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Influence of hydrofluoric acid etching time and concentration on shear bond strength of metal brackets to ceramic surfaces

Influência do tempo e concentração do ácido fluorídrico na resistência de união do braquete metálico à superfície cerâmica

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Resumo

Introdução: O tratamento de superfície prévio a colagem de braquetes a cerâmica com ácido fluorídrico, é indicado devido a sua capacidade de promover alterações morfológicas necessárias para a adesão. **Objetivo:** Avaliar a resistência de união ao cisalhamento (RUC) de braquetes metálicos unidos à superfície cerâmica feldspática sob a ação do ácido fluorídrico (AF), em diferentes concentrações (5% e 10%) e tempos de aplicação distintos (30 e 60 segundos). **Material e método:** Foram utilizados quatro blocos metálicos em níquel-cromo, que receberam a aplicação de cerâmica feldspática, onde foram colados 80 braquetes metálicos (Abzil/3M), divididos em 4 grupos conforme o condicionamento ácido (n=20). Foram condicionados com ácido fluorídrico 5%, por 30 e 60 segundos (AF5/30 e AF5/60) e ácido fluorídrico 10%, por 30 e 60 segundos (AF10/30, AF10/60). O compósito resinoso utilizado foi o Transbond XT (3M) e na superfície cerâmica foi mantido a presença do glazer. Os espécimes foram posicionados na máquina de ensaio universal Instron 4411 (Instron Corp, EUA) com um cinzel adaptado, para teste de cisalhamento, com velocidade de 1mm/min. Os dados foram submetidos a análise de variância (ANOVA) e o Índice de Remanescente Adesivo (IRA) foi avaliado. **Resultado:** No tempo de 30 segundos não ocorreu diferença significativa para as concentrações de 5% e 10% do ácido fluorídrico. No tempo de 60 segundos, para a concentração de 10% houve uma força de cisalhamento significativamente maior ($p<0,05$). O IRA apresentou predominância dos escores 1 e 2. **Conclusão:** Concluiu-se que o ácido fluorídrico 10% apresentou os maiores valores de resistência de união ao cisalhamento em 60 segundos de condicionamento, enquanto, o ácido fluorídrico 5% não demonstrou diferença significativa entre os tempos.

Descritores: Condicionamento ácido; cerâmica; resistência ao cisalhamento; braquete ortodôntico.

Abstract

Introduction: Surface treatment prior to bonding ceramic brackets with hydrofluoric acid is indicated because of its ability to promote morphological changes necessary for adhesion. **Objective:** To evaluate the shear bond strength (RUC) of metal brackets bonded to the feldspar ceramic surface under the action of hydrofluoric acid (AF), in different concentrations (5% and 10%) and different application times (30 and 60 seconds). **Material and method:** Four nickel-chrome metal blocks that received an application of feldspathic ceramic were used, to which 80 metal brackets (Abzil/3M) were bonded and divided into 4 Groups (n=20) according to the acid etching procedure. The blocks were etched with 5% hydrofluoric acid for 30 and 60 seconds (AF5/30 and AF5/60, respectively) and 10% hydrofluoric acid for 30 and 60 seconds (AF10/30, AF10/60, respectively). The resin composite used was Transbond XT (3M) and the presence of a glazer was maintained on the ceramic surface. The specimens were placed on a Universal test machine Instron 4411 (Instron Corp, USA) to which a chisel was adapted to perform the shear test at a speed of

1mm/min. The data were submitted to the analysis of variance (ANOVA) and the Adhesive Remnant Index was evaluated. **Result:** In the time interval of 30 seconds, there was no significant difference for the 5% and 10% hydrofluoric acid concentrations. In the 60-second time interval, the 10% concentration showed significantly higher shear bond strength values ($p<0.05$). The ARI showed predominance of scores 1 and 2. **Conclusion:** It was concluded that 10% hydrofluoric acid showed higher shear bond strength values in 60 seconds of etching, while 5% hydrofluoric acid showed no significant difference between the etching times.

Descriptors: Acid etching; ceramic; shear bond strength; orthodontic bracket.

INTRODUCTION

Hydrofluoric acid is cited as being a ceramic surface conditioner because of its capacity to promote the morphological changes necessary for bonding[1-5]. The growing need for esthetic rehabilitation has raised the demand for orthodontic treatment and increasingly improved alignment of teeth. Therefore, orthodontists need to bond orthodontic brackets to various types of restorations, including those fabricated of porcelain[5].

The literature has suggested surface treatment options for the purpose of promoting enamel surface roughness before bracket bonding[1,6]. Roughness may be achieved by mechanical means using diamond tips or airborne particle abrasion with aluminum oxide particles[1,6]. However, the literature has demonstrated crack propagation within the ceramic, and consequent weakening of the part, making it unfeasible to use this form of roughening[7]. Furthermore, treatment with hydrofluoric acid, saline application or even a combination of these treatments has also been indicated[5,7,8].

Hydrofluoric acid has been shown to be an effective option for preparing surfaces that will receive metal orthodontic accessories[1-5], because it promotes dissolution of the vitreous portion of ceramic. This leads to change in its morphology by creating irregularities that will help the wetting proposed by saline, and consequently, a more effective bond[4,5,9], intensifying the bond strength[10]. Criticism of the use of hydrofluoric acid arises from its caustic power, capable of promoting soft tissue lesions, in addition to the possibility of weakening the ceramic[6,9]. Commercially, it is found in various concentrations, therefore it is necessary to conduct studies to establish an adequate application protocol, so that the intended retention will be obtained without weakening the ceramic part[11-13].

The concentration and etching time with hydrofluoric acid have an influence on the bond strength, because the changes provided on the ceramic surface may act directly on the bond to the orthodontic accessory[9,10,14]. The size and number of irregularities crated at the time of etching are associated with the acid formulation and dilution, as well as the etching time[10]. The degree of dissolution of the vitreous matrix is proportional to the time of exposure to the acid and promotes an increase in the number of irregularities on the ceramic surface and area of contact with the resin cement[11]. This leads to the belief that an increase in etching time would lead to an increase in bond strength[1,10].

Starting with the hypothesis that the hydrofluoric acid concentrating and its etching time would influence the end result of the adhesive process, the aim of this study was to evaluate the shear bond strength (SBS) of metal brackets bonded to feldspathic ceramic surfaces under the action of hydrofluoric acid (5% and 10%), in two time intervals of application (30 and 60 seconds).

MATERIAL AND METHOD

To conduct the present in vitro study, four specimens constructed in the molds for fabricating metal-ceramic prosthetic parts were used, which were produced in a prosthesis laboratory. Each specimen was made with the use of four No.9 wax plates (Asfer) measuring 35 mm high, 16 mm wide and 3 mm thick. In each plate, a central vertical channel was sculpted, with the following measurements: 35mm high, 6mm wide and 1.5mm deep.

The united plates formed a rectangle made of wax, which was included in a silicone ring and filled by coating (Belavest/BEGO). After this, the set was taken to an electric furnace (Bravac) at a temperature from 900 °C to 930 °C, for the purpose of obtaining evaporation of the wax and gases contained in the coating, through a canal known as a sprue. This canal also served as a feed line for inserting the cast metal. Nickel-chrome casting, which initially occurred at approximately 1300 °C, was performed in a crucible specifically for this purpose, using a blow torch and butane gas. After this silicone was inserted into the ring through a centrifuge. From this casting process, four metal blocks were originated, which were cooled naturally to ambient temperature.

Each nickel-chrome metal alloy block (Fit Cast-V/Talmax) obtained, was machined with aluminum oxide stone (Talmax) to regularize the surface, with the aim of avoiding contamination of the alloy. Airborne particle abrasion with 120-micron aluminum oxide particles (Bio-Art) was performed under 2 bar pressure at a distance of 10 mm in a chamber specifically for conditioning of the metal. After conditioning, feldspathic ceramic (VITA) was applied in a vertical and central channel situated on each side of the metal block, simulating a metal-ceramic crown. Ceramic application began with the application of two opaque layers for sealing and obliterating the metal. After this ceramic in the form of paste, obtained by adding distilled water to the ceramic powder, was applied with the use of a laboratory paint brush. After this a sequence of two to three firings were performed in a furnace specifically for ceramics (Atlantis Pro/Kota) at a temperature of approximately 920 °C for sintering, as specified by the manufacturer.

The specimens obtained were initially cleaned with pumice stone and rubber cup, and afterwards cleaned with water, dried with compressed air for 30 seconds, and then etched with hydrofluoric acid (HA) (FGM, DENTSCARE LTDA, Joinville/SC, Brazil). On each of the 4 test specimens, a bonding protocol was performed, by varying the concentration and application time of HA, classified as follows: AF5/30 (5% hydrofluoric acid for 30 seconds), AF5/60 (5% hydrofluoric acid for 60 seconds), AF10/30 (10% hydrofluoric acid for 30 seconds) and AF10/60 (10% hydrofluoric acid for 60 seconds). After application of hydrofluoric acid, the specimens were cleaned with water and dried for 30 seconds.

A coat of silane Rely X Ceramic Primer (3M ESPE, St. Paul, MN, USA) was applied on the ceramic portion of all the specimens. After an interval of 60 seconds a coat of the adhesive system Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA) was applied and light activated for 20 seconds. In total 80 maxillary central incisor brackets of the Abzil brand (3M) (lot 1732200533) were used. These were distributed among the four specimens, so that 20 brackets were placed on each specimen (n=20); and 5 brackets were disposed on each side of the rectangle with a space of 3mm between them (Figure 1). The resin composite used was Transbond XT (3M-Unitek) and light polymerization was performed with the Optilight Max 440 (Gnatus) appliance, (Wavelength 420 to 480nm, Irradiance of 1200 mW/cm^2) for 40 seconds.

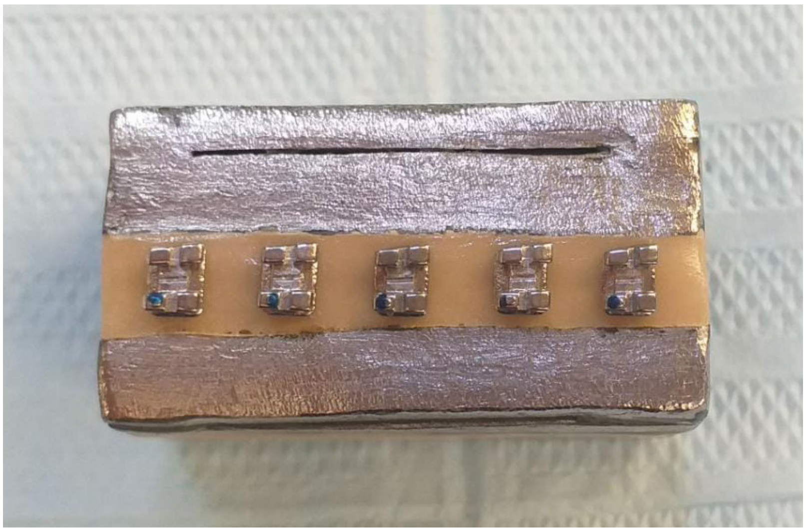

Figure 1. Specimen obtained for positioning the supports for the tensile test.

The prepared specimens were stored in deionized water at 37C° for 24 hours[8,15]. The shear bond strength test was performed with a Universal Test machine INSTRON 4411 (Instron Corp, USA) with a chisel adapted for stabilization, and the speed used was 1mm/min. (Figure 2).

Data were submitted to two-way Analysis of Variance and the Tukey Test, at 5% level of significance.

Figure 2. Specimens, during the test.

The adhesive remnant Index (ARI) was evaluated with the aid of a magnifying glass with a 25x magnification lens[4,15], in accordance with the method proposed by Artun, Bergland[16] and was determined by scores that ranged from 0 to 3:

0 - Absence of any adhesive layer residue on the enamel;

1 - Presence of less than half of the resin residue on the enamel;

2 - Presence of more than half of the resin residue on the enamel;

3 - Presence of all the resin residue on the enamel, together with the impression of the design on the base of the support[5,6,8,17,18].

RESULT

According to the data submitted to analysis of variance (ANOVA), the 5% hydrofluoric acid showed no significant difference between the etching times of 30 and 60 seconds ($p>0.05$). While for the 10% concentration, the etching time of 60 seconds generated significantly higher bond strength values in the time of 30 seconds. There was no significant difference between the concentrations of 5% and 10% hydrofluoric acid used for 30 seconds, differing from the application for 60 seconds, which demonstrated significantly higher SBS values when 10% hydrofluoric acid was used (Table 1).

Table 1. Mean Shear Bond Strength (MPa) of metal brackets bonded to ceramic surfaces considering the concentration and etching time

Concentration	Etching time	
	30 seconds	60 seconds
5%	6.84 (2.4) Ba	5.78 (2.0) Bb
10%	7.28 (2.2) Ba	8.89 (1.6) Aa
p(conc)=0.0002; p(time)=0.4956; p(interaction)=0.004		

Means followed by different letters, (capitals in the horizontal and lower case in the vertical) differ between them ($p\leq0.05$). Area of bracket=12mm^2.

The ARI evaluation demonstrated predominance of score 1 in all the groups, in which all the resin remained adhered to the ceramic surface, followed by Score 2, present in all the Groups, except AF5/30, as may be observed in Table 2. No evidence of fracture was shown on the ceramic surface of the Groups studied.

Table 2. Adhesive Remnant Index on ceramic surface etched with hydrofluoric acid

	AF 5/30	AF5/60	AF10/30	AF10/60
0	0%	0%	0%	0%
1	100%	75%	75%	50%
2	0%	25%	25%	50%
3	0%	0%	0%	0%

DISCUSSION

Porcelain is an inert material that needs differentiated surface preparation to fulfill the proposed prosthetic and orthodontic requirements. For this purpose, the ideal bond strength, either for fracture repair or for bonding orthodontic accessories must be investigated. According to Pannes et al.[19], based on a review of the literature, they verified that the ideal bond strength of clinically successful bonds between orthodontic accessories and ceramic surfaces would be between 6 and 8 MPa. In the present study, specimens etched with hydrofluoric acid in a concentration of 5% and 10%, and in two etching time intervals, 30 and 60 seconds, showed SBS values between 5.78 and 8.98 MPa, demonstrating that the concentration and etching time influenced the SBS between the orthodontic accessory and ceramic surface, therefore, the hypothesis tested in our study was accepted.

Hydrofluoric acid has been cited as a surface conditioner capable of promoting roughness on the ceramic surface resulting from dissolution of the vitreous portion by exposure of the silicon dioxide -SiO2[9], creating the interlocking necessary for the stages of bonding. The increase in etching time to 60 seconds demonstrated that the SBS of the group that received 10% hydrofluoric acid was above the clinical ideal value proposed by Pannes et al.[19].

Although various studies have agreed that the increase in etching time of hydrofluoric acid was responsible for the increase in SBS[1,3,6,11,18], in the present study, the results with reference to 5% hydrofluoric acid demonstrated that in spite of the increase in etching time made, a reduction in SBS could be observed after 60 seconds when compared with the SBS after 30 seconds. However, when the concentration of the hydrofluoric acid was considered sufficient for dissolution of the vitreous matrix of the ceramic and favorable to the mechanical interlocking by the irregularities promoted, an increase in SBS could be observed. Therefore, the increase in etching time is proportional to the increase in SBS, which is in agreement with our findings, as it was possible to observe that Group AF10/60 showed higher SBS values than those of Group AF10/30.

Seeing that there are many concentration of hydrofluoric acid available on the market and that the literature is not conclusive with regard to its protocol of use[13], various studies with the aim of investigating the best way to prepare the ceramic surface for the purpose of repairing structural damage or for bonding orthodontic accessories have reported positive results with 10% hydrofluoric acid used for an etching time of 60 seconds[3,9,10,18]. A previous systematic review verified that the best protocol to use referred to the application of 9.6% hydrofluoric acid for 60 seconds[20].

ARI evaluation demonstrated the presence and predominance of Score 1 in all the studied groups, while Score 2 was present in all the groups with the exception of Group AF5/30. According to Ramos et al.[21], the ideal would be for debonding to result in adhesive failure, which would show evidence of an intact original glassy ceramic surface, which would be in agreement with the findings of the present study, in which even with the predominance of Scores 1 and 2, it was not possible to observe any damage on the ceramic in any of the groups. For Stella et al.[22], Score 3 of the ARI, in which all the adhesive remainder would be bonded to the ceramic surface after debonding the accessory, would be the safest option for preservation of the ceramic surface, in spite of the need for using rotary instruments to remove the residual resin, which must be performed with care.

In this context, the results of the present study suggested that 5% hydrofluoric acid with an etching time of 30 seconds would be feasible for bonding accessories and maintaining the integrity of the ceramic surface. Moreover, the SBS values, irrespective of etching time used were without the standard of clinical success proposed by the literature[22], and in agreement with another study that considered reduction in the concentration an advantageous means of avoiding weakening of the ceramic and caustic dangers pertinent to HA[9].

Orthodontists must recognize the importance of resorting to clinical ceramic surface conditioning protocols that offer safe results free of risks pertinent to bonding procedures, thereby avoiding undesirable effects during the orthodontic treatment of previously rehabilitated patients. After conducting this study, the authors noted the need for investigating intermediate hydrofluoric acid concentrations (between 5 and 10%) and associate them with new *in vivo* studies.

CONCLUSION

It was concluded that 10% hydrofluoric acid applied for 60 seconds was the protocol that produced the highest SBS values between orthodontic brackets and the ceramic surface. While 5% hydrofluoric acid showed a clinically acceptable SBS irrespective of the etching time interval applied.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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