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Catina Prochnow

DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO: EFEITO NA ADESÃO E COMPORTAMENTO EM FADIGA DE UMA CERÂMICA DE DISSILICATO DE LÍTIO

Santa Maria, RS
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Tese apresentada ao Curso de Doutorado 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 **Doutor em Ciências Odontológicas**.

Orientador: Prof. Dr. Luiz Felipe Valandro
Coorientador: Prof. Dr. Gabriel Kalil Rocha Pereira

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Aprovada em 06 de setembro de 2018:

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Santa Maria, RS
2018

DEDICATÓRIA

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"Ver na vida algum motivo pra sonhar,

ter um sonho todo azul,

azul da cor do mar" Tim Maia

RESUMO

DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO: EFEITO NA ADESÃO E COMPORTAMENTO EM FADIGA DE UMA CERÂMICA DE DISSILICATO DE LÍTIO

AUTORA: Catina Prochnow
ORIENTADOR: Luiz Felipe Valandro
COORIENTADOR: Gabriel Kalil Rocha Pereira

Três estudos compõe a presente tese. Primeiramente, o efeito de diferentes concentrações de ácido fluorídrico (HF; 1%, 3%, 5% e 10%) na resistência de união ao microcisalhamento entre uma cerâmica de dissilicato de lítio e um cimento resinoso foi avaliado. Amostras (12×14×2mm) de dissilicato de lítio foram condicionadas com as respectivas concentrações de ácido, silanizadas e cilindros de cimento resinoso (Ø=0.72mm) foram construídos sobre as superfícies. Metade das amostras foi testada após 24 h, e a outra metade foi submetida ao envelhecimento (150 dias + 12.000 ciclos térmicos – 5° e 55°C) prévio ao ensaio. Ângulo de contato, microscopia de força atômica e topografia de superfície foram realizadas. Na condição inicial, HF3=HF5=HF10 (13.9–15.9MPa) ($p>.05$), e HF1<HF5. Após o envelhecimento, o grupo HF1 apresentou a menor resistência de união (HF3=HF5=HF10>HF1). Os grupos HF3, HF5 e HF10 apresentaram menores valores de ângulo de contato (7.8–10.4°). Maiores concentrações de HF promoveram maiores alterações topográficas e consequentemente, superfícies mais rugosas. Em termos de adesão, concentrações de 3%, 5% e 10% parecem ser adequadas para o condicionamento do dissilicato de lítio. Segundamente, o efeito de diferentes concentrações de HF na carga cíclica para falha de discos de dissilicato de lítio cimentados a um material análogo de dentina foi investigado. Discos cerâmicos (Ø=10mm; espessura=1.5mm) e discos de um material análogo de dentina (G10; Ø=10 mm, espessura=2mm) foram produzidos. A exceção do controle (CTRL), os discos cerâmicos foram condicionados (HF3), (HF5) ou (HF10) e posteriormente silanizados, enquanto os discos de G10 receberam a aplicação de um primer adesivo. Após a cimentação adesiva, a carga cíclica para falha (500.000 ciclos, 20Hz, carga inicial=720N, incremento=70N) foi obtida através do método da escada, na presença de água. Um pistão de aço hemisférico (Ø=40mm) aplicou a carga no centro dos espécimes. Análise fractográfica e topográfica foram realizadas. Concentrações intermediárias de ácido fluorídrico (3% e 5%) apresentaram maiores valores de carga cíclica para falha, e o grupo controle (não condicionado) apresentou o pior comportamento em fadiga [HF3(1355.0) = HF5(1335.0) > HF10(1175.0) > CTRL(965.0)]. Todas as falhas observadas foram trincas radiais iniciadas a partir da superfície de cimentação. Por fim, o efeito de diferentes concentrações de HF foi avaliado na carga cíclica para falha de restaurações monolíticas de dissilicato de lítio usinadas pelo sistema CAD-CAM adesivamente cimentadas a preparos protéticos simplificados (G10). O método da escada foi utilizado para os ensaios de fadiga utilizando a mesma configuração de teste e delineamento experimental do estudo anterior. Análises topográfica, fractográfica e de dimensão fractal foram realizadas. Não houve diferença estatística entre os grupos testados [CTRL(805.00) = HF3(781.25) = HF5(755.00) = HF10(833.75)]. Apesar das análises de dimensão fractal e topografia de superfície terem mostrado superfícies mais complexas para HF3 e HF10, o padrão topográfico criado pela usinagem foi preponderante nas coroas de dissilicato de lítio. Todas as falhas encontradas foram trincas radiais iniciadas a partir da superfície de cimentação. Baseado nos resultados da presente tese, em termos de adesão e comportamento em fadiga, a cerâmica testada pode ser condicionada com HF 3% e 5%.

Palavras-chave: Ácido Fluorídrico. Adesão. Ângulo de Contato. Cad/Cam. Carregamento Cíclico Dissilicato de Lítio. Fadiga. Força Atômica. Microcisalhamento. Resistência de União. Rugosidade.

ABSTRACT

DIFFERENT HYDROFLUORIC ACID CONCENTRATIONS: EFFECT ON ADHESION AND FATIGUE BEHAVIOR OF A LITHIUM DISILICATE CERAMIC

AUTHOR: Catina Prochnow
PROMOTER: Luiz Felipe Valandro
CO-PROMOTER: Gabriel Kalil Rocha Pereira

The studies compound the present thesis. Firstly, the effect of different hydrofluoric acid (HF) concentrations (1%, 3%, 5% and 10%) on the microshear bond strength between a lithium disilicate ceramic and a resin cement was evaluated. Samples (12×14×2mm) of lithium disilicate were etched with the respective HF concentrations, silanized, and resin cement cylinders (Ø=0.72mm) were built up over the surfaces. A half of the samples was tested after 24h, and the other half was submitted to aging (150 days + 12,000 thermocycles – 5° and 55°C) previously to testing. Contact angle, atomic force and surface topography analysis were performed. On the "baseline" condition, HF3=HF5=HF10 (13.9–15.9MPa) ($p>.05$), and HF1<HF5. After aging, the group HF1 presented the lowest bond strength (HF3=HF5=HF10>HF1). The groups HF3, HF5, and HF10 presented lower contact angle values (7.8–10.4°). Higher HF concentrations promoted higher topographic alterations and consequently, rougher surfaces. In terms of adhesion, concentrations of 3%, 5% and 10% seem to be adequate for etching lithium disilicate. Secondly, the effect of different HF concentrations on the cyclic load-to-failure of lithium disilicate discs luted to a dentin analogue material was investigated. Ceramic discs (Ø=10mm; thickness=1.5mm) and of a dentin analogue material (G10; Ø=10 mm, thickness=2mm) were produced. With exception of the control group (CRTL), the ceramic discs were etched (HF3), (HF5) or (HF10) and later silanized, meanwhile the G10 discs received an adhesive primer application. After the adhesive luting, the cyclic load-to-failure (500,000 cycles, 20Hz, initial load=720N, step size=70N) was obtained by the staircase method, under water. A stainless-steel piston (Ø=40mm) applied the load in the center of the samples. Fractographic and topographic analysis were performed. Intermediate HF concentrations (3% and 5%) presented higher cyclic load-to-failure values, and the control group (non-etched) presented the worst behavior under fatigue [HF3(1355.0) = HF5(1335.0) > HF10(1175.0) > CTRL(965.0)]. All failures observed were radial cracks starting from the cementation surface. Lastly, the effect of different HF concentrations was evaluated on the cyclic load-to-failure of monolithic lithium disilicate restorations machined by CAD-CAM adhesively luted to simplified prosthetic preparations (G10). The staircase method was used for the tests following the same test assembly and experimental design of the previous study. Topographic, fractographic and fractal dimension analyses were performed. There was no statistical difference among the tested groups [CTRL(805.00) = HF3(781.25) = HF5(755.00) = HF10(833.75)]. Despite the fractal dimension and surface topography analyses have shown complex surfaces to HF3 and HF10, the topographic path created by machining was overweight on the lithium disilicate crowns. All failures found were radial cracks started from the luted surface. Based on the findings of the present thesis, in terms of adhesion and fatigue behavior, the ceramic tested can be etched with 3 and 5% HF.

Keywords: Adhesion. Atomic Force. Bond Strength. Cad/Cam. Contact Angle. Cyclic Load. Fatigue. Hydrofluoric Acid. Lithium Disilicate. Microshear. Roughness.

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1 INTRODUÇÃO GERAL

Devido a sua estética favorável, biocompatibilidade e excelentes propriedades mecânicas, cerâmicas odontológicas têm sido o material de escolha em grande parte das restaurações indiretas (KITAYAMA et al., 2010), apresentando altas taxas de sucesso de aproximadamente 95% (PIEGER et al., 2014; SAILER et al., 2015). Contudo, a longevidade e o sucesso clínico destas restaurações são resultado de uma combinação de diferentes fatores, tais como propriedades mecânicas dos materiais e sua confiabilidade (GUESS et al., 2009), danos causados pelo processamento (fundição, usinagem, sinterização, entre outros) e cargas cíclicas às quais os materiais são submetidos quando em função (ZHANG et al., 2006).

Cerâmicas produzidas industrialmente (pré-fabricadas) destacam-se por diminuído tempo laboratorial e melhor controle de qualidade entre as peças protéticas (MIYAZAKI; HOTTA, 2009), pois são materiais estruturalmente mais homogêneos e confiáveis quando comparados às cerâmicas estratificadas manualmente pelo técnico (TINSCHERT et al., 2000). Neste cenário, a cerâmica vítrea à base de dissilicato de lítio no formato de blocos pré-cristalizados para sistemas CAD/CAM (Computer Aided-Desing/Computer Aided-Manufacturing; IPS e.Max CAD, Ivoclar Vivadent) tem se destacado (BEIER; KAPFERER; DUMFAHRT, 2012) por possuir características estéticas e mecânicas que a tornam muito versátil, principalmente no que tange às suas indicações (restaurações totalmente cerâmicas do tipo *inlay*, *onlay*, faceta, coroa monolítica e próteses fixas de até três elementos) (ALBAKRY; GUAZZATO, 2004).

Os excelentes resultados estéticos e alta resistência observados na cerâmica de dissilicato de lítio estão relacionados a sua peculiar microestrutura de ‘cristais em forma de agulha’ (TYSOWSKY, 2009), sendo que a cimentação adesiva parece potencializar a resistência à fratura de coroas deste material (POWERS et al., 2009). Para o procedimento de cimentação adesiva, a superfície interna das restaurações vitrocerâmicas deve ser condicionada pelo ácido fluorídrico (HORN, 1983) e silanizada (ABOUSHELIEB; SLEEM, 2014), uma vez que a aplicação de somente um destes tratamentos de superfície parece não promover resultados satisfatórios (MATINLINNA et al., 2004; SHIMADA; YAMAGUCHI; TAGAMI, 2002).

O papel do condicionamento com ácido fluorídrico é a remoção da matriz vítrea, criando alterações topográficas superficiais necessárias para a retenção micromecânica (MENEES et al., 2014), assim, diferentes concentrações de ácido fluorídrico são capazes de promover diferentes padrões de rugosidade superficial e área de superfície disponível para embricamento micromecânico com cimentos resinosos (CHEN et al., 1998; ERDEMIR et al., 2014; SUNDFELD NETO et al., 2015). Como consequência do tratamento de superfície com ácido fluorídrico, a energia de superfície da cerâmica é aumentada e o ângulo de contato entre cerâmica e cimento resinoso diminuídos pela remoção dos defeitos superficiais, promovendo a molhabilidade necessária para o silano e cimento resinoso infiltrarem nas irregularidades da superfície cerâmica condicionada (PHOENIX; SHEN, 1995; LISE et al., 2015). O agente de união silano é uma molécula bifuncional, capaz de prover a união química entre matéria orgânica e inorgânica (LUNG; MATINLINNA, 2012). No caso das cerâmicas vítreas, ele desempenha a função de ligação entre a sílica contida na cerâmica e a matriz orgânica dos cimentos resinosos através de uniões siloxanas (DELLA BONA; MECHOLSKY; ANUSAVICE, 2004).

Além disso, o ácido fluorídrico é altamente tóxico, e sua toxicidade depende da concentração utilizada e tempo de contato com a pele/mucosa, podendo levar à necrose tecidual (OZCAN; ALLAHBEICKARAGHI; DÜNDAR, 2012; DENNERLEI et al., 2016). Existem estudos laboratoriais que avaliaram o efeito de diferentes regimes de condicionamento (tempo e concentração de ácido fluorídrico) na resistência de cerâmicas de dissilicato de lítio, entretanto, estes utilizam testes estáticos de resistência à flexão na forma de barras ou discos (HOOSHMAND; PARVIZI; KESHVAD, 2008; ZOGHEIB et al., 2011; MENEES et al., 2014; PROCHNOW et al., 2017). Desta forma, os materiais não falham devido a um dano cumulativo (fadiga), como ocorre quando os materiais estão em função, sob a ação de cargas mastigatórias intermitentes (SCHERRER et al., 2003). Assim, os ensaios de fadiga cíclica são um complemento aos ensaios monotônicos tradicionais, pois reproduzem uma condição mais próxima do que ocorre clinicamente (WISKOTT; NICHOLLS; BELSER, 1995). Além disso, os estudos com espécimes geométricos (barras, discos) não levam em consideração a influência da geometria da restauração na distribuição de tensões, o método de confecção dos corpos-de-prova (KELLY, 1999), nem tampouco a condição de cimentação dos espécimes e sua influência no comportamento mecânico global. Consequentemente, corpos-de-prova usinados na forma de restaurações (GRESSLER MAY et al., 2015; VENTURINI et al., 2018), ou mesmo discos cerâmicos cimentados adesivamente (CHEN et al., 2014; MONTEIRO et al., 2018; SCHERER et al., 2018), os quais contemplam um maior número de variáveis encontradas clinicamente, estão indicados quando se pretende prever com maior confiabilidade o comportamento dos sistemas cerâmicos a partir de ensaios laboratoriais.

Cargas cíclicas em um meio úmido são consideradas as principais responsáveis pelo desenvolvimento e crescimento lento de trincas, que diminuem a resistência dos materiais restauradores, ou ainda causam a sua falha (KELLY, 1999; DRUMMOND et al., 2009). Essas trincas geralmente se originam na superfície interna da região oclusal, a qual possui uma maior concentração de tensões de tração (MAY et al., 2015) e onde é realizado o condicionamento com ácido fluorídrico, que modifica a topografia da superfície cerâmica, e consequentemente a população de defeitos (MENEES et al., 2014). Esta premissa é confirmada através da análise fractográfica de coroas que falharam clinicamente e também da análise de elementos finitos, que indicam que a superfície de cimentação da restauração cerâmica concentra grande parte das tensões de tração responsáveis pelo início da falha nessas restaurações (KELLY et al., 1990; THOMPSON; ANUSAVICE, 1994; QUINN, 2007; MAY et al., 2012; MONTEIRO et al., 2018). Assim, Addison (2007) afirma existirem claras evidências entre a natureza das modificações dos defeitos de superfície em função do tempo de condicionamento do ácido fluorídrico e sua concentração. Além disso, apesar de criar alterações topográficas propícias à adesão, estudos têm mostrado que o condicionamento com diferentes concentrações deste ácido afeta negativamente a resistência mecânica de cerâmicas de dissilicato de lítio (HOOSHMAND; PARVIZI; KESHVAD, 2008; ZOGHEIB et al., 2011). Neste contexto, a avaliação da variação de concentração deste ácido no comportamento de resistência à fadiga é um requisito importante, pois esse procedimento pode influenciar diretamente o sucesso clínico de restaurações cerâmicas.

Assim, torna-se pertinente avaliar *in vitro* o efeito de diferentes protocolos de condicionamento ácido na resistência de união e resistência à fadiga de restaurações de uma cerâmica vítrea à base de dissilicato de lítio. Considerando o contexto exposto, a presente tese de doutorado será dividida em três artigos:

O primeiro deles, intitulado **“*Adhesion to a lithium disilicate glass ceramic etched with hydrofluoric acid at distinct concentrations*”**, se propôs avaliar o efeito de diferentes concentrações de ácido fluorídrico (1%, 3%, 5% e 10%) na resistência de união ao microcilhamento entre uma cerâmica à base de dissilicato de lítio e um cimento resinoso.

O segundo artigo, o qual teve origem a partir dos resultados obtidos no precursor , é intitulado ***“How does hydrofluoric acid etching affect the cyclic load-to-failure of lithium disilicate restorations?”*** e objetivou investigar o efeito de diferentes concentrações de ácido fluorídrico (3%, 5% e 10%) na carga cíclica para falha de restaurações simplificadas de dissilicato de lítio cimentados adesivamente a um material análogo de dentina, através de uma metodologia simplificada (adaptada de CHEN et al., 2014).

Por fim, o terceiro artigo, intitulado **“*Hydrofluoric acid concentrations: effect on the cyclic load-to-failure of machined lithium disilicate restorations*”** teve como propósito avaliar as mesmas concentrações de ácido fluorídrico supracitadas (3%, 5% e 10%) na carga cíclica para falha de coroas monolíticas de dissilicato de lítio usinadas a partir do sistema CAD/CAM, cimentadas adesivamente a um material análogo de dentina, através do método de escada (*staircase*).

2 ARTIGO 1 - ADHESION TO A LITHIUM DISILICATE GLASS CERAMIC ETCHED WITH HYDROFLUORIC ACID AT DISTINCT CONCENTRATIONS

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Adhesion to a lithium disilicate glass ceramic etched with hydrofluoric acid at distinct concentrations

Short title: HF etching and bond strength to a glass-ceramic

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SUMMARY

This study evaluated the effect of different hydrofluoric acid (HF) concentrations on the bond strength between a lithium disilicate-based glass ceramic and a resin cement. Eighty ceramic blocks (12×7×2 mm) of IPS e.Max CAD (Ivoclar Vivadent) were produced and randomly assigned to 8 groups, considering 2 study factors: HF concentration in 4 levels, i.e., 1% (HF1), 3% (HF3), 5% (HF5), and 10% (HF10), and storage in 2 levels, i.e., baseline (tests were performed 24h after cementation), and aged (storage for 150 days + 12,000 thermal-cycles at 5°C and 55°C). Acid etching (20 s) was performed, followed by washing, drying, and silanization. Four resin cement cylinders (Ø= 0.96 mm) were built-up from starch matrices on each ceramic sample (n= 40). Additional ceramic samples were etched and analyzed for contact angle, micro-morphology, and roughness. In baseline condition, the HF3, HF5, and HF10 groups showed similar bond strength values (13.9 – 15.9 MPa), and HF1 presented lower values than HF5, being that statistically different (p= 0.012). After aging, all the mean bond strengths statistically decreased, being that HF3, HF5, and HF10 were similar and higher than HF1 (p= 0.0001). For contact angle, HF3, HF5, and HF10 presented similar values (7.8 – 10.4°), lower than HF1 and CTRL groups. HF5 and HF10 presented rougher surfaces than other conditions. For better bond strength results, the tested ceramic may be etched by HF acid in concentrations of 3%, 5%, and 10%.

Keywords: atomic force microscopy, conditioning, microshear, surface treatment, vitreous ceramic.

INTRODUCTION

The addition of lithium disilicate crystals in glass ceramics improves strength and durability over conventional dental ceramics (1). Nonetheless, the longevity and success of lithium disilicate ceramic restorations is directly related to achievement of the bonding process (2). Prior to resin cementation, the protocol for glass ceramic restorations requires etching with hydrofluoric acid (HF) and silane application on the intaglio ceramic surface (3). Retention is well achieved with these procedures (2,4) and, according to a recent study, when used separately, they do not promote satisfactory results (5).

Ceramic etching is a dynamic process and its result varies owing to acid concentration, etching time, substrate constitution, physical structure, and surface topography (6). Basically, the acid reacts with the ceramic glass matrix, selectively removing it and exposing the crystalline structure (5). The inner surface of all ceramic restoration becomes rough, with increased surface area available for bonding and with undercuts, promoting micromechanical interlocking with resin cement (4,7).

As consequence of the surface treatment, the ceramics' surface energy is increased and the contact angle between the ceramic and resin cement decreased by removal or stabilization of surface defects, providing the needed wettability for the silane and resin cement to infiltrate into irregularities of the conditioned ceramic surface (5). The previous application of silane allows resin cement to chemically bond to the intaglio ceramic surface (3). Silane works as a bi-functional molecule, in which one extremity reacts with a glassy phase of the ceramic surface, while the other extremity copolymerizes with methacrylate groups within the organic matrix of resin cements by siloxane bonds, determining an adhesive cementation (4). In this way, silane improves the durability and bond strength of ceramic restorations (4).

On the other hand, it has been pondered that over-etching can weaken glass-ceramics (6,8), because removal of the glassy matrix and the defects population created on the ceramic surface are related to time and concentration of hydrofluoric acid (8). According to Addison et al. (6), HF acid etching increases the maximum peak to trough amplitude, thus increasing the pre-existing asymmetry in the survival probability distributions and reliability of the fracture strength data. Also, the presence of defects, as created by HF etching, is associated with the stress propagation from flaws at the bonding surface of dental porcelain restorations (6,8).

Moreover, HF is capable of causing severe trauma to soft tissues, considering it is a hazardous substance and extremely corrosive. Epithelial necrosis was observed in rat skin 24 h after HF exposure (9). In addition, skin damage showed a strong extension into deeper skin layers with increasing HF concentration and exposure duration (10).

Taking into account these premises and concepts, an optimal concentration of hydrofluoric acid required to promote durable bond strengths without weakening glass ceramics remains uncertain. Thus, could etching with hydrofluoric acid at distinct concentrations (1%, 3%, 5%, and 10%) promote similar resin adhesion to lithium disilicate ceramic? For this reason, the purpose of the current study was to examine the effects of different HF concentrations on the contact angle, roughness, and durability of bond strength between a lithium disilicate-based glass ceramic (IPS e.Max CAD) and a resin cement. The hypotheses tested were: 1) HF acid at different concentrations will influence the values of bond strength, roughness, and contact angle and 2) storage and thermocycling will decrease bond strength values when compared to dry conditions.

MATERIALS AND METHODS

Contact Angle Measurement

Twenty-five ceramic slices (12×7×2 mm^3) were prepared from prefabricated ceramic blocks IPS e.Max CAD (lvoclar Vivadent, Schaan, Oberland, Liechtenstein). Ceramic blocks were sectioned using a diamond disc at low-speed, under water-cooling, and in a cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). The surface was flattened and polished using silicon carbide papers (#400- #600-, and #1200-grit; 3M, Sumare, SP, Brazil). Then, all ceramic slices were crystallized (P500, Ivoclar-Vivadent; 840° C, vacuum – 7 min), followed by cleaning in an ultrasonic device (Vitasonic, Vita Zanhfabrik, Bad Sackingen, BW, Germany) for 10 min using isopropyl alcohol.

The ceramic samples were randomly assigned (www.randomizer.org) to 5 groups (n=5) according to the surface conditioning method (Table 1). The experimental ceramic surfaces were etched using 4 concentrations of hydrofluoric acid with the same procedures: etching for 20 s, rinsing with air-water spray for 30 s, drying for 30 s, and ultrasonic cleaning (Vitasonic, Vita Zanhfabrik) in distilled water for 5 min, meanwhile the control group (without hydrofluoric acid etching) was just ultrasonic cleaned.

The contact angle via the sessile drop technique was measured using a goniometer (Drop Shape analysis, model DSA 30S, Kruss GmbH, Hamburg, HH, Germany), which was connected to a computer with dedicated software (DSA3, V1 .0.3-08, Kruss GmbH) to assess the contact angles. At room temperature (±24°C), one drop (11 µl) of distilled water was placed at the center of the untreated and treated ceramic surfaces (Table 1) using a needle. The contact angle was measured after 5 s.

Microshear Bond Strength Test

Preparation of the samples: Eighty (N=80) ceramic blocks (12×7×2 mm^3) were prepared as aforementioned. The blocks were embedded in plastic rings with self-curing acrylic resin (JET Clássico; Campo Lindo Paulista, SP, Brazil). Then, all the samples were cleaned in an ultrasonic device (Vitasonic, Vita Zanhfabrik) with isopropyl alcohol for 10 min.

Study design (ceramic surface conditioning): The ceramic blocks were randomly assigned (www.randomizer.org) to 8 study groups (10 blocks per group), according to the hydrofluoric acid concentration, in 4 levels, namely 1% (HF1), 3% (HF3), 5% (HF5), and 10% (HF10), and storage at 2 sublevels (baseline and aging condition) (Table 1). Microshear bond strength tests did not have a control group (without hydrofluoric acid etching). The experimental unit was the resin cement cylinder.

The etching procedures were made as aforementioned for contact angle analysis (etching for 20 s, rinsing with air-water spray for 30 s, and drying for 30 s). The blocks were then cleaned in an ultrasonic bath with distilled water (5 min) to remove debris and precipitates; the bonding surface was air-dried and the silane coupling agent (Monobond Plus, Ivoclar Vivadent) was applied actively for 15 s on the surfaces, and kept reacting for more 45 s, as recommended by the manufacturer.

Cementation: Four starch tubes (0.96 mm internal diameter; 1 mm high; Renata, Pastificio Selmi, Londrina; PR, Brazil) (11) were placed over each bonding surface (n=40). The tubes were fixed with sticky wax (Lysanda, Sao Paulo, SP, Brazil), and the dual cure resin cement (Multilink, Ivoclar Vivadent) was applied inside the tubes and photo-cured (Radii-cal, SDI, Bayswater, WA, Australia) for 40 s. Then, the samples were stored in distilled water at 37 °C for 24 h. After this period, the starch tubes were carefully removed, and the specimens were analyzed using a stereomicroscope (Discovery V20, Carl-Zeiss, Gottingen, NI, Germany) at 40× magnification for examination of the margin of the adhesive zone. Specimens with gaps, bubble inclusions or other defects at the margin were discarded before testing.

Storage and microshear tests: Samples from baseline condition were tested in shear (24 h after cementation), while samples under aging condition were stored for 150 days in distilled water at 37 °C and thermocycled (12,000 cycles; 5 °C – 55 °C; dwelling time: 30 s; transfer time: 2 s) (Nova Etica, Vargem Grande do Sul, SP, Brazil).

For microshear testing, the samples were placed in a jig attached to a universal testing machine (EMIC DL-2000, EMIC, Sao Jose dos Pinhais, PR, Brazil). A thin stainless-steel wire loop (Ø= 0.20 mm) was placed as close as possible to the ceramic surface for contact with the lower half-circle of the cylinder. The load was applied (load cell 0.1 KN) at a crosshead speed of 0.5 mm/min until fracture occurred. Care was taken to keep the resin cement cylinder in line with the center of the load cell and the wire loop parallel to the load cell's movement direction and to the bonding interface. The bond strength (R in MPa) was calculated by equation: R = F/A; where "F" is the load required for failure of the specimen (N), and "A" is the interface area of the specimen (mm^2). The bonded area (A in mm^2) was equal for all samples and was obtained by A= π*$(r)^2$, where π= 3.1416 and r= 0.48 mm; i.e., A= 3.1416*$(0.48)^2$ = 0.72 mm^2.

Fracture Pattern

The fracture pattern was determined under a stereomicroscope (Discovery V20, Carl-Zeiss), and classified into two types: 1) predominantly adhesive failure at the interfacial region between the resin cement and ceramic (ADHES); 2) cohesive failure at the cement (COHES-cem).

Micromorphological Analysis

In order to observe the surface alterations of the etched ceramic using different HF concentrations, specimens without hydrofluoric acid etching and etched with all tested HF concentrations were evaluated under field-emission scanning electron microscopy (FE-SEM) (FEI Inspect F50, FEI, Hillsboro, OR, USA) at distinct magnifications. For these analyzes, the specimens were sputter-coated with gold-palladium.

Roughness Analysis by Atomic Force Microscopy (AFM)

Surface roughness measurements (10 µm × 10 µm) were obtained in four specimens per group (Table 1; control group and etched with 1, 3, 5 and 10% hydrofluoric acid), using a non-contact mode and PPP-NCL probes (Nanosensors, force constant = 48 N/m) mode of the AFM device (Agilent 5500 Equipment, Agilent Technologies, Santa Clara, CA, USA). When using the AFM in non-contact mode, surface features are observed at a nanoscale level and any detected roughness is shown as small grains or particles. The use of this method has many advantages; the most important being the ability to collect 3D surface analysis and phase type of data, as well as the numeric data of surface properties. The numeric surface roughness parameters evaluated were average surface roughness (Sa) and the average distance among the 5 highest peaks and the 5 major valleys (Sz) values. AFM micrographs were analyzed using scanning probe microscopy data analysis software (GwyddionT" version 2.33, GNU, Free Software Foundation, Boston, MA, USA). In addition, topographical images were also collected.

Statistical Analysis

A resin cement cylinder was used as the experimental unit for the bond data analysis. Cohesive failures were excluded from the statistical analysis, since those failures did not represent the real bond strength. All the data were presented as mean and standard deviation (SD) values. A normal distribution was assumed after the Shapiro-Wilk test for bond strength data.

Consequently, the two-way ANOVA and Bonferroni tests (α=.05) were performed to compare results from baseline and aging conditions and to compare data among the different groups. One-way ANOVA and the post-hoc Tukey's test were used to evaluate the contact angle and AFM roughness data (α=.05). All statistical analyses were performed by Software Stata 14.2 (Stata Corp, College Station, TX, USA)

RESULTS

Contact Angle

The non-etched group (CTRL) achieved the highest contact angles, followed by the HF1 group. The lowest contact angle values were HF3, HF5, and HF10, which were statistically similar (Table 2). Figure 1 presents representative contact angle images for each surface treatment.

Microshear Bond Strength

The total number of tested samples in baseline and aged groups, and the number/percentages of specimens that failed during the aging process (pre-test failures) are listed in Table 2. The HF1 group had the highest percentage of pre-test failure. To provide a fair comparative evaluation among the tested groups, the specimens that failed prior to testing were considered in the statistical analysis. For this purpose, the minimum value of µSBS obtained in each group was assigned to each prematurely debonded specimen (12).

Two-way ANOVA revealed that HF concentration ($p<0.0001$), aging ($p<0.0001$), and interaction ($p<0.0001$) had a statistically significant effect on the bond results. For the baseline condition, the HF3, HF5, and HF10 groups were statistically similar. Bonferroni's test showed that HF5 was significantly higher than HF1 ($p=.012$); however, HF1 was statistically similar to HF3 and HF10. After aging, HF3, HF5, and HF10 were statistically similar and had the highest bond results, while HF1 had the lowest ($p=.0001$) (Table 2).

In terms of bond stability, the storage/thermo-cycling had a negative effect for all HF acid concentrations: the bond values reduced after aging, compared to their counterpart baseline conditions (Table 2).

Fracture Pattern

Even with a different number of tested specimens, all groups presented similar percentiles of adhesive and cohesive (in cement) failures (Table 3).

Micromorphological Analysis

FE-SEM analyses (Figure 2) showed slight topographical changes in the lithium disilicate surface etched with HF1 acid, when compared to the non-etched condition (CTRL). HF acid at

3% concentration was able to produce slight topographical changes, but as expected, higher HF acid concentrations (5 and 10% hydrofluoric acid) created more irregular and porous surfaces by a stronger removal of the glassy matrix and with crystals pulling out from the surface.

Roughness analysis by AFM

HF acid in concentrations of 5% and 10% promoted rougher surfaces for *Sa* and *Sz* parameters. 1% HF acid was not able to change the surface when compared to with the non-etched surface (CTRL) (Table 2; Figure 3).

DISCUSSION

The first hypothesis of the present study was partially accepted, since different HF acid concentrations significantly influenced the contact angle values (Table 2; Figure 1), and bond strength, but 1% and 3% hydrofluoric acid were not able to promote changes on parameters evaluated for roughness. All groups presented a statistically significant decrease in bond strength values after aging, meaning that the second hypothesis was accepted. Considering the bond data after aging, HF acid at 3%, 5%, and 10% concentrations promoted higher values of microshear bond strength, when compared with 1% HF acid, which had a high percentage of pre-test failures during aging (90%), evidencing the weak and unstable resin bond by etching with 1% HF acid, probably by slight (limited) ceramic surface alterations and absence of micro-mechanical bond.

It is well known that micro-morphological alterations of the ceramic surfaces promote a better bond strength (5). HF acid is a modifier and an etching agent indicated for ceramics that contain silica (13) to dissolve the glassy phase, exposing the crystals and resulting in micro- and nano-morphological changes in the ceramic structure, which promotes interlocking effects (mechanical bond) (7,8,14). This modification provides increased surface area, improving bonding quality, and a better contact between the ceramic material and resin cement (14). On the other hand, these surface alterations might be also related to a weakening effect on flexural strength, owing to the introduction of new defects on the surface, being crack initiators for fracture (6,8). As the defect population (shape and size) is a predictor for failure of brittle materials (such as glass ceramics) (15), HF acid might play an important role in ceramic failure, since this acid promotes surface alterations of the intaglio surface of restorations, depending on their concentration and etching time (6-8). Thus, the optimal concentration should be one promoting surface alterations without weakening the ceramic.

The scientific community has been finding another option to promote topographic changes on glass-ceramic surfaces owing to HF acid toxicity (10). According to Carpena and

Ballarin (16), the use of HF acid by clinicians is banned in some countries, and the dental laboratory is responsible for applying HF acid as per the ceramic manufacturer's instructions (17). Besides, a recent *ex vivo* study showed its hazardous potential, even with concentrations of <3% HF acid (10). In this sense, some studies have tested different ceramic surface treatments as alternative ones to HF acid, however, these alternative surface treatments were not preferable to HF etching associated with silane application (17), or were poorly supported by the literature, thus requiring more studies. In the present study, HF acid in different concentrations (3%, 5%, and 10%) was able to promote surface changes, but even for the groups with higher matrix removal and changes in the ceramic surface, after 150 days of storage and thermocycling, there was a significant decrease in bond strength data.

Several factors interact in the process of establishing a strong bond between two different materials. Even with rougher surfaces to micromechanical interlocking, the chemical bond can be deteriorated by hydrolysis of siloxane bonds over time, since the resins are permeable to water (18). It is related to a fast increase in the amount of water absorbed by the composite materials causing degradation of the silane, damaging adhesion between the tested materials. Water storage and thermocycling are described as detrimental to the silane-ceramic bond (19). Also, silanized interfaces appear to be unstable in humid conditions and the silane bond was found to deteriorate in moisture. However, *in vitro* studies either lack the aging conditions or employ short-term water storage and/or thermocycling (20).

The results of the present study demonstrated that thermocycling reduced adhesive resistance. The pre-test failures were observed in all the tested groups, even for groups with rougher ceramic surfaces. It can be attributed to the hydrolytical deterioration of siloxane bonds in the storage period (21). In this sense, the group conditioned by 1% hydrofluoric acid lost 36 specimens (90%), demonstrating that weak adhesion takes place when slight topographic alterations of the surface ceramic occur, even with a primer containing silane and functional monomers as MDP (10-methacryloyloxydecyl dihydrogen phosphate). The negative effect of thermal cycling on adhesion can be explained by the fact that materials with different lineal thermal expansion coefficient (LTEC) agent also presented different degrees of shrinkage and expansion – this process promotes the fatigue phenomenon of the materials, leading to rupture of the bond and interface (22). Another factor that might have contributed to the bond decrease is the small dimension of the bonded area receiving larger influence of the thermocycling effects on its surface (23). Shono et al. (24) verified that adhesive joints in samples with different dimensions presented reductions of bond strength in samples with small areas, after similar periods of storage and thermocycling.

An increase in surface area caused by HF acid etching induces an increase in wettability, which is associated with a lower contact angle and greater bonding potential (17). The differences showed in the contact angle measurements are explained by the higher roughness (seen by AFM: Table 2; Figure 2), obtained in groups conditioned with HF acid at concentrations of 3%, 5%, and 10%, and by the higher and similar bond strength values. The subsequent application of a silane coupling agent after HF acid etching enhances the bonding potential of resin cement to lithium disilicate, in agreement with other findings (2,4,5).

HF acid etching produced micro porosities, grooves, and striations (Figure 2) by partially dissolving the glassy phase, and higher HF acid concentrations were able to not only promote the loss of the glassy phase, but also areas with grain pullout (5 and 10% HF acid). It resulted in a different surface pattern, with an increase in the size of the pores and its quantity, and elongated grooves, characteristic of this ceramic material after HF acid etching. Consequently, for the tested lithium disilicate ceramic, etching with HF at concentrations of 5% and 10% produced higher values of roughness and increased surface modifications, as seen in FEG images, and also verified by Ramakrishnaiah et al. (7).

As limitations of this current study, it can be displayed the absence of simulating intermittent clinical loading forces and environmental issues, which occur on teeth/restorations under clinical service. Also, microshear/shear bond strength tests widely used to analyze the bond to lithium disilicate ceramics (5,25) apply a monotonic load and do not cause only shear stress on the tested specimens, thus, fracture analysis is imperative for data interpretation. From this viewpoint, we excluded cohesive failures from the statistical analysis. In addition, our study did not simulate a CAD/CAM machined ceramic surface, but it can be considered as a positive condition, since the isolate effect of HF acid concentrations on adhesion could be fairly assessed.

Within the limitations of this *in vitro* study, it can be concluded that hydrofluoric acid at 3%, 5%, and 10% concentrations promoted higher resin bond to lithium disilicate ceramic after aging. The micromorphological changes promoted by etching with these acids play a crucial role in micro-mechanical bond improvements, and they can be used to conditioning lithium disilicate based glass-ceramic. Hydrofluoric acid at 3% should be considered with caution, since it promoted slight topographical changes on the ceramic surface, and also had lower roughness values than 5 and 10% concentrations. Hydrofluoric acid at a 1% concentration led to a very weak bond after aging.

SUMMARY IN PORTUGUESE

Este estudo avaliou o efeito de diferentes concentrações de ácido fluorídrico (HF) na resistência de união entre uma cerâmica vítrea à base de dissilicato de lítio e um cimento resinoso. Blocos cerâmicos (12×7×2 mm) de IPS e.Max CAD (Ivoclar Vivadent) foram produzidos e distribuídos aleatoriamente em 8 grupos (N=80), considerando 2 fatores de estudo: concentração de HF em 4 níveis, isto é, 1% (HF1), 3% (HF3), 5% (HF5), e 10% (HF10), e armazenamento em 2 subníveis, isto é, controle (testes foram realizados 24 h após a cimentação), e envelhecidos (150 dias de armazenamento + 12.000 ciclos térmicos a 5°C e 55°C). Condicionamento ácido (20 s) foi realizado, seguido por lavagem, secagem e silanização. Quatro cilindros de cimento resinoso (Ø= 0.96 mm) foram construídos a partir de matrizes de amido em cada amostra cerâmica (n= 40). Amostras cerâmicas adicionais foram condicionadas e analisadas quanto ao ângulo de contato, micro-morfologia e rugosidade. Na condição inicial (sem envelhecimento), os grupos HF3, HF5, e HF10 mostraram valores de resistência de união similares (13.9 – 15.9 MPa), e HF1 apresentou valores menores que HF5, sendo estatisticamente diferente (p= 0.012). Após o envelhecimento, todas as médias de resistência de união diminuíram estatisticamente, sendo que HF3, HF5 e HF10 foram similares e maiores que HF1 (p= 0.0001). Para o ângulo de contato, HF3, HF5 e HF10 apresentaram valores similares (7.8 – 10.4°), menores que os grupos HF1 e CTRL. HF5 e HF10 apresentaram superfícies mais rugosas que as outras condições. Para melhores resultados de resistência de união, a cerâmica testada pode ser condicionada com ácido fluorídrico nas concentrações de 3%, 5% e 10%.

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TABLES

Table 1 Experimental design.

	Surface conditioning for contact angle	Surface conditioning for roughness	Surface conditioning for µSBS	Storage condition*
CTRL	Non-etched control		-	-
				-
HF1	Etching with hydrofluoric acid 1%**			Without
				With
HF3	Etching with hydrofluoric acid 3%**			Without
				With
HF5	Etching with hydrofluoric acid 5%**			Without
				With
HF10	Etching with hydrofluoric acid 10%***			Without
				With

*Storage (37°C for 150 days) and thermocycling (12,000 cycles; 5°C and 55°C).
**Experimentally formulated, FGM.
***Condac Porcelana 10%, FGM; Santa Catarina, Brazil.

Table 2 Means and standard deviation of bond strength data (MPa) as a function of storage condition, total number of tested samples in conditions without and with aging, total number of pre-tested failures (PTF) during thermocycling, contact angle and roughness data (*Sa* and *Sz* parameters – in nm).

	Mean bond strength (MPa)		Total tested samples without aging	Total tested samples with aging	Total number (%) of PTF during TC	Contact angle	Roughness	
	Without aging*	With aging*					*Sa* (nm)	*Sz* (nm)
CTRL	-	-	-	-	-	28.4 ± 1.5^{A}	9.2 ± 3.2^{C}	134.5 ± 25.1^{B}
HF1	11.2 ± 4.5^{Ba}	1.8 ± 2.9^{Bb}	40**	4	36 (90.0%)	15.9 ± 2.5^{B}	24.9 ± 3.1^{BC}	205.3 ± 29.6^{B}
HF3	13.9 ± 3.9^{ABa}	7.8 ± 6.1^{Ab}	38**	25	15 (37.5%)	7.8 ± 0.4^{C}	30.4 ± 2.4^{B}	275.0 ± 43.3^{B}
HF5	15.9 ± 2.9^{Aa}	11.0 ± 7.5^{Ab}	39**	33	7 (17.5%)	8.3 ± 2.9^{C}	56.6 ± 18.2^{A}	563.8 ± 185.5^{A}
HF10	14.5 ± 5.2^{ABa}	9.8 ± 7.5^{Ab}	37**	27	13 (32.5%)	10.4 ± 2.1^{C}	52.5 ± 2.4^{A}	469.8 ± 25.4^{A}

*Storage (37°C for 150 days) and thermocycling (12,000 cycles; 5°C and 55°C).

Means in the same column with the same capital letter are statistically similar.
Different lowercase letters mean statistical difference between storage conditions.
**Forty (40) resin cement cylinders were produced for each condition; for baseline groups, specimens with bubbles and defects at the interface were not tested; for aged groups, all the specimens were checked before the storage period, being the non-tested samples all pre-test failures (during thermocycling).

Table 3 Types of failure evaluated after the bond strength tests.

	Type of failure	
	Adhes	Cohes-cem
HF1	34 (77.3%)	10 (22.7%)
HF3	58 (92.1%)	5 (7.9%)
HF5	63 (87.5%)	9 (12.5%)
HF10	53 (82.8%)	11 (17.2%)
Total	266 (85.3%)	46 (14.7%)

FIGURES

Figure 1 Images and means ± standard deviations (in degrees) of contact angle measurements of surfaces subjected to the following conditions: non-etched (CTRL); etched for 20 s with 1%, 3%, 5%, and 10% hydrofluoric acid. The same superscript letters indicate no significant differences (Tukey's test; α=0.05).

Control (non-etched) 28.4 ± 1.5^{A}	1% hydrofluoric acid 15.9 ± 2.5^{B}	3% hydrofluoric acid 7.8 ± 0.4^{C}	5% hydrofluoric acid 8.3 ± 2.9^{C}	10% hydrofluoric acid 10.4 ± 2.1^{C}
				

Figure 2 Representative FEG images of different ceramic surface conditioning: non-etched; etched for 20 s with 1% HF; 3% HF; 5% HF; and 10% HF.

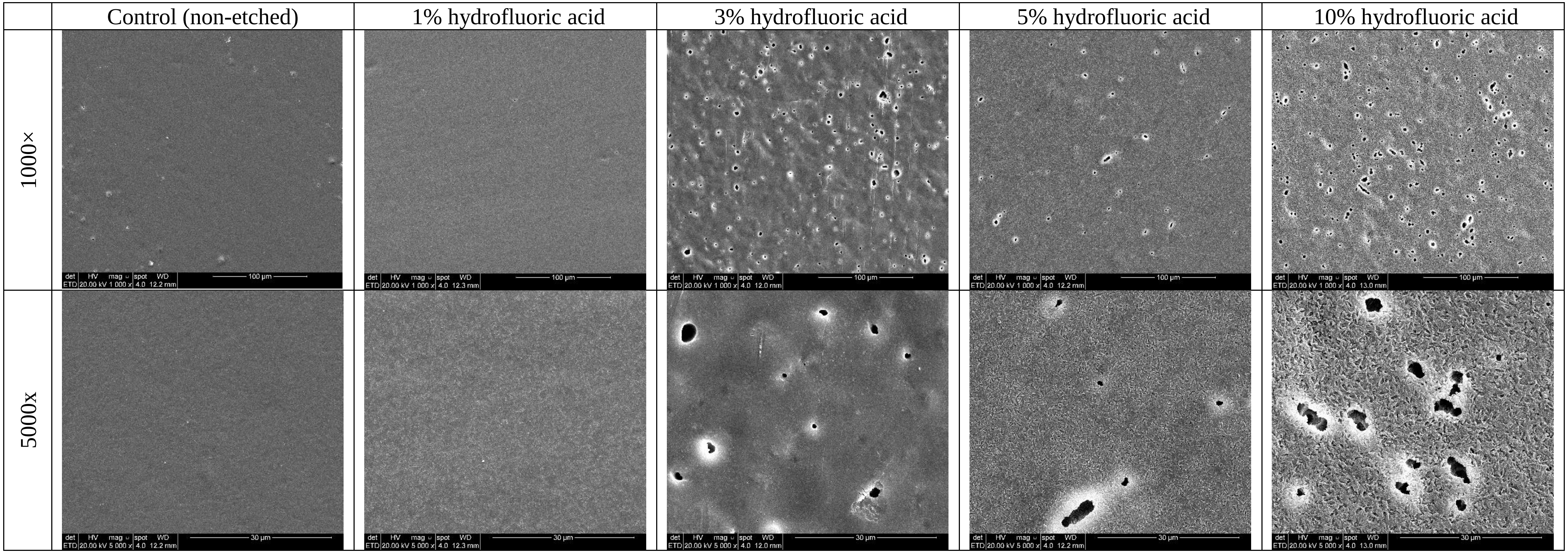
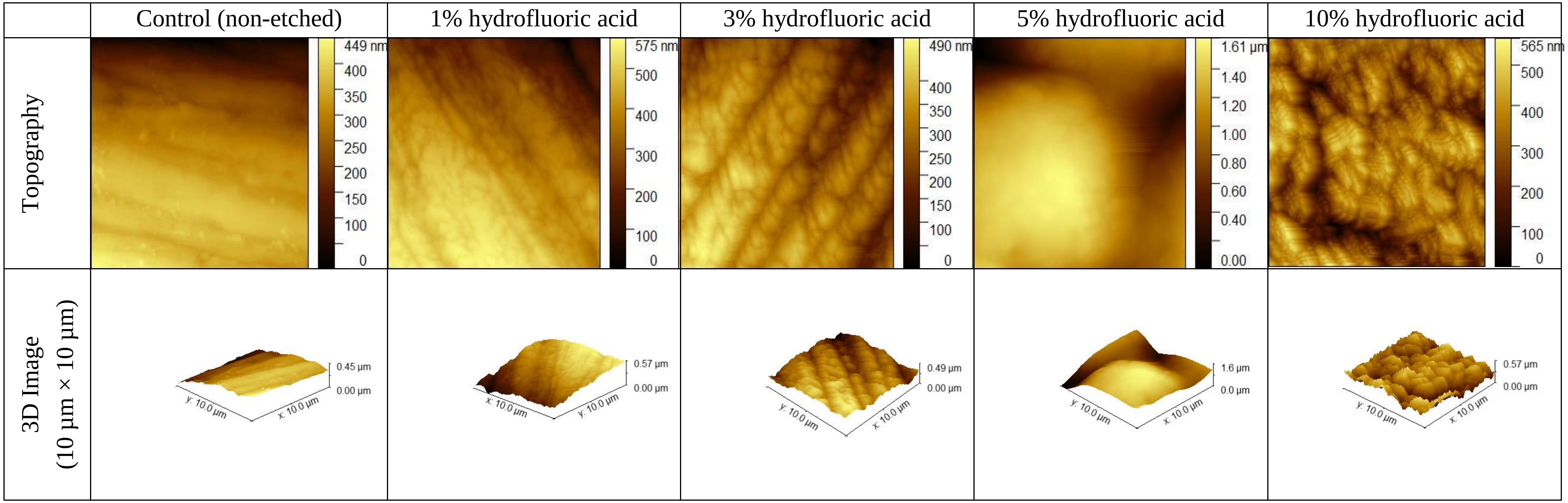


Figure 3 Representative topographic and 3D images of atomic force microscopy (AFM).



3. ARTIGO 2 - HOW DOES HYDROFLUORIC ACID ETCHING AFFECT THE CYCLIC LOAD-TO-FAILURE OF LITHIUM DISILICATE RESTORATIONS?

Este artigo está publicado no periódico *Journal of Mechanical Behavior of Biomedical Materials*, ISSN 1751-6161, fator de impacto de 3.239, Qualis CAPES A2. As normas para publicação estão descritas no Anexo B.

How does hydrofluoric acid etching affect the cyclic load-to-failure of lithium disilicate restorations?

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HIGHLIGHTS

- The effect of HF acid on fatigue of adhesively cemented simplified lithium disilicate ceramic restorations was evaluated.
- The higher the HF acid concentration, the more pronounced the topographical changes.
- Intermediate HF acid concentrations (3 and 5%) depicted the best load-bearing capability under fatigue.

ABSTRACT

This study investigated the effect of etching with distinct hydrofluoric (HF) acid concentrations on the cyclic load-to-failure (C_{Lf}) of simplified lithium disilicate glass-ceramic restorations adhesively cemented to a dentin analogue material. Eighty pairs of dentin analogue (G10; Ø= 10 mm; thickness= 2.0 mm) and lithium disilicate discs (IPS e.Max CAD; Ø= 10 mm; thickness= 1.5 mm) were produced. For luting, the dentin analogue discs were etched (10% HF for 60 s) and received a primer coating. The inner surface of the ceramic discs was treated as follows (n= 20): non-etched/control (CTRL) or etched for 20 s with HF acid at 3% (HF3), 5% (HF5), or 10% (HF10). A silane coating was then applied onto the ceramic surfaces. Fatigue tests followed the staircase approach (initial load= 720 N; step-size= 70 N; 500,000 cycles per sample; 20 Hz) using a hemispheric stainless-steel piston (Ø= 40 mm) under water. The C_{Lf} data were analyzed using Dixon and Mood method. Topographic and fractographic analyses were conducted. C_{Lf} (in N) of HF3 (1355 ± 32.0) and HF5 (1335 ± 58.8) groups were the highest and statistically similar; HF10 presented intermediate C_{Lf} (1175 ± 132.9), while the non-etched group had the lowest one (965 ± 145.0). Topographical analysis showed that the higher the HF acid concentration, the more pronounced the topographical changes. All failures (radial cracks) started from the inner surface of the ceramic discs. Topographical changes promoted by intermediate HF acid concentrations (3 and 5%) may improve fatigue performance for adhesively-cemented lithium disilicate restorations.

Keywords: Accelerated fatigue. Etching. Fatigue. Hydrofluoric acid. Lithium disilicate. Staircase. Surface conditioning. Vitreous ceramic.

1. Introduction

Among the various dental ceramics available to produce monolithic restorations, lithium disilicate presents favorable aesthetic results combined with a higher strength than other vitreous ceramics (Barizon et al., 2014). A recent systematic review has shown that adhesively-cemented lithium disilicate single crowns present excellent survival rates of 96.7% up to 10 years (Pieger et al., 2014). In order to obtain better predictability of long-term success for those restorations, adhesive cementation is required (Valenti, Valenti, 2009).

Etching the intaglio surface of lithium disilicate restorations with HF acid is commonly recommended for enhanced adhesion (Klosa et al., 2009; Colares et al., 2013). HF acid selectively dissolves the silica matrix, exposing numerous elongated or bean-like crystals at the lithium disilicate surface. (Belli et al., 2017; Prochnow et al., 2017). In response to that, it increases the roughness and wettability, enabling mechanical interlocking and chemical reactivity which will ensure long-term bond between luting composites and lithium disilicate (Sundfeld et al., 2018; Prochnow et al., 2018).

The manufacturer recommendation is to etch the lithium disilicate with 5% hydrofluoric acid for 20 s (Ivoclar Vivadent). Despite that, some researches has been conducted attempting to reduce such concentration without altering the performance, as hydrofluoric acid toxicity is concentration-based (Ozcan et al., 2012). In this sense, Sundfeld and collaborators (2018) have found similar bond strength results between lithium disilicate and two different resin cements when HF etching is executed with 1% and 5% concentrations. Also, another study has not found statistical differences in the bond strength between resin cement and the same ceramic aforementioned, comparing 3 and 5% HF concentrations after thermocycling and a storage period of 150 days (Prochnow et al., 2018).

Besides that, the HF acid etching effect on the mechanical integrity of lithium disilicate glass-ceramics remains a controversial topic. Zogheib et al. (2011) reported a decrease in flexural strength when HF acid was applied in higher concentrations and for long periods of time. However, Menees et al. (2014) and Prochnow et al. (2017) found that the mechanical strength of etched and non-etched lithium disilicate surfaces was equal. Meanwhile, knowing that HF acid etching increases roughness by increasing the defects population; filling these defects with a resin cement could increase the strength of ceramic restorations, thus homogeneously transmitting the stress to the support material (May et al., 2012; Posritong et al., 2013; Venturini et al., 2018a). On the other hand, unfilled defects may potentiate slow-crack growth mechanisms impairing the materials’ mechanical performance (Anusavice, Hojitatie, 1992).

In this context, studies report that failures in cemented glass-ceramics restorations start from the intaglio surface (i.e., cementation surface), on which high tensile stresses are seen during load applications, and where the HF acid etching is performed (Kelly et al., 1999; Kelly et al., 2010; Gressler May et al., 2015; Monteiro et al., 2018a; Venturini et al., 2018a). In this sense, higher HF acid concentrations could compromise the final strength of the restorations. Still, up to now, there is no consensus regarding the actual and isolated influence of HF etching on the lithium disilicate surface and its inherent consequences on the fracture mechanical; also well-designed studies that consider adhesive resin cementation and the elastic modulus of substrate as study variables are still needed to clarify this clinically relevant issue.

Thus, it becomes relevant to evaluate the influence of different HF acid concentrations (3, 5, and 10%) on the cyclic load-to-failure of a lithium disilicate glass-ceramic adhesively cemented to a dentin analogue material. The null hypotheses tested were: (1) the distinct HF acid concentrations would not influence the fatigue behavior of lithium disilicate ceramic restorations, and (2) non-etched lithium disilicate ceramic restorations will perform similarly to HF etched ones.

2. Materials and Methods

2.1 Experimental groups and specimens' preparation

The test assembly employed in the present study has been widely used in the literature (Monteiro et al., 2018a; Monteiro et al., 2018b; Venturini et al., 2018b; Scherer et al., 2018). It consisted of a simplified occlusal restoration for a posterior tooth with a final diameter of 10 mm (average dimension of molars; Ferrario et al., 1999) and a final thickness of 3.5 mm (taking into consideration the ceramic and epoxy resin thickness). According to Sulieman et al. (2005), this is the average thickness equivalent from the roof of the pulp chamber to the occlusal teeth surface.

2.1.1 Dentin analogue discs

G10 (fiberglass mesh embedded in an epoxy resin; NEMA class G10, Accurate Plastics, Inc., New York, NY, USA) sticks were transformed into cylinders of 10 mm in diameter with SiC grit papers (#400) in a polishing machine (Ecomet/Automet 250, Buehler, Lake Bluff, IL, USA). The cylinders were cut under water-cooling (Isomet 1000, Buehler) into 2.0-mm thick slices. After cutting, the discs were polished (SiC paper; #600 grit) and cleaned in an ultrasonic bath with distilled water for 5 min.

2.1.2 Ceramic discs

Blocks of a lithium disilicate glass-ceramic for CAD/CAM systems (IPS e.Max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were transformed into cylinders using the technique described by Scherer et al. 2018. The cylinders were cut (IsoMet 1000, Buehler) under water-cooling in 1.5-mm thick slices. The occlusal surface of ceramic discs was wet-polished (SiC paper; #600-

, #1200-grit); meanwhile, their intaglio surface was kept as-cut. After crystallization, the discs were cleaned in an ultrasonic bath (isopropyl alcohol – 5 min).

2.2 Surface treatments and cementation procedure

The ceramic discs were randomly allocated (www.randomizer.org) according to the surface treatment (n=20): kept as-cut (CTRL), or etched for 20 s with experimentally formulated HF acid (FGM, Joinvile, SC, Brazil) at 3% (HF3), 5% (HF5), or 10% (HF10). The ceramic discs were then cleaned, submitted to an ultrasonic bath (distilled water for 5 min), and a silane agent (Monobond Plus, Ivoclar Vivadent) was applied on the ceramic surfaces.

The cementation surface of the dentin analogue material discs was treated with 10% HF acid (Condac Porcelana, FGM) for 1 min, followed by air-water washing (30 s) and ultrasonic cleaning (distilled water for 5 min). Then, a mixture of A and B primers (ratio 1:1; Multilink Automix, Ivoclar Vivadent) was scrubbed onto the surfaces for 30 s and air-dried until a thin layer was obtained.

A dual-cure resin cement (Multilink Automix, Ivoclar Vivadent) was manipulated and applied onto the intaglio surface of the dentin analogue discs. Each ceramic disc was placed over its dentin analogue pair, and a load (2.5 N) was applied on the occlusal ceramic surface for 10 min. The resin cement excesses were removed after seatment of the discs. The assemblies were then light-cured (Radii-cal LED curing light, SDI, Bayswater, Australia) for 20 s around the set (0º, 90º, 180º, 270º) and in the occlusal surface after load removal. All the samples were stored for 7 to 14 days in distilled water (37ºC) until the fatigue tests were carried out.

2.3 Cyclic load-to-failure tests (Staircase Method) and data analysis

The parameters for the cyclic load-to-failure tests (initial load= 720 N; step-size= 70 N) followed the data from the monotonic test described by Prochnow et al., 2018b, using the same ceramic material and loading assembly. For the fatigue tests (staircase method) (Instron ElectroPuls E3000, Instron Corporation, Norwood, MA, USA), the cemented specimens were loaded by a hemispherical stainless-steel piston 40 mm in diameter (Anami et al., 2016; Monteiro et al., 2018a; Monteiro et al., 2018b; Scherer et al., 2018). The piston was positioned in the center of the occlusal ceramic surface stabilized in a flat steel base under water. An attached cylindrical metal ring was used to control specimen positioning, ensuring the load application occurred in the center of the specimens (Monteiro et al., 2018a; Monteiro et al., 2018b). Before testing, an adhesive tape (110 µm) was fixed on the occlusal side of each specimen to improve contact with the piston and to prevent contact surface damage, which could result in cracks from surface damage, i.e., Hertzian cone cracks (Wachtman et al., 1972).

An additional thin sheet of a non-rigid material (cellophane, 2.50 µm) was placed between the piston and the specimen to enhance stress distribution (Venturini et al., 2018b).

Cyclic loads (500,000 cycles at 20 Hz) were applied with amplitudes ranging from a minimum of 10 N (to maintain the piston in contact with the specimen) to the maximum load for every cycle using the staircase method described by Collins (1993). For each group, the first specimen was tested at the initial load level selected (720 N) until it either failed or runout at predetermined cycles. A step size load (70 N) was applied up or down to the next specimen, according to survival or failure of the previous specimen. This procedure was repeated until at least 15 samples per group were obtained after the stair reversal (minimum number of samples necessary to obtain a precise estimation using this methodology; Collins, 1993).

After the cyclic load-to-failure tests, the mean cyclic load-to-failure (C_{Lf}), standard deviation (SD), and 95 % confidence interval (CI) were obtained using the Dixon and Mood method (1948), which involves the maximum-likelihood estimation (overlapping of confidence intervals) and assumes that data follows a normal distribution (Collins, 1993). This procedure, established by Collins (1993) was well-described previously (Pereira et al., 2016; Monteiro et al., 2018a; Monteiro et al., 2018b; Prochnow et al., 2018b), and takes into consideration the least frequent event (survival or failure) that occurred during the fatigue tests.

2.4 Topographic analysis

Additional ceramic discs were produced for each tested group, gold-coated and analyzed by secondary electron (SE) detector at 30.00 kV (Scanning Electron Microscopy - SEM; VEGA3, Tescan; Czech Republic) under different magnifications.

2.5 Fractographic analysis

After testing, the specimens were submitted to oblique light transmission to visually inspect for initial cracks. The samples were cut (Isomet 1000, Buehler) perpendicularly to the failure found and analyzed under a stereomicroscope (Discovery V20, Carl Zeiss, Gottingen, Germany). Two representative samples of each group were randomly selected for further SEM analyses (VEGA3, Tescan).

3 RESULTS

3.1 Cyclic load-to-failure tests (Staircase Method)

Cyclic load-to-failure results are displayed in Table 1. The patterns of runouts (survivals) and failures for each group are presented in Figure 1.

The CTRL group presented the lowest mean cyclic load-to-failure (965 N). Etching with 10% HF acid promoted intermediate values of cyclic load-to-failure (1175 N), while at 3%

(1355 N) and 5% (1335 N) produced the highest load bearing ability for the tested lithium disilicate ceramic.

3.2 Topographic analysis

Figure 2 shows SEM images of the ceramic surfaces. The non-etched (control) ceramic surface was homogeneous and smooth. Meanwhile, HF acid etching at distinct concentrations led to a well-known ceramic surface pattern. The observed topographic changes are related to HF acid concentration: the higher the HF acid concentration, the greater the glassy-matrix dissolution and the pulling out of lithium disilicate grains. In this sense, voids can be observed in SEM images from higher HF acid concentrations tested (5 and 10%), that were not observed when etching the lithium disilicate surface with 3% HF acid neither in the control group.

3.3 Fractographic analysis

The analysis of the failed discs has provided evidence that failures do not involved surface damage or wear. In the same way as proposed by Kelly et al. (1999) evaluating failed clinically ceramic crowns, all failures started as radial cracks from the cementation surface, it means, from defects provided by hydrofluoric acid etching at different concentrations or bubbles into the resin cement that act as crack initiators (Figure 3). It can be observed that the cracks propagated in a perpendicular direction to the main tensile stress point, and the cracks started from a superficial defect located on the cementation surface (tensile side of the ceramic discs in the tested assembly).

4 DISCUSSION

The present study evaluated the influence of different HF acid concentrations on the cyclic load-to-failure of a lithium disilicate glass-ceramic adhesively cemented to a dentin analogue material. The first null hypothesis was partially accepted, since 3% and 5% HF acid etching presented similar cyclic loads-to-failure values; meanwhile, etching with 10% HF presented a lower cyclic load-load-failure value. The non-etched group had the lowest cyclic fatigue value, rejecting the second hypothesis.

Adhesive cementation is required for long-term success of glass-ceramic restorations, since it assists in transmitting loads through the supporting substrate (Addison, Fleming, 2004). As a result, HF acid etching is responsible for removal of the glassy matrix, producing not only a rough surface to enable micromechanical interlocking with resin cement, but also increasing the surface area available to adhesion and improving surface wettability (Prochnow et al., 2018a). Exposure of the crystal phase and removal of the glassy matrix are directly related to HF acid concentration and exposure time (Zogheib et al., 2011; Prochnow et al., 2017).

Although HF acid etching promotes desirable topographical changes and increase in resin adhesion when applied to glass-ceramic surfaces, HF acid is a toxic and hazardous chemical reagent (Ozcan et al., 2012). The use of HF acid has been related to necrosis of the skin and bones; eye irritation, among other collateral effects, that are time and concentration dependents (Derelanko et a., 1985; Dennerlein et al., 2016). Also, it is reported that HF acid concentrations and etching times decrease the final strength of glass-ceramics (Hooshmand et al., 2008; Zogheib et al., 2011; Venturini et al., 2015a). Thus, the potential benefits of reduction on HF acid concentration have been explored, aiming to maintain the desired enhanced bonding characteristics to resin under the lowest possible acid concentration (Venturini et al., 2015b; Sundfeld Neto et al., 2015). In this way, a few studies have been carried out searching for a new etching protocol that provides the same results in terms of resin bond to glass-ceramics (El-Damanhoury et al., 2017; Siqueira et al., 2016; Wille et al., 2017), even though, HF acid etching followed by a silane coupling agent application remains the gold-standard bonding protocol (Prado et al., 2018).

In the present study, the fatigue behavior of different HF acid concentrations on the lithium disilicate surface was investigated when adhesive cementation was performed. Our data support the absence of micromechanical interlocking being related to a lower load-bearing ability under fatigue (Group CTRL - only silane application). Moreover, use of a high HF acid concentration (10%) promoted lower fatigue failure values than intermediate concentrations (3% and 5%). The result of the present study cannot be extrapolated for other glass-ceramic materials since there is a great amount of glass-ceramics commercially available with different microstructural composition, and by that, they do behave differently when HF acid etched (Ramakrishnaiah et al., 2016; Belli et al., 2017).

Performance under fatigue of glass-ceramic restorations is not only related to the applied surface treatment and its changes on the ceramic intaglio surface, but also with the filling-up of the defects promoted by the surface treatment with resin cement (Venturini et al., 2018a). In the current study, defects without resin cement impregnation were not detected; however, 10% HF acid etching decreased the fatigue failure values compared to 5% and 3% HF etching; it appears that the shape of defects obtained by 10% HF acid etching acted as crack initiators. SEM findings corroborate that higher concentrations promote an extensive removal of the glassy matrix and the pulling out lithium disilicate crystals, as previously reported (Prochnow et al., 2017; Belli et al., 2017).

Among the failure types reported in the literature, radial cracks have been identified as a primary source of premature failure in all-ceramic restorations (Kelly, 1999). According to

Kelly et al. (1996), cracks starting from flaws at the intaglio ceramic surface are especially deleterious, since they can spread easily, thus fracturing the ceramic layer. Wendler et al. (2018) stated that, when loaded, ceramic layers bonded and supported by a less stiff material (i.e., tooth dentin or dentin substitute) develop tensile stresses in the ceramic material at the interface with resin cement, below the load application. When this tensile stress exceeds the strength of the ceramic layer, radial cracks are formed in the internal ceramic surface. Our fractographical findings support that statement, since the outcome found for all the distinct ceramic surface treatments were radial cracks starting from the cementation surface, where HF acid etching was performed.

The adhesive cementation and, consequently, the micromechanical interlocking among ceramic material and resin cement are related to better load-bearing ability of all-ceramic restorations (May et al., 2012), allowing the ceramic material to transmit the received loads throughout the whole system. The absence of this interlocking in the control group (only chemical adhesion) resulted in lower fatigue values than the etched groups; even the final mean cyclic load-to-failure of the control group (965 N) was able to support the maximum loads reported in the oral environment (~ 800 N) (Nishigawa et al., 2001). However, siloxane bonds suffer hydrolytic degradation by water storage and thermocycling (Kern, Thompson, 1995). One could expect that water storage and thermocycling would modify the cement bonding quality and, perhaps negatively influence the fatigue behavior of the control group, facts that were not analyzed in the present study.

There is evidence that the CAD/CAM machining process can reshape the ceramic surface and introduce new flaws, thus importantly changing the critical defect populations (Addison et al., 2012) related to damage of those restorations and decreased ceramic strength (Coldea et al., 2015). Those defects expected by the machining process were not reproduced herein. Besides the absence of fully reproducing the machined surface produced by CAD/CAM technology or pressed ceramic restorations, it can be stated that the application of only axial loads, and the use of simplified ceramic restorations were considered as limitations of the present study. The test frequency selected to perform fatigue tests is higher than the masticatory frequency, but Fraga et al. (2016) stated that fatigue results do not change when using 20 Hz frequency compared to 2 Hz, testing an Y-TZP ceramics. Also, the epoxy resin used as an analogue dentin material did not simulate a condition properly observed clinically, but this dentin-like material has recently been used extensively (Gressler May., 2015; Monteiro et al., 2018a; Monteiro et al., 2018b; Venturini et al., 2018a; Venturini et al., 2018b). Kelly et al. (2010) found bond strength and elastic modulus values to be quite similar to hydrated dentin.

CONCLUSIONS

- The use of intermediate HF acid concentrations (3 and 5%) led to a higher fatigue load bearing capability of adhesively-cemented lithium disilicate ceramic restorations.
- Etching a lithium disilicate glass-ceramic with a high HF acid concentration (10%) creates critical defects, thus reducing the mechanical performance.
- No acid etching promotes weak resin adhesion, thus diminishing fatigue load-to-failure.

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TABLES

Table 1 Group codes, surface treatments and results of cyclic loads-to-failure with their respective confidence intervals (CI) of the simplified lithium disilicate ceramic restorations subject to 500,000 cycles of fatigue at 20 Hz.

Group	**Surface treatment**	**Mean**	**95% CI**
CTRL	Non-etched control (only silane application)	965.0^{C}	872.5 – 1057.5
HF3	3% HF acid* (20 s) + silane application	1355.0^{A}	1332.9 – 1377.1
HF5	5% HF acid* (20 s) + silane application	1335.0^{A}	1294.5 – 1375.5
HF10	10% HF acid* (20 s) + silane application	1175.0^{B}	1080.5 – 1269.5

*Experimentally formulated (FGM Produtos Odontológicos, Joinville, SC, Brazil).
Different superscript letters indicate statistically significant differences based on the interval confidences overlap (Dixon, Mood, 1948)

FIGURES

Figure 1 Staircase sensitive results after 500,000 cycles at 20 Hz. The lines indicate the mean cyclic load-to-failure, the black filled squares represent survived specimens, the empty squares indicate failed specimens, and the red squares are the specimens in which the staircase tests initiated according to Collins (1993).

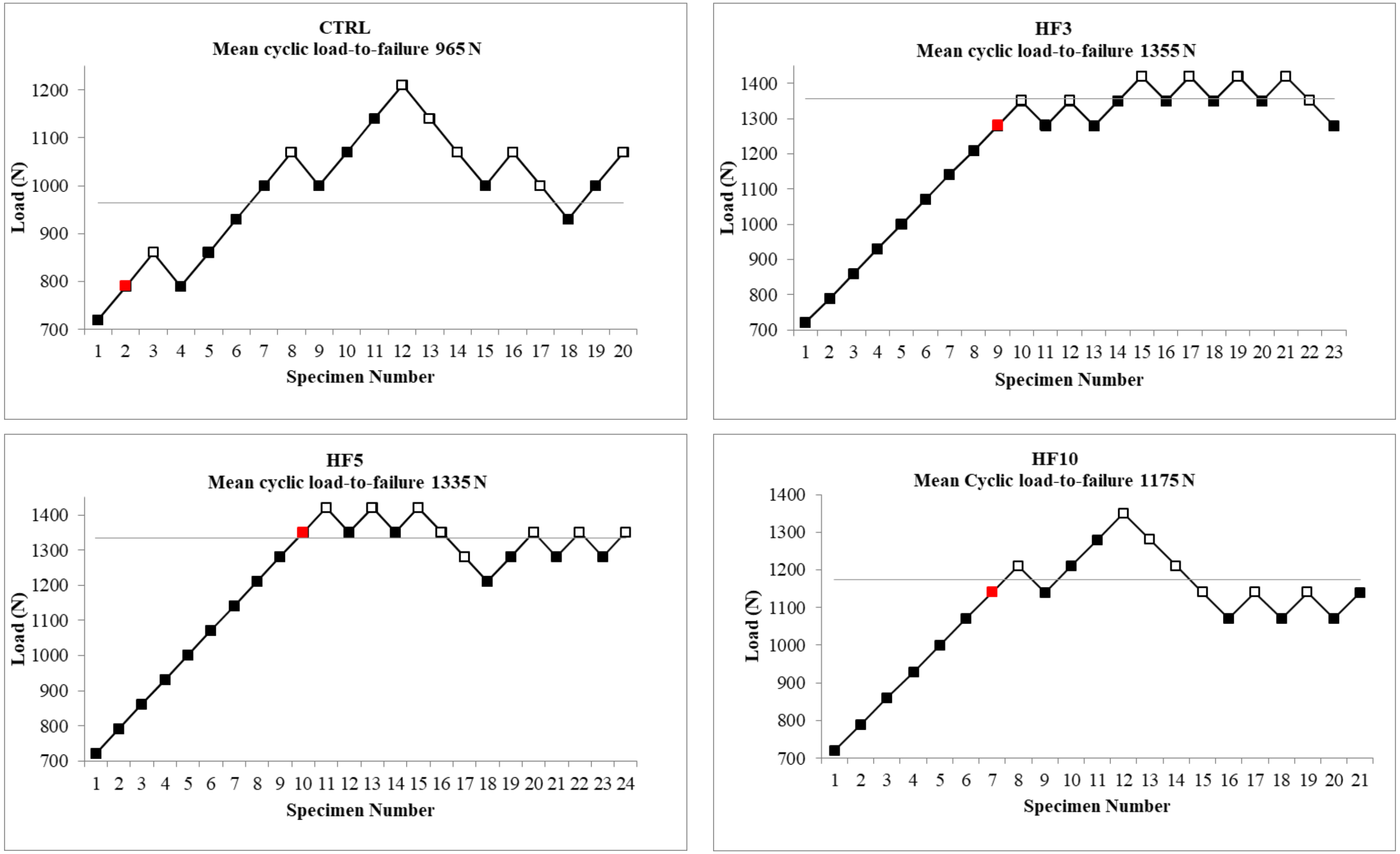


Figure 2 Topographic analysis (2,000× and 8,000× magnifications) of the different surface methods: nonetched control (CTRL), and 20 s of etching with 3% (HF3), 5% (HF5) and 10% (HF10) hydrofluoric acid. The well-known pattern of lithium disilicate grains pulling out from the etched ceramic surface is clearly when 5% and 10% hydrofluoric acid are used, also empty voids can be seeing when a high HF acid concentration is applied (10%; yellow pointers).

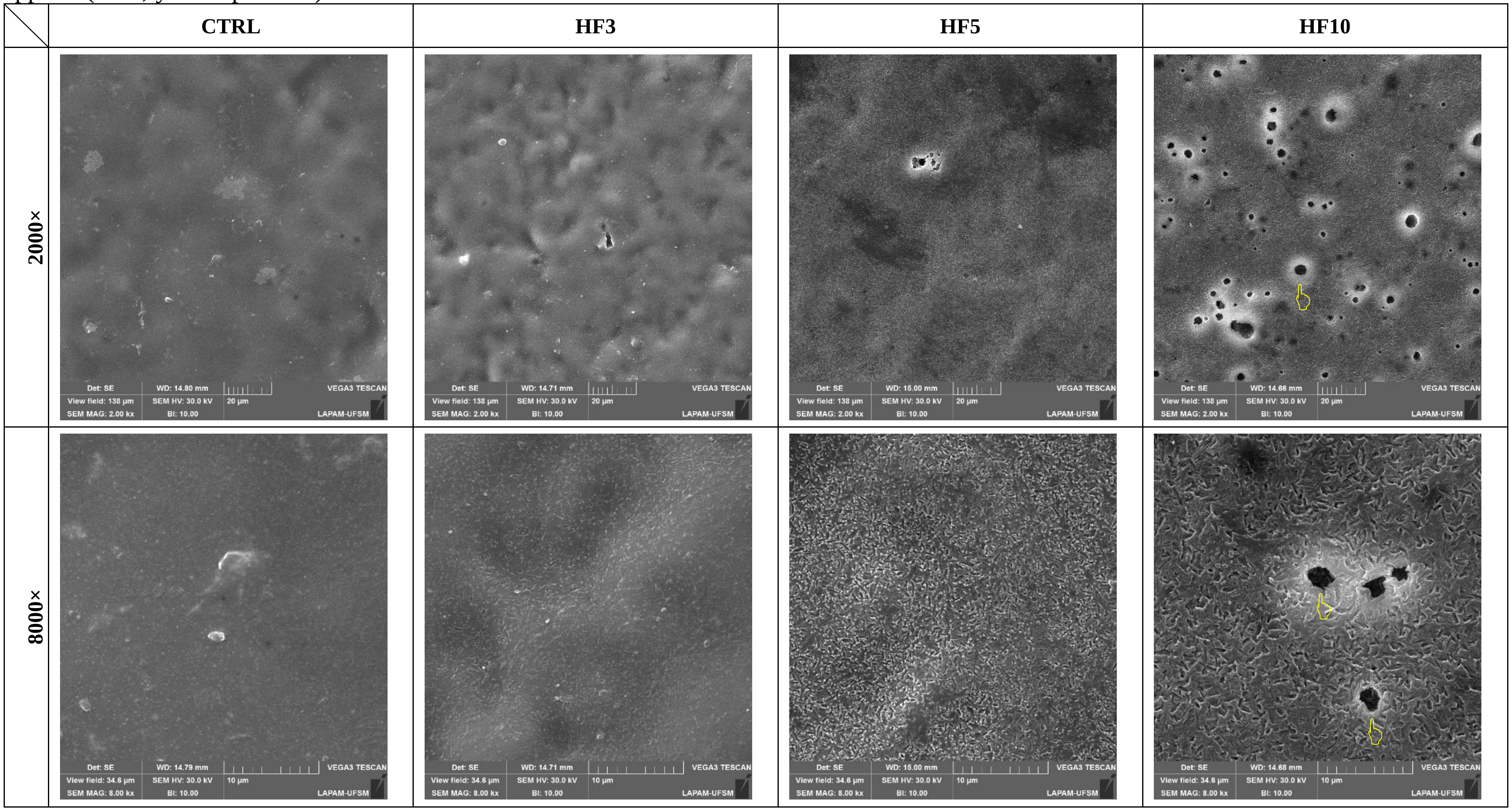
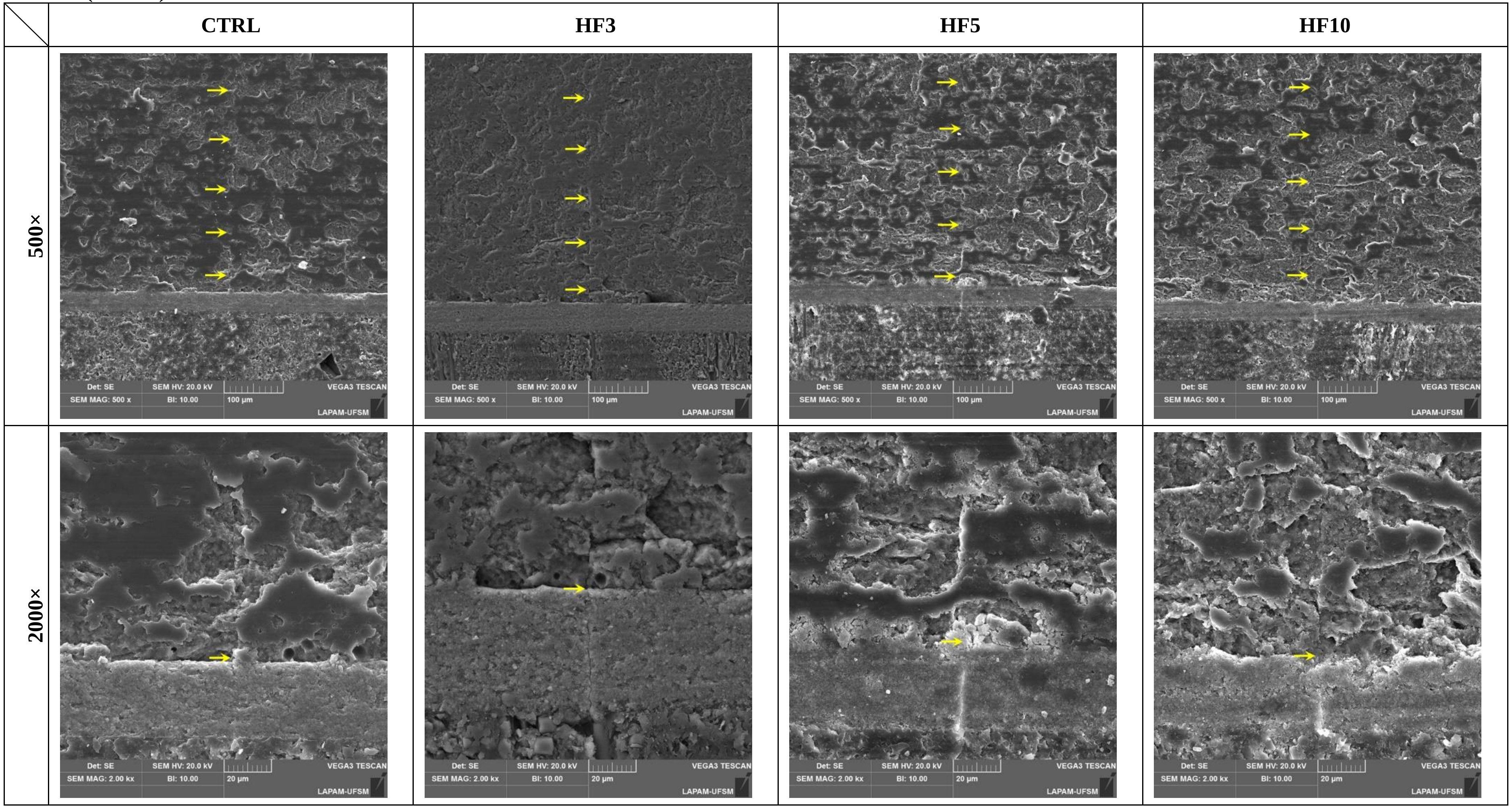


Figure 3 Fractographic examination of representative failed samples after cyclic loads under different magnifications. The yellow arrows indicate the path of crack propagation through the samples (500×), where the crack origin is highlighted under higher magnification at the intaglio ceramic surface (2000×).



4. ARTIGO 3 - HYDROFLUORIC ACID CONCENTRATIONS: EFFECT ON THE CYCLIC LOAD-TO-FAILURE OF MACHINED LITHIUM DISILICATE RESTORATIONS

Este artigo está publicado no periódico *Dental Materials*, ISSN 0109-5641, fator de impacto 4.039, Qualis CAPES A1. As normas para publicação estão descritas no Anexo C.

Hydrofluoric acid concentrations: effect on the cyclic load-to-failure of machined lithium disilicate restorations

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ABSTRACT

Objectives: To evaluate the effects of the etching with different hydrofluoric acid (HF) concentrations on the cyclic load-to-failure (C_{Lf}) of machined lithium disilicate crowns cemented to dentin analogue material.

Methods: Pairs of dentin analogue prosthetic preparations and lithium disilicate ceramic crowns with simplified, standardized designs, were machined (n=18). The preparations were etched with 10% HF (60 s), followed by primer application. The intaglio surface of the ceramic crowns was treated as follows: non-etched (control, CTRL); or etched for 20 s with different HF concentrations – 3% (HF3), or 5% (HF5), or 10% (HF10). A silane coating was then applied onto the treated ceramic surfaces, and they were adhesively cemented to the preparations. To perform the fatigue tests (staircase approach), a hemispheric stainless-steel piston (Ø = 40 mm) applied cyclic loads in the center of the crowns under water (initial load: 720 N; step-size: 70 N; cycles: 500,000; frequency: 20 Hz). Additionally, topographic, fractographic, and fractal analyses were carried out. The fatigue data were analyzed using the Dixon and Mood method.

Results: Although the topographic and fractal analyses depicted the action of HF etching altering the superficial complexity and topography, the preponderant topography pattern was established by machining on CAD/CAM. All groups showed similar C_{Lf} (in N) (CTRL= 805.00 ± 91.23; HF3= 781.25 ± 29.87; HF5= 755.00 ± 154.49; HF10= 833.75 ± 100.74).

Significance: Etching with different HF acid concentrations did not promote a deleterious effect on the cyclic load-to-failure of machined lithium disilicate crowns.

Keywords: Fatigue. Glass ceramic. IPS e.Max CAD. Mechanical cycling. Monolithic crown. Surface treatment.

1 INTRODUCTION

With an increasing demand for aesthetics and mechanical reliability, lithium disilicate ceramic has been meeting an outstanding role among the glass-ceramics available for monolithic restorations. This ceramic material, feasible to machining by CAD/CAM (Computer-aided design/Computer-aided manufacturing) presents a good success rate (~ 95%) for single crowns for up to 10 years of follow-up [1-3].

As a relevant issue, a strong bond between the ceramic restoration and tooth structure provides good support for the restoration and actively transmits functional loads through the bonded interface, thus improving clinical success [4,5]. Regarding vitreous ceramic restorations, to achieve successful bonding, the glass matrix of the intaglio ceramic surface may be chemically modified by hydrofluoric acid (HF) etching in order to promote an irregular surface to provide a mechanical interlocking with resin cement [6-9]. A silane coupling agent is applied on the etched surface to promote a chemical bond between the inorganic phase of the ceramic (i.e., silica), and organic phase (i.e., polymer) of resin cement [10-12]. Studies have shown that HF etching or silane coating, when applied separately, do not promote stable bond strength [13,14].

The manufacturer's recommendation to etch lithium disilicate is with 5% HF acid for 20 s [15]. Despite the increase in ceramic surface roughness to promote micromechanical interlocking, HF acid is capable of decreasing the ceramic material's strength, as a consequence of the modification of the resident flaw population [16-21]. Since there is a direct association between defects on the bulk structure of ceramic materials and their resistance [22,23], over-etching (i.e., higher exposure time and acid concentration) could negatively influence the long-term success of ceramic restorations. Moreover, HF acid is also a hazardous substance; exposure to it can lead to damage to the eyes, soft tissues, necrosis, and bone decalcification, also related to its time and concentration [24-26].

Some authors have researched about the exact concentration and time applicable to lithium disilicate ceramic, but these studies only applied static loads (monotonic tests), using specimens as bars or discs, and did not consider the geometry of an all-ceramic restoration [17,18,27,28]. Cyclic fatigue tests under intermittent loads are crucial to assessing the ceramic material's mechanical performance, thus providing information about their fatigue strength and also influence of the different factors related to it. These tests have been employed to more closely reproduce the clinical conditions under which restorative materials are subjected during function [29,30].

It is important to elucidate the increase of a glass-ceramic's strength when adhesively cemented [31], but also its degradation when the surface is roughened [28,32]. According to Seydler and Schmitter [33], CAD/CAM systems produce ceramic restorations with few defects due to industrially-prepared ceramics blocks, which have reduced internal defects and flaws [34]. However, some studies have shown that the machining process may introduce new defects on ceramic surfaces and sub-surfaces, leading to a decrease in mechanical strength [35,36].

Therefore, adequate HF etching protocol for micromechanical retention and resin cement bonding without weakening the ceramic should be investigated. Moreover, the final strength of etched ceramics evaluated by monotonic tests and the use of disc- or bar-shaped specimens may not exactly reflect the real strength of all-ceramic restorations, since the defects produced by conditioning could be filled-up by resin cement [21,37]. Limited information is available regarding the influence of different HF acid concentrations under cyclic fatigue of adhesively cemented lithium disilicate crowns, as well as about the combinatorial effect of hard machining.

Besides, fractal analysis has been applied in dentistry to assess the complexity of oral mucosa, the roughness of implants, the surface characterization of zirconia (Y-TZP) ceramics, and also to analyze failures of clinically-failed all-ceramic restorations [38-41]. This mathematical tool can provide a new way to account for complexity of the topographical pattern of the treated ceramic surface. Likewise, the fractal dimension, which is a measurement of how complex the topography is when increased by 10%, causes the surface stiffness to decrease more than one order of magnitude [42].

Herewith, the present study aims to evaluate the cyclic load-to-failure (C_{Lf}) of simplified lithium disilicate crowns machined by the CAD/CAM system etched with different HF acid concentrations. Additionally, topographic, fractographic, and fractal analyses have been carried out. The assumed null hypotheses were: (1) different HF acid concentrations would not promote different cyclic loads to failure, and (2) HF acid etching would not reduce the cyclic loads-to-failure when compared with non-etched crowns.

2 MATERIALS AND METHODS

The general description of the materials used in the present study, their manufacturers, composition, and batch numbers are listed in Table 1.

2.1 Specimen preparation

Initially, a model was machined from a dentin analogue material (epoxy resin, G10, FR4 Laminate Round Rods Epoxyglass; NEMA grade FR4, Accurate Plastics, Inc., Yonkers, NY, USA) in a mechanical lathe (Diplomat 3001, Nardini, Americana, Brazil), simulating a molar tooth with a simplified prosthetic preparation (5.32 mm high; internal angle radii of 0.5 mm; axial walls with 8° inclination – total occlusal convergence angle of 16° with rounded corners; 1.2 mm of chamfer; as previously described by Venturini et al., 2018) [21].

As all the preparations were identical, only one was scanned using the inEos Blue equipment (Sirona Dental Systems GmbH; Bensheim, Germany) of the CAD/CAM CEREC InLab MCXL system. The image was transferred to the software (CEREC in-Lab 3D, v4.1, Sirona Dental Systems GmbH), and a simplified molar crown (adapted from Gressler May et al. 2015; Venturini et al., 2018) was designed, considering a cementation space of 80 µm, and a 1.5 mm final occlusal thickness [21,43].

The simplified ceramic (IPS e.Max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) crowns (N=72) were machined (CEREC inLab MC XL; Dentsply Sirona Dental Systems GmbH), using diamond rotary instruments (Step bur 12S - left side of the CEREC machine and a Cylinder pointed bur 12S - right side of the CEREC machine) under water-cooling with a cleaner/lubricant (Dentatec, Sirona Dental Systems GmbH). For the crystallization process, the flow paste IPS Object Fix Putty/Flow (Ivoclar Vivadent) was applied in the intaglio surface of the crowns, and they were submitted to a firing cycle (840°C – vacuum, 7 min) as recommended by the manufacturer (Ivoclar Vivadent). After cleaning the flow paste, the fitting of each crown to its respective preparation was tested. Also, the occlusal thickness was measured by a digital caliper to ensure that all the crowns had 1.5 mm. No adjustments were needed, and before the cementation procedure, the crowns were cleaned in an ultrasonic bath (Vitasonic, Vita Zanhfabrik; Bad Säckingen, Germany) with isopropyl alcohol (5 min), and the preparations with distilled water (3 min).

2.2 Surface treatments and cementation procedure

The simplified ceramic crowns and preparations were randomly assigned (www.randomizer.org) to four groups (n=18). The inner surface of the ceramic crowns received one of the respective treatments, as follows: non-etched (control, CTRL), etching with HF acid at concentrations of 3% (HF3), 5% (HF5), or 10% (HF10) (experimentally formulated, FGM, Joinville, Brazil) for 20 s, and rinsed with air-water spray (30 s). Upon completion of the surface treatments, all the restorations were subjected to ultrasonic bath (distilled water; 5 min), air-dried, and they received an application of a silane-based primer (Monobond Plus, Ivoclar Vivadent) as recommended by the manufacturer (1 min of reaction).

HF acid at 10% (FGM) was applied on the surface of the dentin analogue material preparations (1 min), followed by rinsing with air-water spray (30 s). The preparations were then cleaned in ultrasonic bath with distilled water (5 min). A mixture of Primers A + B (ratio 1:1; Ivoclar Vivadent) was rubbed onto the preparations' surface with a microbrush (30 s), and air-dried until a thin layer was obtained.

Then, one centimeter of dual-cure resin cement (Multilink Automix, Ivoclar Vivadent) was mixed according to the manufacturer's instructions and applied on the intaglio surface of the crowns. Next, the crowns were adhesively cemented to their respective preparations. With an adapted surveyor (B2, BioArt, Sao Carlos, Brazil), a load of 7.5 N was applied to the crowns (for 10 min), the excess cement was removed, and light curing (1200 mW/cm^2, Radii-Cal, SDI, Bayswater, Australia) was performed for 20 s on each surface (0°, 90°, 180°, 270°, and the occlusal surface). After cementation, the specimens were embedded in cylinders with polyurethane (F16, Fast Cast Polyurethane; Axson Technologies, SikaAxson US, Madison Heights, WI, USA) until 2 mm below the cervical margin to position on the fatigue machine [21]. The specimens were stored in distilled water (37°C) for 7-14 days before the cyclic load-to-failure tests were conducted.

2.3 Cyclic load-to-failure tests

Monotonic load-to-failure tests (EMIC DL 2000, Sao Jose dos Pinhais, Brazil) applying an increased load (1 mm/min) were performed in three specimens (etched with HF 10%) to determine the initial load and step-size for the cyclic fatigue tests. The load was applied by a hemispheric stainless-steel piston (Ø = 40 mm) until a radial crack fracture occurred.

The cyclic load-to-failure (C_{Lf}) was determined by the staircase sensitivity approach [44] in a mechanical testing machine (Instron ElectroPuls E3000; Instron, Norwood, MA, USA), under water. A hemispheric stainless-steel piston (Ø = 40 mm) [45-47] was used to apply the load in the center of the occlusal surface of each crown and was stabilized in a device that ensured that the load was normal to the crown's occlusal surface. An adhesive tape (110 µm) was fixed on the occlusal surface to improve contact with the piston [46-48], thus providing better stress distribution and preventing contact surface damage, which could result in cone-crack damage. Also, a non-rigid sheet (cellophane, 2.50 µm) was placed between the piston and specimen to enhance stress distribution [46,47,49,50] (Figure 1).

Cyclic loads (500,000 load pulses at a frequency of 20 Hz) were applied to the ceramic crowns with amplitudes ranging between 10 N (to maintain the piston in intimate contact with the specimen) and the maximum load for every sample, using the staircase approach described by Collins [44]. The first specimen of each group was tested at an initial load level close to the estimated cyclic load-to-failure (720 N, i.e., 60% of the mean monotonic load-to-failure of ~ 1200 N), until it either failed or ran out at predetermined cycles. A step-size load of approximately 10% (70 N) of the initial load level was applied up or down to the next specimen, according to survival or failure (i.e., subsurface radial crack seen by transillumination) of the previous tested specimen. As described by Collins [44], this procedure needed to be repeated for at least 15 samples per group after the first stair reversal, as this was the minimum amount of specimens to obtain an accurate estimation.

2.5 Data analysis

After the cyclic fatigue tests, cyclic load-to-failure (C_{Lf}) standard deviation (SD), and 95% confidence intervals (CI, $\alpha= 0.05$) were calculated according to Collins [44], based on the data of the less frequent event (survival or failure), as previously described [46,47,51,52].

The data were subjected to the Dixon and Mood method [53] involving maximum-likelihood estimation techniques for analytical solutions to the problem of determining the mean and standard deviation. This method assumes that the fatigue values follow a normal distribution. A sorting index (i) was attributed for each load value (step-size) on the data of the less frequent event, being $i= 0$ correspondent to the lower load value observed, $i= 1$

correspondent to the follow load, and so on. The number of samples tested into each load value (step-size) was expressed by *ni*. The multiplications of *ini* and i^2ni equated to the sorting index multiplicated by the samples number in each load value (step-size).

The equations (1) and (2) were used to calculate the mean cyclic load-to-failure (C_{Lf}) and standard deviation (SD):

Equation (1) $C_{Lf} = C_{Lfi} + d\left(\frac{A}{N} \pm \frac{1}{2}\right)$

Equation (2) $\mathrm{SD} = 1.62d\left\{\left[\frac{\mathrm{NB} - \mathrm{A}^2}{\mathrm{N}^2}\right] + 0.029\right\}$ if $\frac{\mathrm{NB} - \mathrm{A}^2}{\mathrm{N}^2} \geq 0.3$

$\mathrm{SD} = 0.53d$ if $\frac{\mathrm{NB} - \mathrm{A}^2}{\mathrm{N}^2} < 0.3$

Where *d* is the step-size (in N), C_{Lfi} is the load value to *I* = 0; *N*, the sum of *ni*; *A*, the sum of *ini* and *B*, the sum of i^2ni. In equation 1, the (+) sign was used when the less frequent event was survival; the (−) sign was used when the less frequent event observed was failure.

2.6 Topographic analysis

Four additional machined crowns were prepared for field emission scanning electron microscopy (FE-SEM, Sigma 300 VP, Carl Zeiss Ltd., Cambridge, England) at different magnifications. The inner surface of the crowns was treated as aforementioned for the fatigue tests and sputtered with a gold-palladium alloy before examination.

2.7 Fractographic analysis

Fractographic analysis was performed to correlate the direction of crack propagation, pointing back to the origin or cause of failure as a result of cyclic load-to-failure of the tested conditions. After the fatigue tests, all the fractured specimens were assessed under a stereomicroscope (Discovery V20, Carl Zeiss, Gottingen, Germany) to determine the crack direction, and they were sectioned into two halves perpendicularly to the direction of the cracks using a diamond blade under water-cooling (Isomet 1000, Buehler, Lake Bluff, IL, USA). The samples were analyzed with a stereomicroscope to identify the origin and cracks propagation, and representative fractures from each evaluated condition were selected for further analysis under SEM (Vega3, Tescan, Czech Republic) (Figure 3).

2.8 Evaluation of the fractal dimension

The qualitative evaluation of the fractal dimension was carried out for five images at different magnifications of the ceramic surface treatments by the box counting method. The algorithm iteratively halves an initial cubic cell with the edge length L equal to the scan length into smaller cubes, and counts N(L) – the number of all cubes that contain at least one sample of a 2D topography. The process continues until L approaches the image resolution, i.e., the distance between two adjacent samples. Since N(L) is proportional to L^{-D}, the slope of a log-log plot of N(L) versus L gives the fractal dimension referred to as the cube count fractal dimension D_{BC}. Fractal dimension data were submitted to 1-way ANOVA and Tukey's test (α= 0.05).

3 RESULTS

The cyclic load-to-failure means, and staircase graphs are presented in Table 2 and Figure 2, respectively. According to the confidence interval overlapping, all groups were statistically similar.

The FE-SEM images indicate that even after HF acid etching at different concentrations, hard machining appears to predominate as a surface pattern, indicating its high potential to induce defects on the surface of lithium disilicate ceramic, which are not completely removed or modified after HF etching. Also, no pullout of lithium disilicate crystals could be observed after etching, even at higher magnifications. (Figure 3).

All failures after fatigue testing were radial cracks starting from the cemented surface. Representative SEM micrographs of the fractured surfaces are presented in Figure 4.

Even without statistical differences based on 1-way ANOVA and Tukey's test, fractal analysis showed a surface less complex after 5% HF etching, as compared to 3% and 10% HF;

the surface's complexity after 5% HF etching was quite similar to the control group (as-machined) (Table 2).

4 DISCUSSION

The aim of the present study was to evaluate the effects of distinct HF acid etching concentrations on the cyclic load-to-failure (C_{Lf}) of machined lithium disilicate crowns cemented to dentin analogue material. The first null hypothesis was accepted, since all HF acid concentrations tested (3%, 5%, and 10%) led to statistically similar cyclic loads-to-failure. Etching the inner surface (i.e., intaglio) of machined lithium disilicate crowns with HF acid at three distinct concentrations promoted similar cyclic load-to-failure values when compared to non-etched crowns, accepting the second null hypothesis.

Lithium disilicate is an acid-sensitive glass ceramic available to the hard machining process [54], which requires high removal forces during machining [55]. These high forces introduce large critical defects that can contribute to initiating failure in the intaglio ceramic surface. Fraga and collaborators (2017) stated that hard machining of lithium disilicate produces higher roughness values and lower fatigue strength when compared to the same machined ceramic after polishing [36]. The findings of the present study support that even after HF acid etching with different concentrations; the surface pattern generated by machining still predominates in topography images, and the effect of etching is localized and is on a smaller scale (Figure 3).

Research studies have been conducted with ceramics of different chemical composition to determine the effect of HF etching on the mechanical properties of restorative assemblies [21,46,50]. It seems that the high heterogeneity of data corroborating a positive or negative effect might be explained by the influence and interaction of different factors converging to define the final load-bearing capability of the restorative assembly. In this sense, factors, such as the assumed specimen geometry simulating or not a posterior restoration, the degree of convergence of the axial walls on tooth preparations (or analogue material), the presence *versus* absence of CAD/CAM machining, the surface treatment of the ceramic restoration (HF etching, etchant viscosity, the protocol used, i.e., etching times and concentrations), the ceramic assumed topography, as well as the properties of the resin cement (e.g., viscosity, reinforcement filler particles size, among other characteristics) will interact and define the final strength of the restoration. Also, these factors will determine the potential of the cement to fill-up all the spaces produced by the machining/surface treatment, and at the end, will generate a theoretically-integrated restorative assembly. Notably, air bubbles, reminiscent defects, and areas not completely filled-up by the resin cement may act as critical defects leading to stress concentration; consequently, leading to premature failure of the assembly [21].

Critical defects in the ceramic surface and sub-surface that could decrease the final strength of the assembly can be promoted as a consequence of hard machining process, internal adjustments after the ceramic crystallization process, and also by HF acid etching (time and concentration dependent) [21,28,36,50,56]. With regard to HF acid etching, even showing high bond strength values and adhesion durability when associated with silanization, the literature remains controversial whether HF acid etching negatively affects ceramic strength [17,18,27,28].

Recently, Prochnow and collaborators [28] tested the isolated effects of different HF acid concentrations (1, 3, 5, and 10%) in the flexural strength, mechanical reliability, and roughness of a lithium disilicate glass-ceramic. No statistical difference in roughness or flexural strength were found among the tested groups, even with the higher acid concentrations presenting a more aggressive glassy matrix removal (SEM and atomic force microscope data). However, that study did not consider the processing methodology (CAD/CAM machining) for obtaining restorations of such material (lithium disilicate). Therefore, in the present study, all the specimens were hard-machined. In the FE-SEM images (Figure 3), is clear that CAD/CAM machining potentially changes the lithium disilicate ceramic surface, and when HF acid etching is performed, the surfaces change in a different way and do not clearly promote the pullout of lithium disilicate crystals. Our findings show that for machined lithium disilicate glass-ceramics, machining overlaps the HF etching in terms of topographical changes (Figure 3). However, the pattern found for the surfaces machined and after conditioned is completely different than the one reported by the current literature when using HF acid etching only [9,18,28].

Fractal analysis may be a powerful tool to assess differences in surface complexity after distinct surface treatments [38-41]. In this way, it can be stated that 3% and 10% HF concentrations for etching depicted the most complex final topographies (Figure 3; Table 2). Even do not presenting statistical differences, roughly the fractal dimension increases 5% when comparing the control group with the 3% of HF etching set, or 10% comparing the control group with 10% HF etching set. We recall that even small differences in fractal dimensions such as those measured here can result in a significant decreasing in stiffness [42]. On the other hand, 5% HF acid etching set has a fractal dimension similar to the control group (only machined), as can be verified in FE-SEM images (Figure 3).

Adhesively cementation has a crucial role, not only for sealing the restoration's margins to avoid resin cement solubility, but also to provide reinforcement for glass-ceramic restorations [57]. Accordingly, the final strength of glass-ceramic restorations relies highly on the adhesion protocol used and on the resin cement's ability to fill-up the defects at the intaglio ceramic surface, transmitting the loads through the assembly [21,31,37]. It is possible that even when producing distinct surface patterns, as shown by fractal analysis and FE-SEM images (Figure 3; Table 2), the used resin cement was able to fill up all the defects. It resulted in assemblies with the same ability to transmit the loads, promoting similar fatigue performance for the testing groups. In this sense, the viscosity of the resin cement employed could play an important role, and the results could be different if a different resin cement was used. Besides, May et al. [31] showed that with the increase in thickness of the cement layer on the bond interface, the influence of an adhesive *versus* a non-adhesive cementation strategy decreases, thus compromising the theoretical reinforcement of mechanical properties. With a thicker layer of cement on the interface, the probability of air bubbles and the presence of critical defects on such a layer also increases.

The aqueous oral environment can reduce the number of cycles required to produce fatigue failure in glass-ceramics [58,59]. For posterior restorations, one year of clinical service is related to 250,000 cycles at only 13.6 N under fatigue [60]. In mechanical aging, it is crucial to simulate the stress/load as close as possible to the *in vivo* situation [29]; however, fatigue testing with low loads and frequency is time-consuming [61]. Therefore, in order to acquire more clinically relevant data with regard to the effect of HF acid etching, hard machining, and cementation of lithium disilicate ceramic crowns, an accelerated fatigue test was used in this study. According to Fraga and collaborators [61], there is no significant difference between fatigue tests under the frequency of 2 and 20 Hz for yttria-partially stabilized zirconia. The hemispheric stainless-steel piston used to apply the loads in our study produces tensile stresses in the cementation surface of the crowns. Consequently, it was observed that all the failures were radial cracks starting from that surface, which is the same type of failure observed clinically [30,62,63]. Employing accelerated fatigue and the metallic piston have been well-accepted in the literature, and several studies have been recently published [21,43,46,47,50,52,61,63].

The authors fully acknowledge that the application of only axial loads (without sliding), the absence of thermocycling and longer periods of sample storage are important limitations of the present investigation. Moreover, the simplified ceramic crowns and preparations, as well as the use of a dentin analogue material in place of a tooth substrate were used in previous studies [21,43], but they also do not fully mimic the clinical scenario; in other words, the results of the present study should be taken with caution.

CONCLUSIONS

Distinct hydrofluoric acid concentrations do not affect the fatigue behavior of machined lithium disilicate ceramic crowns.

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TABLES

Table 1 General description about the materials used in the present study, their manufacturers, composition and batch number.

Material (Manufacturer)	**Composition**	**Batch number**
IPS e.Max CAD (Ivoclar Vivadent)	Lithium disilicate glass-ceramic (SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, other and colouring oxides)	W31404
NEMA grade G10 (Accurate Plastics)	Continuous filament woven fiberglass bonded with epoxy resin	-
Hydrofluoric acid (3, 5, and 10 % FGM)	3, 5 and 10% hydrofluoric acid, water, thickener, surfactant and colouring	12/2016
Monobond Plus (Ivoclar Vivadent)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate	W10892
Multilink Automix (Ivoclar Vivadent)	Dimethacrylates, HEMA, barium glass filler, Ba-Al-fluoro-silicate glass, ytterbium trifluoride, highly dispersed silica, catalysts and stabilizer, pigments	V08514
Multilink Primer A and B (Ivoclar Vivadent)	Primer A: water, initiators; Primer B: phosphonic acid acrylate, hydroxyethyl methacrylate, methacrylate mod. polyacrylic acid, stabiliser	V10775-V18752

Table 2 Mean cyclic load-to-failure (C_{Lf}), standard deviation (SD), and 95% confidence interval of lithium disilicate ceramic crowns based in Dixon and Mood method [53]. Mean fractal dimension values estimated by the box counting (D_{BC}) method of the tested ceramic surfaces.

Group	**C_{Lf}(SD)***	**Confidence Interval**	**D_{BC} (SD)**
CTRL	805.00 (91.23)^{A}	738.77 – 871.23	1.81 (0.06)^{A}
HF3	781.25 (29.87)^{A}	758.13 – 804.47	1.85 (0.05)^{A}
HF5	755.00 (154.49)^{A}	661.13 – 848.87	1.82 (0.08)^{A}
HF10	833.75 (100.74)^{A}	766.03 – 901.47	1.88 (0.01)^{A}

*Similar superscript letters indicate statistically significant equality based on confidence interval overlapping.

FIGURES

Figure 1 Representative image of the test assembly on the fatigue machine.

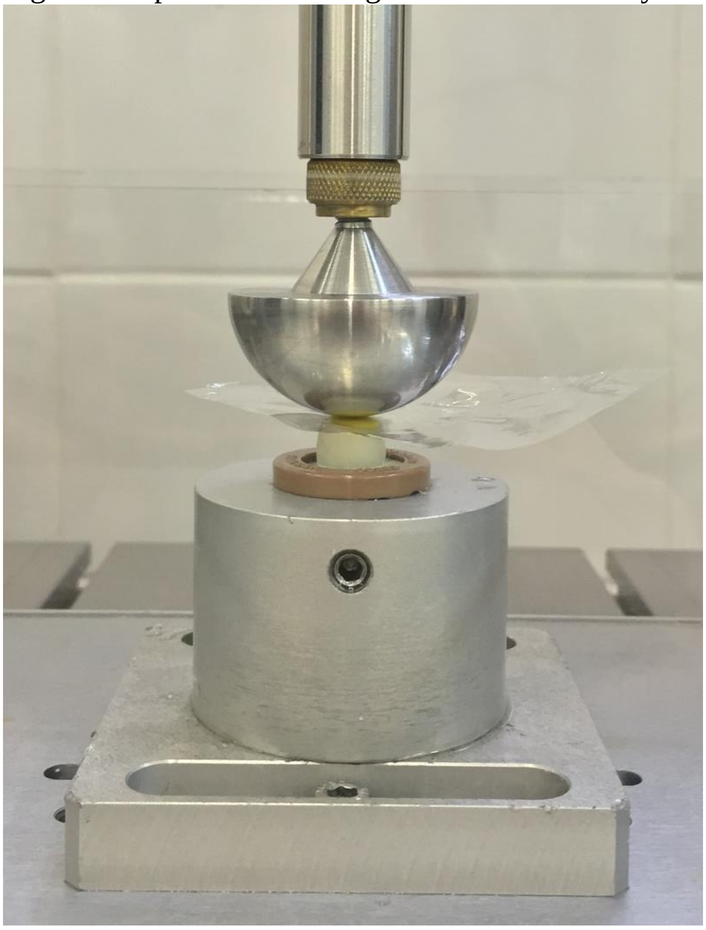


Figure 2 Staircase sensitive results after cyclic loads-to-failure (500,000 cycles; 20 Hz). Horizontal lines indicate de mean cyclic load-to-failure, the red diamonds indicate the load level at which the up-and-down character started (ladder reversed), filled diamonds show survival, empty diamonds are failure.

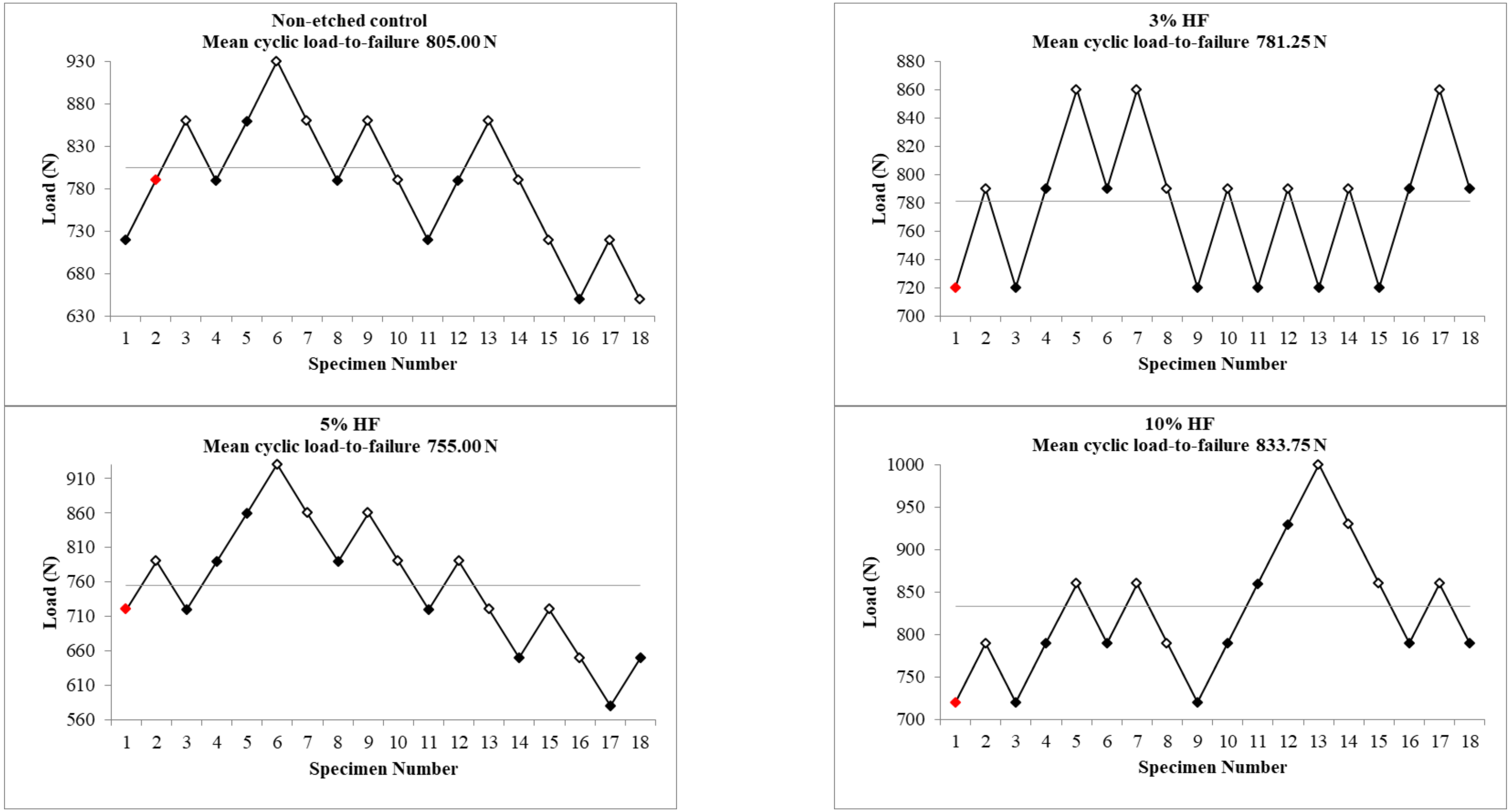


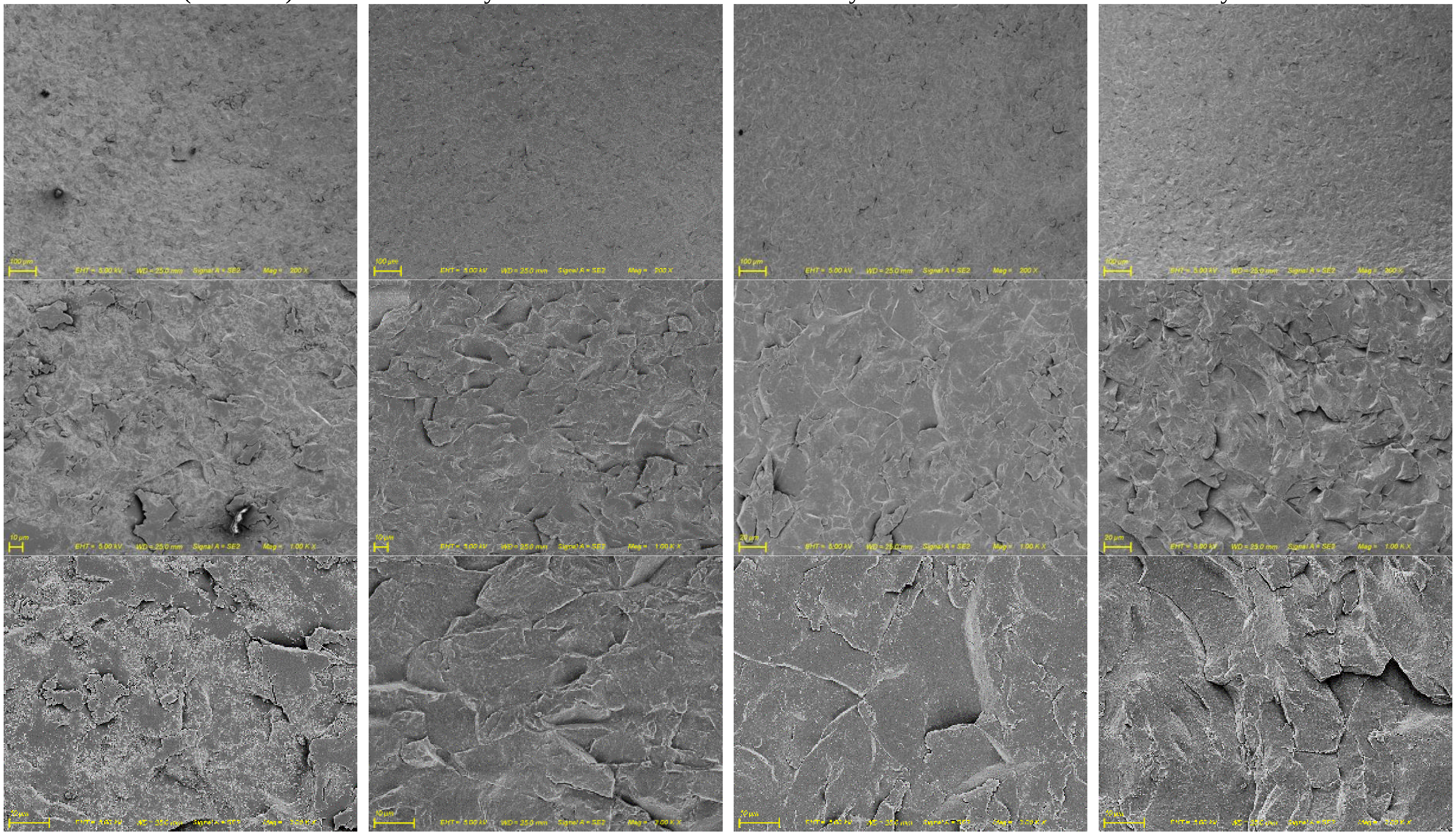
Figure 3 Topographic analysis of the ceramic crowns' intaglio surfaces under FE-SEM at different magnifications.

Non-etched (as milled) | 3% hydrofluoric acid | 5% hydrofluoric acid | 10% hydrofluoric acid

200×

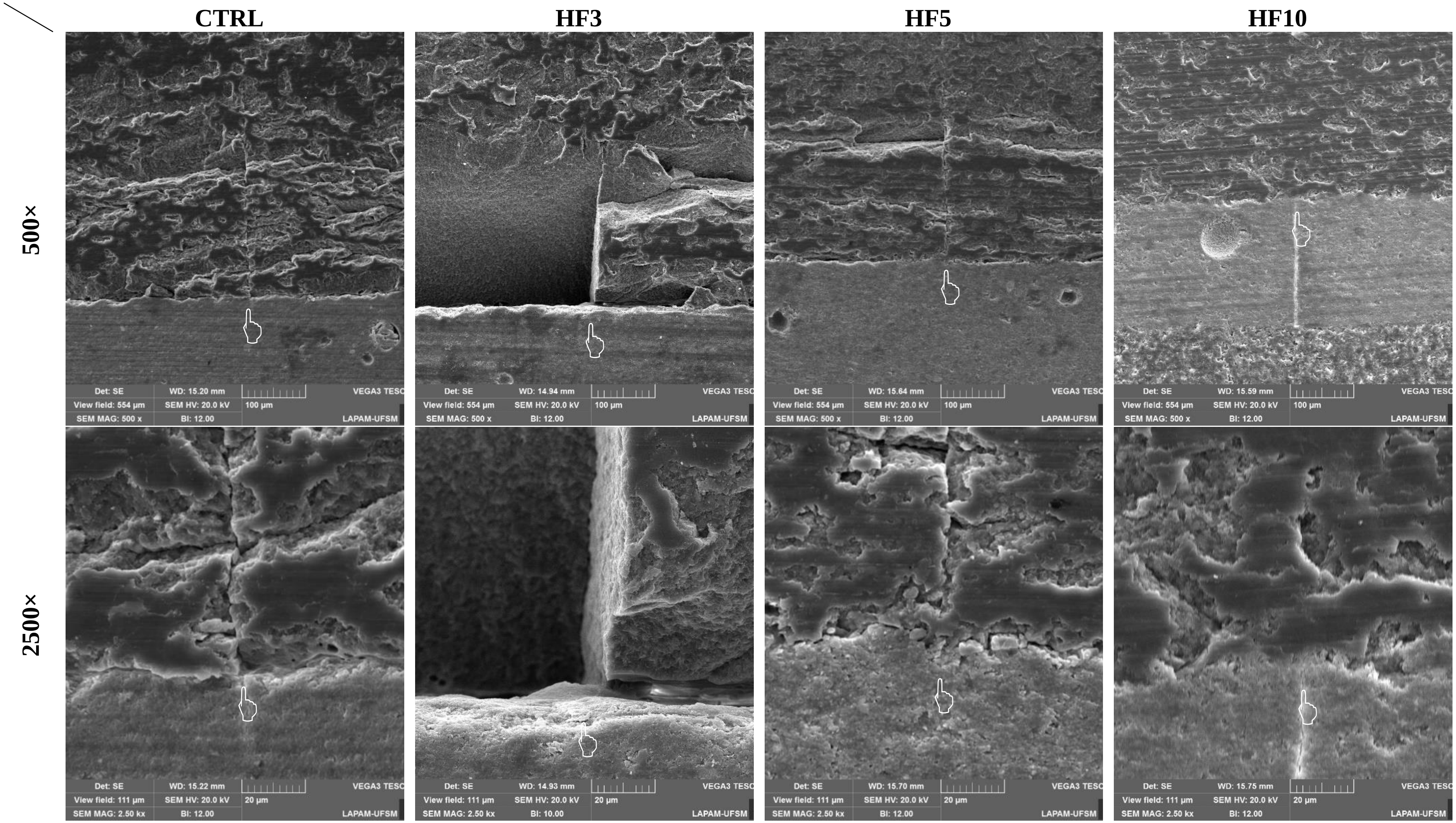
1000×

3000×



CTRL (non-etched, as milled); HF3, HF5 and HF10 (etched with 3%, 5% and 10% hydrofluoric acid, respectivelly).

Figure 4 Representative images of the specimen failures under different magnifications. The white pointers indicate the failure origin.



5 DISCUSSÃO GERAL

Um efetivo condicionamento ácido da superfície cerâmica é considerado um passo essencial para o sucesso clínico de restaurações cerâmicas (DELLA BONA; ANUSAVICE, 2002). Assim, análises microestruturais e da superfície de cerâmicas condicionadas têm mostrado que diferentes padrões topográficos são criados de acordo com a concentração, tempo de aplicação e tipo de ácido utilizado (RAMAKRISHNAIAH et al., 2016; PROCHNOW et al., 2017). Apesar disso, a literatura mostra que um sobre condicionamento é capaz de diminuir a resistência da cerâmica condicionada, devido a um aumento na população de defeitos promovido pela excessiva remoção de matriz vítrea (HOOSHMAND; PARVIZI; KESHVAD, 2008; ZOGHEIB et al., 2011). Conjuntamente, o ácido fluorídrico é uma substância altamente tóxica (OZCAN; ALLAHBEICKARAGHI, DÜNDAR, 2012; DENNERLEI et al., 2016). Com isso, a presente tese se propôs a avaliar o efeito de diferentes concentrações de ácido fluorídrico na adesão em comportamento em fadiga de uma cerâmica de dissilicato de lítio, em busca de menores concentrações capazes de promover não apenas a durabilidade da resistência de união, mas também um comportamento em fadiga semelhante ao encontrado com a concentração de ácido fluorídrico recomentada pelo fabricante (5%; IVOCLAR VIVADENT).

Num primeiro momento, objetivou-se avaliar o efeito de diferentes concentrações de ácido fluorídrico na resistência de união ao microcisalhamento entre uma cerâmica de dissilicato de lítio e um cimento resinoso. Quando a degradação hidrolítica e a ciclagem térmica não estiveram presentes, todas as concentrações testadas mostraram resistência de união semelhante, sendo apenas o grupo 1% inferior ao condicionamento com HF 5%. Após 150 dias de armazenagem e ciclagem térmica, o grupo com uma menor alteração superficial e portanto, área disponível para adesão (1%) apresentou menores valores de resistência de união, corroborando os achados da literatura que mostram que alterações micromorfológicas promovem melhores resultados em termos de resistência de união (LISE et al., 2015). Esses achados são ratificados pelas imagens em MEV (Microscópio Eletrônico de Varredura), que mostram uma maior remoção da matriz vítrea e descolamento dos cristais de dissilicato de lítio quando maiores concentrações de ácido fluorídrico foram utilizadas.

No mesmo sentido, a cerâmica condicionada com HF nas concentrações de 3, 5 e 10% apresentou menores valores de ângulo de contato, indicando que estes tratamentos promoveram um aumento da área disponível para adesão. De acordo com Della Bona et al. (2004), as alterações promovidas pelo condicionamento ácido promovem um aumento na área superficial e assim, um melhor contato entre a superfície cerâmica e o cimento resinoso (DELLA BONA; SHEN; ANUSAVICE, 2004). Da mesma forma, os valores de rugosidade obtidos a partir de microscopia de força atômica mostraram superfícies mais rugosas para o condicionamento com HF 5 e 10%, corroborando não apenas os achados em termos de adesão, mas também as imagens obtidas em MEV.

Após a obtenção de uma união estável entre cimento resinoso e dissilicato de lítio condicionado com as diferentes concentrações de ácido fluorídrico, se tornou pertinente avaliar o desempenho de tais tratamentos de superfície em cenários mais complexos, que pudessem se aproximar devidamente da realidade clínica. Para tal, o condicionamento com ácido fluorídrico nas concentrações de 3%, 5% e 10% foi avaliado na carga cíclica para falha de uma cerâmica de dissilicato de lítio cimentada adesivamente a um material análogo de dentina. Em um primeiro momento, uma metodologia de teste simplificada já consolidada na literatura foi utilizada (CHEN et al., 2014; MONTEIRO et al., 2018; SCHERER et al., 2018).

O uso de restaurações simplificadas de dissilicato de lítio tornou possível a avaliação do efeito isolado do condicionamento com HF para estas restaurações em ambiente úmido. Assim, as alterações topográficas obtidas foram semelhantes àquelas encontradas na literatura (PROCHNOW et al., 2017; RAMAKRISHNAIA et al., 2016). Sabe-se que a cimentação adesiva apresenta um papel importante na transmissão de cargas ao conjunto restaurador (MAY et al., 2012), além disso, quando os defeitos causados pelo condicionamento ácido/processamento são completamente preenchidos por um cimento resinoso, o conjunto se torna mais resistente (VENTURINI et al., 2018). Sendo assim, concentrações intermediárias de ácido fluorídrico apresentaram maiores valores de carga cíclica para falha, quando comparadas ao condicionamento com ácido fluorídrico a 10% e à ausência de condicionamento, mostrando que a ausência de embricamento micromecânico é deletéria para o conjunto, da mesma forma que o aumento da população de defeitos, muito provavelmente não preenchido pelo cimento resinoso.

Apesar de esta assembleia de ensaio ter sido muito difundida na literatura ultimamente, a ausência de uma geometria da restauração e outras variáveis presentes clinicamente como a usinagem em CAD/CAM não são contempladas. Consequentemente, se fez pertinente avaliar em um segundo momento o efeito das mesmas concentrações de ácido fluorídrico (3%, 5% e 10%) na carga para falha em fadiga de restaurações de dissilicato de lítio usinadas pelo sistema CAD/CAM e cimentadas adesivamente a preparos protéticos simplificados de um material análogo de dentina.

Sabe-se que o procedimento de usinagem, como a realizada no dissilicato de lítio, requer forças para a usinagem, e por conseguinte, promove defeitos na superfície interna das restaurações (SONG; REN; YIN, 2016). Os achados do terceiro estudo demonstram que tais alterações produzidas pela usinagem em CAD/CAM são preponderantes ao condicionamento ácido na cerâmica de dissilicato de lítio. Os achados de microscopia mostram um padrão topográfico diferente daquele normalmente encontrado quando o condicionamento é realizado na mesma cerâmica polida ou “mantida como cortada” (ZOGHEIB et al., 2011; RAMAKRISHNAIAH et al., 2016; PROCHNOW et al., 2017), onde um descolamento dos cristais de dissilicato de lítio é imperceptível mesmo em maiores aumentos.

Assim, quando as coroas de dissilicato de lítio usinadas foram submetidas à fadiga, as diferentes concentrações de ácido fluorídrico testadas apresentaram valores similares de carga cíclica para falha, da mesma forma que o grupo controle negativo (apenas silanizado), indicando, que neste caso, a população de defeitos criada pela usinagem é hegemônica. Em busca de informações mais detalhadas em relação às superfícies obtidas após usinagem e condicionamento, a análise da dimensão fractal (complexidade da superfície das amostras) também foi realizada. Apesar de as amostras condicionadas com ácido fluorídrico nas concentrações 3% e 10% terem apresentado uma maior complexidade superficial de ordem numérica, os valores encontrados não foram estatisticamente diferentes entre si, corroborando os achados de carga cíclica para falha.

Ressalta-se que o intuito da presente tese foi encontrar uma concentração de ácido fluorídrico mais baixa que fosse capaz de promover durabilidade da resistência de união e um bom desempenho em fadiga, tendo em vista que o ácido fluorídrico é uma substância altamente tóxica, concentração e tempo dependente (OZCAN; ALLAHBEICKARAGHI, DÜNDAR, 2012).

Como limitações da presente tese, podem-se ressaltar o uso de superfícies polidas que não condizem com o observado clinicamente, na superfície de cimentação de próteses cerâmicas, para o ensaio de resistência de união ao microcisalhamento e ensaio em fadiga com discos produzidos em laboratório. Desta maneira, torna-se importante salientar a busca por métodos laboratoriais alternativos que sejam capazes de simular a topografia de superfície obtida após usinagem em sistemas CAD/CAM, como o método sugerido por RODRIGUES et al. (2018). Em relação aos artigos de fadiga, o uso de apenas cargas axiais, a ausência de uma superfície dentária como material de suporte, o ensaio de fadiga acelerado e o uso de geometrias de teste simplificadas devem ser ressaltados.

6 CONSIDERAÇÕES FINAIS

Baseado nos achados da presente tese, pode-se concluir que em termos de adesão, a cerâmica de dissilicato de lítio pode ser condicionada com ácido fluorídrico nas concentrações 3%, 5% e 10%. Em relação ao comportamento em fadiga, quando o efeito isolado do condicionamento ácido é testado (sem o efeito combinado da usinagem e da geometria do espécime, simulando uma coroa), concentrações intermediárias de ácido fluorídrico (3% e 5%) promovem um melhor comportamento em fadiga, em linha com a recomendação do fabricante. Já em coroas de dissilicato de lítio usinadas pelo sistema CAD/CAM, o efeito topográfico preponderante nas superfícies condicionadas observado em microscopia é aquele obtido pela usinagem. Sendo assim, as diferentes concentrações de ácido fluorídrico testadas não se mostram capazes de afetar o comportamento em fadiga de coroas de dissilicato de lítio usinadas cimentadas adesivamente.

Portanto, com base nos achados dos três artigos componentes da presente tese, a aplicação de ácido fluorídrico em concentrações reduzidas de 1 % não demostrou um efeito adequado, sendo necessário concentrações de pelo menos 3% para alcançar um comportamento otimizado frente à adesão e resistência da restauração cerâmica. Salienta-se ainda que o aumento de concentração superior a 3% (5% e 10%) não se mostrou efetivo em otimizar a performance em nenhum dos desfechos avaliados, evidenciando-se a utilização do ácido fluorídrico na concentração de 3%, inferior àquela recomendada pelo fabricante, com uma menor toxicidade e resultados em termos de adesão e fadiga semelhantes às concentrações de 5 e 10%.

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ANEXO A – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *BRAZILIAN DENTAL JOURNAL*

Form and preparation of manuscripts

THE FOLLOWING GUIDELINES MUST BE FOLLOWED CAREFULLY.

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Pages should be numbered consecutively, starting with the summary.

Full-length manuscripts are assembled in the following sections: 1) Title Page; 2) Summary and Key Words 3) Introduction; Material and Methods; Results; Discussion 4) Summary in Portuguese (an item necessary for Latin American Indexing Services that will be provided for non-Brazilian authors by the Journal) 5) Acknowledgements (if any) 6) References 7) Tables 8) Figure captions 9) Figures

All titles of sections (Introduction, Material and Methods, etc) must be capitalized in regular font type (not bold).

Results and Discussion **MUST NOT** be joined in a single section.

Short Communications and Case Reports should be divided into appropriate sections.

Products, equipments and materials: the trade name must be followed by the manufacturer's name, city, state and country, within parentheses upon first mention. For further mentions, only the manufacturer's name is required.

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Results: Present the results in a logical sequence in the text, tables and figures, emphasizing the important information. Do not repeat in the text data contained in the tables and illustrations. The important observations should be emphasized. Do not repeat the same data in tables and figures. Describe the statistical data in this section.

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Acknowledgements: Financial support by government agencies should be acknowledged. If appropriate, technical assistance or assistance from colleagues may be acknowledged.

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Book 2. Shafer WG, Hine MK, Levy BM. A Textbook of Oral Pathology. 4th ed. Philadelphia: WB Saunders; 1983.

Chapter in a Book 3. Walton RE, Rotstein I. Bleaching discolored teeth: internal and external. In: Principles and Practice of Endodontics. Walton RE (Editor). 2nd ed. Philadelphia: WB Saunders; 1996. p 385-400.

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ANEXO B – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS*

GUIDE FOR AUTHORS

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