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ORIGINAL ARTICLE
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Effect of etching with different hydrofluoric acid concentrations on bond strength between glazed feldspathic ceramic and metal brackets

Efeito de diferentes concentrações de ácido fluorídrico na resistência adesiva entre uma cerâmica feldspatica glazeada e bráquetes metálicos

Cristiane Frantz AREND[1], André GÜNDEL[2], Renésio Armindo GREHS[1], Vilmar Antônio FERRAZZO[1], Luiz Felipe VALANDRO[3]

1 – Graduate Program in Oral Science (Orthodontic Unit) – Federal University of Santa Maria – Santa Maria – RS – Brazil.

2 – Physic School – Federal University of Pampa –Bagé – RS – Brazil.

3 – Graduate Program in Oral Science (Prosthodontic Unit) – Federal University of Santa Maria – Santa Maria – RS – Brazil.

ABSTRACT

Objective: The aim of this study was evaluate the effect of etching with different hydrofluoric acid (HF) concentrations on the shear bond strength between glazed feldspathic ceramic and metal brackets. **Material and Methods:** –Seventy-five blocks of glazed feldspathic ceramic were produced and randomly allocated to 5 groups: Ctrl- silane application only; HF1- HF1%+silane; HF3- HF3%+silane; HF5- HF5%+silane; HF10- HF10%+silane (standard procedures: etching for 1 min + washing + drying + silanization). Metal brackets for upper central incisors (Edgewise Standard) were bonded on the ceramic surface with the use of an adhesive system and light-cured composite resin (TransbondTM XT, 3M). The specimens were aged for 60 days (thermocycling: 10000x 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 using the non-parametric Kruskal-Wallis test. One-way ANOVA and post-hoc Tukey's tests were applied to the contact angle data ($p<0.05$). **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. **Conclusion**

RESUMO

Objetivo: O objetivo deste estudo foi avaliar o efeito do condicionamento com diferentes concentrações de ácido fluorídrico (AF) na resistência de união ao cisalhamento entre cerâmica feldspática glazeada e bráquetes metálicos. **Material e Métodos:** Setenta e cinco blocos de cerâmica feldspática glazeada foram produzidos e distribuídos aleatoriamente em 5 grupos: Ctrl- silano; HF1- AF 1% + silano; HF3- AF 3% + silano; HF5- AF 5% + silano; HF10- AF 10% + silano (procedimento padrão: condicionamento por 1 min + lavagem + secagem + silanização). Bráquetes metálicos de incisivo central superior (Edgewise Standard) foram colados sobre a superfície da cerâmica com o uso de um sistema adesivo e resina composta fotopolimerizável (TransbondTM XT, 3M). Os espécimes foram envelhecidos por 60 dias (termociclagem: 10000x a 5-55°C; estocados em água destilada a 37°C). O teste de cisalhamento foi realizado e espécimes foram classificados quanto ao seu Índice de Remanescente Adesivo (IRA). Inspeção topográfica e análise do ângulo de contato da superfície da cerâmica condicionada foram realizados. Dados de resistência adesiva foram analisados estatisticamente usando o teste de Kruskal-Wallis, enquanto os dados de ângulo de contato foram analisados com ANOVA 1-fator e teste de Tukey ($p<0.05$). **Resultados:** Nenhuma diferença significativa foi detectada para a resistência de união entre os grupos; mas o condicionamento da superfície teve uma influência significativa sobre os resultados de ângulo de contato ($p<0.00001$). O grupo controle apresentou a mais alta média de ângulo de contato (61,8± 17,2°). Todos os espécimes apresentaram falhas adesivas na interface

The tested HF concentrations did not significantly influence the obtained shear bond strength of metal brackets adhered to glazed feldspathic ceramic surfaces.

KEYWORDS

Ceramics; Hydrofluoric acid; Brackets.

cerâmica-resina. **Conclusão:** As concentrações de AF testadas não influenciaram significativamente a resistência de união de bráquetes metálicos aderidos na superfície de cerâmica feldspática glazeada.

PALAVRAS-CHAVE

Cerâmicas; Ácido Fluorídrico; Bráquetes.

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 [1,2].

Some alternative mechanical and/or chemical surface treatmentshave 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 [1-5].

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. 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 [6-8].

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 [2,9,10].

At the same time, it is known that 10% HF is extremely toxic to oral tissues [11] and that it can promote weakening of the ceramic [12-14]. Therefore, some published studies used different concentrations of HF in the surface conditioning of glazed feldspathic ceramic [10,15-18].

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.

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™ XT primer	3M, Unitek, USA (#N396510)	Bisphenol A diglycidyl ether dimethacrylate, triethylene glycol dimethacrylate
Transbond™ 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

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%).

Block ceramic production

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 past was inserted in a 12.5 × 12.5 × 10mm (width × length × 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 ground 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 testing groups, considering the surface treatment (Table 2).

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%).

Bonding procedures

Specimens were first cleaned in an ultrasonic bath under distilled water.HF gel (FGM, Joinville, Brazil) was applied for 1 min, washed with an air/water spray for 10 s and dried with air free of contamination, moisture and oil. The etched surface was silanized using MPS-based silane(3-methacryloxypropyltrim ethoxysilane in ethanol) (Rely XTM ceramic primer, 3M ESPE, USA) for 5 min. The different acid concentrations were made with acid from the same manufacturer. Following the International Organization for Standardization's (ISO's) recommendations, black square stickers with an internal opening of 5 × 5mm were fixed on the conditioned surface to define the area of adhesion [19]. Adhesive (TransbondTM XT, 3M Unitek, USA) was applied on the exposed surface and light-cured for 30 s with an LED curing light(Radii-cal SDI, Australia) with an output of 1200 mW/cm².

The bracket base (Edgewise Standard slot 022” × 030”, Dental Morelli, Sorocaba, Brazil) was covered with light-cured composite resin (TransbondTM 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 s 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 s – 10 s 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 [20]. All specimens were stored in distilled water for 24 h at 37 °C.

Aging

The specimens were submitted to thermocycling (10000 cycles, between 5-55 °C), with a dwell time of 30 s in each bath, according ISO [19], 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 [19]. Force obtained in Newtons (N), which was divided by the bracket area (mm²) 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 [21], 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 min on surface, washed by 10 s 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 s [13] 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.

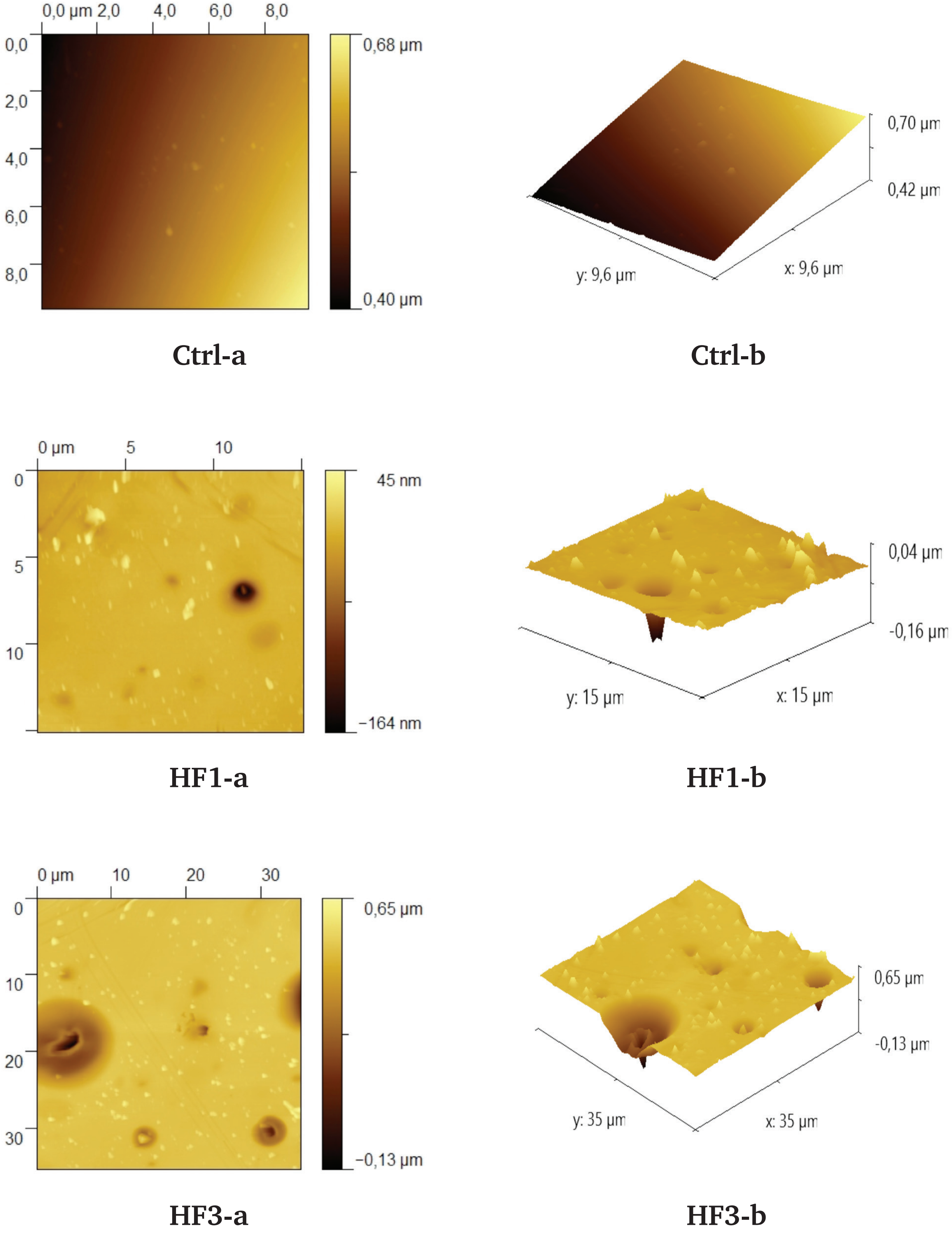
RESULTS

Shear bond strength

Shear bond strength means and standard deviations of the different groups are shown in Table 2. The bond strengths of the groups were statistically similar (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.


0,0 µm 2,0
4,0
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HF1-a
0,04 µm
-0,16 µm
y: 15 µm
x: 15 µm
HF1-b
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HF3-a
0,65 µm
-0,13 µm
y: 35 µm
x: 35 µm
HF3-b

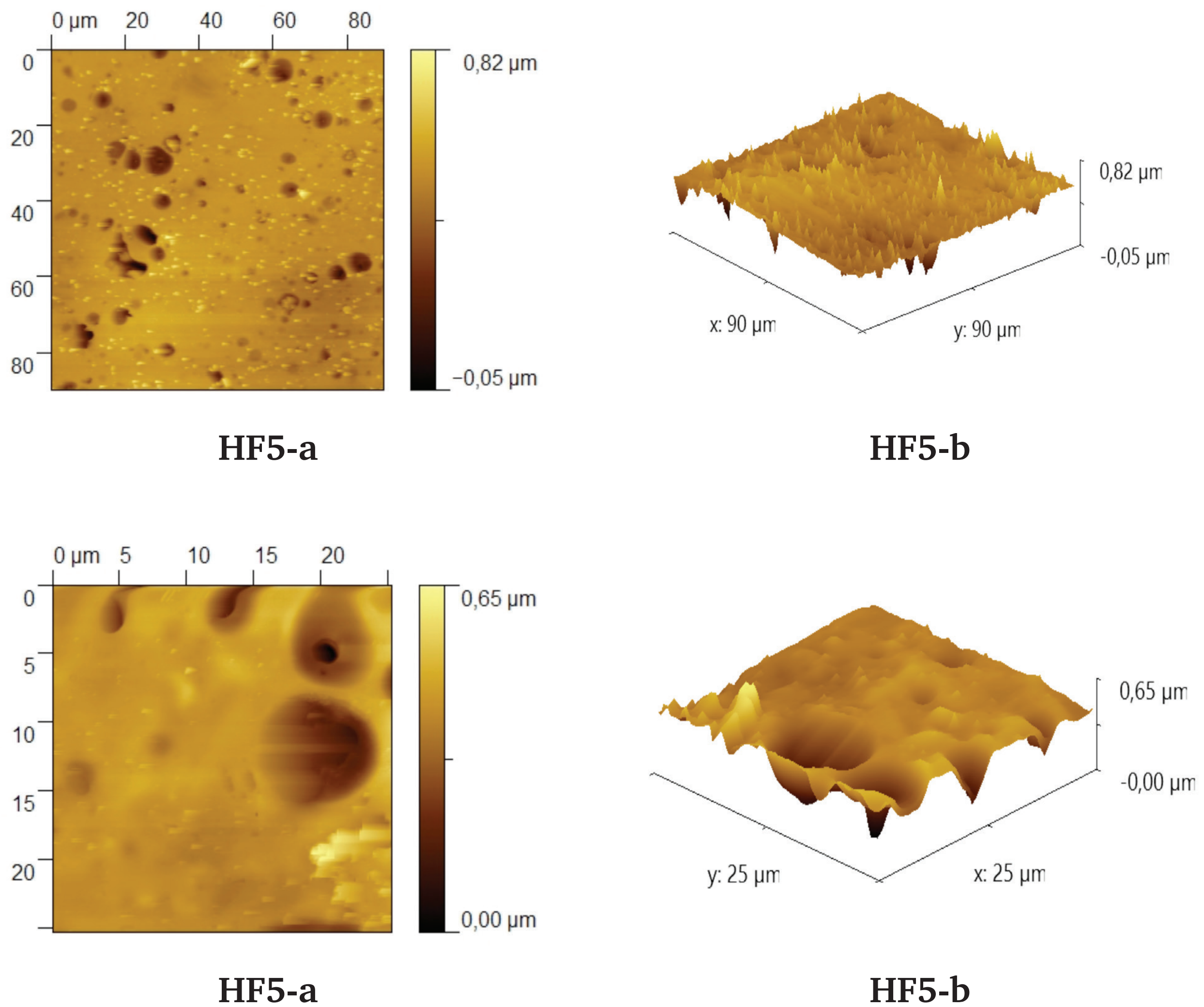

Figure 1 - Representative imagens obtained by an AFM of different ceramic surface conditioning by 1 minute (a: two-dimensional image; b: three-dimensional image). Ctrl- glazed ceramic; HF1- HF 1%; HF3- HF 3%; HF5- HF 5%; HF10- HF 10%.

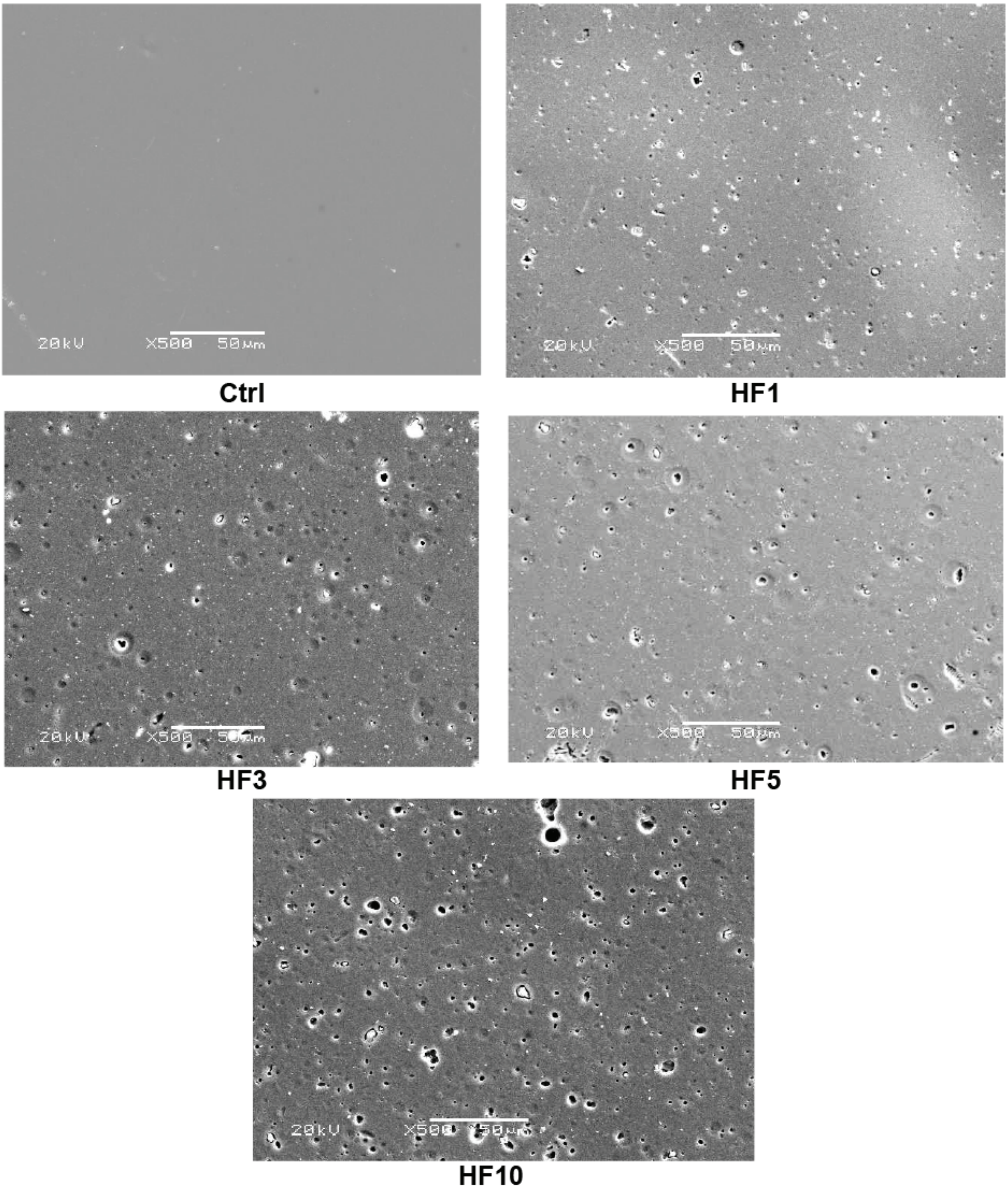

Figure 2 - Representative images obtained in SEM of different ceramic surface conditioning for 1minute followed by washing Ü drying (500×). Ctrl- glazed ceramic; HF1- HF 1%; HF3- HF 3%; HF5- HF 5%; HF10- HF 10%.

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.

DISCUSSION

Hydrofluoric acid is applied to glazed feldspathic ceramic surfaces to increase micromechanical retention, and to prepare the surface prior to silane application [22]. 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 Trakyaliet al. [18],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) [2-16].

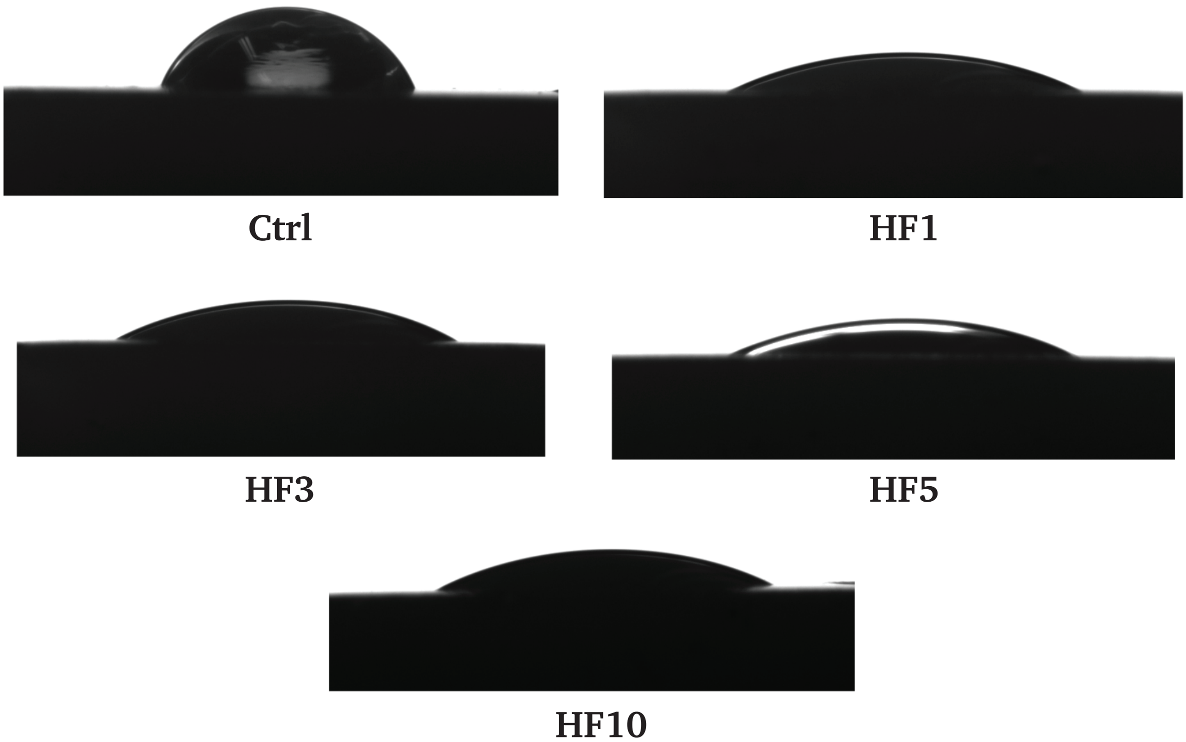

Figure 3 - Photographs of contact angle on feldspathic surfaces submitted to: Ctrl- silane application only; HF1- HF 1%; HF3- HF 3%; HF5- HF 5%; HF10- HF 10%. HF was applied for 1minute on glazed surface.

This could be due to the long aging process, as this procedure decreases the bond strength [2]. In this study, we submitted specimens to 10000 cycles, whereas Bourke, Rock [2]used 500 cycles and found adequate shear bond strengths, confirming the findings of other authors [23,24].

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 [25].

Given that the materials used in this study have different thermal expansion coefficients (feldspathic ceramic: 8.8-9.2 × 10^{-6} K-1 at 25-500 °C; composite resin: 14-50 × 10^{-6} K-1 at 20 °C and metal brackets: 17.3 × 10^{-6} K-1 at 20 °C) 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 [26].

The silane composition can also have an influence on the bond strength [18]. RelyXceramic primer (3M ESPE, USA) a prehydrolyzedsilane consisting <1% aminosilano, 70-80% ethanol and 20-30% water, is the most stable [27]. 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 [28].

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, weakening the structure [12]. 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 [5].

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, increasing the potential of adhesion [7,8]. 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.

There is no consensus about HF action time on glazed ceramics in the literature; studies advocated a 1-4 min application [1,2,4,5,9,18,22-24].In this study, HF was used for 1 min 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 strength, however, an increase in etching time may lead to a reduction in the mechanical strength of the ceramic material. Addison et al.studied the impact of HF surface etching on flexural strength of feldspathic ceramics and they observed a significant strength reduction with increasing etching time [13].

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 reviewstates that HF has a hightissue penetration power, which may cause irritation, burns, haemorrhages, necrosis, and death, depending on the tissue involved, and acid quantity and concentration [22].

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 [29]. 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.

CONCLUSIONS

The different concentrations of hydrofluoric acid did not significantly influence the shear bond strength of metal brackets adhered to glazed feldspathic ceramic surfaces. The hypothesis tested that 10% HF promotes the highest shear bond strength was rejected.

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The authors claim no conflict of interest.

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Luiz Felipe Valandro
(Corresponding address)
D.D.S., M.Sci.D., Ph.D., Associate Professor,
Federal University of Santa Maria
Faculty of Odontology
R. Floriano Peixoto, 1184, 97015-372,
Rio Grande do Sul State, Santa Maria, Brazil.
E-mail: lfvalandro@hotmail.com

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