

Active and Passive Application of the Phosphoric Acid on the Bond Strength of Lithium Disilicate

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The objective of this study was to evaluate the effect of passive or active phosphoric acid (PA) application after hydrofluoric acid (HA) treatment on the microshear bond strength of lithium disilicate. Thirty ceramic discs were made with IPS Emax 2 (10 mm thick and 10 mm diameter). The specimens were divided into 3 groups, A: 9.6% HA application; AF: 9.6% HA application + cleaning with 37% PA in passive mode and AFF: 9.6% HA application + cleaning with 37% PA in active mode. For the microshear test, four tygons (0.9 mm diameter and 0.2 mm high) were filled with resin cement (RelyX Ultimate) and placed on the ceramic disks. After testing, the fracture modes were examined under scanning electron microscopy. Data were analyzed by one-way ANOVA and Tukey's post test ($\alpha=0.05$). The bond strength values were significantly higher in Group AFF (11.0 ± 2.5 MPa) compared with group A (8.1 ± 2.6 MPa) ($p<0.002$). AF group was not statistically different (9.4 ± 2.5 MPa) from Group A. It was concluded that the active application of 37% PA after 9.6% HA increases the microshear bond strength values between the resin cement and lithium disilicate ceramic.

Key-Words: ceramics, hydrofluoric acid, phosphoric acid, shear strength.

Introduction

Great development of all ceramic systems for dental restoration occurred in the last 20 years, which provided the clinician a better aesthetic alternative for anterior and posterior restorations (1). This preference is directly related to the success of ceramic-resin bond, which contributes to the restoration's longevity (2).

Reinforced ceramics by lithium disilicate, feldspathics, leucite-reinforced feldspathics or fluorapatite are acid sensitive, in other words, they undergo morphological changes with acid treatment in different concentrations (3). This sensitivity changes the ceramic structure (2) and may decrease the shear bond strength to enamel and/or dentin and/or resin cement. In order to increase the bond strength values, ceramic inner surface treatments should be performed (4,5), including abrasion with a diamond bur instrument (6), microsanding (7), cleaning after hydrofluoric acid (HA) application. Cleaning the vitreous dissolution is important because it increases the contact with silane and increases bond strength. Moreover, it is known that HA is toxic (8), and to wash out this agent in order to remove the excess acid may be an alternative to reduce its toxicity.

Martins et al. (9) evaluated the effect of different cleaning media on the bond strength of resin cement to ceramic after etching with HA. Increased bond strength values were found when the ceramic specimens were

cleaned for 4 min by ultrasound in distilled water. However, this is an additional time-consuming step and also requires investment in an ultrasound machine. Although ultrasonic cleaning is advantageous, a high contact angle with this type of cleaning should be obtained (10).

The purpose of the current study was to evaluate the effect of passive or active cleaning with phosphoric acid (PA) application after HA application on the microshear bond strength between resin cement and lithium disilicate ceramic. The tested null hypothesis was that the different strategies of cleaning would not influence microshear bond strength values between the resin cement and lithium disilicate ceramic specimens.

Material and Methods

Specimen Preparation

Thirty discs were made with IPS Emax 2 ceramic (Ivoclar Vivadent, Liechtenstein, Swiss). Each ceramic disc was embedded in a polyvinyl chloride (PVC) tube fixed with acrylic resin.

Experimental Design

The specimens were randomly divided into 3 groups ($n=10$):

Group A (control): A 9.6% HA (EUFAR, Bogota, Colombia) was applied for 20 s and cleaned with tap water for 30 s. The ceramic surface was air-dried and a silane coat was

applied (Angelus, Londrina, PR, Brazil) with a microbrush (Angelus), keeping it in contact with the surface for 60 s and gently air dried.

Group AF: HA was applied for 20 s, cleaned with 37% PA (PA: SDI Super etch, Bayswater, Australia), for 30 s without rubbing (passive mode). A silane coat was applied with a microbrush (Angelus) keeping it in contact with the surface for 60 s and gently air dried.

Group AFF: HA was applied for 20 s and cleaned with PA for 30 s, agitated on the surface and the pressure was calibrated in an analytical balance (up to 250 g). A silane coat was applied with a microbrush (Angelus), keeping it in contact with the surface for 60 s and gently air dried (Table 1).

In all groups, after the ceramic surface treatments, the adhesive (Adper Single Bond 2; 3M ESPE, St. Paul, MN, USA) was applied with 2 coats, gently agitated and a slight air jet was applied to evaporate the solvent. Then, the adhesive was light cured for 10 s, according to the manufacturer's instructions.

Microshear Bond Strength Test

After treatment of the ceramic surfaces, tygons (Angiocath BD, Cundinamarca, Colombia) with 0.9 mm diameter and 2 mm high were positioned on the treated ceramic surfaces to be filled by the resin cement (RelyX Ultimate, 3M ESPE). The tygons (4 tygons per ceramic specimen) were positioned on the treated ceramic surface with a minimum distance of 2 mm from each other, according to each group (Fig. 1), and light-cured for 40 s at light intensity of 1,200 mw/cm² (Radii Plus; SDI), according to the manufacturer's instructions. The tygons were then carefully removed with a sharp blade.

The PVC tubes were adapted to a universal testing

machine (Kratos 500, São Paulo, SP, Brazil). A blade was positioned as close as possible to the resin/enamel interface. A shear load was applied to each tygon, at a crosshead speed of 0.5 mm/min, until specimen fractured. The values were expressed in MPa. After fracture, the ceramic surfaces were evaluated under a stereoscopic zoom microscope (SMZ800, Nikon Corporation, Tokyo, Japan) at 40× magnification to classify the failure mode as adhesive (at the resin cement/ceramic interface, including pretesting failure) or mixed (with both adhesive and cohesive failures).

Scanning Electron Microscopy (SEM) Analysis

Two discs from each group were prepared for scanning electron microscopy (SEM) analysis. Samples were dehydrated for 48 h in a desiccator (Dry Keeper Simulate Corp., Tokyo, Japan) and sputter coated with a 10 nm platinum layer (Polaron Equipment Ltd., Hertfordshire, England, UK). A scanning electronic microscope (SEM - Zeiss EVO MA 25; Carl Zeiss, Jena, Germany) was used for analysis of the morphology of the lithium disilicate surfaces in each group.

Statistical Analysis

Descriptive statistics used the mean of microshear bond strength (SBS) values (in megapascal) and the standard deviation (SD). D'Agostino test was used for data normality, and the values were analyzed by one-way ANOVA and Tukey post test ($\alpha=0.05$).

Results

Microshear bond strength values were significantly higher in Group AFF (11.01 ± 02.57 MPa) compared to control Group A (08.14±2.67 MPa) ($p<0.002$), while the group AF was not statistically different (09.42±2.52 MPa) from Group A (Fig. 2). The types of fractures are presented in Table 2. Adhesive fractures were predominant in all experimental groups (A: 75%, AF: 92% and AFF: 60%).

SEM photographs showed the ceramic surface after the

Table 1. Product, manufacturer and composition of the materials used in the study

Product	Manufacturer	Composition
Hydrofluoric acid	Eufar	9.6% hydrofluoric acid
Phosphoric acid	SDI	37% phosphoric acid
Silane	Angelus	Silane and ethanol
Adhesive (Adper Single Bond 2)	3M ESPE	Bis-GMA, HEMA, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids
Cement (RelyX Ultimate)	3M ESPE	Methacrylate monomers, radiopaque silanated filler and alkaline filler, initiator, stabilizers, pigments



Figure 1. Positioning of Tygons on the ceramic discs surface.

procedures in each group. In Group A it may be visualized the exposed vitreous matrix, thus resulting in an increased vitreous layer (Figs. 3A, 4A). For Group AF, an overlying vitreous matrix is exposed without vitreous layer (Figs. 3B, 4B). Group AFF exhibited an exposed vitreous matrix, fixed on the matrix surface, but more irregular and without a vitreous layer (Figs. 3C, 4C).

Discussion

The micromechanical retention of the ceramic surface is crucial to adhesive bonding with a resin luting cement. The ceramic surface has to be rough and clean (11), which may increase bond strength between the ceramic surface and the resin cement (12). When the ceramic surface of IPS Empress 2 was treated with hydrofluoric acid, stretched crystals and superficial irregularities were clearly observed. According to Holand et al. (13), the main crystal phase of IPS Empress 2 glass ceramic is formed by elongated crystals of lithium disilicate. A second phase is composed of lithium orthophosphate. A glass matrix surrounds both crystalline phases. Hydrofluoric acid is able to remove the glass matrix and the second crystalline phase, thus creating irregularities within the lithium disilicate crystals. The same results were observed in this study, and it was also noticed that the vitreous layer influenced bond strength values.

A previous study (14) found that the higher the pores, microcracks and irregularities, (analyzed micromorphologically), the higher the surface area and bonding potential of the ceramic surface. In this study, this increased surface micro roughness was shown in the

AFF group, with active PA application after the HA. PA acts as a neutralizing agent. Some studies (15,16) showed that this neutralization improves bond strength between the ceramic and resin cement, which was also observed in this study: bond strength was significantly higher with active PA application after HA application, being an effective method to remove or eliminate the vitreous precipitates.

However, the literature shows controversial results in terms of PA application as a cleaning agent (17,18). In these studies, the acid was only left on the ceramic surface, without agitation. This is the first study that investigates the active mode. Active PA application could act as a booster cleaning agent, superior to the passive mode, by promoting a deeper contact of the acid with the vitreous debris. Thus, when the PA is washed, the debris would be simultaneously removed, consequently enhancing matrix exposition prior to luting procedure.

It has been reported that these precipitates left by HA may be ultrasonically cleaned (19); however, other strategies for clinicians who do not have this device in their offices to clean the ceramics may be the active application of 37% PA, in order to increase the bond strength values between the resin cement and lithium disilicate ceramic surface.

SEM images confirm the findings of this study: the Figures "A" (only with 9.6% HA application) show the ceramic surface with a high amount of vitreous debris, represented by the white circles. Also, in the Figures "B" (with passive PA application after 9.6% HA), it may be seen a high number of organized vitreous debris, overlaying the cement interface. In contrast, in Figures "C" (with the active PA application after 9.6% HA) the surface is cleaner and rougher, which allowed a better adhesion to the resin cement, confirming the bond strength values found in this last group.

A recent systematic review (8) showed that although bonding between resin cements and glass ceramic improved in the recent years, HA is toxic, and alternative methods should be explored. One could speculate that the active PA application would be an interesting approach, as it acts like a cleaning agent; however, more studies, specifically biological studies, are required to confirm this speculation.

The methodology that was employed in this study to

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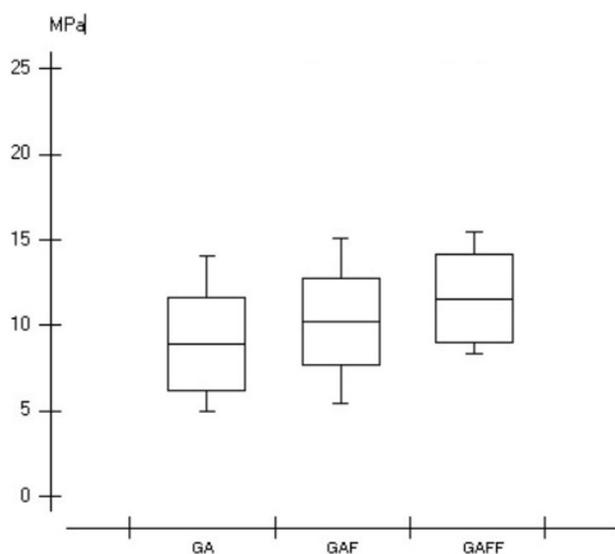


Figure 2. Mean and standard deviation (SD) of the experimental groups (in MPa). Group A: 9.6% HA application; Group AF: 9.6% HA application + cleaning with 37% PA in passive mode; Group AFF: 9.6% HA application + cleaning with 37% PA in active mode.

Table 2. Fracture type (in %) showed in the experimental groups

	Fracture type	
	Adhesive	Mixed
Group A	75	25
Group AF	92	8
Group AFF	60	40

evaluate bond strength was the microshear test, because ceramics are brittle materials, which could not be sectioned to produce specimens as for microtensile tests. Comparing with "macrotests", microshear requires only a small bonding area, leading to greater uniformity of stress distribution (8). Other advantages are the easier specimen preparation and handling, better dimension control and bond area of each specimen, as the tygon diameter is previously recognized (20). On the other hand, some testing parameters may affect the microshear bond strength values, like the time for adhesive photopolymerization and composite placement inside the tygons (21). In this study, care was taken to avoid the influences in the results: the adhesive was light cured before the tygon/resin cement placement, and the tygons were filled after positioning in ceramic treated surfaces.

The fracture mode of the specimens was predominantly adhesive or mixed, which is an advantage of the microshear

test, as no sectioning is required to produce the specimens (8).

A limitation of this study is the lack of mechanical or thermal aging before the microshear bond test. It is well recognized that aging in water (22), mechanical (23) or thermocycling (24) may influence negatively the bond strength values between ceramic and resin cements. A recent study found that the only group which has maintained its bond strength values between a hybrid ceramic and resin cement after 3 weeks was the one treated with HA (25). It seems appropriate that future studies analyze the bond strength obtained over longer periods (up to 6 months), in order to observe if there will be a real advantage in using the active application of 37% PA prior to 9.6% HA.

Also, it is worth mentioning that this study evaluated only one type of resin cement; so clinicians should be

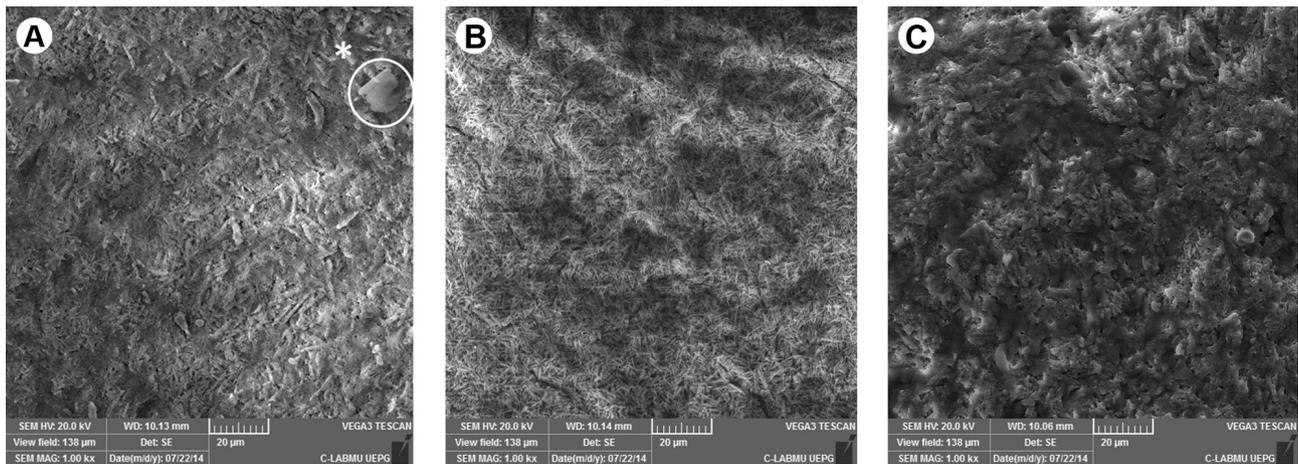


Figure 3. SEM images obtained from each experimental group (Original magnification 1000x): A: Group A; B: Group AF; C: Group AFF. The white circles show the exposed vitreous matrix.

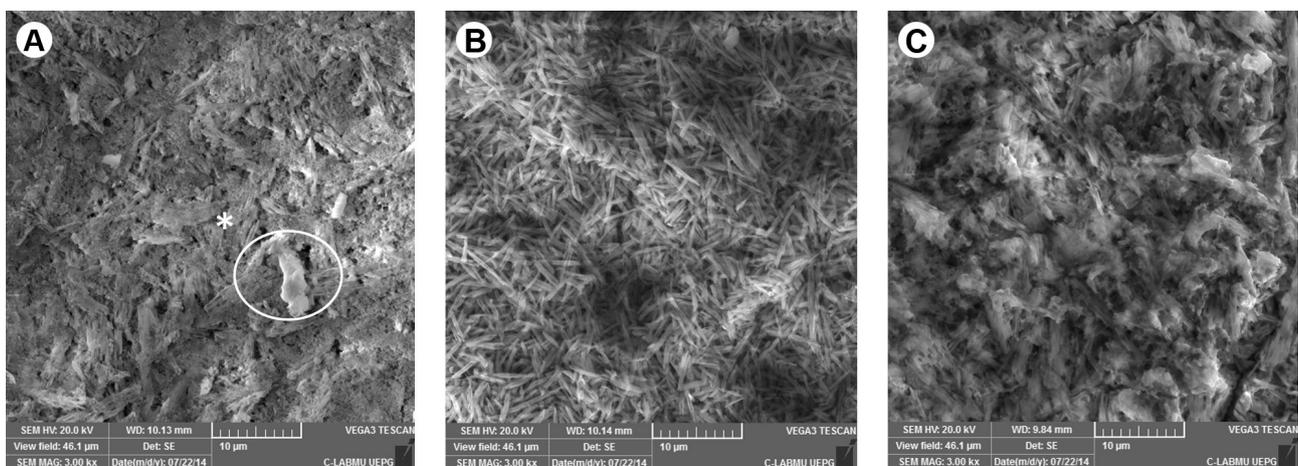


Figure 4. SEM images obtained from each experimental (Original magnification 3000x): A: Group A; B: Group AF; C: Group AFF. The white circles show the exposed vitreous matrix.

aware of extrapolating these results to other resin cement materials. Further studies are required to verify if different resin cements would provide the same results as found in the present study.

Based on the results, the null hypothesis could not be accepted. It was concluded that the active application of 37% PA after 9.6% HA application increased the microshear bond strength values between the resin cement and lithium disilicate ceramics.

Resumo

O objetivo desse estudo foi avaliar o efeito da aplicação passiva ou ativa do ácido fosfórico após o tratamento com ácido hidrófluorídrico na resistência de união ao microcissalhamento entre cimento resinoso e dissilicato de lítio. Trinta discos foram confeccionados com IPS Emax 2 (10 mm de espessura e 10 mm de diâmetro). Os espécimes foram divididos em três grupos (A: aplicação do ácido hidrófluorídrico 9,6%; AF: aplicação do ácido hidrófluorídrico 9,6% + limpeza com ácido fosfórico 37% em modo passivo; AFF: aplicação do ácido hidrófluorídrico 9,6% + limpeza com ácido fosfórico 37% no modo ativo). Para o teste de microcissalhamento, quatro *tygons* (0,9 de diâmetro e 0,2 mm de altura) foram preenchidos com cimento resinoso (RelyX Ultimate) e dispostos sobre os discos de cerâmica. Após o teste, os modos de fratura foram examinados por microscopia eletrônica de varredura. Os dados foram analisados estatisticamente por ANOVA e pós-teste de Tukey ($\alpha=0,05$). Os valores de resistência de união foram significativamente superiores para o grupo AFF ($11,0 \pm 2,5$ MPa), comparado ao grupo A ($8,1 \pm 2,6$ MPa) ($p < 0,002$), mas este não estatisticamente diferente do grupo AF ($9,4 \pm 2,5$ MPa). Concluiu-se que a aplicação ativa do ácido fosfórico 37% após o uso do ácido hidrófluorídrico 9,6% aumenta a resistência de união entre o cimento resinoso e a cerâmica de dissilicato de lítio.

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