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Laboratory Research

Effect of Hydrofluoric Acid Concentration and Etching Time on Bond Strength to Lithium Disilicate Glass Ceramic

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Clinical Relevance

The minimum HF concentration and time for proper bonding to lithium disilicate ceramics is 5% and 20 seconds.

SUMMARY

The aim of this study was to evaluate the influence of different concentrations of hydrofluoric acid (HF) associated with varied etching times on the microshear bond strength (μSBS) of a resin cement to a lithium disilicate glass ceramic. Two hundred seventy-five ceramic blocks (IPS e.max Press [EMX], Ivoclar Vivadent), measuring 8 mm × 3 mm thickness, were randomly distributed into five groups according to the HF concentrations (n=50): 1%, 2.5%, 5%, 7.5%, and 10%. Further random distribution into subgroups was performed according to the following etching times (n=10): 20, 40, 60, 120, and 20 + 20 seconds. After etching, all blocks were treated with a silane coupling agent followed by a thin layer of an unfilled resin. Three resin cement cylinders

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(=1 mm) were made on each EMX surface, which was then stored in deionized water at 37°C for 24 hours before testing. The μSBS was in a universal testing machine at a crosshead speed of 1 mm/min until failure. Data were submitted to two-way analysis of variance, and multiple comparisons were performed using the Tukey post hoc test (α=0.05). One representative EMX sample was etched according to the description of each subgroup and evaluated using scanning electron microscopy for surface characterization. The HF concentrations of 5%, 7.5%, and 10% provided significantly higher μSBS values than 1% and 2.5% (*p*<0.05), regardless of the etching times. For 1% and 2.5% HF, the etching times from 40 to 120 seconds increased the μSBS values compared with 20 seconds (*p*<0.05), but etching periods did not differ within the 5%, 7.5%, and 10% HF groups (*p*>0.05). The effect of re-etching was more evident for 1% and 2.5% HF (*p*<0.05). Different HF concentrations/etching times directly influenced the bond strength and surface morphology of EMX.

INTRODUCTION

Glass ceramic materials have been widely used in dentistry to restore lost/fractured/decayed teeth. Their optimum properties, adhesion ability to dental tissues,[1] high esthetics, biocompatibility, and thermal expansion similar to the tooth structure are the key factors for their adoption by dental practioners.[2-4] Glass ceramic reinforced by lithium disilicate crystals has shown excellent clinical outcomes with great optical/mechanical properties[5-7] and high survival rates over time.[8,9]

The clinical success of ceramic restorations depends on several factors, such as ceramic composition and luting procedures.[10] The ideal bonding to lithium disilicate glass ceramic is achieved with the sum of etching with hydrofluoric acid (HF) followed by a silane coupling agent. This protocol has been recognized as the most accepted surface treatment for glass ceramics.[3,4,11,12] HF has the ability to condition the ceramic surface, since it removes the glassy matrix and exposes the lithium disilicate crystals.[4,13-15] As a consequence, an increased surface area for micromechanical entanglement is promoted, improving the interaction between ceramic and resin cement with increased bond strength.[1,3,4,15-17] Also, the use of a thin layer of unfilled resin prior to the resin cement improved bond strength and the interfacial quality between lithium disilicate glass ceramic and resin cement by promoting a better infiltration to the superficial irregularities of the etched ceramic surfaces.[4]

The etching efficiency of HF depends on the concentration, etching time, temperature, and dilution of the acid solution.[4,10,11,15,18] The manufacturer of IPS e.max Press (EMX; Ivoclar Vivadent, Schaan, Liechtenstein), a commercial brand that presents ±70 vol% of lithium disilicate crystals dispersed in an amorphous vitreous phase, recommends that EMX should be etched with a 4.8% HF concentration for 20 seconds. However, several clinical reports and *in vitro* studies, published from 2011 to date, have reported different HF concentrations and etching periods on lithium disilicate ceramic, such as 10% for 15 seconds,[19] 10% for 20 seconds,[6,7,10,20-22] 9.6% for 30 seconds,[23] 9.5% for 60 seconds,[3] 4.8% for 60 seconds,[24,25] 5% for 60 seconds,[26] and 4.8% and 5% for 20 seconds.[27-29] Thus, a consensus regarding the most suitable etching protocol for glass ceramics is not clear,[30] especially for lithium disilicate glass ceramics.

Although the effects of HF on glass ceramics are elucidated in the literature, there have been no reports that specifically associated different HF concentrations with increased etching times on the bond strength of EMX. In addition, given the hazardous nature of HF,[17,18] it is important to discuss how lower HF concentrations and different etching times would influence the bonding characteristics of EMX and, consequently, guide the clinician toward the more adequate etching protocol to be adopted.

Therefore, the purpose of this study was to evaluate the effect of different HF concentrations associated with varied etching times on the micro-shear bond strength (μSBS) and surface morphology (scanning electron microscopy [SEM] analysis) of EMX luted with a resin cement. The hypotheses tested were 1) HF concentrations affect the μSBS and the etched surface morphology and 2) different etching times affect the μSBS and the etched surface morphology.

METHODS AND MATERIALS

Fabrication of the EMX Specimens

Two hundred seventy-five ceramic blocks of EMX (8 mm × 8 mm × 3 mm thickness), shade LT A2, were fabricated in accordance with the manufacturer's instructions and detailed in a previous study.[4]

After divestment, the EMX blocks were wet polished with silicon-carbide abrasive papers (600-, 1200- and 2000-grit; Norton SA, São Paulo, SP, Brazil) in an automatic polisher (APL4; Arotec, Cotia, SP, Brazil) to obtain a flat and polished surface. Then, all EMX blocks were ultrasonically cleaned (MaxiClean 750; Unique, Indaiatuba, SP, Brasil) in deionized water for 20 minutes and dried with compressed air.

EMX Surface Treatments

The EMX blocks were randomly assigned into five groups (n=50) according to the HF concentrations: 1%, 2.5%, 5%, 7.5%, and 10% (Fórmula & Ação, São Paulo, SP, Brazil). A new random distribution was performed into five subgroups (n=10) according to the etching times: 20, 40, 60, 120, and 20 + 20 seconds. After etching, each EMX surface was rinsed with oil-free compressed air/water spray for 30 seconds. For the re-etched group (20 + 20 seconds), the EMX surface was rinsed as described previously and air dried before the second etching procedure, which focused on simulating the retreatment after clinical contamination of the etched surface.[18] After etching, all EMX blocks were ultrasonically cleaned (MaxiClean 750) in deionized water for 20 minutes and dried for 30 seconds.

One layer of a silane coupling agent (RelyX Ceramic Primer, 3M ESPE, St Paul, MN, USA) was applied to the entire etched EMX surface and left in contact for 60 seconds, followed by air heat drying for 60 seconds to accelerate the water/alcohol evaporation. Then, all EMX surfaces received a thin layer of photo-activated unfilled resin[4] (Scotchbond MultiPurpose, 3M ESPE). The excess was removed with a microbrush and dry air from a dental syringe for five seconds and light cured for 20 seconds using a light-emitting diode (LED) device (Valo Cordless, standard mode; Ultradent Inc, South Jordan, UT, USA) with an irradiance of 1000 mW/cm^2.

μSBS Testing Procedures

The setup design of the μSBS test has been previously described by Naves and others[18] and Sundfeld Neto and others[4] and is represented in Figure 1. Round molds (1-mm thick) containing three cylinder-shaped orifices (1 mm in diameter) were made with elastomer (Oranwash L, Zhermack, Badia Polesine, Italy) and placed onto the EMX surface for the delimitation of the bonding area. The cylindrical orifices were filled with resin cement (Variolink II, Shade Transparent; Ivoclar Vivadent). A mylar strip and a glass slab were placed over the filled mold, followed by a vertical load of 250 g applied for 2 minutes to standardize the height of the resin cement cylinders.[4] Afterward, both vertical load and glass slab were removed and the resin cement cylinders were light-cured for 40 seconds using an LED source with an irradiance of 1000 mW/cm^2 (Valo Cordless). All specimens were stored in deionized water for 24 hours at 37°C. Then, the elastomer mold was sectioned with a No. 11 scalpel blade and carefully removed. Each resin cement cylinder interface was analyzed with optical microscopy (Olympus Corp, Tokyo, Japan) at 40× magnification, and those presenting any flaws or defects were eliminated and replaced. Three cylinders were built on each EMX surface, totaling 30 cylinders for each group.

The μSBS test was performed in a universal mechanical testing machine (model 4411; Instron, Canton, MA, USA) using a thin steel wire (0.2 mm in diameter) at a crosshead speed of 1 mm/min until failure. A mounting jig was used for the parallel alignment of the ceramic-resin cylinder interface to the testing device. The steel wire was looped around each resin cement cylinder and aligned with the bonding interface. The fractured specimens were observed under optical microscopy (Olympus) at 40× magnification, and the mode of failure was classified as follows: adhesive (mode 1), cohesive within ceramic (mode 2), cohesive within resin cement (mode 3), and mixed, involving resin cement and ceramic (mode 4).

SEM Analysis

One representative etched EMX specimen of each group was submitted to SEM analysis to depict the etched surface morphology. The EMX blocks were mounted on aluminum stubs, sputter coated with gold (SCD 050; Balzers, Schaan, Liechtenstein) for 120 seconds at 40 mA, and examined by SEM (JSM 5600 LV; JEOL, Tokyo, Japan) with 2000× magnification and operated at 15 kV by the same operator.

Statistical Analysis

Values of μSBS were calculated and the data provided in megapascals. The average value of the three cylinders was recorded as the bond strength for each specimen. Thus, the mean bond strength values of each group represented the mean of the 10 specimens. Exploratory data analyses were performed prior to applying an analysis of variance (ANOVA). The data of μSBS were submitted to two-way ANOVA (HF concentration × etching time), and multiple comparisons were performed using the Tukey post hoc test (α=0.05).

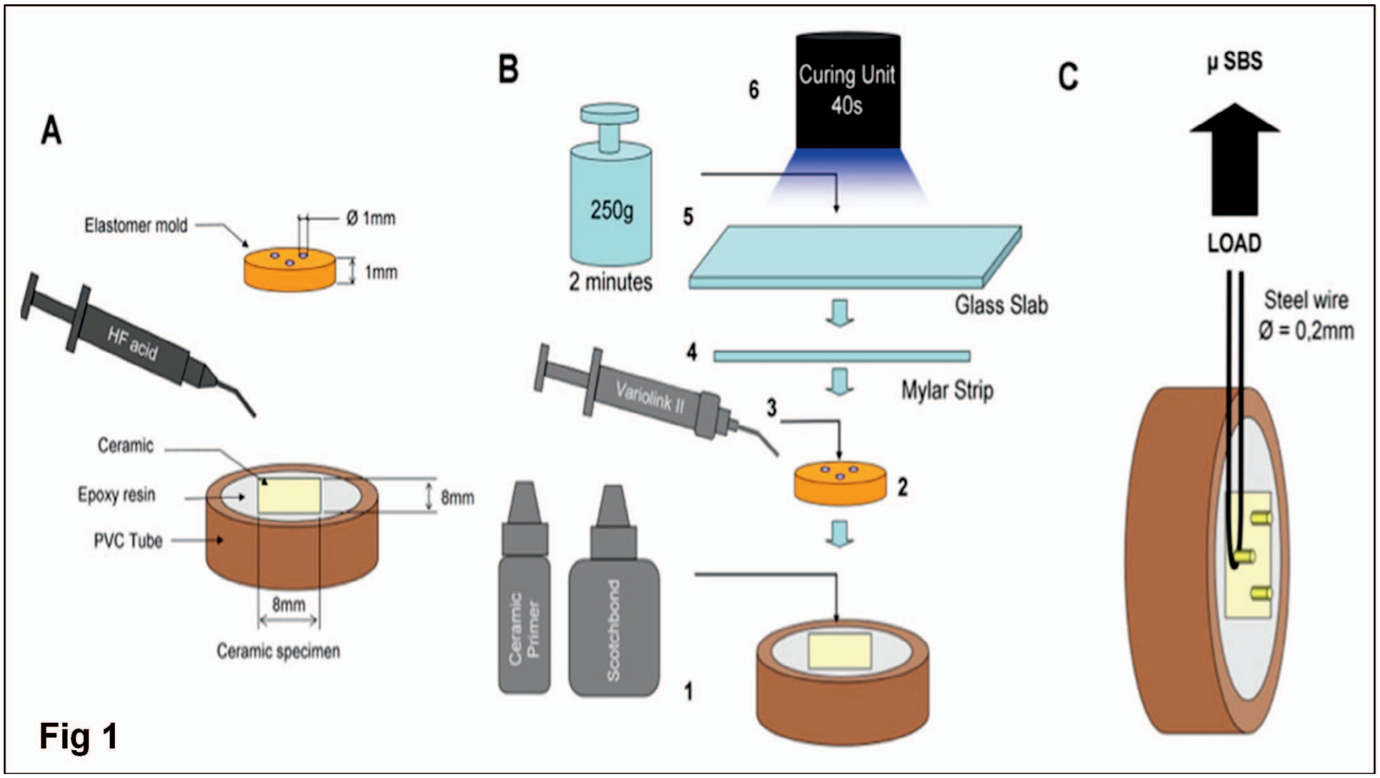

Figure 1. *Experimental design of the study. After etching (A), 1) silane was applied to the ceramic surface, followed by a thin layer of unfilled resin light-cured for 20 seconds; 2) an elastomer mold with cylinder-shaped orifices was positioned onto the surface; 3) orifices were filled with resin cement (Variolink II); 4) a mylar strip and glass slab were placed over the filled mold; 5) cementation load was applied for 2 minutes; 6) light curing of the resin cement was carried out for 40 seconds. (C) Microshear bond strength testing.*

RESULTS

µSBS

The mean µSBS values are shown in Table 1. The interaction between the HF concentrations and etching times ($p<0.01399$) was significant. The HF concentrations ($p<0.0001$) and etching times ($p<0.0001$) directly influenced the µSBS values.

HF concentrations of 5%, 7.5%, and 10% provided significantly higher µSBS values than 1% and 2.5% ($p<0.05$), regardless of the etching times. When the specimens were etched from 40 to 120 seconds, the µSBS for 1% and 2.5% HF concentrations was significantly higher than 20 seconds ($p<0.05$). For 2.5% HF concentrations, no statistical differences were found among the etching times of 40, 60, 120, and 20 + 20 seconds ($p>0.05$). No statistical difference was found among 5%, 7.5%, and 10% HF concentrations ($p>0.05$). When etching times were compared, no statistical differences were found among 5% and 10% HF concentrations ($p>0.05$).

Failure Analysis

A descriptive analysis of the failure modes is shown in Table 2. A predominance of adhesive failures (mode 1) was detected for 1% and 2.5% HF

Table 1: *Means of µSBS (MPa) (±SD) for All Groups*[a]

Hydrofluoric Acid Concentrations (%)	Etching Times (s)				
	20	40	60	120	20 + 20
1	10.2 (±2.1) cC	16.2 (±1.1) cB	14.1 (±1.9) cBC	22.0 (±1.1) bA	16.3 (±1.2) cB
2.5	16.8 (±2.6) bB	21.8 (±2.9) bA	24.2 (±1.2) bA	25.9 (±1.3) bA	22.0 (±1.8) bA
5	27.8 (±1.6) aA	28.4 (±1.2) aA	30.1 (±2.2) aA	30.9 (±1.4) aA	28.9 (±1.7) aA
7.5	28.1 (±1.6) aB	29.9 (±1.3) aAB	32.3 (±1.8) aAB	33.0 (±1.6) aA	30.2 (±2.8) aAB
10	31.1 (±1.7) aA	31.0 (±1.4) aA	32.9 (±2.2) aA	33.8 (±1.5) aA	30.7 (±1.3) aA

[a] *Means followed by different lowercase letters in each column and upper capital letters in each line differ statistically by Tukey's test at 5%.*

Table 2: *Distribution of Failure Modes Among Groups (%)*[a]

HF Concentrations × Etching Times	Failure Modes			
	Mode 1	Mode 2	Mode 3	Mode 4
1%				
20 s	61	0	0	39
40 s	72	0	0	28
60 s	78	0	0	22
120 s	69	0	0	31
20 + 20 s	72	0	0	28
2.5%				
20 s	83	0	0	17
40 s	58	0	0	42
60 s	56	0	0	44
120 s	64	0	0	36
20 + 20 s	75	0	0	25
5%				
20 s	48	0	0	52
40 s	51	0	0	49
60 s	36	0	0	64
120 s	36	0	3	61
20 + 20 s	36	0	0	64
7.5%				
20 s	44	0	0	56
40 s	56	0	0	44
60 s	47	0	0	53
120 s	42	0	0	58
20 + 20 s	50	0	0	50
10%				
20 s	42	0	0	58
40 s	50	0	0	50
60 s	47	0	0	53
120 s	47	0	3	50
20 + 20 s	56	0	0	44

[a] *Failure modes: adhesive (mode 1), cohesive within ceramic (mode 2), cohesive within resin cement (mode 3), and mixed, involving resin cement and ceramic (mode 4).*

concentrations, regardless of the etching times. Conversely, for 5%, 7.5%, and 10% HF concentrations, a slight predominance of mixed failures (mode 4) were verified, regardless of the etching times. No cohesive failures within resin cement or ceramic were recorded.

SEM Evaluation

The SEM images of the etched EMX surfaces are shown in Figures 2, 3, 4, 5, and 6. The HF concentrations and etching times directly influenced the etching morphology of EMX. Different etching patterns were observed for different etching periods for 1% HF concentration, with a slight increase of glassy matrix removal with higher etching times (Figures 2A-E). The same effect was verified for 2.5% HF (Figures 3A-E). For 5% HF concentration, the glassy matrix removal and the consequent exposure of lithium disilicate crystals were more marked as the etching times increased (Figures 4A-E). For 7.5% and 10% HF concentrations, different etching times seemed to not have affected the EMX etched morphology to the same extent as lower HF concentrations/etching times (Figs. 5 and 6). Re-etching (20 + 20 seconds) with 2.5% HF concentration showed a slight increase in the glassy matrix removal compared with the other re-etched groups.

DISCUSSION

The present study assessed the influence of several HF concentrations associated with different etching times on the surface morphology/μSBS of lithium disilicate glass ceramic to resin cement. Through SEM and μSBS analysis, a clear effect of HF concentrations and etching times was verified.

The results indicate that the first tested hypothesis was confirmed. The 5%, 7.5%, and 10% HF concentrations showed the highest μSBS values, regardless of the etching times. This was directly associated with a greater dissolution of the vitreous phase and exposure of lithium disilicate crystals (needle-like appearance; Figs. 4-6) than 1% and 2.5% HF concentrations (Figs. 2 and 3). These findings are in accordance with previous studies, which have reported higher μSBS values for HF concentrations ranging from 5% to 10% applied for 20 seconds on EMX at room temperature.[4,15] The etching reaction of HF on EMX is elucidated by the equation $4HF + SiO_2 \rightarrow SiF_4 + 2H_2O$ (1).[31] The selective glassy matrix removal occurs because the affinity of fluoride to silicon is greater than to oxygen, which makes possible the attack of ionized HF to silanol (silicon-oxygen bonds, SiO_2) presented in the glass ceramic.[31,32] Therefore, the higher the concentration of HF, the greater the glassy matrix removal owing to the higher content of ionized HF available to react with SiO_2.

Different etching times were also evaluated. The results confirmed the second hypothesis as well as higher etching times increased the conditioning ability of HF to EMX, especially for 1% and 2.5% HF concentrations. This was evidenced in the SEM images (Figs. 2 and 3; 1% and 2.5% HF, respectively), in which the etching times of 40, 60, 120, and 20 + 20 seconds promoted a gradual increase on the vitreous phase dissolution/bond strength compared with 20 seconds (Table 1). Probably, as HF stays in

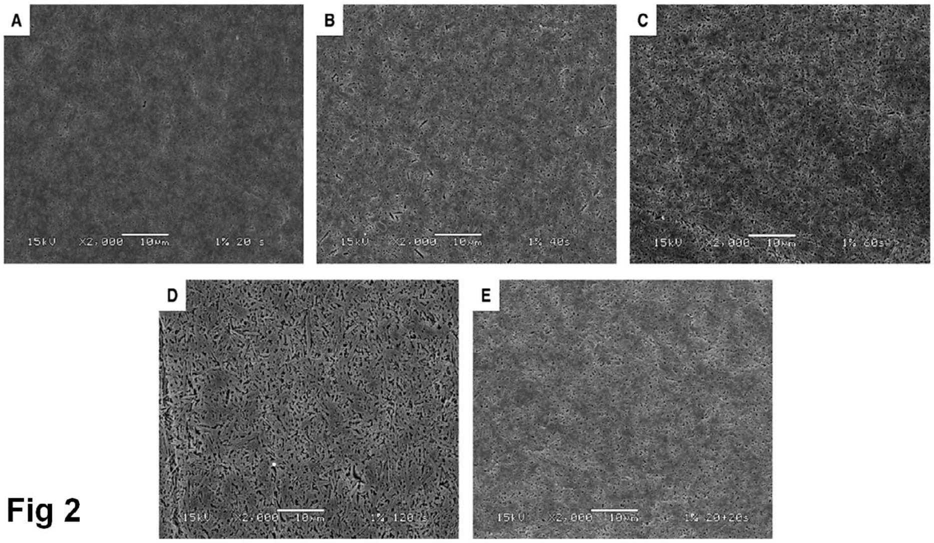

Fig 2

Figure 2. *SEM images (2000× magnification) of etched EMX surfaces made with 1% HF concentration. Different etching patterns were observed for different etching periods. Poor vitreous phase dissolution can be observed in (A) (1%, 20 seconds), (B) (1%, 40 seconds), (C) (1%, 60 seconds), and (E) (1%, 20 + 20 seconds), whereas (D) (1%, 120 seconds) shows slightly greater vitreous phase dissolution than all other etching times.*

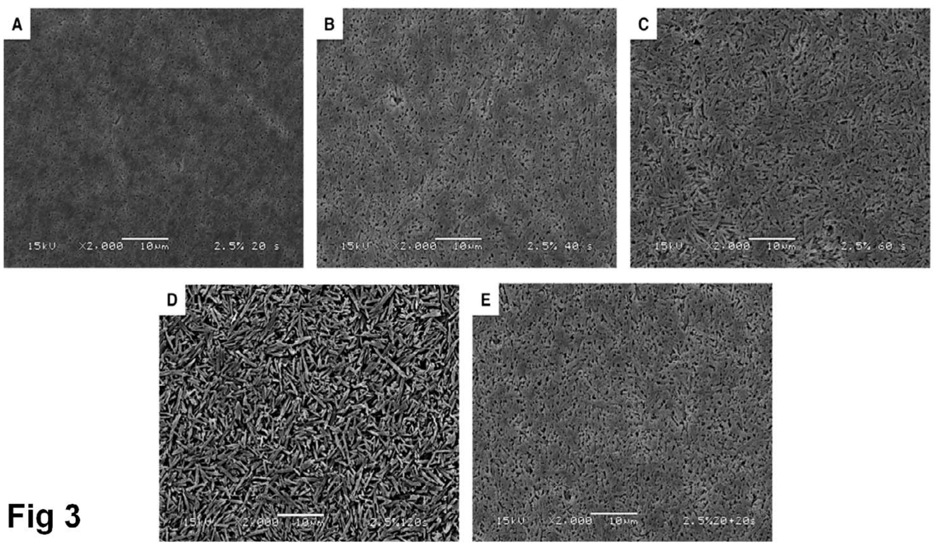

Fig 3

Figure 3. *SEM images (2000× magnification) of etched EMX surfaces made with 2.5% HF concentration. Different etching patterns were observed for different etching periods. (A) (2.5%, 20 seconds) and (B) (2.5%, 40 seconds) show poor vitreous phase dissolution, while (C) (2.5%, 60 seconds) shows partial vitreous phase dissolution, exposing lithium disilicate crystals. (D) (2.5%, 120 seconds) shows a greater vitreous phase dissolution/exposure of lithium disilicate crystals than all other etching times. (E) (2.5%, 20 + 20 seconds) shows a higher vitreous phase dissolution compared with 20 seconds only.*

contact for longer periods with the EMX surface, there will be more time available for ionized HF to react with silicon (glassy matrix), thus removing more vitreous phase and, consequently, creating more surface irregularities for the mechanical entanglement with the resin cement by the greater exposure of lithium disilicate crystals. HF etching with 2.5% for 120 seconds was the concentration below 5% that mostly resembled 5% HF applied for 20 seconds. Although the lowest concentration of HF is desired because of the hazardous nature of HF,[31] etching for 120 seconds is time-consuming, which is not efficient for the clinical office.

The results of the present study are not in agreement with those of previous reports, which have found decreased bond strength when a leucite-reinforced glass ceramic was etched with 10% HF concentration for more than 60 seconds[18] or for 90-180 seconds.[33] Such difference reaffirms that the etching effect of HF depends on the composition of the glass ceramic. Since EMX has a lower content of glassy matrix (±30 vol%) than leucite-reinforced glass ceramic (±70 vol%), a lower formation of silicon tetrafluoride (SiF_4) is expected (see reaction 1); SiF_4 is a crystalline residue deposited on the glass ceramic surface[18,30,34] that acts as a physical barrier and may hamper further HF etching.

The increased etching times did not lead to higher μSBS values for 5%, 7.5%, and 10% HF concentrations. It may be claimed that 1) higher HF concentrations do not need extended etching times, as the amount of ionized HF is enough to properly dissolve the glassy matrix of EMX for 20 seconds, and 2) the limited effect of HF concentrations is due

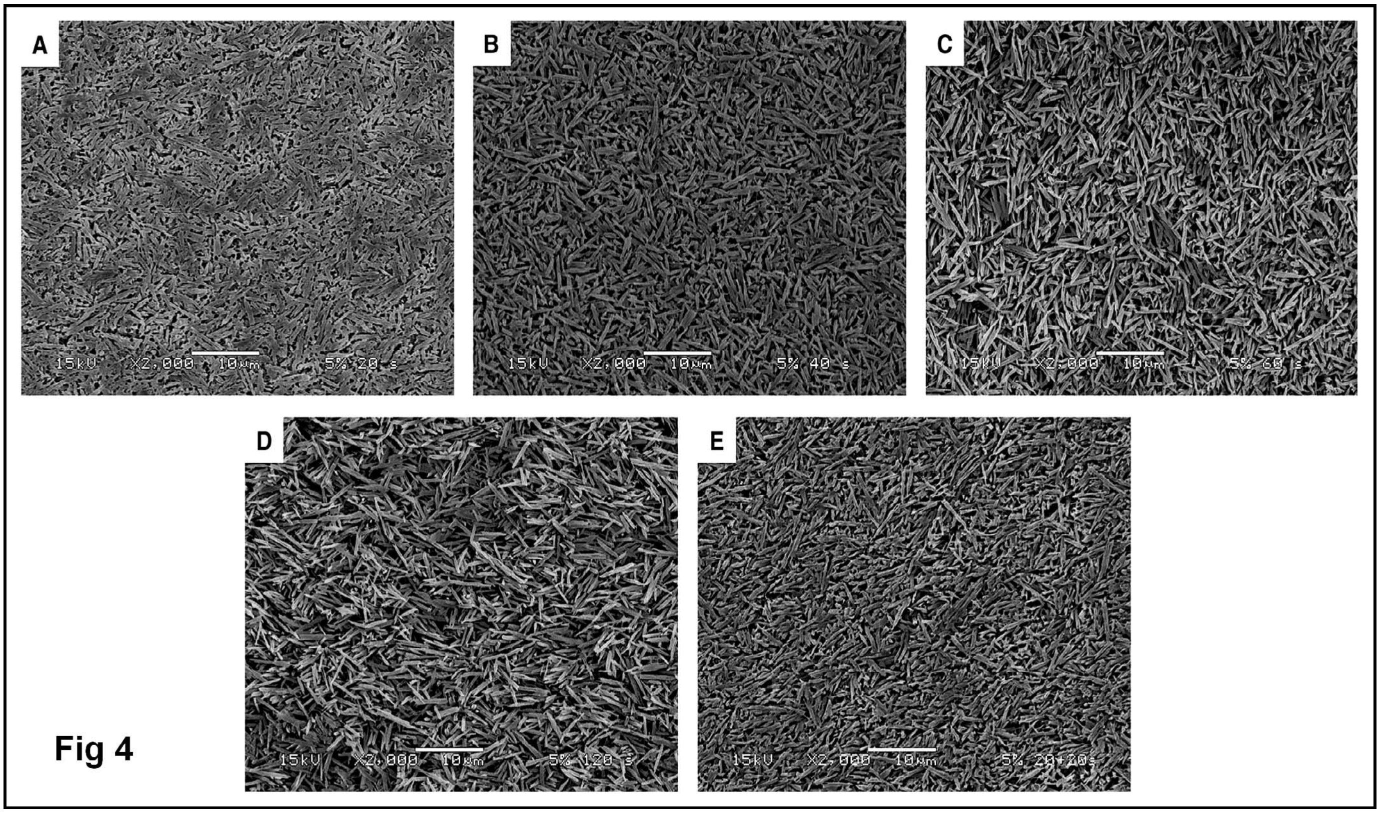

Figure 4. *SEM images (2000× magnification) of etched EMX surfaces made with 5% HF concentration. Different etching patterns can be observed for different etching periods. (B) (5%, 40 seconds), (C) (5%, 60 seconds), and (D) (5%, 120 seconds) show a gradual increase of the vitreous phase dissolution and the exposure of lithium disilicate crystals compared with (A) (5%, 20 seconds), which shows the lowest glassy matrix removal. (E) (5%, 20 + 20) shows a similar etching pattern to (B) (5%).*

to the combination of buffering and dilution of the etchant by the crystalline residue.[18] For 1% and 2.5% HF concentrations, the bond strength was higher when the etching period was extended, which might be explained from the reduced effect of SiF_4 (assumed for the higher content of vitreous phase seen on the EMX surface; Figs. 2 and 3) on the etching capability of HF. Thus, it may be hypothesized that 1% and 2.5% HF concentrations initially form a lower amount of SiF_4 because of the lower content of ionized HF available to react with the silicon. Hence, a lower initial physical barrier is created, which allows HF to continue reacting with the glassy matrix for increased etching times, leading to higher μSBS values.

It was expected that the re-etching would increase the aggressiveness and depth of the glassy matrix dissolution.[18] Furthermore, re-etching was also expected to possibly attenuate the effect of SiF_4, as it is soluble in water[35] and the air/water spray would have removed it from the etched surface before the further new conditioning. However, re-etching provided higher bond strength values for 1% and 2.5% only, which presented statistically similar bond strength values to 40 seconds. Thus, renewing the HF may be done in the case of accidental contaminations of the etched surface, but if the clinician seeks higher bond strength values, re-etching is not necessary.

A slight increase of mixed failures was observed for 5%, 7.5%, and 10% HF concentrations, irrespective of the etching times. The glassy matrix dissolution is higher and the penetration of the resin cement is adequate, although not complete.[4,30] On the other hand, adhesive failures were more frequent for the HF concentrations of 1% and 2.5%, which might point to poor bonding quality for lower HF concentrations, a situation also found by Sundfeld Neto and others.[4]

In summary, the μSBS results and SEM analysis have shown that different etching times and HF concentrations have significant effects on the surface morphology/μSBS of EMX. Adequate etching times and HF concentrations are crucial for optimal bonding properties and clinical performance/longevity. Dental professionals should take care during

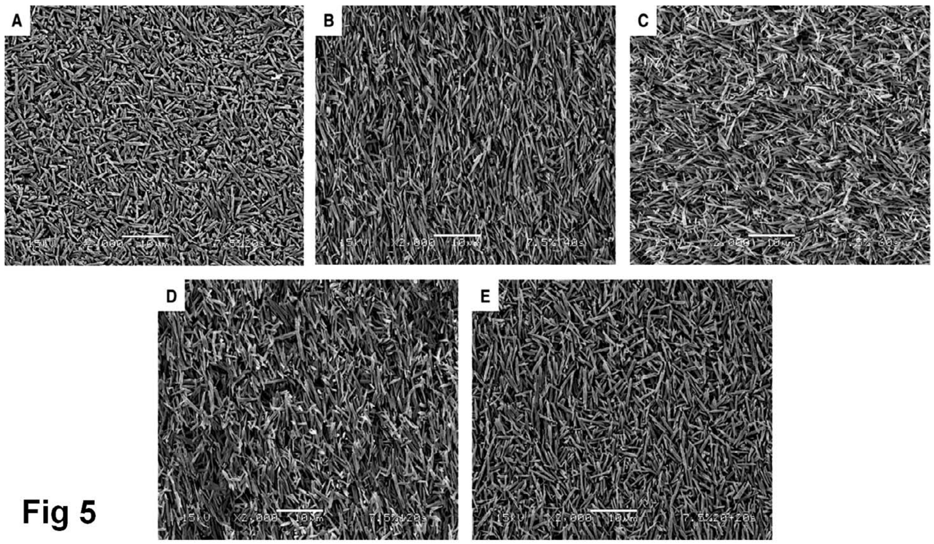
Fig 5

Figure 5. *SEM images (2000× magnification) of etched EMX surfaces made with 7.5% HF concentration. Similar etching patterns (glassy matrix removal and exposure of lithium disilicate crystals) can be observed, regardless of the etching times (A: 20 seconds, B: 40 seconds, C: 60 seconds, D: 120 seconds, and E: 20 + 20 seconds).*

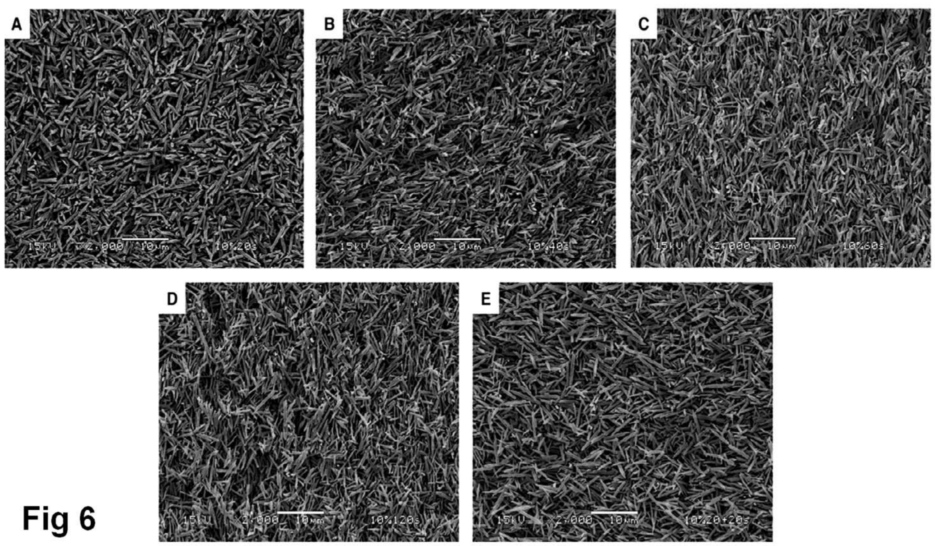
Fig 6

Figure 6. *SEM images (2000× magnification) of etched EMX surfaces made with 10% HF concentration. Similar etching patterns (glassy matrix removal and exposure of lithium disilicate crystals) can be observed, regardless of the etching times (A: 20 seconds, B: 40 seconds, C: 60 seconds, D: 120 seconds, and E: 20 + 20 seconds).*

bonding procedures because HF is a hazardous etchant.[31] Future studies should be carried out to investigate other possible factors, such as viscosities of the resin cement, thermal cycling, and fatigue that may affect the performance of bonded ceramic restorations.

CONCLUSIONS

The effect of HF on the bonding characteristics of lithium disilicate glass ceramic was concentration/time dependent. As the concentration of HF decreased, the etching time had to be increased for greater bond strength. The adequate surface treatment for lithium disilicate glass ceramic was achieved with the HF concentration of 5% applied for 20 seconds, therefore attesting that it is not necessary to use higher concentrations of HF and/or increased etching times.

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Conflict of Interest

The authors certify that they 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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