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EFEITO DO CONDICIONAMENTO COM DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO NA ADESÃO E NA RESISTÊNCIA À FLEXÃO DE UMA CERÂMICA FELDSPÁTICA

DISSERTAÇÃO DE MESTRADO

Andressa Borin Venturini

Santa Maria, RS, Brasil

2014

EFEITO DO CONDICIONAMENTO COM DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO NA ADESÃO E NA RESISTÊNCIA À FLEXÃO DE UMA CERÂMICA FELDSPÁTICA

Andressa Borin Venturini

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Orientador: Prof. Dr. Luiz Felipe Valandro

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como requisito parcial para obtenção do grau de
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"A mente que se abre a uma nova ideia
jamais voltará ao seu tamanho original."
Albert Einstein

RESUMO

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

EFEITO DO CONDICIONAMENTO COM DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO NA ADESÃO E NA RESISTÊNCIA À FLEXÃO DE UMA CERÂMICA FELDSPÁTICA
AUTORA: ANDRESSA BORIN VENTURINI
ORIENTADOR: LUIZ FELIPE VALANDRO
Data e Local da Qualificação: Santa Maria, 06 de agosto de 2014.

Objetivo: Avaliar o efeito de diferentes concentrações de ácido fluorídrico (HF) no ângulo de contato e na durabilidade da resistência adesiva entre uma cerâmica feldspática e um cimento resinoso, bem como o impacto sobre a rugosidade e resistência à flexão desta cerâmica. **Materiais e Métodos:** Vinte e cinco blocos cerâmicos (VitaBlocks Mark II) (12 x 10 x 2.4 mm) foram produzidos para a análise do ângulo de contato, 40 blocos cerâmicos (12 x 10 x 4 mm) para resistência de união à microtração (MTBS) e 150 espécimes em forma de barra de cerâmica (14 x 4 x 1,2 mm) para avaliar rugosidade e resistência à flexão. Os espécimes foram divididos aleatoriamente em 5 grupos, excluindo o grupo controle para MTBS: SC (controle) - sem tratamento da superfície cerâmica; condicionamento com ácido HF 1% (HF1), 3% (HF3), 5% (HF5) ou 10% (HF10) por 60 s. As medidas de ângulo de contato foram realizadas no Goniômetro e o teste MTBS em uma máquina de ensaio universal, sendo metade dos espécimes de cada bloco testados imediatamente e a outra metade submetida à armazenagem/termociclagem. Todas as amostras em forma de barra foram analisadas em um rugosímetro e carregadas até a falha usando um teste de flexão de três pontos. Os dados foram submetidos à análise estatística. **Resultados:** SC obteve o maior ângulo de contato (61,4° ± 5°), enquanto que HF10 apresentou o menor valor (17,5° ± 4°). Em condições secas, diferentes concentrações de ácido HF promoveram resistências adesivas estatisticamente semelhantes (14,2-15,7 MPa) ($p<0,05$), mas quando os espécimes foram envelhecidos, apenas a adesão do grupo HF1 reduziu estatisticamente (14,5-10,2 MPa). Todos os grupos produziram superfícies significativamente mais rugosas do que o grupo controle (SC) ($p<0,05$). No entanto, os valores médios de resistência à flexão não foram estatisticamente diferentes entre os grupos condicionados (106,47-102,02 MPa). **Conclusão:** Em termos de adesão, a cerâmica testada pode ser condicionada com ácido HF em concentrações de 3%, 5% ou 10%. As diferentes concentrações de ácido não afetaram a resistência à flexão da cerâmica testada. O condicionamento ácido parece ter um efeito de enfraquecimento sobre a superfície cerâmica, se comparado com o grupo não tratado.

Palavras-chave: ácido fluorídrico; cerâmica feldspatica; resistência de união; resistência à flexão.

ABSTRACT

Master's Degree Dissertation
Post Graduate Program in Dental Science
Federal University of Santa Maria

EFFECT OF ETCHING WITH DIFFERENT CONCENTRATIONS OF HYDROFLUORIC ACID ON THE ADHESION AND THE FLEXURAL STRENGTH OF A FELDSPATHIC CERAMIC
AUTHOR: ANDRESSA BORIN VENTURINI
ADVISER: LUIZ FELIPE VALANDRO
Date and Local of Defense: August 06, 2014, Santa Maria.

Purpose: To evaluate the effect of different hydrofluoric (HF) acid concentrations in the contact angle and in the durability of bond strength between feldspathic ceramic and resin cement, as well as the impact on the roughness and flexural strength of this ceramic. **Materials and Methods**: Twenty-five ceramic blocks (VitaBlocks Mark II) (12 x 10 x 2.4 mm) were produced for contact angle analysis, 40 ceramic blocks (12 x 10 x 4 mm) for microtensile bond strength (MTBS), and 150 ceramic bar-shaped specimens (14 x 4 x 1.2 mm) to evaluate the roughness and flexural strength. Specimens were randomly divided into 5 groups, excluding the control group for MTBS: SC (control) - no ceramic surface treatment; etching with HF acid 1% (HF1), 3% (HF3), 5% (HF5) and 10% (HF10) for 60. The contact angle measurements were performed on a Goniometer and the MTBS test in a universal testing machine. All bar-shaped specimens were analyzed in a profilometry and loaded to failure using a 3-point bending test. Data were submitted to statistical analysis. **Results**: SC had the highest contact angle (61.4°± 5°), whereas HF10 showed the lowest value (17.5° ± 4°). In dry conditions, different HF acid concentrations promoted similar bond strength statistically (14.2 to 15.7 MPa) ($p<0.05$), but when the specimens were aged, only the bond from the HF1 group decreased statistically (14.5 to 10.2 MPa). All groups produced significantly rougher surfaces than the control group (SC) ($p<0.05$). However, the mean flexural strength values were not statistically different among the etched groups (106.47 to 102.02 MPa). **Conclusion:** In terms of adhesion, the tested ceramic can be etched with HF acid in concentrations of 3%, 5% and 10%. Different acid concentrations did not affect the flexural strength of the tested ceramic. Acid etching appear to have a weakening effect on the ceramic surface, if compared to the untreated group.

Keywords: hydrofluoric acid; ceramic feldspathic; bond strength; flexural strength.

SUMÁRIO

1. INTRODUÇÃO

Recentemente, as abordagens restauradoras minimamente invasivas foram impulsionadas pela evolução dos sistemas adesivos, dos cimentos resinosos e das cerâmicas odontológicas. Os preparos parciais mais conservadores são considerados menos retentivos e mais adesivo-dependentes (BOTTINO et al., 2009). Nesse contexto, o desempenho clínico das restaurações indiretas feitas de cerâmicas baseadas em sílica (cerâmica feldspática) depende de técnicas adesivas eficientes e duradouras, tanto a esses materiais restauradores quanto aos tecidos dentais (HAYASHI et al., 2000; FRADEANI et al., 2002)

O processo adesivo das cerâmicas baseadas em sílica aos cimentos resinosos parece estar bem estabelecido, visto que a união é proporcionada pelo condicionamento com ácido fluorídrico, potencializada pelo agente silano. O condicionamento com ácido fluorídrico resulta na formação de micro retenções para gerar uma superfície retentiva micro mecanicamente a materiais resinosos (ROULET & DEGRANGE, 1996; THORDRUP et al., 1999).

Apesar do condicionamento com ácido fluorídrico promover a alteração topográfica de superfície necessária para criar retenção micro mecânica, ainda tem sido debatido sobre o efeito de diferentes concentrações deste ácido e tempos de condicionamento na resistência mecânica de cerâmicas. Alguns estudos relatam que este condicionamento poderia causar um efeito de enfraquecimento sobre a superfície cerâmica (DELLA BONA & ANUSAVICE, 2002; DELLA BONA et al, 2002; ADDISON & FLEMING, 2004; ADDISON et al., 2007). Além disso, uma clara evidência existe entre a natureza das modificações dos defeitos de superfície em função do tempo de condicionamento do ácido fluorídrico e sua concentração (ADDISON et al., 2007).

Nesse sentido, embora o protocolo de adesão mencionado esteja bem estabelecido na literatura, ainda existem controvérsias quanto à concentração ideal de ácido fluorídrico por dois motivos principais: dúvida quanto ao efeito da concentração na adesão aos cimentos resinosos; impacto do ácido na resistência mecânica do material cerâmico. Portanto, a presente dissertação está dividida em dois estudos com a finalidade de melhor abordar tanto o impacto das diferentes concentrações do ácido na durabilidade da resistência adesiva a um cimento resinoso quanto no comportamento mecânico do material cerâmico.

2 – OBJETIVOS

O presente trabalho se propôs avaliar:

- O efeito de diferentes concentrações de ácido fluorídrico no ângulo de contato e na resistência adesiva entre um cimento resinoso e uma cerâmica feldspática;
- A influência da armazenagem e ciclagem térmica na durabilidade da resistência adesiva;
- O impacto dos diferentes ácidos na rugosidade superficial e na resistência à flexão de uma cerâmica feldspática.

Para efeitos de apresentação esta Dissertação intitulada **"Efeito do condicionamento com diferentes concentrações de ácido fluorídrico na adesão e na resistência à flexão de uma cerâmica feldspática"** foi formatada e dividida em dois artigos científicos que serão submetidos à publicação nos periódicos *The Journal of Adhesive Dentistry and Journal of Biomedical Materials Research Part B: Applied Biomaterials*

ARTIGO 1– EFFECT OF DIFFERENT CONCENTRATIONS OF HYDROFLUORIC ACID ON THE CONTACT ANGLE, THE TOPOGRAPHICAL CHANGES, AND THE RESIN BOND STRENGTH DURABILITY TO A FELDSPATHIC CERAMIC

ARTIGO 2 – INFLUENCE OF HYDROFLUORIC ACID CONCENTRATION ON THE FLEXURAL STRENGTH OF A FELDSPATHIC CERAMIC

Effect of different concentrations of hydrofluoric acid on the contact angle, the topographic changes, and the resin bond strength durability to a feldspathic ceramic

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Short-title: Adhesion to feldspar ceramic

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Effect of different concentrations of hydrofluoric acid on the contact angle, the topographical changes, and the resin bond strength durability to a feldspathic ceramic

Purpose: To evaluate the effect of different concentrations of hydrofluoric acid (HF) on the contact angle and the resin bond strength durability to feldspathic ceramic. **Materials and Methods**: For the contact-angle analysis, 25 feldspathic ceramic specimens (12 x 10 x 2.4 mm) (VitaBlocks Mark II) were divided into 5 groups (n=5): SC (control) - no ceramic surface treatment, and etching for 60s with various HF concentrations: 1%(HF1), 3%(HF3), 5%(HF5) and 10%(HF10). For bond tests, 40 ceramic-blocks were fabricated (12 x 10 x 4 mm) and subjected to the same surface treatments aforementioned (excluding the untreated-group). The etched-surfaces were silanized and resin cement was applied. After 24h, the blocks were sectioned to produce bar-specimens, which were divided into two conditions (Dry: immediate testing, Aging: storage+thermocycling), and subjected to microtensile testing. Micromorphogical analysis of the treated surfaces was also performed (AFM, SEM). **Results**: SC had the highest contact-angle (61.4°), whereas HF10 showed the lowest value (17.5°). In dry conditions, different acids promoted statistically similar bond-strengths (14.2 to 15.7 MPa) ($p<0.05$); however, after aging, only the bond of the HF1-group decreased statistically (14.5 to 10.2 MPa). **Conclusion**: In terms of adhesion, the tested ceramic can be etched by 3%, 5% or 10% hydrofluoric acid. Topographic changes promoted by these different acids are an important factor for bond improvement to feldspathic-ceramic.

Keywords: acid etching, porcelain, adhesion, topographical changes, wettability.

Clinical relevance: In terms of adhesion, etching of a feldspar ceramic surface using 3%, 5%, or 10% hydrofluoric acid promoted high and stable bonding to resin cement and could be used clinically.

INTRODUCTION

Many aspects of restorative dentistry are being constantly changing, especially the use of ceramic restorations and resin cements. This continuous technological progress has allowed for indirect restorations with a minimally invasive approach, excellent mechanical properties and satisfactory aesthetic results.

The clinical performance of indirect restorations made of silica-based ceramics (such as feldspathic ceramic) depends substantially on a durable bond between resin cements, these ceramic materials and dental tissues.[16,17] For the adhesive cementation procedure, the enamel[24], dentin surfaces[24], and ceramic surfaces[29] must be properly conditioned. The type of etchant, acid concentration and etching time influence the bond strength.[2,3,7,40]

Silica-based ceramics, which are acid-sensitive, undergo surface dissolution by hydrofluoric acid, which selectively attacks the glassy phase of these ceramics, exposing silica oxides (SiO_2) and yielding topographic changes that contribute to micromechanical retention[6] and chemical bonding when using a silane coupling agent and resin cement.[28] The use of only one of these methods, hydrofluoric acid etching or silanization, can be insufficient to promote high and stable bonding.[22,36,39] Silane coupling agents, due to their bifunctional characteristics, are able to promote chemical bonding with organic and inorganic surfaces. Silane provides bonding between the silica oxides present in ceramics to the organic matrix of resin cements through siloxane bonds.[9,10,11,12,21,22,38]

Surface energy is responsible for the physical phenomena associated with the bond strength, which can be naturally or artificially modified by acid etching and silanization[3]. Both conditioning methods promote cement wettability on the ceramic surface,[21,23,29] improving the interfacial contact with resin cements. An increase in the numbers and types of irregularities on etched/silanated ceramic surfaces have been associated with enhancing the bond strength.[29] Thus, increasing the area and the surface free energy reduces the contact angle, facilitates penetration of bonding agents and, consequently, increases the wettability and adhesive potential.[10,29]

Another potential factor that affects adhesion to ceramics is the water absorption by composites, which causes hydrolysis and degradation of the adhesive interface. Water storage and thermocycling have been described as detrimental for the silane-ceramic bond.[27,33] Andreatta Filho et al.[4] reported that the negative effect of thermocycling on bonding can be explained by the fact that materials with different lineal thermal expansion coefficients also presented different degrees of shrinkage and expansion when submitted to thermocycling. Thus, this process promotes the fatigue phenomenon of materials, leading to failure of the bond.[8]

As previously mentioned, ceramic surface etching with hydrofluoric acid provides nano- and micro-morphological alterations that promote mechanical interlocking; on the other hand, acid etching could cause a weakening effect on the ceramic surfaces, with a progressive increase in weakening as a function of the concentration of the hydrofluoric acid used for etching.[1,2,10] There is clear evidence detailing the modifications of surface defects as a function of acid concentration and etching time.[2] This latter study found a significant weakening effect on flexural strength with increasing acid concentrations.

The use of hydrofluoric acid has been questioned due to its possible hazardous and caustic effects to soft tissues.[18,26] Additionally, the use of low concentrations of this acid can be considered as an advantage to the health of patients and, especially, clinicians, who often deal with adhesive cementation.

Therefore, although the adhesion protocol of resin cement to a feldspathic ceramic is well established in the literature, there is still controversy regarding the optimal concentration of hydrofluoric acid necessary to promote durable bond strengths without damaging the health of patients and minimize the weakening effects for the ceramics. The question is: do different hydrofluoric acid concentrations promote different contact angles and differently affect the resin bond strength of feldspar ceramic?

Therefore, this investigation: 1) evaluated the effect of different hydrofluoric acid concentrations on the contact angle and the bond strength between resin cement and a feldspathic ceramic; 2) examined the influence of storage and thermocycling on the bond strength. The research hypotheses were: 1. Surface conditioning of feldspathic ceramic with hydrofluoric acid reduces the contact angle; 2. Different acid concentrations promote different bond strengths; 3. Thermocycling/storage reduces the mean bond strength values.

MATERIALS AND METHODS

Contact Angle Measurement

To accomplish the contact angle evaluation, 25 ceramic slices (12 x 10 x 2.4 mm) were prepared from prefabricated ceramic blocks (VITA Mark II for CEREC/inLab, 2M2C/I12, Vita Zahnfabrik; Bad Säckingen, Germany). The ceramic blocks were sectioned using a diamond disc (ref. 15LC, Buehler; USA) at low-speed, under water cooling, and in a sectioning machine (IsoMet 1000; Buehler; USA). The surface to be analyzed was ground and polished using a sequence of silicone carbide papers . Then, all samples were cleaned in an ultrasonic device (Vitasonic, Vita Zanhfabrik; Germany) using isopropyl alcohol for 10 min.[13]

The ceramic samples were randomly divided into 5 groups (n=5) according to the surface conditioning method (Table 1). The ceramic surfaces were etched using the respective hydrofluoric acid, using the same procedures: etch for 60 s, rinse with air-water spray for 30 s, dry, and ultrasonic cleaning in distilled water for 5 min.

The contact angle was measured using a goniometer (Drop Shape analysis, model DSA 30S, Krüss GmbH; Hamburg, Germany), which was connected to the computer with dedicated software for the assessment of contact angles and surface energy. Under a controlled temperature, one drop of distilled water was put on the center of the untreated and treated ceramic surfaces (Table 1) using a syringe. The contact angle was measured after 5 s.[19]

Microtensile Bond Strength

Forty (N=40) ceramics blocks (12 mm x 10 mm x 4.3 mm) were prepared as described above. Impressions were made from each ceramic block using addition silicone putty (Elite HD, Zhermack, Badia Polesine, Italy; Batch # 122842) to fabricate a mold with a 3 mm gap between the upper portion of the mold and the surface of the ceramic block to allow for the controlled application of resin cement. Thereafter, all blocks were ultrasonically cleaned for 5 min using isopropyl alcohol.

The ceramic blocks were randomly divided into 4 groups (n=10), according to the surface conditioning method (Table 1). The ceramic surfaces were etched with the respective hydrofluoric acids, using the same procedures as described previously: etch for 60 s, rinse with air–water spray for 30 s, dry, ultrasonic cleaning in distilled water for 5 min. Then, the silane coupling agent (ESPE-Sil, 3M ESPE, Minnesota, USA) was applied on all conditioned ceramic surfaces and allowed to sit for 5 min.

Each treated ceramic block was placed in its silicone mold, with the cementation face exposed and untouched. The resin cement (RelyX ARC, 3M/ESPE, Minnesota, USA) was mixed following the manufacturer's instructions and injected onto the treated surface of the ceramic block, using a centrix syringe (DFL; Rio de Janeiro, Brazil).[25,28] The cement in the mold was light polymerized for 40 s (Radii-cal; SDI; Australia). After 10 min, the ceramic block/resin cement assembly was removed from the mold and the cement was once again submitted to light polymerization from five aspects of the block (upper and lateral) for 40 s per side. The blocks were rinsed with water and stored in distilled water at 37°C for 24 h until preparation of the specimens.

The blocks were fixed to a device coupled to the cutting machine (Isomet 1000, Buehler, USA) with the bond surface perpendicular to the diamond disc of the machine (ref. 15LC, Buehler, USA). The first section, measuring approximately 1 mm, was discarded due to the possibility of excess or lack of cement at the interface, which might alter the results.[20,42] Thereafter, six sections that measured approximately 1 mm in thickness were produced. Prior to rotating the specimens 90 degrees for a perpendicular cut to produce five other sections (± 1 mm), light body polyvinylsiloxane (Elite HD, Zhermack; Badia Polesine, Italy, batch # 75011) was used between the slices to minimize stress during cutting. The elimination of the first section was followed for the other five sections; thus, only the inner specimens were used for the experiments. Specimens were of a beam shape, 8mm in length, possessed a non-machined adhesive zone (non-trimmed), and had a bonded area measuring approximately 1 mm^2.

The specimens obtained from each ceramic block were randomly divided into two storage/aging conditions. In the dry condition (Dry), the specimens were submitted to microtensile testing immediately after sectioning. In the aged condition (Aged), specimens were thermocycled (12,000 cycles; 5–55°C; dwell time: 30 s; transfer time: 2 s)[4] (Nova Etica, São Paulo, Brazil) and stored in distilled water at 37°C for 230 days, and then submitted to testing. Considering the “surface conditioning” factor at four levels (HF10, HF1, HF3, HF5), and “storage condition” factor at two levels (dry and aged) (factorial 4 x 2), eight groups were formed (Table 1).

Each specimen was fixed with cyanoacrylate gel (SuperBonder Gel, Loctite; São Paulo, Brazil) to the rods of a device adapted for this test. The specimens were positioned parallel to the long axis of the device in order to reduce the bending stresses. The device was fixed in the universal testing machine (EMIC DL-1000, Santa Maria, Brazil), as parallel as possible in relation to the application of the tensile load, and testing was performed at a crosshead speed of 1 mm/min.

The bond strength was calculated according to the formula: R = F/A; where “R” is the strength (MPa), “F” is the load required for failure of the specimen (N) and “A” is the interface area of the specimen (mm^2), as measured with a digital caliper before the test.

Failure Type Analysis

All specimens submitted to the microtensile test were analyzed under a light microscope (Discovery V20, Carl-Zeiss; Germany) at 50x to 200x magnification.

Some specimens were selected for analysis under a scanning electron microscope (JSM-6360, JEOL, Tokyo, Japan) at 90x, 1000x and 3000x magnification.

Failures were classified into 3 types: 1) predominantly adhesive failure at the interfacial region between the resin cement and ceramic (ADHES); 2) cohesive failure at the cement (COHES-cem); 3) cohesive failure at the ceramic (COHES-cer).

Micro Topographical Analysis Under Scanning Electron Microscopy and Atomic Force Microscopy

Two samples of each group were sputter-coated with gold–palladium alloy prior to being examined under a scanning electron microscope (Jeol-JSM-T330A, Jeol Ltd, Tokyo, Japan) at x1000 and x3000 magnifications. For atomic force microscopy (AFM), images were obtained by atomic force microscopy (AFM) (Agilent Technologies 5500 equipment, Chandler, Arizona, USA). The images (40 µm × 40 µm) were collected using a non-contact mode and PPP-NCL probes (Nanosensors, Force constant = 48 N/m). AFM micrographs were analyzed using scanning probe microscopy data analysis software (Gwyddion™ version 2.33, GNU, Free Software Foundation, Boston, MA, USA).

Data Analysis

The block was used as the experimental unit in the microtensile data analysis (10 blocks per group). Thus, the mean values of the samples from the each block were used for data analysis.

Statistical analysis was performed using the software, Statistix 8.0 for Windows (Analytical Software Inc., Tallahassee, FL, USA). The Anderson-Darling test was applied to test for a normal distribution, and the Bonferroni test was used to verify homoscedasticity. Cohesive failures were excluded from the statistical analysis, since those failures were determined to not represent the real bond strength.

One-way ANOVA and post-hoc Tukey's test were used to evaluate the contact angle data (α=0.05). Bond strength data from dry and aging conditions were separately submitted to one-way analysis of variance (ANOVA) and Tukey test (α=0.05). In addition, the groups were compared 2-2 to elucidate the isolated effect of storage for each surface treatment, using the Student *t*-test (p<0.05).

RESULTS

The Anderson-Darling and Bonferroni tests indicated that the data are considered normal and homogeneous

Contact Angle

One-way ANOVA showed that different conditioning methods had a significant influence (p<0.0001) on the contact angle results; thus, the first hypothesis was accepted.

The unconditioned group (SC) achieved the highest contact angles. The lowest contact angle values were HF1, HF3, HF5 and HF10 (in descending order), which correspond to the conditionings with hydrofluoric acids in 1%, 3%, 5% and 10% concentrations, respectively (Table 2). Figure 2 presents the representative contact angle images for each different surface conditioning.

Micro Topographical Analysis

From the SEM and AFM analysis (Figure 3), higher concentrations promoted deeper and more evident craters and pits, as shown in Figure 3 Q-T (10% HF). However, slight topographic changes were found for 1% (Fig. 3 E-H) when compared to the untreated condition (Fig. 3 A-D).

Bond Strength

Means and standard deviations are presented in Table 3 and Figure 1. One-way ANOVA revealed that the factor “conditioning” ($p= 0.8912$) was not statistically significant for the dry conditions, but it was statistically significant ($p=0.0033$) after aging. For the aged groups, HF1 had the lowest mean bond strength values when compared to the other acid concentrations. Additionally, HF1 was the only group that presented a significant decrease in the bond strength after aging when compared to the results of the similarly treated “dry” group.

Table 4 presents the number and percentages (%) of specimens lost either during thermocycling or cutting prior to microtensile bond strength testing for the eight experimental groups.

The numbers and percentages of the types of failure for the specimens submitted to microtensile testing are presented in Table 5. Failure analysis demonstrated that all groups showed ‘predominantly adhesive’ failures (96.2%), i.e. the majority of fractured surfaces exhibited the presence of some resin cement on the ceramic. SEM micrographs representing the failure types of the debonded specimens are presented in Figure 4.

DISCUSSION

This present study found that hydrofluoric acid etching significantly reduces the contact angle values; therefore, accepting the first hypothesis. Moreover, hydrofluoric acids of 3%, 5% and 10% concentrations promoted similar and stable resin bond strengths to a feldspar ceramic, while the 1% acid generated unstable bond strengths, meaning that the 2nd and 3rd hypothesis were partially accepted.

To promote an effective adhesion between resin cements and ceramics based on silica (feldspathic ceramics), surface conditioning with hydrofluoric acid is necessary for a reaction with the ceramic glass matrix, and thus promoting a micromechanically retentive surface.[6] This surface roughness obtained by acid etching increases the surface energy for silanization, resulting in a greater bond potential of resin cement to a feldspathic ceramic. [7,19]

The contact angle analysis of a liquid on a substrate consists of using a medium to determine to the surface energy and wettability of a given surface.[43] An increase in surface area caused by HF etching induces an increase in wettability[29], which is associated with a lower contact angle and a greater bonding potential.[3]

In this present study, HF etching decreased the contact angle in all groups, with the lowest mean contact angle values found for 10% hydrofluoric acid (Table 2 and Fig 2). The current results are in agreement with a study by Jardel et al.[19], which evaluated the effect of surface modifications with gel 10% HF on the surface energy of two feldspathic ceramics. Those authors concluded that, after conditioning, ceramics showed smaller contact angles when compared to the groups that were only polished. However, despite the lower contact angle values for HF10 when compared to HF3 and HF5, the current results did not find an impact on adhesion, since HF3, HF5, and HF10 promoted similar bond strengths. These results might indicate that the resin adhesion to this variety of ceramic is from the micro mechanical nature, ie, that nano- and micro-topographical changes (Fig 3) play a very relevant role in the adhesion process.

Only the group etched with 1% HF showed a significant decrease in bond strength after aging/thermocycling. Higher bond strengths were obtained when ceramic surfaces were conditioned with HF3, HF5 and HF10, without any statistically significant decrease after aging. Similar results were found by Amaral et al.[3], who observed that, independent of the storage condition, bond strength values were significantly higher for groups etched with 9% and 4% hydrofluoric acid gels. An important aspect in the current investigation is that the tested acids had the same viscosity and were produced by the same manufacturer, which is different from the Amaral at al study, which assessed acids with different concentrations and viscosities[8]. Thus, the present study prevented the possible effect of viscosity on the etching pattern of the ceramic surface to control the effects of different concentrations of acid on the contact angle and adhesion.

Representative micrographs clearly demonstrate the effect of increasing the HF acid concentration on the surface topography. However, no differences were found for the bond strength values between 10%, 5% and 3% concentrations. It is possible that bond strength is related to the effective capacity of the silane coupling agent to promote chemical bonding between the resinous materials and ceramics[2,6,28,38] associated with the topographical changes from etching. The silane coupling agents coat the silica oxides present in the ceramic and bind to the organic matrix of resin cements by means of siloxane bonds.[9,22,38] Brentel et al[6] evaluated the durability of the bond strength between resin cement and a feldspathic ceramic submitted to different etching protocols (10% HF acid gel and 1.23% acidulated phosphate fluoride), with and without silane application. They found that the hydrofluoric acid treatment, followed by silanization of the silica-based ceramic, provided greater bond strength values.

Water storage and thermocycling have been found to be detrimental to the silane-ceramic bond.[32,35] Since resins are permeable to water, the bond between silane and resin composite in the current study was expected to deteriorate over time due to hydrolysis.[22] This deterioration will be lower if proper surface preparation is accomplished prior to treatment with silane, to provide nano- and micro-mechanical retention.[35] Therefore, the lack of roughness and the minimal change in topographic patterns after 1% HF acid etching promoted significantly lower bond strengths when compared to the other acid etching approaches, when aged. The silane, when applied in the HF1 group, could not improve the bond strength values due to insufficient micromechanical retention promoted by the minimal acid etching; consequently, the interfaces did not resist hydrolysis. Therefore, the current findings agree with other authors[32,35], who have declared that surface alteration is crucial for enhancing the bond strength.

Regarding the pre-test failures, these occurred only when cutting the ceramic-cement assemblies and can be seen as a limitation of the test protocol, as well as the presence of cohesive failures. These can be explained by the stresses generated during specimen preparation[41], since the ceramic block has high strength and is resistant to cutting, inducing cracks in the ceramic during sectioning. Moreover, microtensile testing is only applicable when the bond strength values were higher than the approximately 5 MPa that is allowed for sectioning the specimens.[6,37] These features might explain the number of pre-test failures observed in the present study and why the group without conditioning could be not tested, since, in a pilot study, was not possible to produce specimens after cutting the specimens of this group.

With regards to the mechanical testing to evaluate the real bond interaction between the materials/substrates/adherent, microtensile bond strength testing is more appropriate for evaluating the adhesive capabilities, as this kind of test generates more homogeneous stress distribution at the interface than other mechanical tests.[5,14,30] Therefore, this "micro" test has less chance of intrinsic defects in the adhesive interface to affect the results of the test. According to the Griffith's theory, the tensile strength of uniform materials decreases when the specimen size is increased, due to a higher probability of critical sized defects than in smaller specimens.[37,41] Thereby, the failure analysis in this present study demonstrated that the most common failure mode of specimens involved the adhesive interface, indicating more real assessment.

It should be noted that the sample had a small area; therefore, it received a greater influence from the thermocycling effects on its surface, which might have contributed to the decrease in the bond strength values. In vitro studies have inherent limitations and some clinical conditions cannot be simulated. Thus, the current findings should be extrapolated to clinical situations with caution. Further studies that utilize more real clinical situations should be conducted, such as evaluating the effect of hydrofluoric acid concentration on the adhesion to ceramic when acid etching is performed on the intaglio surface of inlay restorations used in posterior teeth and applying mechanical cycling.[15,31,34]

CONCLUSIONS

- The tested ceramic can be etched with HF acid in concentrations of 3%, 5% and 10%, since all of those acids promoted stable bond strengths and relevant topographical alterations.
- Greater concentrations of hydrofluoric acid produce more intense alterations of the surface topography of feldspathic ceramic and smaller contact angles.
- Aging/thermocycling decreased statistically the resin-bond only when the ceramic surface was etched with 1% HF concentration.

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FIGURES

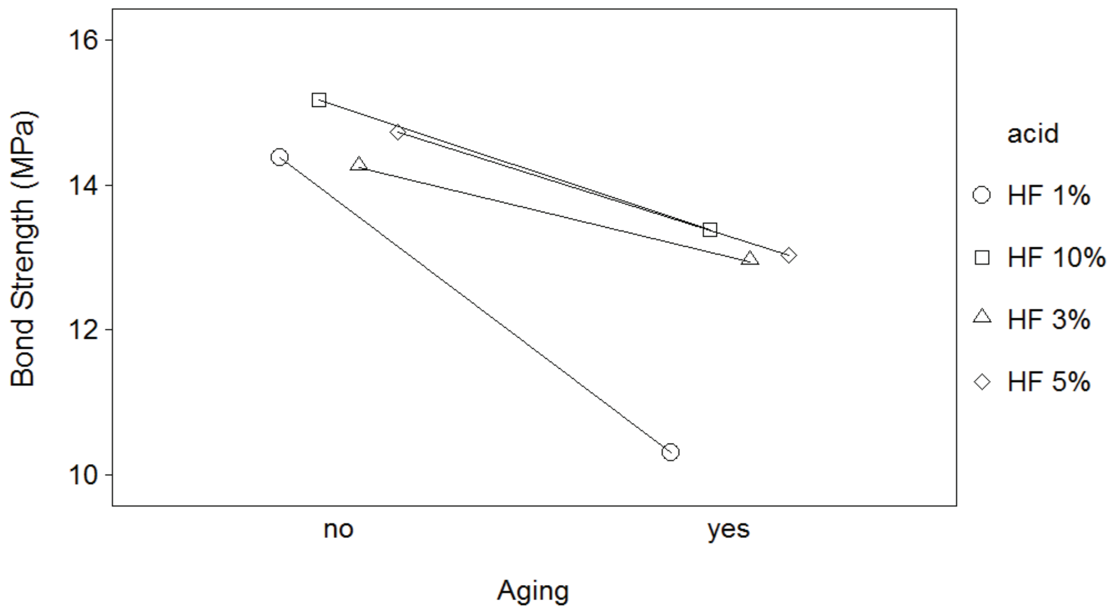


Figure 1 - Means of microtensile bond strength data taking the two factors studied into account.

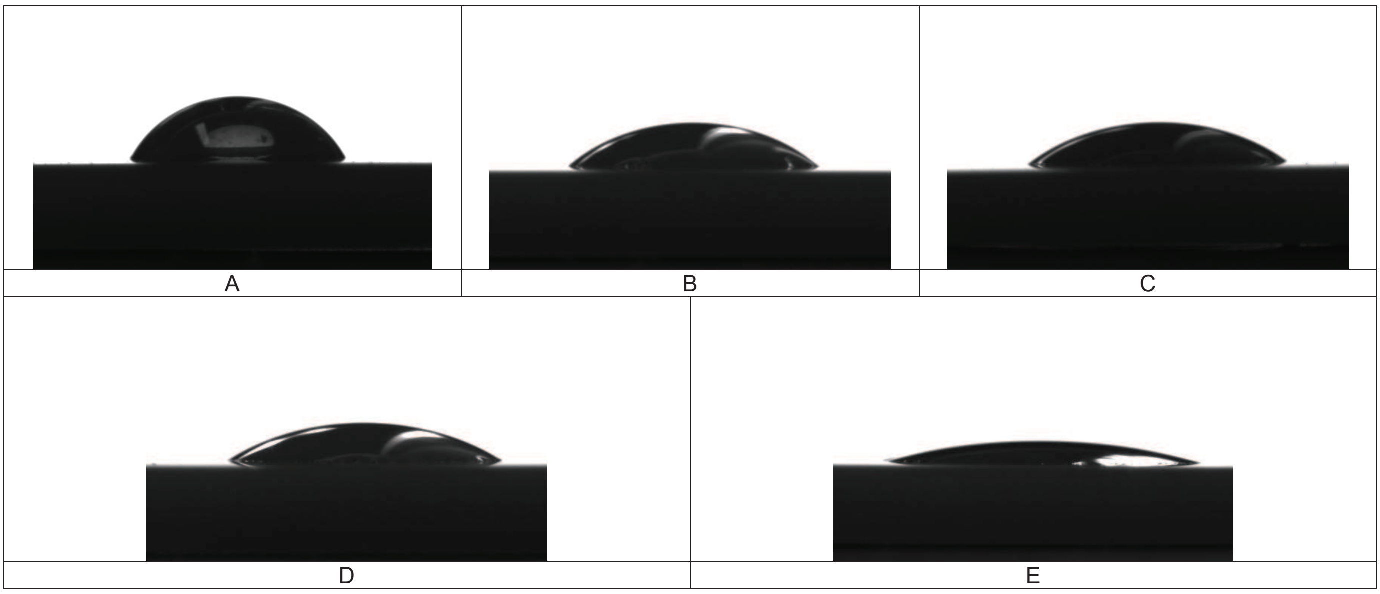
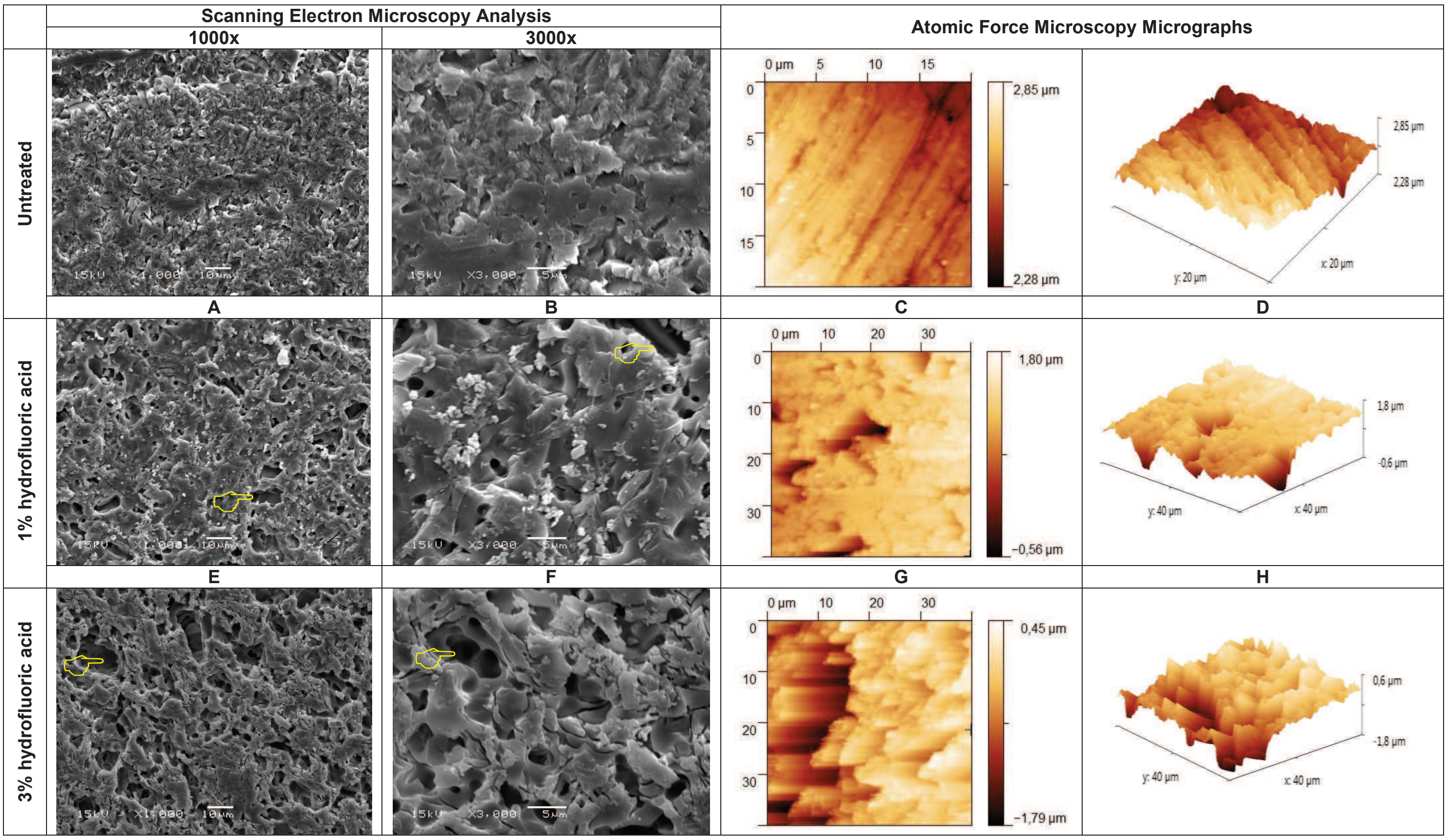


Figure 2 - Photographs of contact angles on surfaces subjected to the following conditions: a) no conditioning; b) 1% HF / 1min + washing / 30s + drying; c) 3% HF / 1min + washing / 30s + drying; d) 5% HF / 1min + washing / 30s + drying; e) 10% HF / 1min + washing / 30s + drying 30s. Surface conditioning reduced the contact angle values with increasing concentration of hydrofluoric acid can be observed.


Scanning Electron Microscopy Analysis
1000x
3000x
Atomic Force Microscopy Micrographs
Untreated
1% hydrofluoric acid
3% hydrofluoric acid
A
B
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F
G
H
15kV X1,000 10μm
15kV X3,000 5μm
0 μm 5 10 15
2,85 μm
2,28 μm
y: 20 μm
x: 20 μm
0 μm 10 20 30
1,80 μm
−0,56 μm
1,8 μm
-0,6 μm
y: 40 μm
x: 40 μm
0,45 μm
−1,79 μm
0,6 μm
-1,8 μm

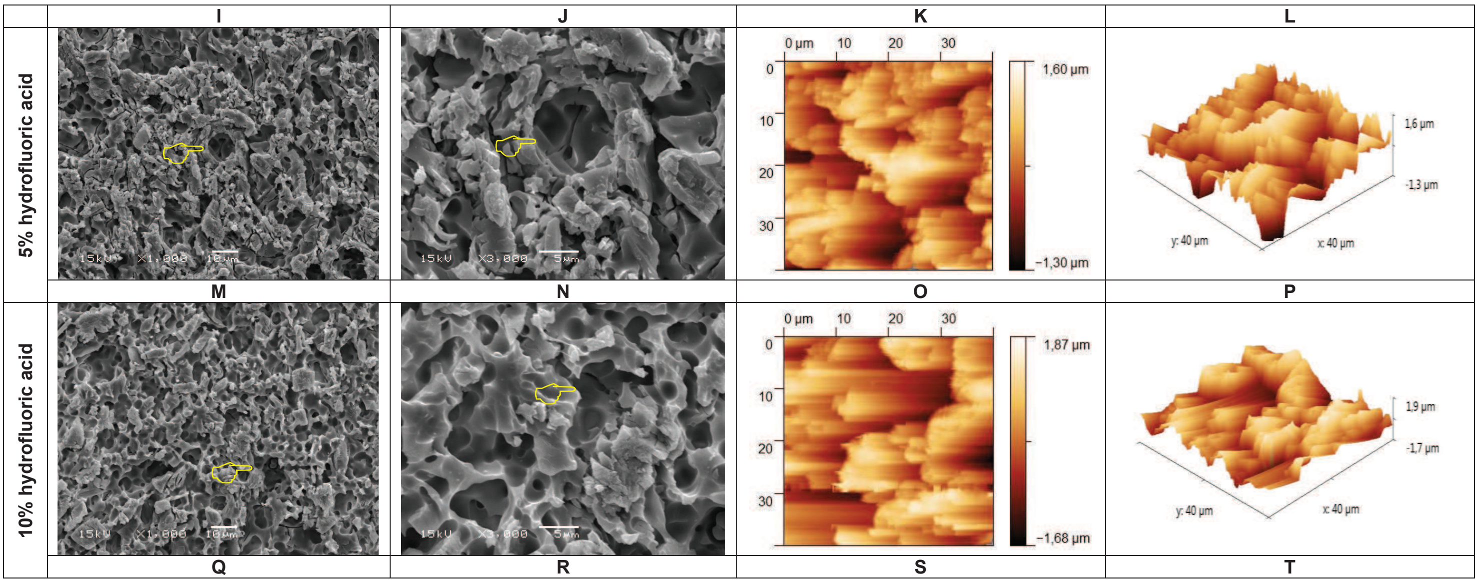

Figure 3 - Representative micrographs of SEM and atomic force images of different ceramic surface conditioning followed by washing + drying: untreated (A-D); etched for 60 s with 1%, 3%, 5% and 10% hydrofluoric acid, which correspond with the letters E-H, I-L, M-P, Q-T, respectively. In SEM micrographs (left: 1000x, right: 3000x), the indicators (☞) show the formation of micro pores and cracks that occur due to degradation of the matrix glass from etching of the ceramic surface.

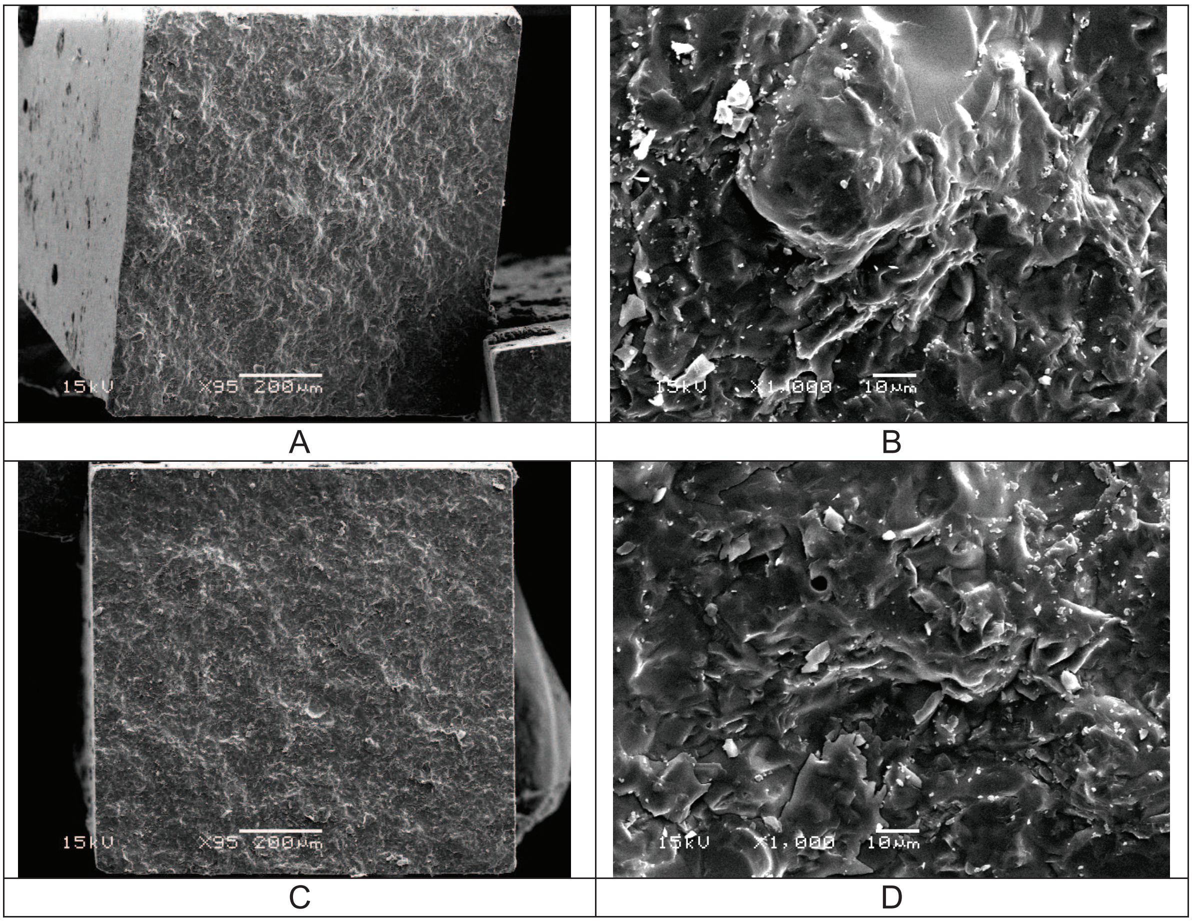

Figure 4 - Representative micrographs of tested specimens. A-D: mainly adhesive fracture between ceramic (A-B) and cement (C-D) of a specimen from HF10 group (95X and 1000X).

TABLES

Table 1 - Surface conditioning for contact angle and microtensile bond strength (MTBS).

	Surface conditioning for *contact angle analysis*	**Surface conditioning for *MTBS***	***Storage condition of the specimens for MTBS***	***Code for MTBS groups***
HF10*	Etching with 10% hydrofluoric acid*		Without aging	HF10-dry
			With aging	HF10-aged
HF1**	Etching with 1% hydrofluoric acid**		Without aging	HF1-dry
			With aging	HF1-aged
HF3**	Etching with 3% hydrofluoric acid**		Without aging	HF3-dry
			With aging	HF3-aged
HF5**	Etching with 5% hydrofluoric acid**		Without aging	HF5-dry
			With aging	HF5-aged
SC	No conditioning	--------------	--------------	--------------

* Condac Porcelana 10% - FGM, Santa Catarina, Brazil.
** Experimentally formulated - FGM, Santa Catarina, Brazil.

Table 2 - Means and standard deviation for contact angle measurements (in degrees). The same superscript letters indicate no significant differences and different letters mean difference significant statistically (Tukey's test, α = 5%).

Groups	**Means ± SD***
HF10	17.5 ± 5.1^{a}
HF1	36.6 ± 5.8^{b}
HF3	36.1 ± 5.2^{b}
HF5	34 ± 4.8^{b}
SC	61.5 ± 5.1^{c}

Table 3 - Means and standard deviation of the bond strength data (MPa), and the comparisons.

Surface Treatment	***Storage condition before testing***		***P values****
	Without aging*	***With aging****	
HF10	15.7 ± 2.8^{a}	13.6 ± 2^{A}	P=0.0732
HF1	14.5 ± 3^{a}	10.2 ± 1.7^{B}	P=0.0010
HF3	14.2 ± 3.3^{a}	13 ± 1.5^{A}	P=0.2941
HF5	14.9 ± 2^{a}	13 ± 2.2^{A}	P=0.0676

*The same letters indicate no significant differences and different letters mean statistically significant differences for comparisons in each column separately. (Tukey's test, α= 5%).
**Student tests for pair comparison between aged and non-aged condition for each surface treatment (P<0.05 were considered to be statistically significant).

Table 4 - Number and percentage (%) of estimated specimens for testing, of pre-test failures (PTF) during cutting and thermocycling (TC) and final number of specimens submitted to the microtensile test (MTBS).

Groups	*Estimated number of bar specimens*	*Number and % of PTF during cutting*	*Number and % of PTF during TC*	*Number and % of tested specimens in MTBS*
HF10-dry	150	69 (46)	0 (0)	81 (54)
HF10-aged	150	66 (44)	0 (0)	84 (56)
HF1-dry	150	86 (57.3)	0 (0)	64 (42.7)
HF1-aged	150	85 (56.7)	0 (0)	65 (43.3)
HF3-dry	150	82 (54.7)	0 (0)	68 (45.3)
HF3-aged	150	82 (54.7)	0 (0)	68 (45.3)
HF5-dry	150	78 (52)	0 (0)	72 (48)
HF5-aged	150	75 (50)	0 (0)	75 (50)

Table 5 - Number and percentages for type of fractures in the beam specimens submitted to the microtensile test.

Groups	*Total number of tested samples*	*Type of Fracture*		
		ADHES	*COHES^{cem}*	*COHES^{cer}*
HF10-dry	81	77 (95,1%)	0 (0%)	4 (4.9%)
HF10-aged	84	79 (94%)	0 (0%)	5 (6%)
HF1-dry	64	61 (95.3%)	0 (0%)	3 (4.7%)
HF1-aged	65	64 (98.5%)	0 (0%)	1 (1,5%)
HF3-dry	68	65 (95.6%)	0 (0%)	3 (4.4%)
HF3-aged	68	67 (98.5%)	0 (0%)	1 (1,5%)
HF5-dry	72	68 (94.4%)	2 (2.8%)	2 (2.8%)
HF5-aged	75	74 (98.7%)	0 (0%)	1 (1,3%)
Total	577 (100%)	555 (96.2%)	2 (0.3%)	20 (3.5%)

***ADHES:** Adhesive fracture at cement/ceramic interface;
COHEScem: cohesive fracture of the resin cement;
COHEScer: cohesive fracture of the ceramic.

Influence of hydrofluoric acid concentration on the flexural strength of a feldspathic ceramic

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Short title: Hydrofluoric acid concentration: impact on ceramic strength

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Influence of hydrofluoric acid concentration on the flexural strength of a feldspathic ceramic

Purpose: To examine the effects of etching with increasing hydrofluoric (HF) acid concentrations on the roughness and flexural strength of a feldspathic ceramic. **Materials and Methods:** One hundred and fifty ceramic specimens (14×4×1.2 mm) were produced from ceramic blocks (VitaBlocks Mark II). All specimens were polished, chamfered and sonically cleaned in isopropyl alcohol. Specimens were randomly divided into 5 groups (n=30): SC (control) no ceramic surface etching; HF1, HF3, HF5 and HF10 ceramic surface etching for 60 s with 1%, 3%, 5% and 10% HF acid concentrations, respectively. Profilometry was performed in all specimens to evaluate roughness prior to flexural strength testing. Data were analyzed using one-way ANOVA and Tukey's test (α=0.05). Weibull module (m) and characteristic stress (σ_0) were also determined. **Results:** HF acid etching, regardless of the concentration used, led to significantly rougher surfaces than the control ($p<0.05$). However, the mean flexural strength values were not statistically different among the etched groups (106.47 to 102.02 MPa). Acid etching significantly reduced the mean flexural strength when compared with the control (143.3 MPa). Weibull modulus of the groups was similar, except for the HF5 group that was higher compared to HF3. **Conclusion:** Flexural strength was similarly affected by the different HF acid concentrations tested, but roughness increased higher the acid concentration. Ceramic etching led to a significant reduction in strength when compared to the untreated ceramic, regardless of its concentration.

Keywords: acid etching, surface treatment, ceramic, strength, AFM

INTRODUCTION

Dental ceramic restorations have been increasingly used, mainly due to their mechanical and optical properties. Technological advances in restorative systems have supplied functional and aesthetic requirements for tooth replacement. Industrially-prepared ceramics for CAD-CAM systems are more structurally reliable for dental applications, because flaws and cracks, which can cause high failure rates under clinical conditions, are reduced to a minimum when compared with the fabrication process at a dental laboratory.[1]

The feldspathic ceramic VitaBlocks Mark II (Vita Zanhfabrik, Bad Säckingen, Germany) is fabricated using fine-grained powders that produce a nearly pore-free ceramic with fine crystals, resulting in improved polishability and increased strength.[2] However, even with superior mechanical properties, bonding between restoration and tooth structure through resin-based adhesive systemsis an essential factor in the clinical success and longevity of dental ceramic restorations [3] For enhanced bonding, the intaglio surface of ceramic restorations should be etched using HF acid[4-5], which promotes surface alterations and mechanical interlocking when associated with a silane coupling agent and a resin-based cement (chemical bond).[6]

Horn[4] was one of the first to suggest the use of HF acid to etch feldspathic laminate veneers in 1983. HF acid selectively etches the glassy phase of the ceramic and exposes siliconoxides (SiO_2) yielding topographic changes (e.g., surface roughness) on the surface, which leads to micromechanical retention when combined with a resin cement.[6-7] This rougher etched surface also contributes to increasing the actual surface area for bonding and enhances the surface energy prior to application of the silane agent.[8]

Different porcelain phases dissolve preferentially depending on the acid concentration and porcelain composition thereby creating a surface more favorable to bonding.[9-10] Ceramic etching is known to be a dynamic process, and its impact is dependent on substrate constitution, surface topography, acid concentration and etching time.[10-11] Meanwhile, since ceramics are brittle materials, the presence of a flaw of critical size has been known to initiate fracture.[12] The propagation of flaws from the intaglio surface (i.e., the cementation side) of ceramic restorations is primarily responsible for their failure.[13] Thompson et al.[14] reported that the fracture strength of a dental ceramic depends on the flaw distribution present in the material, and this distribution can be modified by the surface etching pattern.

Although numerous studies have established the increase in resin bond strength achieved by acid etching as pre-treatment before cementation via micromechanical interlocking, acid etching could have a weakening effect on the material.[11,15] This is due to the extension of flaws that exist on the ceramic surface. The nature of surface flaw modification is a function of etching time and HF acid concentration.[11]

In vitro studies have reported many different combinations between etching periods and acid concentration on bond strength,[7,16-19] roughness[11,21] and flexural strength.[11-12,20-22] However, different ceramics may be more or less sensitive to HF acid, and controversy remains regarding the optimal concentration that results in improvement of the resin bond and preventing a negative effect on ceramic strength. The question is: could higher concentration promote stronger surface alterations and reduce the ceramic strength? It is important to know an acid concentration for adequate micromechanical retention, that does not weaken the ceramic. Lower concentrations of HF also should be considered, because of its hazardous effects.[23-

[24] Currently, there is no study that assesses the effect of HF acid of different concentrations and similar viscosity on the flexural strength of a ceramic.

Thus, the purpose of this study was to evaluate the effect of different HF acid concentrations on the roughness and flexural strength of a feldspathic ceramic. Two hypotheses were tested: 1) acid etching reduces flexural strength compared to no etching; 2) flexural strength decreases as the HF acid concentration increases; 3) higher acid concentrations increases roughness.

MATERIALS AND METHODS

Specimen Preparation

One hundred and fifty bar-shaped specimens ($14 \times 4 \times 1.2$ mm^3) were prepared from sintered ceramic blocks (VitaBlocks Mark II for CEREC/inLab, Vita Zahnfabrik, Bad Säckingen, Germany). The ceramic blocks were sectioned in bar-shaped specimens using a diamond disc at low-speed, under water-cooling, in a sectioning machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). The ceramic bars were wet ground with 400-, 600- and 1200-grit SiC, and then chamfered at a 0.1 mm wide chamfer, as proposed by ISO 6872:2008.[25] All ceramic specimens were sonically cleaned in isopropyl alcohol for 10 min to remove debris.

Surface Conditioning

The specimens were randomly divided into 5 groups (n=30) according to the following ceramic surface treatment: SC (control) no treatment; HF1 etching with 1% HF acid; HF3 etching with 3% HF acid; HF5 etching with 5% HF acid; HF10 etching with 10% HF acid. One side of each ceramic bar was etched with HF acid gel at the different concentrations (FGM, Joinville, Santa Catarina, Brazil) for 60 s. The etched specimens were washed with tap water for 1 min and dried with compressed air for 30 s.

Surface Roughness Analysis

The surface roughness of all specimens was analyzed in a profilometer with a contact-type stylus (Mitutoyo SJ-410, Kanagawa, Japan). Measurement was performed at 3 distinct locations for each specimen according to the ISO 4287:1997[26] parameters (Ra arithmetical mean of the absolute values of peaks and valleys measured from a medium plane (μm) and Rz average distance among the 5 highest peaks and 5 major valleys found in the standard (μm). The values of Ra and Rz were obtained from the average of three readings. Measurements were performed with $\lambda c=0.8$ mm ($0.1<Ra \leq 2.0$), resulting in a total measuring length of 4 mm. Gaussian filter was employed for separation of the defects and shape of the roughness profile.

Three-point Bending Test

The flexural strength was determined using a three-point bending test in a universal testing machine (EMIC DL-1000, EMIC, Sao Jose dos Pinhais, Brazil) and performed according to ISO 6872:2008.[25] The dimensions of each ceramic bar were measured with a digital caliper before the test and numbered on the compression side. The etched surface of the specimens was placed down and flat on a dedicated jig with rounded supporting rods 12 mm apart. The center of the specimens was loaded (load cell 0.5 KN) with a rounded chisel (radius 3 mm) at a crosshead speed of 1 mm/min until fracture. The following equation was used for flexural strength (σ) calculation: $\sigma=3\ Pl/2wb^2$, where P is the fracture load (in N); l is the test span (12 mm); w is the width of the specimen (in mm); and b is the thickness of the specimen (in mm).

Statistical Analysis

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

The strength distributions of brittle materials, such as ceramics, are more properly described by Weibull statistics,[27] which describe reliability of the ceramic material and variation of the resistance.[1] Thus, the Weibull module (m) and the characteristic strength (σ_0) with a confidence interval of 95% were obtained and determined in a lnσ_c - ln[ln 1/(1-F(σ_c)] diagram (according to ENV 843-5):

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

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

Fractographical Analysis

The tested specimens were randomly selected after the flexural test, then the specimens were sputter coated with gold-palladium for examination of the fractured surfaces under a scanning electron microscope (SEM, Jeol-JSM-T330A, Jeol Ltd, Tokyo, Japan).

Topographical Analysis Under Scanning Electron Microscopy and Atomic Force Microscopy

Topographical analysis was performed both by SEM and atomic force microscopy (AFM). For SEM, 2 samples of each group were sputter-coated with gold–palladium alloy prior to SEM imaging at distinct magnifications. AFM images (40 μm × 40 μm) were obtained using an atomic force microscope (AFM Agilent

Technologies 5500, Chandler, Arizona, USA). The images were obtained using a non-contact mode and PPP-NCL probes (Nanosensors, Force constant = 48 N/m). AFM images were analyzed using a dedicated data analysis software (Gwyddion™ version 2.33, GNU, Free Software Foundation, Boston, MA, USA).

RESULTS

Surface Roughness

The mean and standard deviation values for Ra and Rz are presented in Table 1. Significant differences in mean Ra and Rz values were found among all the groups. SC showed the lowest mean Ra and Rz values, while HF10 showed the highest mean Ra and Rz values ($p<0.05$) (Table 1).

Flexural Strength

Significant differences in mean flexural strength values were also found (Table 1 and Fig. 1). The control group showed significantly higher ($p>0.05$) mean values than the other groups. There were no significant differences ($p>0.05$) among the groups HF1, HF3, HF5 and HF10. The Pearson Correlation analysis (Fig. 2) revealed a moderate inverse correlation ($r= -0.5366$; $p < 0.001$) between surface roughness (Ra) and flexural strength (MPa).

The Weibull analysis results are shown in Table 2 and graphically in Fig. 3. The characteristic strength of the untreated group was higher than the etched groups. In general, the Weibull modulus of the groups was similar, except for the HF5 group, which was significantly higher compared to HF3, indicating that 5% HF acid etching can provide more regular surface alterations, thus reducing data scatter.

Fig. 4 shows representative SEM micrographs of the fracture surfaces. In all cases, the initial defect is clearly observed on the tensile surface of the material, probably generated by etching, which creates several defects on the tensile surface. Quinn (2007)[28] referred to this kind of fracture as “zipper cracks”: the mirror zone can be elongated along of the surface due to several defects from the surface.

SEM and AFM images (Figure 5) show that higher HF acid concentrations created deeper, more evident craters and pits (Fig. 5 Q-T; HF10). However, slight topographic changes were found for HF1(Fig. 5 E-H) when compared to the untreated condition (Fig. 5 A-D).

DISCUSSION

Some factors can influence the strength of ceramics, such as the HF acid etching widely used to enhance mechanical interlocking between resin cements and ceramics based on silica. Some studies have reported that microstructural changes promoted by HF acid did not negatively affect the flexural strength of ceramics.[12,14,22] On the other hand, other studies have suggested that both HF acid concentration and etching time have a weakening effect on the strength of ceramics.[11,20,21] In the present study, the ceramic flexural strength decreased after HF acid etching, corroborating the latter investigations. Thus, the first hypothesis was accepted, since the flexural strength of the etched ceramic was lower than that of the untreated ceramic.

Stangel et al.[9] compared the effect of 52% HF acid etching for 90 seconds and 20% HF acid etching for 2.5 minutes and noted distinct differences in the ceramic microstructure. They suggested that the glassy phase would appear to be preferentially dissolved with 52% HF acid, whereas the 20% HF acid dissolved the crystalline phase. Additionally, two other studies[11,15] agree with this, though the etching time was not the same.

Here, HF acid etching increased ceramic roughness, i.e., the higher the acid concentration, the rougher the ceramic surface. The SEM and AFM images clearly revealed the progressive effect of different concentrations of HF acid on the ceramic microstructure (Figure 5 E-T), showing greater surface roughness and the presence of pores in the surface after etching. These pores could act as sources of crack initiation. Thus, minimum surface modification was sufficient for the slight decrease in flexural strength observed in the etched groups. SEM images clearly revealed a lesser amount of glassy matrix in all the experimental groups compared with the control. It should be noted that HF acid preferentially attacks the grain boundaries at the interface of crystals and the glassy matrix, amplifying the ceramic surface roughness with greater concentrations and causing a weakening effect on mechanical strength.[29,30]

However, although the roughness findings and SEM micrographs demonstrated increased surface roughness as a result of acid etching, no statistical differences in flexural strength were noted for the tested acid concentrations. Thus, the second hypothesis was rejected once there was no decrease in flexural strength according to the increased HF acid concentration.

Since ceramics vary widely in their failure rates, a Weibull regression analysis is indicated to evaluate the strength data.[31] Although the Weibull parameters did not show significant differences in flexural strength between the etched groups, the Weibull modulus for the HF5 group was significantly higher compared to HF3. This may have been caused by alterations in size and shape of the initial surface flaws by HF acid etching (e.g., reducing the size and depth of surface flaws, particularly the small and sharp edge or the tips of flaws and the rounded-off bottom of flaws).[14]

In terms of adhesion, one principle for choosing the etching approach should be that it yields maximum bond strength to resin[12], without affecting the strength of the etched ceramic. Pilot data by the current authors (unpublished data), testing the same acid concentrations from this investigation showed that HF acid etching decreased the contact angle in all etched groups, and the highest and durable bond strengths were obtained when ceramic surfaces were conditioned with 3%, 5% and 10% HF acid.

Thus, the most important conclusion is if the flexural strength data depicted in this study and previous bonding findings are taken into account, this suggests that lower concentrations of HF acid (3% and 5%) could be indicated for ceramic surface conditioning due to bond improvement, with no difference in terms of ceramic strength and preventing dangerous effects on health if compared to 10% HF acid.[23-24]

Nonetheless, it should be noted that there are some limitations in this study. First, it was performed under dry and static conditions. Therefore, the 3-point bending apparatus did not simulate the cyclic nature of the oral environment, and the oral fluid exposure, which may cause hydrolysis, affecting mechanical properties of the ceramic. Second, ceramics restorations are resin-bonded, and the unfilled resin coating might improve the ceramic strength. Future studies should consider simulating clinical conditions, to include a wet environment and fatigue testing of resin-bonded ceramic restorations (lifetime,[32] staircase,[33] stepwise[34,35] approaches).

CONCLUSION

Acid etching has a weakening effect on feldspathic ceramic if compared to no treatment. However, different HF acid concentrations do not affect the flexural strength of feldspathic ceramic, in spite of surface changes in roughness.

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FIGURES

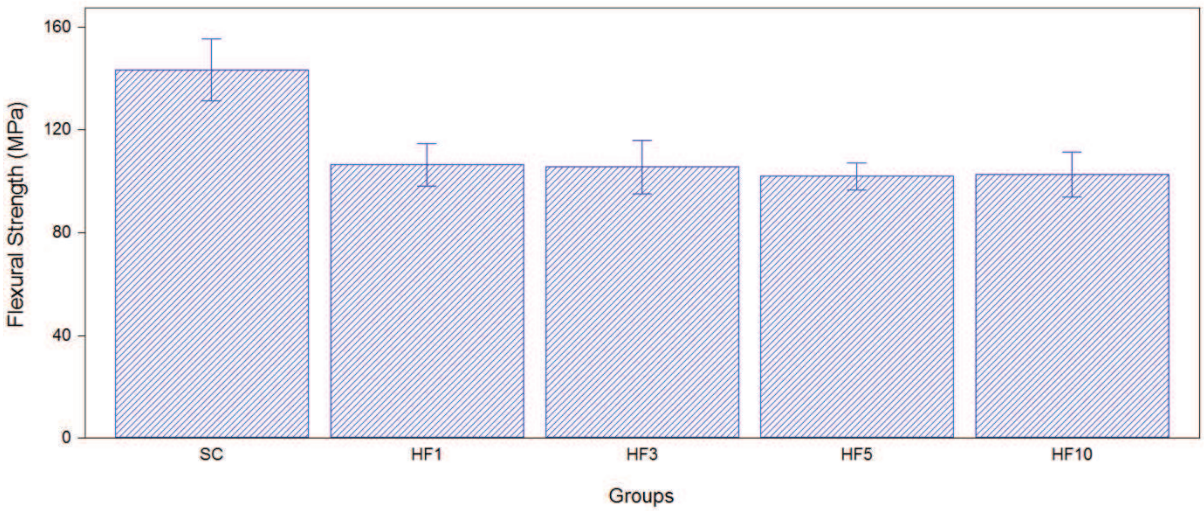


Figure 1 - Column chart (mean ± standard deviation) of the flexural strength (MPa) data.

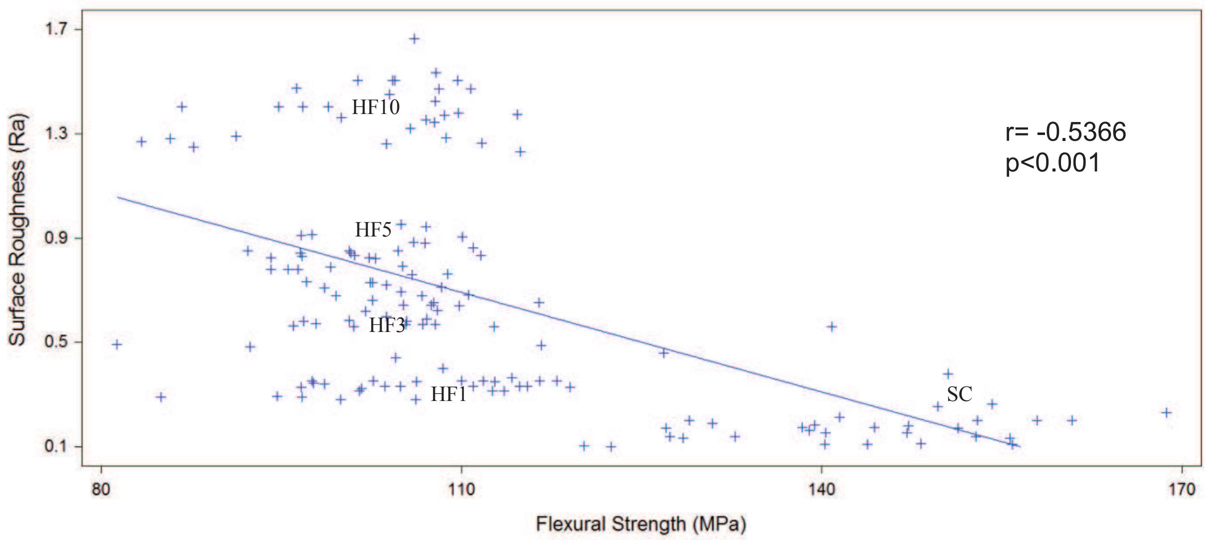


Figure 2 – Scatter Plot for correlation between the surface roughness (Ra) and flexural strength (MPa).

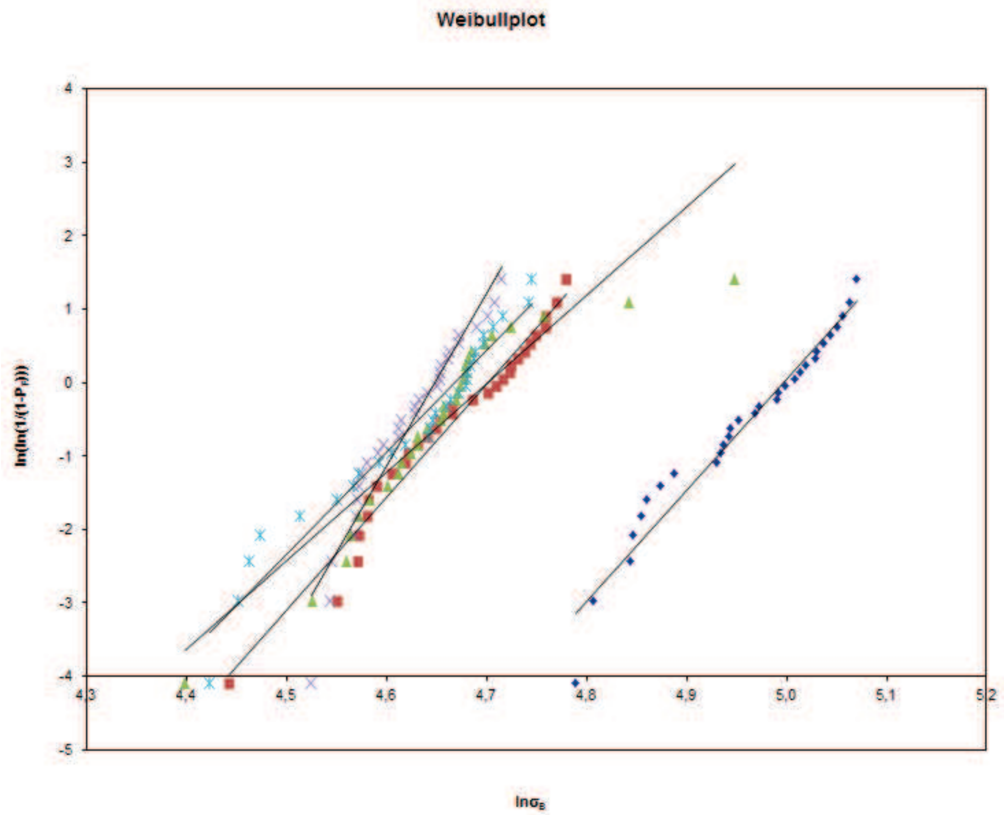


Figure 3 - Weibull distribution for flexural strength (MPa) (Diamond = SC; Square = HF1; Triangle = HF3; X = HF5; Star = HF10).

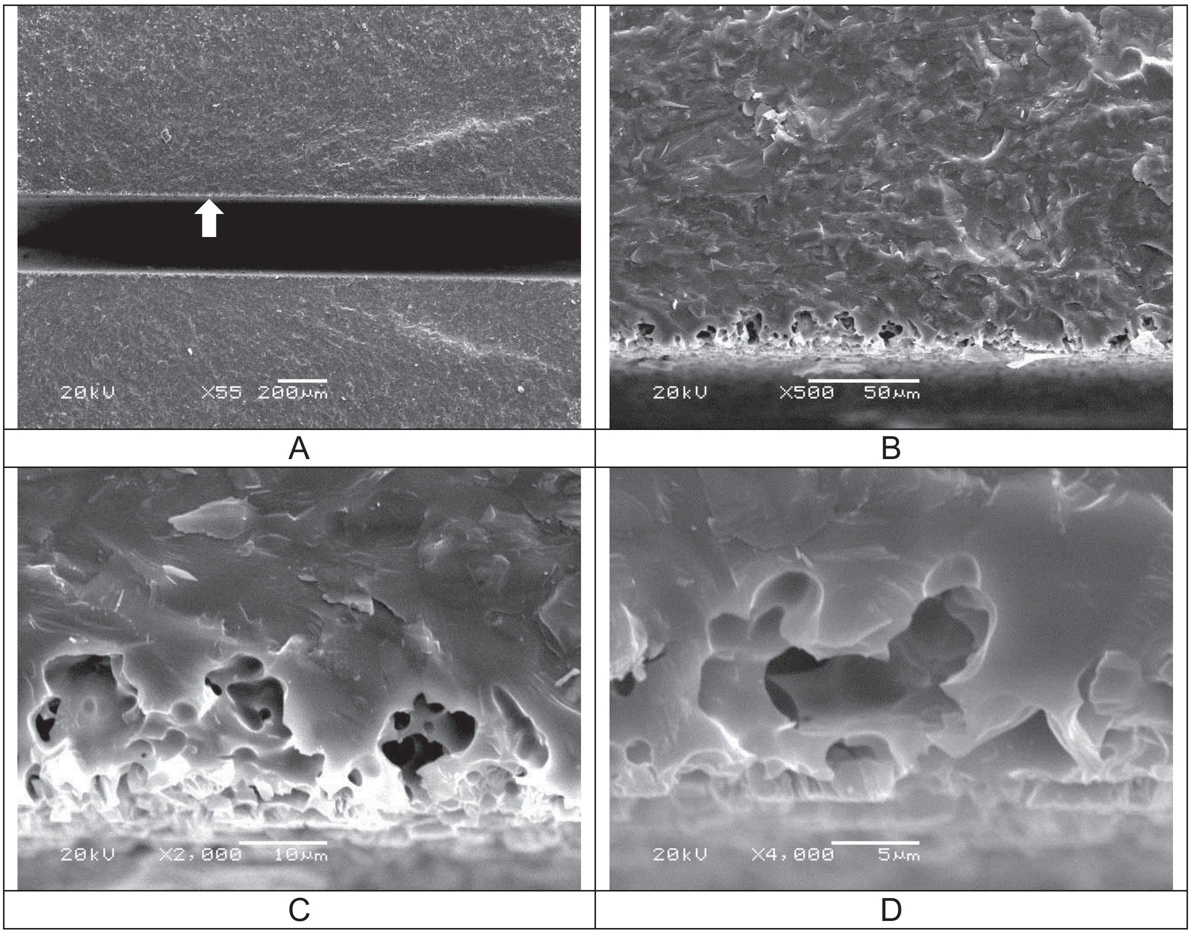
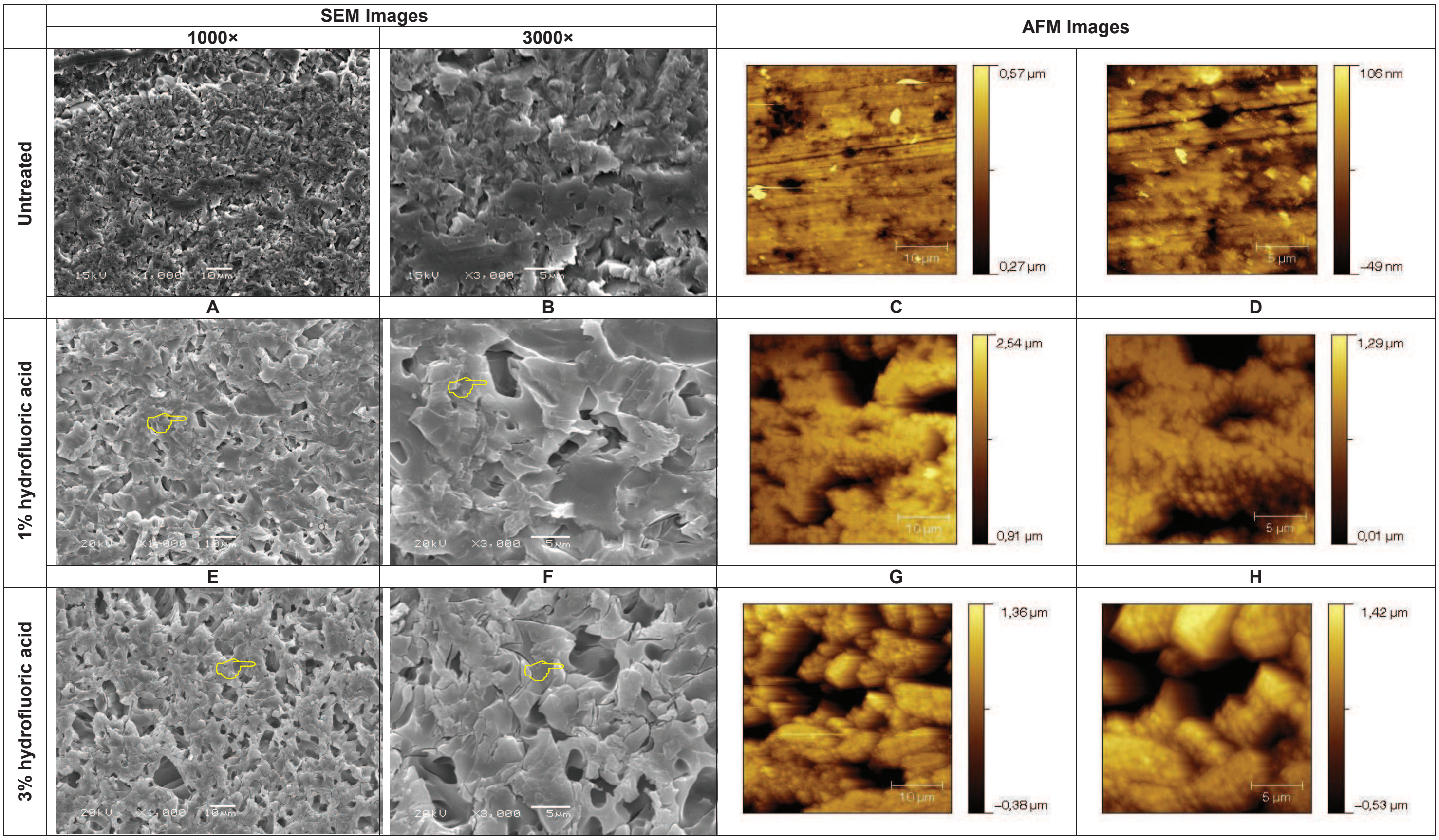


Figure 4 – Representative SEM images of the fracture surface of a specimen from HF10 group. The fracture origin (white arrow) was observed in the etched surface under tensile stresses and the hackles show the direction of crack propagation. Close-up views of image A are presented in images B (500×), C (2000×) and D (4000×) that show numerous microcracks with a honeycombed appearance, which are the fracture origin.


SEM Images
1000×
3000×
AFM Images
Untreated
1% hydrofluoric acid
3% hydrofluoric acid
A
B
C
D
E
F
G
H
0,57 µm
0,27 µm
106 nm
–49 nm
2,54 µm
0,91 µm
1,29 µm
0,01 µm
1,36 µm
–0,38 µm
1,42 µm
–0,53 µm
10 µm
5 µm

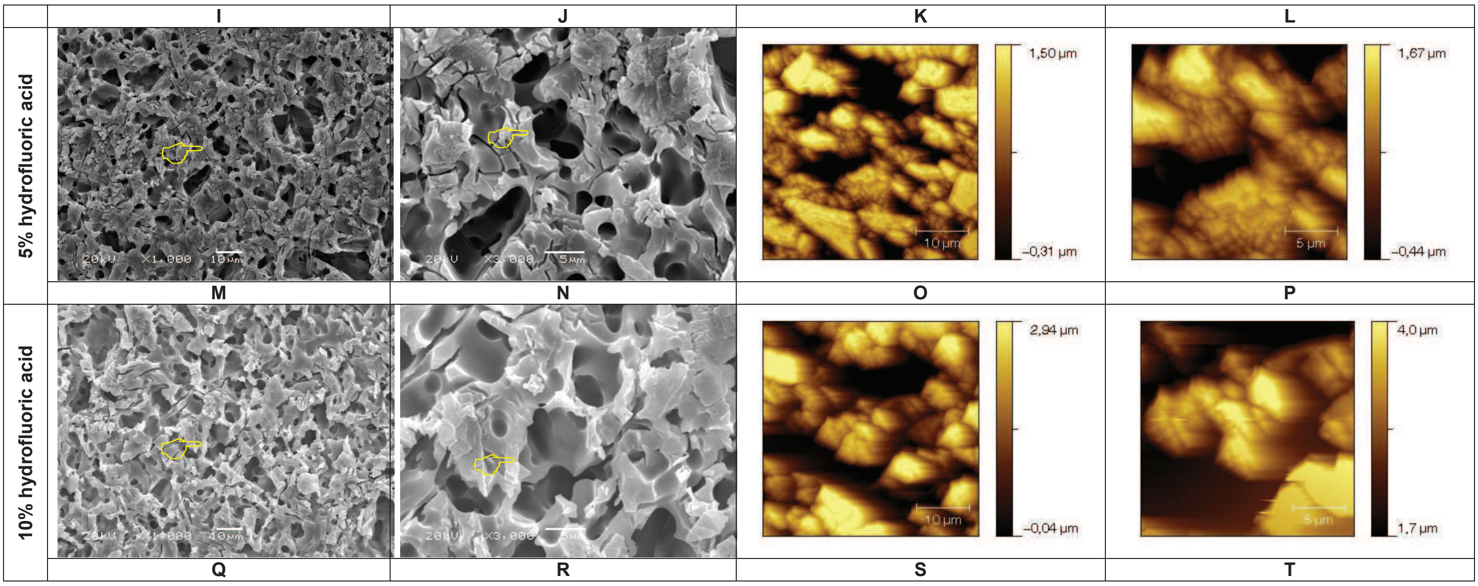


Figure 5 - Representative SEM and AFM images of different ceramic surface conditioning: untreated (A-D); etched for 60 s with 1%, 3%, 5% and 10% hydrofluoric acid (E-H, I-L, M-P, Q-T, respectively). In SEM micrographs, the indicators (☞) show the formation of micropores and cracks that occur due to degradation of the glassy matrix due to HF acid etching.

TABLES

Table 1 - Mean values and standard deviations for surface roughness and flexural strength.

Groups	*Surface roughness (Ra; μm)*	*Surface roughness (Rz; μm)*	*Flexural strength (MPa)*
SC	0.17 ± 0.06 [a]	1.7 ± 0.52 [a]	143.30 ± 12.1 [a]
HF1*	0.34 ± 0.04 [b]	3.64 ± 0.36 [b]	106.47 ± 8.4 [b]
HF3*	0.61 ± 0.07 [c]	5.11 ± 1.05 [c]	105.54 ± 10.4 [b]
HF5*	0.82 ± 0.07 [d]	6.38 ± 0.42 [d]	102.02 ± 5.3 [b]
HF10**	1.39 ± 0.1 [e]	9.81 ± 0.80 [e]	102.64 ± 8.7 [b]

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

Table 2 - HF acid concentration influence on the Weibull parameters.

Groups	**Characteristic strength $\sigma_{63.21\%}$ (MPa)**	**Confidence intervals**	**Weibull modulus *m***	**Confidence intervals**
SC	148.57 [a]	144.13 - 153.02	14.33 [ab]	10.03 - 18.43
HF1	110.15 [b]	107.67 - 113.23	15.32 [ab]	10.72 - 19.69
HF3	110.14 [bc]	106.23 - 114.08	12.04 [a]	8.43 - 15.48
HF5	104.46 [c]	102.50 - 106.34	23.36 [b]	16.35 - 30.03
HF10	106.52 [bc]	103.24 - 109.81	13.91 [ab]	9.73 - 17.88

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

5 – CONSIDERAÇÕES FINAIS

- Quanto mais alta a concentração do ácido fluorídrico, mais intensa é a alteração da topografia de superfície da cerâmica feldspatica condicionada, e menor é o ângulo de contato.
- Em termos de adesão, a cerâmica testada pode ser condicionada com ácido fluorídrico nas concentrações de 3%, 5% e 10%, as quais promoveram resistência adesiva estável e alterações topográficas relevantes para a união cimento/cerâmica.
- A resistência mecânica da cerâmica testada não altera em função do condicionamento com diferentes concentrações de ácido fluorídrico.
- O condicionamento com ácido fluorídrico parece ter um efeito de enfraquecimento sobre a cerâmica feldspática se comparada à cerâmica sem tratamento, independente da concentração utilizada.

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ANEXO 1 – Guidelines for authors

The Journal of Adhesive Dentistry

The Journal of Adhesive Dentistry is a bi-monthly journal that publishes scientifically sound articles of interest to practitioners and researchers in the field of adhesion to hard and soft dental tissues. The Journal publishes several types of peer-reviewed original articles:

1. **Clinical and basic science research reports** – based on original research in adhesive dentistry and related topics.
2. **Reviews topics** – on topics related to adhesive dentistry
3. **Short communications** – of original research in ad-hesive dentistry and related topics. Max. 4 printed pages, including figures and references (max. characters 18,000). High priority will be given to the review of these papers to speed publication.

4a. **Invited focus articles** – presenting a position or hypothesis on a basic science or clinical subject of relevant related topics. These articles are not intended for the presentation of original results, and the authors of the articles are selected by the Editorial Board.

4b. **Invited commentaries** – critiquing a focus article by addressing the strong and weak points of the focus article. These are selected by the Editorial Board in consultation with the focus article author, and the focus article and the commentaries on it are published in sequence in the same issue of the Journal.

5. **Invited guest editorials** – may periodically be solicited by the Editorial Board.
6. **Proceedings of symposia, workshops, or conferences** – covering topics of relevance to adhesive dentistry and related topics.
7. **Letters to the Editor** – may be submitted to the editor-in-chief; these should normally be no more than 500 words in length.

SUBMISSION INSTRUCTIONS

Submission of manuscripts in order of preference:

1. Submission via online submission service (www.manuscriptmanager.com/jadd). Manuscript texts should be uploaded as PC-word files with tables and figures preferably embedded within the PC-word document. A broad range of file formats are acceptable. No paper version required but high resolution photographs or illustrations should be sent to the editorial office (see below). Online submissions are

automatically uploaded into the editorial office's reviewer assignment schedule and are therefore processed immediately upon upload.

2. Submission via e-mail as a PC-word document (wintonowycz@quintessenz.de). Illustrations can be attached in any format that can be opened using Adobe Photoshop, (TIF, GIF, JPG, PSD, EPS etc.) or as Microsoft PowerPoint Documents (ppt). No paper version required but high resolution photographs or illustrations should be sent to the editorial office.

3. One paper copy of the manuscript plus a floppy diskette or CD-ROM (mandatory) containing a PC-word file of the manuscript text, tables and legends. Figures should be included on the disk if possible in any format that can to be opened using Adobe Photoshop, (TIf, GIf, JPG, PSD, EPS etc.) or as a Microsoft PowerPoint Document (ppt).

Mailing address:

Quintessenz Verlags-GmbH, Karin Wintonowycz
The Journal of Adhesive Dentistry,
Ifenpfad 2-4, D–12107 Berlin, Germany

Illustrations that cannot be sent electronically will be scanned at the editorial office so that they can be sent to reviewers via e-mail along with the manuscript to expedite the evaluation process.

Resubmitted manuscripts should also be submitted in the above manner. Please note that supplying electronic versions of your tables and illustrations upon resubmission will assure a faster publication time if the manuscript is accepted.

Review/editing of manuscripts. Manuscripts will be reviewed by the editor-in-chief and at least two reviewers with expertise within the scope of the article. The publisher reserves the right to edit accepted manuscripts to fit the space available and to ensure conciseness, clarity, and stylistic consistency, subject to the author's final approval.

Adherence to guidelines. Manuscripts that are not prepared in accordance with these guidelines will be returned to the author before review.

MANUSCRIPT PREPARATION

• The Journal will follow as much as possible the recommendations of the International Committee of Medical Journal Editors (Vancouver Group) in regard to preparation of manuscripts and authorship (Uniform requirements for manuscripts submitted to biomedical journals. Ann Intern Med 1997;126: 36-47).

• **Title page**. The first page should include the title of the article (descriptive but as concise as possible) and the name, degrees, job title, professional affiliation, contribution to the paper (e.g., idea, hypothesis, experimental design, performed the experiments in partial fulfillment of requirements for a degree, wrote the manuscript, proofread the manuscript, performed a certain test, consulted on and performed statistical evaluation, contributed substantially to discussion, etc.) and full address of all authors. Phone, fax, and e-mail address must also be provided for the corresponding author, who will be assumed to be the first listed author unless otherwise noted. If the paper was presented before an organized group, the name of the organization, location, and date should be included.

• **3-8 keywords**.

• **Structured abstract**. Include a maximum 250-word structured abstract (with headings Purpose, Materials and Methods, Results, Conclusion).

• **Introduction**. Summarize the rationale and purpose of the study, giving only pertinent references. Clearly state the working hypothesis.

• **Materials and Methods**. Present materials and methods in sufficient detail to allow confirmation of the observations. Published methods should be referenced and discussed only briefly, unless modifications have been made. Indicate the statistical methods used, if applicable.

• **Results**. Present results in a logical sequence in the text, tables, and illustrations. Do not repeat in the text all the data in the tables or illustrations; emphasize only important observations.

• **Discussion**. Emphasize the new and important aspects of the study and the conclusions that follow from them. Do not repeat in detail data or other material given in the Introduction or Results section. Relate observations to other relevant studies and point out the implications of the findings and their limitations.

• **Acknowledgments**. Acknowledge persons who have made substantive contributions to the study. Specify grant or other financial support, citing the name of the supporting organization and grant number.

• **Abbreviations**. The full term for which an abbreviation stands should precede its first use in the text unless it is a standard unit of measurement.

• **Trade names**. Generic terms are to be used when ever possible, but trade names and manufacturer should be included parenthetically at first mention.

• **Clinical Relevance**. Please include a very brief (2 sentences or 3 lines) clinical relevance statement.

REFERENCES

• **All references must be cited** in the text, according to the alphabetical and numerical reference list.

• **The reference list** should appear at the end of the article, in alphabetical and numerical sequence.

• **Do not include unpublished data** or personal com-munications in the reference list. Cite such references parenthetically in the text and include a date.

• **Avoid using abstracts** as references.

• **Provide complete information** for each reference, including names of all authors. If the reference is part of a book, also include title of the chapter and names of the book‘s editor(s).

Journal reference style:

1. Turp JC, Kowalski CJ, Stohler CS. Treatment- seeking patters of facial pain patients: Many possibilities, limited satisfaction. J Orofacial Pain 1998;12:61-66.

Book reference style:

1. Hannam AG, Langenbach GEJ, Peck CC. Computer simulations of jaw biomechanics. In: McNeill C (ed). Science and Practice of Occlusion. Chicago: Quintessence, 1997:187-194.

ILLUSTRATIONS

• All illustrations must be numbered and cited in the text in order of appearance.

• Submitted figures should meet the following minimum requirements:

– High-resolution images should have a width of 83 mm and 300 dpi (for column size).

– Graphics (bar diagrams, schematic representations, drawings) wherever possible should be produced in Adobe Illustrator and saved as AI or EPS files.

– All figures and graphics should be separate files – not embedded in Word or Power Point documents.

Upon article acceptance, high-resolution digital image files must be sent via one of the following ways:

1. As an e-mail attachment, if the files are not excessively large (not more than 10 MB), to our production department: Steinbrueck@quintessenz.de
2. Online File Exchange Tool: Please send your figures with our Online File Exchange Tool. This web tool allows you to upload large files (< 350.0 MB) to our server. Please archive your figures with a maximum size of 350 MB first. Then upload these archives with the following link: http://files.qvnet.de/JAD/, password: IAAD. Please name the archive with your name and article number so we can identify the figures.

Line drawings – Figures, charts, and graphs should be professionally drawn and lettered large enough to be read after reduction. Good-quality computer-generated laser prints are acceptable (no photocopies); also provide electronic files (eps, ai) if possible. Lines within graphs should be of a single weight unless special emphasis is needed.
Legends – Figure legends should be grouped on a sep-arate sheet and typed double-spaced.

TABLES
• Each table should be logically organized, on a separate sheet, and numbered consecutively.
• The title and footnotes should be typed on the same sheet as the table.

MANDATORY SUBMISSION FORM
The Mandatory Submission Form, signed by all authors, must accompany all submitted manuscripts before they can be reviewed for publication. Electronic submission: scan the signed form and submit as JPG or TIF file.

PERMISSIONS & WAIVERS
• Permission of author and publisher must be obtained for the direct use of material (text, photos, drawings) under copyright that does not belong to the author.
• Waivers must be obtained for photographs showing persons. When such waivers are not supplied, faces will be masked to prevent identification. For clinical studies the approval of the ethics committee must be presented.

PAGE CHARGE

The first 8 printed pages in an article are free of charge. For excess pages, the charge is €140 per printed page. The approximate number of characters on a printed page is approximately 6,800. Please also consider the number and size of illustrations.

ANEXO 2 – Guidelines for authors

Journal of Biomedical Materials Research Part B: Applied Biomaterials

Aims and Scope

Journal of Biomedical Materials Research Part B: *Applied Biomaterials* is an official journal of the Society for Biomaterials, the Japanese Society for Biomaterials, the Australasian Society for Biomaterials, and the Korean Society for Biomaterials. It is a peer-reviewed journals serving the needs of biomaterials professionals who devise, promote, apply, regulate produce, and market new biomaterials and medical devices. Papers are published on device development, implant retrieval and analysis, manufacturing, regulation of devices, liability and legal issues, standards, reviews of different device areas, and clinical applications. Published manuscript fit into one of six categories: original research reports, clinical device-related articles, short research and development reports, review, special report, or columns and editorials. Manuscripts from all countries are invited but must be in English. Authors are not required to be members of a Society for Biomaterials.

Types of Articles Considered for Publication

Original Research Reports: Full-length papers consisting of complete and detailed descriptions of a research problem, the experimental approach, the findings, and appropriate discussion. Findings should represent significant new additions to knowledge.

Clinical Device-Related Articles: Full-length papers addressing such issues as material processing, device construction, regulatory matters, clinical trials, and device retrieval.

Reviews: Scholarly and critical topic-oriented reviews that present a state-of-the-art view. While most reviews are solicited, persons interested in contributing may contact the Editor.

Special Reports: Reports of special topic-oriented symposia, device retrieval protocols, or other special reports not described in the above categories, yet of interest to the applied biomaterials research and development community. Potential contributors should contact the Editor before submitting special reports.

Columns and Editorials: While columns and guest editorials are preponderantly solicited, persons interested in becoming columnists or contributing editorials are encouraged to contact the Editor.

Submission of Manuscripts

Online Submission:

Journal of Biomedical Materials Research Part B: Applied Biomaterials is now receiving submitted manuscripts online at
http://mc.manuscriptcentral.com/jbmr-b.

Submit all new manuscripts online. Launch your web browser and go to
http://mc.manuscriptcentral.com/jbmr-b.

Check for an existing user account. If you are submitting for the first time, and you do not find an existing account, create a new account. Follow all instructions.

At the end of a successful submission, a confirmation screen with manuscript number will appear and you will receive an e-mail confirming that the manuscript has been received by the journal. If this does not happen, please check your submission and/or contact tech support using the Get Help Now link in the right corner of any screen.

Upon Acceptance: Manuscript files will now automatically be sent to the publisher for production. It is imperative that files be in the correct format to avoid a delay in the production schedule.

JBMR Part B has adopted a policy that requires authors to make a statement concerning potential conflict of interest relating to their submitted articles. The Editorial Board asks authors of original reports and reviews to disclose, at the time of submission: (1) any financial or employment arrangements they may have with a company whose product figures prominently in the submitted manuscript or with a company making a competitive product; and (2) any grants or contracts from a government agency, a non profit foundation, or a company supporting the preparation of the manuscript or the described research. This information will be available to the reviewers of the manuscript. If the article is accepted for publication, the editor will discuss with the authors the manner in which such information may be communicated to the reader.

At the time of submission, *JBMR Part B* asks authors to certify that all animals utilized in their research were cared for according to the policies and principles established by the Animal Welfare Act and the NIH Guide for Care and Use of Laboratory Animals.

Review Process: All original reports and reviews receive critical review by at least two reviewers with expertise in the major subject area of the paper. Reviewers may recommend "Acceptance as is," "Acceptance with modification," or "Rejection." If modification is required, the manuscript is returned to the author(s). The revised manuscript is then re-reviewed by the original reviewers, and even re-revised if necessary. Differences in opinion are resolved by submission either to a third reviewer or the Editor.

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Organization and File Formats

Manuscript: For optimal production, prepare manuscript text in size 12 font on 8-1/2 x 11 inch page, double-spaced, with at least 1-inch margins on all sides. Text files should be formatted as .doc or .rtf files. The results and discussion sections must be written separately and cannot be combined. Refrain from complex formatting; the Publisher will style your manuscript according to the Journal design specifications. Do not use desktop publishing software such as PageMaker orQuark Xpress or other software such as Latex. If you prepared your manuscript with one of these programs, export the text to a word processing format. Please make sure your word processing programs "fast save" feature is turned off. Please do not deliver files that contain hidden text: for example, do not use your word processor's automated features to create footnotes or reference lists. Manuscripts including references (but not figures or tables) should be no longer than 18 pages.

Please be sure to submit your illustrations and tables as separate files; the system will automatically create a pdf file of your paper for the reviewers.

Original research and short reports should appear in the following order: title page (including authors and affiliations), abstract, keywords, introduction, materials and methods, results, discussion, acknowledgments, references, figure legends. Number pages consecutively starting with the title page as page 1. Abbreviations must conform to those listed in Council of Biology Editors' CBE Style Manual, 5th Edition.

When mentioning a material, chemical reagent, instrument, or other product, use the generic name only. If further identification (proprietary name, manufacturer's name and address) is absolutely required, list it in parentheses.

Title Page: List the full title of the paper and each author's full name (first name, middle initial(s), surname), department, institution, city, and state (and country if other than the United States).Indicate the name and address of the author to whom reprint requests should be sent.

Abstract and Keywords: Include an abstract of about 200 words maximum summarizing the aims, findings, and conclusions of the paper. Below the abstract, list five keywords or phrases that best characterize the subject matter of the manuscript.

Running Heads: Supply a short title of no more than65characters, including spaces and punctuations, to be used for running head copy.

References: Number references consecutively as they appear in the text. Material accepted for publication but not yet published may be listed in the References, but unpublished observations, personal communications, and material submitted for publication but not yet accepted should be cited parenthetically within the text (and not included among the numbered references). Style references entries using the Council of Biology Editors Style Manual, 5th Edition formats:

For journal articles:

Alexander A, Green WS. Total hip replacements: A second look. JSocBiomater 1989;45:345–366.

For books/chapters:

Ricci JL, Guichet J-M. Total hip replacement: A third look.CindraAB, Franklin DE, editors. State
of the art orthopaedics, vol 3, Hips.NewYork: Wiley; 1988:56–59.

For abstracts:

Davidson GRH. Total hip replacement: A fifth look. TransABCS1987;22-341–345.

For presentations:

Goodenough T. Total hip replacement: A sixth look. Presented at the 3rd Annu Mtg Orthop Res Soc, Boston, December 5–7, 1989.

Figure Legends: Please supply complete captions for all figures. Captions are to appear on a separate page at the end of the manuscript.

Tables: Please save Tables separately and supply numbers and titles for all. All table columns should have an explanatory heading. Tables should be submitted as doc or rtf files (it is preferred that tables are prepared using Word’s table edit tool.)

Illustrations: When preparing digital art, please consider:

Resolution:

The minimum requirements for resolution are:

1200 DPI/PPI for black and white images, such as line drawings or graphs.

300 DPI/PPI for picture-only photographs

600 DPI/PPI for photographs containing pictures and line elements, i.e., text labels, thin lines, arrows.

These resolutions refer to the output size of the file; if you anticipate that your images will be enlarged or reduced, resolutions should be adjusted accordingly.

Formats:

For the editorial review process, GIF and JPEG files are acceptable; upon submission of a revision, TIFF or EPS files will be required. For the editorial review process, color images may be submitted in RGB color; upon revision, CMYK color will be required. Delivery of production-quality files early in the review process may facilitate smooth and rapid publication once a manuscript has been accepted.

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To ensure that your digital graphics are suitable for print purposes, please go to Rapid Inspector™ at http://rapidinspector.cadmus.com/zwi/index.jsp. This free, stand-alone software application will help you to inspect and verify illustrations right on your computer.

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