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Laboratory Research

The Effect of Hydrofluoric Acid Concentration on the Bond Strength and Morphology of the Surface and Interface of Glass Ceramics to a Resin Cement

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Clinical Relevance

The current results support that 7.5% and 10% concentrations of hydrofluoric acid are more reliable for etching glass ceramics than are higher or lower concentrations. The use of unfilled resin after silane resulted in higher microshear bond strength and provided better interaction between ceramic and resin cement.

SUMMARY

The purpose of this study was to evaluate the influence of various concentrations of hydrofluoric acid (HF) on the surface/interface morphology and μ–shear bond strength (μSBS) between IPS Empress Esthetic (EST) (Ivoclar Vivadent) and IPS e.max Press (EMX) (Ivoclar Vivadent) ceramics and resin cement. Ceramic blocks were divided into 12 groups for each

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kind of ceramic. Six different HF concentrations were evaluated: 1%, 2.5%, 5%, 7.5%, 10%, and 15%. All groups were silanated after etching, and half of the specimens within each group received a thin layer of unfilled resin (UR). Three resin cement cylinders were prepared on each ceramic block for μSBS testing. The specimens were stored in distilled water at 37°C for 24 hours. The μSBS test was carried out in a universal testing machine at a crosshead speed of 0.5 mm/min until fracture. The data were submitted to three-way analysis of variance and multiple comparisons were performed using the Tukey post hoc test ($p<0.05$). The etched surfaces and bonded interfaces were evaluated using scanning electron microscopy. μSBS means (MPa) for 1%, 2.5%, 5%, 7.5%, 10%, and 15% HF concentrations were, respectively, 25.2, 27.2, 30.1, 31.4, 33.3, and 31.8. μSBS means with or without UR application measured 32.24 and 27.4, respectively; EST and EMX measured 29.8 and 29.9, respectively. For the HF concentrations, 10% and 15% showed higher μSBS means than did 1% and 2.5% ($p<0.05$); 7.5% was higher than 1% ($p<0.05$); and no statistical differences were found among the other concentrations ($p>0.05$). When evaluating UR, μSBS mean was significantly higher and better infiltration was observed on the etched surfaces. No statistical difference was found between the ceramics. The HF concentration and UR influenced the bond strength and surface/interface morphology.

INTRODUCTION

The indications for using dental ceramics have increased as a result of their optimal characteristics, such as their ability to mimic the function and esthetics of dental tissues, biocompatibility, color stability, high mechanical resistance, radiopacity, and low thermal conductivity.[1] Among the several types of ceramics used in dentistry, IPS e.max Press (EMX; Ivoclar Vivadent, Schaan, Liechtenstein), which contains lithium disilicate and is reinforced with lithium orthophosphate crystals,[1] and IPS Empress Esthetic (EST; Ivoclar Vivadent), a leucite-reinforced material, can be highlighted, as they are widely utilized and have been evaluated in many studies.[2]

The bond between glass ceramics and resin cements is one of the key factors to long-term clinical success.[3] Additionally, the bonding quality has a direct relation to the ceramic type involved, as well as to the variables that influence ceramic surface etching. Etching is responsible for both an increased contact surface area and improving the interaction between the luting agent and ceramic.[4,5] The size and number of irregularities created on the surface of a glass ceramic as a result of etching have been associated with acid formulation, dilution of the acid,[4,6] and etching time.[5,7-9] Researchers have reported that the EMX surface should be conditioned for 20 seconds[10] and the EST surface for 60 seconds, in both cases with 10% hydrofluoric acid (HF) as a chemical surface conditioner.[11-15] HF requires minimal time for effective application, carries a low cost, and is very efficient at creating surface roughness.[16,17]

On the other hand, HF acid is extremely corrosive and is capable of causing severe trauma to soft tissues. Furthermore, the lesion severity is directly related to the exposure time and acid concentration.[15] Even though HF is not normally applied on soft tissue, less concentrated HF would cause less injury in accidental contact situations. Little is known about the effect of increased or decreased HF concentrations on the surface morphology or bonding ability of this ceramic. Trakyali and others[18] showed that no statistical difference was found in terms of bond strength when using HF concentrations of 5% and 9.6%.

Resin cements need sufficient wettability to completely infiltrate the irregularities of a ceramic surface.[19,20] Normally, manufacturers recommend the use of silane on the internal ceramic surface prior to applying resin cement. With the addition of silica, glass ceramics are able to be adhesively bonded, with chemical bonding to the resin cement due to the previous application of silane, which improves the durability and bond strength.[11,13,21-24] However, it is questionable if the silane and resin cement are efficient in wetting the surface and filling up irregularities when different HF concentrations are used. Although some clinicians apply a layer of unfilled resin on the ceramic surface after the application of silane, the current literature[5] offers little information about luting purposes. It is likely that the use of an unfilled resin will improve bond strength and adaptation of substrates along the ceramic-resin cement interface, as observed by Naves and others.[5]

Therefore, the aim of this study was to evaluate the influence of various HF concentrations on the μ–shear bond strength (μSBS) of EST and EMX ceramics when using a resin cement, with or without the application of an unfilled resin after applying silane, while also evaluating the modes of failure.

Table 1: *Group Descriptions*

Group	Surface Treatment		
	Hydrofluoric Acid Concentration, %	Postetching Treatment	Ceramic
G1	1	Silane	IPS Empress Esthetic
G2	1	Silane + unfilled resin	
G3	2.5	Silane	
G4	2.5	Silane + unfilled resin	
G5	5	Silane	
G6	5	Silane + unfilled resin	
G7	7.5	Silane	
G8	7.5	Silane + unfilled resin	
G9	10	Silane	
G10	10	Silane + unfilled resin	
G11	15	Silane	
G12	15	Silane + unfilled resin	
G13	1	Silane	IPS e.max Press
G14	1	Silane + unfilled resin	
G15	2.5	Silane	
G16	2.5	Silane + unfilled resin	
G17	5	Silane	
G18	5	Silane + unfilled resin	
G19	7.5	Silane	
G20	7.5	Silane + unfilled resin	
G21	10	Silane	
G22	10	Silane + unfilled resin	
G23	15	Silane	
G24	15	Silane + unfilled resin	

This present study also characterized the morphology aspect of the etched surfaces and the interfaces created between the substrates. The hypotheses tested were as follows: 1) Different HF concentrations do affect the μSBS; and 2) The unfilled resin does influence the bond strength and interface homogeneity.

METHODS AND MATERIALS

Ceramic Blocks

One hundred forty-four square ceramic blocks (8 mm × 8 mm × 3.0 mm thick) were fabricated for each type of ceramic, EST and EMX, in accordance with the manufacturer's instructions. Square wax patterns were made, sprued, and invested with phosphate-based material (Esthetic Speed or IPS PressVest Speed, Ivoclar Vivadent), and the wax was eliminated in an automatic burn-out furnace (Vulcan A- 550, Degussa-Ney, Yucaipa, CA, USA) at 850°C for one hour. The EST and EMX ingots were pressed into the investment molds in an automatic press furnace (EP 600, Ivoclar Vivadent). After cooling, the specimens were divested, placed in a horizontal position, embedded in polyester resin (Resapol T208, Difibra/Fiberglass Ltda, Mogi das Cruzes, SP, Brazil) in rigid polyvinyl chloride tubes with a 20-mm diameter and 20-mm height, and submitted to wet polishing with 600-, 1200-, and 2000-grit silicon carbide abrasive papers (Norton SA, São Paulo, SP, Brazil) to obtain a flat surface.

Ceramic Surface Treatments

The ceramic blocks (144 in total) were randomly divided into 24 groups (n=6), as defined by the HF concentrations (1%, 2.5%, 5%, 7.5%, 10%, and 15%; Formula & Ação, São Paulo, SP, Brazil). Table 1 presents a description of the tested groups. In groups 1 through 12, the EST specimens were etched for 60 seconds and rinsed with distilled water for one minute. The specimens in groups 13 to 24, the EMX ceramic blocks were etched for 20 seconds and rinsed with distilled water for one minute. All specimens were then ultrasonically cleaned in distilled water for one minute and dried with compressed oil-free water/air spray. A silane coupling agent (RelyX Ceramic Primer, 3M ESPE, St Paul, MN, USA) was applied onto all ceramic surface specimens and allowed to air dry for 15 seconds, followed by air heat drying for 45 seconds. Half of the specimens from each group received a thin layer of unfilled resin (Scotchbond MultiPurpose, 3M ESPE, Seefeld, Germany) that was light activated for 10 seconds using a LED source (UltraLume 5, Ultradent Inc, South Jordan, UT, USA) with an irradiance of 1.100 mW/cm^2.

Bond Strength Testing

The method used to obtain specimens for the μSBS testing and the design of the test[5,25] are shown in Figure 1. Elastomer molds (Express STD, 3M ESPE), which were 3 mm thick and contained three cylinder-shaped orifices (1 mm in diameter), were placed onto the ceramic surfaces, allowing the delimitation of the bonding area. The orifices were filled with resin cement (Variolink II, shade A3; Ivoclar Vivadent), and a transparent polyester strip and glass plate were placed over the filled mold. A 250*g* cementation load was applied for two minutes. The glass plate was removed and the resin cement was light activated for 40 seconds using a LED source (UltraLume 5, Ultradent). The specimens were then stored in distilled water at 37°C for 24 hours. Three cylinders were built up on each ceramic block, with 18 cylinders tested for each group.

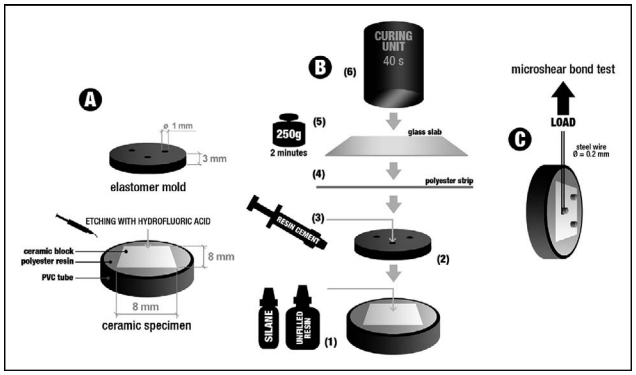

Figure 1. *Experimental setup of the study. After etching, (1) silane was applied to the ceramic surface, and for half of the specimens, an unfilled resin was applied after applying the silane; (2) an elastomer mold with cylinder-shaped orifices was positioned onto the surface and photo-activation of the unfilled resin was performed; (3) orifices were filled with resin cement; (4) a polyester strip and glass slab were placed over the filled mold; (5) cementation load applied for two minutes; (6) photo-activation of the resin cement was facilitated.*

After storage, all resin cement cylinders were checked using optical microscopy (Olympus Corp, Tokyo, Japan) at 40× magnification, and those with irregularities, bonding defects, or flaws were eliminated. For the μSBS testing, a thin steel wire (0.2 mm in diameter) was looped around each cylinder and aligned with the bonding interface (Figure 1). The bonding test was conducted using a mechanical testing machine (model 4411; Instron; Canton, MA, USA) and at a crosshead speed of 0.5 mm/min until failure. The fractured specimens were examined under optical microscopy (Olympus Corp) at 40× magnification. Failure modes were classified as follows: adhesive (mode 1); cohesive within ceramic (mode 2); cohesive within resin cement (mode 3); and mixed, involving resin cement, ceramic, and composite (mode 4).

Statistical Analysis

Values of μSBS were calculated and the data provided in megapascals. For each group, six specimens were tested, and the average value of the three resin cylinders was recorded as the bond strength for each specimen. μSBS data were submitted to three-way analysis of variance, and multiple comparisons were performed using the Tukey post hoc test ($p<0.05$).

Scanning Electron Microscopy (SEM) Evaluation

In order to observe the surface characteristics of the conditioned surfaces, etched specimens for each HF concentration were sputter coated with gold (Balzers-SCD 050, Balzers Union, Aktiengeselischaft, Fürstentun, Liechtenstein) for 180 seconds at 40 mA. The specimens were then mounted on coded brass stubs and examined using SEM (LEO 435 VP, Cambridge, UK), operated at 20 Kv, by a single operator. Samples were examined under magnifications varying from 2000× to 3000×. Additionally, the EST or EMX ceramic blocks were sectioned, etched, and silane coated, and the same kind of ceramics were bonded to each other using resin cement to observe the morphology at the bonding interfaces for each group evaluated. After storage for 24 hours, the specimens were sectioned perpendicular to the bonding interface and embedded in epoxy resin (Buehler, Lake Buff, IL, USA) so that the ceramic-cement interfaces could be viewed. The specimens were wet-polished with 400-, 600-, 1200-, and 2000-grit silicon carbide abrasive papers, followed by polishing with 3-, 1-, and 0.5-μm diamond compounds. After polishing and ultrasonic cleaning, the specimens were gold sputter-coated (Balzers-SCD 050, Balzers Union). The cross-section profiles were examined under SEM (LEO 435 VP), focusing on the depth of etching, micromechanical entanglement and integrity, homogeneity, and continuity along the bonding interface, similar to what has been previously described by Naves and others.[5] Samples were examined under magnifications varying from 2000× to 3000×.

RESULTS

Microshear Bond Strength (μSBS)

The mean values of μSBS are shown in Table 2. Ceramic vs unfilled resin ($p=0.367$), ceramic vs HF concentration ($p=0.100$), and unfilled resin vs HF concentration ($p=0.196$) values did not show significant interaction of factors. The triple interaction between factors was not significant ($p=0.565$). Significant differences for unfilled resin ($p<0.001$) and HF concentration ($p<0.001$) were detected. No difference was detected for the ceramics ($p=0.957$). When unfilled resin was used, the mean value of μSBS was significantly higher when compared to values for the specimens prepared without it ($p<0.05$). When the ceramic material was compared, no statistical difference was found between the EST and EMX ceramics ($p>0.05$). For the HF concentrations, 10% and 15% showed mean values of μSBS that were significantly higher when compared to the 1% and 2.5% values ($p<0.05$). The concentration of 7.5% was significantly higher when compared to that of 1% ($p<0.05$). No statistical differences were found among the other concentrations ($p>0.05$).

Table 2: *Means of Microshear Bond Strength ± Standard Deviation (MPa) for All Groups. Parenthetical Values under Tukey % Indicate the Overall Mean Bond Strength for the Indicated Hydrofluoric Acid Concentration.*

Ceramic	Hydrofluoric Acid Concentration, %	Unfilled Resin[a]		Tukey, %[a]
		With	Without	
IPS Empress Esthetic (29.8)[b]	1	27.6 ± 6.3	20.7 ± 5.3	
	2.5	27.3 ± 7.9	23.8 ± 5.7	
	5	32.5 ± 9.1	31.1 ± 5.9	
	7.5	32.1 ± 3.1	31.8 ± 6.1	1 (25.2) c
	10	39.2 ± 6.7	31.3 ± 5.5	2.5 (27.2) bc
	15	32.3 ± 3.2	27.6 ± 6.2	5 (30.1) abc
IPS e.max Press (29.9)[b]	1	29.2 ± 5.4	23.6 ± 5.6	7.5 (31.4) ab
	2.5	29.4 ± 9.8	28.3 ± 5.7	10 (33.3) a
	5	28.5 ± 3.9	28.1 ± 6.1	15 (31.8) a
	7.5	34.4 ± 3.3	27.3 ± 5.4	
	10	36.1 ± 4.1	26.6 ± 5.7	
	15	38.5 ± 6.1	28.9 ± 3.1	
Tukey		32.24 A	27.4 B	

[a] *Same capital letters indicate no significant differences with or without unfilled resin application ($p>0.05$).*
[b] *No significant differences for ceramic ($p>0.05$), and means followed by different lowercase letters indicate significant differences for hydrofluoric acid concentrations (%) ($p<0.05$).*

Failure Analysis

A descriptive analysis of failure modes is shown in Table 3. A predominance of cohesive within ceramic failure (mode 2) was found for the ceramic EST. For the EMX, a predominance of adhesive failures (mode 1) was detected for the 1% to 5% acid concentrations and failures that were cohesive within resin cement (mode 3) for the 7.5% to 15% acid concentrations.

SEM Evaluation

SEM images of etched surfaces with 1%, 2.5%, 5%, 7.5%, 10%, and 15% HF concentrations are shown in Figures 2 and 3. Figures 4 and 5 present images of the ceramic/resin cement bonding interfaces.

The EMX ceramic presented with greater vitreous phase dissolution and exposure of lithium disilicate crystals with increased HF concentrations. Figure 2A and B exhibited slight vitreous phase dissolution, and Figure 2C and D showed similar patterns, with more evident vitreous dissolution. The images in Figure 2E and F show greater vitreous phase dissolution on the ceramic surface due to the higher HF concentration. Figure 3 presents images of acid etching with the various HF concentrations on the EST ceramic surface. Minimal vitreous phase dissolution can be observed in Figure 3A, while Figure 3B presented slightly greater vitreous phase dissolution. The images in Figure 3C and D present even greater vitreous phase dissolution, causing microporosities. Figure 3F revealed the formation of fissures arising from surface etching.

Figure 4 represents the bonded interface for EMX. The images in Figure 4B, E, and F demonstrated the deficient quality of bonding between ceramic and resin cement with unfilled voids (indicated with a white arrow). When the unfilled resin was used, as in the images in Figure 4A, C, and D, a completely infiltrated interface is seen between ceramic, unfilled resin, and resin cement. The same situation is shown for EST (Figure 5).

DISCUSSION

Alterations in the morphology of the ceramic surface may promote a better bond strength.[26] HF is a modifier and etching agent indicated for ceramic that contains silica,[14,27] acting to dissolve the vitreous phase, exposing crystals and resulting in microporosities on the ceramic structure.[27-30] This provides increased surface area and improved bonding quality[6,11,17,27,28,30,31] and promotes better contact between the restoration material and the resin cement.[4,13,14,28,32]

In this study, the first hypothesis, which stated that different HF concentrations applied on ceramic surface would affect the μSBS between ceramic and resin cement, was accepted. The mean results showed that lower values for μSBS were obtained for HF concentrations of 1% and 2.5% for both EST and EMX, with a statistically significant difference when compared to the values associated with HF concentrations of 10% and 15%. Lower concentrations were not enough to properly dissolve the

Table 3: *Failure Mode Analysis of the Debonded Specimens (%) Among Groups*[a]

Groups	Failure Modes			
	Mode 1	Mode 2	Mode 3	Mode 4
IPS Empress Esthetic (EST)				
1 – EST1	44	56	0	0
2 – EST1 *UR*	50	50	0	0
3 – EST 2.5	28	66	6	0
4 – EST 2.5 *UR*	28	66	0	6
5 – EST 5	0	33	56	11
6 – EST 5 *UR*	6	88	0	6
7 – EST 7.5	0	56	28	16
8 – EST 7.5 *UR*	0	88	6	6
9 – EST 10	0	78	11	11
10 – EST 10 *UR*	11	73	0	16
11 – EST 15	6	94	0	0
12 – EST 15 *UR*	6	61	11	22
IPS e.max Press (EMX)				
13 – EMX 1	100	0	0	0
14 – EMX 1 *UR*	100	0	0	0
15 – EMX 2.5	88	6	6	0
16 – EMX 2.5 *UR*	88	6	6	0
17 – EMX 5	50	0	22	28
18 – EMX 5 *UR*	88	0	6	6
19 – EMX 7.5	33	0	56	11
20 – EMX 7.5 *UR*	44	0	56	0
21 – EMX 10	6	6	44	44
22 – EMX 10 *UR*	11	6	61	22
23 – EMX 15	0	6	50	44
24 – EMX 15 *UR*	28	6	44	22

Abbreviation: UR, unfilled resin.
[a] *Failure modes were classified as follows: adhesive (mode 1); cohesive within ceramic (mode 2); cohesive within resin cement (mode 3); and mixed, involving resin cement, ceramic, and composite (mode 4).*

vitreous phase and exhibited minimal vitreous phase dissolution, in contrast to higher concentrations (Figures 2 and 3). This is likely explained by the presence of fewer microporosities, promoting reduced contact between the ceramic surface and resin cement, resulting in less mechanical interlocking and lower bond strengths, as the shear bond strength is directly influenced by ceramic surface roughness.[4] The degree of ceramic dissolution is proportional to the HF concentration (Figures 2 and 3) and may promote higher values of bond strength.[32] It has been shown that bond strength is more directly influenced by the type of etching agent than it is by the resin cement.[33]

When the ceramics were etched with 7.5% HF, the mean value of μSBS was statistically significantly higher when compared to the values associated with the HF concentration of 1%, although the value associated with the 7.5% concentration was not statistically different from those of the other concentrations. The 7.5% concentration promoted effective dissolution of the vitreous phase in both ceramics. This is likely because 7.5% HF was able to promote sufficient change to the ceramic surface, which improved the mechanical interlocking of the resin cement to the ceramic structure. Therefore, the 7.5% concentration could be as easily indicated for clinical use as the widely used 10%. HF can be harmful and particularly aggressive to soft tissues, but symptoms may not be apparent immediately after exposure because the lesion severity is directly related to the exposure time and the acid concentration.[15] Even though HF is not applied on soft tissue, less concentrated HF would cause less injury in accidental contact situations.

Chen and others[7] and Zogheib and others[34] found rougher feldspathic ceramic surfaces with increased

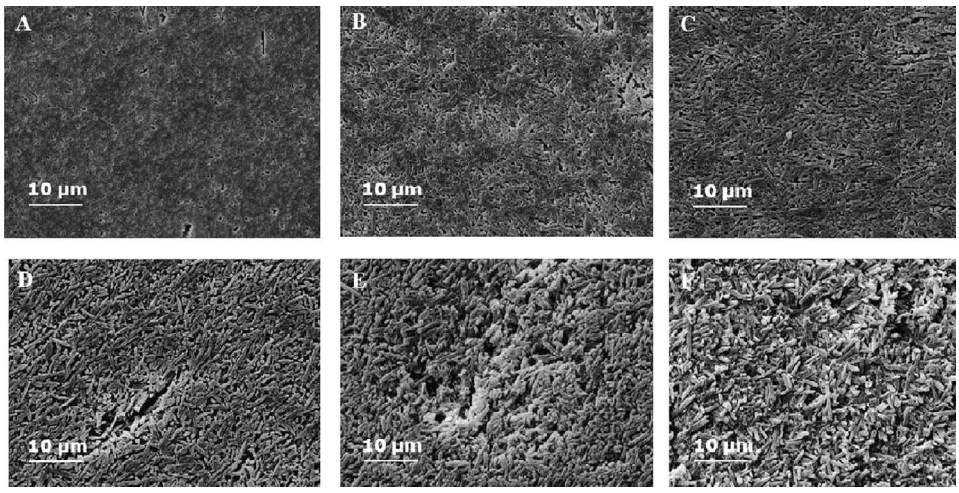

Figure 2. *Images resulting from acid etching with hydrofluoric acid (HF) on ceramic surface (IPS e.max Press). For HF concentrations (A: HF 1%, B: HF 2.5%; C: HF 5%; D: HF 7.5%; E: HF 10%; and F: HF 15%), different etching patterns were found with distinct degrees of vitreous phase dissolution and exposure of lithium disilicate crystals. Images A and B show poor dissolution of the vitreous phase, while there is an increase in the degree of vitreous phase dissolution with higher HF concentrations.*

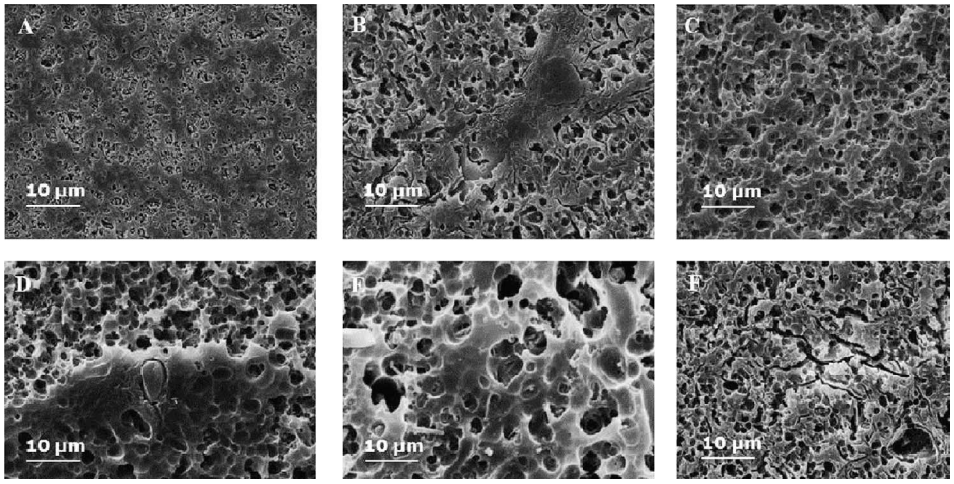

Figure 3. *Images resulting from acid etching with hydrofluoric acid (HF) on ceramic surface (IPS Empress Esthetic). For HF concentrations (A: HF 1%, B: HF 2.5%; C: HF 5%; D: HF 7.5%; E: HF 10%; and F: HF 15%), different etching patterns were found with distinct degrees of vitreous phase dissolution. Image A represents a poor dissolution of vitreous phase, while there is an increase in the degree of vitreous phase dissolution with higher HF concentrations.*

etching times (ranging from 20 to 180 seconds). Thus, if a greater etching time were adopted with the 1% and 2.5% HF used in this current research, it is likely that improved bond strengths could be obtained, providing safer patterns for working with HF. However, if the etching time had been increased, the resulting stronger and deeper ceramic degradation could have weakened its structure.[34,35] Beyond that, the buffering-acid process can increase the pH, as the reactants of the reaction are consumed, decreasing its etching effect. Therefore, dentists should be extremely careful when using an increased etching time with HF, considering that this protocol would not necessarily result in a better etched surface and/or bond strength.

It has been shown[5] that the effectiveness of bonding using only silane depends on the ability of the bonding agent to fill the irregularities and to provide a close contact between the resin cement and the ceramic. However, when an unfilled resin was used, it infiltrated the etched surface irregularities and improved the adaptation of the resin cement/ceramic interface and bond strength. In this present study, when an unfilled resin was used with a silane coupling agent for both ceramics, the mean values of μSBS were significantly higher when compared to those of specimens that received only silane. Therefore, the second hypothesis was also accepted. Similar results were found by Hooshmand and others[22] and Naves and others,[5] who observed greater means of μSBS when an unfilled resin was applied after the application of a silane. Significant evidence was also found when the unfilled resin was applied to the bonded interface and evaluated under SEM (Figures 4A,C,D and 5C,D,E), in which complete penetration of unfilled resin in ceramic irregularities and a homogeneous interface were found between the ceramic and resin cement. This occurred as a result of the lower viscosity presented by the unfilled resin in relation to the resin cement[5] and because of a better interaction of the unfilled resin when applied to the ceramic surface treated

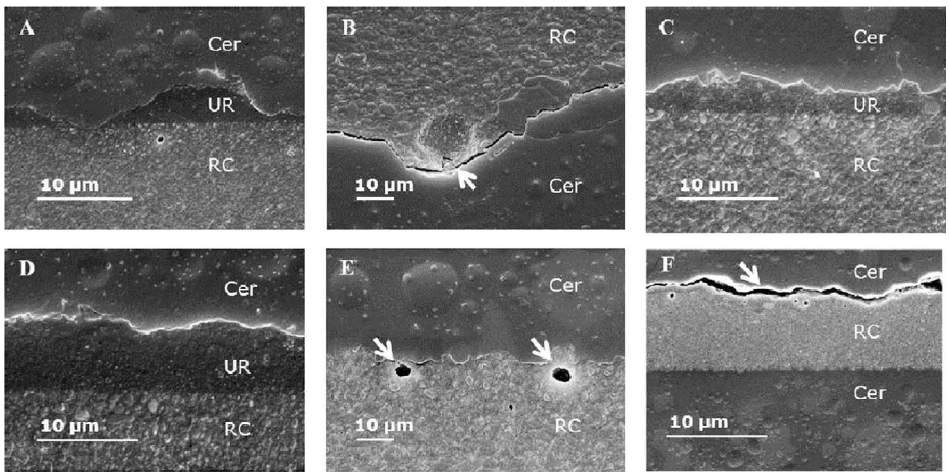

Figure 4. *Images resulting from bond interface analysis (IPS e.max Press). RC, resin cement; Cer, ceramic; UR, unfilled resin; HF, hydrofluoric acid. (A) HF 1%; (B) HF 2.5%; (C) HF 5%; (D) HF 7.5%; (E) HF 10%; (F) HF 15%. Image A shows an interaction without failures among the ceramic, unfilled resin, and resin cement. The white arrow in image B indicates an incomplete interaction between ceramic and resin cement. The unfilled resin was able to penetrate the ceramic in images C and D. The white arrows in images E and F indicate failures at the resin cement-ceramic interface, when the unfilled resin was not applied.*

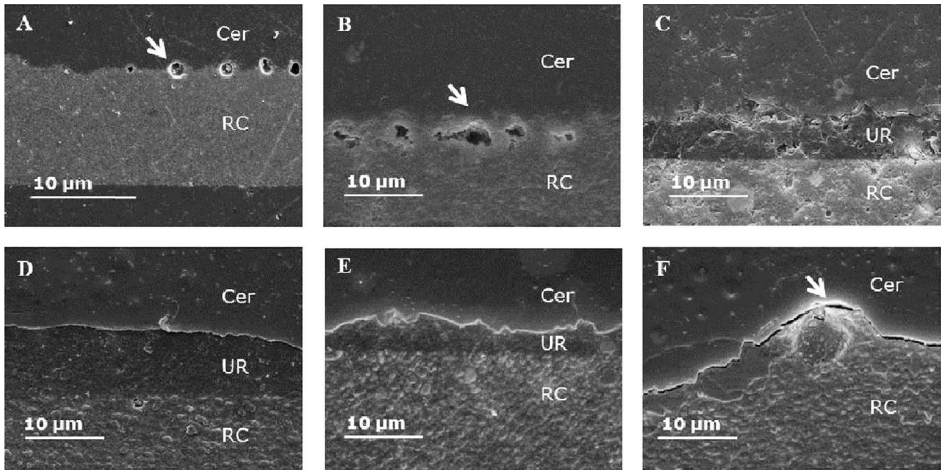

Figure 5. *Images resulting from bond interface analysis (IPS Empress Esthetic). RC, resin cement; Cer, ceramic; UR, unfilled resin; HF, hydrofluoric acid. (A) HF 1%; (B) HF 2.5%; (C) HF 5%; (D) HF 7.5%; (E) HF 10%; (F) HF 15%. Images A and B show incomplete resin cement penetration of surface without the application of the unfilled resin (white arrow). Images C, D, and E show complete penetration of the unfilled resin into ceramic irregularities. However, image F shows an incomplete interaction between the resin cement and ceramic (white arrow).*

with silane.[6,17,23] Under these conditions, the unfilled resin more easily penetrates the etched ceramic surface, when compared to the resin cement, which has filler particles in its composition that hamper its penetration into deeper irregularities on the ceramic surface. According to Naves and others,[5] the unfilled resin fills voids that the resin cement cannot. On the ceramic surfaces that did not receive the application of the unfilled resin, the interface images suggest voids unfilled by the resin cement, causing a nonhomogeneous interface (Figures 4B,E,F and 5A,B,F). This could lead to stress concentration and induce clinical failure since the bonding interface plays an important role in the long-term durability of ceramic restorations.[3] This situation may also have negatively affected the immediate bond strengths.

The analysis of failure modes showed a predominance of cohesive failures within ceramic for the EST ceramic. The EMX presented a predominance of adhesive failures for the 1% to 5% HF concentrations, indicating poor bond quality, and cohesive within–resin cement failures for the 7.5% to 15% HF concentrations (Table 3), most likely due to improved micromechanical interaction between the ceramic and resin cement.[5] This difference can be explained by the low resistance to crack propagation in the EST ceramic due to dissimilar ceramic composition, nonhomogeneous stress distribution at the interface (as produced by the microshear bond test),[36] or the influence of greater HF concentrations on the leucite-based ceramic.

The EST ceramic is a glass-based ceramic reinforced by leucite crystals, which is about 35.5% ± 5% vol and is indicated for inlays, onlays, crowns, and for veneering other core-ceramics. EMX is a lithium disilicate–reinforced glass ceramic, containing about 70% ± 5% vol of crystalline phase. These features provide improved mechanical properties for the EMX;[1] therefore, it is indicated for three-unit fixed partial dentures up to the second premolar.[37] According to de Melo and others,[38] the higher content of the crystalline phase and lower vitreous phase causes fewer cohesive failures in the ceramic. The fact that the EST is submitted to higher etching times compared to EMX might also explain the cohesive ceramic failures found in this current research. Therefore, the bond quality should not only be evaluated by bond strength values but also by its association with failure analysis and fractography to provide a better clinical preview of the performance.[22,36]

The data and images in the present study demonstrate that the HF concentration influenced the μSBS and influenced the surface and ceramic/resin cement bonding interface. Moreover, the application of a low-viscosity unfilled resin after application of a silane may better infiltrate the etched surface of the ceramic, increasing the bond strength, the adaptation of the interface resin cement-ceramic, and possibly its clinical longevity. Therefore, the use of an unfilled resin should be encouraged in clinical practice. However, the thickness of this layer should be as thin as possible. Care should be taken in clinical practice, regardless of the HF concentration used, because HF is toxic and capable of causing severe trauma to soft tissues. Therefore, the etching procedure must be done with personal protective equipment in well-ventilated rooms to avoid any further damage to the professionals. Future studies should be carried out using different cementation loads, viscosities of the resin

cement, thermal cycling, and degradation of the unfilled resin.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be made:

1. The various HF concentrations influenced the bond strength and surface/interface morphology.
2. Application of an unfilled resin increased the μSBS; it also promoted better infiltration of the irregularities of the etched surfaces for both ceramics.
3. No statistical difference was found in μSBS between the two ceramics. The EST showed a tendency for cohesive failure in ceramic, and EMX presented with adhesive and cohesive failures in resin cement.

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Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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