UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

BÁRBARA MARGARIDO BRONDINO

**Lithium Disilicate glass-ceramic surface analysis by Atomic Force
Microscopy and Optical Microscopy**

**Análise de superfície da Cerâmica de Dissilicato de Lítio por Microscopia
de Força Atômica e Microscopia Óptica**

BAURU
2018

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Dissertação constituída por artigo apresentada à Faculdade de Odontologia de Bauru da Universidade de São Paulo para obtenção do título de Mestre em Ciências no Programa de Ciências Odontológicas Aplicadas, na área de concentração Reabilitação Oral.

Orientador: Prof. Dr. José Henrique Rubo

BAURU
2018

Brondino, Bárbara Margarido

Lithium Disilicate glass-ceramic surface analysis
by Atomic Force Microscopy and Optical
Microscopy / Bárbara Margarido Brondino – Bauru,
2018.

Dissertação (Mestrado) – Faculdade de
Odontologia de Bauru. Universidade de São Paulo

Orientador: Prof. Dr. José Henrique Rubo

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DEDICATÓRIA

*À minha mãe, **Cristina**, por todo carinho e amor incondicional que recebi. Uma mulher forte, guerreira, determinada e incrivelmente inteligente, que me inspira todos os dias. Você sempre foi o meu exemplo de humildade, caráter e bondade. Espero ser seu motivo de orgulho e que eu consiga ser, ao menos, metade do que você é. Te admiro muito e te amo infinitamente.*

*Ao meu pai, **Odney**, por ser meu herói até hoje. Uma pessoa extremamente esforçada, que corre atrás de seus objetivos e que teve uma trajetória de vida dura e exemplar. Me espelho em você, na sua determinação e força de vontade. Espero voar tão alto quanto você voou. Tenho o maior orgulho de ser sua filha!! Te amo absurdamente!*

*Aos meus avós, **Zuleica, Nelson, Delcides e Armelindo**, que, apesar de não terem tido muitas oportunidades, fizeram com que meus pais estudassem e me passassem o valor dos estudos. Vocês são meus amores e meu amor é incondicional.*

*Ao meu irmão, **Brunno**, e a todos os meus familiares, que sempre me incentivaram a buscar os meus sonhos e estão comigo em todos os momentos. Amo vocês!!*

AGRADECIMENTOS

*A **Deus**, pelo dom da vida, pela minha saúde e por me proporcionar uma experiência terrena tão maravilhosa e enriquecedora.*

*Ao meu pai, **Odney**, pelo incentivo para fazer a pós-graduação, pelo amparo nos momentos difíceis e por me alegrar sempre. Por não medir esforços e por ser tão carinhoso, sempre confiando no meu trabalho. Foi um dos meus primeiros pacientes... Jamais me esquecerei disso! Obrigada por tudo! Amo você.*

*À minha mãe, **Cristina**, pelo amor incondicional, pela paciência, carinho e por participar de forma tão ativa desta dissertação, realizando (maravilhosamente bem!) a estatística. Você é minha melhor amiga! A responsável por eu ter escolhido a Odontologia, profissão que eu realmente gosto e me sinto realizada. Muito obrigada! Te amo.*

*Ao meu irmão, **Brunno**, que divide a vida comigo há 19 anos. Já tivemos muitos momentos bons e ruins, mas essa é a real vida de irmãos. Saiba que eu sempre estarei com você, apoiando e incentivando em todos os momentos. Pegue muito no seu pé, mas te amo!*

*Ao **Guilherme Sequeto**, meu namorado, melhor amigo e companheiro, sempre tão paciente e disposto a ajudar. Que não mede esforços para me ver bem e que me ajudou demais nas fases difíceis. Espero que esse trabalho te incentive, de alguma forma, a continuar correndo atrás dos seus sonhos... Estarei sempre por perto. Tenho muito orgulho de você. Obrigada por ter chegado à minha vida para ficar. Te amo!*

*À minha cachorrinha, **Kiwi**, por estar dividindo a sua vida comigo e por ter ficado ao meu lado durante todo tempo em que escrevi essa dissertação... Espero ser digna do seu amor e companheirismo!*

*Às minhas famílias, **Margarido (Miguel, Nereide, Isabella, Ana Claudia, Rafael, Lucas, Xixo, Claudia e Pedrinho)** e **Brondino (Rose, Marinho, Pedro, Giovanna, Amanda, Ailton, Susie, Klaen, Allexia e João Alexandre)**, por todo apoio e amor incondicional. Um agradecimento especial às minhas avós, **Zuleica e Delcides**, por toda oração e torcida para realização desse sonho e, é claro, ao meu xodó, meu avô **Nelson**. Amo muito todos vocês!!*

*Ao meu querido avô **Armelindo**, meu bisavô **Antônio** e aos meus saudosos tios, **Carlos e Noquinha**, que, infelizmente, não estão mais neste Plano. Vocês fazem muita falta. Amor eterno por vocês.*

*À minha dupla, amiga de graduação, pós-graduação, “afilhada” e sempre tão parceira: **Giuliana de Campos Chaves Lamarque**. Obrigada, amiga, por todo apoio, por me incentivar e motivar! Você fez a diferença na minha vida. Nossa amizade é pra sempre, já disse isso e faço questão de repetir! Amo você, amiga!*

*À **Beatriz Dantas Marotti** e à **Beatriz Ferreira Carrara**, as “Bias”. 10 anos de amizade é pouco para nós. Obrigada por escutarem os meus desabaços, pelas conversas diárias e por sempre me incentivarem a persistir perante os obstáculos. Muito obrigada por estarem presentes em todos os momentos da minha vida. Amo vocês.*

*Ao **Guilherme Silveira Goulart**, amigo desde o colegial, que torceu por mim e, também, comemorou todas as minhas conquistas comigo. Obrigada pela amizade e parceria.*

*À **Maria Rita Paschoini** que, mesmo distante, sempre torce por mim. Tenho um carinho imenso por você! Obrigada por tudo!*

*À **Nandara Barbosa da Costa**, amiga desde o ensino fundamental e presente até hoje. Obrigada, de coração, pela permanência na minha vida!*

*À **Margarete Fuzaro Alfonso**, que é, para mim, uma amiga, conselheira, cozinheira e uma mãe na mesma pessoa, e ao **Alexys Bruno Alfonso**, que sempre me ajudou*

muito nas disciplinas e a tomar decisões importantes. Vocês são demais! Obrigada por estarem presentes em mais um momento marcante da minha vida e por darem tanto carinho para a minha família!!! Amo vocês!

*Ao Prof. Dr. **José Henrique Rubo**, professor desde o meu segundo ano de graduação e que me orientou nos laboratórios de prótese fixa (e me deixou desesperada algumas vezes – como esquecer?. Não sei se foi graças aos seus puxões de orelha, mas, hoje, a prótese fixa é minha paixão! Obrigada por me dar a oportunidade de ingressar na área acadêmica como sua orientanda e alegrar os dias de clínica e saidinhas do BCCG. Tenho orgulho de fazer parte do seu time!*

*À rubete **Milena Marques**, companheira de mestrado, vizinha de box, que dividiu comigo todos os perrengues e os dias bons na clínica! Super parceira! Ao **Rodrigo Moreira**, minha dupla de Prótese Total, fotografias e desespero e à **Clara Bonachela**, que também dividiu essa jornada comigo e me ajudou muito.*

*Às rubetes, **Brunna Ferrairo e Samira Strelhow**, por todo apoio e ajuda, principalmente, nas primeiras clínicas. Ao novo integrante do BCCG, **Lucas Azevedo**, por trazer ainda mais alegria para nosso grupo.*

*À **Naida Zanini Assem**, colega do doutorado, que me ajudou com a estadia em Araçatuba para realização deste projeto e por me amparar num dia tão difícil. Sempre muito solícita e atenciosa. Muito obrigada!*

*À **Bhenya Ottoni Tostes**, que me orientou em três iniciações científicas com muito carinho e me apresentou o mundo das cerâmicas. Tenho um carinho enorme por você. Obrigada por tudo!!!*

*Ao querido **Rafael Menezes**, por toda ajuda, incentivo e amizade. E por me orientar nas primeiras apresentações do COB, ainda na graduação. Você é demais!*

*Ao Prof. Dr. **Allan Victor Ribeiro**, por todo auxílio e paciência durante a metodologia deste projeto. Por ter abraçado este trabalho comigo e passado dias e dias dentro do*

laboratório para a confecção das imagens.

A todos os professores do departamento de Prótese, em especial à Prof^a Karin Hermana Neppelenbroek, à Prof^a Lucimar Falavinha Vieira e ao Prof. Vinícius Carvalho Porto pela ajuda na disciplina de Prótese Total; ao Prof. Pedro César Garcia de Oliveira, por me auxiliar e enriquecer este projeto durante a qualificação de mestrado; ao Prof. Luiz Fernando Pegoraro, ao Prof. Accácio Lins do Valle e à Prof^a Ana Lúcia Pompéia de Fraga Almeida, pelo auxílio nas clínicas de Reabilitação Oral; e, por fim, ao Prof. Estevam Augusto Bonfante, por abrilhantar nossos seminários com seus conhecimentos.

Aos professores doutores: **Rogério Leone Buchaim, Rafael Francisco Lia Mondelli, Maria Lúcia Rubo de Rezende e Paulo Sérgio da Silva Santos**, por me orientarem durante as iniciações científicas com tanta paciência e zelo.

À Prof^a Dr^a **Ana Flávia Sanches Borges**, por todas as considerações durante a minha qualificação de mestrado.

À **Hebe Freitas**, ajudante das clínicas de pós que sempre me alegrou (até nos dias mais difíceis) durante esses anos. Muito obrigada por tudo. Você é super querida! E a todas as funcionárias da Secretaria de Pós-Graduação da Faculdade de Odontologia de Bauru.

A todos os funcionários do Departamento de Prótese, em especial à **Déborah Andrea Riêra Blasca**, sempre tão solícita e paciente, e à **Cleide Vital Martins**, que sempre me recebeu com um sorriso no rosto.

À **Charlene**, do Departamento de Dentística e Materiais Odontológicos, por cristalizar as minhas amostras.

À **Faculdade de Odontologia de Bauru da USP**, por todo conhecimento de excelência que recebi. Tenho orgulho de ser “uspiana” e por ter minha formação ligada a esse lugar tão maravilhoso.

*Às minhas queridas pacientes, **d. Ana, d. Irene e d. Inês**, pelo apoio, confiança e paciência durante os atendimentos.*

*Ao **CNPq**, Conselho Nacional de Desenvolvimento Científico e Tecnológico, pelo auxílio recebido para realização deste trabalho.*

A todos os alunos de mestrado e doutorado do Departamento de Prótese e Periodontia da FOB-USP.

*A todos os pós-graduandos que participaram da minha graduação e formação. Um agradecimento especial à **Samira Salmeron** por toda ajuda!*

Aos professores membros da banca por aceitarem meu convite e enriquecer este trabalho.

*“Conheça todas as teorias,
Domine todas as técnicas,
Mas, ao tocar uma alma humana,
Seja apenas outra alma humana”*

Carl G. Jung

ABSTRACT

ABSTRACT

Lithium Disilicate glass-ceramic surface analysis by Atomic Force Microscopy and Optical Microscopy

Objective: The primary objective of this study was to evaluate the roughness of CAD/CAM blocks of lithium disilicate glass-ceramic (IPS e.max CAD, Ivoclar Vivadent®, Schaan, Liechtenstein), before and after surface treatment with hydrofluoridric acid 10% and after silane coating by Atomic Force Microscopy and Optical Microscopy. The average size of silane as well as its fit to the valleys after acid etching was also studied.

Methods: Ten specimens of CAD/CAM blocks of lithium disilicate glass-ceramic were analyzed by Atomic Force Microscopy. Before surface treatment with hydrofluoridric acid 10%, the specimens were also analyzed by Atomic Force Microscopy. All specimens were also analyzed by Optical Microscopy after the application of a layer of silane. Free software Gwyddion version 2.51 was used on data processing. The 3D surface measurements were: root mean square roughness, average roughness, maximum peak height, maximum pit depth, maximum height, Skewness and Kurtosis of surface and profile roughnesses. Images provided by Optical Microscopy were used to calculate the size of the silane particle, also using the free software Gwyddion version 2.5.1. Non-parametric tests were done by the free software R version 3.5.1.

Results: Surface roughness and profile roughness were numerically different, but both increased after acid etching. All the skewness measurements concentrated around zero, indicating a more symmetrical behavior after acid conditioning. Silane cross-sectional area measured from $0,0374\mu\text{m}^2$ to $0,424351\mu\text{m}^2$ and its radius ranged from $0,115\mu\text{m}$ to $0,3675\mu\text{m}$ and could fit in on about 77,5% of the conditioned surface valleys.

Significance: Roughness surface and silane layer are important factors to the bond strength between ceramic and resin cements, ensuring the success of restorative treatment.

Keywords: Lithium disilicate glass-ceramic. Hydrofluoridric acid. Silane. Atomic Force Microscopy. Optical Microscopy.

RESUMO

RESUMO

Análise de superfície da cerâmica de Dissilicato de Lítio por Microscopia de Força Atômica e Microscopia Óptica

Objetivo: O principal objetivo deste estudo *in vitro* foi analisar a rugosidade de superfície de blocos de CAD/CAM de cerâmica de dissilicato de lítio, antes e depois do condicionamento com ácido fluorídrico a 10% durante 20 segundos e após a aplicação de uma camada de silano, usando a Microscopia de Força Atômica e Microscopia Óptica. A média do tamanho do silano e como eles se ajustam nos vales da superfície condicionada também foram estudados.

Metodologia: 10 amostras confeccionadas por blocos para CAD/CAM de cerâmica de dissilicato de lítio foram analisadas por um microscópio de força atômica. Depois do tratamento de superfície com ácido fluorídrico a 10% durante 20 segundos, os espécimes foram novamente analisados por Microscopia de Força Atômica. Todas as amostras foram analisadas em microscópio óptico depois da aplicação de uma camada de silano. O software Gwyddion (versão 2.5.1) foi usado para o processamento de dados. As medidas obtidas para as imagens 3D das superfícies foram: raiz quadrada média da rugosidade, rugosidade média, altura do pico mais alto, profundidade do vale mais baixo, máxima altura, assimetria e curtose, tanto para a análise de superfície quanto para a análise do perfil. As imagens obtidas pela Microscopia Óptica foram usadas para calcular o tamanho da partícula de silano, também usando o software Gwyddion (versão 2.5.1.). Testes não-paramétricos foram calculados pelo software R (versão 3.5.1).

Resultados: As rugosidades da superfície e do perfil foram numericamente diferentes, mas ambas aumentaram após o condicionamento ácido. A assimetria concentrou-se próxima a zero, indicando um comportamento mais simétrico após o condicionamento. A área da secção transversal do silano mede entre $0,0374\mu\text{m}^2$ a $0,424351\mu\text{m}^2$ e o seu raio mediu entre $0,115\mu\text{m}$ a $0,3675\mu\text{m}$. Esse tamanho de partícula pode ser acomodado em 77,5% dos vales presentes nas superfícies condicionadas.

Significância: A rugosidade e a camada de silano são fatores importantes para o aumento da união entre a cerâmica e o cimento resinoso, assegurando o sucesso do tratamento restaurador.

Palavras-chave: Dissilicato de lítio. Ácido Fluorídrico. Silano. Microscopia de Força Atômica. Microscopia Óptica.

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1 INTRODUCTION

1 INTRODUCTION

The loss of tooth structure or the loss of one or more teeth causes social disturbances and physical and functional morbidities, impacting the patient's quality of life (KELLY; NISHIMURA; CAMPBELL, 1996).

Currently, dental ceramics are considered effective restorative material for anterior and posterior teeth (SANTOS et al, 2015) because they present favorable properties such as appropriate compressive strength, biocompatibility, tooth-like optical properties, marginal integrity, radiopacity, biomimetism, color stability (AMOROSO et al, 2012), high quality esthetics and clinical longevity (GUESS et al, 2011). Crack propagation, marginal accuracy and wear resistance are limiting factors to their use (CONRAD; SEONG; PESUN, 2007).

The ceramic composition is based on a crystalline phase, involved by a glass phase (GOMES; ASSUNÇÃO; ROCHA; SANTOS, 2008). Dental ceramics can be classified as glass-matrix ceramics, polycrystalline ceramics and resin-matrix ceramics. The glass-matrix ceramics are divided in feldspathic, synthetic (leucite-based, lithium disilicate and derivatives and fluorapatite-based) and glass-infiltrated (alumina, alumina and magnesium and alumina and zirconia). Polycrystalline ceramics are divided in alumina, stabilized zirconia, zirconia-toughened alumina and alumina-toughened zirconia. Lastly, the resin-matrix ceramics are divided in resin nanoceramic, glass-ceramic in a resin interpenetrating matrix and zirconia-silica ceramic in a resin interpenetrating matrix (GRACIS et al, 2015).

Land, in 1903, introduced the first feldspathic ceramic crown (CONRAD; SEONG; PESUN, 2007) used for “jacket” crowns (GOMES; ASSUNÇÃO; ROCHA; SANTOS, 2008), anterior crowns, veneers, inlays and onlays. Feldspathic ceramics present flexural strength between 100-110MPa (AMOROSO et al, 2012). Glass-infiltrated ceramics, such as alumina are indicated for crowns, partial crowns, veneers, anterior and posterior bridges (CONRAD; SEONG; PESUN, 2007) and adhesive prostheses; these porcelain presents flexural strength between 550 to 650 MPa (AMOROSO et al, 2012). Zirconia is more resistant (900-1200MPa)

(AMOROSO et al, 2012) and much less esthetic when compared with glass-matrix ceramics (GUESS et al, 2011). It is used for anterior and posterior ceramic frameworks, anterior and posterior crowns, implant abutments (AMOROSO et al, 2012) and frameworks for implant supported full mouth restorations (SANTOS et al, 2015).

Lithium disilicate glass-ceramic has been widely used in Dentistry in different conditions, such as anterior and posterior (up to second premolar) crowns, veneers, thin veneers, onlays, inlays, three-unit bridges in the anterior region, adhesive prostheses (AMOROSO et al, 2012), implant-supported fixed dental prostheses (TYSOWSKY, 2009) and monolithic restorations (MARTINS et al, 2010) that presents survival rates of 87.9% after 10 years of cementation (KERN; SASSE; WOLFART, 2012). Their flexural strength is around 350-450 MPa (AMOROSO et al, 2012) and these materials are four times more resistant than feldspathic ceramics (MARTINS et al, 2010). Lithium disilicate glass-ceramic is presented in ingots and blocks for CAD/CAM processing (SCHULTHEIS; STRUB; GERDS et al, 2013).

For clinical use, ceramics should receive surface treatments before cementation in order to use adhesive composite resin cements. These treatments are meant to modify the material surface, causing roughness and microporosities which increase adhesion surface area, favoring silane retention, increasing the surface wettability and surface energy. The surface treatment can be done by blasting, laser application and acid conditioning (MALHEIROS; FIALHO; TAVAREZ, 2013).

The acid reacts with the glass-matrix ceramic which is selectively removed, exposing the crystalline phase, leaving a rough surface and increasing the micromechanical retention for adhesive cementation with composite resin cements (CONRAD; SEONG; PESUN, 2007). *In vitro* studies show that hydrofluoridric acid etching modifies the surface of glass-matrix ceramics, because it removes or stabilizes surface defects, increasing roughness topography and adhesive bond (ZOGHEIB et al, 2011). Because the hydrofluoridric acid etching modifies its surface, lithium disilicate glass-matrix ceramic, is called “acid-sensitive” (DE CARVALHO et al, 2011).

A recommended method to increase shear bond strength is the use of silane after hydrofluoridric acid etching (YAVUZ; ERASLAN, 2016). Silane is a bonding agent formed by active organic radical monomers and a water-soluble monovalent

group that promotes a ceramic inorganic phase adhesion with the organic phase of cement on the ceramic surface. Moreover, the silane increases the surface energy of the ceramic substrate and improves the cement damping (DE CARVALHO et al, 2011). Because of these properties, the use of silane is recommended prior to cementing ceramic restorations.

An effective way to analyze qualitatively the effects of hydrofluoridric acid etching and silane coating on ceramic surface is the use of Atomic Force Microscopy (AFM) and Optical Microscopy.

AFM has already been used in the Dentistry to analyze endodontic cements (VALERA et al, 2000), enamel and surface roughness of composite resins (DE OLIVEIRA, 2007), ceramic surface after different surface treatments (such as glaze and polishing with diamond and Shofu points) (VASCONCELLOS, 2003), and primer and acid etching (EL-DAMANHOURY; GAINANTZOPOULOU, 2018). The AFM principles are based on the interaction of atoms present in the Atomic Force Microscope and the specimen atoms. Since all materials are composed of atoms, AFM can produce images from nonconductive or conductive surfaces (PINTO; RAMOS; FONSECA FILHO, 2015) without any specimen preparation, such as metallization. The microscope has a cantilever flexible stem with a microscale tip on its lower side. The AFM analyzes the specimen using a ceramic piezoelectric positioning system that moves on the three main axes (x, y, z) under angstrom precision. For the scanning, the AFM uses an alignment system where a beam laser light affects the cantilever and reflects on a four-quadrant sensor (BERNARDES FILHO; MATTOSO, 2003) that provides the position information to the computer, where the specimen digital topography is built.

Optical Microscopy has been largely used in Dentistry research, such as root canal morphology detection (CRĂCIUNESCU et al, 2016), surface analysis of anchorage miniscrews implants (SEBBAR; BOURZGUI; AAZZAB; ELQUARS, 2011), root resorption studies (FERLINI FILHO, 1999), ceramic analysis (SOUZA; NASCIMENTO; MARTINELLI, 2007) and many other studies.

Since the silane layer is dependent of an optimized surface roughness for adequate bonding, the aim of this *in vitro* study was to compare the lithium disilicate glass-ceramic surface before and after hydrofluoridric acid etching at 10% during 20s, using AFM and analyze the ceramic surface after etching and silanization, using Optical Microscopy.

The null hypothesis is that there will be no differences in roughness before and after acid etching.

2 ARTICLE

2 ARTICLE

The article presented in this Dissertation was written according to the “Dental Materials Journal” instructions and guidelines for article submission (Annex A).

How do silane particles fill roughness on lithium disilicate surface?

ABSTRACT

Objective: The primary objective of this study was to evaluate the roughness of CAD/CAM blocks of lithium disilicate glass-ceramic (IPS e.max CAD, Ivoclar Vivadent®, Schaan, Liechtenstein), before and after surface treatment with hydrofluoridric acid 10% and after silane coating by Atomic Force Microscopy and Optical Microscopy. The average size of silane as well as its fit to the valleys after acid etching was also studied.

Methods: Ten specimens of CAD/CAM blocks of lithium disilicate glass-ceramic were analyzed by Atomic Force Microscopy before and after surface treatment with hydrofluoridric acid 10%. All specimens were also analyzed by Optical Microscopy after the application of a layer of silane. Free software Gwyddion version 2.51 was used on data processing. The 3D surface measurements were: root mean square roughness, average roughness, maximum peak height, maximum pit depth, maximum height, Skewness and Kurtosis of surface and profile roughnesses. Images provided by Optical Microscopy were used to calculate the size of the silane particle, also using Gwyddion version 2.5.1. Non-parametric tests were done by the free software R version 3.5.1.

Results: Surface roughness and profile roughness were numerically different, but both increased after acid etching. All the skewness measurements concentrated around zero, indicating a more symmetrical behavior after acid conditioning. Silane cross-sectional area measured from $0,0374\mu\text{m}^2$ to $0,424351\mu\text{m}^2$ and its radius ranged from $0,115\mu\text{m}$ to $0,3675\mu\text{m}$ and could fit in on about 77,5% of the conditioned surface valleys.

Significance: Roughness surface and silane layer are important factors to the bond strength between ceramic and resin cements, ensuring the success of restorative treatment.

Keywords: Lithium disilicate glass-ceramic. Hydrofluoridric acid. Silane. Atomic Force Microscopy. Optical Microscopy.

1. INTRODUCTION

Dental ceramics are considered effective restorative material for anterior and posterior teeth¹. Ceramics are divided in “Glass-matrix ceramic”, “Polycrystalline ceramic” and “Resin-matrix ceramic” and what differentiate these groups is their composition². Porcelain presents favorable properties such as appropriate compressive strength, biocompatibility, tooth-like optical properties, marginal integrity, radiopacity, color stability³, high quality esthetics and clinical longevity⁴.

Lithium disilicate is a glass-matrix ceramic (presents a glass phase)² and is called “acid-sensitive”, because acid etching modifies the ceramic surface⁵. This ceramic is recommended for anterior and posterior crowns (up to second premolar), veneers, thin veneers, onlays, inlays, three-unit bridges on the anterior region, adhesive prostheses³, implant-supported fixed dental prostheses⁶ and monolithic restorations⁷. Their flexural strength is around 350 to 450 MPa³ being four times more resistant than feldspathic ceramics⁷, the first ceramic used in Dentistry⁸. Lithium disilicate glass-ceramic is presented in ingots and blocks for CAD/CAM processing⁹.

To take advantage of adhesive bond cementation, ceramics must be conditioned. The conditioning can be done by blasting, laser application and acid etching¹⁰. The acid reacts with the glass-matrix of the ceramic which is selectively removed, exposing the crystalline phase, leaving a rough surface and increasing the micromechanical retention for adhesive cementation with composite resin cements⁸. Hydrofluoric acid modifies the material surface, causing roughness and microporosities which increase the adhesion surface area, favoring silane retention, increasing the surface wettability and surface energy¹⁰⁻¹⁴.

Surface treatment can chemically modify the ceramic, as with the use of silane^{15,16}. Silane is a bonding agent that increases the surface energy of the ceramic substrate and improves cement damping⁵. The use of silane after hydrofluoric acid etching before cementation is a recommended method to increase shear bond strength¹⁷⁻¹⁹.

An effective way to analyze qualitatively the effects of hydrofluoric acid etching and silane coating on the ceramic surface is the use of Atomic Force Microscopy (AFM) and Optical Microscopy (OM). AFM has already been used in Dentistry in several studies^{12,20-23}. The microscope analyzes the specimen using a

ceramic piezoelectric positioning system that moves on the three main axes (x, y, z) building 3D images under angstrom precision²⁴. Optical Microscopy has also been largely used in Dentistry²⁵⁻²⁷, providing two-dimensional images of the analyzed surface. Having a better understanding on how the surface treatment modifies the ceramic surface can lead to optimization of resin cement bonding.

The purpose of this *in vitro* study was to compare the lithium disilicate glass-ceramic surface before and after the hydrofluoridric acid etching, using AFM and analyze the ceramic surface after etching and silanization, using Optical Microscopy.

The null hypothesis is that there will be no differences in roughness before and after acid etching.

2. MATERIAL AND METHODS

2.1. Specimen preparation

For preparation of the specimens, CAD/CAM blocks of lithium disilicate glass-ceramic were used (IPS e.max CAD, Ivoclar Vivadent®, Schaan, Liechtenstein). The blocks were cut with diamond disks in 10 specimens (12mm long X 14mm wide X 3mm thick) in a cutting machine (IsoMet® 1000, Buehler, United States). After preparation, all specimens were crystallized in an oven, at a temperature between 840-850°C (1544-1562F). All crystallized specimens were grinded (with 80-, 120-, 150-, 280-, 400- sandpaper granulation) and polished (with 1500- and 2500- silicon carbide paper) in a polishing machine (Politriz Lixadeira Metalográfica “PLF”, Fortel, Brazil) until the surface was smooth and even.

Condensation silicone bases (Fig. 1) were made to keep the specimens with the surfaces as parallel as possible for microscope analysis.

Figure 1: Condensation silicone bases used to hold specimens at microscope

2.2. Atomic Force Microscopy

All specimens were analyzed before surface treatment by an Atomic Force Microscope (Nanosurf Flex AFM, Nanosurf®, Switzerland) with controller (C300, Nanosurf®, Switzerland), characterizing the Control Group (n=10). The parameters used for the Control Group with the C300 Nanosurf Software are presented in Table 1.

Table 1: Parameters used for the Control Group

Z-controller	Image size	50µm
	Time/line	2,4s
	Points/line	512
	Rotation	0
	Setpoint	55%
	P-Gain	4000
	I-Gain	4000
Mode properties	Tip Voltage	0V
	Free	500mV
	Vibration amplitude	

Previously to the second analysis with the AFM, all the specimens were acid etched with hydrofloridric acid 10% (Porcelain Conditioner 10%, Dentsply®, Brazil) for 20 seconds; the acid was removed by a water jet for 20 seconds and air-dried with triple syringe, characterizing the Acid Group (n=10). After this treatment, the

specimens were analyzed by AFM with the C3000 Nanosurf Software. The parameters used are presented in Table 2.

Table 2: Parameters used for the Acid Group

Z-controller	Image size	50μm
	Time/line	1,1s
	Points/line	512
	Rotation	0
	Setpoint	55%
	P-Gain	4000
Mode properties	I-Gain	4000
	Tip Voltage	0V
	Free Vibration amplitude	500mV

2.3. Optical Microscopy

The same specimens used for the Control Group and analyzed by AFM were also analyzed by Optical Microscopy (Axiolab A1 Mat, Zeiss, Germany) in 4 different magnifications: 10x, 20x, 50x and 100x.

A layer of silane coupling agent (RelyX Agent Silane, 3M Espe, Brazil) was applied to all the specimens of the Acid Group. The silane was applied with a brush and, after evaporation, the specimens were analyzed with the Optical Microscope, characterizing the Silane Group.

2.4. Image and Statistical Analysis

Free software Gwyddion version 2.51 was used on imaging analysis. Before calculating roughness measures, data were submitted to background correction and removal of scars. Roughness measures were analyzed in two ways, being one for surface as a whole and another for five profiles chosen randomly.

The considered 3D profile measurements used in this study were: root mean square roughness in nm (Rq), average roughness in nm (Ra), maximum peak height in nm (Rp), maximum pit depth in nm (Rv), maximum height in nm (Rz), Skewness e Kurtosis. To analyze the surface measures, the same measurements were made: Sq, Sa, Sp, Sv, Sz, Skewness and Kurtosis. Zero mean level plans were used as basis to height measures.

For the mensuration of silane particles, 120 measurements were done from 3D images. Assuming that the particles are spherical, the major cross-sectional area of each one was calculated using circle area as basis, taking into account the diameter measured manually with the Gwyddion software, from the Optical Microscope images.

For statistical analysis, non-parametric tests (Friedmann test and Two-sided sign test) were done using the free software R version 3.5.1.

3. RESULTS

3.1. PROFILE AND SURFACE ROUGHNESS

AFM provided 3D images of all samples from the Control Group (Fig. 2) and Acid Group (Fig. 3). Optical microscopy generated 2D images of all specimens of the Control Group (Fig. 4), Acid Group (Fig. 5) and Silane Group (Fig. 6)

Figure 2: AFM image - Control Group

Figure 3: AFM image - Acid Group

Figure 4: OM image - Control Group (100x)

Figure 5: OM image - Acid Group (100x)

Figure 6: OM image - Silane Group (100x)

Aiming to test differences between specimens, a non-parametric Friedmann test considered *Specimen* as the independent variable, and *Group* as the blocking variable to each response was used. The p-values of the effect of specimen tests are

shown on Table 3 and the results suggest that no effect of specimen was observed for all variables.

Table 3: P-values for Friedmann tests

Variable	Ra	Rq	Rt	Rv	Rz	Ry	Rp	Skewness	Kurtosis
p-value	0,639	0,729	0,5159	0,6163	0,7508	0,5824	0,56	0,5378	0,1789

Due to the small sample size, non-parametric tests were applied to compare the variables behavior before and after conditioning. As some variables presented non-symmetrical distributions, a non-parametric two-sided sign test for matched pairs was used. Table 4 shows the descriptive and p-values of the tests comparing roughness measures observed in control and conditioning groups. The p-values observed for tests involving Ra, Rq, Rz, Rt, Rz and Rv are suggestive of significant differences between the roughness observed in the two moments. The medians indicate that all of the roughness parameters increased after conditioning.

Table 4: Profile roughness parameters of IPS e.max® CAD specimens

Group	Ra (nm)	Rq (nm)	Rt (nm)	Rv (nm)	Rp (nm)	Rz (nm)
Control	9,49 ± 5,13	12,88 ± 7,08	81,63 ± 84,21	49,57 ± 25,11	41,05 ± 27,63	56,58 ± 32,36
Acid	49,87 ± 14,11	61,90 ± 17,27	325,04 ± 84,2	171,19 ± 40,18	153,84 ± 45,66	272,13 ± 73,22
p-value	0,639	0,729	0,5159	0,6163	0,56	0,7508

Graphics 1, 2 and 3 show the profile roughness average (Ra), profile root mean square roughness (Rq) and average maximum height of the profile (Rz). Rq is similar to roughness average, but use the mean squared absolute valleys of profile roughness and is more sensitive to deviations of the mean line, being more sensitive to the presence of discrepant peaks and valleys. Tests shows significant difference in the roughness before and after acid etching (p-value = 0,001953 for both roughness). Rz is the average absolute value of the five highest peaks and the five lowest valleys over the evaluation length. This parameter is more sensible to superficial changes than Ra. Ry considers the highest peak and the lowest valley, but they can be apart on profile. Rz considers several peaks and valleys and provides a more realistic measure of roughness. Median roughness of Control Group was smaller than Acid Group, so the acid etching with HF acid increases the roughness.

Graphic 1: Roughness average (Ra)

Graphic 2: Root mean square roughness

Graphic 3: Average maximum height of the profile

Table 5 shows the values of Skewness and Kurtosis observed for the two groups. Skewness is used to measure the symmetry in relation to the mean line. If the symmetry is greater than zero, the surface presents more peaks than valleys and, if less than zero, the surface is more plan, with predominance of valleys. For skewness, the results are suggestive of no difference in the values observed before and after conditioning ($p\text{-value} = 0,3438$) (Graphic 4). After conditioning, all the skewness measures concentrated around zero, indicating a more symmetrical behavior.

Graphic 4: Skewness

Kurtosis is a property of frequency distribution that fits its relationship with Gaussian distribution. When considering kurtosis measures, the results are suggestive of difference between control and conditioning groups (p -value = 0,001953). The kurtosis decreased after conditioning and the median observed for this variable was 2.7805, a value very near Gaussian's Curve of kurtosis. Even for a very large kurtosis observed for a specimen on control group, the kurtosis approximated to 3 after conditioning, i. e., the kurtosis behavior was very similar independently of the initial value of this parameter (Graphic 5).

Graphic 5: Kurtosis

Table 5: Skewness and Kurtosis values

Group	Skewness	Kurtosis
Control	-0,11 ± 0,68	5,21 ± 3,35
Acid	-0,14 ± 0,14	2,83 ± 0,21
p-value	0,3438	0,001953

The same measures were obtained to surface area. The parameters values are shown in Table 6 and Table 7.

Table 6: Surface roughness parameters of IPS e.max® CAD specimens

Group	Sa (nm)	Sq (nm)	St (nm)	Sv (nm)	Sp (nm)	Sz (nm)
Control	13,95 ± 7,7	18,57 ± 10,57	81,63 ± 84,21	64,13 ± 37,15	64,75 ± 38,7	128,89 ± 75,81
Acid	96,1 ± 28,41	120,64 ± 36,56	325,04 ± 84,2	377,74 ± 132,51	375,14 ± 126,94	752,89 ± 259,45
p-value	0,001953	0,001953	0,001953	0,001953	0,001953	0,001953

Table 7: Skewness and Kurtosis values

Group	Skewness	Kurtosis
Control	0,0925 ± 0,385	0,9759 ± 1
Acid	-0,0832 ± 0,209	- 0,745 ± 0,127
p-value	0,001953	0,001953

Graphics 6, 7 and 8 show the surface roughness average (Sa), surface root mean square roughness (Sq) and average maximum height of the surface (Sz).

Graphic 6: Surface roughness average (Sa)

Graphic 7: Surface root mean square roughness (Sq)

Graphic 8: Average maximum height of the surface (Sz)

3.2. SILANE ANALYSIS

Figures 7 and 8 show histograms relative to the transverse areas and silane particles radius, respectively. The graphics shows that the cross-sectional areas are concentrated between 0 and $5\mu\text{m}^2$ and the radius are concentrated in values less

than 1μ . As the values were concentrated in small ranges, percentiles 25 and 50 (median) were used as typical silane sizes.

Figure 7: Histogram of cross-sectional areas of silane

Figure 8: Histogram of radius of silane

Besides the minimum value of cross-sectional area ($0,0374\mu\text{m}^2$), the analyses considered both the 25 and 50 percentiles, corresponding to $0,424351\mu\text{m}^2$ and $0,882788\mu\text{m}^2$, respectively. These percentiles indicate that 25% of measured areas were less or equal to $0,424351\mu\text{m}^2$ and 50% have values less or equal to

$0,882788\mu\text{m}^2$. For the radius, a minimum of $0,115\mu\text{m}$ was observed, while the values $0,3675\mu\text{m}$ and $0,530\mu\text{m}$ were found for 25 and 50 percentiles, respectively. Based on these percentiles and in the small measures found, silane particles with dimensions according to combinations given in (1) were analyzed.

1. $0,0374\mu\text{m}^2 \leq \text{AREA} \leq 0,424351\mu\text{m}^2$ and $0,115\mu\text{m} \leq \text{RADIUS} \leq 0,3675\mu\text{m}$.
2. $0,424351\mu\text{m}^2 < \text{AREA} \leq 0,882788\mu\text{m}^2$ and $0,3675\mu\text{m} < \text{RADIUS} \leq 0,530\mu\text{m}$. (1)
3. $\text{AREA} > 0,882788\mu\text{m}^2$ and $\text{RADIUS} > 0,530\mu\text{m}$.

In the sequence, for each conditioned specimen, maximum depth valleys were selected in the 3D images and their contours were manually marked in the Gwyddion software, according to Figure 9. After marking process, for each measure combination as showed in (1), a tool available on the software selected those valleys that were able to accommodate silane particles with cross-sectional areas and radius as described earlier. After these selections, the number of valleys selected was counted and, between 120 valleys analyzed, 93 would be able to accommodate silane particles with cross-sectional areas ranging from $0,0374\mu\text{m}^2$ to $0,424351\mu\text{m}^2$ and radius ranging from $0,115\mu\text{m}$ to $0,3675\mu\text{m}$. None of the valleys could accommodate particles with cross-sectional area greater than these values, i. e., the biggest silanes would not fit on the studied valleys.

Figure 9: Measures of deep valleys

4. DISCUSSION

A new classification was published in the literature, divided the dental ceramic in three families: glass-matrix ceramics, polycrystalline ceramics and resin-matrix ceramics. Glass-matrix is subdivided in: feldspathic ceramics, synthetic ceramics (as lithium disilicate) and glass-infiltrated ceramics². The success of dental ceramics is related with a good fracture strength, great marginal adaptation, bond strength, crack propagation and esthetic^{8,28, 29}.

For restorations with glass-ceramic and success bonding with composite resin cements, the use of hydrofluoridric acid (HF) etching is recommended^{30,31}. HF modifies the ceramic surface, because the acid removes the glassy matrix, exposes crystalline structure^{12,32}, increases the roughness¹¹ and surface energy¹³ in acid-sensitive ceramics, as lithium disilicate³³, forming hexafluorosilicates³⁴.

HF acid 5% and 10% are the most usual acids used to change the surface ceramics³⁵. The producer of IPS e.max CAD³⁶ suggests hydrofluoridric acid 5% etching during 20 seconds, but a new study shows that no difference between HF acid 10% and 5% in the same time³⁷ and, for micro-shear bond, HF 10% is higher when compared with the HF 5%³⁴. Furthermore, hydrofluoridric acid 5% etching does not remove enough glassy matrix of lithium disilicate to increase the micromechanical retention of the resin cement in the ceramic surface; in contrast, the HF 10% removes³⁵. Hydrofluoridric acid 10% removes the glass-matrix and exposes only lithium disilicate crystals, creating an irregular surface³³. In addition, a positive correlation between wettability and surface roughness exists, when the roughness increase, the wettability increase too³⁸.

Ra (roughness average) is the most common roughness parameter in the literature³⁹ and measures the height of roughness irregularities. Although widely used, other parameters, such as Rq (root mean square), Ry (maximum peak to valley height of the mean line) and Rz (average distance 5 height peaks and 5 deepest valleys) should be used to complement the results⁴⁰. Other parameters can be used, such as Rp (maximum roughness peak height - measure the greater height above mean line), Rv (maximum roughness valley depth measure the greater depth below the mean line) and Rt (maximum height of the roughness – measures the greater distance between peak to valley).

A study published in 2014 observed increase of Ra and Rq, with a less concentrate acid on the surface of IPS e.max Press³⁰. Although the roughness values were different of our study, there was also a difference between the Ra of the control group and the 5%acid¹⁴. A recent study shows that Ra, Rp and Rv of IPS e.max CAD also increase after acid etching 4.8% when compared with the surface without any treatment¹². These results show that roughness increase independently of the lithium disilicate type and acid concentration. Our results corroborate these studies. However, one study shows that there is no significant difference in the Ra and Rz between the control group and conditioned group, regardless of the concentration of the acid⁴¹.

An article published in 2014 found that the Sq of IPS e.max CAD after HF acid 10% is greater than that found for the control group with significant difference⁴². Similar results were obtained by another study, where the Sa in the IPS e.max surface after HF acid 5% etching during 20s increase when compared with the control group³⁸. Thus, our results also corroborate these publications.

In our study, even the specimens that presented heights asymmetrically distributed in the control group, showed similar behavior in skewness after HF acid etching, suggesting that the surface tends to be symmetrical after conditioning. Roughness surface presents greater values when compared with the values of roughness profile, because when the profile is measured, there is no guarantee that the profiler passes through the highest peak or the lowest valley. For this reason, we decided to analyze both roughness profile and roughness surface to have a broader understanding of the problem. Despite this, both indicate the same result: that the roughness increases after HF acid etching.

A study with AFM images of the IPS Empress 2 presented results similar to ours, showing surface irregularities with peaks and valleys after the acid etching with HF acid 5%⁴³. Similar results were found in others studies^{12,41,42}, using AFM images showing higher peaks and deeper valleys after HF acid etching at 10% during 20s, what may have increased the roughness of the IPS e.max CAD.

The use of silane improves the bond between resin cement and glass-ceramic⁴⁴. When HF acid and silane are used together, the bond strength increases, compared with HF or silane alone^{18,45,46}; acid etching and silane together is the most effective surface treatment for porcelain¹⁵. Because of these favorable properties, the

silane coupling agent is considered the gold standard for adhesion of several types of ceramic⁴⁷, being a recommended method to adhesion⁴⁸. On the other hand, some studies *in vitro* show that silane did not show a significant effect in the adhesion, questioning the use of this coupling agent⁴⁹⁻⁵¹.

According to some authors, silane contain at least one silicon atom⁵², which means that it may take more than one atom in the formation of the particle, but the average particle size is not known, as well as the size of the valleys created by the acid etching on the ceramic surface. Therefore, is not clear how silane stays on the ceramic surface after acid etching neither if the roughness created is appropriate to accommodate the silane particles.

A relevant difficulty found in our study was to use the AFM after the silane coating. For some methodology limitation, the AFM probe could not approximate enough to the coated surface without being damaged, what frustrated the attempt to obtain images of the silane particles in their beds. Such images would make it much easier to analyze how the silane spreads over the surface and whether the roughness created by acid etching was effective or not. For that reason, optical microscopy was used to analyze the surface after silane coating. We are currently working on these limitations in order to have a clearer vision of this subject. Another difficulty of this study was to find information on the silane particle size, what was solved by calculating the average size from the 2D images obtained by optical microscopy.

After our study, that analyzed the relationship between presence of valleys on the conditioned surface and size of the silane particle, it was found that the silane dimension must be more investigated. Only 77,5% of the valleys on the conditioned ceramic surface could accommodate silane. None of the valleys could accommodate particles with cross-sectional area greater than $0,424351\mu\text{m}^2$ neither radius greater than $0,3675\mu\text{m}$, i. e., the biggest silanes would not fit on the studied valleys.

Further studies should be performed to allow a proper visualization of silane accommodation in the valleys as well as the optimization of the silane coating technique to guarantee that the best bond strength can be reached between the ceramic and the composite resin cement.

CONCLUSIONS

Within the limitation of this study, it can be concluded that:

The null hypothesis was rejected, because the roughness of lithium disilicate increases after acid etching. Roughness of IPS e.max CAD surface and profile are numerically different, but suggesting the same result;

Additionally, it was found that silane presents a cross-sectional area ranging from $0,0374\mu\text{m}^2$ to $0,424351\mu\text{m}^2$ and radius ranging from $0,115\mu\text{m}$ to $0,3675\mu\text{m}$ and that silane particle can be accommodated in about 77,5% of the conditioned surface valleys.

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3 Discussion

3 DISCUSSION

Dental ceramics have been used in Dentistry over time because of their favorable properties, for example, marginal integrity, compressive strength, biocompatibility, radiopacity, tooth-like optical properties, color stability, aesthetics, and clinical longevity (GUESS et al, 2011; AMOROSO et al, 2012; MARTINS et al, 2010). The success of these materials is related with a good fracture strength, adequate marginal adaptation, bond strength and esthetic (CONRAD; SEONG; PESUN, 2007; CONTREPOIS; SOENEN; BARTALA; LAVIOLE, 2013; SAILER et al, 2015).

Gracis et al (2015) proposed a new classification for ceramic divided in three families: glass-matrix ceramics, polycrystalline ceramics and resin-matrix ceramics. Glass-matrix is subdivided in: feldspathic ceramics, synthetic ceramics and glass-infiltrated ceramics. Lithium disilicate is classified as a synthetic ceramic according to this new classification.

For restorations with glass-ceramic and success bonding between resin cement and porcelain, the use of hydrofluoridric acid (HF) etching is recommended (XIAOPING; DONGFENG; SILIKAS, 2014; KURSOGLU; MOTRO; YURDAGUVEN, 2013). HF modifies the ceramic surface, because the acid removes the glassy matrix, exposes the crystalline structure (EL-DAMANHOURY; GAINANTZOUPOULOU, 2018; MURILLO-GÓMEZ; PALMA-DIBB; DE GOES, 2018), increases the roughness (BORGES et al, 2003) and surface energy (ADDISON; MARQUIS; FLEMING, 2007; BRENTTEL et al, 2007) in acid-sensitive ceramics, as lithium disilicate (VIDOTTI et al, 2013), forming hexafluorosilicates (MOKHTARPOUR; ALAGHEHMAND; KHAFRI, 2017).

Etching time, temperature and concentration of the acid are related to the efficiency of HF etching in the ceramic surface (SUNDFELD et al, 2015; TIAN; TSOI; MATINLINNA; BURROW, 2014; SUNDFELD et al, 2016a). HF acid 5% and 10% are

the most usual acids used to change the surface of ceramics (SUNDFELD et al, 2016b). The producer of IPS e.max CAD (Ivoclar Vivadent, Schaar, Liechtenstein) suggests hydrofluoric acid 5% etching during 20 seconds, but according to Puppini-Rontani et al (2017) there is no difference between HF acid 10% and 5% in the same time and, for micro-shear bond, the HF 10% is more favorable when compared with the HF 5% (SUNDFELD et al, 2016b). Furthermore, hydrofluoric acid 5% etching does not remove enough glassy matrix of lithium disilicate to increase the micromechanical retention of the resin cement on the ceramic surface; as it HF 10% does (SUNDFELD et al, 2016b). Hydrofluoric acid 10% removes the glass-matrix and exposes only lithium disilicate crystals, creating an irregular surface (VIDOTTI et al, 2013). In addition, a positive correlation between wettability and surface roughness exists, when the roughness increase, the wettability increase too (RAMAKRISHNAIAH et al, 2016).

Ra (roughness average) is the most commonly roughness parameter in the literature (AYAD; ROSENSTIEL; HASSAN, 1996) and measure the height of roughness irregularities. Although widely used, other parameters, such as Rq (root mean square), Ry (maximum peak to valley height of the mean line) and Rz (average distance 5 height peaks and 5 deepest valleys) should be used to complement the results (AYAD; FAHMY; ROSENSTIEL, 2008). Other parameters can be used, such as Rp (maximum roughness peak height - measure the greater height above mean line), Rv (maximum roughness valley depth measure the greater depth below the mean line) and Rt (maximum height of the roughness – measure the greater distance between peak to valley).

Xiaoping et al (2014) observed increase of Ra and Rq, but with less concentrate acid in surface of IPS e.max Press. Although the roughness value were different of our study, there was also a difference between the Ra of the control group and the acid 5% group in the article published by Zogheib et al (2011). A recent study shows that Ra, Rp and Rv of IPS e.max CAD also increase after acid etching 4.8% when compared with the surface without any treatment (EL-DAMANHOURY; GAIANTZPOULOU, 2018). These results show that roughness increase independently of the lithium disilicate type and acid concentration. Our results corroborate these studies. However, Prochnow and collaborates (2017) shows that there is no significant difference in the Ra and Rz between the control group and

conditioned group, regardless of the concentration of the acid.

Erdemir et al (2014) found that the Sq of IPS e.max CAD after HF acid 10% is greater than that found in the control group with significant difference. Similar results were obtained by Ramakrishnaiah and collaborators in 2016, where the Sa in the IPS e.max surface after HF acid 5% etching during 20s increase when compared with the control group. Thus, our results corroborate with these publications.

In our study, even the specimens that presented heights asymmetrically distributed in the control group, showed similar behavior in skewness after HF acid etching, suggesting that the surface tends to be symmetrical after conditioning. Roughness surface presents greater values when compared with the values of roughness profile, because when the profile is measured, there is no guarantee that the profiler passes through the highest peak or the lowest valley. For this reason, we decided to analyze both roughness profile and roughness surface to have a broader understanding of the problem. Despite this, both indicate the same result: that the roughness increases after HF acid etching.

Dilber et al (2012) published a study with AFM images of the IPS Empress 2 and the results were similar to ours, showing surface irregularities with peaks and valleys after the acid etching with HF acid 5%. Erdemir et al (2014), Prochnow et al (2017) and El-Damanhoury and Gaintantzopoulou (2018) shows similar results, using AFM images showing higher peaks and deeper valleys after HF acid etching at 10% during 20s, what may have increased the roughness of the IPS e.max CAD.

The use of silane chemically improves the bond between resin cement and glass-ceramic (BLATZ; SADAN; KERN, 2003). Silane is used in Dentistry since 1977, suggest by Eames et al to repair metal ceramic (EAMES et al, 1977).

When HF acid and silane are used together, the bond strength increases, compared with HF or silane alone (BERGOLI et al, 2016; GARBOZA et al, 2016; GRE et al, 2016). Because of these favorable properties, the silane coupling agent is considered the gold standard for adhesion of several types of ceramic (MATINLINNA; LUNG; TSOI, 2017), being a recommended method to adhesion (COLARES et al, 2013; YAVUS; ERASLAN, 2016). On the other hand, some *in vitro* studies show that silane did not have a significant effect on the adhesion, questioning its use (PANAHA; REZAI; AHMADIAN L, 2008; OLIVEIRA; RAMALHO; OGLIARI; MORAES, 2011; DOS SANTOS et al, 2014).

According to some authors, silane contain at least one silicon atom

(KALAVACHARLA et al, 2015), which means that it may take more than one atom in the formation of the particle, but the average particle size is not known, as well as the size of the valleys created by the acid etching on the ceramic surface. Therefore, is not clear how silane stays on the ceramic surface after acid etching neither if the roughness created is appropriate to accommodate the silane particles.

A relevant difficulty found in our study was to use the AFM after the silane coating. For some methodology limitation, the AFM probe could not approximate enough to the coated surface without being damaged, what frustrated the attempt to obtain images of the silane particles in their beds. Such images would make it much easier to analyze how the silane spreads over the surface and whether the roughness created by acid etching was effective or not. For that reason, optical microscopy was used to analyze the surface after silane coating. We are currently working on these limitations in order to have a clearer vision of this subject. Another difficulty of this study was to find information on the silane particle size, what was solved by calculating the average size from the 2D images obtained by optical microscopy.

After our study, that analyzed the relationship between presence of valleys on the conditioned surface and size of the silane particle, it was found that the silane dimension must be more investigated. Only 77,5% of the valleys on conditioned ceramic surface could accomodate silane. None of the valleys could accommodate particles with cross-sectional area greater than $0,424351\mu\text{m}^2$ neither radius greater than $0,3675\mu\text{m}$, i. e., the biggest silanes would not fit on the studied valleys.

Further studies should be performed to improve the silane particles accommodation in the valleys after applying the silane layer.

4 FINAL CONSIDERATIONS

4 FINAL CONSIDERATIONS

Within the limitations of this *in vitro* study, it was concluded that the roughness of ceramic surface increases after the HF 10% acid etching and AFM is a good method to analyze the ceramic surface. The values of surface roughness are numerically greater than profile roughness of the IPS e.max CAD, and surface roughness provides a broader view of the ceramic surface. Nevertheless, both roughnesses suggested the same result. Silane cross-sectional area measured from $0,0374\mu\text{m}^2$ to $0,424351\mu\text{m}^2$ and its radius ranged from $0,115\mu\text{m}$ to $0,3675\mu\text{m}$. Lithium disilicate valleys after conditioning do not accommodate silane particles with cross-sectional area greater than $0,424351\mu\text{m}^2$ neither radius greater than $0,3675\mu\text{m}$.

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ANNEXES

ANNEX A – Guidelines for Dental Materials:

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[3] Mettam GR, Adams LB. How to prepare an electronic version of your article. In: Jones BS, Smith RZ, editors. *Introduction to the electronic age*, New York: E-Publishing Inc; 2009, p. 281–304.

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[4] Cancer Research UK. Cancer statistics reports for the UK, <http://www.cancerresearchuk.org/aboutcancer/statistics/cancerstatsreport/>; 2003 [accessed 13 March 2003].

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