

# Influence of Different Conditioning Protocols on Bond Strength to Glass Ceramic

Fatima A. Shalaby<sup>1</sup>, Nesma M. Elgohary<sup>2</sup>, Noha A. El Wassefy<sup>3</sup>, Ahmed A. Abo EL- Naga<sup>4</sup>

## Abstract:

**Objective:** to detect the effect of different surface treatments on bond strength to machinable glass ceramic. **Materials and Methods:** Sixteen glass ceramic blocks (IPS e.max CAD/CAM) were fabricated by a CAD/CAM machine with dimensions (6mm × 11mm x 13mm) and divided into 4 groups (n=4), according to surface treatments: (G1) Hydrofluoric acid (HF) etching and silane application, (G2) Acidulated phosphate fluoride (APF) and silane application, (G3) Monobond Etch and Prime (MEP), (G4) Tetrabutylammonium dihydrogen trifluoride (TDTF) and silane application. Composite resin blocks (Te economer) were fabricated and cemented to the treated ceramic blocks using adhesive resin cement. All bonded specimens were kept in a water bath for three months and went through 5000 thermal cycles. The bilayered specimens were sectioned into micro-bars (1 mm\* 1 mm in cross-section) using a diamond-coated disc under water cooling using a cutting machine. Microtensile bond strength ( $\mu$ TBS) test was performed. Scanning Electron Microscope (SEM) was used for specimens' examination. **Results:** HF with silane group shows the highest  $\mu$ TBS mean value (39.3±19 MPa) followed by Monobond Etch & Prime (33.0±11.5 MPa). On the other hand, APF with silane showed the lowest  $\mu$ TBS mean value (7.5±3.8 MPa). **Conclusions:** HF acid and Monobond Etch and Prime system are preferred to be used for surface treatment of lithium disilicate ceramics.

## Introduction:

With the demand for indirect esthetic restorations, contemporary ceramic systems have been developed with varying proportions of glass and crystalline phases to ensure a balance between improved mechanical properties, color stability, and radiopacity.<sup>1</sup> Materials such as glass-ceramic (feldspathic, leucite, lithium disilicate reinforced materials), are widely used among the various types of ceramics available in dentistry due to their ability to adhere to the tooth structure increase. They are known for their excellent optical properties, biocompatibility, and surface smoothness, which is an important element of plaque control. They are available in final form using hot press technology or CAD/CAM technology.<sup>2</sup> To optimize the bonding procedures, pretreatment steps for the tooth and ceramic surface are necessary before applying a resin cement.<sup>3</sup> The surface treatment of the porcelain increases the surface area, forms micropores and increases the mechanical retention potential of the luting composite resin. The importance of acid conditioning and silane priming of etchable glass-ceramic restorations in achieving long and strong bonds to tooth structures has long been recognized.<sup>4</sup> The use of orthophosphoric acid, sulfuric acid, N-nitric acid, hydrofluoric acid (HF), acidic hydrofluoric acid (APF) gel, ammonium hydrogendifluoride, and wear by suspended particles for the chemical surface treatment of dental ceramics

are included as different surface treatment protocols.<sup>5</sup> The chemical bonding capacity of silane was high in products containing silanol monomers. Acid etching increases the bond strength to a level that neutralizes the silane contribution of products containing silanol siloxane adducts and siloxane polymers, providing bond strength values similar to silane-free treatments.<sup>6</sup> Among other things, HF acid is considered the gold standard for etching ceramics before the final cementation. Although

widely used, it is a highly corrosive liquid and has many drawbacks that cause tissue rashes, burns, and contact poisons.<sup>7</sup> Etching with hydrofluoric acid (HF) followed by silane coupling agent application is the most common type of pretreatment for glass-ceramic surfaces. Reportedly, this pretreatment provides the highest bond strength values.<sup>8</sup> The use of 4.9% HF for 20 sec proved to be the most effective etching treatment of the intaglio surface.<sup>9</sup> The glassy matrix of silicate ceramics is selectively removed, resulting in a micromorphological three-dimensional porous surface that permits micromechanical interlocking of the luting composite. Micromechanical treatments, such as sandblasting to improve surface roughness, as well as chemical treatments with 10-MDP ceramic primers or tribochemical silica coating, are required for ceramics with a high crystalline content (polycrystalline ceramics). The use of a silane primer can improve this micromechanical treatment.<sup>10</sup> The gold-standard surface treatment for glass ceramics is a combination of mechanical and chemical techniques. HF etching and silanization, in addition to the benefits described above, are quick and simple methods that do not require any additional equipment.<sup>11</sup>

Various topical fluorides such as APF gel are safe for oral tissues and can etch or react with porcelain, glass ionomer, fissure sealant, and composite restorative

<sup>1</sup>Postgraduate MSc student, Department of Fixed Prosthodontics Faculty of Dentistry, Mansoura University, 35516, Mansoura, Egypt. Egypt. [fa85535@gmail.com](mailto:fa85535@gmail.com)

<sup>2</sup>Lecturer, Department of, Faculty of Fixed Prosthodontics Dentistry, Mansoura University, Egypt.

<sup>3</sup>Associate professor, Department of Dental Biomaterials, Faculty of Dentistry, Mansoura University

<sup>4</sup>Professor, Department of Fixed Prosthodontics, Faculty of Dentistry, Mansoura University, Egypt.

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Table 1: The materials used in the study

Material type	Product name	Composition	Manufacturer	Lot number
Lithium disilicate glass ceramic	IPS e.max CAD/CAM	SiO <sub>2</sub> 57 – 80% Li <sub>2</sub> O 11 – 19% K <sub>2</sub> O 0 – 13% P <sub>2</sub> O <sub>5</sub> 0 – 11% ZrO <sub>2</sub> 0 – 8% ZnO 0 – 8% Other oxides and ceramic pigments 0 – 10%	Ivoclar Vivadent, Schaan/Liechten stein	G20677
50 µm Al <sub>2</sub> O <sub>3</sub> for sandblasting	Renfert ALUMINIUM OXID 50 µm	99.7% aluminum oxide	Renfert, Germany Hilzingen in the Lake Constance region.	78247
Ammonium hydrogen difluoride	Ammonium Bifluoride Pure	Assay (acidimetric): 98%, Maximum limits of impurities: Nonvolatile matter: 0.05% Chloride: 0.005%, silica: 0.5% sulphate: 0.05%, iron: 0.005%, lead: 0.01%	Loba Chemie, India Boisar	S12541307
Hydrofluoric acid	Porcelain Etch Ultradent	viscous, buffered 9% hydrofluoric acid	West Ultradent Drive South Jordan, UT	BJ381
Silane	Ultradent	Organosilane (MPS) 5-15% Isopropyl Alcohol 92% Acetic acid <1%	West Ultradent Drive South Jordan, UT	BJ381
Acidulated phosphate fluoride gel	Dharma Ionite Fluoride Gel	Acidulated phosphate fluoride 1.23% Sodium fluoride 2.72% Xylitol	Dharma Ionite Walmart Rogers, Ark.	
Universal primer	Monobond Etch & Prime	Ammonium polyfluoride silane system based on trimethoxypropyl methacrylate Solvents: alcohols and water	ivoclar vivadent Schaan/Liechten stein, Germany	W40212
Light cure composite resin	Te econome	Bis-GMA, UDMA, Bis-EMA and Barium aluminum borosilicate	ivoclar vivadent Schaan/Liechten stein, Germany	Y36520
Self-curing acrylic resin	Acrostone Cold Cure	Powder: PMMA, Benzoyl peroxide, Pigments (1%) Liquid: MMA, Ethylene glycol, Dimethacrylate (10%), Hydroquinone (>1%)	Acrostone, Egypt	
Dual curing adhesive composite	Vita Adiva Full Adhesive	Bis-GMA-based resins, catalysts, stabilizers, pigments, and inorganic filler particles. Filler 61% by weight	VITA Zahnfabrik, Germany	78380

materials.<sup>12</sup> A self-etching primer is another pretreatment that was created to decrease method sensitivity and make acid etching of glass ceramic restorations easier. Monobond Etch and Prime is a combination of silane and a priming agent in a single container, allowing surface etching and silanization in a single process.<sup>13</sup> Surface treatment with self-etching primers resulted in similar fatigue behavior compared to the use of hydrofluoric acid and silane using the same treatment technique, but tended to provide higher mechanical reliability.<sup>7</sup>

The combination of TDTF + silane has been marketed as a clinically acceptable product for strengthening the bonds of glass ceramics.<sup>14</sup> TDTF is also acidic and has some dangerous effects. Therefore, due to the potential

danger to human tissue, care must be taken when handling and applying self-etching primers containing less than ten percent TDTF.<sup>15</sup> The type of surface treatment is an important factor that affects the adhesive strength of glass-ceramics. Therefore, this in vitro study aimed to investigate the effects of various surface treatments on bonding to machinable glass-ceramic.

#### Materials and methods:

**Materials:** The materials that were used in this study are illustrated in Table 1.

**Methods:** In a previous study<sup>16</sup> the response within each subject group was normally distributed with standard deviation 7 and the true difference in the

experimental and control means was 11.1 Based on these estimates using software (Power and Sample Size Calculations v3.1.2; Informer Technologies, Inc), a sample of at least 7 in each group was required to provide a power of 0.08. The Type I error probability associated with this test was 0.05.

**Preparation of ceramic blocks:** A total of sixteen blocks of glass ceramic (IPS e.max) with the dimensions of (6x11x15 mm) were fabricated using CAD/CAM technology, by preparing a wax pattern with dental modeling wax (Perfect Wax, Bilkim, Turkey) using a specially designed Teflon mold then scanning using Ceramill Map 400+. The glass ceramics were then wet milled from IPS e.max by using ceramill® Motion 2 CAD/CAM machine (Amann Girrbach, Austria). After milling, the sintering of the glass ceramic blocks was done using high-temperature furnace (ceramill® Therm, Amann Girrbach) according to manufacturer's instructions.

**Composite resin blocks preparation:**

Composite resin blocks were duplicated using putty impression material (speedex Coltène/Whaledent). The prepared holes were incrementally filled with composite resin (te econom plus, ivoclar vivadent, Korea) (shade B2) that was polymerized with light-curing unit (liteQ LD-107, MONITEX, Taiwan) for 20sec for each increment to fabricate the resin blocks. Composite resin blocks were inspected for any errors after removal, finished and polished. The bonding surfaces of the Composite resin blocks were sandblasted using 50 µm Al<sub>2</sub>O<sub>3</sub> particles (Renfert-Technologie, Germany) under a pressure of 2 bars for 15 seconds with 10 mm distance perpendicular to block bonding surfaces using Renfert Basic sandblaster (Renfert, Germany).

**Surface treatment of glass ceramic blocks:** Ceramic blocks were randomized and divided according to the type of surface treatment into four groups (n=4) each one was subjected to a certain protocol: Group 1: ceramic blocks were etched with porcelain etch HF for 90 seconds, rinsed, and dried; a puddle coat of silane was applied for 60 seconds and dried. Group 2: ceramic blocks were etched with acidulated phosphate fluoride gel for five minutes, rinsed and dried. a puddle coat of silane was added to the inside surface for sixty seconds and dried. Group 3: ceramics blocks were agitated with Monobond Etch & Prime for twenty seconds and remained for another forty seconds. Group 4: ceramic blocks were etched with NH<sub>4</sub>HF<sub>2</sub> as follows: Crystals of NH<sub>4</sub>HF<sub>2</sub> were ground using mortar and pestle to produce NH<sub>4</sub>HF<sub>2</sub> fine powder. 4.2mg of the powder was used to form viscous slurries by mixing with 1 ml distilled water then the formed slurries were spread on the bonding surfaces of glass ceramic blocks. The blocks were heated in a preheated furnace for 5 minutes at 175°C. The blocks were then bench-cooled after removal from the oven. After etching, the blocks were rinsed in water and then ultrasonically cleaned in ethanol (ethyl alcohol 95%) for 5 min and air dried before cementation.

**Bonding of ceramic blocks to composite resin blocks:** The bonding of composite resin blocks and

previously treated glass ceramic blocks was performed using adhesive resin cement (VITA Adiva Full Adhesive, Germany) as follows: Glass ceramic blocks were secured to a specially designed device with a lever system to deliver a constant load of 10 Kg on the composite/ glass ceramic blocks assembly during cementation. VITA Adiva Full Adhesive resin cement was mixed according to the manufacturer instructions and applied through the disposable automix tip on the bonding surface of the secured glass ceramic blocks. The composite resin blocks were placed onto the glass ceramic blocks after cement application. The constant load (10 Kg) was applied on the composite/ glass ceramic blocks assembly. Excess resin cement was removed with a micro brush then curing was done using (liteQ LD-107, MONITEX, Taiwan) from four directions for 20 sec. The bonded assembly was kept for 5 min under the static load.

**Artificial aging:** After cementation, bonded specimens were stored in distilled water at 37° C for 3 months followed by thermocycling for 5000 cycles using a thermocycling device (thermocycler, ROBOTA, Alexandria, Egypt) then air dried. Each thermal cycle is composed of a 1minute cold bath 5 °C followed by a 1minute hot bath 55 °C with a 30sec dwell time. After artificial aging procedures, the bonded specimens were fixed in acrylic resin blocks surrounded by wax rings. The bonded specimens were placed within a wax mold with the notched untreated surfaces of the glass ceramics blocks facing upward.<sup>13</sup>

**Micro-tensile Bond Strength (µTBS) Test:** A total of 16 ceramic-resin composite assemblies were sliced into stick-shaped specimens (1 x1 mm) using a low-speed diamond saw (Pico155, pace Technologies, Tucson, AZ, US) under a water cooling system. Exterior sticks, as well as any that appeared to have interface defects, were discarded. The micro-tensile bond strength was tested. Specimens were individually attached to a designed jig connected to universal testing equipment (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) with cyanoacrylate gel glue. At a crosshead speed of 0.5 mm/min, a tensile load was applied until failure. The µTBSs were calculated by dividing the reported load at failure (N) by the bonded area and represented in MPa. Pre-test failures were documented if specimens failed before being tested.

**Scanning Electron Microscope (SEM):** To evaluate the surface characterization of lithium disilicate glass ceramic, two specimens from each group were analyzed by SEM. Each specimen was air dried, mounted on copper stubs, and then coated with a thin layer of gold (Sputter Coating Evaporator, SPI- Sputter Coater, USA) before being inspected with a SEM (JEOL.JSM.6510LV, Japan) at magnifications (x3000).<sup>17</sup> Statistical analyses: Data were analyzed with the statistical package for social science (SPSS) version 22 (SPSS Inc. Chicago, IL, USA). Variables were presented as Mean± SD. Statistical analysis of data was performed in several steps.IL

**Results:**

Micro-tensile Bond Strength (µTBS) results:

Table 2: Mean and SD of  $\mu$ TBS mean values (MPa) for all groups after thermocycling and statistically differences between test groups regarding surface treatment

	HF Etching	APF	Monobond	TDTF	N	Mean	Std. Deviation
HF+Silane					7	39.3	19
Monobond	0.003**		0.003**	0.06	7	33	11.5
TADF+silane	0.7			0.007**	7	14.1	5.7
APF+silane	0.01*				7	7.5	3.8

The difference in the mean values showed significance when the p value  $\leq 0.05$ .

\*Indicate statistically significant difference.

\*\* Indicate highly statistically significant difference.

Statistical analysis: The influence of different surface treatments was tested by One-Way ANOVA test. Whenever one-way ANOVA test showed significance, Post Hoc Tukey test was utilized for comparing the means of each two test groups at ( $p \leq 0.05$ ). One-way ANOVA test showed significant differences in the values of  $\mu$ TBS as a result of using different surface treatment methods ( $p < 0.001$ ).

Post Hoc Tukey test was used for pairwise comparison between different test groups following one-way ANOVA test. It revealed that, there were statistically significant differences between test groups as follow in Table 2. There was high statistically significant difference between (HF, MEP) test groups as a set and (TADF, APF) test groups as another set. There were no statically significant differences in  $\mu$ TBS between HF+silane application and Monobond etch & Prime test groups ( $P = 0.7$ ). Also, there were no significant differences in the  $\mu$ TBS between APF and TADF+s test groups ( $P = 0.06$ ). On the other hand, HF+s test group showed a significant difference to the test group TADF+s, ( $p=0.01$ ) and high significant difference to test group APF+s, ( $p=0.003$ ). Also, MEP test group showed high significant difference to the test group APF+s, ( $p=0.003$ ) and the test group TADF+s, ( $P = 0.007$ ).

Scanning Electron Microscope (SEM): SEM was used for the investigation of surface characterization of ceramic blocks as shown in Figure A-D. The SE photomicrographs of the specimens etched by HF (Figure A) show highly textured surface irregularities with extensive deepened porosities (blue arrows).

The SE photomicrographs of the specimens etched by APF (Figure B) show prismatic rough surface with tiny fissure-like porosities (red arrows).

The SE photomicrographs of the specimens etched by monoband (Figure C) show moderately textured surface irregularities than HF etched group with fewer and shallower porosities (yellow arrows). The SE photomicrographs of the specimens etched by TDTF (Figure D) show shallow texture surface with negligible porosity.

### Discussion:

Due to its excellent esthetic quality, excellent mechanical properties, biocompatibility, and long durability, ceramic restorations are widely used in routine dental practice.<sup>18</sup>

Lithium disilicate materials have been actively pushed due to their excellent esthetics, adhesive properties, capacity to retain tooth structure, and good fracture resistance.<sup>19</sup>

The strength and stability of the bond between ceramic and resin cement determine the clinical outcome of a ceramic restoration. Self-adhesive resin cements combine the benefits of both adhesive and conventional luting agents, overcoming the multi-step adhesive resin cements luting procedure's complexity and technical sensitivity.<sup>20</sup>

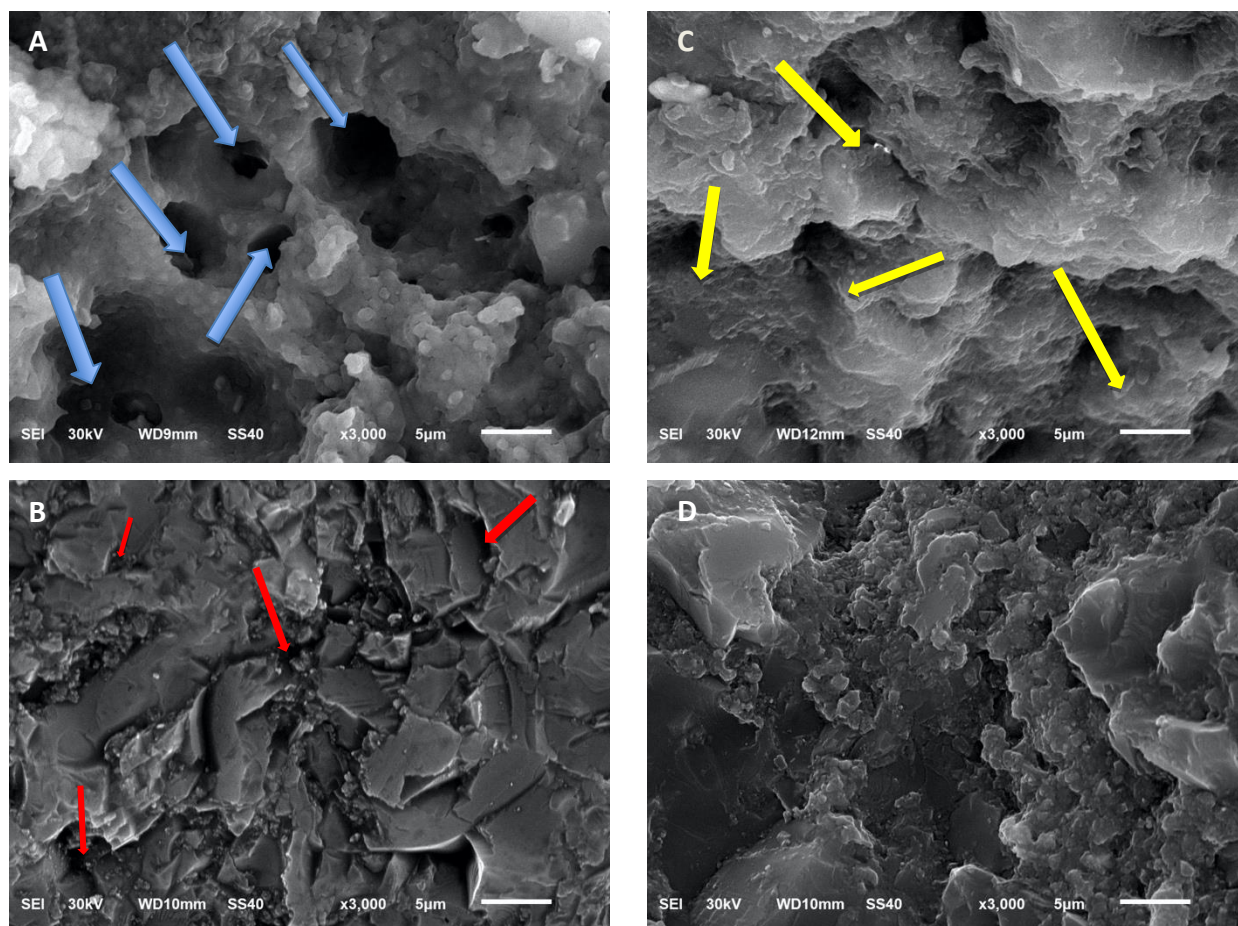
Instead of dental tissues, glass ceramic specimens were glued to composite resin blocks in this study, due to the heterogeneous microstructure of dentin, the composite resin blocks' homogeneous structure would prevent interpretation errors in the bond strength data that could occur when employing tooth tissue.<sup>21</sup>

The most successful surface treatment approach for glass ceramics is the use of hydrofluoric acid followed by the application of silane because it increases the surface area for micromechanical entanglement that promotes and improves the interaction between ceramic and resin cement with increased bond strength.<sup>22</sup>

The results of in vitro studies have indicated that various topical fluorides such as APF gel which is safe for oral tissues may etch or react with porcelain, glass ionomer, fissure sealant, and composite restorative materials. The surface roughness of feldspathic porcelain, low-fusing porcelain, and aluminous porcelain increased after being treated with acidulated phosphate fluoride.<sup>23</sup>

A self-etching primer is another pretreatment that was





**Figure:** Scanning Electron photomicrographs; (A) Surface of lithiumdisilicate glass ceramic specimen etched by HF, (B) Surface of lithiumdisilicate glass ceramic specimen etched by APF, (C) Surface of lithiumdisilicate glass ceramic specimen etched by monobond, (D) Surface of lithiumdisilicate glass ceramic specimen etched by TDTF.

created to minimize method sensitivity and make acid etching of glass ceramic restorations easier. Monobond Etch and Prime is made up of silane and a priming agent in one container, enabling surface etching and silanization in one process.<sup>13</sup>

The combination of TDTF and silane has been introduced to the market as a clinically acceptable product for glass-ceramic reinforced bonding.<sup>14</sup> Thermal cycling and mechanical loading have been shown in laboratory experiments to predict clinical failures, although it appears that appropriate validation for individual materials is required.<sup>24</sup>

In this study, we used microtensile bond strength test because it can concentrate on three-dimensional surfaces that are clinically relevant and has a high capability for discrimination than the ordinary macroshear bond test. Furthermore, it was also shown the possibility to compare the long-term stability of resin adhesion to different parts of the cavity wall of the extracted tooth at different times after placement of the adhesive restoration.<sup>25</sup>

In this in-vitro study, the mode of failure was evaluated to understand in relation to underlying material properties.<sup>26</sup> The results of this study showed that there was a statistically significant difference between the

four surface treatment methods (HF, EP) as a set and (APF, TDTF) as another set. This could be because the chemical composition of the ceramic system affects the strength and durability of the link between ceramic and resin cement, and surface treatments are required

to ensure adhesion between the luting agent and the ceramic surface.<sup>27</sup> Also, the surface treatment is appropriate depending on the ceramic's composition. Results of this research work revealed that, hydrofluoric acid group and Monobond Etch and Prime one showed the highest micro-tensile bond strength mean value. They are too close in the results but hydrofluoric acid group is higher followed by Monobond Etch and Prime one although non-significant. This may be due to HF acid showed greater roughness and irregularities as documented by the SEM results (Figure A); this permits appropriate infiltration of the adhesive resin cement.<sup>28</sup>

A study compared the standard surface conditioning (hydrofluoric acid + silane) with a one-step primer to evaluate the micro-shear bonding strength (SBS) of a composite cement bonded to two machined glass ceramics consistent with Prado et al.<sup>29</sup> who determined the durability of (Monobond Etch & Prime), it was found that hydrofluoric acid + silane had a higher

average  $\mu$ SBS of both ceramics than Monobond Etch and Prime. However, it showed stable adhesion after aging. The "self-priming ceramic primer" has fewer surface changes and the same adhesion efficiency as when the hydrofluoric acid/silane primer is applied individually.<sup>30</sup> The bond strength of a resin composite to lithium disilicate treated by a self-etch primer was inferior under all storage conditions than that of a functional silane and the same primer applied to HF-etched surfaces. Accelerated aging had a significant impact on the self-etch primer group, with values that were even lower than the negative control group (HF-etched substrate without silane). MEP was identical to HF+NS for SBS.<sup>31</sup> Maier et al.<sup>32</sup> informed that glass-ceramic primer that self-etches with Monobond Etch and Prime produced bond strengths that were comparable to HF-etched and silanized specimens. This study is also in line with Lopes et al.<sup>33</sup> who concluded that HF etching followed by a silane solution resulted in stronger bonding than a self-etching ceramic primer. On the other hand, the outcomes of this research are not in agreement with Donmez et al.<sup>34</sup> who concluded that Monobond Etch and Prime may be a preferable method to achieve high bond strength values than hydrofluoric acid and silane. The results of this research work also revealed that Monobond Etch & Prime still had a comparable bond strength to hydrofluoric acid. This could be because the glassy ceramic phase was removed in Etch and Prime, resulting in projecting domains or residual glass and surface texture heterogeneity as noted from SEM results (Figure C) resulting in more surface area for resin bonding and better chemical bonding. These results are in agreement with Dapieve et al.<sup>13</sup> who concluded that for stable fatigue performance of adhesively cemented lithium disilicate restorations, a ceramic surface treatment that promotes micromechanical interlocking and chemical bonding is required. The fatigue performance of the one-step ceramic primer/conditioner was comparable to that of the 5 percent hydrofluoric acid + coupling agent, Levartovsky et al.<sup>35</sup> also concluded that EP treatment did not differ substantially in SBS values when compared to 9% HF for 20 seconds.

On the other hand, etching with acidulated phosphofluoride showed the lowest microtensile bond strength mean values. This may be due to, APF gel etching producing minimal surface topography change and surface roughness. Only a few small pores and undercuts were created as a result, and this is clear in the SEM results (Figure B).

This result is in line with Santos et al.<sup>36</sup> who explained that APF produces an insufficient, uneven, micromechanically retentive surface, but HF can produce the most prominent etching pattern on acid-sensitive ceramics. This is not in line with Barjaktarova-Valjakova et al.<sup>23</sup> and Mallikarjuna et al.<sup>4</sup> who discovered that there was no statistically significant difference in the surface roughness ( $m$ ) and bond strength (MPa) of lithium disilicate discs

(samples) etched with 1.23 percent APF gel and 1 percent APF gel for 10 minutes and etched with 9.6 percent HF for 1 minute. Results of this research work also showed that tetrabutylammonium dihydrogen trifluoride (TDTF)+ silane surface treatment showed a higher bond strength to APF although being statistically non-significant  $p=0.065$ , it can be also noted the relative resemblance in their surface morphology regarding fewer porosities (Figure B and D), that do not permit adequate penetration of the adhesive resin cement. Tetrabutylammonium dihydrogen trifluoride (TDTF) + silane surface treatment had very weak bond strength compared with HF acid surface treatment. This was not in agreement with Ruyter et al.<sup>37</sup> who concluded that good adhesion to zirconia can be achieved by a procedure including etching with selected melted fluoride compounds such as ammonium hydrogen difluoride and potassium hydrogen difluoride.

### Conclusions:

Using Hydrofluoric acid with primer application and Monobond Etch with Prime significantly increased bond strength to lithium disilicate ceramic compared to Tetrabutyl ammonium hydrogen difluoride and acidulated phosphate fluoride.

Acidulated phosphate fluoride demonstrated the lowest microtensile bond strength value when used for surface treatment of lithium disilicate ceramic.

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