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UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA

FERNANDO JOSÉ RIGOLIN FERREIRA

Estudo da resistência de união de cimento resinoso à dois tipos de cerâmicas vítreas, caracterização dos padrões de condicionamento e avaliação da resistência à flexão

Evaluation of surface treatment, flexural strength and resin cement bond strength of two types of glass ceramics

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Tese apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Doutor em Clínica Odontológica , na área de Prótese Dental

Thesis presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Doctor in Dental Clinic, in Dental Prosthesis area.

Orientadora: Celia Marisa Rizzatti Barbosa

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A Ata da defesa com as respectivas assinaturas dos membros encontra-se no processo de vida acadêmica do aluno.

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RESUMO

O objetivo neste estudo foi avaliar o efeito de diferentes protocolos de tratamentos sobre a superfície de cerâmicas vítreas a base de dissilicato de lítio e silicato lítio reforçado por dióxido de zircônia na resistência de união, longevidade da resistência de união e resistência à módulo e flexural.

Foram utilizadas placas de cerâmicas (n=3) a base de dissilicatao de lítio (IPS e.max CAD, Schaan, Liechtenstein) (LDS) e a base de silicato de lítio reforçada por dióxido de zircônia (Suprinity, VITA Zahnfabrick, Bad Säckingen, Germany) (ZLS) nas dimensões de 14 mm de comprimento, 12mm de largura e 2mm de espessura. As superfícies cerâmicas foram tratadas com ácido fluorídrico na concentração de 9% (Ultra etch, Ultradent Inc.) em 10, 20 e 30 segundos e jateamento com óxido de alumínio e analisados e caracterizados através de microscopia eletrônica de varredura (MEV) com aumento de 10000x . Para o teste de resistência de união (n=10), após cada tratamento, as superfícies cerâmicas foram silanizadas (Silane, Ultradent Inc.) e aplicado adesivo (Peak Universal Bond, Ultradent Inc.). Posteriormente, utilizou-se matriz de PVS (Elite, Zermach, Modena, Italy) com dimensões internas de 1,5mm de diâmetro e 4mm de altura para confecção dos espécimes em cimento resinoso fotoativado (NX3, Kerr, USA), sendo os testes realizados em uma máquina de ensaio universal (EZ-Test) após 24 horas e um ano de armazenamento em água deionizada à 37°C. Os valores de resistência de união foram representados em Mpa e os padrões de fraturas foram classificados e analisados em MEV. Para a análise de resistência à módulo e resistência à flexão, obteve-se espécimes cerâmicos em forma de barra com dimensão de 15 mm de comprimento, 0,75mm de altura e 1,5mm de largura e realizados os tratamentos de superfície propostos e submetidos à ensaio de teste de 3 pontos, em máquina de ensaio universal (Instron). As análises dos dados foram realizadas através da Análise de Variância (3 fatores) e teste de Tukey (5%) .

Os tratamentos modificaram a topografia das superfícies cerâmicas, o que pode influenciar a resistência à flexão e de união, dependendo do tipo cerâmico. Jateamento com óxido de aluminio não é recomendado como tratamento de superfície, porque reduziu a resistência à flexão para ZLS e proporcionou resistência de união menor para ambas as cerâmicas independente do tempo de armazenamento. Por outro lado, diferentes tempos de condicionamento com HF não influenciaram as propriedades mecânicas da cerâmica. Considerando ambos os tempos de avaliação para resistência de união, o tratamento com HF por 20 segundos obteve o melhor desempenho para ambas cerâmicas. O armazenamento interferiu negativamente na resistência de união para cerâmicas LDS.

PALAVRAS CHAVE: Dissilicato de lítio, Zircônia reforçada com silicato de lítio, cisalhamento, resistência flexural.

ABSTRACT

The aim of this study was to assess the effect of different protocols of treatments over the surface, the Microshear bond strength (MSBS), durability and flexural strength of glass ceramic based on Lithium disilicate and lithium silicate reinforced with zirconia.

Ceramic plates (n=3) were used based on lithium disilicate (IPS e.max CAD, Schaan, Liechtenstein) (LDS) and based on lithium silicate reinforced by zirconia (Suprinity, VITA, Zahnafabrick, Bad Säckingen, Germany) (ZLS) with a 14 mm x 12 mm x 2 mm. The surfaces of the plates were treated with Fluorhydric acid (FH) 9% (Ultra etch, Ultradent Inc.) for 10, 20 and 30 seconds, and sandblasted with Alumnia, the samples were assessed with SEM at 10000X. For the MSBS (n=10), after the surface treatment, the plates were silanized (Silane, Ultradent Inc) and the adhesive system (Peak Universal Bond, Ultradent Inc.) was applied. A PVS matrix (Elite, Zermach, Modena, Italy) with a diameter of 1.5 mm x 4 mm for the build up with resin cement (NX3, Kerr, USA), the MSBS test was realized with a EZ-Test universal machine, after 24 h and 1 year storage in water. The MSBS were obtained in MPa and the failure patterns classified in SEM. The Flexural strength and Elastic modulus were obtained with a sample of 15 mm x 0.75 mm x 1.5 mm and the surface treatment applied. The test was done in a 3 points test in a universal essay machine (Instron). The data was evaluated with three-way ANOVA post-hoc Tukey (5%).

The protocols of the surface treatment modified the topographical surface of the glass ceramic. This can influence the MSBS, depending on the ceramic kind. The sandblasting with alumina is not recommended as a surface treatment, because reduce the flexural strength for the ZLS and the MSBS was reduced in both ceramics. On the other hand, different times of etching with HF did not influence the mechanical properties of the ceramics. Considering both times of etching, being 20 s the best treatment for LDS.

Keywords: Lithium disilicate, zirconia reinforced lithium silicate, shear, flexural strength.

SUMÁRIO

1. INTRODUÇÃO

As cerâmicas odontológicas são materiais que estão em constante modificações pela busca do natural na reposição do substrato dental perdido. Neste quesito observa-se as qualidades ópticas, estabilidade química, biocompatibilidade, propriedades mecânicas e adesivas, que estes materiais possam ter ao substrato dentinário (Nakamura *et al.*, 2004). Dentre os que mais se destacam, são a cerâmicas vítreas reforçadas por partículas cristalinas que abrangem grande parte das propriedades citadas anteriormente. Neste lanço, as cerâmicas a base de dissilicato lítio ganharam enorme popularidade, sendo primeiramente descrito por Brodkin *et al.*, (1998), originalmente compostas por 65% de dissilicato de lítio como composto cristalino conferindo-lhes alta resistência à flexão e excelentes propriedades ópticas (opalescência e translucidez). Foram comercializadas pela empresa Ivoclar Vivadent e denominadas Sistema Empress II, sendo seu processo de fabricação através da técnica da cera perdida (Albakri *et al.*, 2003).

Entre as opções disponíveis no mercado atual, podemos citar o Sistema IPS e.max Press (Ivoclar Vivadent), que é confeccionado através da técnica da cera perdida, e o IPS e.max Cad (Ivoclar Vivadent), que é processado pela técnica de usinagem dos blocos (Denry & Holloway , 2010), estas últimas, uma evolução da cerâmica anterior descrita, também a base de dissilicato de lítio em sua composição .

A principal diferença do IPS e.max Press e da sua versão anterior (IPS Empress II) é a quantidade cristalina aumentada para 70% em um processo de dupla nucleação controlada, em que torna os cristais mais homogêneos, melhorando propriedades mecânicas de 340 para 400 Mpa. Há também uma gama maior de opções de pastilhas com graus variáveis de opacidade e translucidez com o objetivo de ampliar as indicações em áreas estéticas (Gehrt *et al.*, 2013).

O IPS e.max CAD possui duas fases para facilitar sua fresagem, a primeira composta por uma base de precipitado de metassilicato de lítio sob nucleação dupla controlada e prensado na forma de bloco, tendo aspecto azulado. Nesta fase, possui resistência flexural aproximada em torno de 130 a 150 Mpa, suficiente para suportar o estresse da fresagem, evitando possíveis lascamentos da margem da infraestrutura. Na segunda fase, a infraestrutura é submetida à um processo de cristalização à 850°C, transformando-se em dissilicato de lítio. Apresenta nesta fase resistência flexural aumentada em torno de 360 à 420 MPa e cor semelhante ao dente natural. Possuem em sua composição 70% em volume cristais de dissilicato de lítio em forma agulhada com granulação fina com comprimento de 1,5µm embebidos em uma matriz vítrea, em que a principal diferença com o IPS e.max Press é possuir estes cristais em torno de até 7µm. Vale salientar que, os blocos CAD /CAM poderem possuir estruturas mais homogêneas advindas do seu processo de pré-fabricação, além de seus cristais de dissilicato de lítio são mais aderidos à matriz cerâmica (Höland *et al*., 2008).

Recentemente foi introduzido no mercado uma nova categoria em cerâmicas vítreas, as cerâmicas a base de lítio reforçadas com dióxido de zircônia, como exemplo comercial: Suprinity (VITA) e o Celtra DUO (Dentsply). Até o momento são fabricadas em blocos para a técnica do CAD/CAM; a empresa (VITA) alega melhor resistência e estética, com indicações similares ao dissilicato de lítio. Sua microestrutura é refinada e homogênea, o que lhe confere excelentes características ópticas e de resistência mecânica, aumentando a gama de aplicações clínicas (da Cunha *et al*., 2015). A inclusão de 10% de dióxido de zircônia, dissolvida na matriz vítrea, tem como mecanismo de agir como agente de nucleação, ou seja, o processo de cristalização inicia-se em sua superfície (Krueger, 2013), resultando em formação de cristais de silicato quatro vezes menores, o que implica elevado teor de vidro, configurando ganho em propriedades ópticas de translucidez quando comparados ao dissilicato de lítio (Awad *et al*., 2015). Isto leva a formação de metadissilicato de lítio e dissilicato de lítio em uma matriz contendo partículas de dióxido de zircônia, esta é a principal diferença em relação ao IPS e.max CAD e Press, que apenas produz dissilicato de lítio (Denry&Kelly, 2014). A referida cerâmica possui duas fases: a primeira, o vidro cerâmico na primeira etapa pode ser pré-cristalizado contendo apenas cristais de metasilicato de lítio, o que facilita seu desgaste para confecção no sistema CAD/CAM; a segunda, refere-se à fase de cristalização, o que leva à dupla microestrutura de dissilicato de lítio, este é obtido após curto tratamento térmico a 840°C durante 8 minutos. (Denry&Kelly, 2014) Vale ressaltar, que é encontrado também comercialmente blocos já cristalizados, como ocorre na cerâmica Suprinity (VITA).

A adesão da cerâmica à estrutura dental pode ser realizada através de ligação mecânico-quimica entre o material restaurador, o substrato dental e o agente cimentante

(Pattanaik & Wadkar, 2011). Para isto é necessário que se condicione a superfície interna da restauração cerâmica. Dentre as modalidades utilizadas são: ácido fluorídrico, fluoreto de fosfato acidulado ou jateamento com óxido de alumínio. Após o condicionamento desta superfície é frequentemente utilizado agente de união silano para estabelecer uma união química entre o cimento resinoso e a cerâmica através de ligações siloxano covalentes (Adisson *et al*., 2007; Guess *et al*., 2011).

Para Borges *et al*., (2003), os métodos de tratamento de superfície para cerâmicas a base de disssilicato de lítio podem ser realizados com jateamento com partículas de óxido de alumínio 50µm ou HF a 10% por 20 segundos, pois ambos promovem irregularidades na superfície cerâmica, sendo fator necessário para o imbricamento do cimento resinoso. A utilização do HF como meio de tratamento de superfície com a finalidade de melhorar esta ligação está bem relatada na literatura (Della Bona *et al*., 2002; Della Bona *et al*., 2006), porém há relatos de seus efeitos deletérios tanto para o clínico que irá manipulá-lo (Bertolini, 1992), como para influenciar a resistência à flexão de certas cerâmicas (Xiaoping *et al*., 2015).

O tratamento ácido na superfície das cerâmicas vítreas é uma alternativa em que se promove alteração na topografia da superfície, gerando um aumento desta e uma maior área de união (Della Bona *et al*., 2002). Esta confere a obtenção de união desejável entre o cimento resinoso e a superfície cerâmica (Ozcan & Vallittu, 2003). O referido processo é dado pela reação do ácido fluorídrico com a sílica presente na cerâmica, havendo a dissolução da mesma, criando microporosidades (Borges *et al*., 2003). Os espaços micrométricos são preenchidos posteriormente por resina fluida que faz a ligação ao cimento resinoso, gerando imbricamento mecânico após sua polimerização. O que se discute na literatura em relação ao tratamento de superfície são fatores como a composição, a concentração e o tempo de exposição do ácido a ser utilizado.

Além disso, a relação entre a composição química da cerâmica e os procedimentos adesivos influencia no resultado final de união para se obter padrão ideal de resistência de união e sua longevidade (Calamia & Simonsen, 1983). Muitos testes têm sido empregados para avaliar a resistência de união em ensaios de adesão, tais como: cisalhamento, tração, microtração e microcisalhamento (Foong *et al*., 2006). A geometria da interface adesiva é fator importante para a mensuração dos valores de resistência de união (van Noort *et al*., 1991), sendo que o estresse gerado no teste é sensível a esta geometria em relação a forma de carregamento, módulo de elasticidade e dimensões do material aderente (van Noort *et al*., 1989). Com isto, a resistência do material a ser utilizado pode se tornar um viés dependendo do teste a ser realizado. Aliado a isto, vale ressaltar, a importância da avaliação do potencial de resistência de união após um período de armazenamento, para que se observe possíveis falhas em relação ao comportamento ao longo prazo desta união, simulando situações de envelhecimento (de Sá Barbosa et al., 2013). O envelhecimento artificial resulta frequentemente em forças de união significativamente menores, especialmente quando a força da ligação das amostras de armazenamento a curto prazo era ligeiramente menor em relação ao controle positivo (Klosa *et al*., 2009) .

A saturação por água do cimento resinoso também conduz a efeitos hidrolíticos deletérios na interface de ligação, atuando como agente plastificante no interior da matriz polimérica (Marocho *et al*., 2013), promovendo a formação de ligações hidroliticamente instáveis. Por conseguinte, os testes pré-clínicos de envelhecimento artificial da aplicabilidade dos novos sistemas de ligação parece obrigatória, uma vez que os ensaios a curto prazo podem conduzir a conclusões enganosas (Klosa *et al*., 2013). As variações na composição química, na capacidade de umedecimento, na viscosidade e nas propriedades mecânicas de cada cimento de resina também podem ser fatores responsáveis pelas variações na capacidade de ligação ao substrato cerâmico e pelas diferentes taxas de falha pós degradação (Marocho *et al.,* 2013).

Há dúvida sobre o uso de condicionamento ácido HF e seus possíveis efeitos deletérios sobre a resistência da cerâmica vítrea após o tratamento, proporcionando possíveis erosões, podendo causar microrupturas durante o processo de cimentação (Xiaoping *et al*., 2014). Menees *et al*.,2014, observaram no estudo decréscimo de valores de resistência à flexão para cerâmicas a base de dissilicato de lítio, quando se utiliza jateamento com óxido de alumínio com pressão superior a 100 KPa.

Esta nova classe de cerâmica vítrea, a base de silicato de lítio reforçado por dióxido de zircônia possui um questionamento em relação ao tratamento adequado em relação ao condicionamento de superfície e sua influência em relação a resistência de união à longo tempo e a resistência à flexão pós tratamento. Desta forma, o objetivo neste estudo foi avaliar o efeito do tempo de tratamento com HF após união com cimento resinoso fotoativado em cerâmicas a base de dissilicato de lítio e a base de silicato de lítio reforçado por dióxido de zircônia em 24 horas e após um ano de armazenamento, a caracterização das superfícies cerâmicas pós tratamentos e sua resistência à flexão .

2. ARTIGO

Effects of sandblasting and hydrofluoric acid etching on surface topography, flexural strength, modulus and bond strength of resin cement to ceramics

ABSTRACT

Statement of problem: Bonding procedures to glass ceramics include sandblasting (SBL), hydrofluoric acid etching (HF) and silanization. However, acid etching time vary among different types of ceramics because their compositions, and these procedures may alter some mechanical properties of such prosthetic materials.

Purpose: The aim of this study was to evaluate the influence of different surface treatment protocols on surface topography, shear bond strength, flexural strength and elastic modulus of two dental glass ceramics.

Material and Methods: Two glass ceramics were evaluated: lithium disilicate (LDS) (IPS e.max CAD, Ivoclar Vivadent) and zirconia-reinforced lithium silicate (ZLS) (Suprinity, Vita). Ceramic samples were obtained by sectioning CAD/CAM blocks with a low-speed diamond impregnated saw under refrigeration. Ceramic surfaces were polished by using a sequence of silicon sandpapers of different grits (180, 320, 600 and 1200) prior to sintering and treated as follows: SBL (Al_2O_3 particles of 50 µm), 9% HF for 10s (HF10), 20s (HF20) or 30s (HF30). The treated surfaces of ceramics were analyzed by scanning electron microscopy (SEM) (n=3). For shear bond strength test, ceramic samples were silanized (after each surface treatment protocol), and an adhesive system was applied to the surface (Peak Universal Bond). Afterwards, a silicone mold was used to build resin cement cylinders (4mm high, 1.5mm diameter), which were tested in tension in a universal testing machine after 24 hours or one year of water storage at 37ºC (n=10). Flexural strength and elastic modulus were assessed by 3-point-bending test. Ceramics samples of 15x1.5x0.75 mm (n=15) were tested in a universal testing machine. Data was subjected to statistical analysis at a pre-set alpha of 0.05.

Results: The surface treatments resulted in different surface topographies, which were more evident when comparing SBL to HF for both ceramics. The bond strength of resin cement to LDS significantly reduced after one year for all surface treatments, while for ZLS, HF10 and HF20 resulted in no statistical difference after 1 year of storage. SBL yielded the lowest bond strength for both ceramics and significantly reduced the flexural strength of ZLS. Flexural strength and elastic modulus of ceramics were not affect by different etching times.

Conclusion: Surface treatment protocols modified the surface topography of glass ceramics. SBL can reduce the flexural strength and was not adequate as ceramic treatment for bonding. The bond strength of resin cement was not stable after one year for LDS and for two groups of ZLS, in which HF20 was the best treatment considering both evaluation times.

Clinical Implication: SBL is not the surface treatment of choice and a specific acid etching time might be required for bonding procedures of ceramics tested.

INTRODUCTION

Dental ceramics are in constant research and development aiming for a restorative material that would meet functionality and esthetics of natural dentition. In order to restore a substrate with a minimally invasive approach, it is primordial that restorative dental ceramics possess appropriate optical properties, biocompatibility, chemical stability, high fracture strength and bonding potential. [1,2] Among commercially available glass ceramics, lithium disilicate (LDS) based ceramics has proven to be a viable option that meets previously described requirements. [3]

Cementation techniques of traditional glass ceramics involve hydrofluoric acid etching (HF) for a certain period of time that, depending on the composition of each ceramic, varies in a range of 20 to 60 seconds. HF dissolves the glass matrix phase of these ceramics increasing its surface energy and generating microporosities, resulting in a retentive and very reactive surface. Besides acid etching, the topography of glass ceramics can also be modified by air-abrasion with aluminum oxide particle (or sandblasting). [4] After each surface treatment of glass ceramics, silanization is required for bonding resin cement to ceramics. Silane is a bi-functional molecule, with a silanol group that chemically reacts with hydroxyl groups present on treated ceramic surfaces, while the methacrylate group co-polymerizes with resin monomers. [5, 6] This reaction results in a desirable chemical bond between resin cement and ceramic surface, which provides the marginal sealing, increased fracture strength and clinical longevity of the indirect restoration. [7, 8]

Besides LDS, zirconia-reinforced lithium silicate (ZLS) ceramic has emerged as an option for indirect restorative procedures. This glass ceramic is based on an addition of 10% zirconium dioxide, which act as a nucleating agent. The process of crystallization starts on its surface, resulting in the formation of silicate crystals four times smaller than LDS, which leads to a higher glass content and higher translucency. [9] For ZLS, few information about etching time is available, as well as the optimal etching pattern for bonding procedures, bonding stability over time, clinical performance and mechanical properties. [13-15]

The aim of this study was to evaluate the influence of different surface treatment protocols on surface topography, shear bond strength, flexural strength and elastic modulus of two dental glass ceramics. The null hypotheses tested were that sandblasting and different etching times (1) would not influence the surface morphology, flexural strength and modulus of glass ceramics and (2) would not affect the bond strength of resin cement to glass ceramics after water storage for one year.

MATERIALS AND METHODS

Specimen preparation and experimental groups

For this study, two glass ceramics were tested: lithium disilicate ceramic (LDS) (shade HT A2/C14, IPS e.max CAD, Ivoclar Vivadent, Schaan, Leichtenstein) and zirconia-reinforced lithium silicate ceramic (ZLS) (shade A2, Supriniy, Vita Zahnfabrik, Bad Säckingen, Germany). Specimens (15 mm length x 12 mm width x 2 mm thickness) of both dental glass ceramics were obtained from computer-aided design/computer-aided manufacturing (CAD/CAM) blocks, which were sectioned by using with a low concentration diamond saw, attached to a low-speed cutting machine (Isomet 2000, Buehler Ltd, Lake Bluff, IL, USA) under copious water irrigation. Then, ceramic samples were manually finished and polished using a sequence of silicon carbide abrasive papers (#400, #500, #600, #1200, #2000), followed by ultrasonic bath in distilled water for 15 minutes, in order to eliminate any remaining residue. After cleaning, specimens were crystallized in a dental furnace as recommended by each respective manufacturer.

Samples were randomly assigned to each of the following surface treatments:

sandblasting with 50 µm aluminum oxide particles (Danville Materials, San Ramon, CA, USA) for 10 s (air pressure: 0.552 MPa; distance from the tip: 1.5 cm); 9% HF (Porcelain Etch, Ultradent Products Inc., South Jordan, UT, USA) 10s (HF10), 20s (HF20) or 30s (HF30). After sandblasting or etching, ceramic plates were water rinsed for 30 s, subjected to ultrasonic bath in distilled water for 5 minutes and air-dried.

Surface topography analysis

To analyze the ceramic etching patterns and sandblasting effects created by surface treatments, twenty-four ceramic samples were used (n = 3). After surface treatment protocols, as previously described, samples were mounted on metallic stubs, sputter coated with gold (MED 010, Balzers, Balzers, Liechtenstein) and observed in a scanning electron microscope (VP 435, Leo, Oberkochen, Germany) at 10.000X magnification (15 kV, 15 mm working distance).

Shear bond strength test

Eighty ceramics samples (n = 10) were embedded in epoxy resin cylinders, leaving a ceramic surface exposed. After surface treatments (HF or sandblasting), ceramic samples were silanized (Silane, Ultradent Products Inc., South Jordan, UT, USA) followed by adhesive application (Peak Universal Bond, Ultradent Products Inc., South Jordan, UT, USA). Bonded surface was gently air-dried and light-activated for 10 seconds using a polywave LED light curing unit (irradiance 1,000 mW/cm², Valo, Ultradent Products Inc., South Jordan, UT, USA). Polyvinyl siloxane molds (Virtual Heavy Body, Ivoclar Vivadent, Schaan, Liechtenstein) of 4 mm high and 1.5 mm diameter were positioned on the ceramic surface and filled with resin cement (NX3, Kerr Corporation, Orange, CA, USA). Resin cement was light activated for 20 seconds in regular mode 1,200mW/cm^2 (Valo, Ultradent Products Inc., South Jordan, UT, USA) and molds were gently removed, obtaining four resin cement cylinders on each ceramic sample surface. Ceramic samples were stored in distilled water at 37°C for 24 hs and two cylinders were tested, while the remaining two cylinders were tested after one year of storage at 37°C in distilled water, which was changed monthly.

For shear bond strength testing, a test base clamp held (Ultradent Inc., South Jordan, UT, USA) the epoxy resin cylinder that contained the ceramic plates embedded and test base clamp was attached to an universal testing machine (EZ-Test, Shimadzu, Kyoto, Japan). Shear load was applied to the bonded interface with a chisel edge plunger at a crosshead speed of 0.5 mm/min until failure. Maximum load values were divided by bonded area of each resin cement cylinder in order to calculate shear bond strength (in MPa). Bond strength was calculated as an average of two resin cement cylinders per sample in the same storage time. Data were analyzed using a three-way analysis of variance in order to determine the effect of “type of ceramic”, “surface treatment”, and “storage time” on bond strength. Tukey’s test was used to detect differences among experimental groups. All statistical analysis was performed at a preset alpha of 0.05.

After shear bond testing, ceramic samples were mounted on stubs, sputter coated with gold (MED 010, Balzers, Balzers, Liechtenstein) and observed using a scanning electron microscope (VP 435, Leo, Oberkochen, Germany) at 25X to 50X magnification. Failure patterns were classified as: 1- adhesive failure; 2- mixed failure and 3- cohesive failure within resin cement.

Flexural strength and elastic modulus

Three-point bending test was performed using ceramic samples (n = 15) of 15.0 mm length x 1.5 mm width x 0.75 mm thickness (ISO 178) and span length was 12 mm. Test was performed using an universal testing machine (model 4411, Instron, Norwood, MA, USA), at a crosshead speed of 0.5 mm/min. Collected data comprised of maximum load until failure and flexural strength (FS) was calculated using the following equation:

$$FS = 3PL/2BD^2$$

where, P is the fracture load, L is the roller span, B is the width and D is the thickness of the bar.

Elastic modulus (E) was calculated from three-point bending test using following equation:

$$E = PL^3 / 4BD^3{}_d$$

where, D is the deflexion corresponding to load P.

Flexural strength and elastic modulus data were analyzed using two–way analysis of variance and Tukey's test (preset alpha of 0.05).

RESULTS

Sandblasted surfaces for LDS and ZLS demonstrated similar topographies (Figure A1 and B1). Different times of HF did not yield significant difference in terms of surface topography for ZTL (Figures A2 to A4). However, increasing HF time for LDS resulted in more dissolution of glassy phase (Figures B2 to B4). Ten seconds of HF did not completely remove the superficial layer of LDS (Figure B2), while 30 s completely exposed the crystals of lithium disilicate (Figure B4).

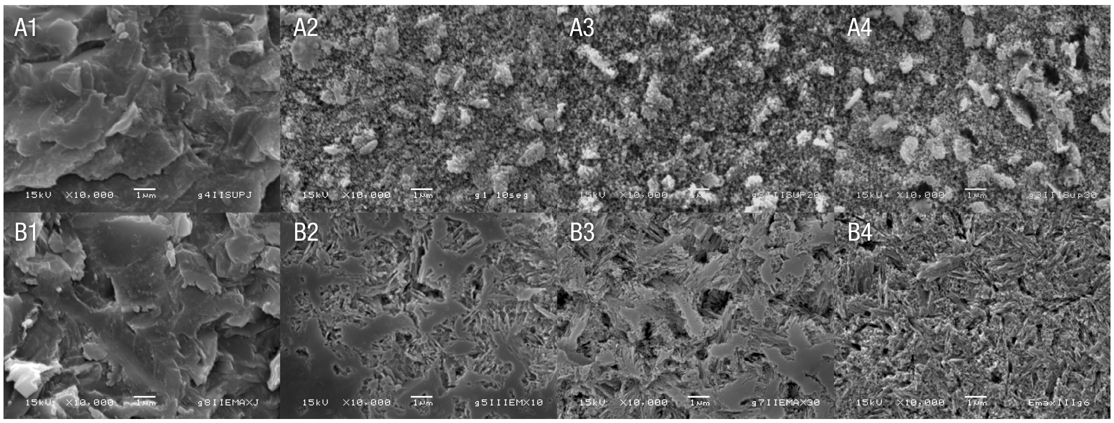

Figure 1. Representative SEM images obtained for ZLS (A1 to A4) and LDS (B1 to B4) glass ceramics subjected to different treatments (X10,000). A1 and B1- after sandblasting; A2 and B2- after etching with HF for 10 seconds; A3 and B3- after etching with HF for 20 seconds and A4 and B4- after etching with HF for 30 seconds.

Mean (SD) shear bond strength values are showed in Table 1. Three-way analysis of variance revealed statistically significant differences for the factors "type of ceramic" ($p = 0.0002$), "surface treatment" ($p < 0.0001$) and "storage time" ($p < 0.0001$). All double interactions were also statistically significant, but triple interaction was not ($p = 0.0443$). The lowest bond strength was observed with sandblasting surface treatment for both materials. Etching time did not influence bond strength for LDS, for both storage times. For ZLS, 20 s of HF yielded higher bond strength when compared to 10 s at 24 h, while 30 s etching was higher than 30 s after one year. One year of storage time reduced the shear bond strength values for LDS. For ZLS, one-year storage decreased the bond strength when specimens were sandblasted or etched for 30 s.

When comparing the bond strength of resin cement, no difference between ceramics was observed at 24 hours, regardless treatment (sandblasting and HF). However, the bond strength of resin cement to ZLS with HF10 and HF20 was higher than those same groups for LDS.

Table 1.Shear bond strength of a resin cement to glass ceramics according to surface treatments and storage time.

Glass Ceramic	Treatment	Storage Time	
		24 hs	1 year
LDS	Sandblasting	7.0 (3.4) A b	1.3 (0.9) B b
	HF 10 sec	20.4 (6.4) A a	*11.2 (5.1) B a
	HF 20 sec	30.6 (12.8) A a	*13.8 (5.4) B a
	HF 30 sec	29.9 (6.4) A a	15.8 (2.8) B a
ZLS	Sandblasting	6.6 (2.2) A c	2.7 (1.4) B c
	HF 10 sec	23.5 (11.1) A b	25.7 (5.1) A ab
	HF 20 sec	35.6 (12.2) A a	31.9 (8.8) A a
	HF 30 sec	28.1 (9.0) A ab	19.7 (6.1) B b

Upper case letter compare storage time within the same treatment (row) and lower case letters compare treatments within the storage time (column).

* Differ from ZLS within the same treatment and storage time

Abbreviations: HF: hydrofluoric acid, LDS: lithium disilicate, ZTL: zirconia reinforced lithium disilicate

Adhesive and mixed failures were predominant for both glass ceramics, while few cohesive failures within resin cement were observed (Figures 2 and 3). For LDS, a higher percentage of adhesive failure was observed than mixed failures, while the opposite was observed for ZLS. One year of water storage affected the incidence of the type of failure for both ceramics.

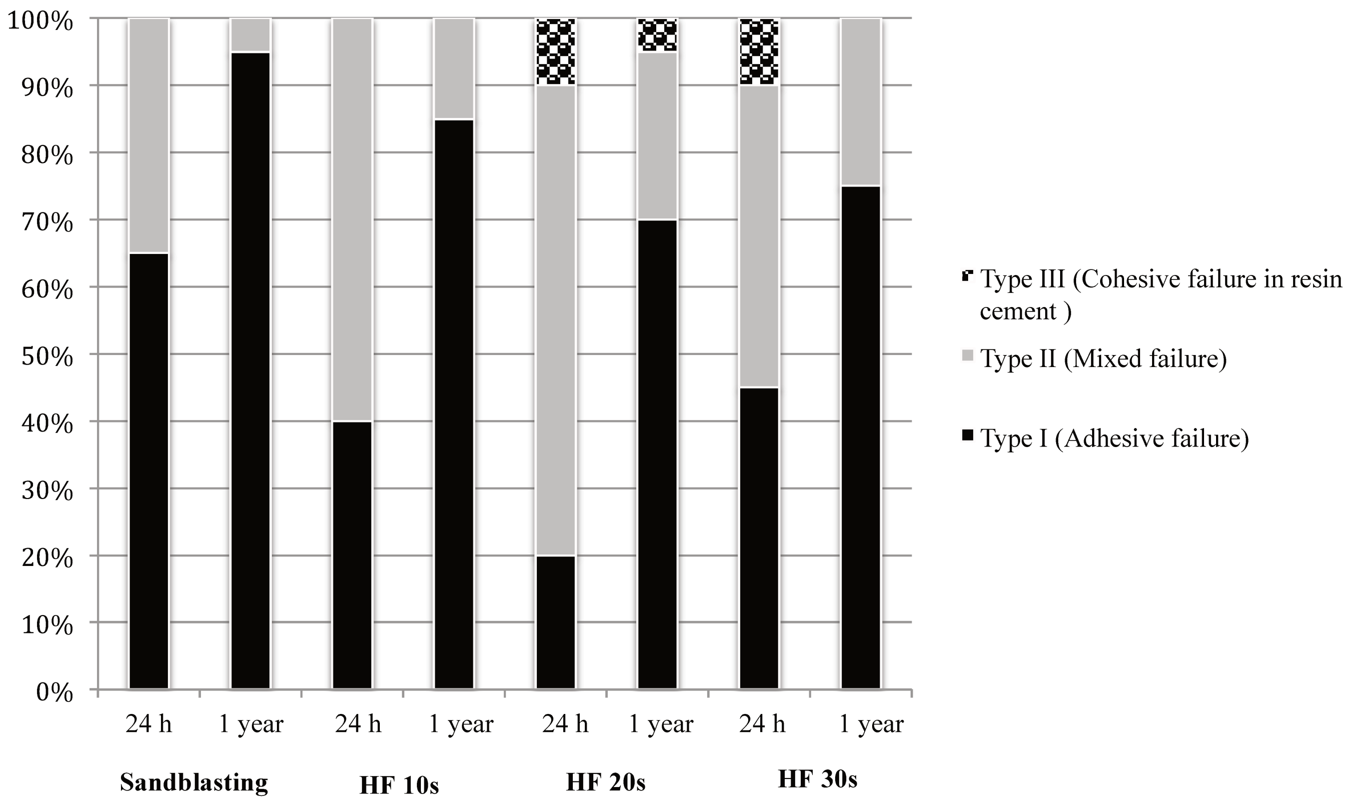

Figure 2. Failure modes of LDS.

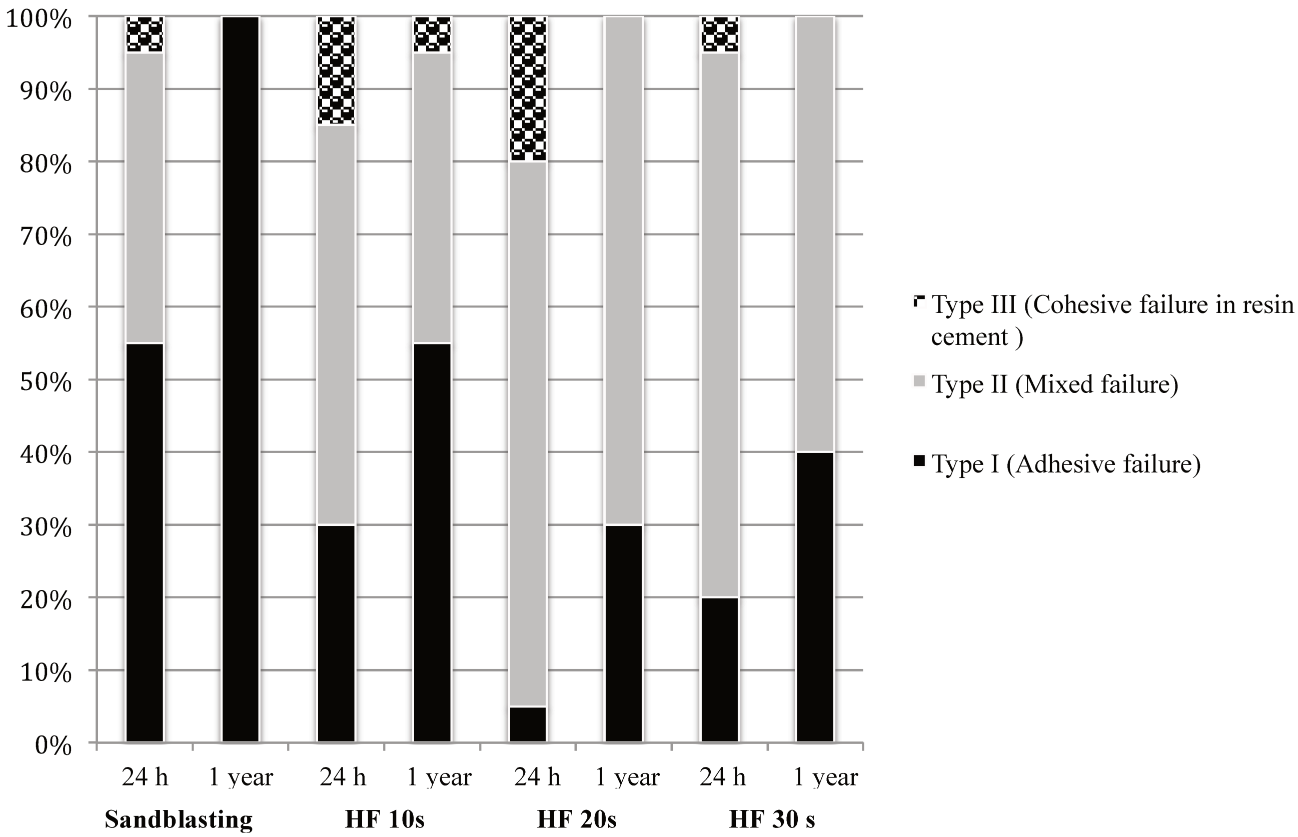

Figure 3. Failure modes of ZLS.

Mean (SD) flexural strength values are demonstrated in Table 2. Two-way analysis of variance revealed statistically significant differences for the factors “type of ceramic” ($p < 0.0001$), “surface treatment” ($p=0.0102$) and for the factor interaction ($p = 0.0011$). LDS surface treatments did not influence the flexural strength values. For ZLS, sandblasting treatment decreased flexural strength when compared to HF, regardless etching time. No statistical difference was obtained between glass ceramics when HF was used.

Table 2.Flexural strength means (SD) of the glass ceramics for different treatments (in MPa).

Treatment	Glass Ceramic	
	LDS	ZLS
Sandblasting	296.8 (35.9) A a	230.6 (38.2) B b
HF 10 sec	289.4 (34.9) A a	288.0 (25.8) A a
HF 20 sec	291.0 (36.2) A a	289.8 (17.3) A a
HF 30 sec	300.5 (33.5) A a	288.8 (27.8) A a

Upper case letter compare glass ceramic within the same treatment (row) and lower case letters compare treatments for the same glass ceramic (column).

*Abbreviations: HF: hydrofluoric acid, LDS: lithium disilicate, ZLS: zirconia reinforced lithium disilicate

Mean (SD) elastic modulus values are shown in Table 3. Two-way analysis of variance revealed no statistically significant differences for the factors “type of ceramic” ($p = 0.2012$), “surface treatment” ($p = 0.1002$) and for the factor interaction ($p = 0.1100$). Surface treatments of LDS and ZLS did not influence elastic modulus values. No statistical difference for elastic modulus was observed between glass ceramics, regardless the treatment.

Table 3.Flexural modulus means (SD) of the glass ceramics for different treatments (in GPa).

Treatment	Glass Ceramic	
	LDS	ZLS
Sandblasting	39.3 (10.7) A a	43.1 (4.6) A a
HF 10 sec	36.8 (7.3) A a	39.6 (7.0) A a
HF 20 sec	39.0 (6.3) A a	41.5 (4.3) A a
HF 30 sec	40.1 (9.3) A a	41.3 (6.7) A a

Upper case letter compare glass ceramic within the same treatment (row) and lower case letters compare treatments for the same glass ceramic (column).

*Abbreviations: HF: hydrofluoric acid, LDS: lithium disilicate, ZLS: zirconia reinforced lithium disilicate

DISCUSSION

Both null hypotheses were rejected because the sandblasting and different etching times resulted in different surface morphologies for both glass ceramics, the sandblasting reduced the flexural strength of ZLS and the decreased of bond strength of resin cement was observed for LDS and two treatments (sandblasting and HF30) for ZLS. When the surface of both ceramics was sandblasted with Al_2O_3, the lowest bond strength was obtained. Also, the bond strength of the samples treated with sandblasting significantly reduced after one-year of water storage. Sandblasting modified the ceramic surfaces, creating a very irregular surface (Figures 1A1 and 1B1), which was quite different than those etched by HF. The surface alteration promoted by Al_2O_3 abrasive particles resulted in high incidence of adhesive failures, mainly after one year, suggesting low interaction between the resin cement and sandblasted ceramic surfaces. Thus, considering the reduction in flexural strength for ZLS and comparing sandblasting with HF, it is not recommended for treating tested ceramics for bonding.

Etching with HF selectively dissolved the glass matrix phase and exposed the crystalline portion of ceramics as showed in the Figure 1 (A2, A3, A4, B2, B3 and B4). This ceramic surface alterations creates microporosities, where the uncured resin monomers penetrate, providing durable micromechanical interlocking and strong bond strength of resin cement to ceramics. [(6)] Depending on the composition of glass ceramics, different etching patterns can be obtained. The zirconia particles present in ZLS yielded a more regular etching pattern, because they were not removed following HF. [(20)] Different etching times seemed not to affect the etching pattern of ZLS, which showed an irregular surface with the glassy matrix dissolution and the exposition crystalline phase, represented by zirconium oxide particles (Figures A2 to A4). Conversely, different etching times resulted in different etching patterns for LDS ceramic. [(5,11,12)] HF10 resulted in a mild dissolution of glassy phase (Figure B2), while HF30 altered the entire ceramic surface, exposing lithium disilicate crystals (Figure B4). Other study also demonstrated that etching for a short time resulted in small pores, while etching for long time resulted in wider and irregular grooves, increased of surface roughness and wettability. [(14)]

Although different etching patterns were observed for LDS following different HF etching times, no influence on bond strength was observed at 24 hours or at 1 year. However, bond strength of resin cement to LDS significantly decreased after 1 year for all treatments (sandblasting and HF), which was not observed for ZLS, when the etching time was 10 and 20 seconds. Thus, it was possible speculate that bond strength of resin cement to LDS was not stable after one year and specific etching times (HF10 and HF20) were important for a durable adhesion of resin cement to ZLS. Additionally, these etching times yielded higher bond strength comparing to LDS after one year, which represented the only differences between the ceramics.

Sato et al. also showed that the etching time for 20 seconds with HF seem be the optimal time to treat ZLS for bonding of resin cement. (13) However, this study also evaluated the bond strength after for one year, which adds information and consolidates the indication of this etching time for ZLS ceramic.

Previous studies have reported that HF improved bond strength between the LDS and resin-based materials, promoting long-lasting indirect restorations. (16-18) However, this study used LDS CAD/CAM blocks, which has different composition, than those prepared by pressing ingots into molds. Comparing both modes of LDS sample preparations, Alkadi and Ruse found that the pressed samples demonstrated higher fracture toughness than those fabricated by CAD/CAM. (19) For ZLS, only CAD/CAM blocks are available for clinical use.

Regarding flexural strength and elastic modulus, no difference was observed between LDS and ZLS ceramics when they treated with HF and regardless etching times. Although ZLS glass ceramic is enriched with zirconia (approximately 10 % by weight), this composition was not able to increase the flexural strength compared to LDS. Because ZLS ceramic demonstrates more linear crystals (20) when compared to LDS, sandblasting treatment resulted in non-linear irregularities on its surface, which can generate more “edge-like” fissures that may accumulate more stress under mechanic loading. Thus, more agglomerated crystals after sandblasting (as observed in Figure 1A1) can generated more tension and therefore lead to lower strength than that showed with LDS.

1. CONCLUSION

Surface treatment protocols modified the surface topography of glass ceramics, which can influence the flexural strength and bond strength, depending on type of ceramic. Sandblasting is not recommended as surface treatment, because reduced the flexural strength for ZLS and yielded lower bond strength of resin cement to both glass ceramics. On the other hand, different etching times did not influence the mechanical properties of ceramics. Considering both evaluation times, HF20 was the better surface treatment for LDS.

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CONCLUSÃO

Dentro das limitações deste estudo "*in vitro*", as seguintes conclusões podem ser emitidas:

1. Não houve diferença estatística na resistência de união ao cisalhamento para ambas as cerâmicas em armazenamento imediato (24 horas), sendo que após o período de armazenamento por 1 ano os valores da cerâmica LDS tiveram um decréscimo acentuado quando utilizado HF 10 e 20 segundos;
2. Cerâmicas a base de ZLS apresentaram maior resistência de união pós armazenamento;
3. Não é recomendável a utilização do jateamento com óxido de alumínio como protocolo de tratamento de superfície por proporcionar redução da resistência flexural para a cerâmica ZLS e resistência de união reduzida para ambas as cerâmicas.

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APÊNDICE 1 - Figuras

Figura 1: Sequência de obtenção dos corpos de prova

Figura 2: Máquina de ensaio Universal (EZ-S TEST), ensaio de cisalhamento

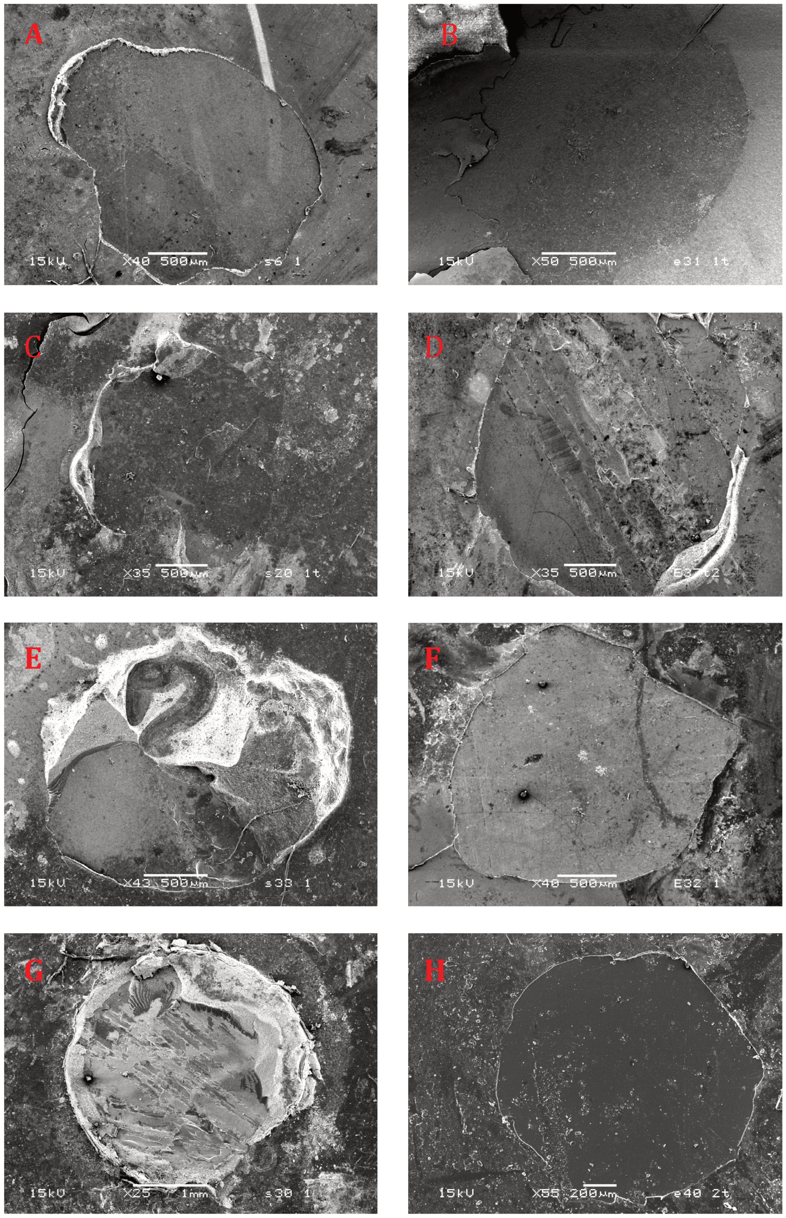

Figura 3. Padrões de fratura representativo por grupo em relação ao tratamento de superfície e armazenamento: **A- Suprinity (ZLS)** Jateamento (24hs) –falha adesiva ; **B- e.max Cad (LD**) jateamento (1 ano) **–** falha adesiva ; **C- Suprinity (ZLS)** HF10% (1 ano) - falha mista **; D- e.max Cad (LD)** HF 10% (24hs) –falha mista; **E- Suprinity (ZLS)** HF 20% (1 ano) - falha mista; **F- e.max Cad (LD) HF 20%** (1 ano)- falha adesiva; **G- Suprinity (ZLS)** HF 30% (24hs) – falha coesiva (no cimento); **H- e.max Cad (LD)** HF30% (1 ano) – falha adesiva.

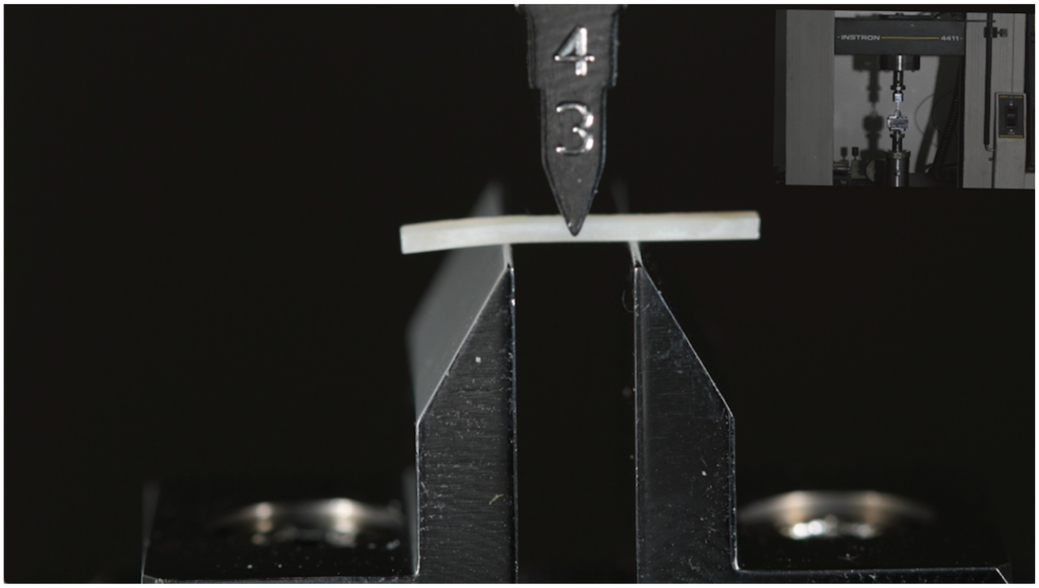

Figura 4: Máquina de ensaio Universal (Instron), ensaio de resistência flexural (3 pontos)

ANEXO 1 - Comprovante de Submissão do artigo

03/02/17 01:23

Dear Dr. Fernando José Rigolin Ferreira,

We have received your article "Effects of sandblasting and hydrofluoric acid etching on surface topography, flexural strength, modulus and bond strength of resin cement to ceramics" for consideration for publication in The Journal of Prosthetic Dentistry.

Your manuscript will be given a reference number once an editor has been assigned.

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Figura 1: Submissão do artigo