

# Effects of Hydrofluoric Acid Concentrations, Commercial Brands, and Adhesive Application on the Bond Strength of a Resin Luting Agent to Lithium Disilicate Glass Ceramic

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## Clinical Relevance

The concentration and commercial brands of hydrofluoric acid affect the bond strength of resin luting agents to lithium disilicate glass ceramic. The application of an adhesive layer seems beneficial and eliminates the effect of hydrofluoric acid concentrations and commercial brands.

## SUMMARY

**Objectives:** To evaluate the surface topography/roughness and bond strength of a resin luting agent

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to a lithium disilicate glass ceramic after etching with different concentrations of hydrofluoric acid (HF) and commercial brands.

**Methods:** For bond strength evaluation, 260

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Lithium disilicate glass ceramic (EMX) discs were randomly distributed into 13 groups based on concentrations of HF and commercial brands ( $n=20$ ): 5% and 10%, Lysanda (LY5 and LY10); 5% and 10%, Maquira (MA5 and MA10); 5% and 10%, FGM (FG5 and FG10); 4.8%, Ivoclar Vivadent (IV5); 5% and 10%, PHS do Brasil (PH5 and PH10); 5% and 10%, BM4 (BM5 and BM10); 9%, Ultradent Inc (UL10); and Dentsply (DE10). A further random distribution ( $n=10$ ) was made based on the application (+) or absence (-) of an adhesive layer. Resin luting agent cylinders (1 mm in diameter) were added on EMX surfaces, light-cured, and stored for 24 hours in deionized water at 37°C. On a universal testing machine (DL 500, EMIC), specimens were submitted to a microshear bond strength test at a crosshead speed of 1 mm/min until failure. A representative etched EMX disc from each group underwent surface topography analysis using field-emission scanning electron microscopy ( $n=1$ ), and five ( $n=5$ ) etched EMX discs from each group were tested for surface roughness. Data were statistically analyzed using analysis of variance and Tukey test ( $\alpha=0.05$ ).

**Results:** A less conditioned and smoother surface was observed for 5% HF compared to 10%. Additionally, commercial brands of HF were shown to affect bond strength. When the adhesive layer was not used (-), a 10% concentration promoted higher bond strengths to EMX. However, when adhesive was applied (+), the concentrations of HF and commercial brands had no effect on bond strength results.

**Conclusions:** A 10% concentration of HF results in higher bond strength than a 5% concentration. If an adhesive layer is applied, neither this distinction nor the influence of commercial brands is observed.

## INTRODUCTION

Indirect dental rehabilitation entails the fabrication of restorations in a dental laboratory, which are then cemented or bonded to the patient's teeth. These restorations must mimic dental form and function to restore lost or defective dental tissues due to carious lesions, fractures, wear, trauma, and dental anomalies and to improve dental esthetics in some cases such as diastemas, microdontia, and dental discolorations. The use of dental ceramic restorations for these applications is accelerating and consolidating, with indications for

full coverage (crowns), partial coverage (inlays, onlays, overlays, veneers), and components for fixed prostheses on teeth and implants.<sup>1-3</sup>

According to their crystalline content, dental ceramics can be classified as either glass ceramics (feldspathic, leucite, and lithium disilicate) or polycrystalline ceramics (zirconia and alumina). Lithium disilicate-based ceramics have distinguished themselves among glass ceramics due to their high ability to mimic the optical properties of dental tissues, their high mechanical properties after bonding protocols, their successful clinical outcomes, and their relative ease of fabrication.<sup>4-11</sup> Long-term clinical success requires proper adhesion between dental tissues (enamel and dentin) and resin luting agents, as well as between resin luting agents and ceramic intaglio surfaces.<sup>12</sup>

For bonding to enamel and dentin, phosphoric acid and adhesive systems have been extensively studied and well established in the literature.<sup>13-16</sup> The gold standard protocol for bonding to glass ceramics involves etching the intaglio surface with hydrofluoric acid (HF) and then applying a silane coupling agent.<sup>17,18</sup> HF can modify the surface topography of lithium disilicate reinforced glass ceramics by dissolving their glassy matrix thereby exposing lithium disilicate crystals, increasing surface roughness, and hence, favoring a micromechanical retention between the ceramic and the resin luting agent.<sup>18-22</sup>

Several studies have investigated the application of HF to lithium disilicate ceramic at varying concentrations, application times, and temperatures.<sup>19-23</sup> *In vitro* and review studies<sup>22,24-26</sup> have evaluated the impact of applying an adhesive layer after silane to improve the interaction between glass ceramics and resin luting agents, with conflicting findings. Moreover, the clinician or dental laboratory has access to a variety of commercial brands and concentrations, whose interactions were not properly investigated so far.

The objective of this *in vitro* study was to determine the effect of two concentrations of HF (5% and 10%) applied to the intaglio surface of a lithium disilicate reinforced glass ceramic on the microshear bond strength ( $\mu$ SBS) to a resin luting agent. Additionally, the effect of an adhesive layer after silane application was evaluated. The following research hypotheses were tested: 1) different HF concentrations influence the  $\mu$ SBS of resin luting agents to lithium disilicate reinforced glass ceramics; 2) different commercial brands influence the  $\mu$ SBS of resin luting agents to lithium disilicate reinforced glass ceramics; and 3) an adhesive layer influences the  $\mu$ SBS of resin luting agents to lithium disilicate reinforced glass ceramics.

## METHODS AND MATERIALS

### Specimen Preparation

A total of 343 ceramic specimens (10-mm diameter x 3-mm thickness) of a lithium disilicate reinforced glass ceramic (IPS e.max Press [EMX], color LT A2, Ivoclar Vivadent, Schaan, Liechtenstein) were fabricated in accordance with the manufacturer's instructions.<sup>22</sup> To embed EMX specimens in acrylic resin, 20 mm in diameter by 20 mm in height polyvinyl siloxane molds were used. The specimens were finished and polished using silicon carbide abrasive papers (#150 and #600, 3M ESPE, St Paul, MN, USA) in a water-cooled automatic grinder polisher (Metaserv 250, Buehler, Lake Buff, IL, USA) to achieve flat, uniform, and smooth surfaces. All specimens were rinsed with water, immersed in water, ultrasonically agitated for 10 minutes, and then air-dried with oil-free compressed air.

### Ceramic Surface Treatments

The EMX specimens were randomly distributed into 13 groups (n=20) according to the HF concentration and commercial brand (Table 1). Ceramic surfaces were etched for 20 seconds (using HF according to group assignments), rinsed with air/water for 20 seconds, and dried with oil-free compressed air for 20 seconds. Using a disposable microbrush, a silane coupling bonding agent (Monobond-N, Ivoclar Vivadent) (Table 1) was applied to the EMX surfaces for 15 seconds. The silane was allowed to react for 1 minute before being air-dried for 20 seconds. Silanized specimens were distributed randomly according to adhesive application (+) or no adhesive application (-) (n=10). For the groups that received an adhesive layer (+), a hydrophobic/unfilled adhesive (Scotchbond MP; 3M Oral Care, St Paul, MN, USA) (Table 1) was actively applied using a disposable microbrush for 10 seconds. Light activation was performed for 10 seconds using a multipeak light-emitting diode (LED) light-curing unit (Valo Cordless, Ultradent Inc, South Jordan, UT, USA) with an irradiance of 1000 mW/cm<sup>2</sup>.

### Field-emission Scanning Electron Microscopy

The etching topography was verified using a field-emission scanning electron microscope (FE-SEM) on representative EMX specimens (n=1) from each group. The surfaces were finished and polished with silicon abrasive paper (#150 and #600, 3M ESPE), ultrasonically cleaned for 10 minutes in water, and air-dried with oil-free compressed air. The specimen surfaces were then etched (using the respective HF) for 20 seconds, air-water sprayed for 20 seconds, and

air-dried. The specimens were mounted on aluminum stubs and coated with gold (Bal-TEC-SCD 050, Balzers Union AG, Fürstentum, Liechtenstein) for 120 seconds at 40 mA. One operator observed the EMX surfaces using FE-SEM (FEI Quanta 250, FEI Company, Eindhoven, Netherlands) with magnifications ranging from 2000× to 7000× at 15-25 kV.

### Microshear Bond Strength Tests

Translucent polyvinyl chloride tubes of 1.0 mm in diameter and 2 mm in height (nontoxic, Embramed, SP, Brazil) were sectioned and positioned on the specimen surface to define the bonding area. The tubes were filled at room temperature with a light-curing resin luting agent (Variolink Esthetic, shade "Light," Ivoclar Vivadent) (Table 1) and activated for 40 seconds using the same multipeak LED. On each specimen, three cylinders were created, resulting in 30 cylinders being tested for each group. Specimens were stored in deionized water at 37°C for 24 hours. Using a #11 scalpel blade, the tubes were carefully sectioned and removed after storage. All cylinders of resin luting agent were examined for flaws or defects, and those that were flawed were discarded and remade. For the  $\mu$ SBS tests, a 0.20-mm-diameter stainless-steel orthodontic wire was placed at the bonding interface and around each resin luting agent cylinder. A universal testing machine (DL 500, EMIC, São José dos Pinhais, PR, Brazil) equipped with a 100 N load cell and a crosshead speed of 1.0 mm/min was used to conduct tests until failure. Using the following equation, the values obtained in kilogram/force (KgF) were converted to megapascals (MPa):

$$BS = \frac{F \times 9.8}{A}$$

where F is the force, in kilograms (KgF); 9.8 is a constant used to convert the kilogram force to newtons; and A is the adhesive interface area ( $\pi r^2$ , where r is the radius of the resin luting agent cylinder).<sup>27</sup> The mean value of three resin luting agent cylinders was considered the mean  $\mu$ SBS (MPa) value for each specimen and was subsequently statistically evaluated. The failure patterns of fractured surfaces were classified as 1) adhesive, 2) cohesive in EMX, 3) cohesive in resin luting agent, 4) or mixed.

### Surface Roughness Analysis

A total of 70 EMX specimens were fabricated, and their surfaces were finished and polished as described previously. The specimens were randomly distributed into 14 groups based on concentration and commercial

Table 1: Information of the Materials Used in This Study

Material	Brand Name (Manufacturer)	Composition
Lithium disilicate glass ceramic	IPS e.max Press (Ivoclar Vivadent)	SiO <sub>2</sub> , Li <sub>2</sub> O, K <sub>2</sub> O, P <sub>2</sub> O <sub>5</sub> , ZrO <sub>2</sub> , ZnO, other oxides and ceramic pigments
Hydrofluoric acid	5% acid conditioner for Porcelain (Lysanda)	Hydrofluoric acid, distilled water, carbomer, pigments
Hydrofluoric acid	10% acid conditioner for Porcelain (Lysanda)	Hydrofluoric acid, distilled water, carbomer, pigments
Hydrofluoric acid	5% Acid Gel (Maquira)	Hydrofluoric acid, purified water, thickening agents, pigments
Hydrofluoric acid	10% Acid Gel (Maquira)	Hydrofluoric acid, purified water, thickening agents, pigments
Hydrofluoric acid	5% Condac Porcelana (FGM Dental Group)	Hydrofluoric acid, water, thickening agents, pigments
Hydrofluoric acid	10% Condac Porcelana (FGM Dental Group)	Hydrofluoric acid, water, thickening agents, pigments
Hydrofluoric acid	4.8% IPS Ceramic Etching Gel (Ivoclar Vivadent)	Hydrofluoric acid
Hydrofluoric acid	10% Porcelain Conditioner (Dentsply Industria e Comercio Ltda)	Hydrofluoric acid, water, thickening agents, pigments
Hydrofluoric acid	5% Potenza Attacco (PHS do Brasil)	Hydrofluoric acid, pigments, thickeners, deionized water
Hydrofluoric acid	10% Potenza Attacco (PHS do Brasil)	Hydrofluoric acid, pigments, thickeners, deionized water
Hydrofluoric acid	5% Power C-etching (BM4)	Hydrofluoric acid, water, thickening agents, pigments
Hydrofluoric acid	10% Power C-etching (BM4)	Hydrofluoric acid, water, thickening agents, pigments
Hydrofluoric acid	Porcelain Etch (Ultradent Inc)	Buffered 9% hydrofluoric acid
Silane coupling agent	Monobond-N (Ivoclar Vivadent)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, and sulphide methacrylate
Adhesive resin (bond)	Scotchbond MP (3M Oral Care)	Bisphenol A diglycidyl dimethacrylate, 2-hydroxyethyl methacrylate, amines, photoinitiator
Light-cured resin luting agent	Variolink Esthetic (Ivoclar Vivadent)	Urethane dimethacrylate and methacrylate monomers, ytterbium trifluoride and spheroid mixed oxide, initiators, stabilizers, pigments

brand of HF (n=5). The EMX surfaces were etched for 20 seconds, rinsed with air-water spray for 20 seconds, and air-dried for 20 seconds. For surface roughness analysis (Ra, average surface roughness), a diamond stylus with a 5- $\mu$ m tip radius was moved across the etched EMX surface in three directions (parallel, perpendicular, and oblique) with a speed of 0.25 mm/seconds and a range of 1.00 mm. Digital profilometry was used for the analysis (SurfTest SJ-410, Mitutoyo

Corp, Tokyo, Japan). Each specimen's mean surface roughness was determined by averaging the three values (Ra).

#### Statistical Analysis

The data from the  $\mu$ SBS tests were analyzed using a one- and two-way analysis of variance (ANOVA) followed by Tukey *post hoc* test to compare HF concentrations, commercial brand within each concentration, and

the effect of adhesive application (+ or -) within each commercial brand and HF concentration. Data on surface roughness were analyzed using a one-way ANOVA, followed by Tukey *post hoc* test to compare HF concentrations. All analyses were conducted using a statistical analysis program (Prism 9.4.1, GraphPad Software, LLC, Boston, MA, USA) with a pre-set alpha of 0.05.

## RESULTS

### Microshear Bond Strength

Table 2 shows the means and standard deviations for  $\mu$ SBS using 5% HF with or without the application of an adhesive layer. All evaluated factors had a significant influence on the results: commercial brand ( $p < 0.0001$ ), adhesive application ( $p = 0.0012$ ), and factor interaction ( $p = 0.0397$ ). The  $\mu$ SBS values for groups LY5 (Lysanda), MA5 (Maquira), and IV5 (Ivoclar Vivadent) were significantly greater than those of groups FG5 (FGM), PH5 (PHS do Brasil), and BM5 (BM4) without adhesive application (-). When an adhesive layer was applied (+), IV5 showed the highest values, which statistically did not differ from LY5, MA5, FG5, and PH5. BM5 had the lowest (+) values, which statistically did not differ from LY5, MA5, FG5, and PH5. The application of an adhesive layer (+) resulted in significantly higher values for PH5.

Table 3 shows the mean (SD)  $\mu$ SBS values when using 10% HF with or without adhesive application. Different commercial brands had a significant influence on

Table 2:  $\mu$ SBS Mean Values (Standard Deviation) for Different HF Commercial Brands at a Concentration of 5% With (+) or Without (-) Adhesive Application

Group	Adhesive Application <sup>a</sup>	
	-	+
LY5	41.8 (7.6) Aa	40.2 (5.1) ABa
MA5	41.3 (6.1) Aa	42.6 (4.8) ABa
FG5	33.8 (6.8) Ba	39.0 (5.1) ABa
IV5	44.1 (5.9) Aa	45.1 (4.9) Aa
PH5	33.3 (4.6) Bb	42.6 (3.7) ABa
BM5	30.5 (5.2) Ba	35.6 (5.5) Ba

Abbreviations: LY5, Acid Conditioner for Porcelain (Lysanda); MA5, Acido Gel (Maquira); FG5, Condac Porcelana (FGM); IV5, IPS Etching Gel (Ivoclar Vivadent); PH5, Potenza Attaco (PHS do Brasil); BM5, Power C-etching (BM4).

<sup>a</sup>Similar uppercase letters indicate no statistical differences ( $p > 0.05$ ) within a given column (commercial brands), and similar lowercase letters indicate no statistical differences ( $p > 0.05$ ) within a given row (adhesive application).

Table 3:  $\mu$ SBS Mean Values (Standard Deviation) for Different HF Commercial Brands at a Concentration of 10% With (+) or Without (-) Adhesive Application

Group	Adhesive Application <sup>a</sup>	
	-	+
LY10	40.3 (7.2) BCa	42.7 (3.7) ABa
MA10	47.6 (2.5) Aa	42.7 (5.3) ABa
FG10	46.6 (5.5) ABa	44.8 (4.1) ABa
DE10	46.0 (4.1) ABa	43.3 (6.5) ABa
UL10	39.8 (4.5) BCb	48.9 (4.2) Aa
PH10	46.7 (5.2) ABa	46.5 (7.1) ABa
BM10	38.8 (5.4) Ca	41.5 (5.4) Ba

Abbreviations: LY10, Acid Conditioner for Porcelain (Lysanda); MA10, Acido Gel (Maquira); FG10, Condac Porcelana (FGM); DE10, Porcelain Conditioner (Dentsply); UL10, Porcelain Etch (Ultradent Inc); PH10, Potenza Attaco (PHS do Brasil); BM10, Power C-etching (BM4).

<sup>a</sup>Similar uppercase letters indicate no statistical differences ( $p > 0.05$ ) within a given column (commercial brands), and similar lowercase letters indicate no statistical differences ( $p > 0.05$ ) within a given row (adhesive application).

$\mu$ SBS results ( $p = 0.0030$ ), with a significant interaction between factors ( $p = 0.0006$ ). MA10 showed the highest  $\mu$ SBS values when no adhesive layer was applied (-), which did not statistically differ from FG10, DE10, and PH10. BM10 showed the lowest values (-), which statistically did not differ from LY10 and UL10. On application of an adhesive layer (+), the highest  $\mu$ SBS values were found for UL10, which statistically did not differ from LY10, MA10, FG10, DE10, and PH10. BM10 showed the lowest (+) values, but statistically did not differ from LY10, MA10, FG10, DE10, and PH10. Adding an adhesive layer (+) increased UL10 values significantly.

Table 4 compares 5% and 10% HF concentrations from the same manufacturer with (+) or without (-) application of adhesive. The highest SBS values were observed for HF at 10% concentration across all manufacturers ( $p < 0.0001$ ), except for LY ( $p = 0.7922$ ), where no statistically significant differences were observed for different concentrations or adhesive application. The application of an adhesive layer did not increase  $\mu$ SBS values, except for PH5 ( $p = 0.0112$ ). The lowest  $\mu$ SBS values were found for 5% concentration without adhesive application for MA, FG, PH, and BM. However, adhesive application on MA5, PH5, and BM5 resulted in no statistical differences in relation to their respective 10% groups. The majority of failures were adhesive (98%), while mixed failures were observed but with a lower frequency (2%).

Table 4:  $\mu$ SBS Mean Values (Standard Deviation) for Different HF Commercial Brands at 5% or 10% Concentrations, With (+) or Without (-) Adhesive Application

Manufacturer	Adhesive	Concentration (%) <sup>a</sup>	
		5	10
LY	-	41.77 (7.61) A	40.29 (7.21) A
	+	40.24 (5.16) A	42.75 (3.75) A
MA	-	41.27 (6.14) BC	47.58 (2.51) A
	+	42.59 (4.77) AB	42.71 (5.34) AB
FG	-	33.83 (6.86) C	46.67 (5.56) A
	+	39.00 (5.12) BC	44.81 (4.15) AB
PH	-	33.35 (4.66) B	46.75 (5.19) A
	+	42.63 (3.71) A	46.48 (7.15) A
BM	-	30.51 (5.26) BC	38.81 (5.46) A
	+	35.61 (5.55) AB	41.52 (5.42) A

Abbreviations: LY, Lysanda; MA, Maquira; FG, FGM; PH, PHS do Brasil; BM, BM4.  
<sup>a</sup>Similar uppercase letters indicate no statistical differences ( $p>0.05$ ) within commercial brands.

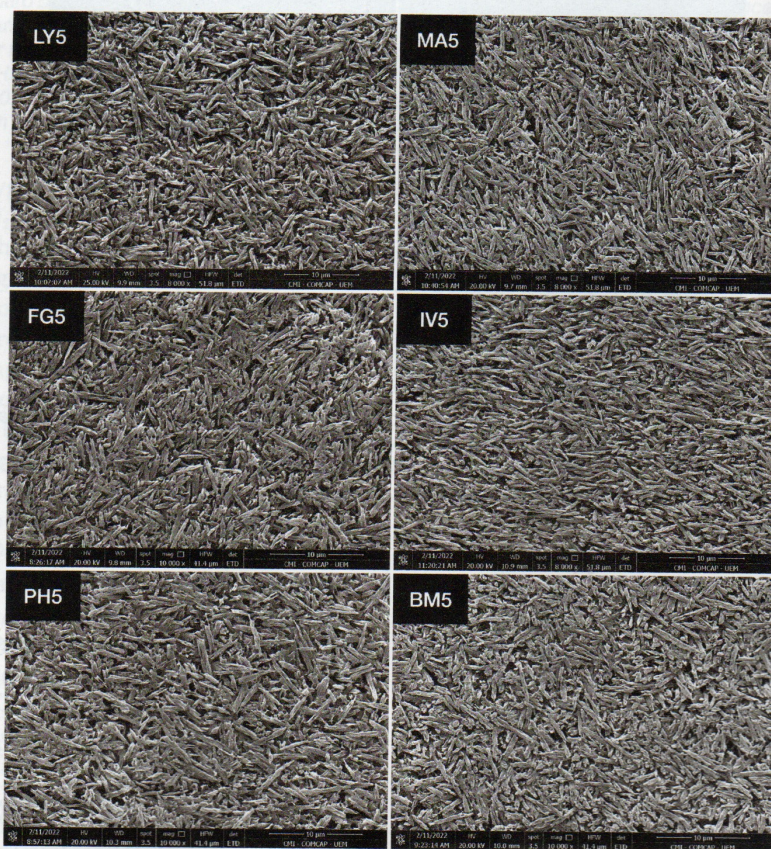


Figure 1. Field-emission scanning electron microscopy images resulting from hydrofluoric acid etching at concentrations ranging from 4.8% to 5% of various commercial brands. Abbreviations: LY5, Acid Conditioner for Porcelain (Lysanda); MA5, 5% Acido Gel (Maquira); FG5, 5% Condac Porcelana (FGM); IV5, 5% IPS Etching Gel (Ivoclar Vivadent); PH5, 5% Potenza Attaco (PHS do Brasil); BM5, 5% Power C-etching (BM4).



Figure 2. Field-emission scanning electron microscopy images resulting from hydrofluoric acid etching at concentrations ranging from 9% to 10% of various commercial brands. Abbreviations: LY10, Acid Conditioner for Porcelain (Lysanda); MA10, 10% Acido Gel (Maquira); FG10, 10% Condac Porcelana (FGM); DE10, 10% Porcelain Conditioner (Dentsply); UL10, 10% Porcelain Etch (Ultradent Inc); PH10, Potenza Attacco (PHS do Brasil); BM10, 10% Power C-etching (BM4).

### Scanning Electron Microscopy

Figures 1 and 2 present FE-SEM images of the etched EMX surfaces. The 10% concentration of HF showed greater glass matrix dissolution and, consequently, a greater exposure of lithium disilicate crystals than the 5% concentration, regardless of the commercial brand. At the same concentration, various commercial brands of HF produced comparable etching patterns on EMX surfaces.

### Surface Roughness Analysis

All commercial brands at 10% concentration resulted in rougher surfaces than the control group ( $p < 0.05$ ; Figure 3). Only FG5 and LY5 produced a rougher

surface than the control group at a concentration of 5% ( $p < 0.05$ ). The surface roughness of EMX surfaces etched with 10% HF was greater than 5% concentration for all commercial brands ( $p < 0.05$ ), except LY5, which did not differ from FG10 ( $p > 0.05$ ). Within each HF concentration (5% or 10%), there were no statistically significant differences among manufacturers.

### DISCUSSION

Because different concentrations of HF had a direct effect on the  $\mu$ SBS of a resin luting agent to lithium disilicate, the first research hypothesis was confirmed. HF, when applied to glass ceramics, attacks the bond

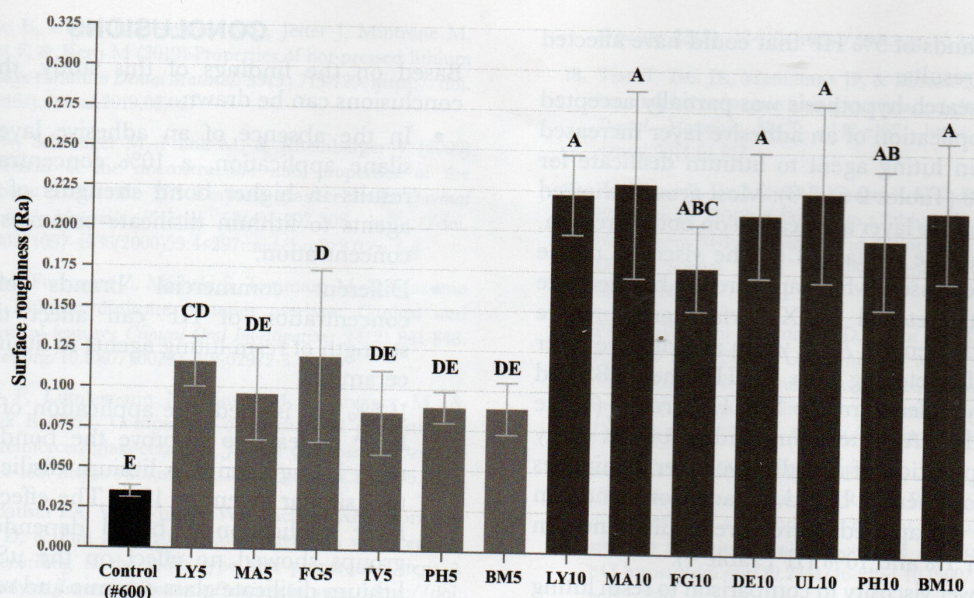


Figure 3. Means and standard deviation of surface roughness (Ra) for lithium disilicate etched with various concentrations of hydrofluoric acid and control group (#600 grit). Similar uppercase letters indicate no statistical differences ( $p > 0.05$ ) within commercial brands. Abbreviations: LY5, 5% Acid Conditioner for Porcelain (Lysanda); MA5, 5% Acido Gel (Maquirá); FG5, 5% Condac Porcelana (FGM); IV5, 5% IPS Etching Gel (Ivoclar Vivadent); PH5, 5% Potenza Attaco (PHS do Brasil); BM5, 5% Power C-etching (BM4); LY10, 10% Acid Conditioner for Porcelain (Lysanda); MA10, 10% Acido Gel (Maquirá); FG10, 10% Condac Porcelana (FGM); DE10, 10% Porcelain Conditioner (Dentsply); UL10, 10% Porcelain Etch (Ultradent Inc); PH10, 10% Potenza Attaco (PHS do Brasil); BM10, 10% Power C-etching (BM4).

between silica and oxygen<sup>28</sup> due to the higher affinity of fluoride for silica ( $\text{SiO}_2$ ) than oxygen<sup>29</sup> and is capable of selectively removing the glassy matrix.<sup>23</sup> When applied to lithium disilicate glass ceramics, HF removes the glassy matrix and exposes the lithium disilicate crystals thereby creating microsities that improve mechanical retention of resin luting agents.<sup>21-23</sup>

According to Table 4, the 5% concentration of HF exhibited lower bond strength values than the 10% concentration, except for LY. In general, the efficacy of HF depends on concentration, reaction time, substrate composition, temperature, and acid solubility.<sup>19,20,22,23,30,31</sup> Due to the increased amount of ionized HF available to react with the silica, the higher the HF concentration, the greater the removal of the glassy matrix.<sup>23</sup> As a result, the glass ceramic becomes rougher, with a greater amount of surface area and increased energy. This increased surface energy enhances the wetting of silane and resinous materials that infiltrate the surface and improves the adhesion to resin luting agents.<sup>19,32,33</sup> The etching potentials of 5% and 10% concentrations are evident in FE-SEM images (Figures 1 and 2) and surface Ra results (Figure 3). When a concentration of 5% was used, it is notable that greater amounts of glassy matrix remained on the ceramic surfaces. More distinct and visible lithium

disilicate crystals with a larger surface area can be seen in the micrographs at 10% HF concentration. When compared to 5% or the control, Figure 3 shows an increase in surface roughness for all groups etched with 10% HF. Similar findings have been reported from prior *in vitro* studies.<sup>19,21,22,30</sup>

The second research hypothesis was accepted because different commercial brands within a given concentration affected  $\mu\text{SBS}$  of the same resin luting agent to lithium disilicate. Table 2 shows that  $\mu\text{SBS}$  varies through commercial brands at 5% HF concentration when no adhesive system has been applied (-). The 10% concentration produced similar outcomes, as shown in Table 3. Their varying chemical compositions can explain these findings. Despite their similar compositions (Table 1), manufacturers are not required to disclose the precise composition of their products, such as the proportion of each component or type of thickener used. Given that each manufacturer is attempting to secure its intellectual property, this is understandable. However, regardless of brand, 5% HF concentrations exhibited similar etching pattern topographies (Figure 1) and surface roughness (Figure 3). It can be assumed that FE-SEM and surface roughness evaluations failed to identify subtle differences in the etching pattern among various

commercial brands of 5% HF that could have affected bond strength results.

The third research hypothesis was partially accepted because the application of an adhesive layer increased  $\mu$ SBS of a resin luting agent to lithium disilicate for PH5 and UL10 (Tables 2 and 3). Most groups showed no effect of adhesive layer application on bond strength. This result may be explained by the viscosity of the resin luting agent used, which appeared to be adequate to penetrate the etched EMX surface and provide adequate bond strength. Also, when an adhesive layer was applied after etching with 5% HF, the  $\mu$ SBS did not statistically differ from the 10% HF groups (Table 4), except for FG. An interesting finding of this study is that the application of an adhesive layer minimizes the effect of commercial brand. In addition, when an adhesive layer was applied, there were no differences in  $\mu$ SBS between 5% and 10% HF (Table 4).

Due to its lower viscosity in comparison to resin luting agents, the adhesive layer may better infiltrate the etched glass ceramic surface, resulting in a more homogeneous interface between the resin luting agent and the glass ceramic.<sup>22,24,25</sup> This can improve adhesion of the resin luting agent to the ceramic. However, a recent systematic review and meta-analysis of *in vitro* studies revealed no effect of the adhesive layer on the bond strength to glass ceramics.<sup>26</sup> It can be assumed that, despite not showing a significant effect on bond strength, a more homogeneous bonding interface, because of improved resin infiltration into glass ceramics, will contribute positively to the clinical longevity of glass ceramic restorations. Flaws at the interface between resin luting agents and glass ceramics<sup>22,24,25</sup> may be considered as stressing points that can initiate cohesive fractures of resin luting agents and/or glass ceramics, resulting in debonding or fracture.<sup>34</sup>

HF is a hazardous and toxic solution capable of causing severe trauma to soft tissues.<sup>29,35</sup> Because some commercial brands may perform better than others at the same concentration, selecting lower concentrations of HF may be less hazardous for dental professionals and patients. Moreover, the application of an adhesive layer after using 5% HF improved the bond strength of resin luting agents to lithium disilicate. Thus, clinicians may choose using 5% HF concentrations followed by an adhesive layer, instead of 10%. Future research should assess the effect of various thickening agents in HF gels and the resulting bonding quality between glass ceramics and resin luting agents. Furthermore, it is important to emphasize that the type of adhesive layer used may influence these results. Thus, studies further investigating this technique would be beneficial.

## CONCLUSIONS

Based on the findings of this study, the following conclusions can be drawn.

- In the absence of an adhesive layer and after silane application, a 10% concentration of HF results in higher bond strengths of resin luting agents to lithium disilicate ceramics than a 5% concentration.
- Different commercial brands of a given concentration of HF can affect the bonding strength of resin luting agents to lithium disilicate ceramics.
- If 5% HF is used, the application of an adhesive layer appears to improve the bond strength of resin luting agents to lithium disilicate ceramics to a similar extent as 10%. The effect of adhesive layer application is brand dependent as most groups showed no effect on the  $\mu$ SBS between lithium disilicate glass ceramic and resin cement.

## Conflict of Interest

The authors of this manuscript 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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