

# Effect of hydrofluoric acid concentration and etching time on resin-bond strength to different glass ceramics

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**Abstract:** The objective of this study was to evaluate the effect of the hydrofluoric acid (HF) concentration and time of acid conditioning on bond strength of three glass ceramics to a resin cement. Thus, fifty blocks (10 mm x 5 mm x 2 mm) of each ceramic (LDCAD: IPS e.max CAD; LCAD: IPS Empress CAD and LDHP: IPS e.max Press) were made and embedded in acrylic resin. The surfaces were polished with sandpaper (#600, 800, 1000, and 1200 grits) and blocks were randomly divided into 15 groups (n = 10) according to the following factors: Concentration of HF (10% and 5%), conditioning time (20 s and 60 s) and ceramic (LDCAD, LDHP, and L). After conditioning, silane (Prosil / FGM) was applied and after 2 min, cylinders (Ø = 2 mm; h = 2 mm) of dual resin cement (AllCem / FGM) were made in the center of each block using a Teflon strip as matrix and light cured for 40 s (1,200 mW/cm<sup>2</sup>). Then, the samples were thermocycled (10,000 cycles, 5/55°C, 30s) and submitted to the shear bond test (50 KgF, 0.5 mm/min). The data (MPa) were analyzed with 3-way ANOVA and Tukey's test (5%). Failure analysis was performed using a stereomicroscope (20x) and a scanning electron microscope (SEM). ANOVA revealed that the "concentration" factor (p = 0.01) and the interaction "acid concentration X ceramic" (p = 0.009) had a significant effect, however, the "ceramic" (p = 0.897) and "conditioning time" (p = 0.260) factors did not influence the results. The LDHP10%<sub>60s</sub> (10.98 MPa)<sup>aA\*</sup> group presented significantly higher bond strength than LDHP10%<sub>20s</sub> (6.57 MPa)<sup>bA</sup>, LCAD5%<sub>20s</sub> (6,90 ± 3,5)<sup>aB</sup> and LDHP5%<sub>60s</sub> (5.66 ± 2,9MPa)<sup>aA\*</sup> groups (Tukey). Failure analysis revealed that 100% of specimens had mixed failure. In conclusion, etching with 5% HF for 20 seconds is recommended for lithium disilicate and leucite-reinforced CAD/CAM ceramics. However, for pressed lithium disilicate ceramic, 10% HF for 60 s showed significantly higher bond strength to resin cement.

**Keywords:** Ceramics; Acid Etching, Dental; Shear Strength.

## Introduction

Due to its excellent aesthetic properties, wear and fracture resistance, biocompatibility, and low thermal conductivity,<sup>1,2,3,4,5</sup> glass ceramics have been used in several clinical situations, such as for the manufacture of indirect restorations,<sup>6</sup> including inlays and onlays,<sup>2,7</sup> veneers,<sup>5</sup> anterior



crowns,<sup>2,5</sup> and fixed partial dentures.<sup>2,8</sup> Several glass ceramics are currently available, including feldspathic ceramics, leucite-reinforced ceramics, lithium disilicate, lithium silicate reinforced with zirconia, and polymer-infiltrated ceramics.<sup>4,5</sup> Lithium disilicate ceramics have become popular because they offer good aesthetics and greater resistance to crack propagation when compared to leucite-reinforced ceramics.<sup>7</sup> When used in the CAD / CAM system, these ceramics are versatile and a structurally dense material, reducing the risk of defects and failures such as fractures and delamination,<sup>1,2,3,9</sup> and increasing the ceramic reliability.

Several studies have evaluated the clinical longevity of feldspathic and lithium disilicate ceramics restorations and observed a longevity rate of up to 100% in 2 years for monolithic crowns<sup>10</sup> and more than 94% for partial restorations in 3 years.<sup>11,12</sup> However, failures of ceramic materials have been reported in the literature, especially fractures, loss of retention and detachment, hypersensitivity, and caries in the adhesive interface.<sup>12</sup>

Prior to cementation of a glass ceramic restoration with a resin cement, the ceramic surface is treated to increase its adhesion to the cement. The procedure involves acid conditioning using hydrofluoric acid (HF) and the application of a silane primer.<sup>13</sup> This matrix is selectively dissolved and its crystal microstructures are exposed.<sup>14,15</sup> resulting in a rough ceramic surface that provides increased micromechanical retention with the resin cement.<sup>14,16</sup> In addition, the increased roughness increases the surface energy and the interaction between the bonding agent and silane,<sup>15,17</sup> thus promoting a chemical-mechanical adhesion at the ceramic / silane / cement interface.

While HF increases the bond strength of the cement to the ceramic, acid conditioning can also decrease the mechanical resistance of the material, depending on the acid concentration and conditioning time.<sup>17</sup> These factors may also alter the bond strength between resin cement and glass ceramics.<sup>15,16,17,18,19,20,21</sup> As an example, the exposure of lithium disilicate to HF for more than 20 seconds may lead to the weakening of its structure.<sup>17,18,19</sup> As for feldspathic ceramics, an acid conditioning time ranging from

1 to 2 minutes is recommended.<sup>16,20,21</sup> Additionally, restorations with disilicate ceramic restorations can be fabricated by the press technique, where ceramic ingots are melted and injected into the mold in a specific furnace. Differently, through the CAD / CAM technique, the partially sintered block of disilicate is milled and its final resistance only occurs after its complete sintering.<sup>22</sup>

There is a lack of consensus in the literature concerning the influence of the hydrofluoric acid concentration and etching time on the resin bond strength to lithium disilicate ceramics produced by the different techniques. Thus, the objective of the present study was to evaluate the influence of HF concentration (5% and 10%), conditioning time (20 or 60s), and ceramic type (leucite reinforced feldspathic glass and lithium disilicate) on the bond strength to a resin cement. The hypotheses tested were: a. the ceramic type influences the results, regardless the HF concentration and time of exposure, b. different concentrations of HF affect the bond strength values, and c. the conditioning time influences the bond strength values.

## Methodology

The commercial name, manufacturers, chemical composition, and batch number of the materials used in this study are listed in Table 1.

### Production of specimens

Blocks were made with three glass ceramics: CAD / CAM lithium disilicate (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein), pressed lithium disilicate (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) and leucite-reinforced feldspathic ceramic (IPS Empress CAD, Ivoclar Vivadent, Schaan, Liechtenstein). The press and sintering process (Programat EP5000, Ivoclar Vivadent AG) of the press lithium disilicate was performed according to the manufacturer's recommendations.

The ceramic blocks were cut into rectangular slices using a diamond disk (Microdont, São Paulo, Brazil, 34.570), mounted on a straight micro-motor handpiece (LB100 Beltec, São Paulo, Brazil) under air/water cooling, resulting in 50 samples for

each type of ceramic (10 mm x 5 mm x 2 mm). The dimensions were verified with digital caliper (Eccofer, Curitiba, Brazil) and surfaces were regularized with sand paper (600 grit). After surface regularization, the milled lithium disilicate blocks were sintered according to the manufacturer's recommendation.

### Embedding samples

The 150 blocks were embedded in chemically activated acrylic resin (JET, Dental Articles Classic,

Brazil) using a silicone mold (Master-Talmax silicone, Curitiba, Brazil). After resin polymerization, the surfaces of ceramic blocks were polished with sandpaper of increasing grit (#600, 800, 1000, and 1200) in a polishing machine (Labpol 8-12, Extec, USA) until the resin and ceramic surfaces were leveled. Then, the samples were randomly divided into fifteen groups (n = 10), according to the factors: "HF concentration (5 and 10%)", "conditioning time (20 and 60s)", and "ceramic type" as presented in Table 2.

**Table 1.** Commercial name, manufacturers, material, chemical composition, batch number of materials used in this study.

Commercial name	Manufacturer	Material	Composition	Batch
IPS EMAX CAD	Ivoclar Vivadent.			
CAD/CAM Lithium disilicate-based ceramic (LDCAD)	Schaan, Liechtenstein	Lithium disilicate	SiO <sub>2</sub> . Li <sub>2</sub> O. K <sub>2</sub> O. MgO. Al <sub>2</sub> O <sub>3</sub> . P <sub>2</sub> O <sub>5</sub>	T38577
IPS Emax Press		Lithium disilicate	SiO <sub>2</sub> . Li <sub>2</sub> O. K <sub>2</sub> O. MgO. ZnO. Al <sub>2</sub> O <sub>3</sub> . P <sub>2</sub> O <sub>5</sub>	N/A
IPS Emress CAD		Leucite reinforced feldspathic	SiO <sub>2</sub> . Al <sub>2</sub> O <sub>3</sub> . K <sub>2</sub> O. Na <sub>2</sub> O	T28994
Condac porcelain 10%		Hydrofluoric acid 10%	10% hydrofluoric acid 10%, water, thickener, surfactant and dye	120815
Condac porcelain 5%		Hydrofluoric acid 5%	5% hydrofluoric, water, thickener, surfactant and dye	50815
Prosil	FGM, Joinville, SC, Brazil	Silane	3- Methacryloxypropyltrimethoxysilane, ethanol, water	40815
Allcem		Dual resin cement	Bis-GMA. Bis-EMA. TEGDMA. Co-initiators, Initiators (camphorquinone and dibenzoyl peroxide), stabilizers, barium-silicate glass microparticles, and silicon dioxide nanoparticles	200815

**Table 2.** Characteristics of study groups.

Group name	Ceramic	HF concentration (%)	Conditioning time (seconds)
LDCAD5% <sub>20s</sub>	CAD/CAM lithium disilicate-based ceramic	5	20
LDCAD5% <sub>60s</sub>			60
LDCAD10% <sub>20s</sub>		10	20
LDCAD10% <sub>60s</sub>			60
LDHP5% <sub>20s</sub>	Heat pressed lithium disilicate-based ceramic	5	20
LDHP5% <sub>60s</sub>			60
LDHP10% <sub>20s</sub>		10	20
LDHP10% <sub>60s</sub>			60
LCAD5% <sub>20s</sub>	CAD/CAM leucite-based ceramic	5	20
LCAD5% <sub>60s</sub>			60
LCAD10% <sub>20s</sub>		10	20
LCAD10% <sub>60s</sub>			60

## Surface treatments

Prior to surface treatment procedures, all ceramic blocks were immersed in distilled water and ultrasonically cleaned for 5 min (Cristófoli Equipamentos de Biossegurança LTDA, Campo Mourão, Brazil). The blocks were kept on a piece of gauze (~ 10 min) until complete water evaporation.

The HF was applied to the surfaces of groups as shown in Table 2. The area to receive the acid was delimited by an adhesive tape with a 3-mm perforation. HF (5 or 10%, FGM, Joinville, Brazil) was actively applied with a microbrush (Dentsply, New York, USA) for 20 or 60 seconds, washed with air / water spray for 30 seconds, and dried with a jet of air for 30 seconds. Afterwards, a layer of silane agent (Prosil, FGM; Joinville, Brazil) was applied to all samples with a microbrush (Dentsply, New York, USA) and left for 2 minutes for solvent evaporation according to the manufacturer's recommendations.

## Manufacture of resin cement cylinders

After acid conditioning, a cylinder of resin cement (AllCem Dual, FGM; Joinville, Brazil) was made on the ceramic surfaces. A Teflon matrix ( $\varnothing = 2$  mm and  $h = 2$  mm) (Ultradent Jig, Ultradent, South Jordan, USA) was used to standardize the adhesive area and height of the cylinder. After fitting the matrix to the surfaces, the base and catalyst pastes of the resin cement were mixed and added to the matrix, light cured for 20 s (1,200 mW / cm<sup>2</sup> - Rádi Cal, SDI, Australia), and left for a 5-minute chemical curing as recommended by the manufacturer. The matrices were removed and the sets (block + resin cement cylinder) were submitted to thermocycling.

## Thermocycling and shear bond strength testing

The thermocycling protocol consisted of 10,000 cycles of alternate 30-s baths at 5°C and 55°C, with a 5-s interval between immersions using a thermocycler (Nova Etica, São Paulo, Brazil, 10.000TC). Then, samples were submitted to a shear bond strength test (50 KgF, 0.5mm / min) in a universal testing machine (DL-1000, EMIC, São José dos Campos, Brazil). Specimens had the cement / ceramic interface hold perpendicularly

to the horizontal plane by a device. The load was applied at the base of the cylinder on the adhesive interface, using an orthodontic wire (0.2 mm diameter) at a speed of 0.5 mm/min and load cell of 50 KgF until fracture of the specimen.

The calculation of the bond strength was performed by the formula:  $R = F / A$ , where R = adhesive strength (MPa); F = force (N); A = interfacial area (mm). The adhesive area of each ceramic block was defined by the area of a circle using the formula  $A = \pi r^2$ , where  $\pi = 3.14$  and  $r = 1$  mm (radius of the cylinder), resulting in a cross-sectional area of 3.14 mm<sup>2</sup>.

## Failure analysis

The fractured surfaces of the specimens were examined using an optical stereomicroscope (Stereo Discovery V20, Zeiss, Göttingen, Germany) and representative failure modes were analyzed by Scanning Electron Microscopy (SEM, Inspect S50, FEI, Czech Republic). For SEM observations, the specimens were gold-sputtered for 80 seconds at 40 mA, creating a 30 nm thick layer. The failure modes were classified as: a) adhesive at the ceramic / cement interface (ADHES cer / cem); b) cohesive in ceramic (COHES cer); c) cohesive in cement (COHES cem); and D) mixed adhesive/cohesive failure (adhesive failure at the ceramic/cement interface + predominantly cohesive failure in cement or in ceramic).<sup>23</sup>

## Surface topography analysis (SEM)

Four samples of each group were examined by SEM (Hitachi TM 3000, Tokyo, Japan) with a magnification of 1,500 X to observe how the surfaces of the ceramics behaved after different HF concentrations and times of exposure.

## Statistical analysis

Analysis of variance (3-way ANOVA) and Tukey's test (5%) were used to compare data from the groups. The normality of the data was verified by the Kolmogorov-Smirnov ( $p > 0.05$ ), Kuiper ( $p > 0.10$ ), and Lilliefors ( $p > 0.05$ ) tests. The computer program Assistat 7.7 PT (Campina Grande, Brazil) was used. Using the OpenEpi website, a power of 96.96% was calculated using a two-sided 95% confidence interval.

## Results

### Shear bond strength

Three-way ANOVA revealed that the factor “acid concentration” ( $p = 0.01$ ) and the interaction “acid concentration X ceramic” ( $p = 0.009$ ) were significant. However, the “ceramic” ( $p = 0.897$ ) and “conditioning time” ( $p = 0.260$ ) factors did not influence the results (Table 3).

When all experimental groups were compared, the use of 10% HF for 60 s resulted in significantly higher bond strength values for the pressed lithium disilicate ceramic (LDHP10%<sub>60s</sub> = 10.98 MPa)<sup>aA\*</sup> that was significantly higher than LDHP10%<sub>20s</sub> (6.57 MPa)<sup>bA</sup>, LCAD5%<sub>20s</sub> (6.90±3.5)<sup>aB</sup> and LDHP5%<sub>60s</sub> (5.66 MPa)<sup>aA\*</sup> groups. The LDCAD10%<sub>20s</sub> (8.78 ±3.6)<sup>aA\*</sup> had significantly higher SBS than the LDCAD5%<sub>20s</sub> (5.30 ±2.9)<sup>aA</sup>. The means (± SD) of shear bond strength

and the comparison between the experimental groups are shown in Table 4.

### Failure analysis

Failure analysis revealed that all samples had mixed failures: predominantly cohesive in cement and adhesive at cement/ceramic interface (Figure 1A) or predominantly cohesive in ceramic and adhesive at cement/ceramic interface (Figure 1B)

### SEM analysis

Samples conditioned with 5% HF for 20 s presented less porous topographies compared to the other groups. For the CAD / CAM lithium disilicate ceramics, groups conditioned with 5% HF for 60 s and 10% HF for 20 and 60 s presented homogeneous pore topography. In contrast, both pressed lithium disilicate and leucite-reinforced feldspathic ceramics

**Table 3.** 3-way ANOVA results for shear bond strength.

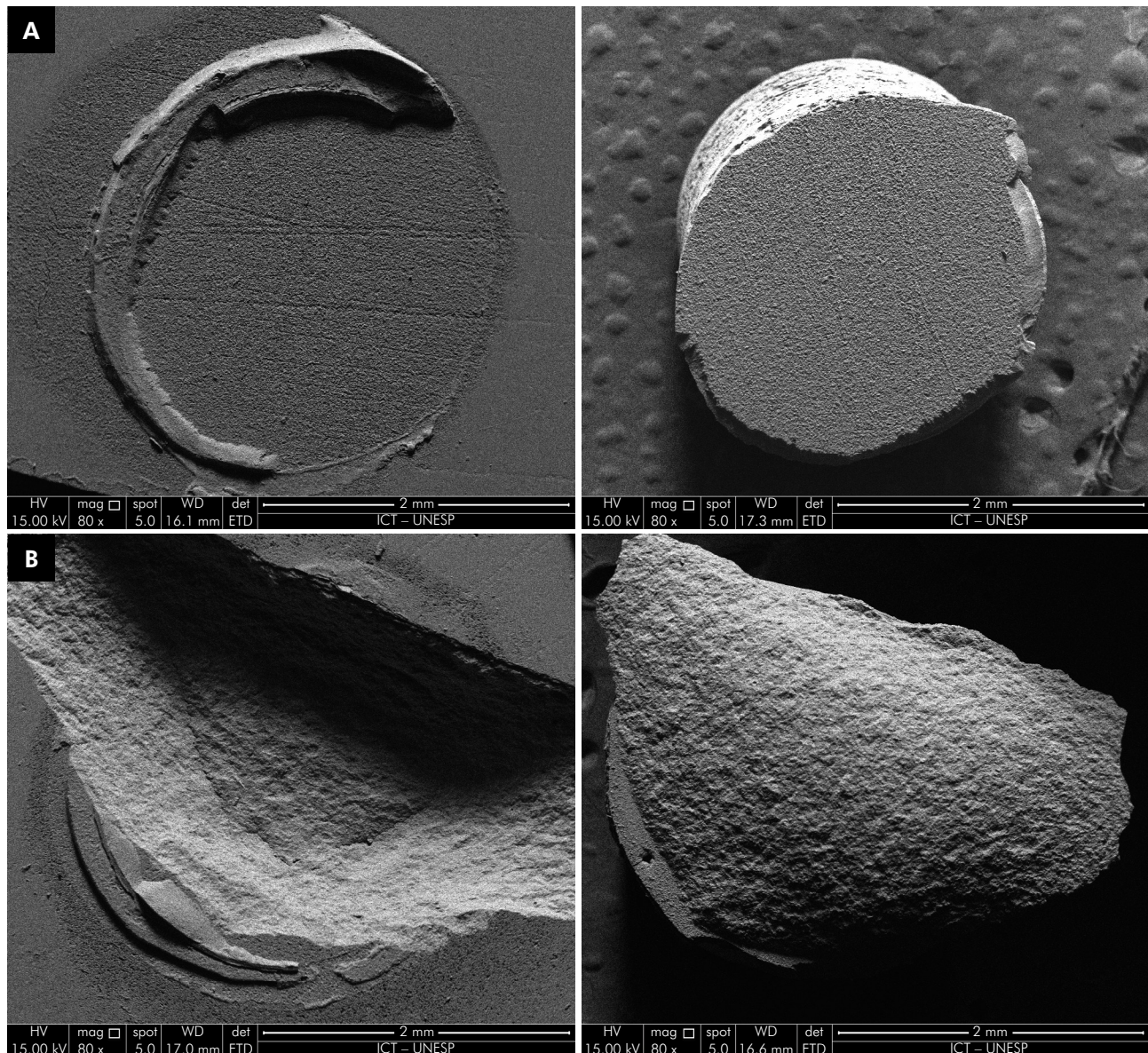
Source	GL	TSS	MSS	F	p-value
Acid concentration	1	6.805.614	6.805.614	58.999	0.0168*
Conditioning time	1	1.479.114	1.479.114	12.823	0.2598
Ceramic	2	250.393	125.197	0.1085	0.8973
Acid concentration X conditioning time	1	454.352	454.352	0.3939	0.5314
Acid concentration X ceramic	2	8.010.881	4.005.441	34.724	0.0345*
Conditioning time X ceramic	2	6.370.656	3.185.328	27.614	0.0676
Acid Concentration X conditioning time X ceramic	2	7.045.778	3.185.328	30.540	0.0513
Error	108	124.580.059	1.153.519		
Total	119	154.996.848			

\*Statistical significance ( $p < 0.05$ ); DF = Degrees of freedom; TSS = Total sum of squares; MSS = mean sum of squares; F = F statistics.

**Table 4.** Shear bond strength (means ± SD) values (MPa) of ceramics submitted to different hydrofluoric acid (HF) concentrations and exposure times.

Acid concentration	Conditioning time	Ceramic		
		LCAD	LDHP	LDCAD
10%	20 seconds	7.17 ± 2.9 aA	6.57 ± 1.6 bA	8.78 ± 3.6 aA*
	60 seconds	6.90 ± 3.5 aB	10.9 ± 4.1 aA*	7.92 ± 4.3 aB
5%	20 seconds	8.66 ± 5.1 aA	5.21 ± 1.6 aA*	5.30 ± 2.9 aA*
	60 seconds	6.66 ± 3.5 aA	5.66 ± 2.9 aA	7.79 ± 2.9 aA

HF: Hydrofluoric acid. Different upper case letters indicate statistically significant differences between different ceramics for the same HF exposure times and concentrations ( $p < 0.05$ ); Different lowercase letters indicate statistically significant differences between the same HF concentration and ceramic submitted to different conditioning times ( $p < 0.05$ ); \*indicates statistically significant difference between the same ceramic and acid exposure time at different HF concentrations.



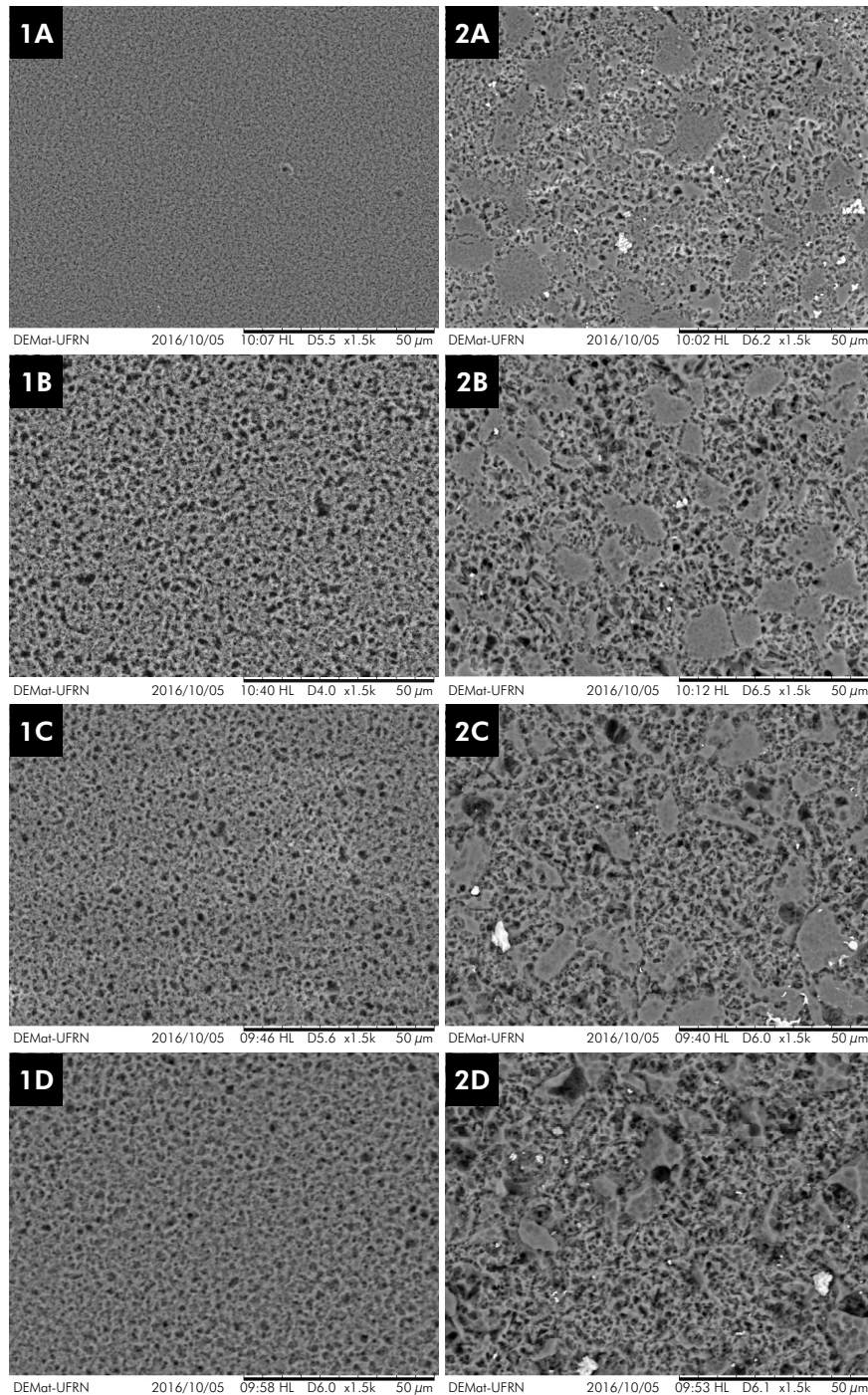
**Figure 1.** (A) MEV micrographs (80 x) of failure modes of cement and resin cement cylinders: cohesive failure in cement and adhesive failure at the cement/ceramic interface. Ceramics (\*), resin cement (#). (B) MEV micrographs (80 x) of failure modes of cement and resin cement cylinders: predominant cohesive failures in ceramic and adhesive at the cement/ceramic interface. Ceramics (\*), resin cement (#).

had greater superficial porosity in the 10% HF for 60s group. Representative SEM images are presented in Figures 2 (1A to 3D).

## Discussion

The objective of this study was to evaluate the influence of different HF concentrations and

exposure times on the bond strength of three ceramic systems to the resin cement. In this study, it was used the shear bond strength test, which is an easy methodology, has low cost, and is widely used in adhesion studies.<sup>24</sup> Other assays may also be used, such as micro-tension and micro-shear, which have a small adhesive area, resulting in few internal defects in the adhesive zone and few superficial failure.<sup>25</sup>



**Figure 2.** (1A) MEV micrographs (1,500x) of surface treatments of the ceramics IPS e.max CAD; A- 5% HF for 20s. (1B) MEV micrographs (1,500x) of surface treatments of the ceramic IPS e.max CAD - 5% HF for 60s. (1C) MEV micrographs (1,500x) of surface treatments of the ceramic IPS e.max CAD - 10% HF for 20s. (1D) MEV micrographs (1,500x) of surface treatments of the ceramic IPS e.max CAD - 10% HF for 60s. (2A) MEV micrographs (1,500x) of surface treatments of the ceramic IPS e.max Press A- 5% HF for 20s. (2B) MEV micrographs (1,500x) of surface treatments of the ceramic IPS e.max Press: 5% HF for 60s. (2C) MEV micrographs (1,500x) of surface treatments of the ceramic IPS e.max Press: 10% HF for 20s. (2D) MEV micrographs (1,500x) of surface treatments of the ceramic IPS e.max Press: 10% HF for 60s. (3A) MEV micrographs (1,500x) of the surface treatments of the ceramic Empress CAD A- 5% HF for 20s. (3B) MEV micrographs (1,500x) of surface treatments of the ceramic Empress CAD: 5% HF for 60s. (3C) MEV micrographs (1,500x) of surface treatments of the ceramics Empress CAD: 10% HF for 20s. (3D) MEV micrographs (1,500 x) of surface treatments of the ceramics Empress CAD: 10% HF for 60s.

However, procedures associated with ceramic cutting in the micro-tension test can induce early interface failure, reducing the effectiveness of the assay.<sup>26</sup> Some authors have reported that shear stress is the most prevalent stress on the cement layer, justifying the use of this in vitro test that evaluate this.<sup>27</sup> Thus, to overcome this limitations, we used a smaller adhesive area (2 mm<sup>2</sup>), which gives more reliable results<sup>4</sup>. In addition, an orthodontic wire was used for the test with the aim of exerting a more uniform shear force on the adhesive interface.<sup>28</sup>

According to our results, the first hypothesis that different ceramic types significantly affects bond strength with varying HF concentrations and times of exposure was partially accepted. It was tested three ceramics, two of which were lithium disilicate-based glass ceramics, differing only in the processing method, CAD / CAM and pressing, and a leucite-reinforced feldspathic glass (IPS Empress CAD). Using the same conditioning time and concentration of HF, no difference was observed between ceramics.<sup>29</sup> However, pressed disilicate had the highest bond strength values when higher concentrations of HF were used for a longer time (LDHP10%<sub>60s</sub>). Although the chemical compositions of the pressed and the CAD / CAM disilicate are the same, the pressed disilicate (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein), which is partially crystallized at its manufacturing, showed greater susceptibility to surface treatment, since it presented the highest values of adhesive strength. This may be related to the press process after the ceramic is melted, which can create a surface with more irregularities and consequently provide a greater surface contact and mechanical bonding to the resin cement.<sup>29</sup>

The second hypothesis that different concentrations of HF effect the bond strength was partially accepted, as only the pressed disilicate (LDHP) had a significant increase in bond resistance with the use of 10% HF for 60s. Sundfeld et al.<sup>30</sup> who also evaluated 5 and 10% HF in pressed disilicate, observed that 5% HF promoted a lower bond strength compared to the 10% conditioning. The authors state that the 10% HF removes a larger amount of the glass matrix and exposes more lithium disilicate crystals compared to the 5% HF because it has a higher amount of ionized

HF available. By containing half of the ionized HF, the 5% HF did not remove enough glass matrix to produce a suitable micromechanical bond between the ceramic and the resin cement,<sup>30</sup> which may explain the results of our study. Sundfeld Neto et al.<sup>31</sup> also investigated different HF concentrations and observed that higher concentrations provided higher bond strengths between disilicate ceramic and resin cement, but for leucite-reinforced ceramics the variation did not yield significant differences, corroborating our findings. In our study, for leucite-reinforced and lithium disilicate (CAD / CAM) ceramics, the variation of HF concentration and exposure time did not significantly interfere in the bond strength, similar to previous similar studies.<sup>25,31,32</sup>

The third hypothesis that time of acid conditioning affects the bond strength values was also partially accepted. Some authors have correlated higher surface roughness of glass ceramics to the prolonged acid conditioning time.<sup>19,31,33</sup> In our study, SEM images showed different ceramic surface topographies, with considerably higher number of irregularities in the conditioned groups compared to the untreated group. The pressed disilicate exposed to 10% HF for 60 s resulted in a more porous and rough surface and higher adhesion values compared to the 20 s, as seen in SEM images. Zogheib et al.<sup>19</sup> demonstrated that if HF conditioning time exceeded 20 seconds (60, 90, or 180 s), ceramic degradation and weakening may occur. According to the authors, the increase in exposure time to HF affects surface roughness proportionally, causing larger and deeper grooves on the surface, observed in SEM micrographs,<sup>19</sup> and at the same time reducing the flexural strength of lithium disilicate. Ramakrishnaiah et al.<sup>33</sup> evaluated the effect of different HF conditioning times on the surface characteristics of glass ceramics finding changes in porosity and roughness pattern, and concluded that longer exposures resulted in wider and irregular grooves, increasing surface roughness.<sup>25</sup>

The leucite-reinforced feldspathic ceramic presents a lower percentage of the crystal phase compared to the lithium disilicate ceramics (35.55 ± 5 and 70 ± 5%, respectively)<sup>7,8</sup> and thus, disilicate is less friable and presents higher surface resistance even



after conditioning, due to less crack propagation<sup>34</sup>. The manufacturer recommends a 20-s conditioning with 10% HF for disilicate, regardless of the production process used.<sup>35</sup> However, in our study it was observed that for pressed disilicate, a more aggressive conditioning protocol (longer time and higher concentration) yields greater bond strength to the cement, and these findings are supported by previous similar studies.<sup>29</sup> Although it has been reported in the literature that an increase in acid exposure time and concentration may decrease the flexural strength of ceramics leading to more cohesive failures in the ceramic, this generally applies to very long exposure times (90s and 120s) that can cause visible changes in the surfaces resulting in a significant dissolution of the ceramic network, weakening its structure.<sup>36</sup> The deep dissolution caused by HF was measured by a recent study,<sup>37</sup> which observed that HF etching dissolves glass material not only superficially, but also internally and in an uneven manner. In addition, the use of 10% HF for 60 seconds can produce greater and deeper dissolution (600 µm for lithium disilicate and 400 µm for leucite based ceramic), increasing the crating of defects and consequently the crack propagation risk. Thus, the present protocol increased the bond strength for lithium disilicate pressed ceramic, but less aggressive protocols such as 5% HF for 20 s can be used for a suitable adhesion for CAD/CAM lithium disilicate and leucite-based ceramics.

In our study, 100% of the samples had mixed failures, with predominance of cohesive failure in cement or ceramic and adhesive at cement/ceramic interface (Figure 1B-C). According to Melo et al.<sup>37</sup>

several factors can be related to this finding. The larger volume of crystal phase of lithium disilicate ceramics results in less cohesive failures compared to leucite-reinforced ceramics that have a lower volume of the crystal phase and higher glass phase, which may contribute to the higher number of mixed failures in ceramic found in our study. Problems associated with cohesive failures of ceramics during a shear test have been reported, and may be associated with the failures found in our study. However, this type of failure was not regular in all groups, confirming the assumptions cited above.

Additional studies evaluating the effect of oral conditions such as pH variations and masticatory load, as well as depth of acid degradation on the ceramic surface, should be performed. Moreover, clinical studies should be carried out to verify the longevity of restorations submitted to different surface treatments.

## Conclusion

Etching with 5% HF for 20 seconds can be recommended for lithium disilicate and leucite-reinforced CAD/CAM ceramics. However, for pressed lithium disilicate ceramic, 10% HF for 60 s showed significantly higher bond strength to resin cement.

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