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EFEITO DO CONDICIONAMENTO COM DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO SOBRE A RESISTÊNCIA DE UNIÃO ENTRE CERÂMICA E BRÁQUETES METÁLICOS

DISSERTAÇÃO DE MESTRADO

Cristiane Frantz Arend

Santa Maria, RS, Brasil

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EFEITO DO CONDICIONAMENTO COM DIFERENTES CONCENTRAÇÕES DE ÁCIDO FLUORÍDRICO SOBRE A RESISTÊNCIA DE UNIÃO ENTRE CERÂMICA E BRÁQUETES METÁLICOS

Cristiane Frantz Arend

Dissertação apresentada ao Curso de Mestrado do Programa de Pós-Graduação em Ciências Odontológicas, Área de Concentração em Odontologia, Ênfase em Ortodontia, da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para a obtenção do grau de **Mestre em Ciências Odontológicas**

Orientador: Prof. Dr. Renésio Armindo Grehs

Santa Maria, RS, Brasil

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elaborada por
Cristiane Frantz Arend

como requisito parcial para obtenção do grau de
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DEDICATÓRIA

*A **Deus** por ter me dado a vida e saúde. Por me dar*
discernimento nas horas de dúvida,
paz nas horas de conflito
e amor nas horas de revolta e indignação.

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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 SOBRE A RESISTÊNCIA DE UNIÃO ENTRE CERÂMICA E BRÁQUETES METÁLICOS

AUTORA: CRISTIANE FRANTZ AREND
ORIENTADOR: RENÉSIO ARMINDO GREHS
Data e Local da Defesa: Santa Maria, 26 de agosto de 2014.

Condicionamento prévio da superfície da cerâmica feldspática é necessário para proporcionar suficiente adesão de bráquetes nessa superfície. O objetivo deste estudo foi avaliar o efeito do condicionamento com diferentes concentrações do ácido fluorídrico (AF) na resistência de união ao cisalhamento de bráquetes metálicos aderidos na superfície de cerâmica feldspática glazeada. Setenta e cinco blocos de cerâmica feldspática glazeada (VM9, Vita Zahnfabrik, Germany) foram confeccionados e randomizados em 5 grupos experimentais: **Ctrl**- silano; **HF1**- AF 1% + silano; **HF3**- AF 3% + silano; **HF5**- AF 5% + silano; **HF10**- AF 10% + silano. AF e silano foram aplicados durante 1 e 5 minutos, respectivamente. Bráquetes metálicos de incisivo central superior (Edgewise Standard slot 022”x030”, Dental Morelli, Brasil) foram colados na superfície da cerâmica com uso de sistema adesivo e resina composta (Transbond^{TM} XT, 3M Unitek, Brasil) e fotopolimerizados. Os espécimes foram envelhecidos por 60 dias (termociclagem 10.000 ciclos 5-55°C; armazenagem em água destilada a 37°C). Teste de cisalhamento foi realizado em uma máquina de ensaio universal e espécimes foram classificados quanto ao Índice de Remanescente Adesivo (IRA). Análises de topografia e ângulo de contato da surfície cerâmica condicionada foram realizadas. Dados foram estatisticamente analisados ($p < 0.05$). Teste de Kuskal-Wallis não detectou diferenças significantes entre os grupos para os valores de resistência de união. ANOVA 1-fator mostrou que o condicionamento teve uma influência significante sobre os resultados de ângulo de contato ($p < 0.00001$), sendo que o grupo controle apresentou a mais alta média (61.8 ± 17.2°) quando comparado aos outros grupos (teste de Tukey). Todos os espécimes apresentaram falha adesiva na interface cerâmica-resina. Com base nos resultados, podemos concluir que o uso do ácido fluorídrico não influenciou significativamente sobre a resistência de união ao cisalhamento de bráquetes metálicos aderidos na superfície de cerâmica felspática glazeada, mesmo que o seu uso promoveu aumento do ângulo de contato superficial e mudou a topografia da superfície.

Palavras-chave: Cerâmica. Ácido fluorídrico. Estresse Mecânico.

ABSTRACT

Master Thesis
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EFFECT OF ETCHING WITH DIFFERENT HYDROFLUORIC ACID CONCENTRATIONS ON BOND STRENGTH BETWEEN GLAZED FELDSPATHIC CERAMIC AND METAL BRACKETS

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ADVISER: RENÉSIO ARMINDO GREHS
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Prior conditioning of the feldspathic ceramic surface is necessary to provide sufficient adhesion of brackets in this surface. The aim of this study was to evaluate the effect of etching with different hydrofluoric acid (HF) concentrations on the shear bond strength between glazed feldspathic ceramic and metal brackets. Seventy-five blocks of glazed feldspathic ceramic (VM9, Vita Zahnfabrik, Germany) were produced and randomized to 5 experimental groups: **Ctrl-** silane application only; **HF1-** HF 1% + silane; **HF3-** HF 3% + silane; **HF5-** HF 5% + silane; **HF10-** HF 10% + silane. HF and silane were applied for 1 and 5 minutes, respectively. Metal brackets of upper central incisor (Edgewise Standard 022"X030" slot, Dental Morelli, Brazil) were bonded on the ceramic surface, using an adhesive system and composite resin (TransbondTM XT, 3M Unitek, Brazil) and light-cured. Specimens were aged for 60 days (thermocycling 10000 cycles 5-55 °C; storage in distilled water at 37 °C). Shear testing was performed in a universal testing machine and specimens were classified for their Adhesive Remnant Index (ARI). Topographical inspection and contact angle analysis of the etched ceramic surfaces were performed. Data were statistically analyzed ($p < 0,05$). Kruskal-Wallis test did not detect significant differences among groups for bond strength values. ANOVA 1-factor showed that the etching had a significant influence on the contact angle results ($p < 0.00001$), since the control group presented the highest mean (61.8 ± 17.2°) when compared with the other groups (Tukey test). All specimens showed adhesive failures at the resin-ceramic interface. Based on the results, we can conclude that the use of HF did not influence significantly the shear bond strength of metal brackets bonded to the glazed feldsphatic ceramic surface, even though its use promoted the increase of the contact angle and changed of the topography surface.

Key Words: Ceramics. Hydrofluoric Acid. Stress, Mechanical.

SUMÁRIO

INTRODUÇÃO

Desde 1955, com a introdução do condicionamento ácido por Buonocore, a colagem de acessórios ortodônticos na superfície de esmalte vem sendo amplamente utilizada como procedimento de rotina na Ortodontia Corretiva (BUONOCORE, 1955).

Pacientes adultos geralmente exibem vários tipos de materiais restauradores na cavidade oral, incluindo cerâmicas. Com a crescente demanda de tratamento ortodôntico corretivo em pacientes adultos (NATTRASS e SANDY, 1995), a colagem de bráquetes sobre a superfície de restaurações tem se tornado um desafio. Isso porque o protocolo atual de tratamento de superfície do esmalte não fornece suficiente adesão quando realizado sobre a superfície da cerâmica (BOURKE e ROCK, 1999; ABU ALHAIJA e AL-WAHADNI, 2007).

Algumas alternativas de tratamento de superfície mecânicos e/ou químicos (ABU ALHAIJA e AL-WAHADNI, 2007) vêm sendo estudados na tentativa de aumentar a adesão entre bráquete-cerâmica. O aumento da rugosidade da superfície da cerâmica com o uso de brocas diamantadas (PRATT et al., 1989; TÜRK et al., 2006) ou jateamento com partículas de óxidos (ZACHRISSON, ZACHRISSON e BUYUKYILMAZ, 1996; KOCADERELI, CANAY e AKCA, 2001) são alguns tratamentos mecânicos sugeridos na literatura. O condicionamento ácido da superfície e/ou o uso de silano (ZACHRISSON, ZACHRISSON e BUYUKYILMAZ, 1996; BOURKE e ROCK, 1999; AMARAL et al., 2008; AMARAL et al., 2011) facilitam a união química da sílica proveniente da cerâmica com a do sistema adesivo e, por isso, são considerados tratamentos químicos da superfície.

Para determinar o tipo de tratamento de superfície a ser escolhido, proporcionando uma resistência de união satisfatória, é importante levar em consideração a composição do material a ser condicionado (VALANDRO et al., 2005; VALANDRO et al., 2006; BOTTINO, FARIA e VALANDRO, 2009). As cerâmicas odontológicas, em geral, são compostas basicamente por duas fases: uma vítrea e uma cristalina. A fase vítrea é formada por uma matriz a base de sílica (óxido de sílica) e a fase cristalina é formada por partículas que podem ser de óxido de alumínio, óxido de zircônia, leucita, dissilicato de lítio, entre outras, as quais são incorporadas à matriz vítrea e têm a finalidade primordial de melhorar as propriedades mecânicas da cerâmica (CONCEIÇÃO, 2005).

Cerâmicas que possuem grande quantidade de matriz vítrea em sua composição, como por exemplo as feldspáticas, são classificadas em cerâmicas ácido sensíveis no que se refere à sensibilidade da superfície diante do condicionamento com ácido fluorídrico (BOTTINO, FARIA e VALANDRO, 2009). Isso porque a sílica da matriz é facilmente atacada quando em contato com esse ácido (ÖZCAN, ALKUMRU e GEMALMAZ, 2001). No entanto, em relação às cerâmicas com baixo ou nenhum conteúdo de sílica, o ácido fluorídrico não contribui para melhorar a capacidade de união dessas cerâmicas aos bráquetes ortodônticos, porque a superfície não se degrada diante do condicionamento com ácido fluorídrico e, assim, tratamentos mecânicos são requeridos (ÖZCAN, ALKUMRU e GEMALMAZ, 2001; BOTTINO, FARIA e VALANDRO, 2009).

Os estudos descrevem que o protocolo de tratamento da superfície interna de coroas feldspáticas com ácido fluorídrico 10% e posterior aplicação de silano resulta em valores de resistência de união adequados (DELLA BONA, ANUSAVICE e SHEN, 2000; BRENTEL et al., 2007), principalmente pela ação do silano que promove adesão química da sílica da cerâmica feldspática com a do sistema adesivo (LU et al., 1992) e, aumenta significativamente, os valores de resistência de união (KAO e JOHNSTON, 1991; KOCADERELI, CANAY e AKCA, 2001).

As colagens de bráquetes ortodônticos são realizadas na superfície externa da cerâmica, a qual geralmente apresenta-se glazeada. O glaze é um material aplicado nas cerâmicas odontológicas para reforçá-la e obter uma superfície menos rugosa, melhorando suas propriedades. Como este material é composto de matriz vítrea, também torna-se suscetível ao condicionamento com ácido fluorídrico (PHILLIPS, 1993). Desta maneira, mesmo não existindo um protocolo bem definido na literatura para o condicionamento de superfície das cerâmicas feldspáticas glazeadas, o ácido fluorídrico em associação com silano, parece promover valores de resistência de união satisfatórios (MAJOR, KOEHLER e MANNING, 1995; GILLIS e REDLICH, 1998; KOCADERELI, CANAY e AKCA, 2001; HARARI et al., 2003; TÜRKKAHRAMAN e KÜÇÜKESMEN, 2006). Contudo, sabe-se que o ácido fluorídrico a 10% é um ácido extremamente tóxico aos tecidos bucais (HAYAKAWA et al., 1992) e que pode promover o enfraquecimento da cerâmica (BRENTEL et al., 2007). Por isso, alguns trabalhos publicados utilizaram diferentes concentrações do ácido fluorídrico no condicionamento superficial da cerâmica feldspática glazeada (MAJOR, KOEHLER e MANNING, 1995; GILLIS e REDLICH, 1998; HARARI et al., 2003; SCHMAGE et al., 2003; TRAKYALI et al., 2009), obtendo valores de resistência de união aceitáveis.

O teste de cisalhamento em uma máquina de ensaio universal é um método eficaz para mensurar resistência de união de bráquetes na superfície de cerâmicas (ZACHRISSON, ZACHRISSON e BUYUKYILMAZ, 1996; COCHRAN et al., 1997). Porém, as análises dos resultados bem como dos tipos de falha, devem ser criteriosos, já que a distribuição de tensões na área de colagem se dá de maneira heterogênea (VAN NOORT et al., 1989; PHILLIPS, 1993; DELLA BONA, ANUSAVICE e MECHOLSKY, 2003; BRAGA et al., 2010).

Apesar de uma transferência direta dos valores de resistência de união para a situação clínica não ser totalmente aceita, porque a adesão bráquete-cerâmica é influenciada por muitos fatores ambientais (ZACHRISSON, 2000), valores de resistência de união máximos não são desejados para tratamento ortodôntico, uma vez que os acessórios são temporários (BOURKE E ROCK, 1999). A adesão ideal deve ser suficientemente forte para suportar as forças ortodônticas e mastigatórias e, ao mesmo tempo adequada para permitir a remoção dos bráquetes, causando mínimo de danos possíveis à superfície da cerâmica (BOURKE e ROCK, 1999). Valores de resistência de união ao cisalhamento entre 6-10 Mpa têm sido relatados, em estudos prévios, como uma força de adesão suficiente para manter bráquetes aderidos à superfície da cerâmica feldspática (COCHRAN et al., 1997; GILLIS e REDLICH, 1998; BOURKE e ROCK, 1999). Forças acima de 13 Mpa poderiam causar falhas coesivas na cerâmica (THURMOND, BARKMEIER e WILWERDING, 1994). Por isso, falhas adesivas na interface adesivo/cerâmica são desejáveis no procedimento de descolagem dos acessórios ortodônticos para evitar fraturas na cerâmica e deixar a superfície livre de resíduos resinosos (SMITH et al., 1988). Essa observação é clinicamente relevante, uma vez que danos macroscópicos à superfície da cerâmica é uma indicação da diminuição da longevidade da restauração (HARARI et al., 2003).

Apesar de existirem estudos que utilizam diferentes concentrações de ácido fluorídrico, poucos autores comparam o efeito de diferentes concentrações desse ácido nos valores de resistência de união ao cisalhamento (TRAKYALI et al., 2009).

O objetivo deste trabalho foi avaliar a resistência de união ao cisalhamento de bráquetes metálicos aderidos na superfície de cerâmica feldspática glazeada condicionada com diferentes concentrações de ácido fluorídrico.

ARTIGO – EFFECT OF THE ETCHING WITH DIFFERENT HYDROFLUORIC ACID CONCENTRATIONS ON BOND STRENGTH BETWEEN GLAZED FELDSPHATIC CERAMIC AND METAL BRACKETS

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Title Page

Effect of Etching with Different Hydrofluoric Acid Concentrations on Bond Strength Between Glazed Feldspathic Ceramic and Metal Brackets

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Abstract

Purpose: To evaluate the effect of etching with different hydrofluoric acid (HF) concentrations on the shear bond strength between glazed feldspathic ceramic and metal brackets.

Materials and Methods: Seventy-five blocks of glazed feldspathic ceramic (VM9, Vita Zahnfabrik, Germany) were produced and randomly allocated to 5 experimental groups: **Ctrl-** silane application only; **HF1-** HF 1% + silane; **HF3-** HF 3% + silane; **HF5-** HF 5% + silane; **HF10-** HF 10% + silane. The ceramic was acid etched for 1 minute, followed by water washing and air spray drying. Metal brackets suitable for upper central incisors (Edgewise Standard 022”x030” slot, Dental Morelli, Brazil) were bonded on the ceramic surface with the use of an adhesive system and light-cured composite resin (Transbond^{TM} XT, 3M Unitek, USA). The specimens were aged for 60 days (thermocycling: 10000 cycles at 5-55 °C; stored in distilled water at 37 °C). Shear testing was performed, and specimens were classified for their Adhesive Remnant Index (ARI). Topographical inspection and contact angle analysis of the etched ceramic surfaces were performed. Data were statistically analyzed ($p < 0.05$).

Results: No significant difference was detected between the shear bond strength of the groups; but surface etching had a significant influence on the contact angle results ($p < 0.00001$). The control group presented the highest mean contact angle (61.8 ± 17.2 °). All specimens showed adhesive failure at the resin-ceramic interface.

Conclusions: HF etching for 1 minute could be considered unnecessary, as it did not significantly influence the shear bond strength.

Keywords: Ceramics. Hydrofluoric Acid. Adhesion. Brackets. Silanization.

Introduction

Patients with ceramic restorations (inlay, metal-ceramic crowns and metal-free) may require orthodontic treatment, and, because the current phosphoric acid enamel surface conditioning protocol does not provide sufficient adhesion when performed on the surface of the ceramic, adhesion problems between the bracket and the ceramic have been observed[3,12].

Some alternative mechanical and/or chemical surface treatments[1,3,8,12,19,25,31,38,42] have been studied in an attempt to increase bracket-ceramic adhesion, however, the type of surface treatment chosen must take into account the composition of the material to be conditioned[17,39,40].

Traditionally, the method of treating the internal surface of the restoration with hydrofluoric acid (HF) followed silanization for the cementation of feldspathic restorations, has provided high bond strength with resin cements[14,24,26]. The mechanism of adhesion in this context is known: HF selectively attacks the silica matrix, generating important micromorphological changes in the ceramic surface to create a micromechanical adhesion, while the silane provides a chemical bond between silica and the resinous material[14,17].

However, when a glazed feldspathic ceramic is conditioned with HF, the context can become hostile, as this surface is rich in silicon dioxide (glass matrix), and therefore, etching of the ceramic surface is not preferably selective, because the whole surface is almost uniformly attacked. Thus, the micromorphological changes may not be effective in promoting sufficient mechanical microretention[6,12,18].

At the same time, it is known that 10% HF is extremely toxic to oral tissues[21] and that it can promote weakening of the ceramic[4,5,22]. Therefore, some published studies used different concentrations of HF in the surface conditioning of glazed feldspathic ceramic[18,20,27,34,35].

Thus, in the context of Orthodontics, there is a clear dichotomy: the need to promote proper adhesion of orthodontic accessories on the ceramic surface and the requirement to prevent negative effects on the mechanical strength of the conditioned material. Accordingly, the study of different concentrations of HF becomes relevant in assessing the potential for micromorphological modification induced by acid application on the surface. This modification is important in creating micromechanical adhesion and it is importante to seek alternatives with lower acid concentrations, which are less harmful to the mechanical strength of the ceramic material, and, as the etching is intraoral, have a lower risk to the patient.

The aim of this study was to evaluate the effect of different HF concentrations on the shear bond strength between the ceramic surface and metal bracket. The research hypothesis tested is that 10% HF promotes the highest shear bond strength.

Materials and Methods

The materials, their manufacturers and respectives compositions are shown in Table 1.

Sample size calculation

Sample size calculations were performed with the program PS (Power and Sample Size 2.1.30), using shear bond strengths obtained from a pilot study (α = 5%; power = 80%).

Seventy-five blocks of feldspathic ceramic (VM9 enamel, Vita Zahnfabrik, Germany) (n = 15) were manufactured by a single operator mixing powder and liquid modeler (VITA MODELLING FLUID, Vita Zahnfabrik, Germany). The homogeneous mass was inserted in a 12.5 × 12.5 × 10mm (width x length x depth) metal template, which was previously lubricated with mineral oil (Quimidrol, Joinville, Brazil). The ceramic mass was compacted using a metal piston, with slightly smaller dimensions than those of the template, aided by disposable tissue paper (Kleenex® Classic, Kimberly-Clark, São Paulo, Brazil), which kept contact with the ceramic mass for removing excess fluid.

The blocks were sintered in a ceramic furnace (VITA VACUMAT 6000MP, Vita Zahnfabrik, Germany) using a firing cycle recommended by the manufacturer. The ceramics shrank assuming final dimensions of 9 × 9 × 4mm.

The top of the feldspathic ceramic blocks was sanded manually with 220 grit sandpaper (3M ESPE, USA) until it was flat. All specimens were marked with a waterproof pen (Sharpie ® permanent marker, São Paulo, Brazil) on the opposite face from sanding, cleaned in an ultrasonic bath (Vitasonic, Vita Zanhfabrik, Germany) with distilled water for 10 minutes, dried, and glazed.

The surface glazing was performed through the application of glaze obtained by mixing powder (AKZENT GLAZE®, Vita Zahnfabrik, Germany) and liquid (FLUID LIQUID VITA, Vita Zahnfabrik, Germany). The specimens were submitted to glaze firing as recommended by the manufacturer.

The glazed surface was examined in a stereomicroscope (Discovery V20, Carl Zeiss, Göttingen, Germany) at 7.5× magnification, and specimens that showed bubbles or surface flaws were replaced. All specimens were embedded in acrylic resin (Classic, São Paulo, Brazil), keeping only the glazed surface exposed. Specimens were randomly allocated (Random Allocation Software 1.00) to 5 experimental groups, considering the surface treatment (Table 2).

Bonding procedures

Specimens were first cleaned in an ultrasonic bath under distilled water. HF gel (FGM, Joinville, Brazil) was applied for 1 minute, washed with an air/water spray for 10 seconds and dried with air free of contamination, moisture and oil. The etched surface was silanized using MPS-based silane (3-methacryloxypropyltrimethoxysilane in ethanol) (Rely X™ ceramic primer, 3M ESPE, USA) for 5 minutes. The different acid concentrations were made with acid from the same manufacturer. Following the International Organization for Standardization's (ISO's) recommendations[23], black square stickers with an internal opening of 5 × 5mm were fixed on the conditioned surface to define the area of adhesion. Adhesive (Transbond™ XT, 3M Unitek, USA) was applied on the exposed surface and light-cured for 30 seconds with an LED curing light[36] (Radii-cal SDI, Australia) with an output of 1200 mW/cm².

The bracket base (Edgewise Standard slot 022”x030”, Dental Morelli, Sorocaba, Brazil) was covered with light-cured composite resin (Transbond™ XT, 3M Unitek, Monrovia, California, USA) and positioned on the exposed ceramic surface. Excess composite was removed with an explorer (Duflex, Brazil) while the bracket was stabilized by applying a 600g load for 10 seconds with a Gilmore needle to allow the thickness of the composite resin in the bracket-ceramic interface to standardize, and then light-cured for 40 seconds – 10 seconds on each side of the bracket. The total surface area of bracket base, provided by the manufacturer, is 14.8 mm². This bracket was selected due to its regular base and nearly flat geometry, important considerations when performing shear tests[33]. All specimens were stored in distilled water for 24 hours at 37 °C.

Aging

The specimens were submitted to thermocycling (10000 cycles, between 5-55 °C), with a dwell time of 30 seconds in each bath, according ISO[22], as well as 50 days of storage under distilled water at 37 °C. The water was changed every 7 days.

To prevent damage whilst moving the specimens, they were fixed in the receiver of the thermocycler.

Shear bond strength

Specimens were placed in a fixed device in a universal testing machine (EMIC DL-1000, São José dos Pinhais, Brazil) and positioned parallel to the long axis of the load application device. The bracket was carefully positioned such that the point of load application was perpendicular to the crosshead. Load was applied by a flat rod positioned between the base, and the wings of the bracket closest to the adhesive interface, until fracture occured. The load was applied at a speed of 1mm/min[22]. Force obtained in Newtons (N), which was divided by the bracket area (mm^2) to calculate the shear bond strength (MPa).

Failure analysis

After shear testing, the ceramic surfaces were analyzed under a stereomicroscope at 7.5× magnification for scoring the Adhesive Remnant Index (ARI), initially proposed by Artun and Berglan[11], and suitable for use on ceramic surfaces. Each specimen was scored to establish the amount of composite on the feldspathic surface, according to the following classification:

0 = no adhesive left on the ceramic surface

1 = less than half of the adhesive left on the ceramic surface

2 = more than half of the adhesive left on the ceramic surface

3 = all adhesive left on the ceramic surface

4 = fracture of ceramic

Topography inspection

Ten additional specimens (n = 2/group) were manufactured for analysis by Atomic Force Microscopy (AFM) (Agilent Technologies 5500 equipment, Chandler, Arizona, USA) and Scanning Electron Microscopy (SEM) (Jeol-JSM-T330A, Jeol Ltd, Tokyo, Japan).

AFM images were collected in non-contact mode using PPP-NCL probes (Nanosensors, Force constant = 48N/m). AFM micrographs were analyzed using scanning probe microscopy data analysis software (GwyddionTM version 2.33, GNU, Free Software Foundation, Boston MA, USA).

SEM scans an electron beam over the specimen surface in x and y lines. Specimens were gold coated prior to analysis. The obtained images were standardized at 500× magnification.

AFM and SEM images were obtained to illustrate surface topography. AFM and SEM analyzes were carried out only with the application of hydrofluoric acid by 1 minute on surface, washed by 10 seconds and dried.

Contact angle analysis

Ten additional specimens (n = 2/group) were manufactured for contact angle analysis. Hydrofluoric acid were applied on surface, washed and dried. The values were obtained using a goniometer (Krüss; Hamburg, Germany) under controlled temperature. One drop of distilled water was put on the ceramic surface with a syringe, and after 5 seconds[24] an image was taken and the contact angle calculated by software analysis. Five measurements were made each specimen, and an average per group is reported (Table 2). Representative images were captured.

Statistical Analysis

Data were statistically analyzed, using Statistix 8.0 (Analytical Software Inc., Tallahassee, FL, USA), considering a significance level of 5%. Tests of normality and homogeneity of variances were performed with the shear bond strengths and contact angles. The non-parametric Kruskal-Wallis test was applied to the shear bond strength data. One-way ANOVA and post-hoc Tukey's tests were applied to the contact angle data.

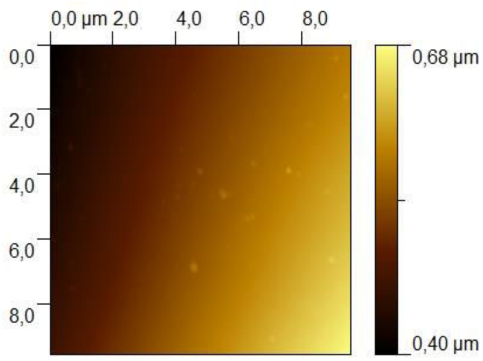
Results

Shear bond strength

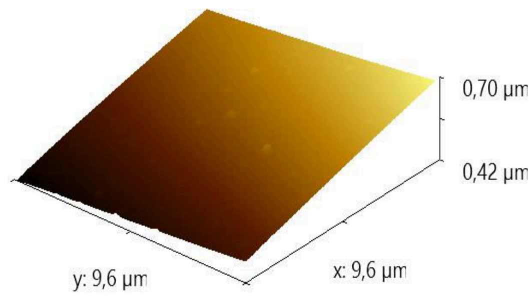
Shear bond strength means and standard deviations of the different groups are shown in Table 2. The HF1 group presented the highest mean (4.1 MPa), whilst the HF10 presented the lowest mean (1.1 MPa). Kruskal-Wallis test showed no significant differences of shear bond strength within groups ($p = 0.31$).

According to the ARI, all specimens received a score of 0, because all composite remained bonded on the bracket base, i.e. there was complete adhesive failure at the resin-ceramic interface (Table 2).

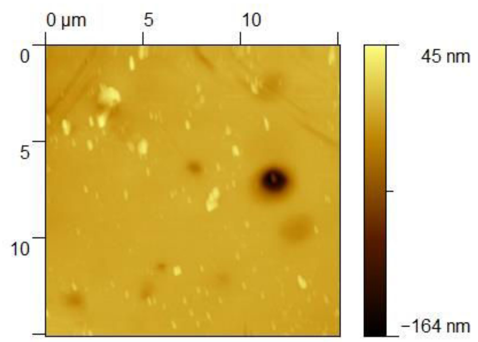
Changes in the surface topography of glazed feldspathic ceramic submitted to different etchings are shown in Figure 1 and Figure 2. Note that etching did not occur homogeneously on ceramic surfaces, which may have occurred due to a lack of standardization of glazing and acid application, even if the application time and the viscosity of the acid were controlled.



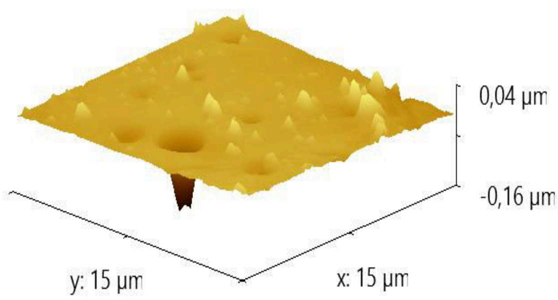
Ctrl-a



Ctrl-b



HF1-a



HF1-b

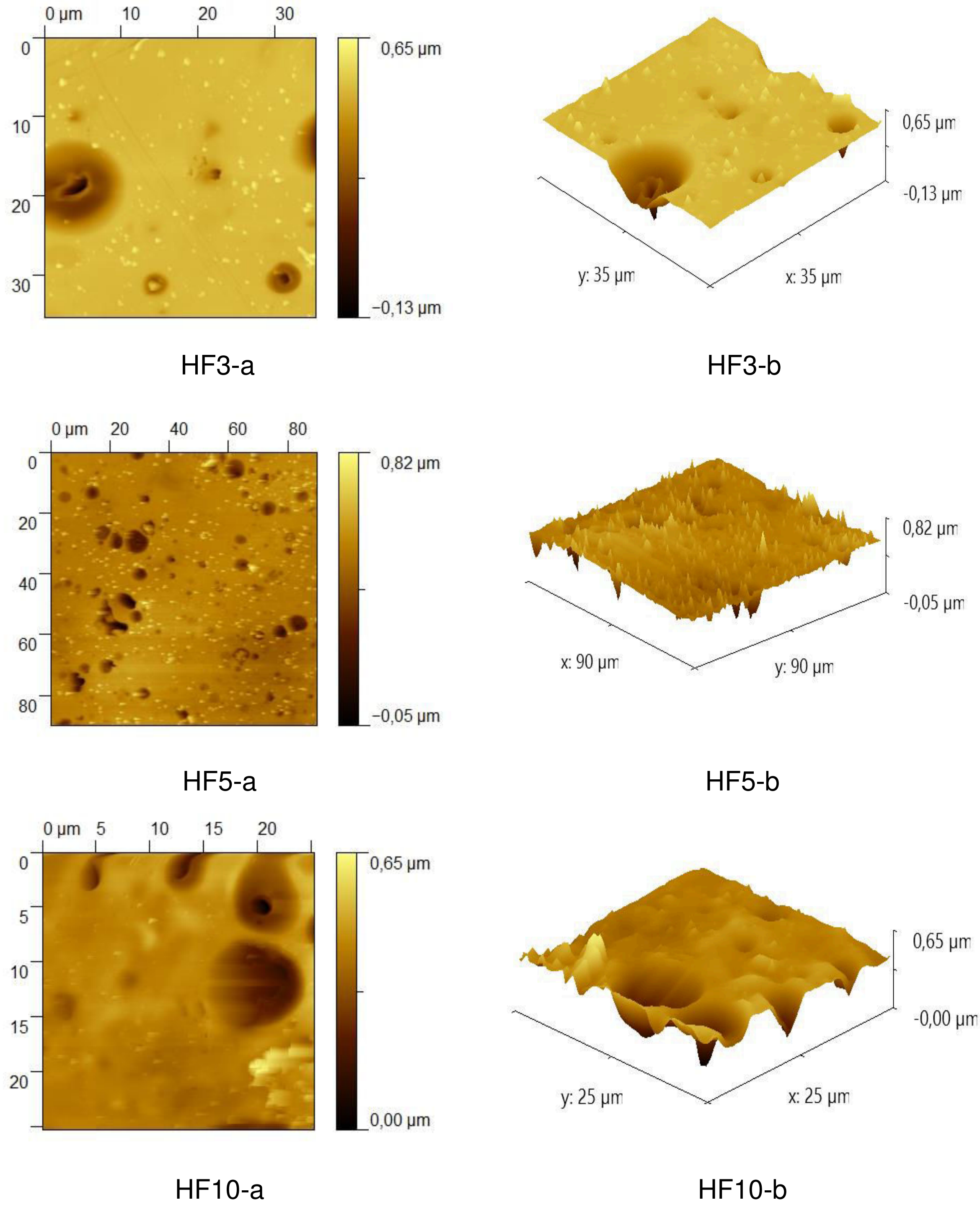


Figure 1 – Representative imagens obtained by an AFM of different ceramic surface conditioning by 1minute (a: two-dimensional image; b: three-dimensional image). Ctrl- glazed ceramic; HF1- HF 1%; HF3- HF 3%; HF5- HF 5%; HF10- HF 10%.

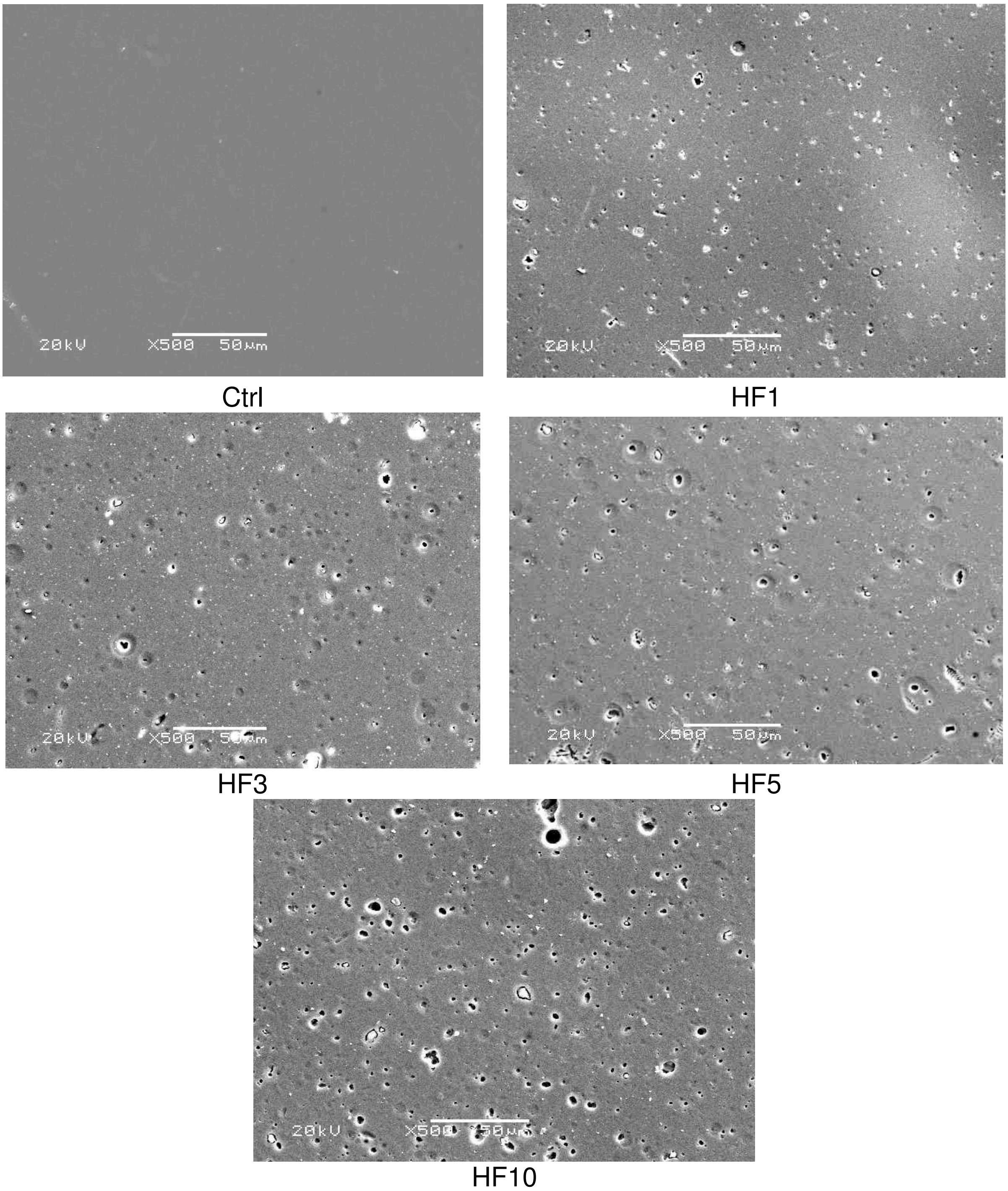


Figure 2 – Representative images obtained in SEM of different ceramic surface conditioning for 1minute followed by washing + drying (500×). Ctrl- glazed ceramic; HF1- HF 1%; HF3- HF 3%; HF5- HF 5%; HF10- HF 10%.

Contact angle

Contact angle means and standard deviations are shown in Table 2. The one-way ANOVA test showed that surface etching had a significant influence on the contact angle ($p < 0.00001$). Tukey’s test showed that statistically the control group presented the highest mean values.

These results reveal that despite the use of HF to change the surface of the glazed feldspathic ceramic, it was not sufficient to promote an increase in shear bond strength.

Representative images of contact angle analysis can be seen in Figure 3.

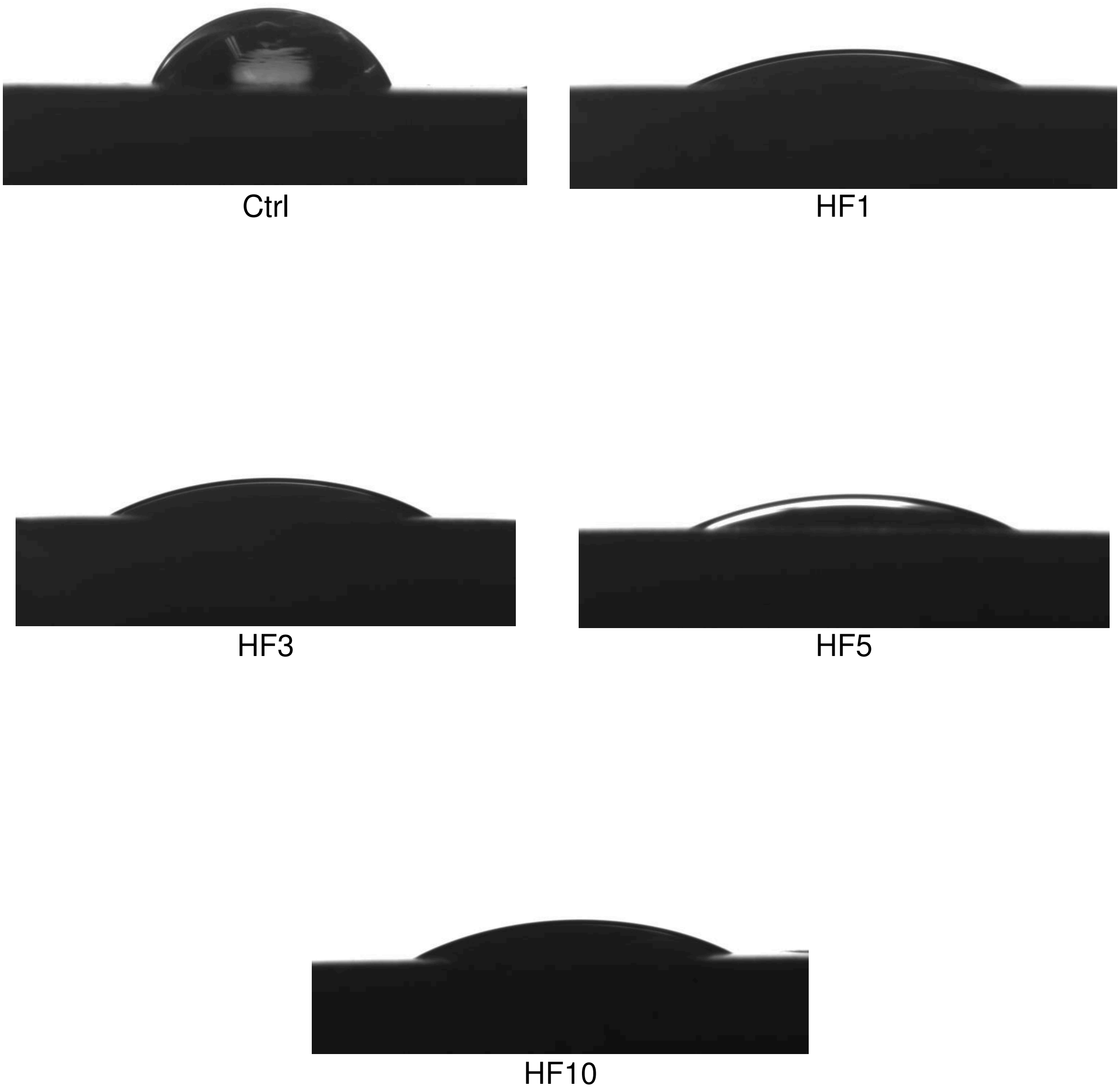


Figure 3 – Photographs of contact angle on feldspathic surfaces submitted to: Ctrl- silane application only; HF1- HF 1%; HF3- HF 3%; HF5- HF 5%; HF10- HF 10%. HF was applied for 1minute on glazed surface.

Discussion

Hydrofluoric acid is applied to glazed feldspathic ceramic surfaces to increase micromechanical retention, and to prepare the surface prior to silane application[7,29]. The results of this study showed that the HF concentrations tested did not significantly influence the obtained shear bond strength of metal brackets adhered to glazed feldspathic ceramic surfaces. This is in agreement with the findings of Trakyali et al.[35] which compared HF concentrations of 5% and 9.6%. 10% HF did not show the highest mean shear bond strength, and, as such, the tested hypothesis was rejected.

Hydrofluoric acid changes the topography of glazed feldspathic ceramic surfaces (Figure 1 and Figure 2). The SEM images show that HF dissolves the glass matrix of the glaze layer, creating pits on the surface, and that the changes created by applying 1%, 3% and 5% HF does not appear to be regular. However, we note that the HF does not uniformly attack the glazed ceramic surface. This could be explained by the lack of standardization of the glaze layer thickness, amount of powder and liquid in the glaze mass.

Low shear bond strengths were found in this study (1.1-4.1 MPa); lower than those reported in previous studies (6-10 MPa)[12,15,18]. This could be due to the long aging process, as this procedure decreases the bond strength[12,41]. In this study, we submitted specimens to 10000 cycles, whereas Bourke and Rock[12] used 500 cycles and found adequate shear bond strengths, confirming the findings of other authors[2,37].

The artificial aging effect induced by thermocycling can degrade the interface by two mechanisms: (1) hot water may accelerate hydrolysis of poorly polymerized resin oligomers; and (2) repetitive contraction/expansion stresses can be generated in the interface[16].

Given that the materials used in this study have different thermal expansion coefficients (feldspathic ceramic: 8.8-9.2 x 10^{-6} K^{-1} at 25-500 °C; composite resin: 14-50 x 10^{-6} K^{-1} at 20 °C and metal brackets: 17.3 x 10^{-6} K^{-1} at 20 °C)[10] and that they were tested during thermocycling, the stress generated at the interface may have contributed to the appearance of gaps and a more severe degradation of the bracket-ceramic interface than that by water action alone.

The silane composition can also have an influence on the bond strength[35]. Rely X ceramic primer (3M ESPE, USA) a prehydrolyzed silane consisting <1% aminosilano, 70-80% ethanol and 20-30% water[28], is the most stable. This material can cause adhesion instability in moist conditions, because a hydrolyzed silane is less stable in the container, and, due to its high affinity for atmospheric humidity, can degrade before use[9].

10% HF showed the lowest shear bond strength and this can be explained by the fact that 10% HF is a strong and aggressive acid, and created deep pores in the ceramic surface that couldn't be filled by the adhesive[29], weakening the structure[14]. An ARI score of 0 was given, as no cohesive failure was observed after debonding (Table 1). It showing that the measured shear bond strength is representative of adhesion between the ceramic and adhesive system[42].

The contact angles decreased significantly after the application of HF, as the acid increases the surface free energy of the substrate and the wettability of the adherent on the ceramic surface[26], increasing the potential of adhesion[24]. Contact angle findings were not reflected in the shear bond strength results.

Both shear bond strength and contact angle results exhibited high standard deviations (±5.9 to shear bond strength; ±19.9 to contact angle) and could be attributed to variations in operator technique in glazing and lack of uniformity in etching[31].

There is no consensus about HF action time on glazed ceramics in the literature; studies advocated a 1-4 minute application[2,3,6,7,12,35,37,38,41,42]. In this study, HF was used for 1 minute on the glazed feldspathic ceramic, following the manufacturer's instructions, and the exposure time did not appear to be enough to create adequate micromechanical retention.

Increasing the time of HF application on the ceramic surface could be an alternative to enhance adherence and to increase the shear bond strengt, however, an increase in etching time may lead to a reduction in the mechanical strength of the ceramic material. Addison et al.[5] studied the impact of HF surface etching on flexural strength of feldspathic ceramics and they observed a significant strength reduction with increasing etching time.

Also, due to the high toxicity of HF, there is difficulty in managing this acid in the oral cavity. No studies were found on the hazardous effects due to HF exposure in the dental literature, but a published review[30] states that HF has a high tissue penetration power, which may cause irritation, burns, haemorrhages, necrosis, and death, depending on the tissue involved, and acid quantity and concentration.

A question still seems unclear: Which superficial changes are needed to promote adequate bond strength, considering that the bonding of brackets is temporary?

In terms of bond mechanical tests, the current investigation used the shear test, which presents a main limitation: non-homogeneous stress distribution at the interfaces[13]. Even with this limitation, the observed failures, as previously mentioned, were adhesive failures at the resin-ceramic interface, which means the real shear bond strength was evaluated by this study.

Conclusion

It is clearly observed that the adhesion of brackets on glazed surfaces is weak and unstable, as the etching with 10% HF for 1 minute followed by silanization showed low shear bond strength.

The methodology employed in this study were not satisfactory to create a adequate adhesion. The findings of this study should be interpreted with caution, because *in vitro* studies have limits, as it is difficult to reproduce of the complex oral environment.

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Clinical Relevance

The use of HF appears to be unnecessary, as it did not increase the shear bond strength. However, an increase of etching time could be considered. Randomized clinical trials are required to extrapolate these findings to clinical practice.

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Table 1 – Materials, manufacturer and composition

Material	Manufacturer (*#batch*)	Composition
VM9 (ceramic)	Vita Zanhfabrik, Germany (#29220, #38590, #38780)	60-64% silic oxide (powder)
Vita Akzent (glaze)	Vita Zanhfabrik, Germany (#23750)	56-58% silic oxide (powder) and about 99% polyhydric alcohol (liquid)
Hydrofluoric acid gel	FGM, Brazil (#150812 and #07102013)	1%, 3%, 5% and 10% HF, water, thickener, surfactant and dye
Ceramic primer (silane)	3M ESPE, USA (#N167818)	<1% aminosilano, 70-80% etanol and 20-30% water
Transbond^{TM} XT primer	3M, Unitek, USA (#N396510)	Bisphenol A diglycidyl ether dimethacrylate, triethylene glycol dimethacrylate
Transbond^{TM} XT paste	3M, Unitek, USA (#9HG)	Silane-treated quartz, bisphenol A diglycidyl ether dimethacrylate, bisphenol A bis(2-hydroxyethyl ether) dimethacrylate, dichlorodimethylsilane reaction product with silica
Edgewise brackets	Dental Morelli, Brazil (#1809112)	alloy of chromium and nickel

Table 2 – Means and standard deviation of the shear bond strength and contact angle of experimental groups and Adhesive Remnant Index (ARI).

Groups	Surface treatment	Bond strength (Mpa)	Contact Angle (angle)	ARI
Ctrl	No etching + silane	1.7 ± 3.3	61.8 ± 17.2^{A}	100% score 0
HF1	HF 1% + silane	4.1 ± 4.4	33.2 ± 19.9^{B}	100% score 0
HF3	HF 3% + silane	1.3 ± 1.8	30.3 ± 7.3^{B}	100% score 0
HF5	HF 5% + silane	3.2 ± 5.9	30.6 ± 9.0^{B}	100% score 0
HF10	HF 10% + silane	1.1 ± 1.7	28.4 ± 6.0^{B}	100% score 0

The same superscript letters indicate no significant differences and different letters mean significant statistical difference for contact angle values (Tukey's test, α=5%).

CONCLUSÃO

Apesar dos objetivos propostos para este estudo serem todos cumpridos, baixos valores de resistência de união ao cisalhamento foram encontrados, mais baixos do que aqueles reportados em estudos prévios como uma força ideal para manter bráquetes aderidos na superfície da cerâmica (6-10MPa) (BOURKE e ROCK, 1999; COCHRAN et al., 1997; GILLIS E REDLICH, 1998).

Talvez, a execução de novas pesquisas que testassem a variável tempo de aplicação em associação com as diferentes concentrações do ácido fluorídrico sobre a superfície da cerâmica feldspática glazeada seria uma alternativa para tentar melhorar esses valores de resistência de união.

Com base nos resultados deste estudo, o uso do ácido fluorídrico poderia ser considerado desnecessário, uma vez que não influenciou significativamente na resistência de união ao cisalhamento.

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ANEXOS

Anexo A – Normas para publicação no periódico *The Journal of Adhesive Dentistry*

The Journal of
Adhesive Dentistry

GUIDELINES FOR AUTHORS

The Journal of Adhesive Dentistry is a bi-monthly jornal 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 adhesive 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 servisse (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 hotographs 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 PCword 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).

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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 ffiliation, 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 communications 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 separate 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 uthor.
• 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.