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Effect of Acid Etching, Silane and Thermal Cycling on the Bond Strength of Metallic Brackets to Ceramic

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The aim of this study was to evaluate the effect of silanes, thermal cycling and acid etching on the shear bond strength (SBS) of metallic brackets to feldspathic ceramic. Feldspathic ceramic cylinders (Groups 1, 2, 5 and 6) were etched for 60 s with 10% hydrofluoric acid and Groups 3, 4, 7 and 8, without acid etching. Two layers of silane Clearfil Ceramic Primer (CCP, Groups 1 to 4) and two layers of RelyX Ceramic Primer (RCP, groups 5 to 8) were applied and dried for 60 s. Brackets were bonded to the cylinders with Transbond XT and light-activated for 40 s with Bluephase G2. All specimens were stored in deionized water at 37 °C for 24 h, and the specimens of groups 1, 3, 5 and 7 were submitted to 7,000 thermal cycles (5 °C/55 °C). After storage, the SBS test was performed at a crosshead speed of 1 mm/min. Data were subjected to three-way ANOVA and Tukey's post hoc test (α=0.05). The adhesive remnant index (ARI) was evaluated at 8x magnification. The SBS of CCP was significantly greater than of RCP ($p<0.05$), with or without thermal cycling. Thermal cycling significantly reduced the SBS ($p<0.05$). The groups submitted to acid etching showed significantly higher SBS than those without acid etching ($p<0.05$). In conclusion, thermal cycling reduced SBS for all groups. The best ceramic surface treatment for bracket bonding was achieved by acid etching and CCP silane. The ARI results showed predominance of score 0 for all groups.

Key Words: ceramic, silane, etching acid, thermal cycling, shear bond strength

Introduction

In the last years, the number of young and old patients with ceramic restorations looking for orthodontic treatment has been increasing. In addition to dental enamel, ceramic restorations may serve as substrates for bonding of orthodontic brackets in clinical conditions. Bonding orthodontic brackets to ceramic surfaces may exhibit a higher degree of failure in relation to bonding to enamel. However, no negative effects occurred on the surface of ceramics, as most failures were adhesive between the bonding material and the ceramic surface with no damage to the ceramic (1-3).

The hydrofluoric acid (HF) has been used in orthodontics for etching ceramic surfaces where the orthodontic bracket is bonded, for 60 s (1). The HF acid promotes dissolution of the glass ceramic providing a rough surface, increased contact surface area to the ceramic, producing a stronger bond between dental ceramics and composite resin (4,5). The bond strength between brackets and ceramic surface may also be increased by the use of silanes, which are able to form chemical bonds with both inorganic and organic surfaces (6), and improve durability and bonding strength (1,6,7). However, HF is highly toxic, reactive and corrosive (8) and it may be harmful for the dental personnel and patients (5). Thus, it has been alleged that a specific primer adhesive named Clearfil Ceramic Primer (CCP, Kuraray America) for dental ceramics could be used without the need of HF acid etching. The CCP is a single-component adhesive primer used to enhance the bond strength between resin-based materials and some types of restorative ceramics and composites. The CCP contains the MDP phosphate monomer, which bonds very strongly to metal oxides, and MPS silane coupling agent that promotes strong adhesion to esthetic restoration materials (9,10), without association with HF acid.

Conversely, when orthodontic brackets are bonded to ceramic surface and exposed to the oral environment, failure may occur in the interface among ceramic, bonding material and orthodontic brackets due to thermal changes and heavy forces produced by the archwire in the oral cavity during the orthodontic procedure (2). Thermal cycling regimens between 500 and 7,000 cycles have been used to verify if temperature variations promoted stresses in the light-cured materials deteriorated in the simulated oral conditions before the mechanical test (2,11,12).

However, the literature is still not conclusive about specific primer adhesive without combination of HF acid etching and thermal cycling. The aim of this study was to evaluate the effect of silanes, thermal cycling and acid etching on the bond strength of metallic brackets to feldspathic ceramic. The hypotheses tested were: 1) The acid etching would not affect the shear bond strength; 2) The silane application would not influence the shear bond strength; and 3) Thermal cycling would not affect the shear bond strength.

Material and Methods

The surface of eight feldspathic ceramic cylinders (Certec Advanced Ceramics, Barueri, SP, Brazil) 20 mm high x 15 mm diameter were cleaned using pumice water slurry (S.S. White, Petropolis, RJ, Brazil) for 20 s with rubber cup on low speed hand piece (Kavo do Brasil, Joinville, SC, Brazil), rinsed with air-water spray for 20 s and air dried for 20 s. The rubber cup was replaced after each cylinder cleaning. Cylinders (Groups 1, 2, 5 and 6, Table 1) were etched with 10% hydrofluoric acid gel for 60 s (Dentsply Caulk, Milford, DE, USA), rinsed with air-water spray for 30 s and air dried for 30 s. Two layers of Clearfil Ceramic Primer silane (CCP - Kuraray Medical Inc., Kurashiki, Okayama, Japan) (Groups 1 to 4, Table 1) and two layers of RelyX Ceramic Primer silane (RCP - 3M ESPE, St. Paul, MN, USA) (Groups 5 to 8, Table 1) were applied and air dried for 60 s.

Standard stainless steel maxillary premolar brackets (Abzil; 3M ESPE, São Jose do Rio Preto, SP, Brazil) were bonded and firmly adhered to the curved area of the ceramic cylinders using Transbond XT light-cured bonding resin (3M Unitek, Monrovia, CO, USA), following the manufacturer's instructions. Microbrushes removed excess of bonding resin .

Light-activation was carried out with 4 exposures (10 s), one on each side of the bracket for a total exposure time of 40 s using LED (Bluephase G2; Ivoclar Vivadent, Schaan, Liechtenstein), with a radiance of 1,100 mW/cm^2, measured by a power meter (Ophir Optronics Inc., Danvers, MA, USA), and a computer-controlled spectrometer (USB2000; Ocean Optics, Dunedin, FL, USA). The radiation exposure was 44 J/cm^2.

Table 1. Experimental groups according to acid etching, silane and thermal cycling

Group	Silane	Acid etching	Thermal cycling
1	Clearfil Ceramic Primer	Yes	Yes
2	Clearfil Ceramic Primer	Yes	No
3	Clearfil Ceramic Primer	No	Yes
4	Clearfil Ceramic Primer	No	No
5	Ceramic Primer	Yes	Yes
6	Ceramic Primer	Yes	No
7	Ceramic Primer	No	Yes
8	Ceramic Primer	No	No

Metallic brackets were bonded to each ceramic cylinder (n=20) for each acid etching, silane and termal cycling, totalizing 160 bonded brackets. A punch-holed strip of black plastic was used to avoid light exposure to adjacent brackets, restricting the polymerization light to the specimen being bonded (2,3). The specimens were stored in deionized water at 37 °C for 24 h. Next, the specimens of the groups 1, 3, 5 and 7 underwent 7,000 thermal cycles (2) in a thermal cycler (MSCT 3; Marnucci ME, São Carlos, SP, Brazil) with deionized water between 5 and 55 °C, dwell time of 30 s and transfer time of 10 s between baths.

After storage and thermal cycling, a mounting jig was used to align the ceramic bracket interface parallel to the testing device. The shear bond strength (SBS) test was made in a mechanical testing machine (Model 4411; Instron, Canton, MA, USA), the shear load applied by a knife-edged rod at a crosshead speed of 1.0 mm/min until failure. Exploratory data analyses were performed prior to applying analysis of variance (ANOVA). The preliminary results by the Kolmogorov-Smirnov test indicated the data were normally distributed ($p>0.05$). The values of shear bond strength data were calculated in MPa and subjected to three-way analysis of variance (silane, thermal cycling and acid etching), and multiple comparisons were performed using the Tukey's post hoc test ($\alpha=0.05$).

After debonding, the ceramic and bracket surfaces were observed under optical microscope (Olympus Corp, Tokyo, Japan) at 8× magnification. The adhesive remnant index (ARI), from a modified Årtun and Bergland's method (13) was used to classify the failure modes, as follows: Score 0: indicated that no bonding resin remained on the ceramic; Score 1: indicated that less than half the bonding resin remained on the ceramic; Score 2: indicated that more than half the bonding resin remained on the ceramic; and Score 3: indicated that all bonding resin remained on the ceramic, with a clear impression of the bracket mesh.

Results

The results of shear bond strength are in the Tables 2 and 3. Significant influences of silane ($p<0.0001$), thermal cycling ($p<0.0001$) and acid etching ($p<0.0001$) were detected. The interaction between silane and acid etching ($p=0.96436$), acid etching and thermal cycling ($p=0.51080$), and silane, acid etching and thermal cycling ($p=0.96661$) factors were not significant. Silane and thermal cycling interaction was significant ($p=0.00753$).

When silanes were compared (Table 2), the mean SBS of CCP was significantly greater than of RCP, with or without thermal cycling ($p<0.05$). The groups submitted to thermal cycling demonstrated lower SBS values relative to the groups without thermal cycling (p<0.05). The specimens submitted to acid etching showed significantly higher SBS than those without acid etching (p<0.05), regardless of silane or thermal cycling (Table 3). Figure 1 shows the distribution frequency of ARI. Score 0 was observed in all groups.

Discussion

The first hypothesis tested in this study was rejected. The current study showed that the acid etching influenced significantly the SBS, regardless of silane or thermal cycling. The ceramic surface etching by hydrofluoric acid was shown to be more effective in relation to other treatments. Previous studies have shown that the bond strength for the specimens etched with HF acid was higher than the samples submitted to other treatments, such as aluminum oxide blast, phosphoric acid etched or without etching (6,14,15). The specific surface treatments by HF acid are used to modify the morphology of the ceramic by the dissolution of the glassy matrix, providing micromechanical retention to the ceramic substrate (4,15,16). Modification of ceramic surface morphology may be performed to increase bond strength (4,15,16) and a better contact between bonding material and ceramics (17). A previous study showed that the HF acid in contact with the ceramic surface reacts preferentially with the silica phase, creating retentive microchannels (18). Conversely, the HF acid is an extremely corrosive, toxic and reactive inorganic acid (8). It may cause severe complications for patients and dental personnel (5). Another study showed that the severity depends on the exposure time and acid concentration, and penetration into the exposed tissue (19).

In this study, both types of silane were used to evaluate their influence on the shear bond strength between orthodontic brackets and bonding materials. The data showed that CCP was significantly greater than RCP, with or without thermal cycling. Thus, the second hypothesis was also rejected. Silane agents are usually monomeric species where silicon links to reactive organic radicals and hydrolysable ester groups. The reactive organic groups are chemically bonded to the resin molecules. Hydrolysable monovalent groups bond chemically to silicon in the glass matrix and lithium disilicate (6). Thus, a chemical bond forms between the silane coupling agent and silica layer on the ceramic surface or the bonding materials.

Conversely, as HF acid may be harmful for the dental personnel and patients (3), the CCP for dental ceramics may be used without HF acid etching. The CCP contains MDP phosphate monomer that bonds very strongly to metal oxides, it contains the MPS silane coupling agent that provides strong adhesion to esthetic restorative materials (9,10). Previous studies have shown that the mixture of silane with acidic monomers may accelerate the hydrolysis reaction of the alkoxy groups into silanol, increasing the reactivity of the silanes (20,21). Thus, probably due to this fact a significant difference was observed in relation to RCP in the current study.

Durability of the bond strength between orthodontic brackets and the ceramic surface using composite resin may be influenced by some factors like thermal cycling, mechanical fatigue, silane and mechanical properties

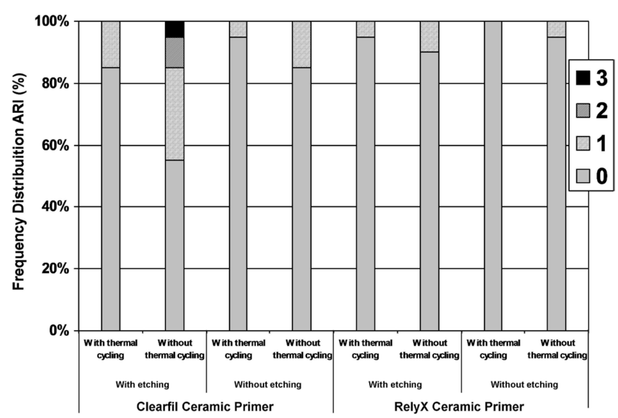

Figure 1. Frequency distributions (%) of the adhesive remnant index (ARI) scores for all groups. Score 0 indicates that no bonding resin remained on the ceramic; Score 1 indicates that less than half the bonding resin remained on the ceramic; Score 2 indicates that more than half of bonding resin remained on the ceramic; and Score 3 indicates that all bonding resin remained on the ceramic, with a clear impression of the bracket mesh.

Table 2. Mean shear strength values (S.D.) in MPa for silane, with and without thermal cycling, regardless of acid etching.

Silane	Thermal cycling	
	Without	With
Clearfil Ceramic Primer	12.8 ± 1.2 a, A	9.3 ± 0.8 a, B
RelyX Ceramic Primer	11.1 ± 1.1 b, A	5.9 ± 0.6 b, B

Means followed by different uppercase letters in the same row and lowercase letters in the same column indicate statistically significant difference (p<0.05).

Table 3. Mean shear strength values (S.D.) in MPa with or without acid etching, regardless of thermal cycling and silane.

Acid etching	Shear bond strength (MPa)
With	11.4 ± 0.9 a
Without	8.2 ±1.2 b

Means followed by different lowercase letters in the column indicate statistically significant difference (p<0.05).

of the composite resin (1,7,12). The quality of the bond defines the specific treatment to improve bonding between bracket-bonding resins (2) and to promote chemical and micromechanical retention to ceramic (22). Thermal cycling has been used to determine if changes of temperature may influence the reduction of the bond strength between bracket-bonding material substrate associations. Reduction of the mechanical properties of the bonding resin is probably the result of a continuous action of water on the interface of the orthodontic bracket-bonding resin substrate or by abrupt temperature fall in the bonded materials with different expansion coefficients, producing thermal stresses at the interface (23). In addition, a reduction in the bond strength may be due to hydrolytic degradation of the interface components (24).

The specimens submitted to thermal cycling showed lower SBS values between ceramic and bonding material in relation to the group without thermal cycling, regardless of acid etching. Thus, the third hypothesis was rejected. This finding is in agreement with another study showing significant differences in bond strength for specimens subjected to thermal cycling (2). Conversely, other studies have found no significant difference for bond strength after thermal cycling (7,12). However, in these studies the specimens were submitted to a small number of cycles, while in the current study a larger number of cycles was used, as advocated by previous studies (2,11). It has been alleged that a larger number of cycles is necessary to obtain accelerated simulation of the bond degradation (11).

Bond strength values in the range of 6 to 8 MPa are required for orthodontic procedures in the oral environment (25). In this study, bracket bonding to ceramic with strength values below 6.0 MPa were obtained for RCC submitted to thermal cycling. Therefore, care is especially required when RCC is used, because this silane has no acceptable potential to resist clinical forces during orthodontic treatment. ARI values indicated predominance of debonding failures with score 0, when no bonding resin on the ceramic surface was observed. This may be clinically advantageous, because there is less adhesive to remove from the ceramic surface after bracket debonding (1,2).

In summary, the results showed that CCP silane was more effective than RCP silane. Acid etching influenced significantly the SBS. However, the specimens without acid etching, regardless of silane or thermal cycling showed SBS values greater than 6 MPa (25). Therefore, the CCP silane may be used without previous acid etching of the ceramic. Further studies should be developed to investigate other possible factors affecting the clinical performance of the bonding brackets to ceramic restorations, like silane types and different bonding materials. In conclusion, thermal cycling reduced the SBS for all groups. The best ceramic surface treatment for bracket bonding was provided by acid etching and CCP silane. The ARI results showed prevailance of score 0 for all groups.

Resumo

O objetivo neste estudo foi avaliar o efeito de diferentes silanos, ciclagem térmica e condicionamento ácido na resistência da união ao cisalhamento (RUC) de bráquetes metálicos a cerâmica feldspática. Cilindros da cerâmica feldspática (Grupos 1, 2, 5 e 6) foram condicionados por 60 s com ácido hidrofluorídrico a 10% e os Grupos 3, 4, 7 e 8 sem condicionamento ácido. Duas camadas do silano Clearfil Ceramic Primer (CCP, Grupos 1 a 4) e duas camadas do RelyX Ceramic Primer (RCP, Grupos 5 a 8) foram aplicadas e secas por 60 s. Bráquetes foram fixados aos cilindros cerâmicos usando Transbond XT e fotoativado por 40 s com Bluephase G2. Todas as amostras foram armazenadas em água deionizada a 37 °C por 24 h, sendo as amostras dos grupos 1, 3, 5 e 7 submetidas a 7.000 ciclos térmicos (5 °C/55 °C). Após a armazenagem, a RUC foi verificada a velocidade de 1 mm/min. Os dados foram submetidos à ANOVA 3 fatores e teste de Tukey post hoc (α=0,05). O Índice de Remanescente do Adesivo (IRA) foi avaliado com aumento de 8x. A RUC do CCP foi significantemente maior do que o RCP (p<0,05), com ou sem ciclagem térmica. A ciclagem térmica reduziu significantemente a RUC (p<0,05). Os grupos submetidos ao condicionamento ácido mostraram valores de RUC significantemente maiores em relação os grupos sem condicionamento ácido (p<0,05). Concluindo, a ciclagem térmica reduziu a RUC para todos os grupos. O melhor tratamento de superfície da cerâmica para colagem de bráquetes foi obtido pelo condicionamento ácido e pelo silano CCP. O IRA mostrou predominância de escore 0 para todos os grupos.

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