



# Effect of Hydrofluoric Acid Concentration and Thermal Cycling on the Bond Strength of Brackets to Ceramic

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This study to evaluate the effects of different hydrofluoric acid (HF) concentrations and thermal cycling on the shear bond strength (SBS) of brackets to ceramic. Cylinders of ceramic were divided into 10 groups (n=15), according to HF concentrations: 1-1%;2-2.5%;3-5%;4-7.5%;5-10% (storage 24 h); 6-1%;7-2.5%;8-5%;9-7.5%; and, 10-10% (thermal cycling). All cylinders were etched for 60s and received one layer of silane. Metallic brackets were bonded to the cylinders using Transbond-XT, light activated for 40 s, using a LED (Radii Plus) and stored in deionized water at 37° C for 24h. The groups 6 to 10 were submitted to thermal cycling (7,000 cycles - 5°/55°C). SBS was performed in an Instron at crosshead speed of 1.0 mm/min. Data were submitted to two-way ANOVA and Tukey's post-hoc test ( $\alpha=0.05$ ). The Adhesive Remaining Index (ARI) was evaluated at 40x magnification. The different HF acid concentrations influenced on the SBS of the brackets to ceramic ( $p<0.05$ ). The thermal cycling decreased the SBS of the brackets to ceramic for all acid concentrations ( $p<0.05$ ). The ARI showed a predominance of scores 0 for all groups, with an increase in scores 1, 2 and 3 for the group storage for 24 h. In conclusion, the different HF acid concentrations 5.0%, 7.5% and 10% influenced on the SBS of brackets to ceramic. The thermal cycling decreased significantly the SBS of brackets to ceramic.

Key Words: ceramic, dental materials, hydrofluoric acid, thermal cycling, orthodontic

## Introduction

Glass ceramic material has been used in restorative dentistry and may serve as substrates for bonding orthodontic brackets. The ideal bonding to ceramic surface is obtained with hydrofluoric acid (HF) etching and silane application (1). The 60 s etching time for bonding to feldspathic ceramic surface is commonly recommended (2).

The HF acid has the ability to conditioning and dissolving the glass ceramic, cause physical alteration of the surface creating a rough surface, and increased contact surface area in the ceramic bonding area (3-6). Strong bonds are formed between resin-based materials and dental ceramics etched by HF acid (3,4). However, the etching efficiency depends on the concentration of the HF acid and etching time (3,4,7). Besides, HF as highly hazardous chemical due to its toxicity, corrosiveness and reactivity (8), and can be harmful for patient and is capable of causing severe trauma to soft tissues and dental personal (6). The effect can continue for several days, causing increased tissue damage at a later stage. The severity and damage are directly related to the acid concentration, exposure time, and penetration to the exposed tissue (6). Although HF is not applied on soft tissue, less concentrated HF would cause less injury in accidental contact situations (4). However,

little is known about the effect of increased or decreased HF concentrations on the bonding ability of the brackets to ceramic. HF acid application is followed by silane coupling agent on the ceramic surface before to applying bonding material and may also increase the bonding strength between ceramic and orthodontic brackets.

Clinically, when the orthodontic brackets bonded on the ceramic are exposed to the oral environment failure can occur due to exposed to thermal, physical and chemical changes in the oral cavity during the orthodontic use (1). Thermal cycling is an alternative method to induce stress and regimens between 500 and 7,000 cycles have been used to understand the mechanisms that can cause deterioration and reduction in strength in the oral conditions before the mechanical tests (1,9-11). The difference in the thermal expansion coefficients between different bonding materials can increase the stress and cause adhesion failure due to temperature variations (12).

Therefore, the purpose of this study was to evaluate the influence of different HF acid concentrations and thermal cycling on the shear bond strength (SBS) of metallic brackets to ceramic. The hypotheses tested were: 1) The HF acid concentrations would not affect the SBS of the brackets to ceramic; and 2) The thermal cycling would not affect the SBS.

## Material and Methods

### Preparation of the Specimens

Cylinders of feldspathic ceramic (Certe Advanced Ceramics, Barueri, SP, Brazil) with 10 mm in height x 13 mm in diameter were used in this study. The surfaces were cleaned with a rubber cup (KG Sorensen, Cotia, SP, Brazil) and pumice-water slurry (S.S. White, Petropolis, RJ, Brazil) for 20 s, rinsed with air-water spray for 20 s and dried with air for 20 s. A rubber cup was used for each cylinder.

Ceramic cylinders were divided into 10 groups (n=15), according to the HF acid concentrations: Groups: 1) 1%;2) 2.5%;3) 5%;4) 7.5%;5) 10% (storage for 24 h); 6) 1%;7) 2.5%;8) 5%; 9) 7.5%; and, 10) 10% (thermal cycling). All cylinders surface were etched with each HF acid concentrations (Formula & Action, Sao Paulo, SP, Brazil) for 60 s at room temperature, and rinsed with oil-free compressed air-water spray for 60 s. All specimens were ultrasonically cleaned (MaxiClean 750) in deionized water for 15 min and dried for 60 s.

One layer of a silane coupling agent (RelyX Ceramic Primer; 3M ESPE, St. Paul, MN, USA) was applied to the etched cylinders surface and left in contact for 60 s, followed by compressed air for 60 s to accelerate the water/alcohol evaporation. After, the surface of all specimens received one coat of Transbond XT light cure adhesive primer (3M Unitek, Monrovia, CA, USA) and light-cured for 10 s, using a LED source (Radii Plus; SDI Limited, Bayswater, Victoria, Australia) with an irradiance of 1,100 mW/cm<sup>2</sup> as measured using a curing radiometer (Model 100, Demetron Research Corporation, Danbury, CT). Standard maxillary premolar metallic brackets (Abzil, 3M, Sao Jose do Rio Preto, SP, Brazil) were positioned and bonded to the ceramic cylinders surface with light cure adhesive paste (Transbond XT; 3M Unitek), according to manufacturer's instructions. The excess of adhesive paste was removed using a microbrush before light-curing for 10 s on each side of the bracket (four activations) with the light guide tip positioned at the interface ceramic / brackets using a LED source (Radii Plus; SDI). A punch-holed strip of black adhesive tape was used to restricting the polymerization light to the specimen that is being bonded to avoiding light exposure to adjacent bracket (2).

### Storage and Bonding Testing

All the specimens were stored in deionized water at 37 °C for 24 h and then Groups 1 to 5 were tested. The specimens in groups 6 to 10 were then submitted to 7,000 thermal cycles (10) in a thermal cyler (MSCT 3, Marnucci ME, Sao Carlos, SP, Brazil) with water between 5 °C and 55 °C (dwell time of 30 s) and transfer time of 6 s between baths. After storage for 24 h and thermal cycling, the SBS test was performed in a universal testing machine (Model

4411; Instron, Canton, MA, USA) using a mounting jig to align the ceramic-bracket interface and a knife-edged rod at 1.0 mm/min until failure.

The experimental unit was the ceramic cylinder, with each group containing fifteen cylinders. Two brackets were bonded to each cylinder ceramic, totalizing thirty brackets for each group. The mean of the SBS values in each group represented the mean of the fifteen experimental units. The bond strength mean values were calculated and the data supplied in MPa. Data were tested for normality (Shapiro-Wilk) and equal variances (Levene) prior to being analyzed with two-way ANOVA (acid concentration x treatment). Multiple comparisons were performed using the Tukey post-hoc test ( $\alpha=0.05$ ).

The bracket and ceramic interfaces were observed by optical microscopy at 40x magnification (Olympus Corp, Tokyo, Japan) and the failure mode was classified according to ARI (13), as follows: score 0: indicated that no resin remained on the ceramic; score 1: indicated that less than half the resin remained on the ceramic; score 2: indicated that more than half the resin remained on the ceramic; and score 3: indicated that all resin remained on the ceramic, with a clear impression of the bracket mesh.

## Results

SBS mean values are shown in Table 1. Significant differences in SBS for acid concentrations ( $p<0.0001$ ) and treatment ( $p<0.0001$ ) were detected. The interaction between acid concentrations and thermal cycling ( $p=0.081$ ) was not significant. Thus, multiple comparisons were made with separate Tukey's tests within each acid concentrations and within each treatment. After 24 h storage and thermal cycling, the SBS of 10% HF acid was significantly higher than 5% HF, 2.5% HF and 1% HF ( $p<0.05$ ). The HF acid 7.5% and 5% presented significantly higher SBS than 2.5% HF and 1% HF ( $p<0.05$ ). No statistical difference was found

Table 1. Mean of shear bond strength (SBS)  $\pm$  Standard Deviation (MPa) for the HF acid concentrations 10%, 7.5%, 5%, 2.5% and 1% after storage for 24 h and thermal cycling

HF Acid concentrations (%)	SBS (MPa)	
	24 h	Thermal Cycling
10	10.7 $\pm$ 0.7 Aa	9.0 $\pm$ 0.4 Ba
7.5	10.2 $\pm$ 0.8 Aab	8.7 $\pm$ 0.6 Bab
5	10.1 $\pm$ 0.6 Ab	8.3 $\pm$ 0.4 Bb
2.5	5.8 $\pm$ 0.4 Ac	4.8 $\pm$ 0.4 Bc
1	5.6 $\pm$ 0.6 Ac	4.5 $\pm$ 0.9 Bc

Values followed by the same upper-case in the same row and lower-case within the same column are statistically similar ( $\alpha=5\%$ ).

between HF acid 10% HF and 7.5% HF, and 7.5 HF and 5% HF, and 2.5% HF and 1% HF ( $p>0.05$ ). The mean values of SBS obtained at 24 h for all HF acid concentrations were significantly higher than following thermal cycling ( $p<0.05$ ).

Failure mode results (ARI) are shown in Figure 1. A predominance of failures score 0 was observed in all groups with increased for scores 1, 2 and 3 for the specimens 24 h.

## Discussion

HF acid etching modifies the morphology of the ceramic by the dissolution of the glassy matrix, resulting in microporosities on the ceramic surface (3,4,14-16), improve bond strength (3,4,16,17), and better contact between bonding material and ceramic (18,19). However, HF acid is a toxicity, corrosiveness and reactivity inorganic acid (8) and can lead to several health complications for patient and dental personal (6,20). The severity depends on the acid concentration and exposure time, and penetration to the exposed tissue (6). A decrease of HF acid concentrations would be advantageous and can cause less risk to the patient and dentist.

In the current study, different HF acid concentrations applied on ceramic surface promoted different results, indicating that the first hypothesis was rejected. The data showed that lower values for SBS were obtained for 1% HF and 2.5% HF acid concentrations with statistically significant difference in relation to 5% HF, 7.5% HF and 10% HF. The 10% HF acid showed the highest SBS values. These results are in line with those of previous studies, which also found different SBS values for concentrations of 10% HF in relation to 1% HF and 2% HF (3,4;14,21). Probably, lower HF acid concentrations were not enough to dissolve the vitreous phase promoting lesser dissolution than higher

concentration (3,4,14). Others study showed that fewer microporosities were found with low HF concentration with reduced contact area between ceramic and bonding materials, resulting in little mechanical interlocking and lower bond strength (22). Another study, showed that 1% and 2.5% HF concentrations showed reduced effect on vitreous phase, because of the lower content of ionized HF available to react with vitreous phase (3). Although 5% HF acid concentration showed SBS intermediate values, it was effective to promote changes in the ceramic surface, improved the mechanical interlocking of the bonding material to ceramic surface. In this way, 5% HF acid concentration could be indicated for clinical use in orthodontic. On the other hand, previous study showed that hydrofluoric acid can be aggressive and harmful to soft tissues, however the symptoms like pain may not be immediately after exposure, but after several h and the lesion severity is directly related to the acid concentration (6). Although HF acid is not applied on soft tissues, in clinical situations by accidental contact the lower acid concentration would cause less injury (4).

Some factors as thermal cycling, silane application, fatigue, and artificial ageing may compromise the durability of the bond strength between orthodontic brackets and ceramic surface using bonding materials (2,10,11,23). The quality of the bond is determined by the specific treatment bonding used to improve bonding mechanisms between bracket-bonding materials (1).

In this study, thermal cycling has been used to determine if changes of temperature might reduce the bond strength between bracket and bonding material. The reduction of mechanical properties of the bonding resin occurs due to a continuous action of water and temperature changes at the bonded interface, causing different expansion and thermal conductivities that promote stresses on the orthodontic bracket and bonding resin (12). Other study showed that the thermal cycling might promote hydrolytic degradation of the components or differences in the thermal expansions among brackets, bonding material and ceramic (24). The specimens submitted to thermal cycling showed lower SBS values between ceramic and bonding material in relation to the 24 h. Thus, the second hypothesis was rejected. These findings are in agreement with the previous studies, which found significant differences in bond strength after thermal cycling (1,10,21). However, some studies have found no significant difference for bond strength after thermal cycling. These studies submitted the specimens to a small number of cycles while in this current study a larger number of cycles was used (9,11). Another study showed that is necessary a larger number of cycles to accelerate the degradation occurred in the simulated test (9).

High bond strengths values are required to avoid bracket

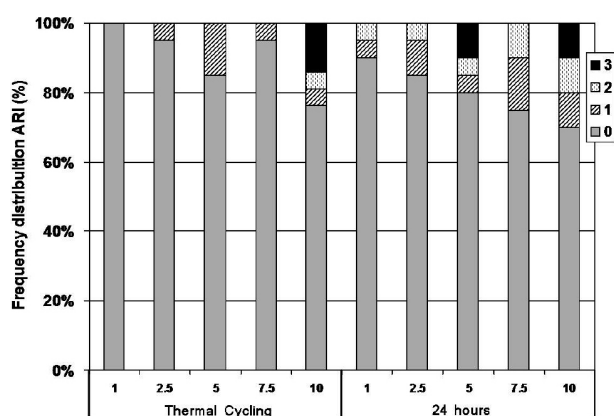


Figure 1. Failure Mode Analysis (ARI) for all groups (%). Score 0: indicated that no resin remained on the ceramic; score 1: indicated that less than half the resin remained on the ceramic; score 2: indicated that more than half the resin remained on the ceramic; and score 3: indicated that all resin remained on the ceramic, with a clear impression of the bracket mesh.

failure during orthodontic treatment. Bond strength values in the range of 6 to 8 MPa are clinically acceptable for orthodontic forces in oral environment (25). In this study, brackets bonding to ceramic with strength values lower than 6.0 MPa were obtained for 1% and 2.5% HF acid concentrations. Therefore, care should be taken when 1% and 2.5% HF acid concentrations were used because these concentrations have not acceptable potential to resist clinical forces during orthodontic treatment. ARI values indicated predominance of failures with score 0; however, an increase in scores 1, 2 and 3 for groups 24 h was observed.

In summary, the results showed that HF acid concentrations and thermal cycling have significant effects on the SBS values. The different HF acid concentrations 5.0%, 7.5% and 10% influenced on the SBS of brackets to ceramic. The thermal cycling decreased the SBS of brackets to ceramic. Adequate HF acid concentration is crucial for obtaining optimal mechanical properties and clinical performance. Care should be taken by clinicians during the etching procedure because HF is toxic and capable of causing severe trauma to soft tissues (4,6). Therefore, the etching procedure must be done with personal protective equipment in well-ventilated rooms to avoid any further damage to the professionals (4). Future studies should be developed to investigate other possible factors such as etching times, bonding material and fatigue that may affect the clinical performance of the bonding brackets to ceramic restorations.

## Resumo

Este estudo avaliou os efeitos de diferentes concentrações de ácido hidrofluorídrico (HF) e ciclagem térmica na resistência de união ao cisalhamento (RUC) de bráquetes metálicos a cerâmica. Cilindros da cerâmica feldspática foram divididos em 10 grupos (n=15). De acordo com as concentrações do ácido HF: 1-1%;2-2,5%;3-5%;4-7,5%; e, 5-10% (armazenagem por 24 h); 6-1%;7-2,5%;8-5%;9-7,5%; e, 10-10% (ciclagem térmica). Todos os cilindros foram condicionados por 60s e receberam uma camada de silano. Bráquetes metálicos foram colados aos cilindros usando o transbond-XT, fotoativado por 40 s, usando o LED (Radii Plus; SDI) e armazenados em água deionizada a 37° C por 24 h. Os Grupos 6 a 10 foram submetidos à ciclagem térmica (7.000 ciclos – 5°/55°C). RUC foram realizadas na Instron a velocidade de 1.0 mm/min. Os dados foram submetidos à análise de variância dois-fatores e ao teste de Tukey's post-hoc ( $\alpha=0,05$ ). O Índice de Remanescente do Adesivo (IRA) foi avaliado com 40x de aumento. As diferentes concentrações do ácido HF influenciaram na RUC dos bráquetes na cerâmica ( $p<0,05$ ). A ciclagem térmica diminuiu a RUC dos bráquetes na cerâmica para todas as concentrações do ácido ( $p<0,05$ ). O IRA mostrou predominância de escores 0 para todos os grupos, com aumento de escores 1, 2 e 3 para os grupos armazenados por 24 h. Concluindo, as concentrações do ácido HF 5,0%, 7,5% e 10% influenciaram na RUC de bráquetes à cerâmica. A ciclagem térmica diminuiu significativamente a RUC dos bráquetes à cerâmica.

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