



Impact of Hydrofluoric Acid Etching Duration on Translucency, Flexural Strength and Surface Topography of Lithium Silicate Based Glass Ceramic

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ABSTRACT

Purpose: The purpose of the present study is to assess the effect of different hydrofluoric acid etching durations on the translucency, flexural strength and surface topography of a lithium silicate-based glass ceramic (LS). **Materials and Methods:** Forty LS (Obsidian) samples will be constructed and divided into four groups (n=10 each) according to the duration of hydrofluoric acid (HF) application including group (I); (control) as received samples without surface treatment, group (II); ceramic surfaces etched with HF for 10 seconds; following the manufacturer's recommendations, group (III); ceramic surfaces etched with HF for 30 seconds and group (IV); ceramic surfaces etched with HF for 60 seconds. All treated samples were evaluated for translucency (TP) using spectrophotometer, flexural strength using universal testing machine and surface topographic analysis using SEM and profilometer. Obtained values were statistically analyzed. **Results:** The highest mean value of translucency and biaxial flexural strength were recorded at the control group. Values then gradually decreased in the subsequent etching times to reach its lowest level after 60 seconds. While the lowest mean value of surface roughness (Ra) was recorded at control group. The mean Ra value gradually increased with the subsequent increase in etching times to reach its highest level after 60 seconds. **Conclusions:** Increasing HF acid etching duration negatively affects the translucency of lithium silicate based glass ceramic; while it has no effect on its biaxial flexural strength or its surface roughness (Ra).

KEYWORDS

Biaxial flexural strength (FS), LS (Obsidian), surface roughness, translucency.

INTRODUCTION

Dental ceramics are materials used for producing dental prostheses that in turn, are used to replace missing or damaged dental structures⁽¹⁾.

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In (1998), a lithium disilicate (LDS) glass ceramic material called "IPS Empress II" was introduced by Ivoclar. It was used back then either as a single or multiple unit frame-works and indicated for the anterior region. The material recognized a huge success back then that was measured through a 5-years study. It recorded a seventy percent success rate when used as a fixed partial denture frame-work⁽²⁾.

LDS used again in 2006 as a pressable ingot and partially crystallized milling block. 170% higher than the previously used leucite-reinforced ceramics was the rate that LDS reached in terms of FS. Afterwards, LDS created a new opportunity in digitized restorative dentistry through the CAD/CAM milling of the frame-work either (zirconium dioxide or metal), the full-contoured crown (lithium disilicate at chair-side or in the laboratory), or an implant abutment⁽³⁾.

In the last few years, a new glass ceramic; LS based was introduced under the name of Obsidian®. It witnessed an innovative combination of more than 20 elemental oxides from the periodic table (including Zirconia). The perfect properties of the Obsidian milling blocks were related to the very high content of the ultra-nanometer size lithium silicate and lithium phosphate crystals which are provided in the semi-crystallized phase to be milled using the above mentioned method, "CAD/CAM methods."⁽⁴⁾

Bonding to the newly introduced glass LS (Obsidian) is still not determined and under laboratory investigation. Manufacturer instructions stated that Obsidian is etched with HF followed by silane coupling agent is necessary to obtain adhesive bonding to resin cements. However, the etchant's effect on different properties of this new glass ceramic material is still unknown.

As concerns remain about the newly introduced Obsidian as a promising dental material; it is important to evaluate its different properties as;

the translucency, flexural strength and surface topography, especially after application of different hydrofluoric acid etching durations.

The null hypotheses of the present study were; different HF etching durations will not affect the translucency, flexural strength, or surface topography of Obsidian glass ceramic.

MATERIALS AND METHODS

Preparation of Obsidian samples

Obsidian blocks were lathe cut in a cylindrical shape of diameter 10.2 mm, using a lathe cutting machine and then sectioned into 1.3 mm thick circular discs by diamond micro-saw. According to the ISO standards 6872; 2015 for ceramic flexural strength testing; the dimensions of finished ceramic discs should be 10 mm diameter and 1.2 mm thickness. Due to the expected shrinkage during crystallization (0.1% according to the manufacturer), Obsidian discs were sectioned with larger dimensions (10.2 mm diameter, 1.3 mm thickness). The dimensions of the samples were confirmed using digital caliper.

Then, the sectioned samples were finished according to the manufacturer's instructions using fine inverted cone diamond burs.

Obsidian samples were crystallized according to the manufacturer's instructions in Vita Vacumat 6000 MP ceramic furnace. Subsequently, the samples were left until the furnace had cooled sufficiently while being protected from any drafts of air.

To simulate the clinical situation, one surface of each sample was polished. To standardize the polishing procedure, each surface was polished in only one direction for 15 seconds by a single operator with light pressure according to the manufacturer's instructions using Blue wheel stone followed by felt wheel with diamond paste and finally goat hair brush for each sample. After that, the dimensions of the sample were confirmed using digital caliper.

Classification of samples

Forty circular discs (N=40) of Obsidian lithium silicate ceramic (10mm diameter X 1.2mm thickness) were randomly divided into 4 groups (n=10 each) according to the duration of hydrofluoric acid (HF) as follows:

1. Group (I): As received samples without surface treatment (control group).
2. Group (II): Obsidian Samples etched with HF for 10 seconds; following the manufacturer's recommendations.
3. Group (III): Obsidian Samples etched with HF for 30 seconds.
4. Group (IV): Obsidian Samples etched with HF for 60 seconds.

Etching of Obsidian samples:

Samples of groups (II, III, and IV) were etched using IPS ceramic etching gel according to the specified etching duration for each group (10, 30 and 60 seconds, respectively). A thin layer of etching gel was applied using a micro-brush on the un-polished surface of each sample followed by washing with an air water spray for 30 seconds.

Testing Procedures:

A) Translucency determination (TP value):

The Translucency Parameter (TP) can be defined as measuring the color difference between a material (Obsidian in this study) of uniform thickness (1.2mm) over a black background and a white one and correlates it directly to a common visual assessment of translucency. The translucency was measured using the portable reflective spectrophotometer where an accurate alignment between samples tested and the device were set. Additionally, the hole size was set at 4 millimeter. The samples were placed in the center of the measuring port and were kept in the same position for the white and black backings.

B) Biaxial Flexural Strength (MPa):

Biaxial flexural strength tests were performed using Bluehill Lite Software from Instron. For measuring the FS of the tested samples, a biaxial flexure test (uniform pressure on disc) with a ball on ring fixture was performed through testing machine with a load-cell of five kN, at a speed of 1mm per min. For accuracy basis, computer software was used to record the data. Other parameters were set to keep the test under control, which are: first is room environment were kept at $30 \pm 1^\circ\text{C}$, and $70\% \pm 5\%$ relative humidity. Secondly, discs diameter kept at 8-mm and circular knife-edge with 3.8mm spherical indenter in the center. Disc was set at the unpolished face where tension was performed, while loading was performed on the polished etched face. A positive relationship between the fracture strength and number of fragments was noticed when samples were loaded under biaxial flexure strength. The greater the fracture stress, the more fragments resulted.

C) Surface Topography:

C.i: Surface Roughness (Ra) (μm):

Samples were photographed using high technical and USB digitalized microscope connected with a camera and IBM compatible PC through a high magnifying characteristic that reach 120X.

Resolution of pictures was kept as high as 1280×1024 pixels. Then the microscopic pictures were edited to 350×400 pixels using simple programs, such as MS picture manager to accurately set the roughness area. Then a program called WSxM software were used to analyze the pictures and determine all parameters in pixels that has been later translated into real units, such as limits, borders, and frames. Each sample was photographed 5 times with 3D technology from areas, the central and the sides. Each area is $10 \mu\text{m} \times 10 \mu\text{m}$. The average of heights (Ra) expressed in μm was measured by the above mentioned software and the figures were assumed to be a reflection of the surface roughness.

C.ii: Scanning Electron Microscope:

Randomly selected samples from each group were scanned with Electron Microscope. The magnification used in this study was X2000 and X 8000.

Statistical analysis:

Results were introduced in form of mean (M) and standard deviation (SD) values. They have been tested in Kolmogorov-Smirnov test of normality. Hence, results highlighted that most of data were normally distributed (parametric data). Therefore, a variance analysis was needed using (ANOVA) to come up with a better comparison among all groups. One more test was then conducted due to the significant difference recorded. Hence, Tukey's post hoc test was conducted. To measure the correlation between roughness and translucency, Pearson correlation coefficient is the best to use. The Pearson correlation coefficient is used to measure the strength of a linear association between two variables, where the value r equals 1 means a perfect positive correlation and the value r equals -1 means a perfect negative correlation.

RESULTS**Statistical analysis of Translucency Parameter (TP):**

The highest mean value was recorded at the control group (13.93 ± 0.8). Values then gradually decreased in the subsequently increased application times to reach its lowest level (12.41 ± 0.38) at 60 seconds, group (IV). The result of ANOVA test was dramatically different than the results of revealed that Tukey's post hoc test. Although ANOVA resulted in extremely statistically significant ($P < 0.0001$) difference between groups, Tukey's post hoc test revealed no significant difference between TP values after 10 seconds, group (II), and after 30 seconds, group (III), and no significant difference between TP values after 30 seconds, group (III), and the value after 60 seconds, group (IV), table (A).

Statistical analysis of Biaxial Flexure Strength

The highest biaxial flexural strength mean value was recorded in the control group (433.6 ± 22.21 MPa), and then gradually decreased in the subsequent application times groups to reach its lowest level at 60 seconds, group (IV), (377.25 ± 62.67 MPa). ANOVA test highlighted that, there was no statistical difference between tested groups ($P = 0.194$), table (B).

Table (A): Mean values (M), standard deviation (SD), and statistical analysis of Translucency parameter (TP) and comparison of different groups (ANOVA and Tukey's test):

| Group | M | SD | SE | 95% Confidence Interval for Mean | | Min | Max | F | P |
|---------|----------------------|------|------|----------------------------------|-------------|-------|-------|-------|----------|
| | | | | Lower Bound | Upper Bound | | | | |
| Control | 13.93 ^a | 0.80 | 0.21 | 13.49 | 14.38 | 12.75 | 15.19 | 21.47 | <0.0001* |
| 10 sec | 13.20 ^b | 0.28 | 0.07 | 13.04 | 13.35 | 12.93 | 14.12 | | |
| 30 sec | 12.83 ^{b,c} | 0.55 | 0.14 | 12.52 | 13.14 | 12.09 | 13.89 | | |
| 60 sec | 12.41 ^c | 0.38 | 0.10 | 12.20 | 12.61 | 11.75 | 13.22 | | |

-Significance level $p < 0.05$, * significant.

-Tukey's post hoc test: No Significant difference recorded between groups having the same superscript letter within same comparison

Table (B): Mean values, standard deviation, and statistical analysis of biaxial flexure strength (MPa) and comparison between different groups (ANOVA test):

| Group | M | SD | SE | 95% Confidence Interval for Mean | | Min | Max | F | P |
|---------|--------|--------|-------|----------------------------------|-------------|--------|--------|------|----------|
| | | | | Lower Bound | Upper Bound | | | | |
| Control | 433.60 | 22.21 | 5.73 | 421.30 | 445.90 | 406.18 | 459.29 | 1.63 | 0.194 ns |
| 10 sec | 419.47 | 69.08 | 17.84 | 381.22 | 457.72 | 308.75 | 490.04 | | |
| 30 sec | 415.44 | 110.53 | 28.54 | 354.23 | 476.65 | 218.52 | 528.47 | | |
| 60 sec | 377.25 | 62.67 | 16.18 | 342.54 | 411.96 | 311.39 | 483.42 | | |

-Significance level $p < 0.05$, ns = non-significant

Results of Surface Topographic examination:

Statistical analysis of Surface Roughness values (Ra) (μm):

The lowest mean (Ra) value ($0.251 \pm 0.013 \mu\text{m}$) was recorded for the control group, group (I). The mean (Ra) values gradually increased with increased application times to reach its highest level after 60 seconds, group (IV), ($0.257 \pm 0.02 \mu\text{m}$). ANOVA test revealed that there is no statistical difference between different groups ($P=0.15$), table (C)

Scanning electron microscopic observations:

SEM images of the representative samples from each group are illustrated in figures (1-4). Comparing SEM images of HF etched groups with those of control group, varying degree of roughness which increase with increase application duration is observed. High magnification ($X=8000$) of etched groups show dissolution of glassy matrix proportional to application duration. Crystals become more prominent with obvious intra-crystalline space.

Table (C): Mean values, standard deviation, and statistical analysis of Ra values (μm) and comparison of different groups (ANOVA test):

| Group | M | SD | SE | 95% Confidence Interval for Mean | | Min | Max | F | P |
|---------|--------|-------|-------|----------------------------------|-------------|-------|-------|------|----------|
| | | | | Lower Bound | Upper Bound | | | | |
| Control | 0.2505 | 0.013 | 0.003 | 0.243 | 0.258 | 0.223 | 0.268 | 1.84 | 0.150 ns |
| 10 sec | 0.2538 | 0.004 | 0.001 | 0.252 | 0.256 | 0.244 | 0.258 | | |
| 30 sec | 0.2546 | 0.013 | 0.003 | 0.247 | 0.262 | 0.209 | 0.266 | | |
| 60 sec | 0.2567 | 0.002 | 0.001 | 0.256 | 0.258 | 0.254 | 0.261 | | |

-Significance level $p < 0.05$, ns = non-significant

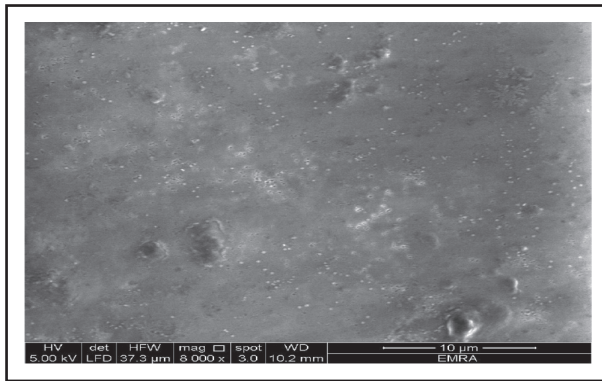


Fig. (1): SEM of (control group) non etched Obsidian, magnification X8000

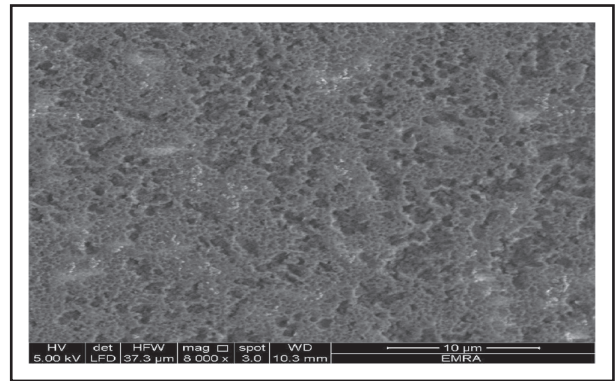


Fig. (2): SEM of 10 sec. HF etched Obsidian, magnification X8000

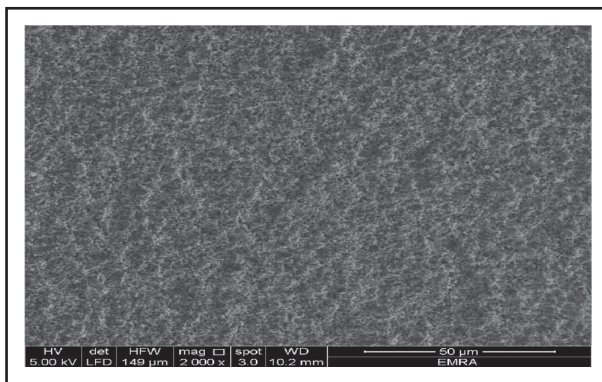


Fig. (3): SEM of 30 sec. HF etched Obsidian, magnification X8000

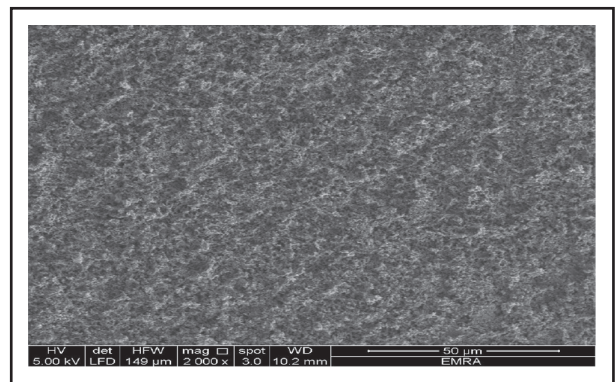


Fig. (4): SEM of 60 sec. HF etched Obsidian, magnification X8000

DISCUSSION

Obsidian is a promising new glass ceramic restorative material ⁽⁵⁾. The Obsidian milling block is supplied in a partially crystallized (nucleated) phase, so crystallization of the discs was done to reach the fully crystallized discs. The expected amount of shrinkage was taken into considerations by sectioning the samples with larger dimensions. Final dimension of the samples was confirmed using the digital caliper.

Flexural strength is influenced by surface roughness ⁽⁶⁾. It was previously reported that flexural strength is inversely proportional to surface roughness and directly proportional to surface finish and defect disappearance ⁽⁷⁾. Previous studies revealed that flexural strength is inversely proportional to the square of the defect size ^(7, 8).

That's why discs' finishing and polishing procedures were done according to standardized procedure in the present study.

In order to simulate the clinical situation of crown restoration, one of the surfaces of the discs was polished according to the manufacturer recommendations while the other surface was etched using hydrofluoric acid (HF). Finishing and polishing procedures were standardized by polishing in single direction for a fixed time by one operator.

In this study; etching was done with 5% HF acid concentration according to manufacturer instructions at four different etching times (0, 10, 30, and 60 sec.). Non-etched samples served as a control baseline group for comparison purposes. Etching for 10 seconds is the manufacturer's recommendation

etching time while 30 and 60 seconds was done to evaluate the effect of increasing the duration of etching time, as in an attempt to improve bonding of glass ceramics with resin cements. Accordingly, the effect of increasing etching time on different properties of the ceramic was investigated.

Concerning the etching time, many studies have been conducted in this field with various ceramics and HF concentrations⁽⁹⁻¹²⁾. One of them, for example⁽¹¹⁾ used two HF concentrations (2.5% and 5%) with 7 different etching intervals (zero, thirty, sixty, ninety, one hundred and twenty, one hundred and fifty, and one hundred and eighty seconds). On another hand, another study⁽¹²⁾ used a higher HF concentration, which is 9.5% with different intervals, 30, 60, 150, and 300 sec. Although there has been difference in HF concentration and different intervals in both studies, they both agreed that there is a positive relationship between HF etching time and ceramic surface roughness. And all other studies conducted in this field revealed same result, the positive correlation has been confirmed^(13,14).

To evaluate the effect of different etching durations on the optical properties of Obsidian; translucency of group samples was evaluated. Translucency is a fateful factor in controlling esthetics, in addition to playing a key role in luting procedures especially during resin polymerization⁽¹⁵⁾. In this study, translucency was evaluated with the calculation of (TP) value, which is defined as; measuring the color difference between a material (Obsidian in this study) of uniform thickness (1.2mm) over a black background and a white one. The higher values of (TP), the greater the translucency⁽¹⁶⁾.

In the present study there was a statistically significant difference in translucency between groups with the highest mean TP value recorded in the control group (13.93 ± 0.8), group (I). Values gradually decreased in the subsequent application times to reach its lowest level (12.41 ± 0.38) after 60 seconds, group (IV). Therefore, the first null

hypothesis of the study was rejected. Increasing application duration of HF etching significantly decrease the translucency of Obsidian glass ceramic.

These results are in accordance with a previous study which stated that the translucencies of lithium disilicate ceramic (e.max CAD) significantly decreased after hydrofluoric acid etching due to the removal of the glassy matrix of the ceramic exposing the crystalline structure⁽¹⁷⁾.

On the contrary, another study⁽¹⁸⁾ found that 5% HF etching have no effect on TP values of glass ceramic laminate veneers with different shades and thicknesses. Taking into account that this study was conducted using IPS e.max Press glass ceramic; the difference in results between both studies could be explained on the bases of the compositional difference.

It was previously stated that material roughness has a significant effect on the optical properties of the restorative materials and directly affect the perceived translucency⁽¹⁹⁾. The smoother the surface of the material, the more it is expected to experience a specular reflection as reflection angel of the light will be at the same angel where it hits the surface. However, the rougher the surface is, the more the light reflection diffuses and reflects in totally different angel⁽²⁰⁾. It was reported that after airborne-particle abrasion lithium disilicate ceramic become opaquer, whereas HF acid etching do not affect the translucency significantly⁽¹⁸⁾. The TP values of the tested groups obtained in the present study could be explained on the basis of the surface topographic changes occurring after surface treatment, as surface roughness and the SEM analysis revealed.

This decrease in translucency with increased etching duration can be attributed to the relative decrease in amount of glass after etching. Relative increase in crystalline percent after glass removal will increase the opacity.

A variety of FS tests have been employed to expect the performance of brittle materials such

as dental ceramics. The standard test over dental ceramic in general has been the three-point bending test. However later on; many defects were recognized on surface sides which threatened the accuracy of the test. On the other side, biaxial flexural reliability aced the uniaxial flexural one. Biaxial flexural has more advantages, such as allowing the maximum principal stress over the area in the center of tensile surface. Not only that, but also biaxial allows an elimination of failures and variation of material strength are witnessed. A one final advantage is the reproduction of clinical mode of all ceramic restorations, i.e., failure from the extension of pre-existing flaws on the internal surface of restorations under tensile stresses ^(21,22).

Within the results of present study, flexural strength mean values were higher in the control group, group (I), compared with the etched groups, groups (II, III &IV); however, the difference between groups was not significant. Hence, we fail to reject the second null hypothesis stating that HF acid etching time would not affect the flexural strength of lithium silicate glass ceramic.

This finding is in agreement with a study ⁽²³⁾ that compared the effects of different etching protocols (5% and 9.5% HF) and time (20 and 120 seconds) on the flexural strength (3-point bending test) of IPS e.max CAD specimens. Authors concluded that HF acid etching decrease the flexural strength of glass ceramic regardless of the protocol used; however, this effect is not statistically significant.

However, opposite findings were reported in a previous study that examined the effect of different acid etching times (20, 60, 90 and 180 seconds) on the flexural strength of IPS e-max CAD glass ceramic. It was found that etching lithium disilicate glass ceramics with HF acid significantly reduces their flexural strength ⁽⁶⁾.

The acidic action on the glassy matrix and the crystal structure creates plenty of porosities and holes which results in creation of a rougher surface. For a better bonding, more roughness is required

which can be resulted from the increase of etching duration ^(6, 12-14). This also has been supported from other studies which confirm a positive relationship between roughness of the surface and bond strength ^(6, 12). However, 2 side notes should be taken into consideration in this respect: firstly, the absolute amount of roughness required for perfect bonding is not known yet. Secondly, over etching can form deep porosities which in turn affect the bond strength ^(9, 13, 14, 24).

Some authors ⁽²³⁾, however, theorized that the shallow consistent pattern resulted from etching with five percentile HF on LDS surface did not generate the needed stress to notably affect the FS.

Surface roughness of dental ceramics can be measured by contact or non-contact methods. The main disadvantage of contact method (through using stylus- profilometer) is the possible surface damage of the samples due to the force applied by the profilometer's stylus. However, non-contact profilometer overcome this disadvantage, given they do not touch the surface of the ceramic specimens and that the small diameter of the laser scanner provides accurate measurement of the surface topography ^(25, 26). Hence, non-contact profilometry was the method of choice for surface roughness measurement in the present study.

In this study, the HF acid etching did not have, on any of the tested sample, a significant effect to raise surface roughness, apart from of the etching duration used. However, Ra values increased non-significantly with increasing application time. In addition, the non-contact profilometry scans in the present study, displayed a pattern of higher peaks and deeper valleys of the etched samples in comparison with the untreated control group. Thus taking into consideration the obtained results of the present study, we fail to reject the null hypothesis that HF acid etching duration would not significantly affect the surface roughness of the lithium silicate glass ceramic.

This result is in agreement with a previous study⁽²⁷⁾ that found that increasing HF acid etching duration does not have a significant effect on the surface roughness of IPS e.max CAD lithium disilicate glass ceramic. Yet, lower roughness values were obtained in that study. This difference could be attributed to the using of different polishing protocols, contact profilometry, and different roughness parameters to report the surface roughness^(6, 28, 29).

However, this result contradicts what was reported in previous studies^(6, 28) that increasing HF acid etching duration has a positive relation with ceramic surface roughness. A previous study⁽²⁸⁾ tested the impact of HF acid etching intervals (20, 40, 80 and 160 sec.) on the surface roughness of rectangular shaped specimens cut from different types of ceramics and found a significant increase in surface roughness with increasing etching duration. While another study⁽⁶⁾ examined the effect of different acid etching times (20, 60, 90 and 180 sec.) on the roughness of IPS e-max CAD glass ceramic and found that increasing HF etching time increased surface roughness. Both studies used different materials, samples shape and etching durations which could explain the different results obtained.

In the present study, SEM images of the etched and un-etched ceramic surfaces noticeably represented the effect of different etching durations on the microstructure of the glass ceramic. SEM images at magnifications (X2000 and X8000) revealed numerous irregularities and voids in the etched ceramic surfaces as well as lithium silicate and phosphate crystals in comparison with the un-etched ceramic surfaces, which displayed homogenous patterns. This can be explained by the selective removal of the glassy matrix in the treated samples as a sequence to HF etching; exposing the underlying crystalline structure.

In addition, as the etching periods increased, the size and number of the voids also increased as was seen in samples etched for 60 seconds (group IV), versus those etched for 10 seconds

(group II), which demonstrated fewer microstructure alterations. These observations are in agreement with some previous studies^(6, 28, 29), which found that non etched ceramic surface is normally smooth and flat, However HF etching can transform it into porous and irregular due to the dissolution of the glass phase. Hence, the longer the etching period, the larger the voids and channels appeared until it reached to an extreme case where disilicate crystals can be easily seen from the glassy matrix.

This present research reflects the limitations associated with the in-vitro studies. It worth mentioning that the 3-point bending test used is just an assumption and does not reflect the real clinical situation in respect of actual flexural strength in due to the difference between environmental and loading conditions. In addition, other conditions that do not apply to the oral environment are witnessed. For example, study was done in static and dry conditions, which contradicts with wet and cyclicity of oral environment. One further limitation is that the effect of only a single concentration of HF was evaluated. Combining different HF concentration and time intervals might have changed the results of the study.

CONCLUSIONS

Keeping the limitations of this study into consideration, the following conclusions can be highlighted as follows:

1. Increasing HF acid etching duration negatively affects the translucency of lithium silicate glass ceramic. However, this effect is non-perceivable.
2. Increasing HF acid etching duration to 60 seconds does not affect biaxial flexural strength of lithium silicate based glass ceramics.
3. Increasing HF acid etching duration to 60 seconds has no effect on surface roughness (Ra) of lithium silicate based glass ceramics. However, increased HF acid etching duration causes removal of the glassy matrix.

4. Optimum combination of translucency and biaxial flexural strength is obtainable by following the recommended HF etching duration.

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