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# EFFECT OF SURFACE PRE-TREATMENTS AND THERMOCYCLING ON MICROTENSILE BOND STRENGTH OF LITHIUM DISILICATE GLASS CERAMICS TO SELF ADHESIVE RESIN CEMENT

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## *ABSTRACT*

**Purpose**: This invitro study investigated the effect of lithium disilicate surface treatment methods and thermocycling condition on the bond strength to resin cement.

**Material and methods**: 12 Lithium disilicate ceramic blocks (7x9x6 mm) were fabricated from IPS e.max CAD blocs. Specimens were allocated in 3 groups (n=4), according to type of surface treatment: ***Group HFI:*** etched with 5 % HF acid followed by Monobond plus, ***Group HFP:*** etched with 9% HF acid followed by Monobond plus and ***Group MEP:*** treated using self-etch primer (Monobond Etch & Prime). Composite blocks of equal size were fabricated and bonded to ceramic blocs using self-adhesive resin cement. Specimens were embedded in acrylic resin blocks then sectioned into beam-shaped specimens (1x1mm). Each group was further subdivided into two subgroups according to aging by thermocycling *Subgroup Immediate (I)*: stored in distilled water at 37$^{o}$ C For 24 hours then tested immediately and *Subgroup Thermocycled (TC)*: stored then subjected to 5000 thermocycles. The µTBS were presented in MPa and failure mode of debonded specimens were examined by stereomicroscope and SEM to investigate surface characterization.

**Results:** The µTBS mean values ranged between (20.19 and 23.09 Mpa) for (I) subgroups and (14.93 and 15.45 Mpa) for (TC) subgroups. HFP-I showed the highest µTBS (23.09MPa). Thermocycling significantly affected the bond strength results (p=0.001) of all groups.

**Conclusion:** HF Etching of Lithium disilicate with increased concentration followed by silane application is optimal for ceramic/resin bonding. Moreover, Single-bottle self-etch ceramic primer proved to be a good alternative protocol.

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## INTRODUCTION

All ceramic restorations are biocompatible materials that can successfully simulate the visual characteristics of natural tooth structure. currently, a wide range of ceramic systems and materials are available in the market for use in dentistry.[1] Glass ceramics among the different ceramic materials are designed to offer high translucency, exceptional aesthetics, optimal strength, wear resistance, low thermal conductivity, and hardness that mimic that of natural teeth.[2]

When it comes to current prosthetic dentistry which demands good aesthetic and mechanical capabilities, Lithium disilicate ($LS_2$) is a category of glass ceramics which is considered one of the materials of choice owing to the remarkable properties and versatility as it could be fabricated using a hot-pressing technique, or CAD-CAM technologies.[3]

It may be essential to select the appropriate bonding methods for each type of glass-ceramic due to the variation in their chemical composition and microstructure.[4] Because of the presence of silica, lithium disilicate is an acid-sensitive ceramic that ensures a high strength of adhesion to the substrate via both chemical and micromechanical bonding mechanisms.[5]

Hydrofluoric acid (HF) etching is currently considered the most common surface treatment procedure for glass-based ceramics. HF etching for 60 seconds is requested for feldspathic and leucite-based ceramics, while Lithium disilicate require shorter time which is 20 seconds (using 5% concentration).[6] The efficiency of Hydrofluoric acid etching is affected by the acid solution's concentration, etching time, temperature, and dilution. [7] Several laboratory investigations have studied the effect of various concentrations of HF acid and etching times on lithia-disilicate based ceramic. As a result, there is no clear consensus on the best etching protocol for glass ceramics, particularly for lithium disilicate glass ceramics.[5-7]

Since 1977, Silane coupling agent has been used to improve the bond between etched ceramic surface and resin cement through the formation of siloxane bond and increasing ceramic surface energy. [8] Bonding of lithium disilicate with a self-adhesive cement without previous HF etching is not recommended as LD benefits from surface treatment with HF and silane application, regardless of the type of resin cement used.[9]

Using different surface pretreatment techniques, bond strength of glass ceramic to resin luting cements varied dramatically. For example, the procedure that produced greater values with feldspathic and lithium disilicate ceramics was the application of HF acid followed by silane.[10] A relatively newly developed universal primer (Monobond Plus) which is addressed by manufacturer to be a truly universal primer which contain different functional groups; phosphoric acid methacrylate, silane methacrylate, and sulfide methacrylate promoting an adhesive and durable bond between luting composites and all indirect ceramic restorations.[11]

Considering the toxicity posed by hydrofluoric acid, Monobond Etch & Prime has been introduced as single-component and one-step self-etching ceramic primer for surface treatment of glass ceramics. The manufacturer claims that this primer is less harmful because there are fewer steps in the application process than in the conventional protocol which requires 2 steps (HF etching then silane coupling agent).[12]

Colombo et al.[13] showed that Self-etching ceramic primer (MBEP) produced fewer surface alterations and comparable bonding strength compared to separate HF etching and silane application. Also, Lyann et al.[14] concluded that, for the surface treatment of lithium disilicate glass ceramics, Monobond Etch & Prime was proved to be a possible substitute for the combination of hydrofluoric acid and Monobond Plus.

Surface pretreatment of ceramic restoration is an important factor that affect the resin/ceramic interface bond strength. Moreover, thermocycling is a deteriorating factor that challenges the durability of bond strength. Hence, this invitro study was to investigate the effect of different surface treatment of lithium disilicate glass ceramic followed by aging condition on the microtensile bond strength to adhesive resin cement.

## MATERIALS AND METHODS

### Glass ceramic specimen fabrication:

Twelve Lithium disilicate ceramic blocks were milled from (IPS™ e.Max CAD; Ivoclar Vivadent) with a dimension of (7x9x6 mm). Subsequently and according to the manufacturer's instructions, all ceramic blocks were crystallised in a Programat ceramic furnace (P500; Ivoclar Vivadent). Low-speed diamond polisher (OptraGloss; Ivoclar Vivadent) was used for polishing of all blocks. Then, all blocks were cleaned in ultrasonic bath for 10 minutes in distilled water and dried for 60 seconds with oil-free air.

According to the type of surface treatment, specimens were randomly assigned to 3 groups (n=4) using Excel software (Excel 2019; Microsoft Corporation). Each group was further subdivided into two subgroups according to aging by thermocycling. The study design is shown in (fig1). The protocol of the study was reviewed and approved by Dental Research Ethics Committee, Faculty of Dentistry, Mansoura University with an approval serial number (A0103023FP).

### Ceramic specimens surface treatments

***Group HFI:*** 5% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) was used to etch ceramic specimens for 20 seconds. HF acid was washed for 30 seconds with an air water spray before being air dried for 30 seconds. A universal primer containing 10-MDP (Monobond Plus, Ivoclar Vivadent)) was rubbed on the specimens' surface with an application time of 15 seconds and left to react for another 60 seconds.

***Group HFP:*** 9% hydrofluoric acid (Porcelain Etch; Ultradent Products, Inc) was used to etch ceramic specimens for 20 second. HF Acid was washed, dried and Monobond Plus was applied as in the HFI group.

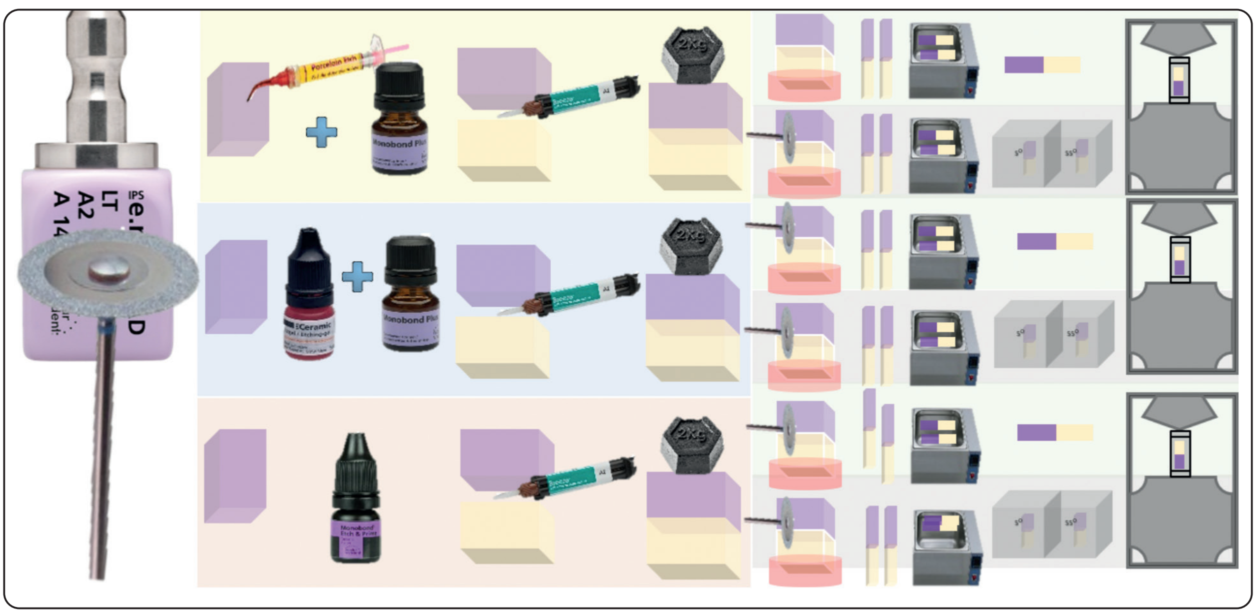

Fig. (1) Study design featuring ceramic specimens fabrication, grouping and bong strength test.

***Group MEP:*** Ceramic specimens were treated using single-component self-etch ceramic primer (Monobond Etch and Prime, Ivoclar Vivadent). MEP was applied with a microbrush to the specimens' surface for 20 seconds and allowed to react for another 40 seconds. It was then washed with an air water spray for 30 seconds before being air dried for 30 seconds.

**Composite blocks fabrication**

In a putty index duplication of ceramic blocks, sex increments of composite resin (Beautiful II; shofu, Japan) were built up. Each increment was applied in a thickness of 1 mm then light cured with a LED light curing unit (BlueLex; Monitex, Taiwan) with power density of 1000 mW/cm$^2$ for 40 seconds.

**Cementation procedures**

Composite resin blocks were sandblasted using 50 μm Al2O3 particles (SHERA Werkstoff-Technologie,Germany) for 15 seconds using 2 bars pressure with 10 mm distance using Renfert Basic sandblaster (Renfert, Germany). Sandblasted composite were bonded to treated lithium disilicate ceramics specimens using adhesive resin cement (Breeze, Pentron, USA) under a load of 2 Kgm using custom-made cementation device. Micro brushes were used to remove excess resin. Then the assembly was light cured using LED light-curing unit (BlueLex; Monitex, Taiwan) for 40 s and the load was continued constantly for 10 min

**Specimen preparation for the Microtensile test:**

All specimens were embedded in the center of auto polymerizing acrylic resin blocks (Acrostone, Egypt). Ceramic/composite blocks were sectioned into beam-shaped specimens (1x1 mm) using a low-speed saw machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). Each group external beams, as well as any beams have any type of defects, were discarded.

**Storage and Thermocycling**

Each group was then subdivided into two sub-groups based on aging by thermocycling:

*Subgroup Immediate (I)*: Half of the sectioned beam-shaped specimens were stored for 24 hours in distilled water at 37$^o$ C then tested immediately.

*Subgroup Thermocycled (TC)*: The other half of sectioned beam-shaped specimens were stored for 1 month in distilled water at 37$^o$ C and subjected to 5000 thermocycles between 5$^o$C 55$^o$C using thermocycling device (thermocycler, ROBOTA, Alexandria, Egypt). Each thermal cycle composed of a 1-minute cold bath followed by a 1-minute hot bath with a 30 second dwell time. Then beam-shaped specimens were air dried and tested.

**Microtensile bond strength test**

Each beam-shaped specimen were positioned in a designed jig which is connected to a universal testing equipment (Model LRX-plus;LIoyd Instruments Ltd., Fareham,UK) with cyanoacrylate gel glue. A tensile load was applied at 0.5 mm/min crosshead speed, till the occurrence of failure. The μTBSs were presented in MPa which were calculated by dividing the load at failure (N) by the adhesive area(1mm$^2$). Pre-test failures were documented if specimens failed before being tested.

**Failure mode**

Debonded specimens were examined by Stereomicroscopy (Stereomicroscope SZ2-ILST, Olympus co., Japan) to determine the mode of failure, which may be adhesive, cohesive or mixed failure.

**Scanning electron microscope (SEM)**

One specimen from each group was selected and was examined using scanning electron microscopy (JEOL.JSM.6510LV) at different magnifications (500x, 1000x, 2000x, 5000x) before bonding to show changes in the surface microstructure. Also, One specimen was selected from each sub-group after debonding for SEM examination to investigate the surface and mode of failure in debonded specimens.

### Statistical Methods

Data was analyzed using SPSS software, version 22 (SPSS Inc., PASW statistics for Windows version 22 SPSS Inc., Chicago). Shapiro Wilk test was used to check normality, then quantitative data were presented using mean and standard deviation for normally distributed data. Significance of obtained results was judged at (≤0.05) level. Paired t test was used to compare 2 paired readings distributed data. To compare more than 2 independent groups, One Way ANOVA was used with Post Hoc Tukey test to detect pair-wise comparison. Two Way ANOVA test was also used to study the combined effect of 2 independent factors on dependent continuous outcome with estimation of R2.

## RESULTS

The mean microtensile Bond Strength (µTBS) and standard deviation for all tested group are presented in. (Table 1)

The mean µTBS values ranged between (20.19 and 23.09 Mpa) for immediately tested subgroups and (14.93 and 15.45 Mpa) for thermocycled subgroups.

For immediately tested subgroup without thermocycling, HF (9%) etching and monobond plus application showed the highest µTBS mean value (23.09±2.53) followed by HF (5%) etching and monobond plus application (21.52±2.53) while Monobond etch and prime showed the lowest µTBS mean value (20.19±3.06). For thermocycled subgroups, Monobond etch and prime showed the highest µTBS mean value (15.45±4.55) while HF (5%) etching and monobond plus application showed the lowest µTBS mean value (14.93±3.31) as shown in (fig 2).

For immediately tested subgroups, One-way ANOVA test showed that there was a statistical significant difference between HFP and MEP groups (P=0.012*). While there were no statistically significant differences between HFI and both HFP & MEP subgroups but increasing the etching concentration in HFP group showed relatively higher µTBS. For thermocycled groups, One way ANOVA test showed that there were no statistically significant differences between all tested groups.

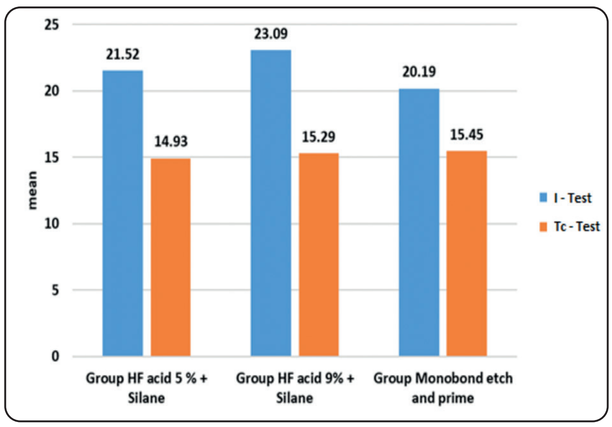


Fig. (2): Means values (in MPa) of the µTBS test of the studied groups.

TABLE (1) Showing Means and Standard deviation SD of the µTBS (in MPa) test

| | Group HFI | Group HFP | Group MEP | Test of significance |
|---|---|---|---|---|
| Immediate | 21.52±2.53 | 23.09±2.5$^{3}$A | 20.19±3.06$^{A}$ | F=4.27 **P=0.012*** |
| Thermocycled | 14.93±3.31 | 15.29±3.53 | 15.45±4.55 | F=0.07 P=0.932 |
| Paired t test | T=11.19 **p<0.001*** | T=8.43 **p<0.001*** | T=5.18 **p<0.001*** | |
| % of change | 30.6% | 33.8% | 23.5% | |

***Similar superscripted letters denote significant difference between studied groups within same row by post Hoc Tukey test , F:One Way ANOVA test , *statistically significant***

Paired t-test showed that there was a high statistical significant differences between immediately tested subgroups and the same subgroups after thermocyling ($p<0.001$). Analysis with serial Two-way ANOVA to test the combined effect of surface treatment and thermocycling on µTBS were not significant ($p=0.208$). (Table 2)

SEM of specimens before bonding showed changes in the surface microstructure which varied according to the type of surface treatments. Etching with 9% and 5% HF acid resulted in the dissolution of the glassy phase with apparent surface irregularities including multiple micropores, grooves, and striations which were evident in HFP and HFI respectively (fig 3a, 3b). While, MEP showed less morphological surface changes with distinct residual layers (fig 3c).

Debonded specimens were examined by Stereomicroscopy to identify the type of the failure which is presented in (Table 3) and shown in (fig 4). Adhesive failure at ceramic/resin interface was recorded as the most common pattern of failure in debonded specimens, while mixed failure was relatively higher in both HFP-I and MEP-TC. Cohesive failures of resin composite presented the lowest frequency among all debonded specimens. Debonded specimens were further examined using SEM at different magnifications to investigate the mode failure as shown in micrographs (fig 5).

TABLE (2). Summary of overall TWO way ANOVA test

| Source | Type III sum of Squares | df | Mean Square | F | p value |
|---|---|---|---|---|---|
| **Corrected Model** | 979.586 | 5 | 195.917 | 17.757 | **.001*** |
| **Intercept** | 30510.893 | 1 | 30510.893 | 2765.425 | **.001*** |
| **Surface treatment** | 29.703 | 2 | 14.851 | 1.346 | .266 |
| **Thermocycling** | 914.573 | 1 | 914.573 | 82.894 | **.001*** |
| **Surface treatment *thermocycling** | 35.310 | 2 | 17.655 | 1.600 | .208 |
| **Error** | 926.771 | 84 | 11.033 | | |
| **Total** | 32417.250 | 90 | | | |
| **Corrected Total** | 1906.357 | 89 | | | |

***a. R Squared = .514 (Adjusted R Squared = .485)***

TABLE (3). Represent different types and numbers of failure in debonded ceramic specimens

| | Adhesive failure Ceramic/resin interface | Cohesive failure Within composite resin | Mixed failure Adhesive and cohesive |
|---|---|---|---|
| **HFI-I** | **7** | **2** | **6** |
| **HFI-TC** | **9** | **1** | **5** |
| **HFP-I** | **6** | **1** | **8** |
| **HFP-TC** | **8** | **0** | **7** |
| **MEP-I** | **10** | **0** | **5** |
| **MEP-Tc** | **5** | **2** | **8** |

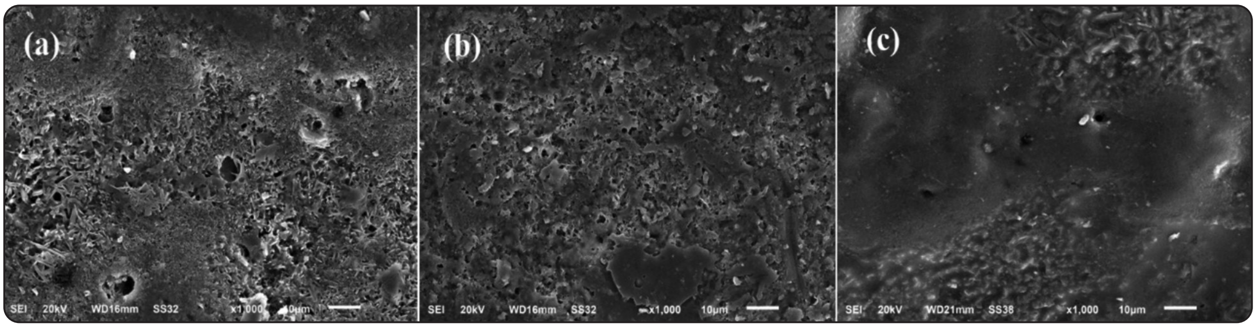


Fig. (3): SEM of ceramic surfaces before bonding at x1000 magnification showing different microstructures induced by etching using 9% HF (a), 5% HFP (b) and MEP (c)

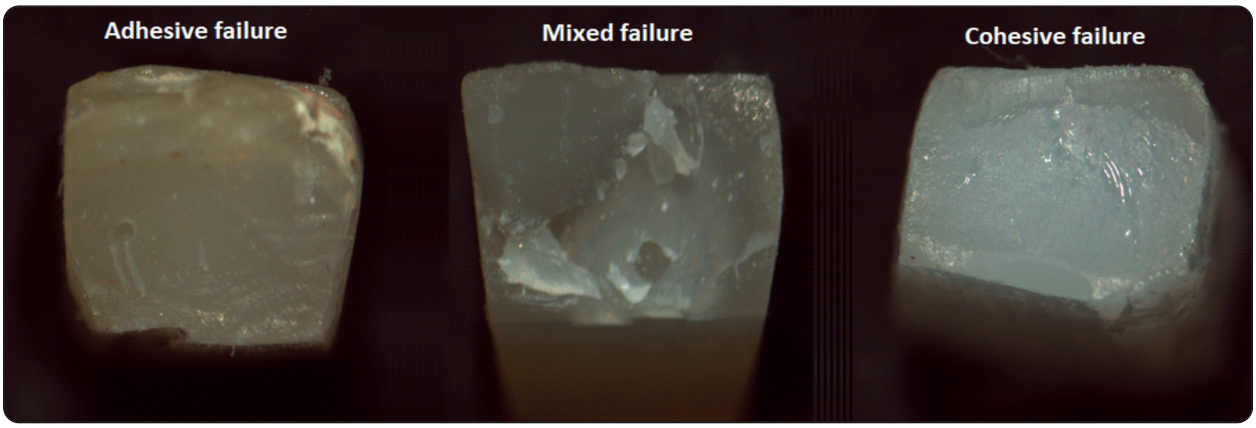


Fig. (4): Stereomicroscopic pictures of debonded ceramic specimens represent different types of failure

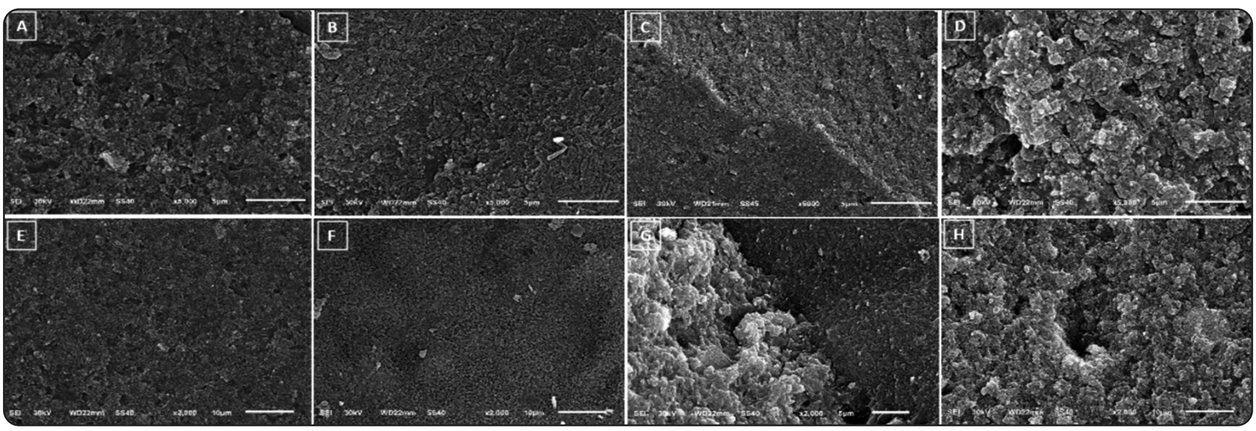


Fig. (5): SEM of debonded ceramic surfaces (A-D) at x5000 and (E-H) at x2000 magnifications showing different modes of failure: (A,B,E,F) Adhesive failure at ceramic/resin interface, (C,G) Mixed failure and (D,H) Cohesive failure within composite resin

## DISCUSSION

The success of indirect ceramic restorations is determined by numerous factors, including restoration design, material selection, occlusal scheme and cementation protocol.[1] Nowadays, glass ceramics are widely used owing to their improved mechanical properties associated to better microstructures and new fabrication methods. The optimal mechanical properties of these materials are reflected in the good durability and survival of such dental restorations.[15]

IPS e.max Press (lithium disilicate based formulation) was released on the market in 2005 as a result of improvements in the processing parameters that allowed the formation of smaller and more evenly distributed crystals, this material was processed by heat-pressed technique and exhibited improved both mechanical and optical properties.[16] Later on, (IPS e.max CAD) ceramic blocks were introduced thanks to advancement of (CAD-CAM) technologies. In this invitro study, Lithium disilicate (IPS e.max CAD) ceramic blocs were used which exhibit fracture resistance suitable for both anterior and posterior monolithic restorations.[3]

In this invitro study, to standardize the specimens, ceramic blocks were bonded onto composite resin blocks instead of teeth substrate due to the heterogeneous microstructure of dentin and to avoid the anticipated greater source of variability of dental substrate compared to a manufactured material such as the composite blocks.[17]

Success of all ceramic restorations is mainly dependent on achieving a reliable and durable bond at ceramic/resin interface.[18] The gold standard for surface treatment of feldspathic and lithium disilicate ceramics has been considered to be HF acid etching, followed by silane application. [6,10,19] These findings are consistent with the manufacturer's bonding protocol of nearly all glass ceramics, and is supported by International Academy of Adhesive Dentistry (IAAD) guidelines.[20]

HF partially dissolves the glass phase of a ceramic matrix, which increases the surface area of contact and interactions between the resin cement and glass ceramic.[21] While silane application increases ceramic surface energy and enhance the cement wettability, thereby inducing microscopic interactions at the interface.[8] In the present study, the appropriate degree of glass matrix dissolving at two hydrofluoric acid concentrations, with the highest bonding performance, has been investigated. Both concentrations (5% and 9%) were applied for an equal time (20 seconds) as recommended by manufacturer. SEM showed adequate etching pattern and proved that this time was optimal to properly condition the ceramic surface for adhesion to resin cement. In accordance with these results, Verissimo et al.[22] concluded that, different etching time had no significant effect on the bond strength of lithium disilicate machinable ceramics to resin cement.

Also, Puppin-Rontani et al.[7] found that, greater concentrations do not require prolonged etching durations and the generated ionised HF is sufficient to dissolve the glass matrix adequately in 20 s, and so, increasing the etching times for 5%, 7.5%, and 10% HF concentrations did not result in higher SBS values. Moreover, Hooshmand et al.[23] concluded that prolonged etching with hydrofluoric acid could impair the mechanical properties of glass ceramic materials, as increasing the etching has a weakening effect on both leucite and lithium disilicate glass ceramic systems.

On the other hand, Avram et al.[24] found that machinable e max CAD ceramic achieved superior results when etched with 9.5% HF acid for 60s. Moreover, no significant differences were found between the bond values of etching time (30 s and 60 s) which led to the fact that; for lithium disilicate ceramic bonding, it is possible to use either a higher concentration HF acid with the recommended manufacturer' time or a higher etching time with the acid concentration recommended by manufacturer.

In the present study, Etching lithium disilicate glass ceramic with HF (5%) for 20 s enhanced the adhesion between etched ceramic and resin cement with a mean μTBS (21.52 Mpa) which is, relatively higher than that of MEP group (20.19 Mpa). In agreement with these findings, El-Damanhoury and Giantantzopoulou[10] revealed that, the use of 5% hydrofluoric acid etching followed by Monobond Plus improved the microtensile bond strength in comparison to conditioning with Monobond Etch and Prime. In another study by Murillo et al.,[25] Combination of 5% HF acid and silanization proved to be the gold standard treatment for Lithia-based glass-ceramic materials.

Increasing the etching concentration was also investigated in the present study through etching using (9 %) HF acid which demonstrated the highest μTBS value (23.09 Mpa). These results may be attributed to greater etching aggressiveness and better surface conditioning pattern for lithium disilicate which was observed in SEM micrographs, showing that glass ceramics benefits from increases in etching concentration. Also, Almiro et al.[26] concluded that, in comparison to Monobond etch and prime, etching lithium disilicate surface with 9.5% HF for 60 seconds and then applying Bis-Silane achieved the highest μTBS. Also, in an invitro study[19], higher values of shear bond strength for e.max CAD specimens etched with 9.5% HF acid for 60 s than for those etched with 5% HF acid for 20 s, but not treated with silane.[19]

Although HF etching combined with silanization has been proved to be an ideal surface treatment for lithium disilicate ceramics, this bonding protocol requires at least three clinical steps, making the technique more sensitive and less user - friendly.[27] Moreover, HF acid is extremely toxic and can cause serious health problems.[28] Also, it produces insoluble surface byproducts consisting of salts of silica fluoride which could disrupt the resin bond strength.[29]

For the above-mentioned reasons, self-etching glass-ceramic primer (MEP) has been used in this study combining the surface etching and silanization of lithium disilicate ceramics into a one step. It contains a methacrylate-functionalised silane, a methacrylate phosphate monomer and tetrabutyl ammonium dihydrogen trifluoride (TADF) which is much weaker than hydrofluoric acid. This was apparent through weaker etching pattern demonstrated by SEM micrographs and the lowest immediate μTBS value (20.19 Mpa). However after thermocycling, MEP group demonstrated the highest μTBS value (15.45 Mpa). In agreement with these results, Tribst et al. [30] concluded that the self-etch ceramic primer is ideal for maintaining adhesive and durable bond to lithia- based glass ceramics after ageing.

Using the single-bottle self-etching glass-ceramic primer reduces technique sensitivity, less time consuming and found to be a good alternative approach to the conventional ceramic surface treatment.[31] However, Almiro et al.[26] found that, the group in which self-etching primere (MEP) was applied have prematurely failed as the bond strength was not enough for the specimens to withstand sectioning into sticks for microtensile bond testing which may be attributed to absence of micromechanical retention.

Comparing the shear bond strength test to tensile bond strength test, the latter seems to be a more reliable method of assessing the resin-ceramic bond strength.[32] Because of the uneven stress distribution along the bonded interface, which increases the possibility of receiving biassed data, the microshear bond strength test has been subject to criticism.[33] In this study, the bond strength was evaluated using microtensile bond testing at interface between ceramic and resin, that was presented by Sano[34] in an effort to remove non uniform stress distribution within the zone of adhesion. This variant of the method, allowed obtaining multiple specimens from a single block, which would provide more consistent results as the testing variance is usually reduced to 10 - 25%.[35]

Intraorally, bonded indirect ceramic restorations are exposed to complex aging that is associated with changes in temperature, immersion in saliva, occlusal forces and beverages exposure.[36] To simulate intra-oral conditions, In this invitro study, the effect of thermocycling on microtensile bond-strength at ceramic/resin interface was tested. Results showed that thermocycled ceramic had an average bond strength of (14.93 and 15.45 Mpa) while immediately tested groups showed an average of (20.19 and 23.09 Mpa) which proved that thermocylcling has a significant effect on the bond strength of LD2 ceramic to resin cement. In agreement with these results, Guarda et al. [37] concluded that, thermocycling cause deterioration of bonding at the ceramic/resin interface, and this should be interpreted as an indication of the potentially low long-term stability of such bonding. Also, Kamada et al.[38] , showed that thermocycling and water storage adversely affect the bond durability between the silanated ceramic surface and the resin cement.

## CONCLUSION

*Within the limitations of this study, the following conclusions could be drawn:*

1- Etching Lithium disilicate with hydrofluoric acid with increased concentration followed by silane application proved to be the ideal protocol for ceramic/resin bonding

2- Considering the potential hazards associated with HF acid, Single-bottle self-etch ceramic primer found to be a good alternative protocol for surface treatment of Lithium disilicate ceramics.

3- Ageing by thermocycling adversely affect the durability of ceramic/resin interfacial bond which is relatively enhanced by using MEP.

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