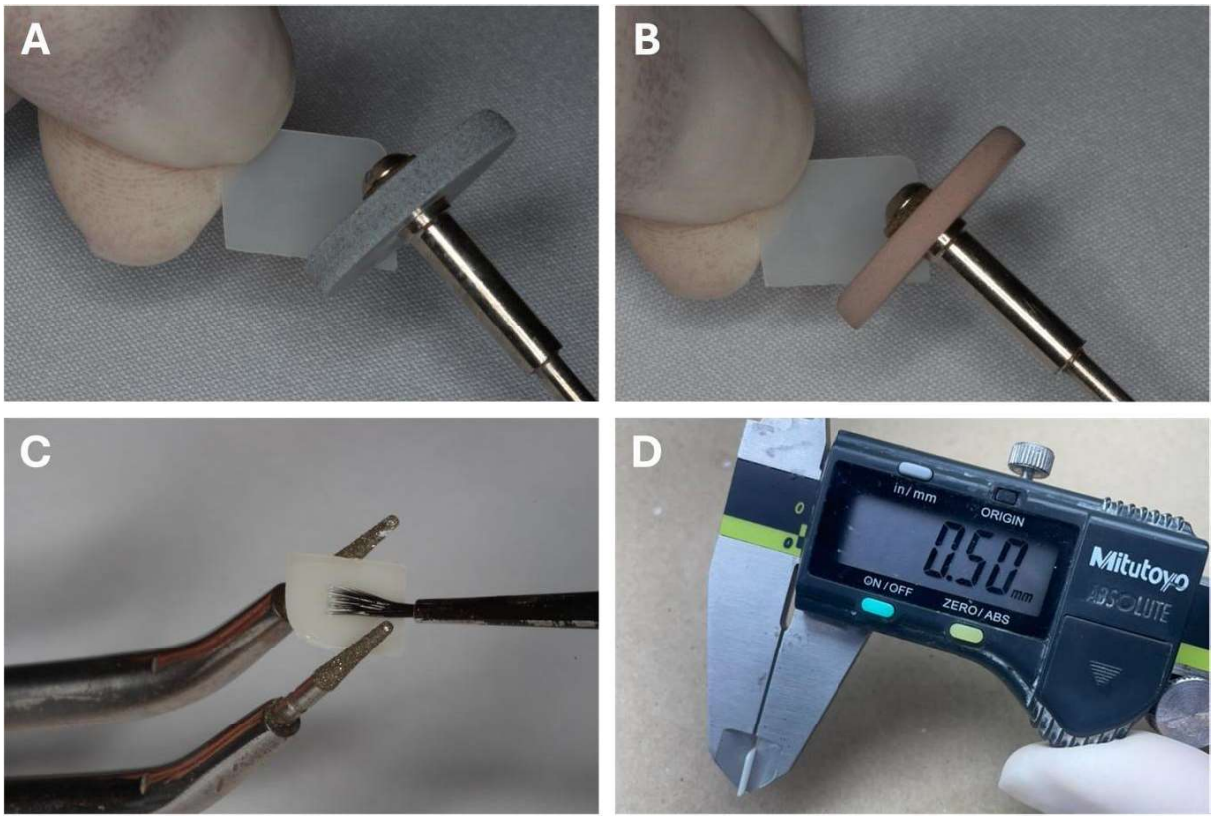

Figura 3. Acabamento e polimento das amostras cerâmicas. (A) Uso de borracha abrasiva 0310UM, (B) uso de borracha abrasiva 0316UM, (C) aplicação de glaze e (D) verificação da espessura final da cerâmica.

Tratamento de Superfície Cerâmica

Para o dissilicato de lítio, a superfície interna foi condicionada com ácido hidrofluorídrico a 10% (Condicionador de porcelana 10%, Dentsply Sirona) por 20 segundos, enxaguada pelo mesmo tempo com spray de água/ar e seca com ar isento de óleo e água (Figura 4). A superfície interna da zircônia foi jateada com óxido de alumínio de 50 µm a 4 bar de pressão, a aproximadamente 10 cm da ponta do jateador (Figura 4).

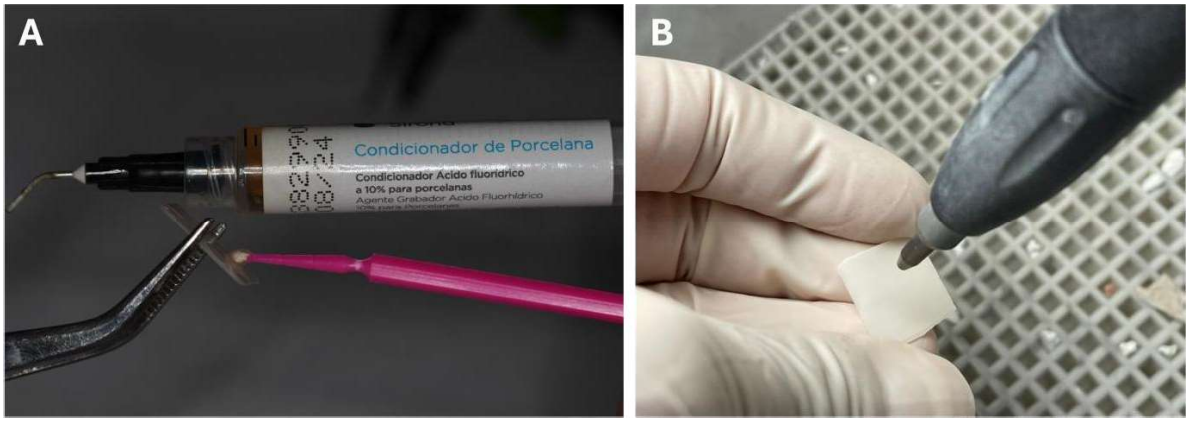

Figura 4. Condicionamento da superfície das cerâmicas. (A) Condicionamento da cerâmica à base de dissilicato de lítio com ácido hidrofluorídrico e (B) condicionamento da cerâmica à base de zircônia com jateamento com óxido de alumínio.

Antes da cimentação, foi aplicada uma camada de primer com MDP (Clearfil Ceramic Primer Plus, Kuraray Noritake Dental Inc., Japão) na superfície interna da zircônia com microbrush. O dissilicato de lítio recebeu um agente de acoplamento silano (Prosil, FGM, Joinville, SC, Brasil), aplicado por 15 segundos, com tempo de reação total de 1 minuto, e depois seco com ar isento de óleo e água. Uma fina camada de adesivo (Optibond FL Adhesive, Kerr Corporation, Nova Zelândia) foi aplicada na superfície de ambas as cerâmicas e a polimerização foi fotoativada por 10 segundos com uma fonte de luz LED (Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein) com irradiância de 800 mW/cm² (Figura 5).

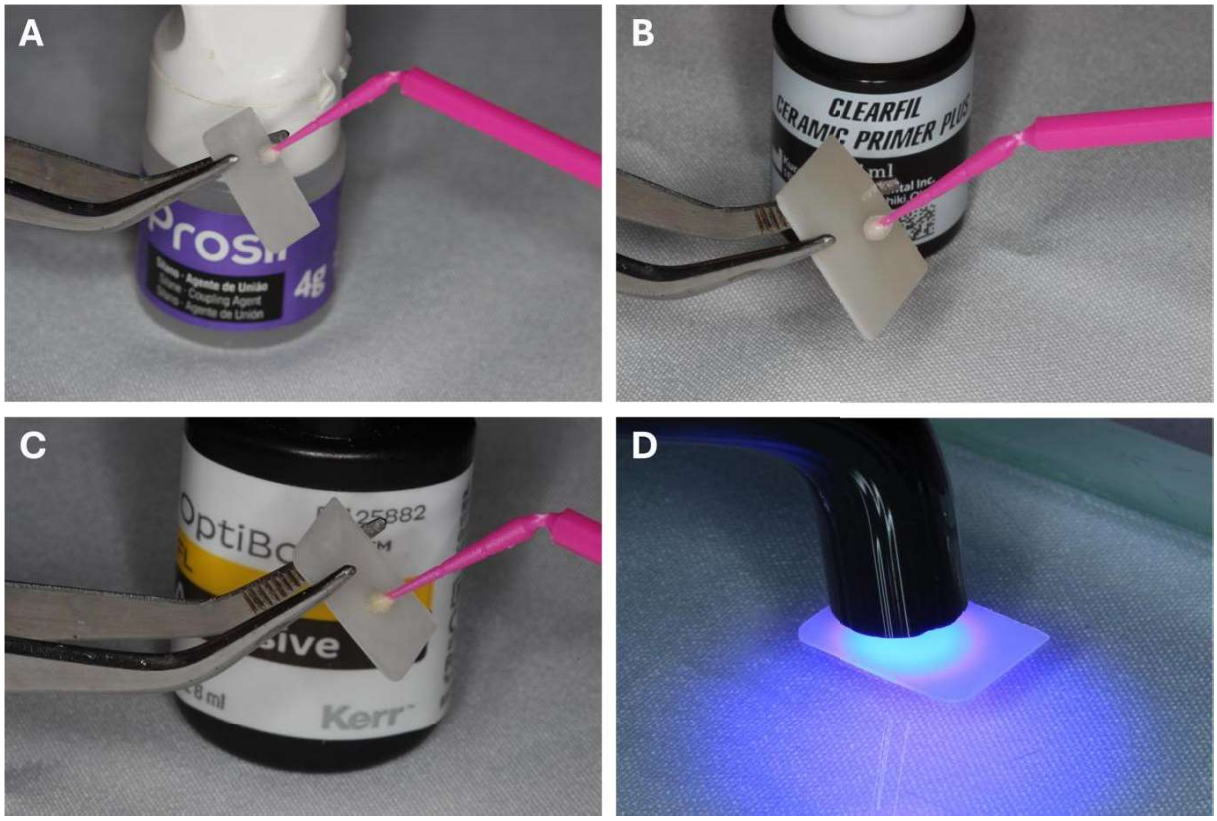

Figura 5. Tratamento das superfícies cerâmicas. (A) Aplicação de agente de acoplamento silano na superfície de cerâmica à base de dissilicato de lítio, (B) aplicação de primer a base de MDP em cerâmica à base de zircônia, (C) aplicação de adesivo de esmalte na superfície cerâmica e (D) fotoativação de polimerização do adesivo.

Preparo dos Corpos de Prova para µSBS

A análise de resistência de união foi realizada usando o teste de microcisalhamento (µSBS). Para a confecção dos espécimes, foi criado um molde de silicone com seis orifícios de 1 mm de diâmetro e 1 mm de altura. O cimento resinoso fotopolimerizável (Variolink Esthetic LC, Ivoclar Vivadent Inc., Ontário, Canadá) foi aplicado em cada orifício, seguido de fotopolimerização por 40 segundos. Após a polimerização, o molde foi cuidadosamente removido, resultando em cilindros de cimento resinoso sobre a cerâmica (Figura 6).

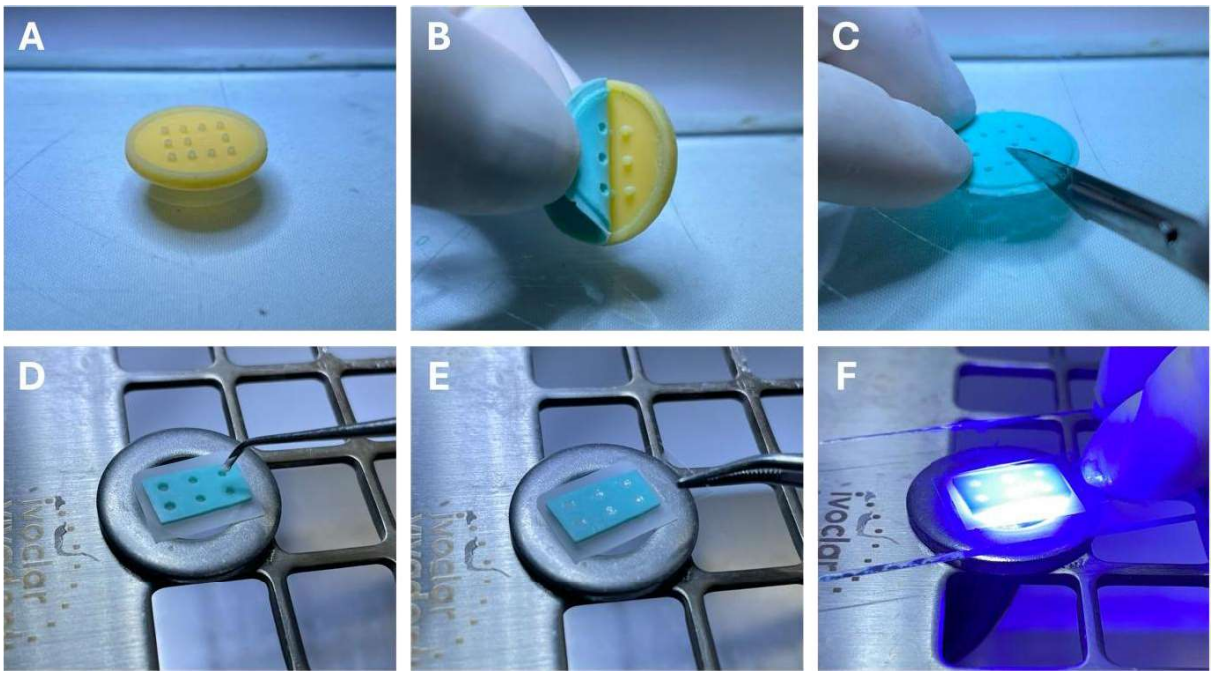

Figura 6. Espécimes para microcisalhamento. (A) Dispositivo molde para confecção da matriz de silicone, (B) remoção da matriz após moldagem do dispositivo com silicone por adição, (C) individualização dos 6 orifícios, (D) posicionamento da matriz de silicone sobre a cerâmica, previamente tratada, e inserção do cimento resinoso, (E) posicionamento da tira de poliéster sobre a matriz e (F) posicionamento da lâmina de vidro e fotoativação de polimerização do cimento resinoso.

Teste de µSBS

Os espécimes foram testados em uma máquina universal de ensaios (EMIC DL3000). Os espécimes foram fixados na base da máquina, onde a área de união ficou perpendicularmente posicionada em relação a ponta do cinzel utilizado. O movimento em direção ao solo foi realizado com célula de carga de 50 N e velocidade de deslocamento de 0,5 mm/min até a falha (Figura 7). Os resultados foram convertidos em MPa pela fórmula:

$$MPa = N / A_b$$

onde N é a força máxima até a falha e A_b é a área da base do cilindro.

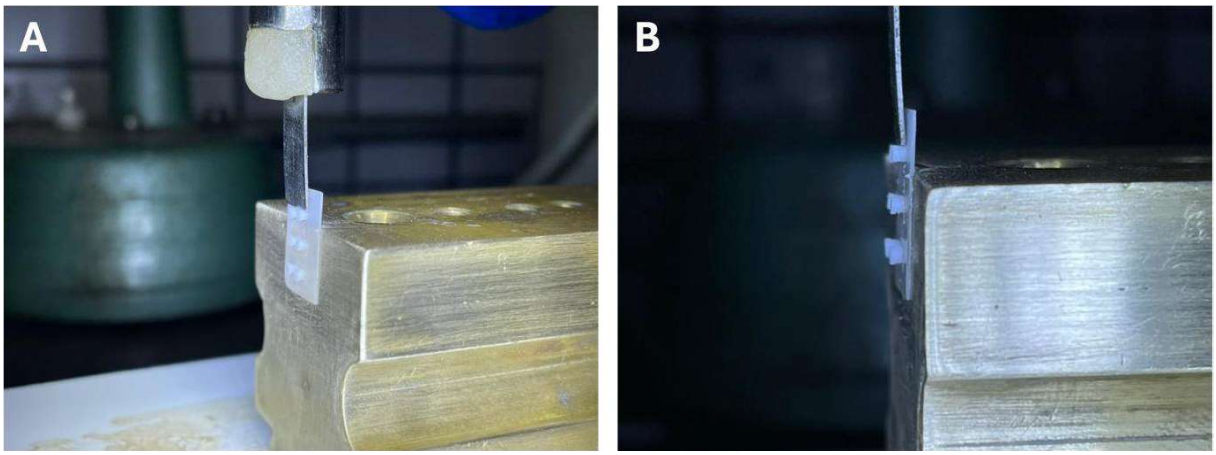

Figura 7. Teste de microcisalhamento. (A) Posicionamento do espécime da base da máquina e (B) posicionamento do cinzel perpendicular à área de união.

Modos de Falha

Após o teste mecânico, as áreas de união foram observadas em microscópio estereoscópico (Ken-a-Vision 4424, Kansas City, EUA) e classificadas como: 1) Adesiva (quando a falha ocorreu na interface de união); 2) Coesiva no cimento resinoso (quando a falha ocorreu dentro do material do cimento resinoso); 3) Coesiva na cerâmica (quando a falha ocorreu dentro da cerâmica); 4) Mista (quando mais de um tipo de falha ocorreu) (Figura 8).

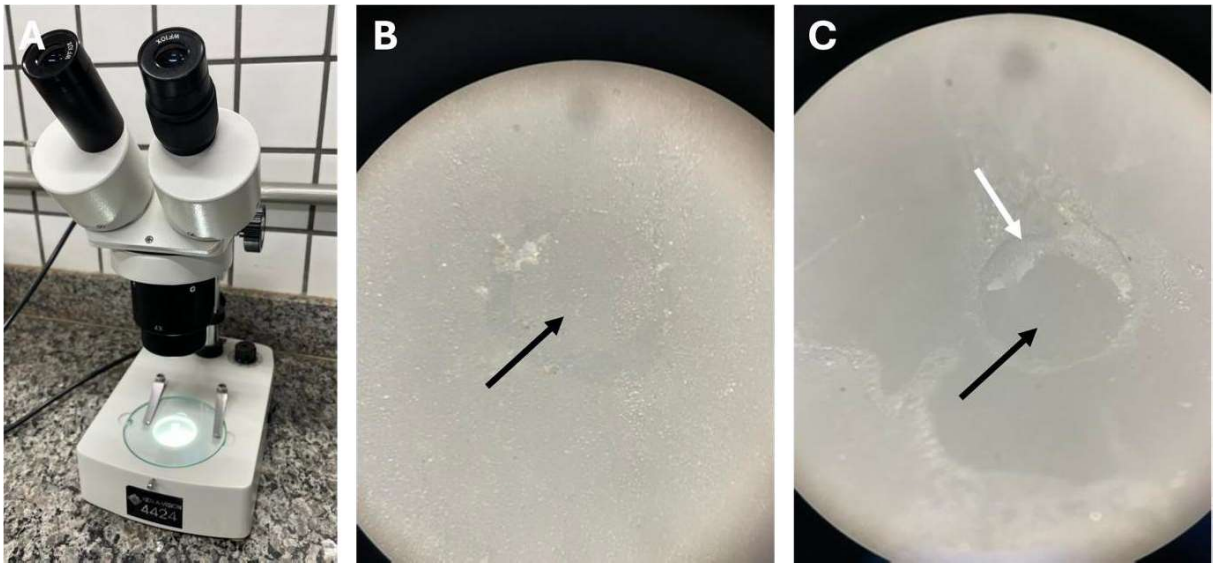

Figura 8. Análise do modo de falha da área de união. (A) Equipamento utilizado (microscópio), (B) exemplo de modo de falha tipo adesiva (seta preta: área de união analisada) e (C) exemplo de modo de falha do tipo mista (seta preta: área de união analisada; seta branca: resíduos de

cimento resinoso na superfície da cerâmica).

Microscopia Eletrônica de Varredura (MEV)

A análise foi realizada para observar a interface de união entre as cerâmicas e o cimento resinoso e feita utilizando a técnica de sanduíche. As superfícies cerâmicas foram tratadas conforme descrito em "Tratamento de Superfície Cerâmica", e o cimento resinoso foi aplicado entre as cerâmicas. Pressão digital foi aplicada, e o excesso de material extravasado foi removido. A fotoativação de polimerização do cimento resinoso foi realizada com a mesma fonte de luz LED por 20 segundos em cada superfície. Em seguida, o sanduíche (cerâmica de dissilicato de lítio / cimento resinoso / cerâmica de zircônia) foi embutido em resina acrílica e tubo de PVC (policloreto de vinila). Após 24 horas, as amostras foram cortadas utilizando uma serra de precisão com disco de diamante e polidas com papeis abrasivos de granulação 600, 1200 e 2000, garantindo uma superfície lisa e uniforme. Após o polimento, todas as amostras foram limpas em um banho ultrassônico por 10 minutos em água destilada para remover resíduos e contaminantes (Figura 9).

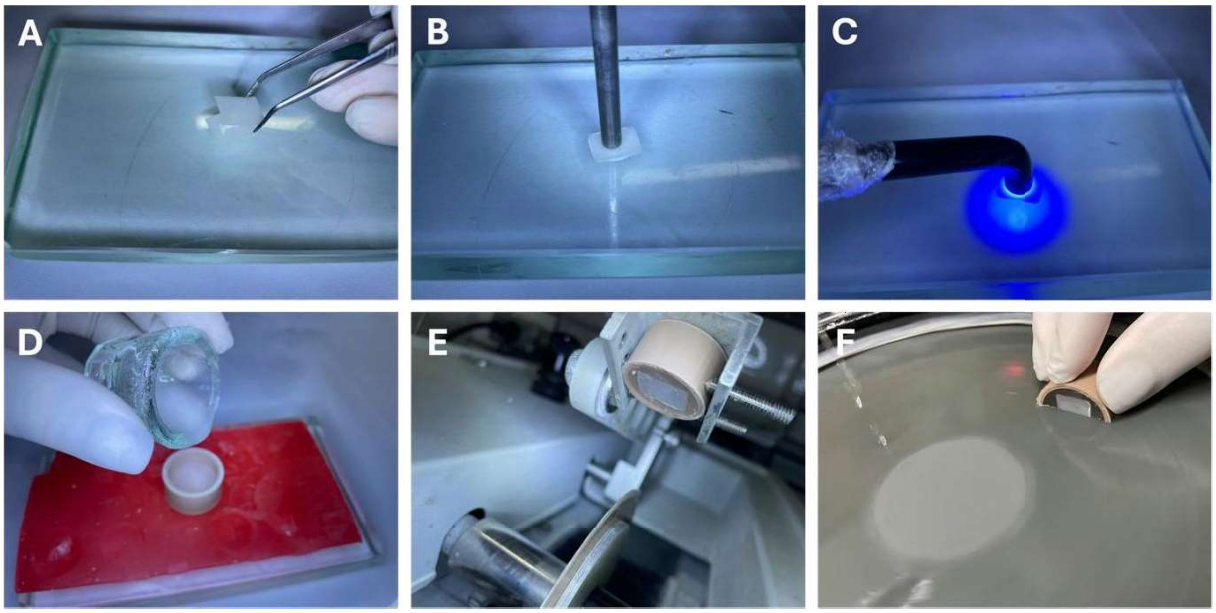

Figura 9. Preparo para análise em MEV. (A) União de duas cerâmicas com cimento resinoso, (B) pressionamento das cerâmicas, (C) fotoativação de polimerização do cimento resinoso, (D) inclusão da amostra com resina acrílica autopolimerizável, (E) corte ao meio da amostra e (F) polimento da interface.

Posteriormente, os espécimes foram revestidos com uma fina camada de ouro usando

um "*sputter coater*" (BAL-TEC MED010, Liechtenstein) para garantir uma condutividade elétrica adequada. As amostras foram analisadas por microscopia eletrônica de varredura (MEV) (JEOL JSM-IT300LV, Tóquio, Japão), operando a 15 kV, com ampliações variando de X1500 a X4000, para observar a interface entre a cerâmica e o cimento resinoso (Figura 10).

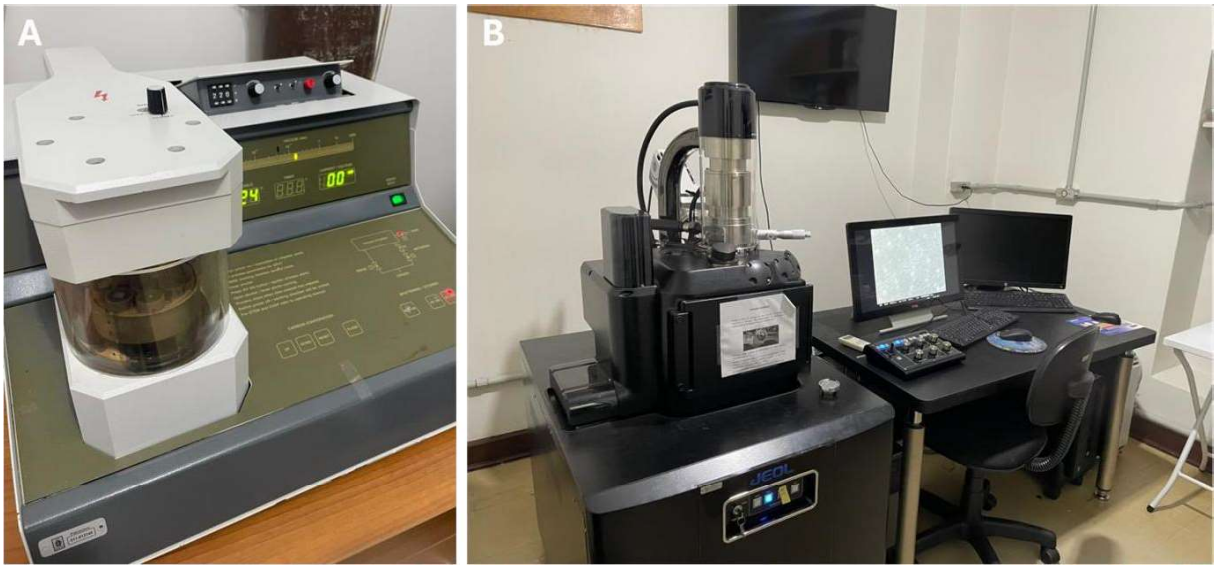

Figura 10. Equipamentos de MEV. (A) Realização da cobertura de ouro sobre as amostras em *sputter coater* e (B) microscópio eletrônico de varredura do laboratório de Microscopia Eletrônica aplicada à Pesquisa Agropecuária (NAP/MEPA) da Escola Superior de Agricultura Luiz de Queiroz da Universidade de São Paulo (ESALQ-USP).