ABSTRACT:

OBJECTIVE: To evaluate the influence of post hydrofluoric acid (HF acid) etch crystalline residue on the bond strength of lithium disilicate (LD) glass ceramics by means of microtensile bond strength (μ-tbs) testing.

METHODOLOGY: Thirty blocks (4x6x8mm) of LD ceramic were made with pressing furnace utilizing the lost wax technique. Out of these 20 ceramic blocks were subjected to edHF acid (9.5% for 90 sec) and rinsed with water (20 sec). The remaining 10 ceramic blocks were left un-etched to be used as negative controls (group A). Half (n=10) of the etched blocks were only rinsed with water (group B) and the other half (n=10) received post etch cleaning (PEC) (37.5% Phosphoric acid for 1 min, rinse with water for 20 seconds and ultrasonic bath immersion) (group C) of the surface. Silane was applied (5 mins) to all specimens. Ceramic and composite resin blocks were bonded using an adhesive resin and light cured restorative composite as a luting agent, under standard conditions. A total of 360 specimen sticks (8 x 1mm²) were subjected to micro-tensile testing. The means of micro-tensile bond strength (µ-tbs) were analyzed with ANOVA.

RESULTS: The lowest and highest bind value was shown by group A (10.81±3.02) Mpa and group C (39.94±2.58) respectively. Post-etch cleaning of the residue formed on ceramic surface showed significant improvements in μ-tbs value (p=0.01) when compared to un-etched and etched ceramic specimens.

CONCLUSIONS: The presence of surface residue and crystalline salts produced after HF acid etch, significantly compromised the microtensile bond strength of LD ceramic to resin. Post etch cleaning of lithium disilicate ceramics significantly improved their microtensile bond strength to resin.

KEYWORDS: Ceramic, Lithium disilicate, Post-etch cleaning, microtensile bond strength, HF acid etch.


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INTRODUCTION

Modern dental ceramic is the esthetic material of choice and has contributed greatly to the increased demand on cosmetic indirect restorations. Adhesive cementation of glass-ceramic restorations with a composite resin of optimum physical properties can withstand higher masticatory forces and has demonstrated improved clinical performance. Bonding of indirect ceramic restorations to tooth structure involves the luting agent to interact with two different surfaces i.e. enamel/dentin and ceramic. As compared to other ceramics lithium disilicate ceramics offer enhanced flexural strength and fracture toughness. Some investigators, due to improved mechanical properties, have also used LD ceramics for 3 unit bridges.

As conditioning of tooth surface is a standard protocol for adhesive bonding, similar is the importance of conditioning the ceramic bonding surface for predictable adhesion. Hydrofluoric acid, as a surface treatment has a long term proven track record of favorable clinical outcome. Similarly, for the formation of a durable chemical bond, silane application is recommended. And many authors consider treatment of ceramic surface both with HF acid and silane as indispensable.

In a recent study by Canay et al., spectroscopic analyses showed formation of crystalline precipitates on the etched ceramic surfaces in the form of Na, K, Ca & Al by-products. These by-products of HF acid etch are readily insoluble in water and possibly contaminate the ceramic bonding surface. In
addition a cleaning regime for etched ceramic surfaces has been recommended\textsuperscript{12}, which includes use of 37.5\% Phosphoric acid for 1 min, rinse with water for 20 seconds and ultrasonic bath immersion. Therefore it is hypothesized that the presence of surface residue and crystalline salts after ceramic etching would compromise the bond between ceramic and resin. In addition, effective removal of crystalline precipitates from etched ceramic surface would result in a durable and predictable ceramic resin bond. Therefore, this study aimed to evaluate the influence of post HF acid etch crystalline residue on bond integrity of LD by means of microtensile bond strength ($\mu$-tbs) testing.

### METHODOLOGY

The methodology of this study is similar to our previously published paper\textsuperscript{20}. Briefly, 30 blocks of lithium disilicate ceramic (IvoclarVivadent) measuring 4x6x8 mm of were prepared, grit blasted with alumina (70$\mu$m glass beads, finished with 240-1200 grit SiC) and cleansed in ultrasonic bath for 10 minutes.

A total of 20 ceramic blocks were etched using 9.5\% HF acid for 90 seconds and rinsed with water for 20 seconds (sec) for removal of HF acid. 10 ceramic blocks were left un-etched to be used as negative controls (Group A). All the experimental ceramic specimens were divided into the following three groups (A,B and C) depending on the surface treatments.

The prepared ceramic blocks were subjected to following surface treatment:

- **Group A:** LD ceramic blocks + no HF acid etch + Silane (S) application (5 minutes)
- **Group B:** LD ceramic blocks + HF acid etch + Rinse with water (20 secs) + Silane (S) application (5 minutes)
- **Group C:** LD ceramic blocks + HF acid etch + Rinse with water (20 secs) + Post etch cleaning (37.5\% phosphoric acid (H3PO4) with agitation for 1 min, water rinse 20 sec and ultrasonic cleaning (US) for 5 min in distilled water bath) + Silane (S) application (5 minutes).

An adhesive (Optibond FL, Kerr) was applied to all the surface treated ceramic blocks. Composite blocks (Hybrid filler) (4x6x8 mm) of same dimensions as ceramic blocks were fabricated using a rubber (Aquasil, putty) copy mould of ceramic blocks. Composite and ceramic blocks were bonded using a 10 sec load application of 1 kilogram and light cured (LED) for 160 sec (650 mWcm\textsuperscript{-2}). The bonded composite-ceramic blocks were sectioned using slow speed diamond wheel saw (Isomet 1000) at a constant speed of 500 rpm at 250 g force. Specimen sticks of 1mm\textsuperscript{2} cross section (non trimming technique) were produced and stored for 24 hours in normal saline. Hundred specimen for each group were randomly selected for microtensile testing. The specimens were attached to the tester jaws using cyanoacrylate adhesive and loaded to failure under tension at a crosshead speed of 0.5mm/min using a microtensile tester. The means of $\mu$-tbs were analyzed with one way ANOVA. Details of the materials and equipment used in the study are presented as appendix A

### RESULTS

The data was normally distributed as revealed by Kolmogrov Smirnov test. The lowest and highest $\mu$-tbs value was shown by group A (10.81±3.02) Mpa and group C (39.94±2.58) respectively. Table no. 1 represents mean $\mu$-tbs values. The difference in the $\mu$-tbs values between different groups is graphically presented in Fig. 1. Comparison using ANOVA revealed a statistically significant difference in the $\mu$-tbs value among the three different groups ($p=0.01$). Post-etch cleaning of the residue formed on ceramic surface showed significant improvements in $\mu$-tbs value ($p=0.01$) when compared to un-etched and etched ceramic specimens.
DISCUSSION

The application of glass ceramic based inlays, onlays and crowns are frequently employed in oral rehabilitation due to tooth conservation. And the ability of glass ceramics to form a durable adhesive bond to tooth structure is critical to its clinical success. The present study was based on the hypothesis that formation of surface residue and crystalline salts due to HF acid etch can potentially interfere with the ceramic adhesive bond. In addition a standard post etch cleaning regime would result in improvement of glass ceramic bond strength to resin composite. Both these hypothesis were found to be acceptable as a result of the present study results.

Resin composite was used as the bonding substrate, instead of tooth structure. The explanation for this is twofold, primarily because the aim was to evaluate contribution of surface treatment steps of ceramics and secondarily, in order to minimize the likely variables in the experiment which includes, quantity and structure of exposed dentine, size and number of tubuli openings and tooth surface treatment. Hydrofluoric acid etching of ceramics was used as it produces a consistent and favorable micro morphology of surface for micromechanical retention. Etching further increases the surface free energy and reduces the surface contact angle, improving wettability for the luting agents. Moreover, microtensile bond strength test was used as it represents the true adhesive bond strength compared to shear bond test, which reflects the strength of base material.

Previous analyses have revealed significant increase in the bond strengths of ceramic materials on silane application. Silanes being bifunctional molecules promote ceramic resin adhesion and facilitate resin penetration into the acid etched ceramic by enhancing the wetting of the surface. One of the previous report have identified silane as having a major effect on bonding of resin to lithium disilicate ceramics. In the present study silanes were included in the adhesive regime in all experimental groups to produce a durable bond and to simulate clinical protocol.

The present study showed that etching with HF acid followed by no standard post etch cleaning regime resulted in bond strength values significantly lower than specimens cleaned with post etch cleaning regime after etching. According to a study using energy dispersive spectroscopy, it was concluded that surface etching of glass ceramics leaves a layer of poorly adherent precipitates composed of reaction products of fluorosilicate of Na, K, Ca and Al. In an earlier study, it was shown that failure to remove these precipitates, resulted in a 50% reduction in microtensile bond strength when bonding feldspathic ceramic to composite resin. In the present study similar findings were observed with regards to lithium disilicate ceramics.

In the present study, post etch cleaning included application of phosphoric acid and ultrasonic bath immersion, significantly improving adhesive bond strength. However, it would be interesting to investigate the individual contribution of ultrasonic cleaning and phosphoric acid on the bond strength in order to simplify the bonding process clinically. Therefore further studies in this regard are warranted. A 90 seconds etch time was employed in the present study, however studies have reported bond strength values for lithium disilicate ceramics four times than other glass ceramics using a 2 minute ceramic etch time. Therefore further studies with varying etching concentrations and durations to improve the ceramic bonding regime are recommended.

Fig. (1). Comparison of Means and SD of microtensile bond strength values among study groups.
CONCLUSIONS

Within the limitations of the study, the following conclusions can be drawn with respect to the materials used:

- The presence of surface residue and crystalline salts produced after ceramic etching significantly compromised the microtensile bond strength of LD ceramic to resin.
- Post etch cleaning of lithium disilicate ceramics significantly improved their microtensile bond strength to resin.

DISCLOSURE

None declared.

REFERENCES