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Fernanda Dalla Nora

EFEITO DE TRATAMENTOS DE SUPERFÍCIE E CIMENTOS RESINOSOS NA RESISTÊNCIA DE UNIÃO E NA CARGA PARA FALHA EM FADIGA DE UMA CERÂMICA VÍTREA

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Dissertação apresentada ao Curso de Mestrado do Programa de Pós-Graduação em Ciências Odontológicas, Área de concentração em Odontologia, ênfase em Prótese Dentária, da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para obtenção do grau de **Mestre em Ciências Odontológicas.**

Orientadora: Profª. Drª. Marília Pivetta Rippe

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Aprovado em 17 de julho de 2019:

Orientador: Prof[a]. Dr[a]. Marília Pivetta Rippe
(Presidente/ Orientador)

Prof[a]. Dr[a]. Andressa Borin Venturini (UFSM)

Prof. Dr. Gabriel Kalil Rocha Pereira (IMED)

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DEDICATÓRIA

Com carinho,
aos meus pais Lino e Elizete, irmãs, vó Lourdes, amigos que torceram para o meu sucesso, professores que me incentivaram e me apoiaram para ingressar na pós-graduação e em especial à Professora Marília.

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RESUMO

Efeito de diferentes tratamentos de superfície e cimentos resinosos na resistência de união e na carga para falha em fadiga de uma cerâmica vítrea

AUTORA: Fernanda Dalla Nora
ORIENTADOR: Profª. Drª. Marília Pivetta Rippe

O objetivo do presente estudo foi avaliar o efeito de diferentes tratamentos de superfície e cimentos resinosos na resistência de união e no comportamento mecânico de uma cerâmica a base de silicato de lítio reforçada por zircônia. Para o teste de resistência de união ao microcisalhamento, blocos de cerâmica (18 × 14 × 12) foram cortados em fatias de formato retangular (1 × 14 × 12 mm), embutidos em resina acrílica e divididos de acordo com os fatores do estudo (n = 36) “tratamento de superfície” (condicionamento com ácido fluorídrico 5%- HF; aplicação de primer autocondicionante- EP; e jateamento com óxido de alumínio revestido por sílica- SB) e “cimentos resinosos” (cimento convencional sem MDP - nMDP; cimento convencional com MDP- MDP; e cimento autoadesivo- SA). Após os respectivos tratamentos de superfície, os cimentos foram aplicados dentro de matrizes de tubos de amido sobre a cerâmica e fotopolimerizados. Esperou-se 24 horas e realizou-se a termociclagem (5-55°C; 5 mil ciclos) de todos os espécimes. Posteriormente, foi realizado o teste de microcisalhamento pela técnica do fio (wire-loop). Os dados foram submetidos aos testes de Kruskal-Wallis e post-hoc Dunn. Para o teste de fadiga foram confeccionados discos cerâmicos (diâmetro (Ø)= 10 mm, espessura= 1,5 mm) e de material análogo de dentina (Ø= 10 mm, espessura= 2,0 mm), os quais foram divididos de acordo com os fatores do estudo (n=15) acima descritos. Todos os discos foram cimentados, termociclados e submetidos ao teste de fadiga (Step-wise) iniciando com uma carga de 400N, à uma frequência de 20Hz por 10.000 ciclos por etapa, com acréscimo de 100N por etapa. A análise estatística foi realizada com testes de Kaplan-Meier e Mantel-Cox (Log Rank) ($p < 0,05$). Para o teste de resistência de união, considerando o tipo de tratamento de superfície, HF e EP proporcionaram maiores valores de resistência de união com o cimento autoadesivo, e o SB com o cimento resinoso convencional e o cimento autoadesivo. Considerando o tipo de cimento, o convencional sem MDP apresentou melhores resultados com os tratamentos HF e SB e os cimentos contendo MDP e autoadesivo apresentaram melhores resultados com o condicionamento com HF. Com relação ao teste de fadiga, o tratamento de superfície com ácido fluorídrico e primer cerâmico apresentaram valores de carga para falha e número de ciclos para fadiga estatisticamente semelhantes e maiores que o jateamento, independentemente do tipo de cimento. Quando o tratamento de superfície foi considerado isoladamente, o melhor cimento para os tratamentos de superfície ácido fluorídrico e jateamento foi o cimento autoadesivo. Contudo para o tratamento cerâmico com o primer, não houve diferença entre os cimentos.

Palavras-chave: Silicato de lítio reforçado por zircônia. Microcisalhamento. Adesão. Carregamento cíclico. Taxa de sobrevivência.

Effect of different surface treatments and resin cements on bond strength and fatigue failure load of a glass ceramic:

AUTHOR: Fernanda Dalla Nora
ADVISOR: Profª. Drª. Marília Pivetta Rippe

The aim of the present study was to evaluate the effect of different surface treatments and resin cements on bond strength and mechanical behavior of a zirconia reinforced lithium silicate ceramic (ZLS). For the micro-shear bond strength test, the ZLS blocks were cut into rectangular (1 × 14 × 12 mm) slices, embedded in acrylic resin and divided according to the study factors (n = 36): “surface treatment” (hydrofluoric acid etching 5%- HF; application of a self-etching primer- EP and sandblasting with silica coated aluminum oxide particles- SB) and “resin cements” (MDP-free cement - nMDP; MDP-containing cement - MDP; and self-adhesive cement - SA). After the surface treatments, the different cements were applied inside starch tubes on the ceramic and photopolymerized. Twenty-four hours after the cementation, all specimens were thermocycled (5-55°C, 5,000 cycles). Subsequently, the wire-loop micro-shear test was carried out. Statistical analysis was performed using Kruskal-Wallis and post-hoc Dunn tests. For the fatigue test, ceramic discs (diameter- Ø = 10 mm, thickness = 1.5 mm) and discs of dentin analog material (Ø = 10 mm, thickness = 20 mm) were made and divided according to the factors of the study (n = 15) described above. The discs were cemented, thermocycled and submitted to the fatigue test (Step-wise), starting with a load of 400N at a frequency of 20Hz for 10,000 cycles for step, plus 100N for step. Statistical analysis was performed using Kaplan-Meier and Mantel-Cox (Log Rank) tests ($p < 0.05$). For the bond strength test, considering the type of surface treatment, the HF and the EP provided higher values of bond strength with the self-adhesive cement, and the SB with the conventional resin cement and the self-adhesive cement. Considering the type of cement, the MDP-free conventional one showed better results with the HF or SB treatments and the MDP-containing and the self-adhesive cements performed better results with HF etching. Regarding the fatigue test, the surface treatment with hydrofluoric acid and ceramic primer showed values of load and number of cycles for failure statistically similar and higher than the sandblasting, regardless of the type of cement. When only the surface treatment was considered, the best cement for hydrofluoric acid and sandblast surface treatments was self-adhesive cement. There was no difference between the cements for the ceramic treatment with the self-etching primer.

Keywords: Zirconia reinforced lithium silicate ceramic Micro shear. Adhesion. Cyclic loading. Survival Rate.

SUMÁRIO

1. INTRODUÇÃO

Restaurações totalmente cerâmicas são amplamente utilizadas e desempenham um papel importante na odontologia estética moderna. Segundo Üstum, Büyükhatipoglu e Seçilmiş [2016] a tecnologia CAD/CAM (computer-aided design and computer-aided manufacturing) tem melhorado a qualidade das restaurações cerâmicas, propiciando o desenvolvimento de materiais mais resistentes e restaurações que apresentam maior longevidade [AL-THAGAFI; AL-ZORDK; SAKER, 2016]. Isso se deve a fabricação totalmente industrial das cerâmicas, o que proporcionou um aprimoramento deste material, e levou a uma melhor estética, biocompatibilidade e durabilidade das coroas protéticas [BELLI et al., 2017; ELSAKA; ELNAGHY, 2016]. Uma opção recentemente introduzida no mercado de materiais cerâmicos para CAD/CAM é a cerâmica de silicato de lítio reforçada com cristais de zircônia (ZLS). A ZLS é uma combinação de estrutura vítrea enriquecida com zircônia, indicada para confecção de inlays, onlays, coroas parciais, laminados, coroas anteriores e posteriores sobre estrutura dental ou implante [ELSAKA; ELNAGHY, 2016; RINKE et al., 2016].

Após sua cristalização, a cerâmica ZLS apresenta uma microestrutura homogênea (Li_2-ZrO_2-SiO_2) muito fina, com um tamanho médio de grãos entre 0,5-0,7μm [RINKE et al., 2016; TRAINI et al., 2016], o que proporciona a este material elevadas propriedades mecânicas e estéticas. Existem na literatura estudos comparando a cerâmica ZLS com diferentes materiais, como a cerâmica de dissilicato de lítio [ELSAKA; ELNAGHY, 2016; HAMZA; SHERIF, 2017] entre outras (cerâmica feldspática, cerâmica vítrea reforçada por leucita, cerâmica vítrea de silicato de lítio reforçada por zircônia pré-sinterizada, cerâmica vítrea de silicato de lítio reforçada por zircônia totalmente sinterizada, resina nanocerâmica e cerâmica híbrida) [WEYHRAUCH ET AL., 2016] onde relatam superioridade em propriedades mecânicas como, resistência à fratura, resistência à flexão, módulo de elasticidade e dureza, para a cerâmica ZLS. Além disso, Zimmermann et al. [2017] concluíram em seu estudo longitudinal que restaurações CAD/CAM em cerâmica ZLS apresentam uma alta taxa de sucesso clínico após 12 meses.

Por ser uma cerâmica de matriz predominantemente vítrea, a ZLS é considerada sensível ao condicionamento ácido [SATO et al., 2016], que é o método de tratamento de superfície mais comumente usado para essa classificação cerâmica [STRASSER et al., 2018]. O principal objetivo deste tratamento é atacar seletivamente a fase vítrea da cerâmica, introduzindo microporosidades que permitirão a penetração e consequentemente o microembricamento do cimento resinoso. Esta exposição da matriz vítrea, também expõe grupos hidroxílicos, que através da aplicação do agente de união silano, possibilitam uma ligação química entre cerâmica e cimento [RAMAKRISHNAIAH et al., 2016; SILVA et al., 2016; STRASSER et al., 2018; TIAN et al., 2014].

No entanto o ácido fluorídrico é um produto de alta toxicidade, além disso, não se sabe até que ponto defeitos introduzidos pelo condicionamento cerâmico são totalmente preenchidos pelo cimento, o que pode resultar em uma diminuição da resistência da restauração, por esses motivos, métodos alternativos de tratamento de superfície vêm sendo desenvolvidos e testados [SATO et al., 2016; WILLE; LEHMANN; KERN, 2017]. O jateamento da superfície cerâmica com partículas de óxido de alumínio revestidas com sílica é uma opção. Al-Thagafi et al [2016] observaram melhores resultados de resistência de união com a cerâmica ZLS, quando o jateamento de partículas de óxido de alumínio revestidas por sílica associado com silano foi utilizado como tratamento de superfície cerâmica, do que quando o ácido fluorídrico associado ao silano foi utilizado. No momento do jateamento, a pressão e a alta energia do choque destas partículas sobre a cerâmica, resulta na incorporação de sílica na superfície do material. Este procedimento, além de aumentar a rugosidade da superfície, também modifica quimicamente a cerâmica, a tornando mais reativa ao cimento resinoso, especialmente através do agente silano [AMARAL et al., 2006; SATO et al., 2016]. Porém Strasser et al. [2018] observaram que o jateamento pode causar propagação de fissuras e micro trincas no material, promovendo uma diminuição da resistência mecânica e longevidade da peça protética. Além disso, Sato et al. [2016] concluíram que o jateamento com sílica não se mostrou eficiente na resistência de união após envelhecimento quando comparado com o condicionamento com ácido fluorídrico, provocando trincas na superfície cerâmica.

Outra alternativa de tratamento de superfície cerâmico é a utilização de um primer cerâmico autocondicionante de passo único, sendo uma opção substitutiva para o uso do ácido fluorídrico e do agente silano [EL-DAMANHOURY; GAINTANTZOPOULOU, 2018; WILLE; LEHMANN; KERN, 2017]. Segundo Wille, Lehmann e Kern [2017] essa alternativa mostrou resultados satisfatórios quando comparado com o condicionamento ácido em uma cerâmica de dissilicato de lítio, já Lopes et al [2018] e Prado et al [2018] reportaram resultado contrário a estes achados, mostraram que o tratamento de superfície com ácido fluorídrico associado com agente silano obteve melhores resultados de resistência de união quando comparado com o primer cerâmico autocondicionante para a mesma cerâmica. O que mostra que não há um consenso na literatura sobre o desempenho deste tipo de tratamento da superfície cerâmica.

Um fator que também deve ser levado em conta para o sucesso do tratamento restaurador é o tipo de cimento. Segundo Bindl, Lüthy e Mörmann [2006], a cimentação adesiva proporciona melhores resultados de resistência à fratura em coroas monolíticas fresadas em sistema CAD/CAM, quando comparado com uma cimentação convencional. Mesmo resultado obtido por Campos et al [2016] em coroas de zircônia. Isso pode ser explicado por um aumento da resistência do conjunto dente/restauração, quando uma adesão efetiva é realizada [SASSE et al., 2015], o que proporciona a otimização da distribuição de estresse na cerâmica, cimento resinoso e estrutura dental [MONTEIRO et al., 2017].

Vários fatores são considerados importantes para o sucesso a longo prazo das restaurações cerâmicas cimentadas, dentre eles as características intrínsecas do cimento resinoso, como a quantidade de carga e viscosidade. Segundo Kern [2009] cimentos resinosos contendo monômeros fosfatados, como o MDP (10-methacryloyloxy-decyl-dihydrogen-phosphate), possuem uma melhor adesão à cerâmica com o cimento resinoso. Isso se explica pela presença de moléculas bifuncionais, que através do seu grupo éster de fosfato se unem diretamente à óxidos de sílica presentes na superfície da cerâmica e grupos de metacrilatos que se ligam à matriz resinosa do cimento, proporcionando uma melhor união entre os componentes [KERN, 2009]. Além disso, a quantidade de carga presente na composição do cimento resinoso influencia diretamente na capacidade de umectação do mesmo [ABOUSHELIB; SLEEM, 2014], assim, um cimento com uma maior viscosidade possui uma menor capacidade de penetração nas irregularidades na superfície cerâmica causadas pelos tratamentos de superfície [HITZ et al., 2012]. Em um estudo comparando cimentos resinosos convencionais com e sem MDP em uma cerâmica vítrea e uma cerâmica de zircônia, concluíram que o uso de cimento com MDP proporciona maior força de união entre ambas cerâmicas e o substrato usado [ABOUSHELIB et al., 2009]. Porém em um estudo de Secilmis, Ustun e Buyukhatipoglu [2016], com ZLS, o cimento sem MDP obteve os melhores resultados.

Já cimentos resinosos autoadesivos, contêm monômeros de metacrilato multifuncionais que são ionizados no momento da mistura, reagindo com a porção mineral de hidroxiapatita do tecido dentário e com a camada de smear layer da dentina subjacente [PEDREIRA et al., 2016]. Além disso, esses cimentos apresentam um menor estresse de contração durante a sua polimerização, resultando em menor tensão interfacial, que é considerada uma fonte de falhas da interface de união [SADIGHPOUR et al., 2018], o que influencia diretamente na sobrevida e estabilidade da restauração.

Além do cimento, o envelhecimento da interface adesiva deve ser levado em consideração quando se trata de longevidade de restaurações. Por estarem em meio aquoso, na presença de constante fluxo salivar e trocas de pH, os cimentos resinosos estão sujeitos a um envelhecimento a longo prazo, o que pode afetar negativamente as suas propriedades mecânicas e adesivas [CENCI et al., 2008; WAHAB; SHAINI; MORGANO, 2003]. A ciclagem térmica é a forma mais eficiente de envelhecimento em laboratório, sendo um método que simula os estímulos encontrados na cavidade bucal, através da ingestão de substâncias quentes e frias, levando ao envelhecimento dos materiais intrabucais que possuem diferentes coeficientes de expansão térmica [CENCI et al., 2008]. Müller, Rohr e Fisher [2017] mostraram em seu estudo que mudanças de temperatura cíclicas podem levar ao estresse do material, como resultado de diferentes coeficientes de expansão e condutividade térmica, levando a micro trincas no cimento resinoso, aumentando sua absorção e dissolução em água, afetando assim suas propriedades.

Segundo Al Akhali et al. [2017] há pouca informação disponível sobre a confiabilidade, longevidade, estabilidade e comportamento de materiais mais recentes, como a cerâmica ZLS, que podem ser influenciados por fatores como microestrutura, carga dinâmica, técnicas de fabricação e método de cimentação. Por estarem suscetíveis a cargas cíclicas [GONZAGA et al., 2015] e em um ambiente úmido [MORIMOTO et al., 2016], o conjunto restaurador pode sofrer diferentes tipos de falhas. Estas falhas podem ser provenientes de defeitos internos do material restaurador e se propagar sob tensão constante e abaixo da resistência crítica do material [ZOGHEIB et al., 2015]; por uma falha na cimentação [YU et al., 2018] ou por um condicionamento deficiente ou inefetivo da superfície cerâmica [ABOUSHELIB et al., 2009], podendo levar a restauração à fratura ou descimentação. Por isso estudos sobre os materiais mais recentes devem ser conduzidos, para se verificar o melhor tipo de cimento e tratamento de superfície da cerâmica, para que a resistência de união e comportamento à fadiga sejam potencializados para uma maior sobrevivência da restauração em boca.

Sendo assim, a presente Dissertação está apresentada sob a forma de dois artigos científicos, cada um com objetivos distintos, a serem submetidos para o periódico *Journal of the mechanical behavior of biomedical materials.*

ARTIGO 1 - "Effect of different surface treatments and resin cements on the bond strength of a zirconia-reinforced lithium silicate ceramic"

Objetivo: Avaliar o efeito de diferentes tratamentos de superfície e cimentos resinosos na resistência de união de uma cerâmica a base de silicato de lítio reforçada por zircônia.

ARTIGO 2 – "Effect of different surface treatments and resin cements on fatigue performance of a zirconia-reinforced lithium silicate ceramic"

Objetivo: Avaliar o efeito de diferentes tratamentos de superfície e cimentos resinosos no comportamento mecânico de uma cerâmica a base de silicato de lítio reforçada por zircônia.

2. ARTIGO 1- EFFECT OF DIFFERENT SURFACE TREATMENTS AND RESIN CEMENTS ON THE BOND STRENGTH OF A ZIRCONIA-REINFORCED LITHIUM SILICATE CERAMIC

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Effect of different surface treatments and resin cements on the bond strength of a zirconia-reinforced lithium silicate ceramic

Dalla Nora F[a]; Guilardi LF[a]; Zucuni CP[a]; Valandro LF[a]; Rippe MP[a]

[a] MSciD and PhD Post-Graduate Program in Oral Science (Prosthodontics Units), Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil
Address: Avenue Roraima 1000, Building 26F, room 2383. Zip Code: 97105-900Santa Maria, Rio Grande do Sul, Brazil.

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Author's e-mails:
Fernanda Dalla Nora (fedn1994@hotmail.com)
Luís Felipe Guilardi (luisfguilardi@hotmail.com)
Camila Pauleski Zucuni (camila-svs@hotmail.com)
Luiz Felipe Valandro (lfvalandro@hotmail.com)
Marília Pivetta Rippe (**Erro! A referência de hiperlink não é válida.**)

*Corresponding author: DDS, MSc, PhD, Adjunct Professor Marília Pivetta Rippe (mariliarippe@mail.ufsm.br), Federal University of Santa Maria, Faculty of Dentistry, MSciD and PhD Post-Graduate Program in Oral Science - Prosthodontics Unit, Avenue Roraima 1000, Building 26F room 2383, Zip Code: 97105-900, Santa Maria, Rio Grande do Sul, Brazil
Phone: +55-55-3220-9276; Fax: +55-55-3220-9272.

Short title: Surface treatment and resin cements on adhesion of a zirconia-reinforced lithium silicate.

Effect of different surface treatments and resin cements on the bond strength of a zirconia-reinforced lithium silicate ceramic

Abstract

Objective: The aim of the present study was to assess the effect of different ceramic surface treatments and resin cements on the bond strength to a zirconia-reinforced lithium silicate (ZLS).

Material and Methods: ZLS ceramic blocks (18 × 14 × 12 mm) were sectioned in rectangular (14 × 12 × 1 mm) slices, embedded in acrylic resin and allocated in 9 groups (n= **36)** according to two factors: 'ceramic surface treatment' (**HF** - hydrofluoric acid etching; **EP** - self-etching ceramic primer; and **SB** - sandblasting with silica-coated alumina) and 'resin cements' (**nMDP** - MDP-free conventional resin cement, **MDP** - MDP-containing conventional resin cement, and **SA** - self-adhesive resin cements). Starch tubes were placed over the treated ceramic surface on which the different types of cements were applied. After 24 h from cementation, all starch tubes were removed and the specimens were thermally aged (5,000 thermal cycles between 55°C and 5°C). The microshear bond strength test was performed using the wire-loop technique. The bond strength values were analyzed by Kruskal-Wallis and Dunn's post-hoc tests. Topographic and failure analyses were also performed.

Results: The microshear bond strength results were statistically influenced by the different ceramic surface treatment and resin cement used. Considering the type of surface treatment, the HF and the EP provided higher values of bond strength with the self-adhesive cement, and the SB with the conventional resin cement and the self-adhesive cement. Considering the type of cement, the MDP-free conventional one showed better results with the HF or SB treatments and the MDP-containing and the self-adhesive cements performed better results with HF etching.

Significance: The type of surface treatment seems dependent on type of cement, EP presented the highest values of bond strength with self-adhesive cement, SB with conventional and self-adhesive resin cement, and HF with all cements investigated in this study, mainly with self-adhesive cement, which presented the best bond strength values.

Keywords: Dental ceramics. Lithium silicate ceramics. Adhesive bonding. Thermocycling. Microshear bond strength.

Highlights:

-The ideal type of surface treatment seems dependent on the cement.

-EP presented the highest values of bond strength with self-adhesive cement and SB with conventional and self-adhesive cement

-HF presented good values of bond strength with all cements investigated in this study, mainly with self-adhesive cement, which presented the best bond strength values.

1. Introduction

Monolithic ceramic restorations have been widely used as consequence of advances in the dentistry, mainly through development of the Computer-Aided Design/Computer-Aided Machining (CAD/CAM) technology, which has enabled clinicians to provide precise high-quality ceramic restorations [Ramakrishnaiah et al., 2016; Hu et al., 2016]. An option of ceramic with a variety of clinical applications is the zirconia-reinforced lithium silicate glass-ceramic (ZLS). This material is a glass ceramic reinforced by lithium-metasilicate (Li_2SiO_3) crystals with zirconium dioxide (ZrO_2; 8-14 wt % [Hamza & Sherif, 2017]) dissolved in the glass matrix [Elsaka & Elnaghy, 2016; Traini et al., 2016], and hence provides good aesthetic and mechanical requirements [Üstün et al., 2016].

However, for the longevity of restorations, not only the mechanical behavior ceramic is important, but also the adhesion between tooth and ceramic, being the surface treatment ceramic indispensable for the success of the set. This procedure improves the free surface energy, increasing the wettability of the surface, which favors the micromechanical bond between resin cement and the dental ceramic [Strasser et al., 2018; Tsujimoto et al., 2017].

The hydrofluoric (HF) acid etching of glass ceramics is well established in the literature as an effective surface treatment, since it dissolves the vitreous matrix selectively, creating micropores on the surface, in which the resin cement penetrates, promoting a mechanical interlocking [Ramakrishnaiah et al., 2016; Zogheib et at., 2011]. In addition, the exposition of the silica crystals interacts with the silane agent that is copolymerized with methacrylate groups of the organic matrix of the resin cements [Suldfeld Neto et al., 2015].

However, according to Al-Thagafi et al., [2016], sandblasting with CoJet system presents superior bond strength results compared to 5% hydrofluoric acid conditioning with ZLS ceramic. In this sense, the sandblastingof the ceramic surface with silica-coated aluminum oxide would be a good surface treatment option [Silva et al., 2016].

Another surface treatment is self-etching ceramic primers, which have emerged as a less toxic and easy-to-apply technique that allows surface etching and silanization in one single step, reducing the clinical time and technique sensitivity [Wille et al., 2017]. Such primer also selectively etches the ceramic surface, but, unlike the HF acid etching, did not cause significant changes in the ZLS ceramic surface, being less likely to leave unfilled flaws [Strasser et al., 2018].

Besides an efficient surface treatment, the cement is equally important for a long-lasting tooth/restoration bonding [Vargas et al., 2011]. In an attempt to provide an easy and fast handling and less-sensitive bonding technique, different simplified resin cement systems (e.g., self-adhesive) have emerged in addition to the conventional resin cements. The self-adhesive resin cements bond to the dental substrate without the need of a tooth pretreatment, since it allows the formation of chemical bonding between the self-adhesive resin and the hydroxyapatite [Pedreira et al., 2016; Tunc et al., 2017], but still requires a ceramic surface pretreatment [Simões et al., 2016].

On the other hand, the conventional resin cements, require a bonding agent between their resinous matrix and the dental surface [Lührs et al., 2010]. Furthermore, some cements have the incorporation of bifunctional phosphate monomers (e.g., 10-methacryloxydecyl dihydrogen phosphate – 10-MDP) in their composition, that chemically bonds to the ceramic surface oxides through its phosphate ester group [Tunc et al., 2017] and to the resin matrix of the cement through its methacrylate group [Kern, 2009]. Despite of some studies indicate a good performance of MDP containing cement resin on bond strength of different ceramic materials [Aboushelib et al 2009; Franco-Tabares et al., 2019; Lumkemann et al., 2019], few studies compared different cements with ZLS ceramic, and according to Secilms et al [2016] the conventional cement without MDP presented better bond strength results than cement with MDP.

Thus, the literature is still not conclusive about different surface treatments for ZLS when using different resin cement systems. Therefore, the purpose of this *in vitro* study was to evaluate the influence of the ceramic surface treatments and the different resin cement systems on the microshear bond strength to a zirconia-reinforced lithium silicate glass-ceramic. The null hypotheses were that the 1) surface treatments and the 2) resin cement type have no influence on the final bond strength results.

2. Materials and Methods

The materials used in this study are described in Table 1.

Table 1 – Materials used in the study and respective characteristics (commercial name, manufacturer and chemical composition); Ceramic surface treatments; Study groups.

MATERIALS

Commercial name and manufacturer	**Chemical composition**
Zirconia-reinforced lithium silicate glass-ceramic, VITA Suprinity – VITA Zahnfabrik	SiO_2; Li_2O; K_2O; P_2O_5; ZrO_2; Al_2O_3; CeO_2; pigments.
Hydrofluoric acid 5% (IPS Ceramic Etching-gel) - Ivoclar Vivadent	Hydrofluoric acid < 5%.
Silane (Prosil) - FGM	3-metacriloxipropiltrimetoxisilano (< 5%); ethanol (> 85%); water (< 10%).
Monobond Echt & Prime - Ivoclar Vivadent	Tetrabutyl ammonium dihydrogen trifluoride, methacrylate phosphoric acid ester, trimethoxysilylpropyl methacrylate, alcohol, water.
MDP-free conventional resin cement (Multilink automix – Ivoclar Vivadent)	Dimethacrylate and hydroxyethyl methacrylate, barium glass and silica filler (68 wt%)[1], ytterbium trifluoride, catalysts, stabilizers, pigments.
MDP-containing conventional resin cement (Panavia F 2.0 – Kuraray Noritake)	10-methacryloxydecyl dihydrogen phosphate, bisphenol-A-polyethoxy dimethacrylate, hydrofobic aliphatic methacrylates, hydrophilic aliphatic methacrylate, silanated silica filler (78 wt %)[2], silanated barium glass filler, sodium fluoride.
Self-adhesive resin cement (RelyX U200 – 3M ESPE)	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers (72 wt %), initiator components, stabilizers, pigments, rheological additives [3].

[1][Baena et al.,2012]; [2][Giti et al., 2016]; [3][Burey et al., 2017]

2.1 Preparation of ceramics specimens

Rectangular slices (14 × 12 × 1 mm^3) were cut from zirconia-reinforced lithium silicate glass-ceramic blocks (18 × 14 × 12 mm^3; Vita Suprinity, Vita Zahnfabrik, BadSäckingen, Germany) with a diamond blade in a cutting machine (Isomet 1000, Buehler, Lake Bluff, Illinois, USA) under constant water cooling. The 36 slices obtained were manually polished in both sides with silicon carbide papers of different grit sizes (#400, #600 and #1200-grit; Norton Abrasives, Saint-Gobain; São Paulo, SP, Brazil). To simulate the roughness obtained from CAD/CAM milling, the specimens were ground single side with a #60-grit silicon carbide sandpaper (Norton Abrasives, Saint-Gobain) (15 times on the x and y axis), providing an initial roughness (mean Ra= 1.98 µm and mean Rz= 12.88 µm), before crystallization, similar to that of CAD/CAM milled restorations [Vichi et al., 2018; Madruga et al., 2019]. The specimens were washed in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ind. And Com. Equip. Med.

Odonto. LTDA, Ribeirão Preto, Brazil) for 10 minutes in distilled water, air-dried for 30 seconds and crystallized in a specific oven according to the manufacturer's instructions (VACUMAT 6000 MP, Vita Zahnfabrik, 840 ˚C, 8 min vacuum).

Each ceramic slice was embedded in a cylindrical polyvinyl chloride (PVC) mold using a self-cured acrylic resin (VIPI Flash, Pirassununga, Brazil). For this, a double-sided tape (3M ESPE, Saint Paul, USA) was used to keep the ground surface free for cementation. After the final polymerization of the acrylic resin, the specimens were washed in an ultrasonic bath, as previously reported, to remove any glue remnant. The slices were randomly assigned (www.randomizer.org) into nine groups (n= 36) according to the study factors: surface treatment (hydrofluoric acid etching, self-etching ceramic primer or sandblasting) and type of resin cement (MDP-free conventional resin cement, MDP-containing conventional resin cement, and self-adhesive resin cements), as shown in Table 2.

Table 2 - Study design and study groups.

Resin cements	**Surface treatments**	**Study groups**
Multilink Automix – **nMDP**	**HF** ❼ 5% hydrofluoric acid etching (IPS Ceramic etching-gel) + Silane (Prosil)	**nMDP+HF** **nMDP+EP** **nMDP+SB**
Panavia F 2.0 - **MDP**	**EP** ❼ Self-etching ceramic primer (Monobond Etch & Prime)	**MDP+HF** **MDP+EP** **MDP+SB**
RelyX U200 –**SA**	**SB** ❼ Sandblasting with silica-coated alumina (CoJet Sand) + Silane (Prosil)	**SA+HF** **SA+EP** **SA+SB**

2.2 Ceramic surface treatments

-Hydrofluoric acid etching (HF)

The hydrofluoric acid (5% IPS Ceramic Etching Gel, Ivoclar Vivadent; Schaan, Liechtenstein) was applied and scrubbed on the ceramic surface with a microbrush for 20 s, as recommended by the manufacturer. After, the specimens were washed with air/water-spray for 30 s, subjected to the ultrasonic bath (1440D, Odontobras, Ind And Com. Equip Med. Odonto Ribeirão Preto, Brazil) with distilled water for 5 min [Venturini et al., 2015] and air-spray dried for 30 s. Right after, the silane coupling agent (Prosil, FGM, Joinville, SC, Brazil) was actively applied for 15 s with a microbrush, kept to react for 60 s and gently air-spray dried for 15 s.

-Etch & Prime ceramic primer (EP)

The self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent) was actively applied on the ceramic surface with a microbrush for 20 s, kept to react for 40 s, washed with air/water-spray for 20 s, and air-spray dried for 30 s.

- *Sandblasting (SB)*

The sandblasting was performed with 30 μm silica-coated aluminum oxide particles (CoJet Sand, 3M ESPE, Seefeld, Germany) on the ceramic surface using a micro-etcher (DENTO-PREP microblaster, Ronvig, Daugaard, Denmark) at a distance of 15 mm from the blast nozzle to the ceramic surface, with a pressure of 2.5 bar [Sato et al., 2016], in oscillatory movements for 15 s [Al-Thagafi et al., 2016]. After that, a gentle air-spray was applied to remove loose particles, and a silane coupling agent (Prosil, FGM) was actively applied for 15 s, kept to react for 60 s and gently air-spray dried for 15 s.

2.3 Microshear resin cement sample

After ceramic surface treatment, 36 starch tubes (12 starch tubes each ceramic slice, 3 ceramic slice each group) (Renata, Pastificio Selmi, Londrina, Brazil) with 1.0 mm of height and 0.96 mm of internal diameter [Tedesco et al., 2013] were placed on the treated ceramic surface and fixed at the external surface with sticky wax (Lysanda, São Paulo, Brazil) to keep them in position.

-Conventional resin cement (nMDP- Multilink Automix)

The base and catalyst pastes of the resin cement were dispensed from the double-push syringe, mixed for 20 s, resulting in a homogeneous mixture and inserted with the aid of a probe into the starch tubes.

MDP-containing conventional resin cement (MDP- Panavia F2.0)

The base and catalyst pastes of the resin cement were dispensed from the syringes, mixed for 20 s, resulting in a homogeneous mixture and inserted with the aid of a probe into the starch tubes.

- Self-adhesive resin cement (SA - RelyX U200)

The base and catalyst pastes of the resin cement were dispensed from the double-push syringe, mixed for 20 s, resulting in a homogeneous mixture and inserted with the aid of a probe into the starch tubes.

The cement excesses were carefully removed with the aid of a microbrush and the cement was polymerized with a LED apparatus (1200 mW/cm^2 of intensity; Radii Cal, SDI; Bayswater, Australia) for 40 s, and the specimens were stored in distilled water in a laboratory incubator at 37°C for 24 h. After that, the starch tubes were decomposed and could be carefully removed with a probe clinical. The adhesive interface of the specimens was analyzed in stereomicroscope (Stereo Discovery V20, Carl-Zeiss, Gottingen, Germany) for verification of bubbles or defects. Cement cylinders with defects in the interface were discarded and substituted. The adhesive procedures were performed by a single trained operator and at a room temperature of 25°C.

2.4 Aging – thermocycling

After 24 hours, all specimens underwent intermittent 5,000 thermal-cycles (Ethik Technology Limited – model 521-6D; Vargem Grande Paulista, SP, Brazil) with temperature ranging from 5 to 55°C with 30 s of dwell time at each temperature and 4 s of transfer time [Üstün et al., 2016; Secilms et al., 2016].

2.5 Microshear bond strength test

PVC cylinders were placed in a jig attached to a universal testing machine (Emic DL1000, São José dos Pinhais, Brazil). A stainless-steel wire loop (Ø= 0.20 mm) was placed juxtaposed against the ceramic surface and in contact with the lower semicircle of the resin cement cylinder in the adhesive interface. The cement cylinder was positioned in alignment with the center of the load cell, and the wire held parallel to the direction of movement of the load cell and to the adhesive interface. The shear load was applied (10 N load cell) at a rate of 0.5 mm/min until failure occurred. The data were recorded in MPa.

2.6 Failure analysis

All specimens submitted to the bond strength test were analyzed in stereomicroscope (Discovery V20, Carl-Zeiss, Germany) with 10-50 × of magnification to verify the type of failure. The failures were classified as adhesive (more than 50% of resin cement free adhesive area) or cohesive (more than 50% of the adhesive area with the presence of resin cement). Representative samples were selected and analyzed in scanning electron microscope (SEM) (TESCAN VEGA3, TESCAN, Brno, Czech Republic) at 100× and 230× of magnification.

2.7 Topographic analysis

Two additional specimens were produced, submitted to the surface treatment under study, ultrasonically cleaned, dried in a laboratory desiccator and gold-sputtered to be analyzed at the SEM (TESCAN VEGA3, TESCAN, Brno, Czech Republic) for the evaluation of ceramic surface characteristics after the different treatments.

2.8 Statistical analysis

The normality (Shapiro-Wilk test) and homoscedasticity (Levene test) of the data were tested and the bond strength was analyzed using the Kruskal-Wallis and Dunn's post-hoc test. All specimens which presented pre-testing failures were substituted a pre-defined criterion for determining values (0.1MPa) [Davies, 1973].

3. Results

The microshear bond strength results were statistically influenced by the different ceramic surface treatment and resin cement used. Considering the type of surface treatment, the HF and the EP provided higher values of bond strength with the self-adhesive cement, and the SB with the conventional resin cement and the self-adhesive cement. Considering the type of cement, the MDP-free conventional one showed better results with the HF or SB treatments and the MDP-containing and the self-adhesive cements performed better results with HF etching (Table 2).

The groups treated with the self-etching ceramic primer showed a considerably greater number of pre-test failures (Table 2). Failure analysis showed a greater number of adhesive failures for all groups (Fig.1 and 2).

Table 2 - Mean and standard deviation of bond strength in MPa (Kruskal-Wallis and Dunn's post-hoc test);

RESIN CEMENTS	**SURFACE TREATMENTS**		
	HF	**EP**	**SB**
nMDP	7.44 (4.21)^{Ab}	3.87 (3.53)^{Bb}	7.51 (4.46)^{Aa}
MDP	7.59 (4.3)^{Ab}	2.7 (3.83)^{Bb}	2.49 (2.61)^{Bb}
SA	14.13 (6.25)^{Aa}	7.03 (7.9)^{Ba}	6.91 (3.98)^{Ba}

‡Different uppercase letters show statistically significant difference between surface treatments for the same resin cement ($p<0.05$).

‡Different lowercase letters show statistically significant difference between the resin cements for the same surface treatment ($p<0.05$).

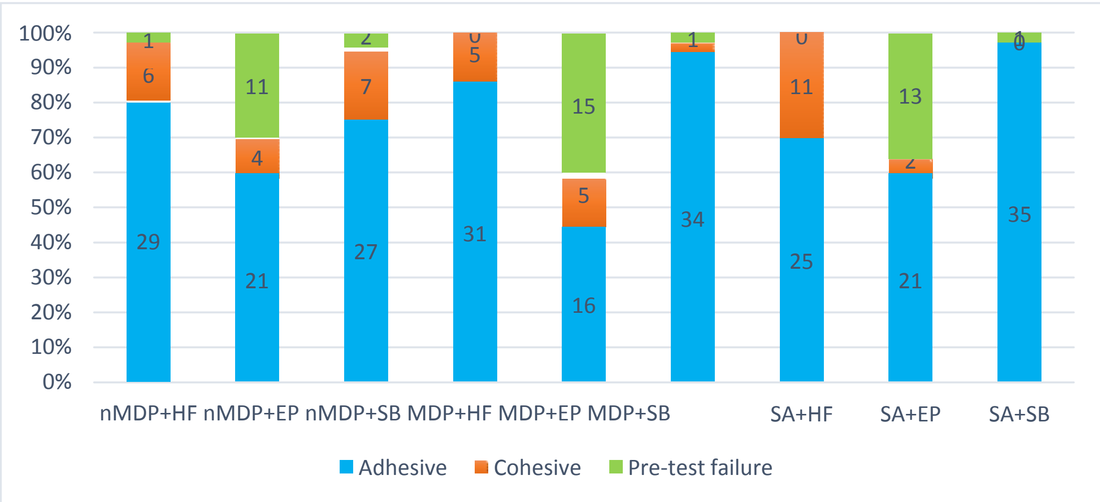

Figure 1 – Number and percentage for types of failure and pre-test failures of each experimental group submitted to the microshear bond strength test.

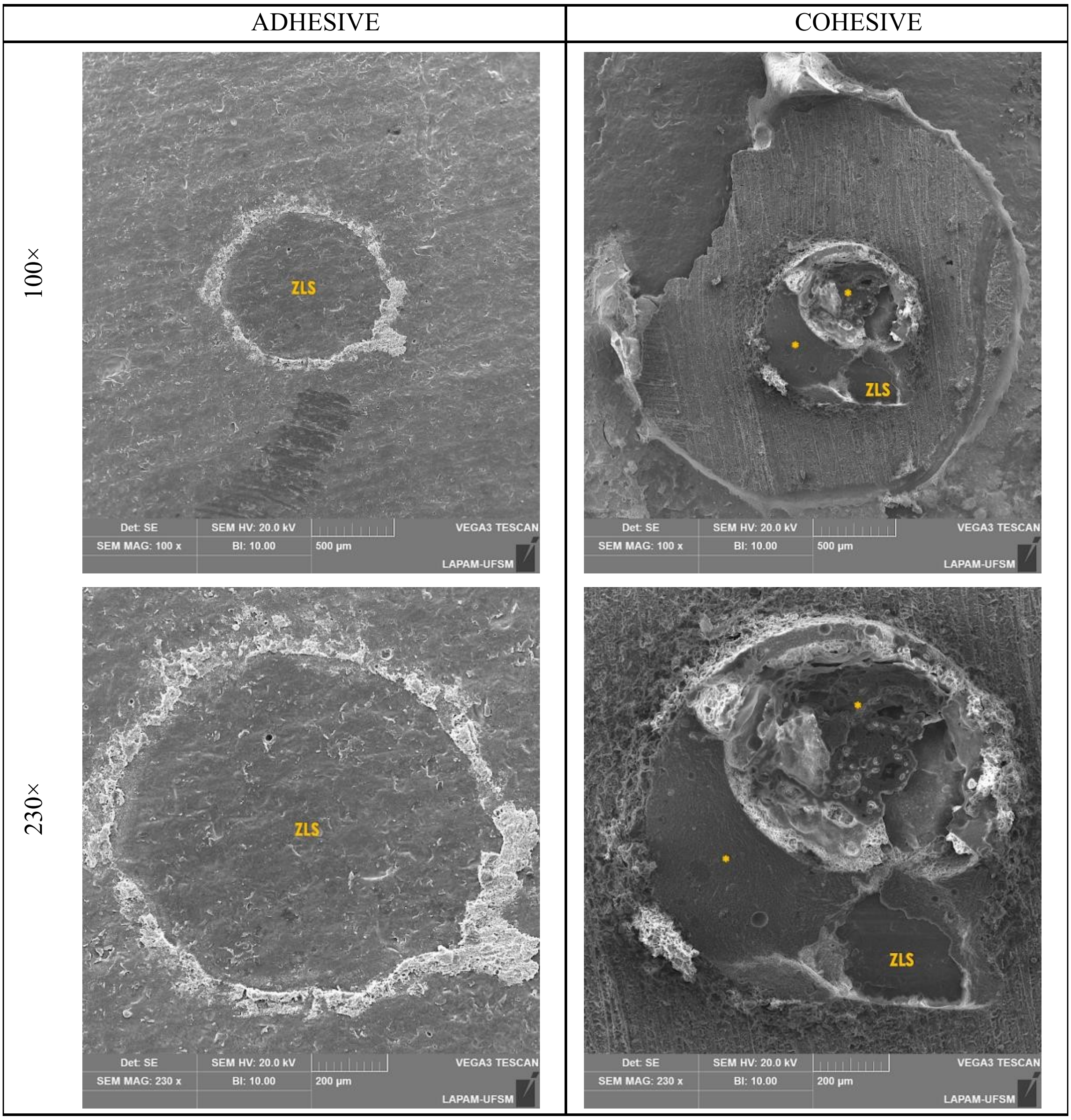

Figure 2 – SEM images (100× and 230× of magnification) of the different types of failure after the microshear bond strength test.

Micrographs show that the different ceramic surface treatments promoted different topographical patterns. Sandblasting created a more irregular surface pattern, introducing flaws and scratches. The hydrofluoric acid etching seems to soften the ceramic surface and the self-etching ceramic primer does not seem to alter the ceramic topography considerably (Fig. 3).

25

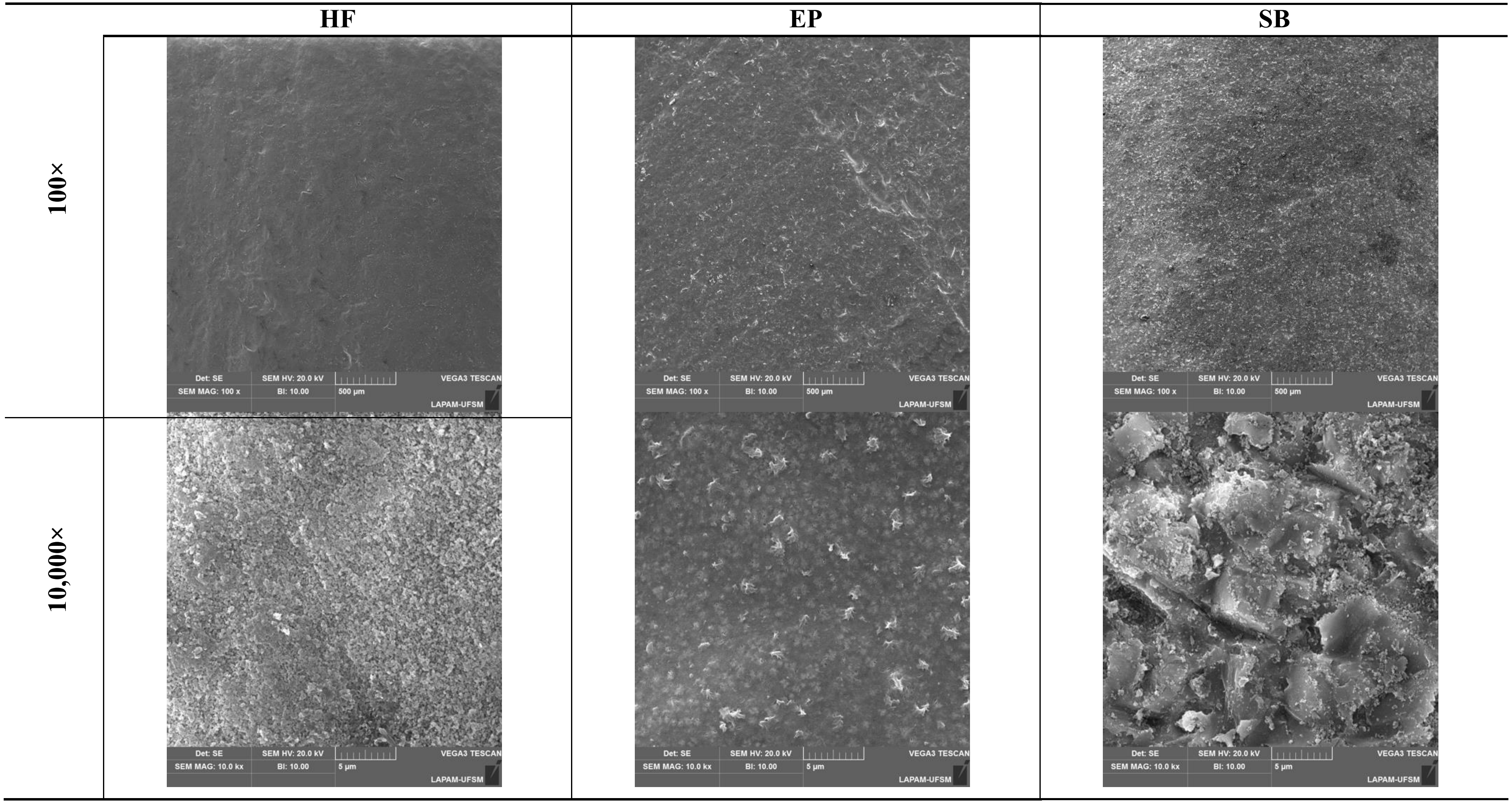

Figure 3 - Topographic images on Scanning Electron Microscopy (100× and 10,000× of magnification) of the ceramic surface after the surface treatments: 5% hydrofluoric acid etching (HF), Monobond Etch & Prime application (EP) and sandblasting (SB).

4. Discussion

The results of the study showed that the bond strength to a ZLS ceramic was directly influenced by the surface treatment and the type of cement applied, so the null hypotheses were rejected. In our study the 5% HF etching plus the silane agent application obtained one of the best values of bond strength for each cement, corroborating with many other studies [El-Damanhoury and Gaintantzopoulou., 2017; Strasser 2018; Prado et al., 2018]. The HF etching followed by the silane agent application still is the most commonly accepted method for the pretreatment of glass ceramics, with conditioning time adapted for each specific material [Strasser et al., 2018].

Due to the HF acid potential toxicity [Özcan et al., 2012], alternative methods have been tested and new materials have emerged. Monobond Etch & Prime is composed of a silane, a ceramic agent and a priming agent (Table 1) in a single bottle, thus allowing surface etching and silanization in a single step, reducing clinical time and toxicity [El-Damanhoury and Gaintantzopoulou., 2017]. However, in our study, the groups treated with the ceramic primer presented one of the lowest bond strength values, the highest number of pre-test failures. Prado et al. [2018] also observed a greater number of pre-test failures for a lithium disilicate treated with this ceramic primer. El-Damanhoury and Gaintantzopoulou [2017] and Lopes et al. [2018] found lower values of bond strength when the lithium disilicate was conditioned with the ceramic primer, compared to HF etching in different concentrations, corroborating with our study. One of the reasons this worse performance of the EP may be attributed to the low capacity of the ceramic primer to create microporosities in the ceramic surface [El-Damanhoury and Gaintantzopoulou., 2017].

In relation to sandblasting, in our study, this surface treatment showed bond strength values similar to HF only for conventional resin cement without MDP, showing low bond strength values with the other cements. Also Sato et al. [2016] reported a significant decrease in the bond strength of a zirconium-reinforced lithium silicate vitreous ceramic when the surface treatment used was silicatization after thermocycling, suggesting that this surface treatment does not provide a stable bond. In addition, a rougher ceramic surface can have a deleterious effect on mechanical properties when defects are not properly filled by the adhesive bonding [Addison & Fleming, 2008; Yi & Kelly., 2011; Spazzin et al., 2016).

The filling capacity presented by each type of cementation system is determined by the type, size and content of the particles and its organic matrix composition, which directly reflects the viscosity of the cement [Di Francescantonio et al., 2013; Lopes et al., 2015]. The lower the viscosity, the greater the capacity of the cement to infiltrate the irregularities present in the ceramic surface, improving the filling of defects [Hitz et al, 2012]. It also provides a thinner cement thickness, allowing a lower shrinkage stress and reducing the possibility of gaps and sorption and solubility of the resin cement [Mese et al., 2008 Oliveira et al., 2010], strengthening the restoration. About the type of resin cement, in our study, better results were found when self-adhesive resin cement was used (Table 2). The self-adhesive resin cement (RelyX U200) has a low viscosity and consequently better wets the ceramic, which gives it a higher capacity to penetrate and seal the defects created by the surface treatment [Aboushelib & Wang, 2010], further protecting the accelerated degradation of the bonding interface due to the thermocycling process, which may compromise the resin cements mechanical properties [Muller et al., 2017; Medeiros et al., 2007]. Also, the methacrylate monomers with phosphoric ester functional groups in the self-adhesive resin cement are able to improve adhesion by creating hydrogen bonds with the ceramic surface [Tunc et al., 2016].

One of the limitations of this *in vitro* study was the pre-test failures of the EP groups. It can be explained due to lower chemical interaction between this surface treatment and the conventional resin cements, despite this, more than the half of the specimens were tested in these groups. Another limitation was the number of cohesive failures, but they are inherent of the microshear tests, which generate a non-homogeneous load distribution on the interface, which is associated with adhesive interface distal failures [Al-Thagafi et al., 2016]. Furthermore, cohesive failures mean the bond within the material itself is stronger than the bond at the interface [Secilms et al., 2016], and it justifies the high microshear bond values. By that, further *in vitro* and clinical studies should be carried out to evaluate this issue, with other surface treatments and cements in an attempt to elucidate the best adhesive approach for zirconia-reinforced lithium silicate glass-ceramics.

5. Conclusions

Based on the finding of this *in vitro* study, it can be concluded that:

- The bond strength to the zirconia-reinforced lithium silicate glass-ceramic is directly influenced by the ceramic surface treatment performed and the resin cement used.
- Applying the self-adhesive resin cement after hydrofluoric acid etching provided better bond strength to the ZLS ceramic.
- Self etching ceramic primer presented a greater number of pre-test failure.

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3. ARTIGO 2- EFFECT OF DIFFERENT SURFACE TREATMENTS AND RESIN CEMENTS ON FATIGUE PERFORMANCE OF A ZIRCONIA-REINFORCED LITHIUM SILICATE CERAMIC

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Effect of different surface treatments and resin cements on fatigue performance of a zirconia-reinforced lithium silicate ceramic

Dalla Nora F[a]; Guilardi LF[a]; Zucuni CP[a]; Valandro LF[a]; Rippe MP[a]

[a] MSciD and PhD Post-Graduate Program in Oral Science (Prosthodontics Units), Faculty of Dentistry, Federal University of Santa Maria (UFSM), Santa Maria, Rio Grande do Sul State, Brazil Address: Roraima Avenue, #1000,26F Building. Zip Code: 97.105-90 Santa Maria, Rio Grande do Sul, Brazil.

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Author's e-mails:
Fernanda Dalla Nora (fedn1994@hotmail.com)
Luís Felipe Guilardi (luisfguilardi@hotmail.com)
Camila Pauleski Zucuni (camila-svs@hotmail.com)
Luiz Felipe Valandro (lfvalandro@hotmail.com)
Marília Pivetta Rippe (mariliarippe@mail.ufsm.br)

*Corresponding author: DDS, MSc, PhD, Adjunct Professor Marília Pivetta Rippe (mariliarippe@mail.ufsm.br), Federal University of Santa Maria, Faculty of Odontology, MDS-PhD Graduate Program in Oral Science - Prosthodontics Unit, Roraima Avenue, #1000,26F Building. Zip Code: 97.105-90, Santa Maria, Rio Grande do Sul, Brazil. Phone: +55-55-3220-9276, Fax: +55-55-3220-9272.

Short title: Cementing strategies and fatigue of zirconia-reinforced lithium silicate ceramic.

Effect of different surface treatments and resin cements on fatigue performance of a zirconia-reinforced lithium silicate ceramic

Abstract

Objective. The aim of the present study was to assess the fatigue failure load, number of cycles for failure, and survival rates of a zirconia-reinforced lithium silicate (ZLS) cemented on dentin analogue with different ceramic surface treatments and resin cements.

Material and Methods. ZLS ceramic discs and dentin analogue (diameter Ø = 10 mm, thickness = 1.5 mm and 2.0 mm respectively) were produced and divided according to the study factors: 'ceramic surface treatment' (HF - hydrofluoric acid etching, EP - self-etching ceramic primer and SB - sandblasting with silica-coated alumina particles) and 'resin cements' (nMDP - MDP-free conventional resin cement, MDP - MDP-containing conventional resin cement, and SA - self-adhesive resin cements). The ceramic discs were cemented on the dentin analogue and all the specimens were aged for 5,000 thermal cycles (5-55 °C) prior to the fatigue test. The stepwise fatigue test (20 Hz frequency) started with a load of 400 N (5,000 cycles) followed by steps of 500, 600, 700, up to 1800 N at a maximum of 10,000 cycles each step. The specimens were loaded until failure detection by light transillumination and visual inspection at the end of each step. Data from fatigue failure load (FFL), number of cycles for failure (CFF) was recorded and submitted to survival statistical analysis Kaplan-Meier (log rank test at α=.05). Topographic and fractographic analyses were also performed.

Results. HF and EP failed at statistically similar loads being higher than SB, regardless of cement type used. When the type of cement was considered singly, the self-adhesive cement obtained statistically greater results for FFL, CFF and survival rates, for the HF and SB treatments, and no difference among cements was found when applying EP. Overall, at 900 N all specimens submitted to sandblasting had already failed, while more than 47% for HF and at least 27% for EP had survived.

Significance. Different surface treatment of the intaglio zirconia-reinforced lithium silicate ceramic and its bonding with different resin cements affect its survival rate and fatigue performance. Sandblasting should be avoided for treating the ZLS surface prior to bonding.

Keywords. Dental ceramics. Lithium silicate glass-ceramic. CAD/CAM. Fatigue performance. Surface treatment.

Highlights

- Hydrofluoric acid and self-etching primer presented the best fatigue performance among the surface treatments tested.
- Sandblasting created the lowest fatigue failure load results, regardless of the cement used.
- Self-adhesive resin cement presented the best fatigue performance among the resin cements tested for hydrofluoric acid and sandblasting surface treatment.

1. Introduction

The zirconia-reinforced lithium silicate glass-ceramic (ZLS) is composed of a glassy matrix reinforced by lithium silicate crystals and dissolved zirconium oxide (56-64% silicon dioxide, 15-21% lithium oxide, 8-12% zirconia), among other components [Gracis et al., 2016; Kruzic et al., 2018]. The reduction in the glass content and the crystal microstructure (0.5 - 0.7 μm) of the lithium silicate creates a material with high strength and easy machining and polishing associated to good aesthetic properties [Rinke et al., 2016; Wendler et al., 2017]. Due to its glassy content, this ceramic is considered acid-sensitive, being the protocol more recommended for adhesive bond with resin cements, the etching with hydrofluoric acid (HF) followed by the silane coupling agent application [Tian et al., 2014; Manicone et al, 2007].

The HF acid partially attacks the glassy matrix and expose the crystalline microstructure of the ceramic surface creating a micromechanical pattern that, added to the siloxane bonds provided by the silane between the exposed silica and methacrylate group of the resin cement [Siqueira et al., 2016], results in a good bond strength [Hu et al., 2016; Manicone et al., 2007]. However, it is not known if all the microporosities and defects created by the HF are completely filled by the cement, which could weaken the restoration by reducing its resistance to intermittent masticatory loading over time [Spazzin et al., 2016]. In addition, the HF acid is considered to be potentially toxic to human health and should be used with caution or even avoided [Ozcan et al., 2012; Tian et al., 2014]. Therefore, it is very important to understand this mechanism and look for safer alternative surface treatments.

A promising possibility for dental glass-ceramic treatment is the use of a self-etching ceramic primer, which is a single-step ceramic initiator that facilitates handling and reduces the clinical time and the technique sensitivity when compared to the conventional HF etching. But there is few information available in the literature about the efficacy of these type of product [Wille et al., 2017; El-Damanhoury & Gaintantzopoulou, 2018]. Wille et al. [2017] showed that the one-step ceramic primer (Monobond Etch & Prime; Ivoclar Vivadent) provided bond strength to the lithium disilicate ceramic comparable to the well-established HF etching plus silane application. Indicating that this surface treatment may be promising for use in glass ceramics. However, in a study of Strasser et al. [2018], the use of this self-etching ceramic primer did not cause significant changes in the surface of a zirconium-reinforced lithium silicate ceramic when compared to 5% HF acid etching. That shows how the literature is still controversial about this subject.

Another alternative for ZLS could be the tribochemical silica-coating (TSC), since Al-Thagafi et al [2016] observed in their study higher bond strength values with ZLS ceramics when surface treatment was carried out with blasting silane-coated aluminum oxide particles associated with silane when compared with conditioning with 5% HF associated with silane. The TSC consists in silica deposition on the ceramic surface through a sandblasting process, and application of a silane-based coupling agent. The CoJet (SiO_2-coated Al_2O_3, 30 µm particles, CoJet Sand, 3M ESPE) system is a versatile and portable option for clinical use, which it creates chemical bonds by applying mechanical energy.

The increase in bond strength is also directly influenced by the cement characteristics like viscosity, elastic modulus and its ability to fill the defects created during surface treatment [Addison et al., 2010; Spazzin et al., 2016]. There is a high tendency to simplify and reduce steps of adhesive bonding process in dentistry, and different simplified resin cements were created (e.g., self-adhesive). This kind of cement was created to chemically interact with the dentin without the need of previous etching it, but it still requires the ceramic surface treatment [Simões et al., 2016]. In addition, they exhibit a lower polymerization shrinkage, resulting in less tooth/restoration interface failure, presenting bond strength values similar to conventional MDP cements when used with hybrid ceramics [Sadighpour et al., 2018].

On the other hand, resin cements containing phosphate monomers, like the 10-MDP (10-Methacryloyloxydecyl dihydrogen phosphate), have in their composition bifunctional molecules that directly bond to silica oxides of the ceramic surface through their phosphate ester group to the resin matrix of the cement through their methacrylate groups, providing a better bond strength between the components [Kern, 2009]. Gungogdu & Aladag [2017], obtained in their study better values of bond strength when a zirconia ceramic was cemented with conventional resin-cement with MDP compared to a self-adhesive resin cement. However, there is no data in the literature about its real effect on glass ceramics such as ZLS.

Furthermore, the data present in the literature evaluating the zirconium-reinforced lithium silicate ceramics with respect to different surface treatments are scarce, especially with regards to the effects on its fatigue behavior. Thus, this study aimed to evaluate the effect of different surface treatments and resin cements on fatigue behavior of a ZLS ceramic cemented to a dentin analogue. The null hypotheses were: 1) surface treatment and 2) cement type will not influence the load for fatigue failure, number of cycles and survival rate of zirconium-reinforced lithium silicate ceramics cemented on dentin analogue.

2. Material and Methods

The materials used in this study are described in Table 1.

Table 1 - Materials used in the study and respective characteristics: commercial name, manufacturer and composition.

Material and manufacturer	Composition
Zirconia-reinforced lithium silicate glass-ceramic, VITA Suprinity - VITA Zahnfabrik	SiO_2; Li_2O; K_2O; P_2O_5; ZrO_2; Al_2O_3; CeO_2; pigments.
Hydrofluoric acid 5% (IPS Ceramic Etching-gel) - Ivoclar Vivadent	Hydrofluoric acid < 5%.
Hydrofluoric acid 10% (Condac Porcelana) – FGM	Hydrofluoric acid 10%, water, thickener, surfactant and coloring.
Monobond Etch &Prime - Ivoclar Vivadent	Tetrabutyl ammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, trimethoxysilylpropyl methacrylate, alcohol, water.
CoJet Sand - 3M ESPE	Aluminum oxide. Free amorphous synthetic silica.
MDP-free conventional resin cement (Multilink Automix - Ivoclar Vivadent)	Base: ytterbium trifluoride, ethoxylated bisphenol A dimethacrylate, Bis-GMA, 2-hema, 2-dimethylamanoethyl mathacrylate. Catalyst: ytterbium trifluoride, ethoxylated bisphenol A dimethacrylate,, urethane dimethacrylate, 2-HEMA, dibenzoyl peroxide and silica filler (68 wt%)[1], pigments.
MDP-containing conventional resin cement (Panavia F 2.0 – Kuraray Noritake)	10-methacryloxydecyl dihydrogen phosphate, bisphenol-A-polyethoxy dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic methacrylate, silanated silica filler, silanated barium glass filler (78 wt%)[2], sodium fluoride.
Self-adhesive resin cement (RelyX U200 - 3M ESPE)	Base paste: Methacrylate monomers containing phosphoric acid groups, Methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste: Methacrylate monomers, alkaline (basic) fillers, silanated fillers (72 wt%)[3], initiator components, stabilizers, pigments, rheological additives.
Silane (Prosil – FGM)	3-Metacriloxipropiltrimetoxisilano (< 5%); ethanol (> 85%); water (< 10%).

[1][Baena et al.,2012]; [2][Giti et al., 2016]; [3][Burey et al., 2017]

2.1 Specimens preparation

2.1.1 Ceramic

Pre-fabricated zirconia-reinforced lithium silicate glass-ceramic blocks (18 × 14 × 12 mm^3; VITA Suprinity, VITA Zahnfabrik, Bad-Säckingen, Germany) were ground into cylinders (Ø = 10 mm) using #150-grit size silicon carbide paper (SiC) (Norton Abrasives, Saint-Gobain; São Paulo, SP, Brazil) in a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA). After, discs with 1.7 mm of thickness were obtained in a diamond saw machine (Isomet 1000, Buehler) under constant water cooling. For the removal of any irregularities inherent to the cutting, the discs were polished with SiC #400-, #600- and #1200-grit (Norton Abrasives, Saint-Gobain) on both sides up to the final thickness of 1.5 mm. To simulate the roughness of CAD/CAM machining, the cementation surface was ground with #60-grit size SiC paper (Norton Abrasives, Saint-Gobain) for 15 times on each axis, *x* and *y*, providing an initial roughness (mean Ra= 2.11µn and mean Rz= 12.77µm) similar to that of CAD/CAM milled restoration [Vichi et al., 2018]].

The specimens dimensions were based on literature data [Kelly, 1999; Kelly et al., 2010; Chen et al., 2014], considering the mean diameter of the occlusal surface of the first permanent molars (Ø= 10 mm) [Ferrario et al., 1999], and their mean thickness based on the distance between the occlusal surface and the dental pulp chamber roof.

After finishing, the discs were washed in an ultrasonic bath (1440D, Odontobras, Ind. and Com. Equip. Med. Odonto., Ribeirão Preto, SP, Brazil) with distilled water for 10 min, dried with air-spray and crystallized in a specific furnace (VACUMAT 6000MP, VITA Zahnfabrik) according to the manufacturer’s instructions (840 ºC, 8 min vacuum).

2.1.2 Dentin analogue

Dentin analogue discs were obtained from an epoxy resin plate (2.5 mm) (elastic modulus of 18 GPa; Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany) by cutting it using a cylindrical diamond drills (Ø = 10 mm) under constant water cooling, and then ground (SiC #400-, #600- and #1200-grit sizes; Norton Abrasives, Saint-Gobain) until obtaining discs with a final thickness of 2.0 mm.

The ceramic and dentin analogue discs were randomly distributed (www.randomized.org) in nine groups (n = 15) according to the factors under study as shown in Table 2.

Table 2 - Study design and study groups.

Resin cements	Surface treatments	Study groups

Multilink Automix – **nMDP**	**HF** ❼ 5% hydrofluoric acid etching (IPS Ceramic etching-gel) + Silane (Prosil)	**nMDP+HF** **nMDP+EP** **nMDP+SB**
Panavia F 2.0 - **MDP**	**EP** ❼ Self-etching ceramic primer (Monobond Etch &Prime)	**MDP+HF** **MDP+EP** **MDP+SB**
RelyX U200 –**SA**	**SB** ❼ Sandblasting with silica-coated alumina (CoJet Sand) + Silane (Prosil)	**SA+HF** **SA+EP** **SA+SB**

2.2 Surface treatments

2.2.1 Ceramic

¬ *Hydrofluoric acid etching – HF*

Hydrofluoric acid (5% IPS Ceramic Etching Gel, Ivoclar Vivadent; Schaan, Liechtenstein) was applied and scrubbed on the ceramic surface with a microbrush for 20 s, as recommended by the manufacturer. After, the specimens were washed with air/water-spray for 30 s, gently air-dried, subjected to the ultrasonic bath (1440D, Odontobras, Ind And Com. Equip Med. Odonto Ribeirão Preto, Brazil) with distilled water for 5 minutes [Venturini et al., 2015] and air-spray dried for 30 s. Right after, the silane coupling agent (Prosil, FGM, Joinville, SC, Brazil) was actively applied for 15 s with a microbrush, kept to react for 60 s and air-dried for 15 s.

¬ *Etch & Prime ceramic primer – EP*

The self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent) was actively applied on the ceramic surface with a microbrush for 20 s, kept to react for 40 s and then washed with air/water-spray for 20 s.

¬ *Sandblasting – SB*

The sandblasting was performed with 30 µm silica-coated aluminum trioxide particles (CoJet Sand, 3M ESPE, Seefeld, Germany) in the intaglio ceramic surface using a micro-etcher (DENTO-PREP microblaster, Ronvig, Daugaard, Denmark) at a distance of 15 mm from the blast nozzle to the ceramic surface, with a blast pressure of 2.5 bar [Sato et al., 2016], in oscillatory movements for 15 s [Al-Thagafi et al., 2016]. After that, a light air-spray was applied to remove loose particles, and a silane coupling agent (Prosil, FGM) was actively applied for 15 s, kept to react for 60 s and air-spray dried for 15 s.

2.2.2 Dentin analogue

To simulate, as close as possible, a clinical arrangement, dentin analogue discs were used to mimic the hydrated human dentine that is similar in terms of bond strength and elastic behavior [Kelly et al., 2010].Prior to cementation, the dentin analog discs were cleaned in an ultrasonic bath (1440D, Odontobras, Ind And Com. Equip Med. Odonto Ribeirão Preto, Brazil) with distilled water for 5 min and air-spray dried. After, they were acid etched with 10% HF (Condac Porcelana, FGM, Joiville, SC, Brazil) for 1 min, that was removed with air/water-spray for 30 s, and the discs were washed in an ultrasonic bath with distilled water for 5 min and air-spray dried for 15 s [Venturini et al., 2015].

2.3 Cementation

¬ *MDP-free conventional resin cement – nMDP (Multilink Automix)*

The Primers A and B of the Multilink Automix system were mixed (1:1) and applied in the dentin analog surface for 30 s with a microbrush, and a light air-spray was applied until reach a thin layer. The base and catalyst pastes of the resin cement were dispensed from the double-push syringe, mixed and applied on the dentin analog surface.

¬ *MDP-containing conventional resin cement – MDP (Panavia F2.0)*

ED Primers liquid A and liquid B were mixed (1:1) and applied on the dentin analog surface for 60 s with a microbrush and dried with a light air-spray. The base and catalyst pastes of the resin cement were dispensed from the syringes, mixed for 20 s and applied on the dentin analog surface.

¬ *Self-adhesive resin cement – U (RelyX U200)*

The base and catalyst pastes of the resin cement were dispensed from the double-push syringe, mixed for 20 s and applied on the dentin analog surface.

After the resin cements being place in the treated dentin analog, the ceramic treated surface was placed over the cement and a constant load of 2.5 N was applied on the ceramic surface to promote a uniform spreading of the cement in order to standardize its thickness [Monteiro et al., 2018]. After removing the cement excesses with a microbrush, the light curing was performed (LED light, 1200 mW/cm^2, 440 - 480 nm, Radii-cal, SDI Limited; Bayswater, Australia) for 40 s on the 'occlusal' surface of the ceramic, followed by 20 s on each lateral side of the interface (0°, 90°, 180° and 270°).

2.4 Aging – thermocycling

After bonding, all the discs were stored submerged in distilled water at 37 °C in a laboratory incubator. After 24 hours, the specimens underwent intermittent 5,000 thermal-cycles (Ethik Technology Limited – model 521-6D; Vargem Grande Paulista, SP, Brazil) with temperature ranging from 5 to 55 °C with 30 s of dwell time at each temperature and 4 s of transfer time [Secilms et al., 2016].

2.5 Fatigue testing – Stepwise method

The specimens (n = 15) were tested submerged in distilled water in an electrodynamic testing machine (Instron ElectroPuls E3000, Instron Corp, Norwood, USA) over a flat metal base and the load was applied on the ceramic surface with a 40 mm diameter stainless steel hemispherical piston [Kelly et al., 2010]. Prior to testing, an adhesive tape (110 μm) was placed between the piston and the ceramic to improve the contact between them and to avoid contact damage [Monteiro et al., 2018]. The test started with a load of 200 N at a frequency of 20 Hz per 5,000 cycles (preconditioning phase to ensure predictable positioning of the piston with the sample), followed by steps of 400 N, 500 N, 600 N, and so on with increments of 100 N up to 1800 N, to a maximum of 10,000 cycles each step. At the end of each step the specimens were analyzed by transillumination to check the presence of crack. The specimens were tested until fracture (presence of a minimal radial crack) or until the end of all steps.

2.6 Topographic analysis

Micrographs of the ceramic after surface treatment were analyzed in topographical and lateral views to identify the differences in the micromechanical pattern after being subjected to the surface treatments under study. Representative specimens (n = 2) were ultrasonically cleaned, gold sputtered and analyzed at a Scanning Electron Microscope (SEM) (VEGA3 Tescan; Brno-Kohoutovice, Czech Republic) on 1,000× and 2,000× magnification.

2.7 Fractographic analysis

After the fatigue testing, the specimens were analyzed in a stereomicroscope (Stereo Discovery V20, Carl-Zeiss; Göttingen, Germany) to identify their crack path, which was marked. Representative specimens of each group were cut under water-cooling (Isomet 1000, Buehler) in the middle and perpendicular to the crack, ultrasonically cleaned in distilled water for 10 min, sputtered with a gold-palladium alloy and analyzed in the SEM (VEGA3 Tescan) to identify the crack features on 100× and 500× magnification.

2.8 Statistical analysis

The fatigue failure load (N), number of cycles for failure and the survival rates of each group were recorded and analyzed by the Kaplan-Meier and Mantel-Cox (log rank test at $\alpha=0.05$).

3. Results

The statistical analysis showed a statistically significant difference between the conditions evaluated for the fatigue failure load results as well as for the number of cycles for failure (Table 3 and Fig. 1). Kaplan-Meier analysis showed that surface treatments with HF and EP presented load values and number of cycles for failure statistically similar to each other and higher than SB, regardless of the type of cement (Table 3). When only the cement was considered, the self-adhesive cement obtained statistically greater results for the HF and SB treatments, and no difference among cements was found when applying EP (Table 3). Regarding the survival rate, considering the load of 900 N or 55,000 cycles, all specimens submitted to sandblasting had already failed, while more than 47% of the HF-treated specimens and at least 27% of the specimens treated with EP had survived (Table 4).

The topographic analysis showed different ceramic surface patterns for the different surface treatments performed. HF etching seems to produce a less roughness than EP, and SB created a more irregular surface than the other treatments (Fig. 3 and 4).

The fractographic analysis showed that the failure pattern was the same for all groups. The origin of the radial cracks was located in the ceramic intaglio/cementation surface and propagated towards the surface of load application (ceramic 'occlusal' side) (Fig. 5). In addition, no failures due to contact damage (Hertzian cone cracks) were observed.

Table 3 - Survival Analysis of the Fatigue Failure Load (FFL) and Number of Cycles for Failure (CFF) of the Fatigue Step-stress test (n=15).

Study groups	^{f} Survival analysis	
	FFL (N) - Mean (CI)	CFF - Mean (CI)
nMDP+HF	973.33 (891 - 1055) ^{B}	72,333 (64,113 – 80,553) ^{B}
nMDP+EP	866.67 (759 - 974) ^{B}	61,666 (50,902 – 72,430) ^{B}
nMDP+SB	546.67 (514 - 579) ^{D}	29,666 (26,428 – 32,905) ^{D}
MDP+HF	986.67 (920 - 1052) ^{B}	75,000 (67,843 - 82156) ^{B}
MDP+EP	1066.67 (993 - 1139) ^{AB}	81,666 (74,341 – 88,991) ^{AB}
MDP+SB	546.67 (520 - 572) ^{D}	29,666 (27,053 – 32,280) ^{D}
SA+HF	1206.67 (1086 - 1326) ^{A}	95,666 (83,650 – 107,683) ^{A}
SA+EP	1026.67 (942 - 1111) ^{AB}	77,666 (69,227 – 86,105) ^{AB}
SA+SB	733.33 (702 - 764) ^{C}	48,333 (45,209 – 51,456) ^{C}

Different uppercase letters in each column indicate significant statistical difference based on the ^{f} Kaplan-Meier and Mantel-Cox (log rank test at α=.05).

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Table 4 – Survival rates (in % - probability of the specimens to exceed the respective fatigue failure load and number of cycles for failure without fail) of the experimental groups and their respective standard error measurements.

Groups	Fatigue failure load (N) / Number of cycles ($\times 10^3$) on the respective step*														
	400 / 15×10^3	500 / 25×10^3	600 / 35×10^3	700 / 45×10^3	800 / 55×10^3	900 / 65×10^3	1000 / 75×10^3	1100 / 85×10^3	1200 / 95×10^3	1300 / 105×10^3	1400 / 115×10^3	1500 / 125×10^3	1600 / 135×10^3	1700 / 145×10^3	1800 / 155×10^3
nMDP+ HF	1	1	1	1	0.73 (0.11)	0.47 (0.13)	0.27 (0.11)	0.20 (0.10)	0.07 (0.06)	0.00 (0.00)	-	-	-	-	-
nMDP+ EP	1	1	1	0.67 (0.12)	0.27 (0.11)	0.27 (0.11)	0.20 (0.10)	0.13 (0.09)	0.07 (0.06)	0.00 (0.00)	0.00 (0.00)	-	-	-	-
nMDP+ SB	1	0.40 (0.12)	0.06 (0.06)	0.00 (0.00)	-	-	-	-	-	-	-	-	-	-	-
MDP+ HF	1	1	1	0.93 (0.06)	0.87 (0.08)	0.73 (0.11)	0.20 (0.10)	0.13 (0.09)	0.00 (0.00)	-	-	-	-	-	-
MDP+ EP	1	1	1	1	0.93 (0.06)	0.80 (0.10)	0.53 (0.12)	0.27 (0.11)	0.13 (0.09)	0.00 (0.00)	-	-	-	-	-
MPD+SB	1	0.47 (0.13)	0.00 (0.00)	-	-	-	-	-	-	-	-	-	-	-	-
SA+HF	1	1	1	1	1	1	0.80 (0.10)	0.47 (0.13)	0.20 (0.10)	0.13 (0.09)	0.13 (0.09)	0.13 (0.09)	0.13 (0.09)	0.06 (0.06)	0.00 (0.00)
SA+EP	1	1	1	1	0.87 (0.09)	0.60 (0.12)	0.40 (0.12)	0.27 (0.11)	0.13 (0.09)	0.00 (0.00)	-	-	-	-	-
SA+SB	1	1	0.93 (0.06)	0.40 (0.12)	0.00 (0.00)	-	-	-	-	-	-	-	-	-	-

The sign '-' indicates absence of specimen being tested on the respective step. *These values are approximated.

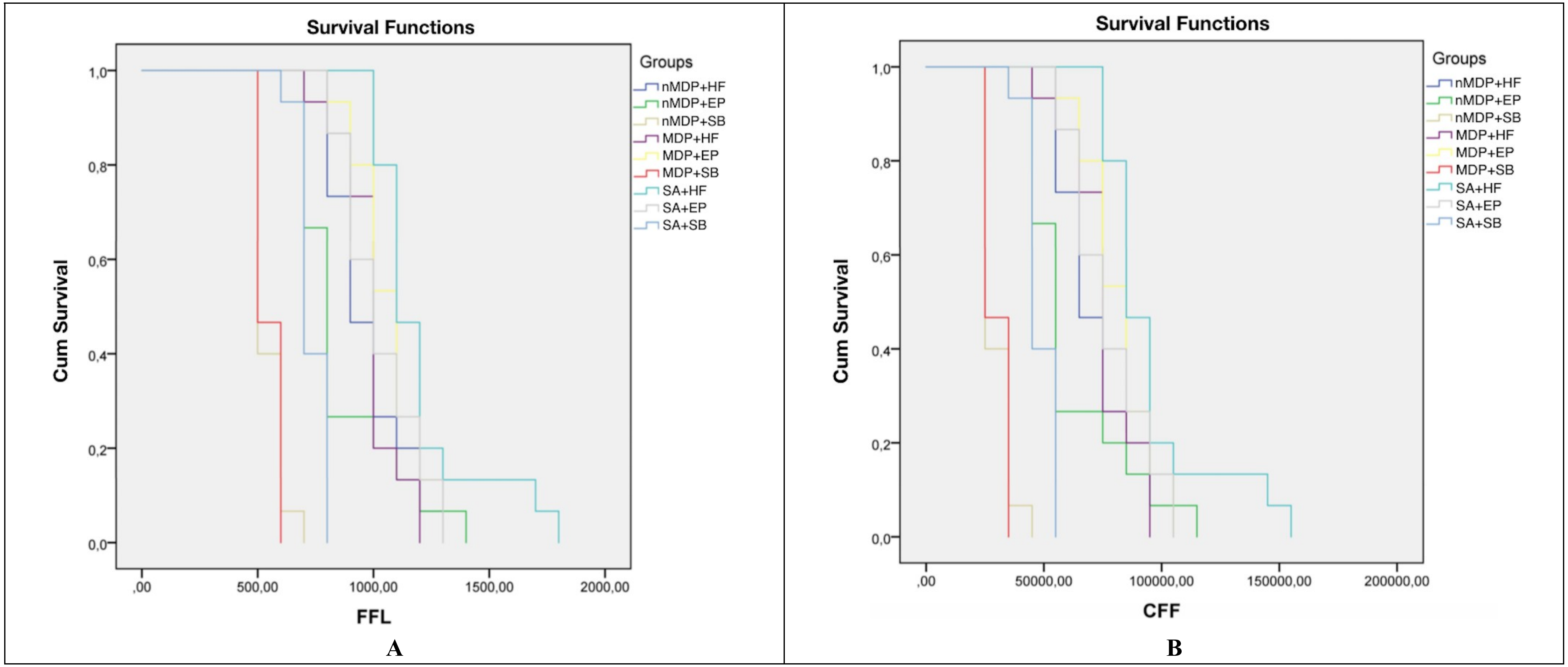

Figure 1 – Survival graphs obtained by Kaplan-Meier and Log-rank (Mantel-Cox) tests for the Fatigue Failure Load (**A**) and number of Cycles for Failure (**B**).

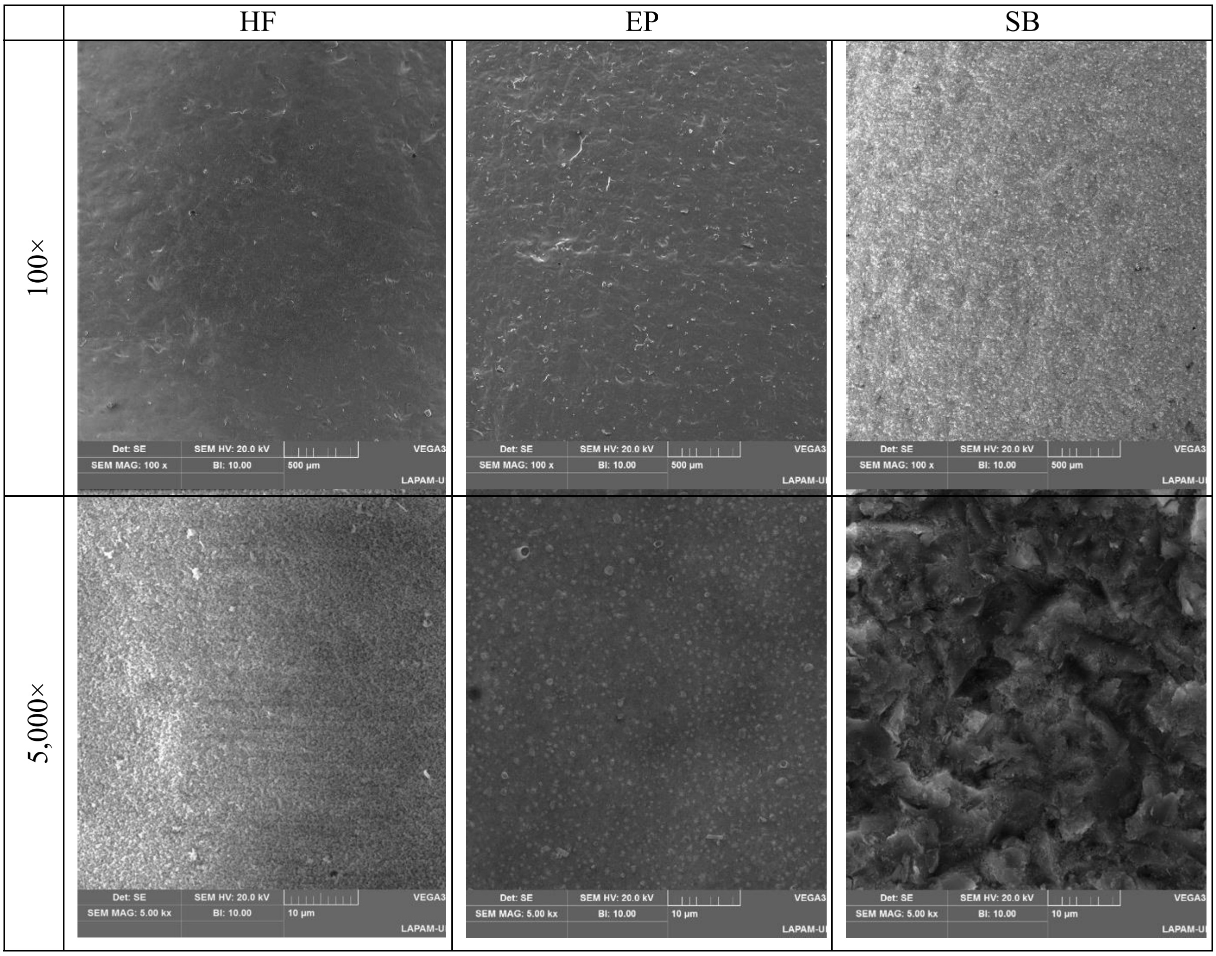

Figure 3 - Topographic images on Scanning Electron Microscopy (100× and 5,000× magnification) of the ceramic surface after the surface treatments: 5% hydrofluoric acid etching (HF), Monobond Etch & Prime application (EP) and sandblasting (SB).

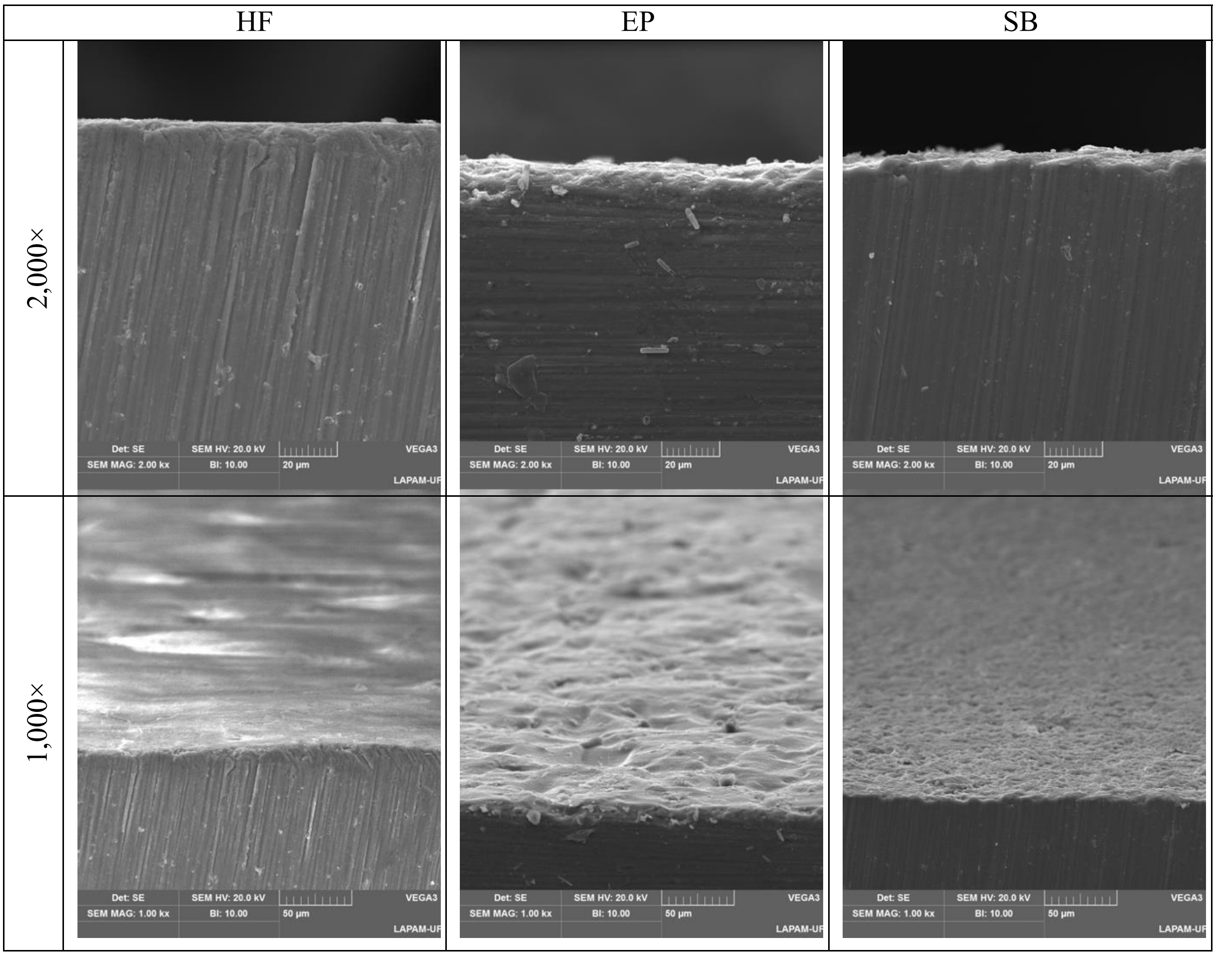

Figure 4 - Representative SEM images (1,000× and 2,000× magnification) on a lateral view of surface treatments performed. HF – 5% HF etching; EP - Application of Etch & Prime; SB - Sandblasting.

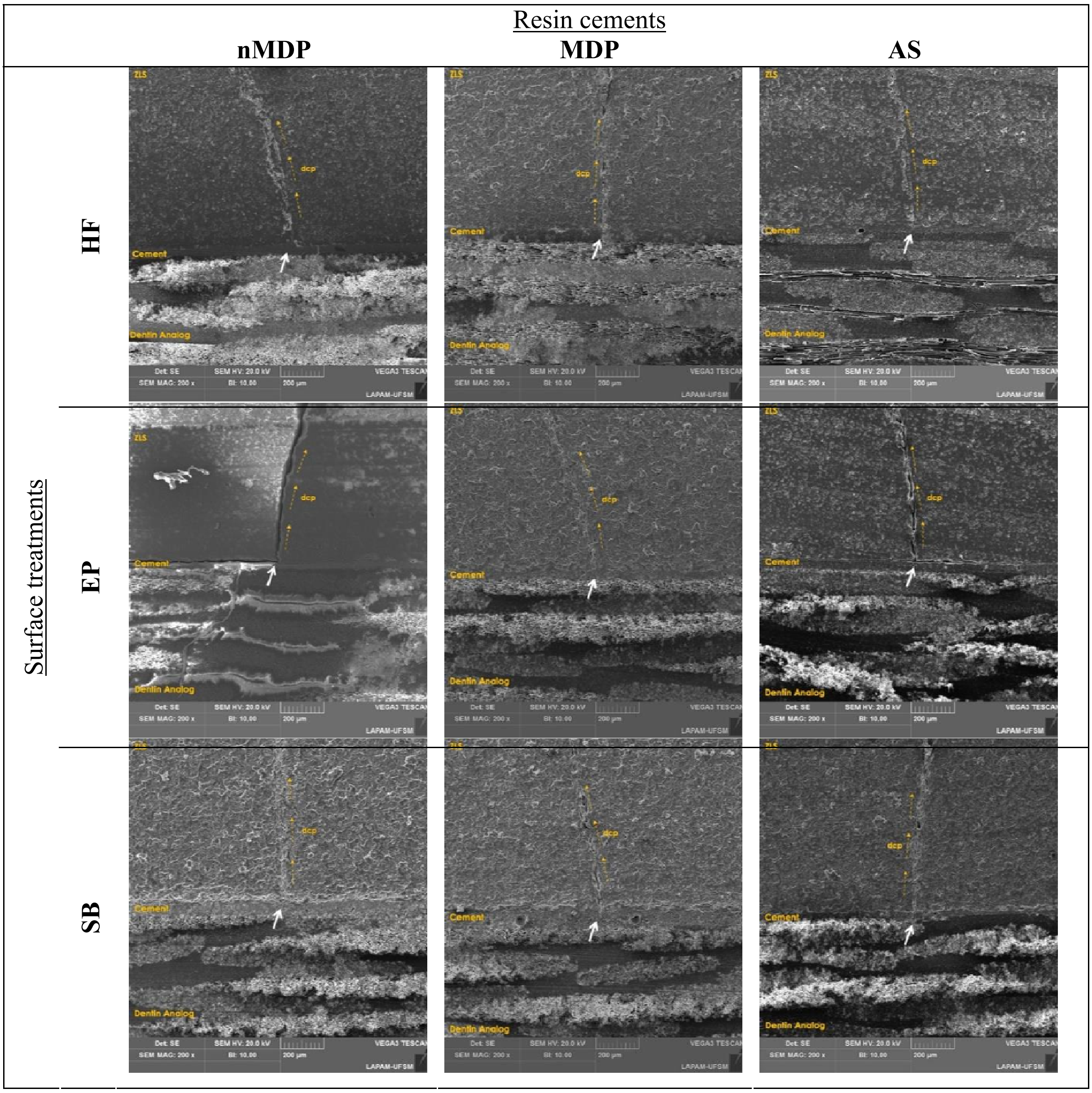

Figure 5 - Fractographic images (200× magnification) on Scanning Electron Microscopy of the study groups showing the fractographic characteristics after failure during the fatigue test. The origin of failure is located at the intaglio ceramic surface (white arrow). ZLS – zirconia-reinforced lithium silicate glass ceramic; dcp – direction of crack propagation.

4. Discussion

The results of the present study show that the surface treatment and the type of cement used influenced the fatigue failure load and the number of cycles for failure of the zirconia-reinforced lithium silicate glass-ceramic (ZLS) (VITA Suprinity). Thus, the null hypotheses were rejected.

As a glass-ceramic, the most recommended surface treatment for ZLS is the conditioning with 5% hydrofluoric acid followed by the silane agent application [Traini et al., 2016]. This treatment promotes selective removal of the glassy matrix, cleans the surface, and creates microretentions, increasing the surface wettability [Sato et al., 2016; Strasser et al., 2018], besides removing and/or smoothening the surface defects resulting from CAD/CAM (Computer-aided design/manufacturing) processing [Thompson & Anusavice, 1994; Sato et al., 2016]. Strasser et al. [2018] stated that the HF etching followed by the silane agent application provided a greater flexural strength when compared to the sandblasting followed by the silane application in a ZLS glass-ceramic, corroborating our results (Table 3).

Differently, during sandblasting with silica-coated alumina particles, the high energy generated during the impact impregnated the silica layer to the ceramic surface [Sato et al., 2016; Traini et al., 2016], modifying it chemically [Michida et al., 2003]. However, the impact of the particles also causes a certain amount of abrasion, creating sharper defects and cracks at the ceramic surface [Strasser et al., 2018], as observed in the present study (Fig. 3 and 4), which could be potential sites for the crack growth during function. According to Griffith's weakest link theory [1921], the greater the number of critical defects, the greater the chance of an earlier crack growth occurring when the material is subjected to intermittent loading, such as those present in the oral environment during chewing. That sentence is corroborated by others [Addison et al., 2008; Yi & Kelly., 2011; Spazzin et al., 2016], who have shown the deleterious effect of a rougher ceramic surface when the defects are not properly filled by the adhesive bonding. In addition, Kern and Thompson [1994] reported that irregularities caused by sandblasting with aluminum oxide are not capable of providing reliable long-term micromechanical retention in zirconia ceramic.

Another alternative for surface conditioning of vitreous ceramics is the application of a self-etching ceramic primer, such as the Monobond Etch & Prime (Ivoclar Vivadent), which consists of a combination of ammonium polyfluoride and silane (trimethoxysilypropyl methacrylate) in a single bottle, simplifying the conditioning technique [Prado et al., 2018]. Studies have shown a poor ability of such ceramic primer to properly modify the surface when compared to 5% hydrofluoric acid etching in a lithium disilicate ceramic [El-Damanhoury & Gaintantzopoulou., 2018; Lopes et al., 2018] and in a ZLS ceramic [Strasser et al., 2018]. However, in our study the EP application seems to keep a greater heterogeneous surface when compared to HF that creates a more homogeneous/smoothened surface (Fig. 3 and 4). In the study of Prado et al. [2018], the use of a self-etching ceramic primer, although providing lower bond strength values than HF etching followed by the silane application, promoted more stable long-term results. According to our results, the surface treatment with EP was effective as much as the HF, regardless the cement applied, and it could be a viable option for the ZLS ceramic surface treatment in terms of mechanical fatigue performance and survival rates.

The use of resin cements to bond the vitreous ceramic restorations has improved their mechanical performance [Della Bona et al., 2004; Fleming et al., 2006; Addison et al., 2008; Spazzin et al., 2016]. This finding has been related to the ability of resin cements to seal and modify the ceramic surface defects, possibly by creating compressive forces at the cracks tip or simply by sealing the critical defects [Fleming et al., 2006]. However, the explanation for this strengthening mechanism has not yet been fully clarified and requires further investigation [Fleming et al., 2006]. The characteristics of the bonding systems may determine their ability to fill these defects, and the type, size and content of the cement filler particles [Meşe et al., 2008] besides the presence of bisphenol-A-diglycidylether dimethacrylate (Bis-GMA) [Di Francescantonio et al., 2013] directly influence such ability. As the increase in the filler content and the presence of such organic matrix (Bis-GMA) result in an increase in their viscosity [Di Francescantonio et al., 2013; Lopes et al., 2015] (filler content Table 1). A low viscous cement is preferable since it generates a better intimacy between the ceramic surface defects and the infiltrating bonding system, resulting in a greater capacity of filling the defects [Hitz et al, 2012; Bulut et al., 2018], and it also provides a thinner cement thickness, resulting in lower shrinkage stress, and reducing the possibility of gaps and resin cement sorption and solubility [Meşe et al., 2008; Oliveira et al., 2010].

The results of our study show a difference between the cements used when the ceramic surface was treated with HF or SB, and the self-adhesive resin cement presented better fatigue performance than the other cements in such groups (Table 3). The self-adhesive resin cement (RelyX U200) has a lower viscosity and consequently better wets the ceramic, which gives it a higher capacity to penetrate and seal the surface and subsurface defects created by the HF and SB [Aboushelib and Wang, 2010], further protecting the accelerated degradation of the bonding interface due to the thermocycling process, which may compromise the resin cements mechanical properties [Muller et al., 2017; Medeiros et al., 2007]. Also, the methacrylate monomers with phosphoric ester functional groups in the self-adhesive resin cement are able to improve adhesion by creating hydrogen bonds with the ceramic surface [Tunc et al., 2016].

Such difference between the cements was not observed when the surface was treated with the ceramic primer (EP) (Table 3). That may be explained since the EP did not create a much irregular surface and all the cements had the same capacity for filling the defects created on such surface. Besides, it is not clear the role that the adhesion between the cement and the dentin analog will have on the mechanical behavior of the restorative set. In our study we adopted the protocol indicated by the manufacturer of each cement system, and perhaps the adhesion achieved among systems will be different, since the mechanism of adhesion that they are based is also different, and not clearly informed by their manufacturer and existing literature.

Although laboratory tests are important in determining properties and characteristics of materials, they do not fully simulate *in vivo* conditions. Simplified restoration, besides of allowing a more standardized approach, it allows for a less complex stress distribution and the factors under study are better evaluated. The present study has some limitations, such as it does not apply the load with sliding contact, to which ceramic prostheses are subjected under normal and tangential loads during chewing and it can generate surface damage accumulation and consequently reduces the ceramic strength [Ren & Zhang, 2014]. In addition, as an accelerated fatigue test method, caution is required when evaluating the results, and additional studies are needed to corroborate them.

5. Conclusion

Based on the findings of this *in vitro* study, it can be concluded that:

- Different surface treatments and resin cements directly influence the survival rate, the fatigue failure load and the number of cycles to failure of a bonded simplified zirconia-reinforced lithium silicate glass-ceramic.

- The hydrofluoric acid etching and self-etching ceramic primer provided better fatigue behavior than sandblasting regardless of the cement considered.

- The self-adhesive resin cement produced statistically greater results for the HF and SB treatments, and no difference among cements was found when applying EP.

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4. DISCUSSÃO

Os resultados encontrados nestes estudos mostram que o tratamento de superfície e o tipo de cimento resinoso utilizados, influenciam diretamente na resistência de união, carga para falha em fadiga, no número de ciclos para falha e na probabilidade de sobrevivência de uma cerâmica vítrea de silicato de lítio reforçada por zircônia (ZLS).

Em ambos estudos o condicionamento da superfície cerâmica com ácido fluorídrico 5% (HF- ácido fluorídrico 5%) associado com o cimento resinoso autoadesivo (SA- RelyX U200) apresentou os melhores resultados. Sendo uma cerâmica de matriz predominantemente vítrea (56-64% de dióxido de silício), o tratamento de superfície mais recomendado para a ZLS é o condicionamento com ácido fluorídrico 5% seguido da aplicação do agente silano [TRAINI et al., 2016], o que promove a remoção seletiva da matriz vítrea, criando irregularidades e aumentando a superfície de contato [SATO et al., 2016], além de remover e/ou suavizar os defeitos superficiais resultantes do processamento de CAD/CAM (computed-aided design/ manufacturing) [THOMPSON; ANUSAVICE, 1994; SATO et al., 2016], corroborando com os nossos achados.

Segundo Al-Thagafi et al. [2016], jateamento com partículas de óxido de alumínio revestidas por sílica associado com silanização pode ser uma alternativa de tratamento de superfície para a cerâmica ZLS, já que apresentou melhores resultados, quando comparado com ácido fluorídrico 5% associado com agente silano. Durante o jateamento com partículas de alumina revestidas de sílica, a alta energia gerada durante o impacto causou a incorporação de partículas sílica na superfície da cerâmica [SATO et al., 2016; TRAINI et al., 2016], modificando-a quimicamente [MICHIDA et al., 2003]. No entanto, o impacto dessas partículas pode criar mais defeitos e inserir microtrincas na superfície da cerâmica [STRASSER et al., 2018], como observado no presente estudo. Obtivemos como resultado para o tratamento de superfície com jateamento a menor média geral de valor, em ambos os estudos. Confirmando que, além de diminuir a resistência para falha em fadiga do conjunto restaurador, as irregularidades causadas pelo jateamento na superfície cerâmica são desprovidas de microrretenções [KERN; THOMPSON, 1994].

Na tentativa de contornar a toxicidade característica do ácido fluorídrico [ÖZCAN; ALLAHBEICKARAGHI; DÜNDAR, 2012], métodos alternativos vem sendo testados, como o primer cerâmico autocondicionante Monobond Etch & Prime, que consiste em uma combinação de polifluoreto de amônio e silano (metacrilato de trimetoxisilpropilo) de passo único, simplificando a técnica de condicionamento [PRADO et al., 2018]. Em nosso estudo os resultados encontrados para os grupos onde o tratamento de superfície foi a aplicação do primer cerâmico, mostraram uma alta taxa de falhas pré teste, menor resistência de união com alguns cimentos, além de uma baixa capacidade de modificação da superfície condicionada, que pode estar relacionada a uma cobertura da superfície cerâmica pelo primer, que causa um revestimento das irregularidades da superfície [STRASSER et al., 2018], resultado que coincide com dados obtidos em outros estudos presentes na literatura [EL-DAMANHOURY; GAINTANTZOPOULOU, 2017; PRADO et al., 2018; STRASSER et al., 2018].

Para a cimentação de materiais frágeis, como as cerâmicas vítreas, os cimentos resinosos são comumente usados porque aumentam a tenacidade à fratura do material restaurador por promoverem ligações químicas e mecânicas [HITZ et al., 2012; DELLA BONA; SHEN; ANUSAVICE, 2004]. As características dos sistemas de união determinam sua capacidade de preenchimento de defeitos, ademais, o tipo, tamanho e conteúdo das partículas de cimento [MEŞE; BURROW; TYAS, 2008], além da presença de dimetacrilato bisfenol-A-diglicidílico (Bis-GMA) [DI FRANCESCANTONIO; CAVALCANTI; DAVANZO, 2013] influenciam diretamente nessa habilidade. Assim, o aumento do teor de carga e a presença dessa matriz orgânica (Bis-GMA) resultam em um aumento da sua viscosidade [DI FRANCESCANTONIO; CAVALCANTI; DAVANZO, 2013; LOPES et al., 2015] (conteúdo de preenchimento Tabela 1, Artigo 1 e 2). Os melhores valores de resistência de união, número de ciclos para falha e carga para falha em fadiga foram encontrados quando utilizou-se o cimento resinoso auto-adesivo (SA- RelyX U200- 3M ESPE). Este cimento tem como característica baixa viscosidade e, consequentemente, maior molhabilidade à cerâmica, o que lhe confere maior capacidade de penetração e selamento dos defeitos superficiais e de subsuperfície criados pelos tratamentos de superfície [ABOUSHELIB; WANG, 2010]. Além disso, possui em sua formulação monômeros metacrilato com grupos funcionais de éster fosfórico, que são capazes de melhorar a força de união , formando ligações de hidrogênio entre a matriz resinosa do cimento e a superfície da cerâmica [TUNC et al., 2017].

Segundo Gale e Darvel [1999] restaurações adesivas estão por prolongados tempos suscetíveis a um ambiente bucal hostil, com mudanças cíclicas de temperatura, fluxo salivar constante e trocas de pH [WAHAB; SHAINI; MORGANO, 2003], que podem gerar degradação da interface adesiva. Por esse motivo, para gerar resultados *in vitro*, de uma forma mais próxima aos encontrados clinicamente, utilizou-se para esse trabalho a ciclagem térmica, que através de trocas bruscas de temperatura, simulam o processo de envelhecimento acelerado da interface adesiva [BLUMER et al., 2015].

Contudo estudos laboratoriais *in vitro* não são capazes de simular genuinamente as condições encontradas clinicamente. No entanto são importantes na geração de dados que determinam as características dos materiais. O presente estudo apresenta algumas limitações, como a não aplicação da carga para fadiga com contato deslizante, a qual as próteses cerâmicas são submetidas durante a mastigação, o que poderia gerar acúmulo de danos na superfície e consequente redução da resistência cerâmica [REN; ZHANG, 2014]. Além das falhas pré-teste relacionadas ao método de teste de microcisalhamento, podendo ter sido consequência de uma fraca adesão. Com isso, mais estudos *in vitro* e clínicos devem ser realizados para avaliar estes assuntos, proporcionando maior número de dados laboratoriais para nortear o uso clínico da cerâmica de silicato de lítio reforçado por zircônia.

5. CONCLUSÃO

Com base nos resultados *in vitro* obtidos em nossos estudos podemos concluir que:

- A resistência de união de uma cerâmica de silicato de lítio reforçada com zircônia é diretamente influenciada pelo tratamento de superfície e pelo cimento resinoso utilizado.
- A aplicação do cimento resinoso autoadesivo após condicionamento com ácido fluorídrico proporcionou melhor resistência adesiva à cerâmica ZLS.
- O primer cerâmico auto-condicionante apresentou maior número de falhas pré-teste.
- Diferentes tratamentos de superfície e cimentos resinosos influenciam diretamente a taxa de sobrevivência, a carga de falha por fadiga e o número de ciclos em fadiga de uma cerâmica de silicato de lítio reforçado com zircônia.
- O ácido fluorídrico e o primer cerâmico autocondicionante proporcionaram melhor comportamento à fadiga do que o jateamento, independente do cimento resinoso considerado.
- O cimento resinoso autoadesivo produziu resultados estatisticamente superiores em fadiga para os tratamentos HF e SB, e não houve diferença entre os cimentos ao se aplicar EP.

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