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DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO E SEU EFEITO NA RESISTÊNCIA FLEXURAL DE UMA CERÂMICA VÍTREA À BASE DE DISSILICATO DE LÍTIO

DISSERTAÇÃO DE MESTRADO

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Santa Maria, RS, Brasil
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DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO E SEU EFEITO NA RESISTÊNCIA FLEXURAL DE UMA CERÂMICA VÍTREA À BASE DE DISSILICATO DE LÍTIO

Catina Prochnow

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**

Orientador: Prof. Dr. Luiz Felipe Valandro

Santa Maria, RS, Brasil
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elaborada por
Catina Prochnow

como requisito parcial para a obtenção do grau de
Mestre em Ciências Odontológicas

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Santa Maria, 08 de julho de 2015.

DEDICATÓRIA

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“Se você ouve uma voz dentro de você dizer
‘você não pode pintar’, então pinte sem
dúvida, e essa voz será silenciada.”

Vincent Van Gogh

RESUMO

Dissertação de Mestrado
Programa de Pós-Graduação em Ciências Odontológicas
Universidade Federal de Santa Maria

DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO E SEU EFEITO NA RESISTÊNCIA FLEXURAL DE UMA CERÂMICA VÍTREA À BASE DE DISSILICATO DE LÍTIO

AUTORA: CATINA PROCHNOW
ORIENTADOR: LUIZ FELIPE VALANDRO
Data e Local da Defesa: Santa Maria – RS, 08 de julho de 2015.

Este estudo teve como objetivo avaliar o efeito de diferentes concentrações de ácido fluorídrico (HF) na resistência flexural de uma cerâmica vítrea à base de dissilicato de lítio. Espécimes cerâmicos na forma de barra (14x4x1,2mm) foram produzidos a partir de blocos cerâmicos (e.max CAD, Ivoclar Vivadent), conforme a ISO 6872. As barras cerâmicas foram aleatoriamente divididas em 5 grupos (n=23): SC (controle) – sem tratamento, HF1, HF3, HF5 e HF10 - condicionadas por 20 s com diferentes concentrações de ácido: 1%, 3%, 5% e 10%, respectivamente. As superfícies condicionadas foram avaliadas em microscópio eletrônico de varredura (MEV) e microscópio de força atômica (MFA). A rugosidade das superfícies tratadas foi aferida e os espécimes foram submetidos ao teste de resistência flexural (3 pontos). Os dados foram analisados utilizando ANOVA 1-fator, Teste de Tukey (α=0.05), Correlação de Pearson e análise de Weibull (módulo e resistência característica σ_o). Nenhuma diferença estatística foi encontrada entre os grupos para rugosidade e resistência flexural, da mesma forma que a correlação entre os dados de rugosidade e resistência flexural não teve significância estatística. A confiabilidade estrutural (modulo de Weilbull) foi semelhante entre os grupos testados, entretanto, HF1 apresentou resistência característica maior que HF10. O condicionamento com diferentes concentrações de HF não afetou a rugosidade superficial e a resistência flexural de uma cerâmica vítrea à base de dissilicato de lítio, quando comparada à cerâmica não tratada, independente da concentração de HF utilizada.

Palavras-chave: Ácido fluorídrico. Cerâmica. Resistência de materiais.

ABSTRACT

Master Course Dissertation
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HYDROFLUORIC ACID AND IT IS EFFECT ON THE FLEXURAL STRENGTH OF A LITHIUM DISILICATE- BASED GLASS CERAMIC

AUTHOR: CATINA PROCHNOW
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Date and Place of Defense: Santa Maria – RS, 2015, July 08.

This study aimed to evaluate the effect of different hydrfluoric acid (HF) concentrations on the flexural strength of a lithium disilicate based glass ceramic. Ceramic bar-specimens (14x4x1.2mm) were produced from ceramic blocks (e.max CAD, Ivoclar Vivadent), according to ISO 6872. The ceramic bars were randomly divided into 5 groups (n=23): SC (control) – without treatment, HF1, HF3, HF5 e HF10 – conditioned for 20 s with different acid concentrations: 1%, 3%, 5% e 10%, respectively. The etched ceramic surfaces were evaluated in a scanning electron microscope (SEM) and atomic force microscope (AFM). The roughness of treated surfaces was measured and the specimens were submitted to the 3-point flexural strength testing. Data were analyzed using 1-way ANOVA, Tukey's test (α=0.05), Pearson correlation and Weibull analyzis (modulus and characteristic strength). No statistical difference was found amoung groups for roughness and flexural strength, and the correlation between the data roughness and flexural strength was not statistically significance. The structural reliability (Weibull modulus) was similar among the tested groups, however, HF1 presented characteristic strength greater than HF10. The conditioning with different HF concentrations did not affect the surface roughness anfd flexural strength to a lithium disilicate based glass ceramic, when compared to untreated ceramic, regardless of the HF concentration used.

Keywords: Ceramic. Hydrofluoric acid. Resistance of materials.

SUMÁRIO

INTRODUÇÃO GERAL

Restaurações totalmente cerâmicas, feitas a partir de uma técnica computadorizada CAD/CAM, oferecem vantagens consideráveis quando comparadas aos sistemas de fabricação convencionais devido a seus aspectos estéticos e um menor número de defeitos/poros, e por consequência, maior densidade e baixa incidência de lascamento (Giordano, 2006; Seydler & Schmitter, 2015). Ademais, cristais de dissilicato de lítio foram adicionados às cerâmicas vítreas e proporcionaram um aumento a resistência, durabilidade e propriedades óticas em relação às cerâmicas convencionais (Zhang et al., 2013), devido à sua microestrutura com pequenos cristais aleatoriamente interligados (Aboushelib & Sleem, 2014). Apesar da otimização microestrutural, a longevidade e o sucesso das restaurações feitas de cerâmica a base de disslicato de lítio parecem estar diretamente relacionados à união destes sistemas cerâmicos e cimentos resinosos aos tecidos dentais (Blatz, Sadan & Kern, 2003; Valenti & Valenti, 2009). Para os procedimentos de cimentação adesiva, superfícies de esmalte e dentina (Nakabayashi & Pashley, 2000), bem como a superfície cerâmica (Phoenix & Shen, 1995) devem ser condicionadas adequadamente.

Neste sentido, o processo de adesão dessas cerâmicas ácido-sensíveis (como a cerâmica à base de dissilicato de lítio) aos materiais resinosos parece estar consolidado na literatura, pois a união é proporcionada pelo condicionamento com ácido fluorídrico (HF) e potencializada pela ação química do agente silano (Colares et al., 2013; Neis et al., 2015), sendo que o emprego de apenas um desses tratamentos parece não promover durabilidade da resistência de união (Stacey, 1993; Shimada et al., 2002; Matinlinna et al., 2004; Brentel et al., 2007).

Segundo Addison et al. (2007), o condicionamento ácido é um processo dinâmico e seu resultado varia de acordo com concentração do ácido, tempo de condicionamento e microestrutura do substrato. Assim, quando a superfície cerâmica é condicionada, o ácido fluorídrico ataca seletivamente a matriz vítrea (SiO_2) desta, expondo os óxidos de sílica e produzindo alterações topográficas que favorecem a união micromecânica e a união química com o agente de ligação silano e o cimento resinoso (Dilber et al., 2012). Como o HF reage com a sílica da cerâmica para formar hexafluorsilicatos, os cristais de dissilicato de lítio são projetados a partir do conteúdo vítreo, dissolvendo a subsuperfície, desempenhando um papel importante para um protocolo de adesão adequado (Höland et al., 2000).

Dentro desse contexto, o agente de união silano é uma molécula bi-funcional capaz de prover união química com superfícies orgânicas e inorgânicas. Ele desempenha função de ligação entre a sílica contida na cerâmica e a matriz orgânica dos cimentos resinosos através de uniões siloxanas (Lu et al., 1992; Söderholm & Shang, 1993; Della Bona et al., 2000; Debnath et al., 2000; Della Bona et al., 2004).

Como responsável pelos fenômenos físicos relacionados à resistência adesiva, está a energia de superfície de um material, que pode ser alterada naturalmente ou artificialmente, pelo condicionamento ácido e silanização. Ambos têm a propriedade de aumentar a molhabilidade do cimento na superfície (Lu et al., 1992; Phoenix & Shen, 1995; Melo, Valandro & Bottino, 2004), facilitando o contato com os cimentos resinosos. O aumento no número e no tipo de irregularidades na superfície de cerâmicas previamente condicionadas, têm sido associado ao aumento da resistência adesiva (Phoenix & Shen, 1995), sendo que a presença destas irregularidades aumenta a energia livre de superfície, reduz o ângulo de contato, facilita a penetração dos agentes de união e, desta forma, aumenta a molhabilidade e o potencial de adesão (Della Bona, Shen & Anusavice, 2004; Pisani-Proenca et al., 2006) .

Apesar de o condicionamento com HF da cerâmica reforçada por dissilicato de lítio proporcionar alterações topográficas para a retenção micromecânica e aumento da área superficial, ele parece ter um efeito de enfraquecimento na resistência flexural da cerâmica reforçada por cristais de dissilicato de lítio (Zogheib et al., 2011; Hooshmand et al., 2008). Além disso, existe uma clara evidência sobre a natureza das modificações dos defeitos da superfície da cerâmica em função do tempo de condicionamento do ácido HF e sua concentração (Addison et al., 2007). Segundo Quinn 2007, poros redondos e livres de falhas não atuam intensificando a tensão, apenas são pontos concentradores de tensão, porém, no caso de cerâmicas condicionadas, numerosos poros interligados por pequenas rachuaduras, propagam-se sobre tensões excessivas, levando a falha do material. Ainda, deve-se levar em consideração o potencial corrosivo do ácido fluorídrico, capaz de causar traumas graves aos tecidos moles, sendo diretamente relacionado à concentração do ácido e ao tempo de exposição (Ozcan, Allahbeickaraghi & Dundar, 2012).

Mesmo que numerosos estudos *in vitro* tenham firmemente estabelecido o aumento da resistência de união obtido pelo condicionamento com ácido fluorídrico (Panah et al., 2008; Erdemir et al., 2014) e o enfraquecimento que este provoca sobre a superfície da cerâmica de dissilicato de lítio (Hooshmand et al., 2008; Zogheib et al., 2011), não se sabe em que magnitude menores concentrações de HF irão afetar as propriedades mecânicas desta cerâmica sem causar prejuízo às alterações topográficas atinentes a adesão. Além disso, diferentes viscosidades e concentrações de ácido fluorídrico podem produzir padrões de condicionamento distintos.

Portanto, este estudo procura estabelecer um protocolo de condicionamento para favorecer a retenção micromecânica sem enfraquecer a cerâmica condicionada, utilizando quatro diferentes concentrações de ácido fluorídrico de mesma viscosidade e fabricante.

1. ARTIGO

Etching with distinct hydrofluoric acid concentrations and its effect on the flexural strength of a lithium disilicate-based glass ceramic

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Short title: Hydrofluoric acid concentration: impact on lithium disilicate strength

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The authors claim no conflicts of interest.

Etching with distinct hydrofluoric acid concentrations and its effect on the flexural strength of a lithium disilicate-based glass ceramic

Abstract

The purpose of this study was to examine the effects of distinct hydrofluoric (HF) acid concentrations on the mechanical behavior of a lithium disilicate-based glass ceramic. Ceramic bar-shaped specimens (14×4×1.2 mm) were produced from ceramic blocks (e.max CAD, Ivoclar Vivadent). All specimens were polished, chamfered, and sonically cleaned in distilled water. The specimens were randomly divided into 5 groups (n=23). SC (control)—no treatment; HF1, HF3, HF5, and HF10 were etched for 20 s with different acid concentrations: 1%, 3%, 5% and 10%, respectively. First, the etched surfaces were analyzed under a scanning electron microscope (SEM). Next, the specimens were observed using an atomic force microscope (AFM). Lastly, the roughness was measured and the 3-point bending flexure test was performed. Data were analyzed using one-way ANOVA and Tukey's test (α=0.05). Weibull modulus (m) and characteristic stress (σ_o) were also determined. No statistical difference for roughness and flexural strength was found among the groups. The structural reliabilities (Weilbull module) were similar to the tested groups; however, HF1 showed higher characteristic strength than HF10. Etching with different HF acid concentrations did not affect the surface roughness and flexural strength of a lithium disilicate-based glass ceramic when compared to the untreated ceramic, regardless of its concentration.

Keywords: Acid etching, AFM, flexural strength, glass ceramic, surface conditioning.

1. INTRODUCTION

The ceramic systems for computer-aided design/computer-aided manufacturing (CAD/CAM) are highly dense and contain a low quantity of inner defects/porositites, making ceramics structurally more reliable (Giordano, 2006); besides the presence of lithium disilicate crystals that improves strength and durability when compared with feldspar-based ceramics (Lawn, Dent and Thompson, 2001). These ceramic properties allow for the fabrication of inlay/onlays/laminates, as well as three-unit fixed partial dentures up to the second premolar (Albakry, Guazzatto and Swain, 2004). In general, the microstructure of lithium disilicate ceramics have two components: silica, which serves as the glassy matrix, and lithium oxide (Li_2O) crystals, which function as a flux used to lower the processing temperature of the glassy matrix from approximately 2000°C to 1100°C (Aboushelib and Sleem, 2014).

In terms of cementation, the restorations made of lithium disilicate ceramic can be satisfactorily conditioned by two major procedures: ceramic surface conditioning using hydrofluoric acid and silane coupling agent application prior to cementation with a resin cement (Hooshmand, Parvizi and Keshvad, 2008; Addison, Marquis and Fleming, 2007; Klosa et al., 2009). The hydrofluoric acid reacts with the glass matrix that contains silica and forms hexafluorosilicates. This glassy matrix is selectively etched and the crystalline microstructure is revealed microscopically. As a result, the ceramic surface becomes rougher for micromechanical retention of the resin cements (Chen, Matsumura and Atsuta, 1998). This roughly etched surface also helps to provide more surface energy prior to combining with the silane agent (Jardel et al., 1999), which promotes the chemical bond between the inorganic matrix of ceramic and the organic matrix of the resin cement, as a bifunctional molecule (Della Bona and Anusavice, 2002).

In vitro studies have reported that the effects of HF acid etching on the surface topography are positive, increasing roughness for adhesive bonding and removing and/or stabilizing surface defects (Erdemir et al., 2014; Neis et al., 2015). However, controversy remains regarding the impact of the etching protocol on the mechanical properties of lithium disilicate ceramic. Menees et al. (2014), evaluated the flexural strength of lithium disilicate ceramic following HF acid etching at two concentrations (i.e., 5% and 9.5%), and concluded that the microstructural changes promoted by HF acid did not negatively affect the flexural strength of this ceramic compared to the untreated ceramic. On the other hand, Zogheib et al. (2011) demonstrated a weakening effect of HF etching on lithium disilicate ceramics.

Apart from the potentially negative effects of HF acid etching on the mechanical properties of glass ceramics, the literature also reports that HF acid can produce acute hazardous effects on the skin and eyes in HF concentrations of 10% or less. In this way, even though there is no report on the incidence of these hazardous effects of HF in dentistry, the risk of toxicity by exposure to low concentrations and high frequency of use should be taken into consideration. (Ozcan, Allahbeickaraghi and Dündar, 2012)

Although some studies have reported the effects of different HF etching times and concentrations on bond strength (Erdemir et al., 2014; Neis et al., 2015), surface roughness (Addison, Marquis and Fleming, 2007) and flexural strength (Zogheib et al., 2011; Menees et al., 2014), none of these studies addressed the following research question: could the different concentrations of HF acid (with the same viscosity) affect the flexural strength of a lithium disilicate-based glass ceramic? It appears to be clinically relevant, since HF acid with lower concentrations could perhaps be used for ceramic etching.

Therefore, the purpose of this study was to examine the effects of distinct HF etching concentrations on the roughness and flexural strength of a lithium disilicate-based glass ceramic (IPS e.max CAD). The following hypotheses were tested: 1) different HF etching concentrations produce the same roughness patterns, 2) HF does not affect the ceramic flexural strength.

2. MATERIALS AND METHODS

2.1 Specimen Preparation

IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein) blocks were sectioned into 115 bar-shaped ceramic specimens (14×4×1.2 mm), using a diamond disc (15LC, Buehler, Lake Bluff, IL, USA) at low-speed, under water-cooling, in a sectioning machine (Isomet 1000, Buehler). The ceramic bars were wet-finished with 400-, 600-, and 1200-grit silicon carbide paper to remove irregular surface scratches and defects in a polishing machine (EcoMet 250, Buehler). After polishing, all edges were manually chamfered using a 0.1 mm-wide chamfer, as proposed by ISO 6872:1999. All ceramic specimens were sonically cleaned (Ultrasonic Cleaner 1440D, Odontobrás, Ribeirão Preto, São Paulo, Brazil) in distilled water for 10 min to remove debris. They were then fired (P500, Ivoclar-Vivadent, Schaan, Liechtenstein) as recommended by the manufacturer for crystallizing IPS e.max CAD.

2.2 Surface Conditioning

The 115 bars were randomly assigned to 5 experimental groups (n=23) according to the different surface treatment (Table 1). The tensile sides (for flexure tests) of the ceramic specimens were etched with hydrofluoric acid gel at different concentrations (FGM, Joinville, Brazil) for 20 s. The etched specimens were rinsed with air-water spray for 30 s and dried with compressed air for 30 s. The treated specimens were then sonicated in distilled water for 5 min.

2.3 Surface Roughness Analysis

For all groups, the surface roughness was determined using a surface roughness tester with a contact-type stylus (Mitutoyo SJ-410, Mitutoyo Corporation, Kanagawa, Japan). Measurement was performed 3 times for each specimen according to the ISO 4287:1997 parameters (Ra-arithmetical mean of the absolute values of peaks and valleys measured from a medium plane in µm, and Rz–average distance between the 5 highest peaks and 5 major valleys found in the standard in µm). The values of Ra and Rz were obtained from the average of three readings. Measurements were performed with $\lambda c=0.8$ mm ($0.1<Ra\leq 2.0$), resulting in a total measurement length of 4 mm.

2.4 Three-point Bending Test

Flexural strength was determined with a three-point bending test in a universal testing machine (EMIC DL-2000, EMIC, São Jose dos Pinhais, Brazil) performed according to ISO 6872:1999. Each ceramic bar was measured with a digital caliper prior to the test and numbered on the compression side. The etching side of the specimens was placed down and then they were placed flat on a mountain jig with rounded supporting rods 12 mm apart, and the center of the specimens was loaded (load cell 0.5 KN) with a rounded chisel (radius 3 mm) at a crosshead speed of 1 mm/min until fracture. The following equation was used for flexural strength (σ) calculation: $\sigma=3Pl/2wb^2$, where **P** is the fracture load (in N); **l** is the test span (12 mm); **w** is the width of the specimen (mm); and **b** is the thickness of the specimen (mm).

2.5 Statistical Analysis

One-way ANOVA and Tukey's test (α=0.05) were used to assess the surface roughness and flexural strength values. The Pearson Correlation analysis was used to verify correlation between the surface roughness and flexural strength.

In addition, the strength distributions of quasi brittle materials, such as ceramics, are more properly described by Weibull (1951) statistics, which define the reliability of the ceramic material and variation of the resistance (Tinschert et al., 2000), obtaining the Weibull module (m) and the characteristic strength (σ_c) with a confidence interval of 95%, determined in a diagram (according to ENV 843-5):

$$\ln \ln \left(\frac{1}{1-F}\right) = m \ln \sigma_c - m \ln \sigma_0$$

Where F is the failure probability, σ_0 the initial strength, σ_c the characteristic strength, and *m* is the Weibull modulus. A higher value of *m* indicates a close grouping of the flexure stress data, expressing reliability of the material, and the characteristic strength is considered to be the strength at a failure probability of approximately 63%.

2.6 Micromorphological Analysis

In order to observe the surface morphology of the different conditioning methods, two others specimens (12x10x7 mm) were produced from each group and imaged under an SEM (Jeol-JSM-T330A, Jeol Ltd; Tokyo, Japan) at distinct magnifications. For these analyzes, the specimens were sputter-coated with gold-palladium. In the same way, ceramic plates (12x10x7 mm) were also prepared for the AFM analysis (Agilent 5500 Equipment, Agilent Technologies, Santa Clara, USA).

The images (40 μm × 40 μm) were collected using a non-contact mode and PPP-NCL probes (Nanosensors, Force constant = 48 N/m). AFM micrographs were analyzed using a scanning probe microscopy data analysis software (Gwyddion™ version 2.33, GNU, Free Software Foundation, Boston, USA).

2.7 Fractographic Analysis

The tested specimens were randomly selected after the flexural strength test and they were prepared for examination of the fractured surfaces under SEM.

3. RESULTS

3.1 Surface Roughness

There were no significant differences in mean Ra (*$p=0.13$*) and Rz *($p=0.27$)* values among all the groups (Table 2).

3.2 Flexural Strength

There was no significant difference in mean flexural strength values among all groups (Table 2). The Pearson Correlation analysis showed no significant correlation between surface roughness (*Ra*) and flexural strength (MPa) for all the tested groups (Table 2).

In general, the Weibull modulus and characteristic strength were similar for all groups (Table 3 and Figure 1); however, there was an exception for the HF1 characteristic strength, which was significantly higher compared to HF10.

Figure 2 shows representative SEM micrographs of the fracture surfaces. In all cases, the initial defect is clearly seen on the tensile surface of the material, probably generated by etching, which creates several semi-elliptical cracks, that have linked. These short tails confirms that the fracture was originated in the tensile region. Quinn

(2007) referred to these cracks as “zipper cracks,” where the mirror zone can be elongated along the surface due to several defects in the surface, which commonly occurs in rectangular flexure specimens.

SEM and AFM images (Fig. 3) display the untreated ceramic surface as smooth and homogeneous, becoming increasingly more porous and irregular due to glass phase dissolution upon HF etching. As a consequence, voids and channels appear larger and deeper, as the HF etchant concentration increases. HF etching patterns appeared more noticeable and aggressive in HF5 and HF10, where lithium disilicate crystals can be easily seen protruding from the glassy matrix (Fig. 3 M to T).

4. DISCUSSION

Although the standard protocol for bonding to glass-based ceramics seems to be well-known and clear, requiring HF acid etching and silanization of the intaglio ceramic surface (Stewart, Jain and Hodges, 2002); it is of vital importance to better understand the mechanical behavior of these ceramics following HF acid etching. The findings from this current investigation indicated that HF etching does not change the mean values of flexural strength and roughness. Therefore, the two hypotheses that were tested were accepted.

Recent studies (Erdemir et al., 2014; Kalavacharla et al., 2015; Lise et al., 2015) have reported that hydrofluoric acid etching improves bond strength between lithium disilicate and resin cements. In brief, etching with HF acid create numerous microporosities, undercuts, and grooves by selective dissolution of the glassy matrix and exposure of the crystalline phase, promoting an increase in the surface area for bonding and micromechanical retention when combined with a resin cement (Colares et al., 2013; Aboushelib and Sleem, 2014).

In the present study, HF etching did not increase ceramic roughness in all experimental groups, regardless of the acid concentration used (1, 3, 5, or 10%). However, different results were reported by Zogheib et al. (2011) and Dilber et al. (2012). Zogheib et al. (2011) observed an increase in ceramic roughness after HF etching even for periods as short as 20 s, which is the etching time recommended by the manufacturer. Thus, it appears difficult to compare the present data to those of previous studies, which used a different roughness tester, ceramics, etching protocols, and HF acid viscosities. On the other hand, the AFM and SEM images showed distinct surface characteristics (i.e., a considerably higher number of irregularities, with deeper valleys and higher peaks) when compared to the untreated group. It might be that these surface treatments not only removed the glassy matrix and exposed the crystalline content, as observed with other ceramic materials but the fine-grains (0.2–1.0 µm) of lithium disilicate were also removed.

Ceramic etching is a dynamic process, and its impact is dependent on substrate constitution, surface topography, acid concentration, and etching time (Della Bona and Anusavice, 2002; Addison and Fleming, 2004). Although HF etching of glass-based ceramics provides the required surface roughness to mechanical interlocking, this process could have a weakening effect on these ceramics (Della Bona and Anusavice, 2002; Addison and Fleming, 2004; Hooshmand, Parvizi and Keshvad, 2009; Zogheib et al., 2011). Therefore, the present study investigated the adequate etching protocol for a lithium disilicate-based glass ceramic, looking for low concentrations of HF to be able to promote micromechanical retention without weakening the ceramic. Notwithstanding, in this study, the etching did not affect the flexural strength of a lithium disilicate-based ceramic. These findings are in agreement with other investigations that have reported no significant difference in flexural strength between the etched and non-etched ceramic surfaces (Thompson and Anusavice, 1994; Menees et al., 2014). However, Venturini et al. (2015), using the same HF concentrations and viscosities but with another ceramic (feldspathic), observed a statistical difference in flexural strength between the untreated ceramic and those etched with HF acid. One needs to consider that lithium disilicate ceramics have an unique microstructure consisting of small interlocking, plate- or needle-like crystals that are randomly oriented, act as crack stoppers, and increase flexural strength as compared to conventional glass ceramics (Aboushelib and Sleem, 2014). The different findings reported by Venturini et al. (2015) might evidence that the high glassy content of feldspathic ceramic (microstructure) makes the ceramic surface more susceptible to HF etching, even in low concentrations.

Similarly, the Weibull modulus (between 6.79 and 8.61) was similar for all tested groups, indicating that structural reliability was not affected by HF acid etching. Concerning characteristic strength (σ_c), which is the value that 63.21% of specimens fail, we found a statistically significant difference between HF1 and HF10. An explanation for this finding can be a homogenization of the defects created by polishing via 1% HF etching for a short time, this acid concentration was not able to develop new defects on the ceramic surface. When 10% HF was used, the surface degradation and removal of the glassy matrix was sufficient to promote a decrease in the σ_c for this group (HF10).

According to Quinn (2007), fractures in brittle materials, such as ceramics, start from pre-existing defects on the surface or within the bulk of the ceramic material, which propagate under excessive tensile stresses. Fractographic analysis identified the type of origins, where fracture was probably generated by acid etching, and it confirmed the presence of characteristic hackles of the fracture process.

Nevertheless, the limitations of this study should be highlighted. First, the 3-point bending test was performed under dry and static conditions, therefore, not closely mimicking the overall wet and cyclic nature of the oral environment. Yet, the flexural strength of the ceramic could be enhanced by unfilled resin treatment (Posritong et al., 2013). In this context, besides bond strength, new studies might search for more real simulations, closer to oral behavior, such as staircase, stepwise, and lifetime fatigue-based protocols.

5. CONCLUSION

Different HF acid concentrations ranging between 1% and 10% promote similar roughness and mean values of flexural strength to a lithium disilicate glass ceramic.

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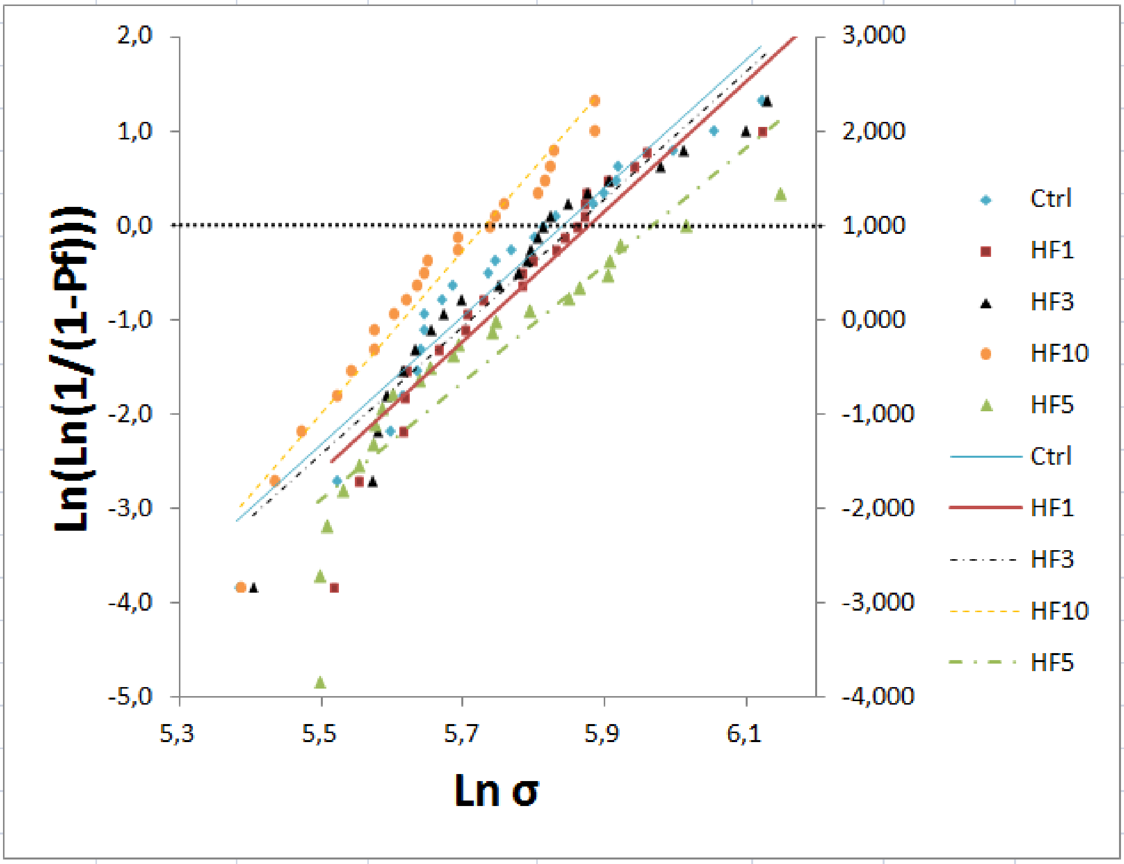


Figure 1. Weibull distribution for flexural strength (MPa) (Diamond: SC; Square: HF1; Black triangle: HF3, Green triangle: HF5 and Ball: HF10)

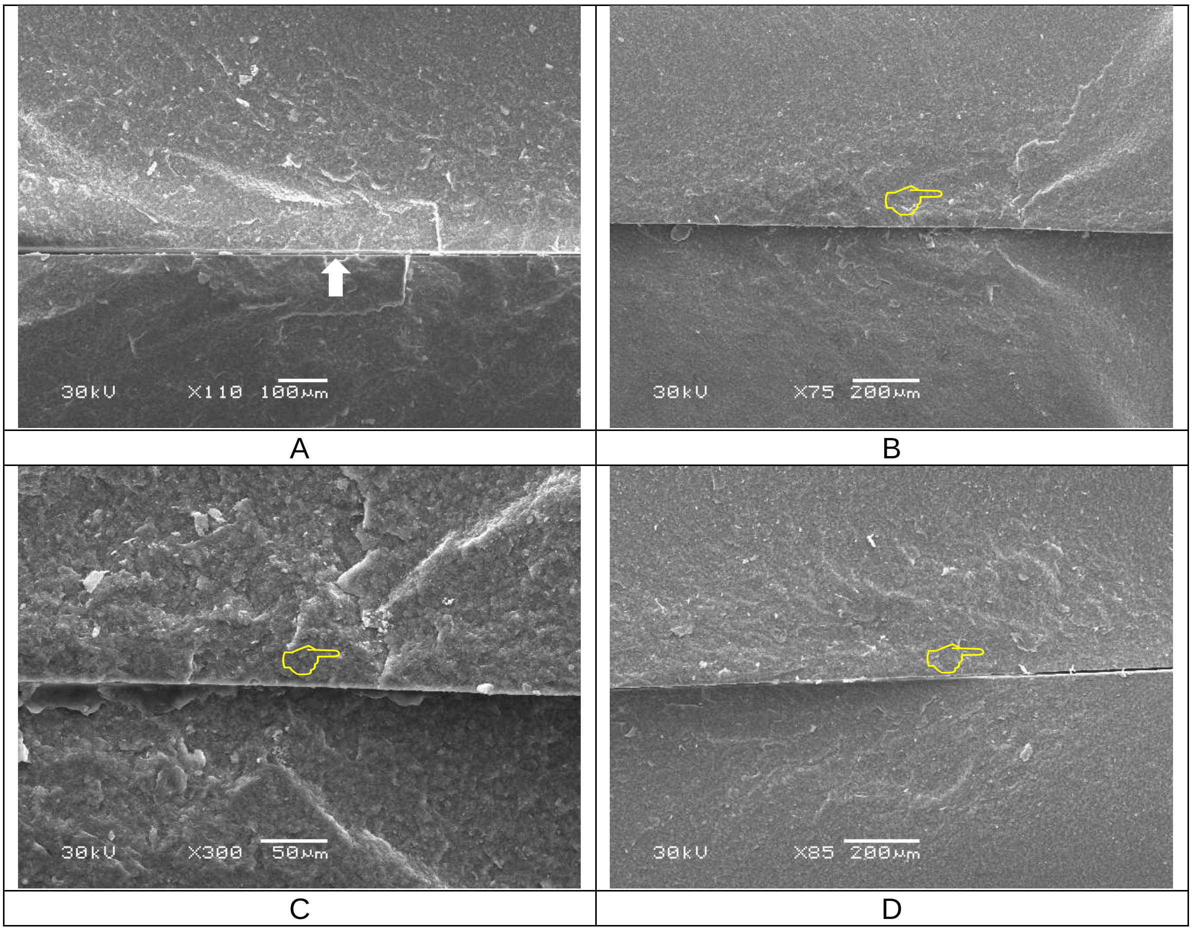
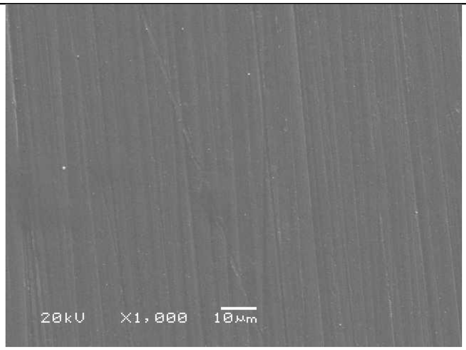
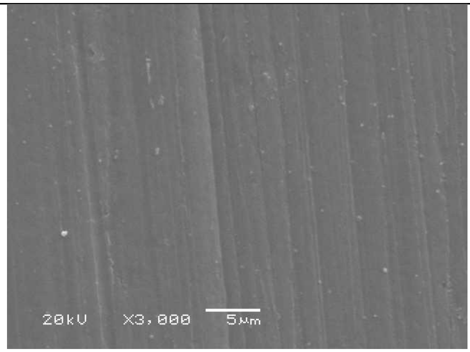
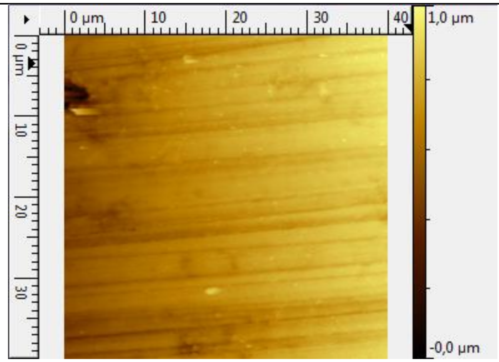
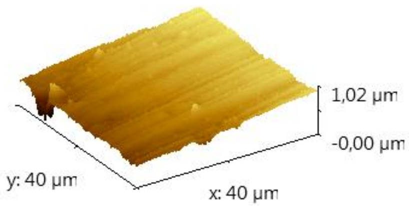
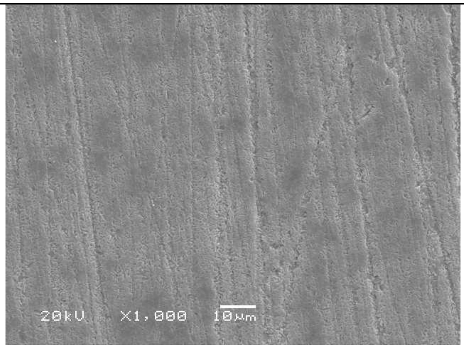
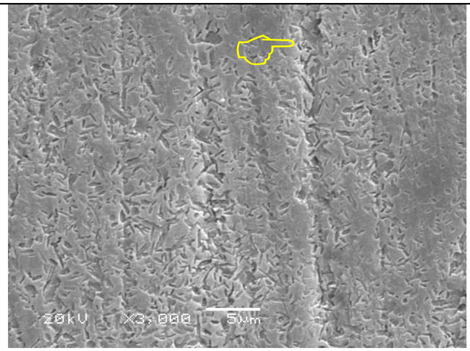
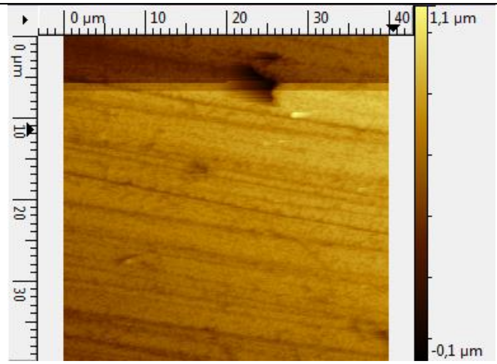
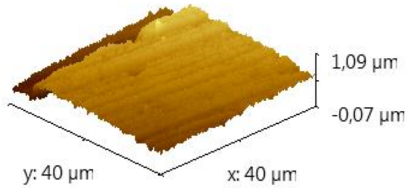
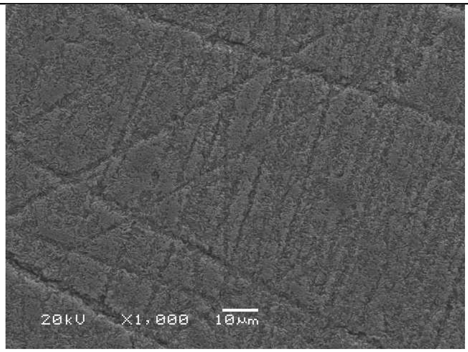
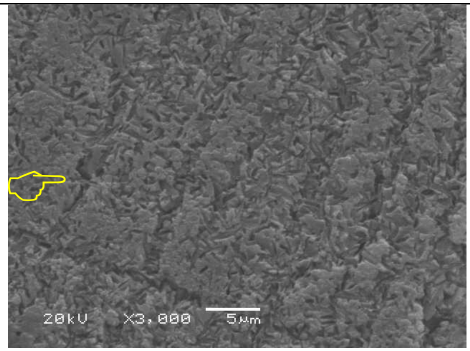
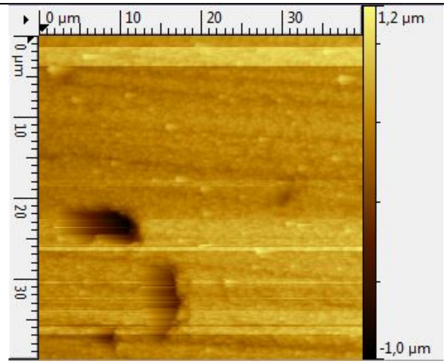
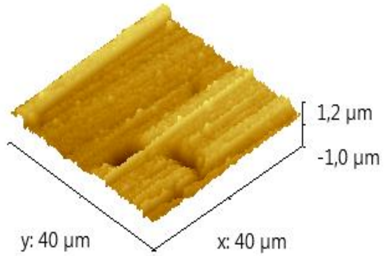


Figure 2. Representative micrographs of the fracture surface of a specimen from HF5 and HF10 group. The fracture origin (white arrow) was observed in the etched surface under tensile stresses and the hackles show the direction of crack propagation. It noted the numerous microcracks with a honeycombed appearance, which are the fracture origins.

	Scanning Electron Microscopy		Atomic Force Microscopy	
	1000×	3000×		
Untreated				
	A	B	C	D
1% hydrofluoric acid				
	E	F	G	H
3% hydrofluoric acid				
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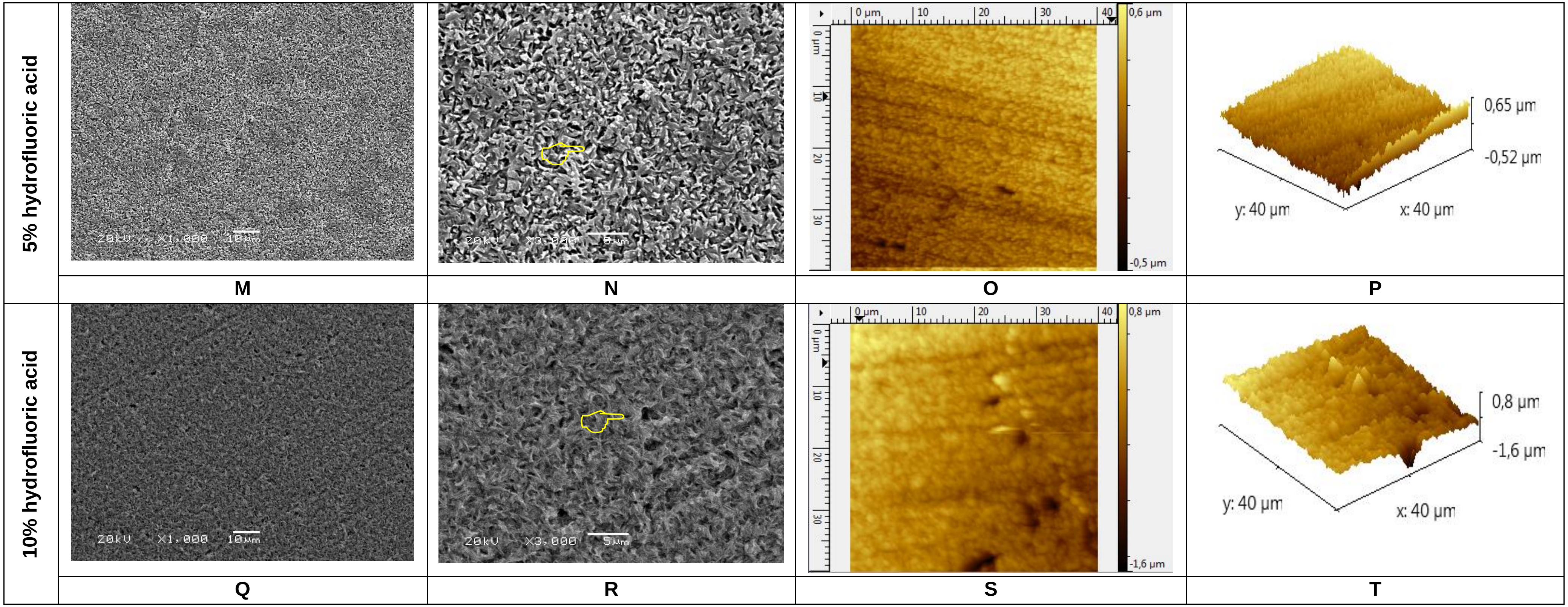

Figure 3 – Representative SEM and AFM images of different ceramic surface conditioning: untreated (A-D); etched for 20 s with HF 1% (E-H); HF 3% (I-L); HF 5% (M-P) and HF 10% (Q-T). In SEM micrographs, the indicators (☞) show the formation of micropores and cracks that occur due to dissolution of the glassy matrix by HF acid etching.

TABLES

Table 1. Surface conditioning for Roughness and Flexural Strength.

Groups	*Surface conditioning for Roughness and Flexural Strength*
SC	No surface treatment
HF1*	Etching with HF 1% 20 s + washing 30 s + drying 30 s
HF3*	Etching with HF 3% 20 s + washing 30 s + drying 30 s
HF5*	Etching with HF 5% 20 s + washing 30 s + drying 30 s
HF10**	Etching with HF 10% 20 s + washing 30 s + drying 30 s

SC: unconditioned control.
* Experimentally formulated - FGM; Santa Catarina, Brazil.
** Condac Porcelana 10%- FGM; Santa Catarina, Brazil.

Table 2. Mean values and standard deviations for Surface Roughness (*Ra* and *Rz*) and Flexural Strength and Pearson Correlation (*p* value) for Surface Roughness (*Ra*) and Flexural Strength.

Groups	*Surface roughness (Ra; µm)*	*Surface roughness (Rz; µm)*	*Flexural strength (MPa)*	*Pearson correlation (p value)*
SC	0.1676 ± 0.08 [a]	1.2587 ± 0.6 [a]	321.88 ± 57.6 [a]	r= 0.0374 (p= 0.8654)
HF1*	0.1291 ± 0.46 [a]	0.9634 ± 0.33 [a]	333.71 ± 59.7 [a]	r= -0.1638 (p= 0.4552)
HF3*	0.1282 ± 0.05 [a]	1.0044 ± 0.42 [a]	326.85 ± 59.2 [a]	r= -0.3804 (p= 0.0733)
HF5*	0.1372 ± 0.07 [a]	1.826 ± 0.59 [a]	308.36 ± 59.1 [a]	r= -0.2618 (p= 0.2276)
HF10**	0.1457 ± 0.04 [a]	1.0802 ± 0.34 [a]	291.48 ± 40.7 [a]	r= 0.1950 (p= 0.3726)

Different letters indicate statistically significant difference (Tukey's test; p<0.05).
* Experimentally formulated - FGM, Santa Catarina, Brazil.
** Condac Porcelana 10% - FGM, Santa Catarina, Brazil

Table 3. HF acid concentration influence on the Weibull parameters.

Groups	**Characteristic strength $\sigma_{63.21\%}$ (MPa)**	**Confidence intervals**	**Weibull modulus m**	**Confidence intervals**
SC	344.57 [ab]	319.21 - 371.39	6.79 [a]	4.41 - 9.03
HF1	357.23 [a]	331.16 - 384.79	6.85 [a]	4.45 - 9.11
HF3	350.12 [ab]	324.13 - 377.63	6.73 [a]	4.38 - 8.95
HF5	332.33 [ab]	305.6 - 360.81	6.19 [a]	4.02 - 8.23
HF10	308.28 [b]	290.25 - 327.05	8.61 [a]	5.6 - 11.45

Same letters correspond to statistical similarity
Different letters correspond to statistical difference

4. CONSIDERAÇÕES FINAIS

Embora o protocolo padrão para união à cerâmica vítrea à base de dissilicato de lítio pareca estar claro, exigindo condicionamento com HF e silanização da superfície interna da cerâmica, julgamos necessária uma melhor compreensão sobre o comportamento mecânico deste materiail submetido ao condicionamento com diferentes concentrações de ácido fluorídrico. Os resultados deste estudo indicaram que o condicionamento com HF não altera os valores médios de resistência à flexão e rugosidade.

Da mesma forma, encontramos resultados de confiabilidade (módulo de Weibull) semelhantes para todos os grupos testados, indicando que a confiabilidade estrutural da cerâmica testada não foi afetada pelos diferentes protocolos de condicionamento. A diferença estatística encontrada na resistência característica entre os grupos HF1 e HF10 pode ser justificada pela maior degradação da superfície cerâmica, quando condicionada com o ácido a 10%, sugerindo que a concentração de 1%, no curto período de condicionamento utulizado (20 s) foi capaz apenas de homogeneizar defeitos pré-existentes na superfície, criados pelo protocolo de polimento.

Por outro lado, as imagens de AFM e MEV apresentaram características distintas das superfícies cerâmicas condicionadas (número consideravelmente mais elevado de irregularidades, com vales mais profundos e picos mais altos) quando comparadas ao grupo controle. Hipotetizamos que o condicionamento não apenas removeu a matriz vítrea e expôs o conteúdo cristalino, conforme observado com outros materiais cerâmicos, mas também os finos grãos (0,2 - 1.0μm) de dissilicato de lítio.

No entanto, as limitações deste estudo devem ser consideradas: o teste de flexão de 3 pontos foi realizado sob condições secas e estáticas, portanto, não mimetizou a natureza úmida e cíclica do ambiente oral. Também, há indícios de um aumento na resistência de restaurações cerâmicas após o procedimento de cimentação adesiva. Assim, sugerimos que novos estudos devem testar, além de resistência de união, simulações mais reais do comportamento deste material em meio oral – através de ensaios de fadiga em ambiente úmido.

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ANEXOS

Anexo A – Normas para publicação no periódico *Journal of the Mechanical Behavior of Biomedical Materials*

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