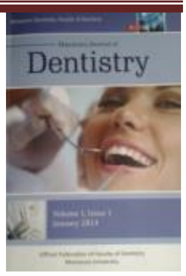


# *Bond strength of resin cements to pressable ceramic: effect of different surface conditioning methods*

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***Abstract:***
**PURPOSE. This study aimed to evaluate the bond strength of two resin cements to lithium disilicate ceramic after different etching and priming protocols. MATERIALS AND METHODS. 64 lithium disilicate discs (IPS e.max press) were fabricated by pressing technique then divided into two groups (n=32) group (I) multi-step adhesive resin cement (Multilink® N); group (II) self-adhesive resin cement (Maxcem Elite®). Each group was subdivided into four subgroups (n=8) according to surface treatment material; (A): Monobond Etch & Prime, (B): Hydrofluoric acid, (C): Acidulated phosphate fluoride (D): Ammonium hydrogen difluoride. Composite resin discs (Nexcomp®) were cemented to treated ceramic discs. All bonded specimens stored in water bath for 5 months and subjected to 5000 thermal cycles. Shear bond strength (SBS) test was performed using a universal testing machine. Scanning Electron Microscope (SEM) was used for specimens' examination. RESULTS. Statistical analysis were done using two-way ANOVA and serial one-way ANOVAs followed by Post Hoc Tukey-HSD test at $\alpha$ =0.05. The highest SBS mean value 17.05±3.99 MPa, 16.59±2.27 MPa was obtained for APF and HF at multi-step adhesive cement. On the other hand, AHDF with self-adhesive resin cement showed the lowest SBS mean value 6.71±1.46 MPa. There was statistically significant difference between AHDF and other used materials. CONCLUSION. Multi-step adhesive resin cement showed superior bond strength than self-adhesive resin cement. Also, HF acid and APF were preferred to be used more than Monobond Etch & Prime for ceramic surface treatment as they significantly affected the durability & bond strength regardless used cement type.**
**Keywords: Hydrofluoric acid, Multilink N, Ammonium hydrogen difluoride, self-adhesive resin cement, lithium disilicate ceramics.**.

## *Introduction*

IPS e-max ceramics consist of about 65% by volume of interlocking lithium disilicate crystals scattered in a glassy matrix, it is a silica-based ceramic available in milled and pressed forms.1 It is suitable to be used for the fabrication of monolithic or veneered restorations in the anterior and posterior areas, because of its natural tooth color and excellent light-optical properties.2
Pressable ceramics demonstrated great success and deserve special attention for increasing the limited knowledge database regarding optimal adhesive bonding techniques.3 Dental lithium disilicate have superior features such as chemical stability, biocompatibility, low thermal conductivity, high pressure resistance, translucency and fluorescence.4
Lithium disilicate glass ceramics are a new generation of heat-pressed ceramics that provide high fracture strength and bending resistance, when compared with feldspar ceramics. The presence of lithium disilicate crystals improves the overall characters of the final ceramic restoration. These features enable the fabrication of onlays, inlays, laminate veneers, crowns and three-unit fixed dental prosthesis bridges extending to the first molar.5 Advances in adhesive dentistry have resulted in the investigation of different surface treatments to achieve high bond strengths.6 Ceramic surface can be conditioned by several methods such as: air-borne particle abrasion, acid etching and silica coating.7
Etching with hydrofluoric acid (HF) dissolves surfaces that contain a glass matrix and crystals. In addition, it helps to increase the surface energy for the implementation of acidified surface silane agent, as a result, the chemical bond between the organic matrix and inorganic matrix of resin cement is enhanced.8 On the other hand, etching leaves insoluble salts (silica-fluoride) in the surface that weakens the bond, thus post-etching ultrasonic cleaning is mandatory with alcohol.9
Acidulated phosphate fluoride (APF) was shown to etch different types of dental materials such as dental composites, ceramics, amalgam and dental cements in vitro.10 It produced only a few shallow pores and undercuts. Moreover, APF gel is considered safe and effective substitute for etching of ceramic surfaces.11 Ammonium hydrogen difluoride also used as glass etchant and it has been considered as an intermediate in the production of Hydrofluoric acid from hexafluorosilicic acid.12
The application of a silane coupling agent after ceramic etching is recommended to achieve chemical bonding.13 Silane coupling agents have a major role as mediators to fulfill the clinical requirements for durable bonding. Nowadays, surface conditioning of dental materials combines with silanation is a standard step before adhesive bonding with resin cement.14 Recently multi-purpose primers are introduced into the dental market to enhance chemical bonding to different prosthetic materials, ceramics, metal alloy, composites and acrylic resin.15
Resin cements are low viscosity composite materials with filler distribution and initiator content adjusted to allow for low film thickness and suitable working and setting time. Most resin cements are radiopaque and release small amount

of fluoride. The resin cements are classified according to curing mode as auto polymerized, light-polymerized and dual-polymerized.16

Multi-step adhesive resin cements are time consuming, technique sensitive, and consequently may compromise bonding effectiveness. On the other hand, self-adhesive resin cements are luting agents with a very simple application procedure, combining the advantages of glass ionomer (adhesion, fluoride release) with mechanical properties of resin cements. They are indicated for cementation of cast alloy restorations, metal ceramic crowns and bridges, ceramics (except veneers) and indirect composite restorations.17

Therefore, the main objective of this in-vitro study was to investigate the shear bond strength of resin cements to pressable lithium disilicate glass ceramic, and the effect of different surface treatment methods on bonding strength of pressable ceramic. The present research work performed under the null hypothesis that, the bond strength of two different resin cements to lithium disilicate ceramic would not influenced by different etching and priming methods.

Materials and methods:

Specimens' preparation:

sixty-four discs (n = 64) of lithium disilicate glass ceramic (8 mm in diameter and 4 mm in thickness) were fabricated by pressing of the discs from Lithium disilicate glass ceramic ingots (IPS e.max Press, Lot No. X48904, Ivoclar Vivadent, Schaan/Liechtenstein). Ceramic discs were designed by fabrication of a wax pattern disc (8×3 mm in dimensions), the wax disc was scanned using Ceramill Map 400+ (Amann Girbach, Germany) for obtaining standardized ceramic discs. EXOCAD designing software was used to check the accuracy of the specimen dimensions and to replicate 64 discs form, then milling of 64 wax discs using Ceramill wax white (Amann Girbach, Germany) by Ceramill mikro device (Amann Girbach, Germany). Wax discs were sprued and by using lost-wax technique, pressing of the discs at 700°C from Lithium disilicate glass ceramic ingots by using Programat EP 3010 Press furnaces machine (Ivoclar, Vivadent, Schaan, Liechtenstein) according to manufacturer's instructions.

Specimens grouping and surface treatment:

Ceramic discs were divided into 2 main groups (n=32) according to the type of the resin cement that used: Group (I): Multistep adhesive resin cement (Multilink® N, Lot No. W40132, Ivoclar, Vivadent, Schaan, Liechtenstein). Group (II): Self-adhesive resin cement (Maxcem Elite, Lot No. 6593728, Kerr,Italy). Each group was subdivided into 4 subgroups (n=8) according to used method of surface treatment before primer application:

Subgroup (A): The discs were etched by Monobond Etch & Prime (MEP, Lot No. W40212, Ivoclar, Vivadent, Schaan, Liechtenstein) for 1 minute. Subgroup (B): The discs were etched by Porcelain Etchant Gel (10% Hydrofluoric acid (HF), Lot No. RF20U, Prime-Dent Chicago, USA) for 1 minute. Subgroup (C): The discs were etched by Porcelain Etchant Gel (1.23% Acidulated phosphate fluoride (APF), Mirage, Myron International, Kansas City, USA) for 3 minutes. Subgroup (D): The discs were etched by ammonium hydrogen diflouride (AHDF, Lot No. S12541307 LobaChmie, India) for 5 minutes at 170° C, then cleaned by water bath.

Application of Ammonium hydrogen difluoride slurry ($NH_4HF_2$) was prepared by grinding of crystal particles into fine powder using mortar and pestle then 4.2 mg $NH_4HF_2$/ ml distilled water was mixed forming viscous slurry. The mix was applied onto one surface of each ceramic disc using small brush.18

The discs with Ammonium hydrogen difluoride slurry were heated in a preheated furnace (VITA VACUMAT 40 T) at a temperature of 170° C for 5 minutes. Etched specimens were steam cleaned afterwards.19

Subgroup A were etched and primed with single bottle Etch &Prime, while the other subgroups, B, C and D after etching procedure a universal ceramic primer (Monobond N, Lot No. X00048, Ivoclar Vivadent, Schaan/Liechtenstein) were applied. All Etched specimens were steam cleaned, ultrasonically rinsed by 95% alcohol (5 min) and dried in air before cementation.19

Composite resin discs preparation:

A total number of 64 composite resin discs were fabricated using metal ring with central hole (4 mm internal diameter and 3 mm thickness),20 then filled with composite resin incrementally (Nexcomp, Lot No. NXC1706191, META® BIOMED, Korea) to fabricate composite resin discs which were polymerized using light curing unit (liteQ LD-107, MONITEX, Taiwan) for 20 sec. for each increment. Then each composite resin disc was removed and inspected for any defects. One surface of each composite resin disc was treated by sandblasting using 50 µm Aluminum Oxide particles (SHERA ALUMINIUM OXID 50 µm, Lot No. 1799872, Werkstoff-Technologie, Germany).21

Cementation:

Composite resin discs were cemented to previously treated lithium disilicate glass ceramic discs using resin cements. Ceramic discs were secured to a specially designed device with lever system to deliver a constant load of 5 Kg for 5 minutes on the composite discs during cementation, excess resin cement was removed with a brush then curing was done using light cure unit from four directions for 40 sec. from each surface for a total of 160 sec. and the constant load was left for 10 min. After cementation, the specimens were stored in water at 37ºC for 5 months then all specimens were subjected to thermo-cycling in thermocycling device (THE-1100, SD-Mechatronik, Germany) for 5000 cycles. Each cycle consisted of 1 min in 5ºC cold bath and 1 min in 55ºC hot bath with a dwell time of 30 s, and then the specimens were air-dried.

Bond Strength measurement:

The bond strength between lithium disilicate glass ceramic discs and composite discs was determined by a shear bond test (SBS). This test was performed using Bluehill Lite Software from Instron (R) (Model 3345: Instron Industrial Product, Norwood, MA, USA) (Fig. 1). Shear test was designed to evaluate the bond strength, the shear strength was calculated by dividing the load at which failure occurred by the bonded surface area of the disc to give the bond strength in MPa.

Mode of failure evaluation:

The mode of failure was determined by examination of bonding surfaces of debonded discs by optical reflection microscope (S300II; Inoue Attachment Corp) at ×8 magnification and was divided into three types:22

1. Adhesive mode of failure: failure between the ceramic and resin cement (at the interface).

2. Cohesive mode of failure: failure took place in the cement layer or in composite resin disc.
3. Mixed mode of failure: including cohesive and adhesive failure.

Scanning electron microscope (SEM):
In order to investigate surface characterization of debonded lithium disilicate glass ceramic, one specimen from each subgroup was examined using SEM (JEOL.JSM.6510LV) at different magnifications (50x, 500x, 1000x, 2000x).
Statistical analysis:
Statistical analysis was conducted using the IBM SPSS software package version 22.0. (SPSS Inc. Chicago, Ill, USA). The normality of data was first tested with Shapiro-Wilk test, variables were presented as Mean ± SD (Standard Deviation). Statistical analysis of data was performed in several steps. Initially, descriptive statistics for each group results, then two-way ANOVA test was used to detect the effect of each variable (Resin cements and surface conditioning methods) on shear bond strength. Tukey (HSD) honest significant difference was used for multiple comparison between different groups. Results:
Shear bond strength results
In Table 1, the mean shear bond strength (SBS) and standard deviation for each tested group were calculated. APF with Multi-step adhesive resin cement group showed the highest SBS mean value (17.05±3.99 MPa) followed by HF with Multi-step adhesive resin cement (16.59±2.27 MPa). On the other hand, AHDF with self-adhesive resin cement showed the lowest SBS mean value (6.71±1.46 MPa). Multi-step adhesive resin cement showed higher mean SBS than Self-adhesive resin cement group. ($p=0.001$) regardless surface treatment used. The significance of the influence of either surface treatment or used cement was tested by One-way ANOVA test. Whenever ANOVA test showed significance, the Post-Hoc Tukey test was utilized for comparing the means of each two tested groups. The level of significance was established at ($p\leq 0.05$). Two-way ANOVA showed significant differences in the values of shear bond strength as a result of applying different surface treatment methods ($p<0.001$), as well as significant difference due to the type of cement used ($p<0.001$). However, the interaction between different surface treatments and cement was not significant. (Table 2) One-way ANOVA showed statistically significant difference between different types of surface treatment ($p<0.001$). There was statistically significant difference in the shear bond strength between tested groups with different cement types ($p=0.001$). There was statistically significant difference between AHDF and other types of surface treatment (HF, ME&P, APF) in self-adhesive resin cement and multi-step adhesive resin cement. There was a statistically significant difference between Self-adhesive and Multi-step adhesive resin cements with different types of surface treatment. (Table 3)
Mode of failure:
Failure patterns of all debonded specimens showed mainly adhesive failure pattern (34 specimens) followed by mixed failure pattern (23 specimens) and the least was cohesive failure pattern (7 specimens) as shown in (Table 4).

Scanning Electron Microscope (SEM) was used for investigation of surface characterization of debonded ceramic discs as shown in (Fig. 2).
Discussion:
Several all-ceramic materials have been available for dental restorations. Among these materials, Lithium Disilicate Glass Ceramic that considered one of the most popular material due to its high strength, good color stability, high resistance to wear, and high biocompatibility.23
The lost-wax and heat-pressing technology were used as the glass-ceramic comprises at certain volume of the glass phase, the material can be pressed into the mold under defined temperature and pressure conditions by viscous flow. 24 In this study we used lithium disilicate ceramic disc designed to simulate the core portion of all-ceramic restoration and used to represent the more complex clinical situation with same thickness can be performed and to minimize such inaccuracies.25
The clinical success of a ceramic restoration depends on the quality and durability of the bond between ceramic and resin cement. The multi-step system was used because is still the gold standard for the marginal adaption of tooth substance and restorative materials.26 On the other hands, Self-adhesive resin cements may combine the advantages of both adhesive and conventional luting agents which overcome the complex and technically sensitive of multi-step adhesive resin cements luting procedure. 27
The general protocol established for cementation is the etching with HF acid and the application of a silane agent. Although the surface treatment with HF acid is widely used and accepted for lithium disilicate ceramics because it increased surface area for micromechanical entanglement is promoted, improving the interaction between ceramic and resin cement with increased bond strength. 28
Etch & Prime as a single-component ceramic primer, has been introduced to the market, as an alternative to hydrofluoric acid etching/silane coupling agent routine treatment. This product integrates the etching and silane priming treatments in a single step. It has been shown to shorten the treatment time of the clinical steps by etching and silanating glass ceramic surfaces in one working step, free of the toxic HF acid, stable and retaining the original silanol activity after aging.29
Acidulated phosphate fluoride treatment increased the surface roughness of feldspathic porcelain, low-fusing porcelain and aluminous porcelain.30 APF gel may be used intra-orally as it was less toxic and safe for oral tissues and may etch or react with porcelain, glass ionomer, fissure sealant, and composite restorative materials.31
Ammonium polyfluoride salts have been discovered and used in industrial processes to treat and etch surface of silica-based materials and have produced smoother etching patterns than HF acid.32
Laboratory simulations of clinical service are often performed because clinical trials are costly and time consuming. Thermal-cycling is an in-vivo process often represented in these simulations, exposing the specimens to thermo-cycling regimens is a common technique for simulating hydrothermal aging. 33
In this study, shear bond strength test (SBS) was used because it is considered the ideal representative of typical clinical

stress. 34 SBS is the most commonly used test to screen new adhesive formulations according to their bonding effectiveness. This test gains its high popularity in companies and research institutes since no further specimen processing is needed after the bonding procedure; thus, it is the easiest and fastest method. 35

In this in-vitro study mode of failure was examined to understood in relation to underlying material properties (modulus, strength, and toughness) and geometrical factors (characteristic contact dimension, layer thickness). 36

The results of this study showed that, there was statistically significant difference between Ammonium hydrogen difluoride and HF acid , Etch &Prime and, also Acidulated phosphate fluoride in Self-adhesive and Multi-step adhesive resin cements. This may be due to, the strength and durability of the bond between ceramic and resin cement depends on the chemical composition of the ceramic system, and surface treatments are necessary to ensure adhesion between the luting agent and the ceramic surface.37 In addition, the composition of the ceramic determines which surface treatment is appropriate.38

Also, this research work revealed that, acidulated phosphate fluoride group showed the highest SBS mean value followed by Hydrofluoric acid with Multi-Step adhesive resin cement. This may be due to, APF gel etching produced minimal surface topography change and surface roughness. It produced only a few shallow pores and undercuts, whereas HF acid showed greater roughness and irregularity.10 These results supported by the study of Sayin et al., (2019)39 who showed that, minimal surface roughness can produced high bond strength and there was no correlation between the increase of surface roughness of porcelain and the bond strength. This study in line with Mallikarjuna et al., (2018)40 who reported that, etching with HF acid alone is not sufficient to produce a strong bond with dental ceramics. Also, lithium disilicate discs etched with 1.23% APF gel and 1% APF gel for 10 min. showed similar surface roughness and bond strength to those etched with 9.6% HF for a 1 min.

On the other hand, Ammonium hydrogen difluoride showed the lowest SBS mean values in both types of resin cements. This may be due to lower roughness values and the wettability modified by the surface treatments may influenced on the bonding ability and resulted in less micromechanical retention of resin cement.41 The ammonium hydrogen difluoride, in reaction with silica matrix creates some silicon tetrafluoride and ammonium fluoride this acid used as a glass etchant.42

This in-vitro study showed that, the Etch & Prime was higher in SBS than AHDF with self-etch and multistep adhesive resin cements. This may be due to removal of the glassy ceramic phase in Etch & Prime, which created the protruding domains or residual glass and surface texture heterogeneity, resulted in more surface area for resin bonding and promoting better chemical bonding. However, the AHDF promotes a weaker etching pattern in the ceramic surface.43

On the other hand, there was a statistically significant difference between Self-adhesive and Multi-step adhesive resin cements with using different types of surface treatment. Results showed that, Multi-step adhesive resin cement regardless used type of surface treatment showed higher SBS mean values than tested groups with Self-adhesive resin cement. This low bond strength recorded for self-adhesive resin cement may be related to the initial low pH and higher viscosity of the self-adhesive cements, the low bond strength recorded for the self-adhesive resin despite the cement's limited ability to demineralize and infiltrate.44 Other study was done by Upadhyaya et al., (2019) 45 who found that, multistep adhesive produced higher bond strength of all-ceramics than that obtained by self-adhesive resin cements. They explained this result due to lower degree of cure and higher water solubility as compare to multistep and existence of low-molecular-weight oligomers that allows water to penetrate the junction of resin cement and ceramic structure.

The null hypothesis of this in-vitro study that, the bond strength of two resin cements to lithium disilicate ceramic wouldn't influenced by different etching and priming methods was rejecte. One of the limitations of the present study is that, in-vitro studies do not reflect clinical conditions. In addition, only one type of lithium disilicate glass ceramic was evaluated, and the results may differ for other types of ceramic material.

Conclusion:

Within the limitations of the present in vitro study, the following conclusions could be drawn: The multi-step adhesive resin cement has a superior bond strength in comparison to self-adhesive resin cement when used for lithium disilicate ceramic bonding regardless the surface treatment used. Using of Hydrofluoric acid & Acidulated phosphate fluoride with primer application significantly increased bond strength to lithium disilicate ceramic compared to Ammonium hydrogen difluoride and Monobond Etch & Prime system. Ammonium hydrogen difluoride demonstrated the lowest SBS value when used for surface treatment of lithium disilicate ceramic regardless type of resin cements used.

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