UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA

VEBER LUIZ BOMFIM AZEVEDO

INFLUÊNCIA DE TÉCNICAS DE CONDICIONAMENTO DE CERÂMICAS CAD/CAM NA RUGOSIDADE DE SUPERFÍCIE, TOPOGRAFIA E RESISTÊNCIA DE UNIÃO DO CIMENTO RESINOSO

INFLUENCE OF ETCHING TECHNIQUES OF CAD/CAM CERAMICS ON SURFACE ROUGHNESS, TOPOGRAPHY AND BOND STRENGTH OF RESIN CEMENT

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

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

Orientadora: Profa. Dra. Vanessa Cavalli Gobbo.

Coorientador: Prof. Dr. Marcelo Giannini

Este exemplar corresponde à versão final da tese defendida pelo aluno Veber Luiz Bomfim Azevedo e orientado pelo Profa. Dra. Vanessa Cavalli Gobbo.

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“Se for possível, está feito! Se for impossível, vamos fazê-lo!; não pergunte do que sou capaz. Apenas dê-me a missão”

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Resumo

Diferentes tratamentos utilizando ácido fluorídrico ou primer cerâmico em diferentes tempos de aplicação, podem influenciar a topografia e a resistência de união de materiais cerâmicos do sistema CAD/CAM. Desta forma, este estudo avaliou a rugosidade de superfície (SA), topografia e resistência de união imediata e de longo prazo de diferentes tratamentos na superfície da cerâmica feldspática (Vitablocks Triluxe [FEL]) e à base de silicato de lítio reforçado por zircônia (Vita Suprinity PC [ZLS]) para CAD/CAM. Duzentas placas (10 mm x 5 mm x 1mm) de FEL e ZLS foram obtidas e submetidas ao condicionamento com ácido fluorídrico (HF – 5% ou 10%) ou aplicação de primer condicionante para cerâmica (MEP - Monobond Etch & Prime) durante diferentes tempos de aplicação (20, 40 ou 60s), de acordo com os grupos: Jateamento (controle); 5%HF 20s; 5%HF 40s; 5%HF 60s; 10%HF 20s; 10%HF 40s; 10%HF 60s; MEP 20s; MEP 40s e MEP 60s. As placas das cerâmicas FEL ou ZLS foram tratadas de acordo os grupos experimentais e dois cilindros de cimento resinoso (1,5 mm de diâmetro x 1,5 mm de altura) foram aderidos a cada placa, e submetidos ao ensaio de resistência de união por cisalhamento (SBS) (n=10), um testado após 24 h e o outro após dezesseis meses de armazenamento em água destilada. A rugosidade de superfície (SA) (n=10) e padrão de condicionamento foram avaliados em microscopia de força atômica (AFM) e eletrônica de varredura (SEM). Os dados foram analisados pela ANOVA dois fatores (rugosidade - SA) e três fatores (cisalhamento - SBS), seguidos pelo teste de Bonferroni ($\alpha = 0,05$). O teste de Pearson verificou a correlação entre a rugosidade e resistência de união dos blocos cerâmicos imediatamente e após armazenamento. O aumento da concentração de HF ou tempo de aplicação (de HF ou MEP) não influenciou significativamente a rugosidade das cerâmicas ($p>0,05$). O jateamento e o MEP 40s promoveram maiores valores de SA para os grupos ZLS e o condicionamento com 10% HF promoveu maior rugosidade para FEL. A resistência de união foi menor para os grupos tratados com MEP e os maiores valores de SBS foram observados para o condicionamento com HF (5% ou 10%) ($p<0.05$). Após 16 meses, todos os grupos mostraram reduções nos valores de SBS, exceto alguns grupos de FEL. Houve correlação positiva entre rugosidade e resistência de união para a cerâmica FEL imediatamente e após armazenamento. As imagens de MEV e AFM revelaram que o condicionamento com HF promoveu maiores alterações na superfície de FEL. Conclui-se que o pré-tratamento proposto para FEL é HF 5% ou 10% por 20 segundos e para ZLS, 10% de HF por 40 segundos.

Palavras-chave: Resistência de união, Projeto auxiliado por computador, Materiais dentários, Cerâmicas odontológicas.

Abstract

Different hydrofluoric acid or ceramic primer treatments and application times might influence surface topography and bond strength of CAD/CAM ceramic systems. Therefore, this study evaluated surface roughness (SA), topography and immediate and long-term shear bond strength (SBS) of different surface pretreatments of CAD / CAM zirconia reinforced lithium silicate (Vita Suprinity PC [ZLS]) and feldspathic glass ceramics (Vitablocs TriLuxe [FEL]). Two hundred slabs (10 mm x 5 mm x 1mm) of FEL and ZLS glass ceramics were obtained and submitted to hydrofluoric acid (HF - 5 or 10%) or ceramic primer (Monobond Etch & Prime - MEP) with different application times (20, 40 and 60s), according to the groups: Sandblasting (control); 5%HF 20s; 5%HF 40s; 5%HF 60s; 10%HF 20s; 10%HF 40s; 10%HF 60s; MEP 20s; MEP 40s and MEP 60s. The ceramic slabs were prepared, treated according to each group and two cylinders of a resin cement (1.5 mm diameter x 1.5 mm height) were bonded to each plate and subjected to the shear bond strength test (SBS) ($n = 10$), one of which was tested after 24 h and the other after sixteen months of water storage. Surface roughness and etching pattern analysis were evaluated by atomic force (AFM) ($n=10$) and scanning electron microscopy (SEM). Data were analyzed by two-way (surface roughness) and three-way (SBS) ANOVA, followed by Bonferroni test ($\alpha = 0.05$). Pearson's test verified correlation between surface roughness and bond strength of the ceramic blocks immediately and after water storage. The HF-concentration and pretreatment time (of HF or MEP) did not significantly increase surface roughness of the ceramics ($p>0.05$). Sandblasting and MEP for 40s showed higher SA values for ZLS groups and 10%HF showed higher roughness for FEL. Bond strength was lower for MEP-treated groups and the greatest SBS values were observed for the HF-treated groups, regardless the acid concentration ($p<0.05$). After sixteen months, SBS values reduced for the groups tested, except for some FEL groups. A positive correlation was found between roughness and bond strength immediately and after water storage for FEL glass ceramic. MEV and AFM images revealed that HF-etching promoted greater surface changes for FEL. In conclusion, the proposed pretreatment of FEL is 5% or 10% HF etching for 20 seconds and for ZLS, 10% HF for 40 seconds.

Key Words: Bond strength, Computer-Aided Design, Dental Materials, Dental ceramics.

SUMÁRIO

1 Introdução

Com o avanço da tecnologia na área Odontológica, houve significativo aumento na utilização de técnicas de desenho e fabricação de peças protéticas assistidas por computador (CAD/CAM) como método para confecções de restaurações cerâmicas em casos com alta demanda estética (Corazza et al., 2013; Ruse e Sadoun, 2014; Alghazzawi, 2016). Ainda, as restaurações cerâmicas em CAD/CAM ganharam popularidade devido às suas propriedades físico-químicas melhoradas (de Carvalho et al., 2015) e ao processo de obtenção dos blocos CAD/CAM. Os blocos do sistema CAD/CAM são produzidos sob condições controladas e oferecem a máxima qualidade em estrutura e composição do material. Portanto, as propriedades mecânicas e ópticas são superiores quando comparadas aos materiais convencionalmente fabricados (Alt et al., 2011).

Cerâmicas são materiais caracterizadas por uma matriz vítrea amorfa que consiste em rede aleatória formada por ligações cruzadas de sílica em disposição tetraédrica incorporadas com quantidades variadas de cristais insolúveis de leucita e feldspato (Dilber et al., 2012). As cerâmicas vítreas feldspáticas são compostas por feldspato de potássio e quartzo, passíveis de condicionamento com ácido fluorídrico, e apresentam adesão química entre a fase inorgânica do material cerâmico, a fase orgânica de agente de ligação e o tecido dentinário. São materiais frequentemente usados como cobertura para estruturas metálicas, restaurações indiretas como *inlays, onlays* e laminados (Yavuz et al., 2013).

Por sua vez, as cerâmicas policristalinas são compostas basicamente por cristais que aumentam a resistência destes materiais. Em contrapartida, a quantidade de cristais em sua composição acentua a opacidade e impede o condicionamento com ácido fluorídrico. Desta forma, são designadas como cerâmicas acidorresistentes e contraindicadas em restaurações que não possuem retenção mecânica (Malheiros et al., 2013; D'Arcangelo et al., 2016). Recentemente, uma nova cerâmica odontológica constituída de silicato de lítio reforçada com dióxido de zircônia (ZLS) foi desenvolvida, sob o argumento de que a incorporação de 8 a 12% de dióxido de zircônia poderia agir como uma fase cristalina reforçando o material; isto é, evitando a propagação de fissuras.

O ZLS representa uma tentativa de unir a resistência mecânica da cerâmica policristalina com a excelente estética das vitrocerâmicas em uma restauração monolítica. Além disso, por ser predominantemente matriz de cerâmica de vidro este material é considerado passível de condicionamento com ácido fluorídrico, ao contrário das cerâmicas policristalinas (Rinke et al., 2015; Al-Thagafi et al., 2016; Elsaka e Elnaghy, 2016; Ramos et al., 2016; Weyhrauch et al., 2016; Rinke et al., 2016; Sato et al., 2016).

O conhecimento atual de adesão dos materiais dentários é baseado em duas teorias: adesão química, ou seja, conexões realizadas pelas interfaces das moléculas; e retenção micromecânica, onde ocorre a adesão como resultado da interpenetração dos componentes nas duas superfícies (Dilber et al., 2012). Desse modo, a adesão entre coroas totalmente cerâmicas e cimentos resinosos apresentam benefícios como melhor retenção, adaptação marginal e resistência à fratura (Cotes et al., 2013). A desejável união entre o cimento e as cerâmicas requer um pré-tratamento, responsável pelo aumento da área de superfície devido à criação de microporosidades, cujo objetivo é aumentar o potencial de retenção micromecânica do cimento e a resistência da restauração (Yavuz et al., 2013).

Entretanto, os dados sobre a resistência de união das recentes cerâmicas CAD/CAM são escassos, especialmente quando diferentes métodos de tratamento da superfície são propostos. Adicionalmente, a resistência de união deve ser estudada não apenas no tempo inicial; porém, por longos períodos de armazenamento, embora até o momento, não há dados na literatura que relatem avaliações por períodos maiores do que 90 dias. Ainda, nenhum estudo determinou a correlação entre a rugosidade promovida pelo pré-tratamento das cerâmicas para o sistema CAD/CAM com a resistência de união.

Portanto, este trabalho determinou a rugosidade de superfície e topografia promovida pelo padrão de condicionamento e a resistência de união por cisalhamento imediata e em longo prazo de uma cerâmica feldspática e o silicato de lítio reforçado por zircônia do sistema CAD/CAM, após diferentes tratamentos de superfície. Ainda, foi determinada a correlação entre rugosidade e resistência de união e os modos de fratura após o teste de resistência de união.

2 ARTIGO:

INFLUENCE OF ETCHING TECHNIQUES OF CAD/CAM CERAMICS ON SURFACE ROUGHNESS, TOPOGRAPHY AND BOND STRENGTH OF RESIN CEMENT

Artigo submetido no periódico JPD

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Abstract

Statement of problem: Different hydrofluoric acid or ceramic primer treatments and application times might influence surface roughness, topography, and immediate and long-term bond strength of CAD/CAM ceramics.

Purpose: To evaluate surface roughness (SA), topography and immediate and long-term shear bond strength (SBS) of different surface pretreatment of CAD / CAM hybrid (Vita Suprinity PC [ZLS]) and feldspathic glass ceramics (Vitablocs TriLuxe [FEL]).

Material and Methods: Two hundred slabs (10 mm x 5 mm x 1mm) of FEL and ZLS CAD/CAM glass ceramics were obtained and submitted to hydrofluoric acid (HF - 5 or 10%) or ceramic primer (Monobond Etch & Prime - MEP) with different application times (20, 40 and 60s), according to the groups: Sandblasting (control); 5%HF 20s; 5%HF 40s; 5%HF 60s; 10%HF 20s; 10%HF 40s; 10%HF 60s; MEP 20s; MEP 40s and MEP 60s. The ceramic slabs were prepared, treated according to each group and two cylinders of a resin cement (1.5 mm diameter x 1.5 mm height) were bonded to each plate and subjected to the shear bond strength test (SBS) (n = 10), one of which was tested after 24 h and the other after sixteen months of water storage. Surface roughness and etching pattern analysis were evaluated by atomic force (AFM) (n=10) and scanning electron microscopy (SEM). Surface roughness data were analyzed by two-way ANOVA and shear bond strength (SBS) by three-way ANOVA, followed by Bonferroni test ($\alpha = 0.05$). Pearson correlation tests verified the correlation between roughness and bond strength of ceramic blocks immediately and after water storage.

Results: The HF-concentration and pretreatment time (of HF or MEP) did not significantly increase surface roughness of the ceramics ($p>0.05$). Sandblasting and MEP for 40s showed higher SA values for ZLS groups and 10%HF showed higher roughness for FEL. Bond strength was lower for MEP-treated groups and the greatest SBS values were observed for the HF-treated groups, regardless of the acid concentration ($p<0.05$). After sixteen months, SBS values reduced for the groups tested, except for some FEL groups. Low and negative correlations between roughness and SBS were found for ZLS immediately (24h) and after water storage for sixteen months, whereas positive correlations were observed for FEL immediately (0.7) and after storage (0.9). MEV and AFM images revealed that HF-etching promoted greater surface changes for FEL.

Conclusions: The most indicated pretreatment of FEL is 5% or 10% HF etching for 20 seconds and for ZLS, 10% HF for 40 seconds.

Key Words: CAD/CAM ceramics, Bond strength, Surface treatment, Self-etching ceramic primer.

Clinical Implications

Hydrofluoric acid etching promotes intense surface changes and superior bond strength of resin cement compared to the ceramic primer (MEP) on glass ceramics surfaces.

Introduction

The use of computer aided designer and manufacture (CAD/CAM) as a method for fabrication of indirect restorations has drastically increased.[1-3] The CAD/CAM ceramic restorations have gained popularity due to their adequate mechanical properties, esthetics and ease of milling.[3] CAD/CAM blocks are manufactured under controlled conditions, producing a material with accurate microstructure. Therefore, it is expected that the mechanical and optical properties of these materials might be superior than those of conventionally sintered and crystalized ceramics.[2]

Conventional glass-ceramics are composed of a main crystalline phase in a vitreous matrix.[4] Due to the great concentration of Si, glass-ceramics are capable of being etched with hydrofluoric acid and the silanization improves the bond strength of the resin cement, due to the formation of the silanol groups (Si-O-H) on the ceramics interface. Feldspathic (FEL) ceramics are frequently used as laminate veneers and as porcelain fused to metal crowns,[5] but with the development of CAD/CAM systems, this material has grown in popularity due to the mechanical properties improvement and decrease of the potential complications and failures promoted by the manufacturing process.[1]

Polycrystalline ceramics such as yttria stabilized tetragonal zirconia polycristal (Y-TZP), are limited from the adhesion perspective as these materials do not present glass matrix that could be etched; therefore the etching step is discarded and the clinical indications in cases of indirect restorations without mechanical retention, are restricted. On the other hand, the high concentration of crystalline phase within the ceramic microstructure, increases its mechanical strength.[6,7]

Novel microstructures like lithium silicate glass-ceramics reinforced with zirconia (ZLS) have been developed in an attempt to combine the flexural strength of the polycrystalline structure (ZiO_2) with the esthetic features provided by glass ceramics in a monolithic restoration. It is expected that zirconia would act as a crystalline phase, reinforcing the ceramic structure, preventing crack propagation.[8-10] Moreover, since ZLS is predominantly a glass ceramic matrix, with 8% - 12% zirconia dioxide, it can be etched with hydrofluoric acid, unlike conventional polycrystalline ceramics.[11-14]

The adhesion concept between mineralized dental tissues and the restorative materials interface is based on two fundamental mechanisms: 1- chemical adhesion, that is the molecular reactions at the interface between the adherent surface and the bonding agent; and 2- micromechanical retention, in which a resin interdiffusion zone is created due to the infiltration of adhesive resins or resin cement.[4] The adhesion between ceramic-based materials and resin cements provides benefits as improved retention, marginal adaptation, marginal sealing and mechanical resistance against fracture.[15,16] However, in order to obtain appropriate adhesion between resin cement and ceramic, surface treatments of ceramics are required.[15,16]

Conventional surface treatment indicated for glass ceramics involves hydrofluoric acid-etching application, increasing the surface area by creating microporosities, followed by the silanization, which is a chemical reaction that forms a covalent Si-O-Si bond.[5] Both phases have been recently combined in a single-step agent with the development of Monobond Etch & Prime (MEP), by Ivoclar Vivadent. MEP contains ammonium polyfluoride, which is an acid salt usually used for etching glasses, creating a rough pattern for micromechanical retention,[17] and a silane methacrylate group. This agent excludes the hydrofluoric acid-etching step[17-22], simplifying the bonding procedure.[18,23] However, due to the existence of different glass ceramics types, it is important to evaluate if MEP would properly prepare the surface of these materials prior to bonding and if the long-term bond strength of resin cement to ceramic is not reduced or compromised.[10,12,16]

Therefore, the purpose of this in vitro study was to evaluate the effect of different surface treatments on roughness, topography and shear bond strength (SBS) of resin cement bonded to CAD/CAM glass ceramics (feldspathic [FEL] and zirconia reinforced lithium silicate [ZLS]). The null hypotheses tested were that different surface treatments would not influence (1) the roughness and topography, as well as (2) the immediate (24 hours) and long-term (sixteen months of water storage) bond strength of a resin cement to FEL or ZLS CAD/CAM materials.

Material and Methods

Specimen preparation and group division

Two ceramic blocks for CAD/CAM were tested: ZLS (VITA SUPRINITY PC, Vita Zahnfabrik) and FEL (VITABLOCS TriLuxe, Vita Zahnfabrik). Commercial names, manufacturers, compositions and batch numbers of all materials are listed on Table 1.

Ceramic blocks were sectioned using a slow-speed diamond-wafering blade (Isomet 1000 Precision Saw; Buehler Co) in order to obtain 200 slabs for each ceramic (10 mm x 5 mm x 1mm). Ceramic samples were wet-ground with silicone carbide abrasives (Norton Abrasives) up to 600-grit, using a polishing machine (Automet 500; Buehler Co). Ceramic slabs were treated for 20 s; 40 s or 60 s with 5% hydrofluoric acid (HF) (IPS Ceramic Etching Gel, Ivoclar Vivadent); 10% HF (Porcelain Conditioner, Dentsply/Sirona) or MEP (Monobond Etch & Prime, Ivoclar Vivadent). Controls consisted of sandblasted ceramics with aluminum oxide (50 µm) using a sandblasting unit (Microetcher II; Danville Engineering Inc.) for 20 s, 10 mm away from the surface at 60 psi. After the treatment, all the samples were washed in ultrasonic bath (ultrasonic cleanser, USC 1400; Unique) in distilled water for 5 min, followed by thorough air-drying. Thus, ten groups for each ceramic were evaluated (n=10): (1) Sandblasting (control); (2) 5% HF for 20 s; (3) 5% HF for 40 s; (4) 5% HF for 60 s; (5) 10% HF for 20 s; (6) 10% HF for 40 s; (7) 10% HF for 60 s; (8) MEP for 20 s; (9) MEP for 40 s and (10) MEP for 60 s.

After HF etching, silane (RelyX Ceramic Primer, 3M Oral Care) followed by an adhesive resin (Scotchbond Multipurpose, 3M Oral Care) were applied to treated ceramics surfaces. For groups with MEP (groups 8 to 10), silane was not used. The surface treatments and adhesive techniques of each experimental group are described on Table 2.

Surface roughness and topography evaluations

Ten ceramic slabs of each material were prepared for surface roughness measurements (n = 10). After sectioning and before the roughness and topography evaluations, the samples were immersed for five minutes in ultrasonic bath (USC 1400; Unique) with distilled water. After cleaning, samples were treated according to the ten groups described previously, except for the application of silane and the bonding resin. Surface roughness analysis was performed using an atomic force microscope (Shimadzu/SPM 9600). The probe was used in contact mode and images of 20 × 20 μm of the surface profile was generated and surface roughness (SA) was calculated according to the Rzjis parameter (ten points means roughness). The surface morphology of the glass-ceramic after treatments was evaluated using a scanning electron microscope (JSM IT 300; JEOL). Ceramic slab samples were fixed on metallic stubs, sputter-coated with gold (SCD 050; Bal-Tec) and micrographs of the indirect material microstructures were taken at 3.000x magnifications.

Shear bond strength test

For SBS test, two circular areas (1.5 mm in diameter), delimited with adhesive tapes served as bonding area for testing bond strength. The areas were treated according to the groups described previously. Silicone molds (Virtual, Ivoclar Vivadent) with internal cylindrical shape hole (1.5 mm diameter and height) were precisely positioned over the tapes, matching with the circular-bonded area.

The orifices were filled up with resin cement (RelyX™ Veneer Cement; 3M Oral Care) and light-cured with LED light (Valo Cordless; Ultradent) for 20 s, resulting in polymerized resin cement cylinders on the ceramic surface. After curing, silicone molds and tapes were carefully removed and the ceramic samples were stored in distilled water at 37°C. One resin cylinder was submitted to SBS test 24 h after water storage, and the other one after sixteen months, with water changed monthly.

After SBS testing, fractured specimens were dried and fixed with cyanoacrylate glue (Super Bonder; Loctite) on the shear test device, attached to the universal testing machine (EZ-Test; Shimadzu). Shear load was applied by an orthodontic wire at the resin cylinder's base with a crosshead speed of 1.0 mm/min, until failure. The force required for failure was recorded in Newton and divided by the surface area (mm^2) to calculate the SBS, in MPa.

Failure mode analysis

The tested specimens were examined with a digital microscope (KH 8700; Hirox) to determine failure modes (140x magnification), classified as: Adhesive failure (A); Cohesive failure within ceramic (CC); Mixed failure within ceramic and resin cement (CR); Mixed failure between adhesive and ceramic (AC); Mixed failure between adhesive and resin cement (AR) or Mixed failure involving adhesive, ceramic and resin cement (ARC). Representative images of the failure's modes were obtained under scanning electron microscope (SEM, JSM IT 300; JEOL, voltage 20.0 kV, beam width 35–60 nm, working distance 10–20 mm). For SEM, the samples were fixed in metallic stubs, sputter-coated with gold (SCD 050; Bal-tec) and micrographs were taken at 55x magnifications.

Statistical analysis

The normality and homoscedasticity assumptions were confirmed by Kolmogorov–Smirnov test ($p > 0,05$). Surface roughness data were analyzed by two-way analysis of variance (factors: *material* and *treatments*) and post-hoc Bonferroni test ($\alpha = 0.05$). The SBS data were analyzed by three-way analysis of variance (repeated measures approach) (factors: *material*, *treatment* and *storage time*) and post-hoc Bonferroni test ($\alpha = 0.05$). Pearson correlation was performed to verify the correlation between roughness and bond strength of ceramic blocks. The statistical analysis was performed by the software SPSS (Version 15.0, SPSS Inc.).

Results

Surface roughness and topography evaluations

The surface roughness means are summarized in Table 3. Sandblasting promoted the highest surface roughness values among surface treatments for ZLS ($p < 0.05$). FEL ceramic surface treatments with 5% and 10% HF exhibited higher roughness than those for ZLS, regardless of the etching time ($p < 0,05$). Only the treatment with MEP 40s yielded higher surface roughness for ZLS compared to FEL ($p < 0.05$).

Figures 1 and 2 exhibit the surface topography and etching patterns obtained under scanning electron and atomic force microscopies, respectively. For ZLS, regular etching patterns were obtained following 5 and 10% HF treatments, while no etching pattern was produced by MEP treatment (Figure 1). Conversely, MEP caused surface alterations for FEL, but porosities and etching patterns were more visible when this ceramic was treated with HF, independent on the etching time. The increase of HF concentration caused more surface changes and a more porous arrangement for both ceramics, especially for FEL. Comparing the effects of HF on surface ceramics, FEL underwent more alterations with the removal of glass-phase than ZLS. Sandblasting promoted similar surface alterations for both ceramics and created an air-abraded topography, but they were different when compared with HF etching and MEP treatment (Figure 2). The images of Figure 2 corroborate with SEM images, showing that MEP produced minor surface alterations compared to 5 and 10% HF etching and sandblasting.

Shear bond strength

The mean SBS values are exhibited in Table 4. There was no statistical difference between ceramics ($p > 0.05$). At 24 h, 5% HF (20 and 60 s) and 10% HF (all etching times) showed higher SBS means than those treatments with sandblasting and MEP (all times) for ZLS ($p < 0.05$). After 16 months, sandblasting and HF etching, regardless of the etching time and acid concentration, presented higher SBS than those obtained with all treatment times of MEP application on ZLS ($p < 0.05$). After 16 months of water storage, the SBS of resin cement to ZLS decreased significantly ($p < 0.05$).

Sandblasting, 5% and 10% HF (all etching times) treatments on FEL ceramics promoted higher SBS than treatment with MEP for 20 and 40 s ($p < 0.05$) at 24 h. After 16 months, sandblasting, 5% HF (all etching times) and 10% HF for 20 s presented higher SBS than those obtained with 10% HF for 40 s and MEP (all times) ($p < 0.05$). The SBS of resin cement to FEL reduced significantly for most of treatments ($p < 0.05$), except for sandblasting and 5% HF for 20 s surface treatments ($p > 0.05$).

Failure mode analysis

Representative images of each failure mode are shown in Figure 3. Failure mode distributions (in %) for all groups tested for SBS are presented in Figure 4. The Figure 3 shows images that represent examples of each failure type: adhesive failure (A), cohesive failure within ceramic (CC), mixed failure within cohesive ceramic and resin cement (CR), mixed failure between adhesive and ceramic (AC), mixed failure between adhesive and resin cement (AR) and mixed failure including all materials involved at the interface (adhesive, ceramic and resin cement -ARC).

According to the Figure 4, adhesive failure (A) occurred for all groups of ZLS and was the most common fracture mode detected when ZLS was treated with MEP, regardless of the application time used. Mixed failure between adhesive and ceramic (AC) was also prevalent when the SBS of resin cement was tested on ZLS surface and this type of failure was observed only for groups treated with HF, regardless of the concentration and etching time.

For FEL groups, high percentage of cohesive failure within ceramic (CC) and mixed failure between adhesive and ceramic (AC) were obtained, however the cohesive failure did not occurred for all groups, including those treated with MEP and specimens tested after sixteen months. Additionally, the long-term water storage also resulted in adhesive failures (A) when FEL was treated with MEP, but the incidence of this failure was not higher than 20%. Water-storage for sixteen months did not change drastically the failure pattern for all ZLS and most of FEL groups. The exception occurred when FEL was treated with MEP for 40s and 60s; these groups exhibited cohesive failure within ceramic (CC) immediately after bonding and after storage, both groups predominantly showed mixed failure between adhesive and ceramic (AC).

Pearson correlation between roughness and bond strength

The Pearson correlation coefficient showed low and negative correlations between surface roughness and bond strength for ZLS immediately (24 h) and after 16 months of water storage: - 0.07 and - 0.09, respectively (Figures 5A and 5B). On the other hand, FEL exhibited positive correlations between roughness and bond strength at 24 h and 16 months of water storage were obtained (0.7 and 0.6, respectively) (Figures 5C and 5D).

Discussion

According to results, sandblasting produced the highest roughness values among ZLS-treated groups while 5% and 10% HF promoted higher roughness for FEL ceramic compared to ZLS groups, regardless of the etching time. In addition, MEP for 40s granted higher surface roughness for ZLS compared to FEL. Therefore, the first null hypothesis was rejected, because surface treatments, including etchants (HF and MEP), times (20, 40 and 60s) and concentrations (5 and 10% HF) influenced the roughness results for both ceramics.

Likewise, other studies showed that HF etching promotes surface topography changes,[17-20] and the increase of HF concentration from 5% to 10% caused deeper changes and more porosities.[17] In the current study, this tendency was also observed for both ceramics, but specially for FEL. In this context, the 10% HF is a more aggressive acidic solution and can dissolve a larger amount of glass phase, creating a rough and retentive surface.[17,26-29]. On the other hand, studies have shown an increase in the surface roughness of the indirect restorative materials submitted to micromechanical sandblasting pretreatment.[5,28-34] This outcome was also observed in the present study for both ceramics, as sandblasting promoted the higher surface roughness values, mainly for ZLS, which presented the highest surface roughness among treatments. Since the ZLS ceramic contains 8-12% of zirconium oxide and zirconia is not affected by HF,[10,11,13,34,35] ZLS is less prone to HF action compared to FEL, as can be seen in the Figure 1 and 2.

No etching pattern on ZLS surface was created by 5% HF for 20s and 40s. Ceramic dissolution and etching pattern was only observed when the application of 5% HF increased to 60s or when 10%HF was applied, independent on the etching time. This CAD/CAM material contains lower volume of glass matrix and increased concentration of highly compacted ceramic crystals than FEL, becoming less etchable. [13,17,35]

Some studies have reported that the increase on surface roughness can simultaneously induce cracks propagation that reduces the resistance of the restoration.[33,36] In addition, increasing the etching time or the concentration of the HF did not significantly increase bond strength of resin cement as observed in this study and others, and it also could adversely affect the mechanical properties of ceramics.[14,18,20,21] As the basic principle of HF or MEP is the removal of the glass matrix and the exposure of the ceramic crystals,[17-19,22,29,33] it can be assumed that up to a certain time, the application of the acid is useful for removal of the ceramic glass matrix. However, after a certain application time, the removal of the glass matrix by acid etching can affect the ceramic structure, reducing the amount of exposed crystals and consequently, decreasing ceramics roughness.

Surface treatments (HF and MEP) affected the SBS of resin cement to FEL and ZLS, both at 24 h and 16 months. Therefore, the second hypothesis was also rejected. There were few differences among ZLS groups etched with 5% and 10% for 20s to 60s at 24 h. In this context, ZLS etching with 5% or 10% HF for 20s seems to be a suitable pretreatment option as it would reduce etching time, exposing minor concentration of ceramic crystals, leading to less damage to the ceramic surface,[14,18,20,21,33,36] without compromising SBS. For FEL, etching with 5%HF for 20s exhibited the lowest SBS values among the groups treated with HF. If 5% is the selected concentration of HF to apply on FEL, then etching time should be for 40 or 60 seconds. However, if one elects to apply 10%HF, the time did not influence on SBS. However, according to the study of Mokhtarpour et al.,[37] etching time should be performed for 20 seconds.

The HF etching increases the ceramic surface energy and wettability prior to the silane application. The surface energy is responsible for improving the chemical bond between adhesive resin or resin luting cement and ceramics. Thus, a specific etching pattern and higher surface energy (lower contact angle) is associated with higher bond strengths,[20] being the best protocol to treat glass ceramics before bonding.

Tribst et al.[20], Román-Rodríguez et al.[23], Cardenas et al.[29] and Wille et al.[38] observed that the use of MEP as a ceramic pretreatment promoted bond strength similar to HF etching. Conversely, other investigations[18-20,22,32] obtained results similar to the present study, attesting higher bond strength when HF is used as pre-treatment of ZLS and FEL.[17,18] This study also showed that MEP promoted lower roughness values for both ceramics, except for the ZLS group submmited to MEP application for 40s. Scherer et al.,[19] reported that ammonium polyfluoride has the potential to change the ceramic topography and this reaction is time-dependent, i.e., longer etching time can create more intense surface alterations, enhancing micromechanical interlocking and, consequently improving bond strength. Conversely, no improvement was observed following the application time when MEP was used.

El-Damanhoury and Gaintantzopoulou[18] have stated that ammonium polyfluoride has milder acidity compared to HF, so a slight alteration on ceramic surface and lower roughness values are expected.[17] The silane system in MEP is based on trimethoxypropyl methacrylate and leaves a chemically bonded thin layer of silane that remains on the surface after washing off with water and air drying.[18] Recent studies using micro-energy dispersive X-ray spectroscopy (EDX), [18,22] found fluoride residue on the ceramic surface after treatment with MEP and this fluoride ions residue could be attributed to the reaction of the material with the glass-phase, producing insoluble silica-fluoride salts, which remains as a deposit on the surface. Besides, Prado et al.,[22] and Tribst et al.,[20] reported that the presence of fluoride in the MEP decreased surface energy of substrate. These findings may explain lower SBS values obtained in this study for both ceramics, when they were treated with MEP.

Thermocycling and/or water storage are aggressive environment conditions that degrades the adhesive interface and decreases bond strength. [19,20,22] Thus, long-term artificial aging is an *in vitro* method to provoke resin hydrolytic degradation of the adhesive interface and resin cement. Several studies have demonstrated that water storage or thermocycling resulted in significant decrease of bond strength between resin cements and ceramic blocks, [14,16,19,20,22,32] corroborating with the results of this study. Methacrylates and filler-polymer interface can be hydrolyzed by water, contributing to the degradation of the polymer chain. In addition, water sorption allows unreacted monomers to be eluted. The reduction of the mechanical properties of the polymer-based materials, make them more susceptible to failure,[39,40] as it could be observed in this study after sixteen months of water storage for all ZLS groups and most of FEL ones. The exception was observed when FEL was sandblasted or etched with 5%HF for 20s.

The SBS test is known to induce cohesive failures within the bonded material due to stress concentration at the location where the load is applied.[40,41] High incidence of cohesive failures within ceramic was observed for FEL, because HF and MEP produced significant surface alterations, which favored the penetration of adhesive resin and resin cement into this ceramic and created a strong interlocking between materials. Various types of mixed failures were reported in this study due to the three materials present at interface (ceramic, adhesive and resin cement). The main mixed failure obtained involved the adhesive layer and resin cement (37%). Regarding the adhesive failure, it might indicate a low interaction between ceramic and resin cement. This type of failure was mainly observed for ZLS and when MEP was used as pretreatment for both ceramics.

The low Pearson correlation coefficient attests the fact that surface roughness and bond strength (immediate or after 16 months water storage) of ZLS has negative correlation (- 0.07; - 0.09, respectively) (Fig. 5A and 5B). Yet, since FEL presents higher amount of glass matrix, it showed higher surface change after etching, demonstrating a positive correlation between roughness and bond strength at 24 h and 16 months of water storage, respectively (0.7; 0.6) (Fig. 5 C-D).

One limitation of this study is that the shear test may underestimate the actual tension due to an irregular tension distribution creating localized high stress areas.[20,41,42] In addition, *in vitro* studies do not accurately reproduce clinical conditions such as occlusal load, saliva, pH, temperature variation and biofilm formation. Therefore, the current findings cannot be directly extrapolated to the clinical situation, however, they can be an indication for future clinical studies to validate the best method of pretreatment of ZLS and FEL.

Based on the results obtained and discussed from surface roughness, topography and immediate and long-term bond strength of ZLS and FEL ceramics submitted to different surface pretreatments we selected 10% HP for 40 seconds as the preferable treatment for ZLS while 5% or 10%HP for 20 seconds could be the pretreatment of choice for FEL ceramics of the CAD/CAM system. This choice was based on the results of all response variables and the fact that they could save time and were the more suitable on keeping long-term SBS.

Conclusion

Surface treatments, including MEP and different HF etching times and concentrations influenced roughness, topography, immediate and long-term bond strengths of resin cement to both ZLS and FEL CAD/CAM ceramics. To save time and considering the effect of long-term water storage on bond strength, this study suggest for 10% HP for 40 s for ZLS, and 5% or 10% HF for 20 s for FEL.

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Tables

Table 1. Materials, manufacturers, compositions and batch numbers used *.

Materials	Manufacturer	Composition	Batch #
Vitablocs TriLuxe (Feldspathic glass ceramic - FEL)	Vita Zahnfabrik	SiO_2 (56–64 wt%), Al_2O_3 (20–23 wt%), Na_2O (6–9 wt%), K_2O (6–8 wt%), CaO (0.3–0.6 wt%), TiO_2 (0.0–0.1 wt%)	34990
Vita Suprinity (Zirconia reinforced lithium silicate glass ceramic - ZLS)	Vita Zahnfabrik	ZrO_2 (8–12 wt%), SiO_2 (56-64 wt%), Li_2O (15-21 wt%), Pigments (< 10 wt%), Various (> 10 wt%)	47530
Porcelain Conditioner 10% (10% HF)	Dentsply Sirona	10% hydrofluoric acid, thickener, colourant, water	332935J
IPS Ceramic Etching Gel (5% HF)	Ivoclar Vivadent	5% hydrofluoric acid, thickener, colourant, water	U41061
Monobond Etch&Prime (MEP)	Ivoclar Vivadent	Butanol (10-<25%), tetrabutylammonium dihydrogen trifluoride (≤10%), methacrylated phosphoric acid ester (3-<10%), bis(triethoxysilyl)ethane (1-<2.5%), silane methacrylate, colourant, ethanol, water	U10661
RelyX Ceramic Primer	3M Oral Care	Ethanol, water, methacryloxypropyltrimethoxysilane	N555194
Scotchbond Multi-Purpose	3M Oral Care	Adhesive (#3): bisphenol a diglycidyl ether dimethacrylate (Bis-GMA), hydroxyethyl methacrylate (HEMA), triphenylantimony	N561539
Relyx™ veneer cement	3M Oral care	Silane treated ceramic (55 – 65 wt%), triethylene glycol dimethacrylate (TEGDMA [10 – 20 wt%]), bisphenol a diglycidyl ether dimethacrylate (Bis-GMA [10 – 20 wt%]), silane treated silica (1 – 10 wt%), reacted polycaprolactone polymer (1 – 10 wt%), titanium dioxide (< 1 wt%), diphenyliodonium hexafluorophosphate (< 0.5 wt%), triphenylantimony (< 0.5 wt%)	N779281

∗ Information obtained from safety data sheet and/or technical data provided by manufacturers.

Table 2. Experimental groups' distribution, surface treatment and application mode (n = 10).

CAD/CAM Ceramics	Treatment	Application Mode
Feldspathic (FEL) or Zirconia reinforced lithium silicate (ZLS)	Sandblasting	Air-abrasion (50 µm aluminum oxide) with a sandblasting unit (Microetcher II; Danville Engineering Inc.) for 10 s, 10 mm away from the surface at 60 psi. Wash the samples in ultrasonic bath (ultrasonic cleanser, USC 1400; Unique) in distilled water for 5 min, followed by thorough air-drying + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose + RelyX™ Veneer cement
	5%HF 20s	Apply HF 5% on the ceramic surface for 20 s and thoroughly wash with water, followed by air-drying + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose + RelyX™ Veneer cement
	5%HF 40s	Apply HF 5% on the ceramic surface for 40 s and thoroughly wash with water, followed by air-drying + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose + RelyX™ Veneer cement
	5%HF 60s	Apply HF 5% on the ceramic surface for 60 s and thoroughly wash with water, followed by air-drying + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose + Relyx™ Veneer cement
	10%HF 20s	Apply HF 10% on the ceramic surface for 20 s and thoroughly wash with water, followed by air-drying + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose + Relyx™ Veneer cement
	10%HF 40s	Apply HF 10% on the ceramic surface for 40 s and thoroughly wash with water, followed by air-drying + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose + RelyX™ Veneer cement
	10%HF 60s	Apply HF 10% on the ceramic surface for 60 s and thoroughly wash with water, followed by air-drying + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose + RelyX™ Veneer cement
	MEP 20s	Actively apply on the ceramic surface for 20 s, let it react for 20 s and wash it with water for 10 s, followed by air-drying + Adhesive-Scotchbond Multipurpose + RelyX™ Veneer cement
	MEP 40s	Actively apply on the ceramic surface for 20 s, let it react for 40 s and wash it with water for 10 s, followed by air-drying + Adhesive-Scotchbond Multipurpose + RelyX™ Veneer cement
	MEP 60s	Actively apply on the ceramic surface for 20 s, let it react for 60 s and wash it with water for 10 s, followed by air-drying + Adhesive-Scotchbond Multipurpose + RelyX™ Veneer cement

Table 3. Roughness means and standard deviations (SD) of CAD/CAM ceramic blocks after surface treatments (in µm).

Treatments	CAD/CAM Ceramics	
	ZLS	FEL
Sandblasting	0.479 (0.1)^{Aa}	0.439 (0.05)^{Ca}
5%HF 20s	0.188 (0.03)^{Ca}	0.470 (0.07)^{Cb}
5%HF 40s	0.155 (0.03)^{Da}	0.456 (0.07)^{Cb}
5%HF 60s	0.039 (0.01)^{Ea}	0.492 (0.1)^{Cb}
10%HF 20s	0.176 (0.04)^{CDa}	0.767 (0.16)^{Ab}
10%HF 40s	0.177 (0.03)^{CDa}	0.830 (0.19)^{Ab}
10%HF 60s	0.169 (0.03)^{CDa}	0.614 (0.09)^{Bb}
MEP 20s	0.111 (0.02)^{Da}	0.136 (0.03)^{DEa}
MEP 40s	0.371 (0.08)^{Ba}	0.158 (0.03)^{Db}
MEP 60s	0.105 (0.02)^{Ea}	0.085 (0.02)^{Ea}

Means followed by different letters (upper case letters compare treatments within the same ceramic and lower-case letters compare ceramics within the same treatment) differ among them (by Bonferroni's test. $p \leq 0.05$). (ZLS: Zirconia reinforced lithium silicate; FEL: Feldspathic; HF: hydrofluoric acid; MEP: Monobond Etch&Prime).

Table 4. Shear bond strength means and standard deviations (SD) of resin cements to CAD/CAM ceramics (in MPa).

Treatments	CAD/CAM Ceramics			
	ZLS		FEL	
	Storage Time			
	24 h	16 months	24 h	16 months
Sandblasting	18.5 (4.5)^{Ca}	10.6 (2.4)^{Bb}	18.2 (3.0)^{ABa}	15.5 (2.9)^{Aa}
5%HF 20s	21.6 (5.2)^{ABa}	10.1 (6.0)^{Bb}	14.4 (2.3)^{Ca}	12.4 (2.7)^{ABa}
5%HF 40s	20.9 (4.9)^{BCa}	11.5 (3.3)^{Bb}	18.1 (2.3)^{ABa}	12.2 (1.9)^{Bb}
5%HF 60s	22.3 (4.3)^{ABa}	12.6 (3.0)^{Bb}	20.4 (4.2)^{Aa}	12.7 (4.2)^{Ab}
10%HF 20s	21.5 (4.6)^{ABCa}	12.4 (3.1)^{Bb}	19.1 (2.1)^{Aa}	14.0 (3.5)^{ABb}
10%HF 40s	23.9 (6.2)^{ABa}	13.3 (4.5)^{ABb}	19.0 (4.6)^{Aa}	10.5 (2.1)^{Cb}
10%HF 60s	24.7 (5.8)^{Aa}	16 (3.0)^{Ab}	19.7 (3.6)^{Aa}	11.4 (3.5)^{BCb}
MEP 20s	10.5 (2.1)^{Ea}	5.6 (1.7)^{Cb}	14.9 (3)^{Ca}	7.2 (2.5)^{Cb}
MEP 40s	14.1 (3.6)^{Da}	5.8 (2.1)^{Cb}	14.0 (3.6)^{Ca}	8.7 (2.0)^{Cb}
MEP 60s	10.8 (1.8)^{Ea}	4.0 (2.0)^{Cb}	15.4 (4.1)^{BCa}	7.7 (2.3)^{Cb}

Means followed by different letters (Upper case letters compare treatments within the same storage time and ceramic. Lower-case letters compare storage time within the same ceramic blocks and treatment) differ among them (by Bonferroni's test. $p \leq 0.05$). (ZLS: Zirconia reinforced lithium silicate; FEL: Feldspathic; HF: hydrofluoric acid; MEP: Monobond Etch&Prime).

Figures

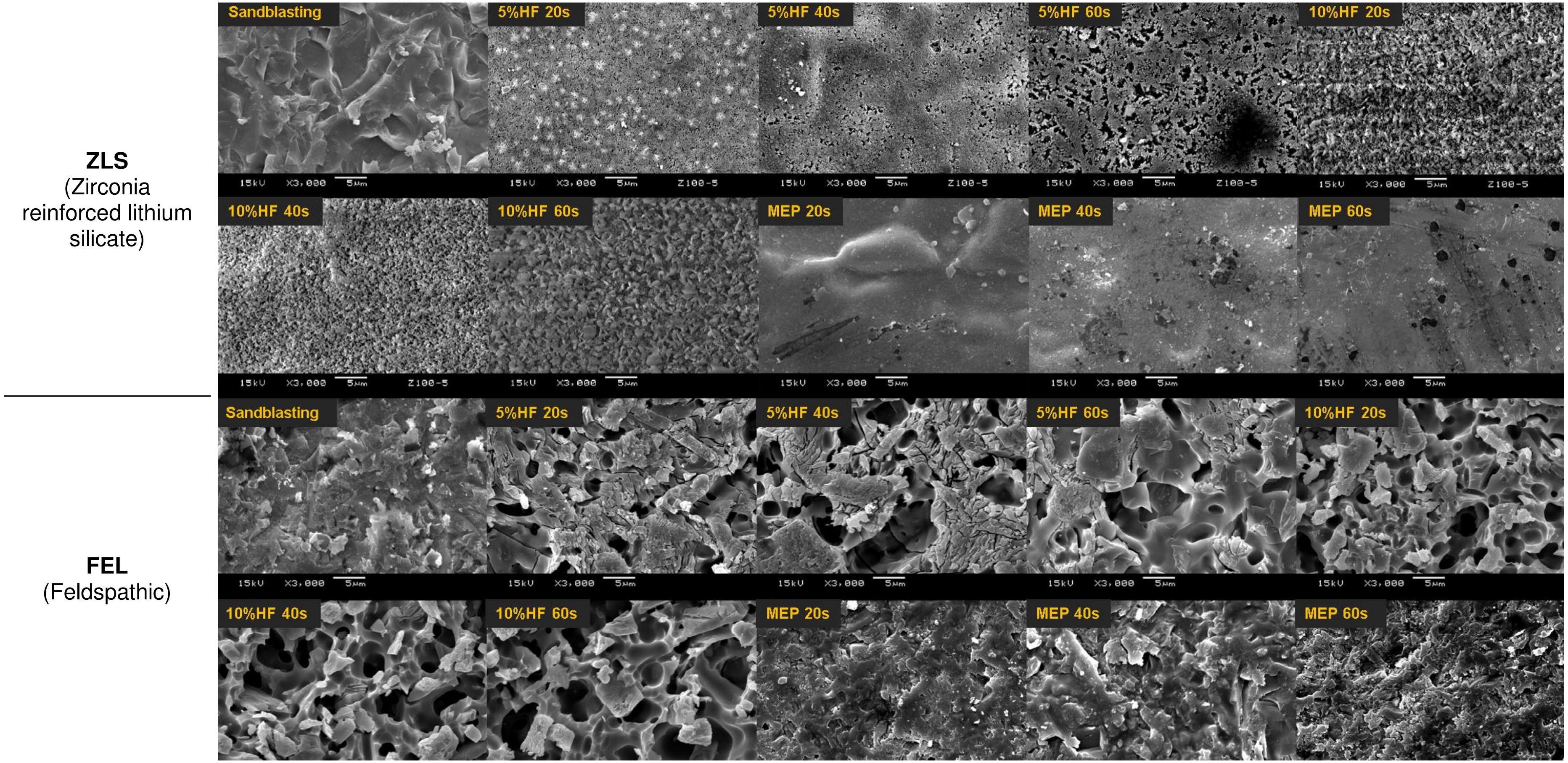

Figure 1. Representative SEM micrographs of ZLS and FEL surfaces after treatments at x3000. Air-abrasion with 50 µm aluminum oxide for 10 s (Sandblasting); HF 5% on the ceramic surface for 20 s (5%HF 20s); HF 5% on the ceramic surface for a0 s (5%HF 40s); HF 5% on the ceramic surface for 60 s (5%HF 60s); Apply HF 10% on the ceramic surface for 20 s (10%HF 20s); HF 10% on the ceramic surface for 40 s (10%HF 40s); HF 10% on the ceramic surface for 60 s (5%HF 20s); Actively apply on the ceramic surface for 20 s, let it react for 20 s (MEP 20s); Actively apply on the ceramic surface for 20 s, let it react for 40 s (MEP 40s); Actively apply on the ceramic surface for 20 s, let it react for 60 s (MEP 60s). (HF: hydrofluoric acid; MEP: Monobond Etch&Prime).

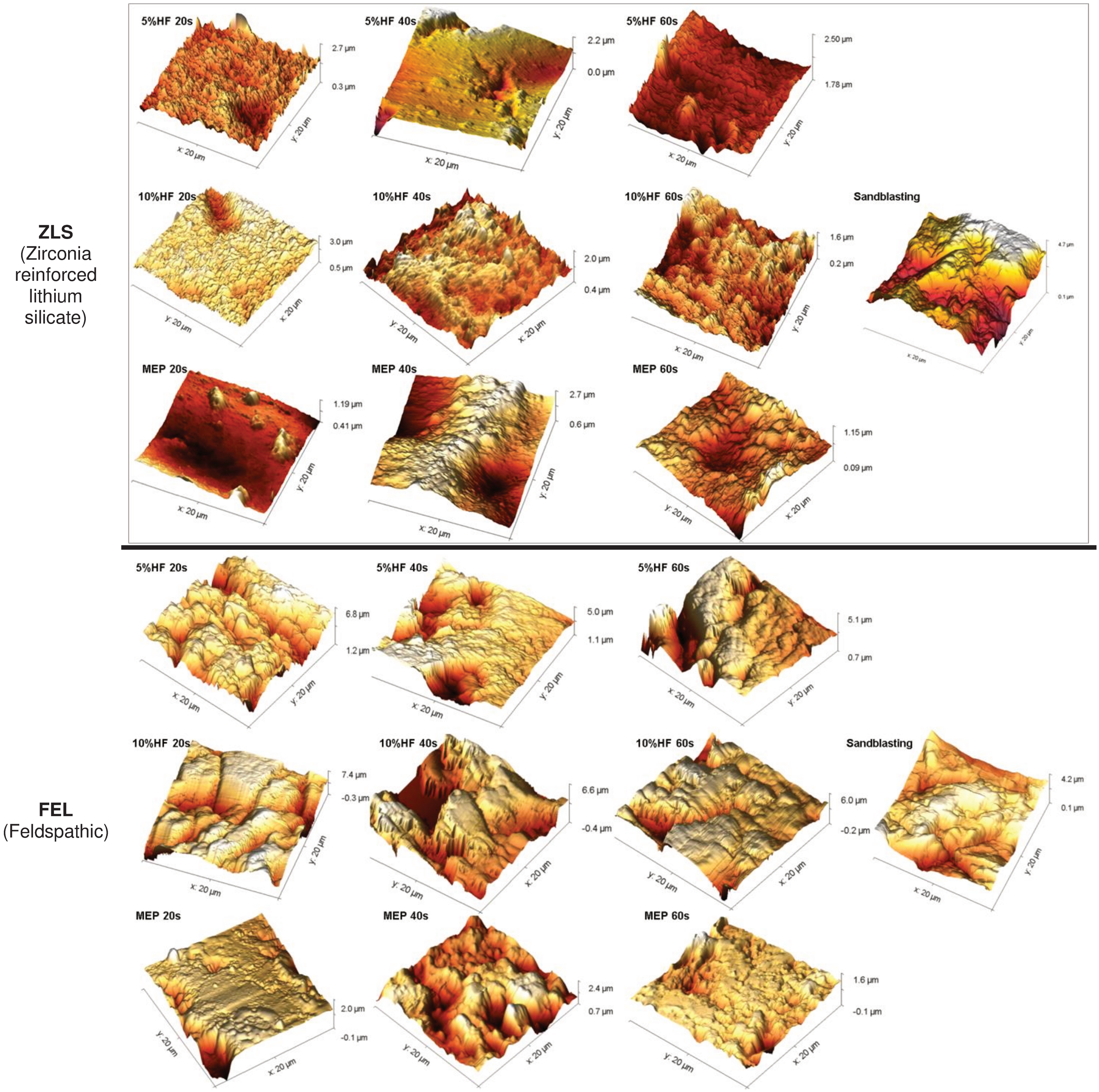

Figure 2. Representative AFM micrographs of ZLS and FEL surface treatments (surface profile image of 20 × 20 µm). Air-abrasion with 50 µm aluminum oxide for 10 s (Sandblasting); Etching with HF 5% on the ceramic surface for 20 s (5%HF 20s); Etching with HF 5% on the ceramic surface for 40 s (5%HF 40s); Etching with HF 5% on the ceramic surface for 60 s (5%HF 60s); Etching with HF 10% on the ceramic surface for 20 s (10%HF 20s); Etching of HF 10% on the ceramic surface for 40 s (10%HF 40s); Etching with HF 10% on the ceramic surface for 60 s (5%HF 20s); Active application of MEP on the surface for 20 s and left undisturbed to react for 20 s (MEP 20s); Active application of MEP on the surface for 40 s and left undisturbed to react for 40 s (MEP 40s); Active application of MEP on the surface for 60 s and left undisturbed to react for 60 s (MEP 60s). (HF: hydrofluoric acid; MEP: Monobond Etch&Prime).

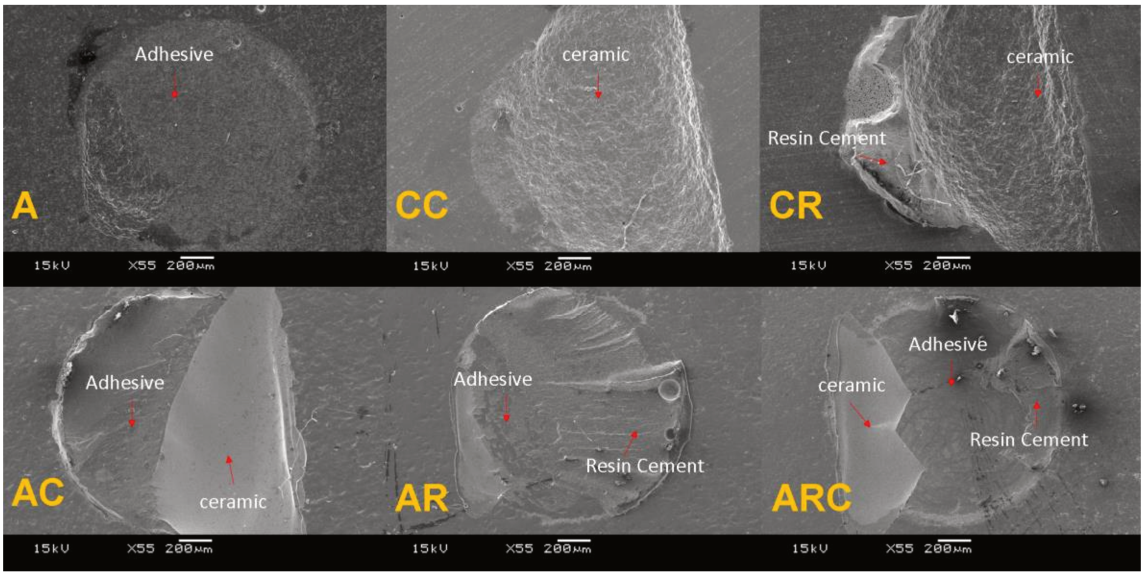

Figure 3. Representative micrographs at x55 magnification of each failure mode: A, adhesive failure; CC, Cohesive failure within ceramic; CR, Mixed failure within ceramic and resin cement; AC, Mixed failure between adhesive and ceramic; AR, Mixed failure between adhesive and resin cement; ARC, Mixed failure involving adhesive, ceramic and resin cement.

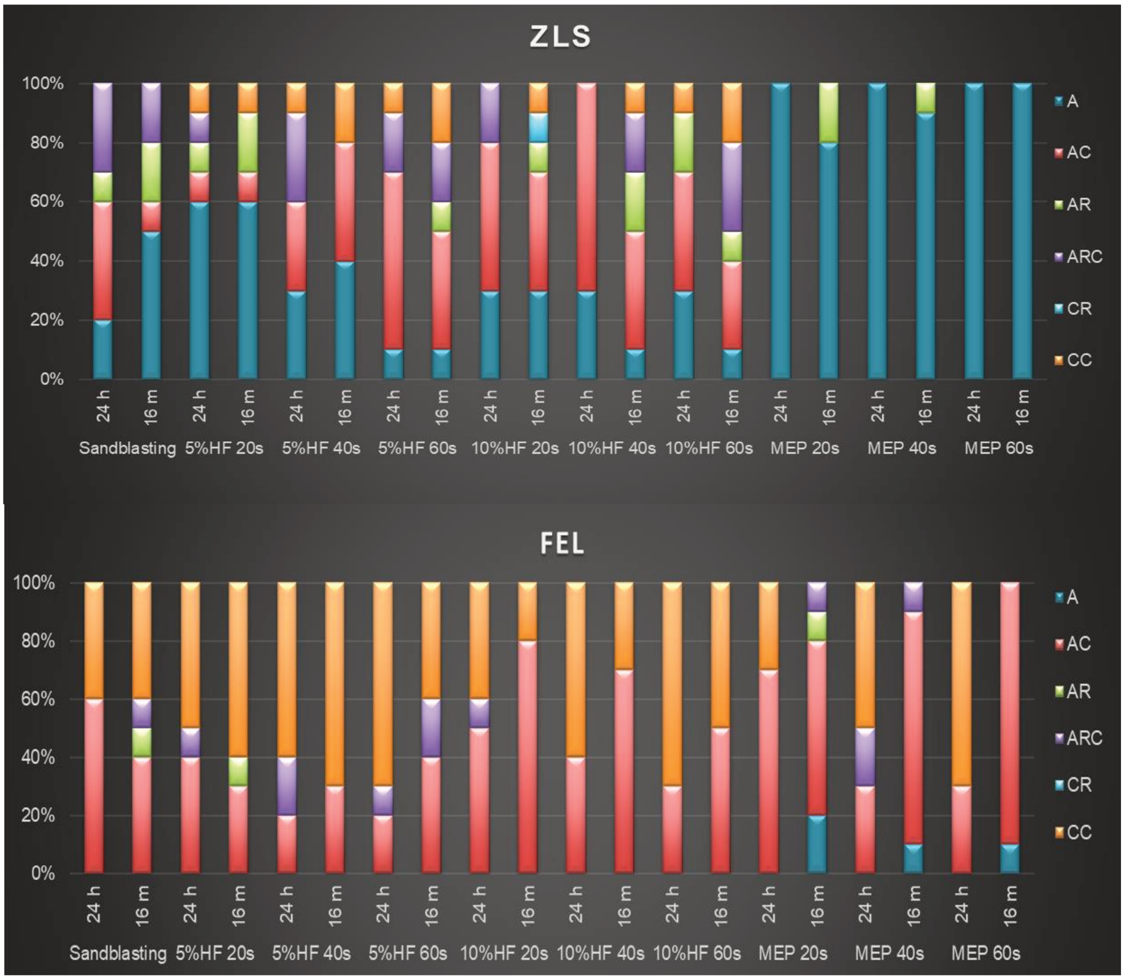

Figure 4. Bar graph presentation of proportional prevalence of fracture modes for resin cements bonded to ceramic blocks. Zirconia reinforced lithium silicate (ZLS); Feldspathic (FEL); Adhesive failure (A); Cohesive failure within ceramic (CC); Mixed failure within cohesive ceramic and resin cement (CR); Mixed failure between adhesive and ceramic (AC); Mixed failure between adhesive and resin cement (AR); Mixed failure involving adhesive, ceramic and resin cement (ARC).

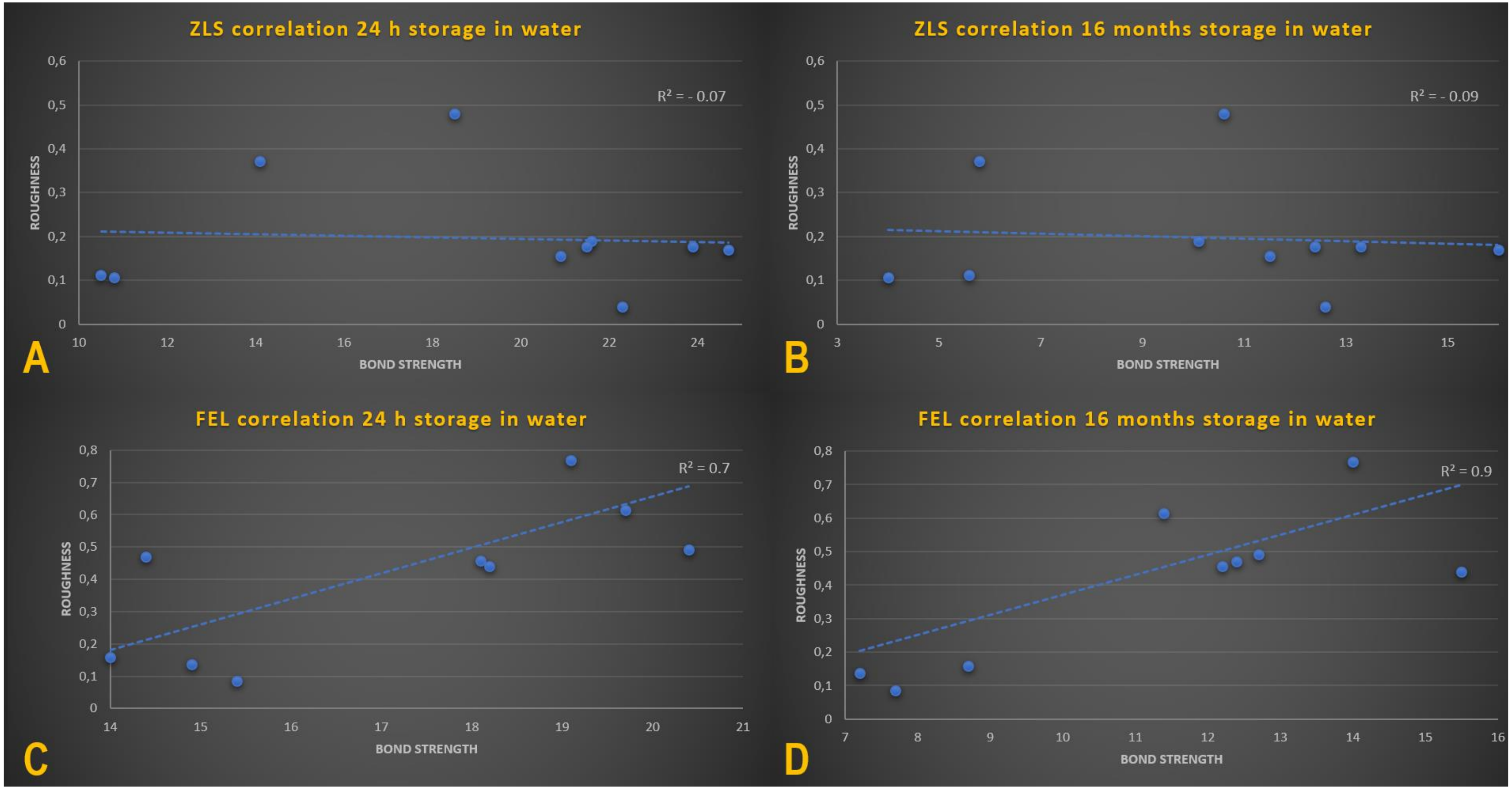

Figure 5. Representative Pearson correlation coefficient (Roughness x Bond Strength) of ZLS (zirconia reinforced lithium silicate) ([A] 24 h and [B] 16 months of water storage); and FEL (feldspatic ceramic) ([C] 24 h and [D] 16 months of water storage).

3 Conclusão

Com base nos resultados deste estudo *in vitro*, o pré-tratamento da superfície influencia a rugosidade, topografia e, resistências de união imediata e de longo prazo da cerâmica feldspática (FEL) e a base de silicato de lítio reforçadas por zircônia (ZLS) do sistema CAD/CAM. O pré-tratamento mais indicado para ZLS é de 10% HF por 40 segundos e 5% ou 10% de HF por 20 segundos para FEL.

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ANEXOS

ANEXO 1 - VERIFICAÇÃO DE ORIGINALIDADE E PREVENÇÃO DE PLÁGIO

INFLUÊNCIA DE TÉCNICAS DE CONDICIONAMENTO DE CERÂMICAS CAD/CAM NA RUGOSIDADE DE SUPERFÍCIE, TOPOGRAFIA E RESISTÊNCIA DE UNIÃO DO CIMENTO RESINOSO

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