EFFECT OF HF ACID ETCHING DURATION ON SURFACE ROUGHNESS OF NEWLY INTRODUCED GLASS CERAMIC MATERIAL

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ABSTRACT

Purpose: The aim of this in-vitro study will be directed to evaluating the effect of different hydrofluoric acid etching durations on the surface roughness of lithium silicate based glass ceramics obsidian and e-max, microstructure changes also color difference.

Materials and methods: Eighty disc samples were grouped according to material type into two groups; obsidian group(n=40) and e-max group(n=40), each group was subdivided into four groups according the duration of hydrofluoric acid (HF) application into: **Subgroup I (control)** (n=10) without surface treatment. **Subgroup II,** (n=10) 20 seconds **Subgroup III,** (n=10): 40 seconds. **Subgroup IV,** (n=10): 60 seconds. All surface treated samples will be subjected to: Surface roughness examination, Surface topographic analysis using SEM and profilometer, and color difference

Results: the surface roughness test revealed that; the highest mean value was recorded in 60 second etch, followed by 40 sec etch then 20 second etch; with the least value recorded in control (no etch). ANOVA test revealed that the difference between etching subgroups was statistically significant (p<0.0001). Also higher mean value was recorded in E-max with statistically significant difference (p=0.014). Effect of etching time on color the highest mean value was recorded in 60 second etch, followed by 40 sec etch then 20 second etch,; with the least value recorded in control (no etch). ANOVA test revealed that the difference between etching time was statistically significant (p<0.0001).Also a higher mean value was recorded in E-max with statistically significant difference (p<0.0001),

Conclusion E-max group showed higher means of surface roughness test and color difference rather than obsidian. Also 60 second etch showed the highest mean values of surface roughness test and color difference followed by 40 second etch followed by 40 second etch.

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INTRODUCTION

Esthetic aspects of dental treatment are becoming increasingly important to patients, thus increasing the demand for all ceramic restorations. Since the introduction of reinforced feldspathic Porcelain in 1965 [1], new materials and processing technology for all ceramic restorations with significantly improved physical and mechanical properties are available at present [2, 3].

One of the early generations of glass ceramics was Leucite-based glass ceramics IPS Empress I (Ivoclar Vivadent) [4, 5]. Its major advantage over other types of ceramic materials is a translucency similar to that of enamel, providing a "chameleon effect" that permits light to be reflected, scattered, and absorbed throughout the restoration and the underlying tooth structure and allows the restoration to blend with the natural tooth [6, 7]. Despite its limited in use to single unit complete-coverage restorations in the anterior segment due to its low mechanical properties [8].

In order to enable glass-ceramics to be used in the fabrication of dental bridges and single crowns in the molar region, the strength and toughness of these materials had to be increased. A significant increase in these parameters was achieved by introducing the Lithium disilicate glass-ceramics [9, 10].

The next generation of lithium disilicates (IPS e.max Press IPS e.max CAD, Ivoclar Vivadent, Amherst, NY, USA) [11, 12]. Is an innovative all-ceramic system that comprises lithium disilicate (LS2) glass ceramic for the press and CAD/CAM technologies [13]. Restorations can be fabricated from this type of resistant glass ceramic with either lost-wax hot pressing techniques or modern CAD/CAD milling procedures [14, 15].

Recently in the past few years a new glass ceramic -lithium silicate based- was introduced under the name of Obsidian. Obsidian is a monolithic restoration. It may use as CAD/ CAM milling blocks or pressed to metal results in a restoration with superior strength. Obsidian blocks are lithium silicate glass ceramic that contain 20 elemental oxides including Zirconia. Additionally, the Obsidian milling block owes its excellent properties due to a very high content of ultra-nanometer-size lithium silicate and lithium phosphate crystals. Obsidian milling block is supplied in a partially crystallized phase, which is to be milled using CEREC MC XL milling machine. Obsidian is highly esthetic and chip resistant. It exhibits excellent translucency, resulting in superior esthetics. It is indicated for crowns, inlays, onlays, and veneers; possesses above average flexural strength and is recommended mainly for anterior and premolar crowns [16].

An important aspect required for the success of such restorations is the establishment of proper adhesion between substrate and adherent [17]. In this sense, the gold-standard protocol for resin bonding to glass ceramics is the etching with hydrofluoric acid (HF) followed by the application of a silane coupling agent (chemical and micro-mechanical bond) [18, 19].

Variations in HF acid etching (time and concentration) have been shown to change the surface micro-morphology of glass ceramics (**Traini et al., 2016**) [20] and resin adhesion (**Leite et al., 2013; Venturini et al., 2015**) [21, 22], for ceramic surface treatment, the acid reacts with the glass matrix that contains silica and forms hexafluorosilicates. This glass matrix is selectively removed and the crystalline structure is exposed. As a result, the surface of the ceramic becomes rough, which is expected for micromechanical retention on the ceramic surface. This roughly etched surface also helps to provide more surface energy prior to combining with the silane solution.

The study have reported positive effects of hydrofluoric acid (HF) etching on the strength of glasses by removing or stabilizing surface defects

and on surface topography increasing roughness for adhesive bonding.

MATERIALS AND METHODS

Forty (N=40) samples for each material (lithium-silicate glass ceramic (Obsidian; Prismatik Dentalcraft, Glidewell Labs.) and (lithium-disilicate glass ceramic (e max CAD Ivoclar Vivadent, Schaan, Liechtenstein). will be constructed of 10.2 mm diameter and 1.3 mm thickness. The dimensions of the samples were confirmed using digital caliper.

The ceramic slices were wet polished using 1000-grit silicon carbide paper and polishing paste to remove external irregular scratches and defects. All ceramic specimens were sonically cleaned in distilled water for 15 min.

Samples are divided into four groups according the duration of hydrofluoric acid (HF) application as follows:

1. Group I (control) (n=10): as received samples without surface treatment.

2. Group II, (n=10): ceramic surfaces etched with HF for 20 seconds; following the manufacturer's recommendations.

3. Group III, (n=10): ceramic surfaces etched with HF for 40 seconds.

4. Group IV, (n=10): ceramic surfaces etched with HF for 60 seconds.

- All surface treated samples will be subjected to:

1. **Surface roughness examination**. Surface Roughness analysis was done using photographed using USB Digital microscope. With a built-in camera (Scope Capture Digital Microscope, Guangdong, and China) connected with an IBM compatible personal computer using a fixed magnification of 120X. The cropped images were analyzed using WSxM software. Calibration was made by comparing an object of known size (a ruler in this study) with a scale generated by the software.

- **Surface topographic analysis using SEM and profilometer.**

Using SEM Model Quanta 250 FEG (Field Emission Gun), with accelerating voltage 30 K.V. and magnification $14x$ up to 1000000. The magnification used in this study was $2000x$.

- **Color stability testing (Δ E).**

A spectrophotometer (FEI Company, Netherlands) was used for the reflectance measurements of the disc samples. The color change value (ΔE) was calculated by the following equation:

$$\Delta E = [(\Delta L^*) 2 + (\Delta a^*) 2 + (\Delta b^*) 2]1/2.$$

L* stands for lightness, a* for green-red and b* for blue-yellow. ΔL^*, Δa^*, Δb^* correspond to the differences between specimens after etching and after bonding

Obtained data will be tabulated and statistically analyzed.

RESULTS

I-Effect of etching time on roughness

a) Comparison within the same group

In E-max group, the highest mean value was recorded in 60 second etch, followed by 40 sec etch then, control (no etch); with the least value recorded in 20 second etch. ANOVA test revealed that the difference between etching time was statistically significant (p<0.0001). Tukey's post hoc test revealed a significant difference between each 2 etching subgroups (table 1, Fig. 1)

In Obsidian group, the highest mean value was recorded in 40 second etch, followed by 60 sec etch then, control (no etch); with the least value recorded in 20 second etch. ANOVA test revealed that the difference between etching subgroups was not statistically significant (p=0.361), (Table 1, Fig. 1)

TABLE (1) Values of roughness and effect of etching time within the same group (ANOVA test)

Roughness		Mean	Std. Dev	Std. Error	95% Confidence Interval for Mean		Min	Max	F	P
					Lower Bound	Upper Bound				
EMAX	Control (No etch)	.2545[c]	.00	.00	.25	.26	.25	.26	108.002	.000*
	20 sec. etch	.2523[d]	.00	.00	.25	.25	.25	.25		
	40 sec. etch	.2567[b]	.00	.00	.26	.26	.26	.26		
	60 sec. etch	.2647[a]	.00	.00	.26	.27	.26	.27		
Obsidian	Control (No etch)	.2538[a]	.00	.00	.25	.26	.24	.26		
	20 sec. etch	.2505[a]	.01	.00	.24	.26	.22	.27	1.089	.361 NS
	40 sec. etch	.2567[a]	.00	.00	.26	.26	.25	.26		
	60 sec. etch	.2546[a]	.01	.00	.25	.26	.21	.27		

*Significance level P<0.05, *Significant, NS=non-significant*

Tukey's post hoc test: Within the same comparison, means sharing the same superscript letter are not significantly different

TABLE (2) Comparison of values of roughness between E-max and Obsidian (independent t test)

Etching	Group	Mean	SD	t	P
Control	E max	.2545	.00	.663	0.516NS
	Obsidian	.2538	.00		
20 sec. etch	E max	.2523	.00	.525	0.608NS
	Obsidian	.2505	.01		
40 sec. etch	E max	.2567	.00	0	1 NS
	Obsidian	.2567	.00		
60 sec etch	E max	.2647	.00	7.35	0.00*
	Obsidian	.2546	.01		

*Significance level P<0.05, *Significant, NS=non-significant*

TABLE (3a) Comparison of Values of roughness in different etch time (2 ways ANOVA)

Etch time	Mean	Std. Error	95% Confidence Interval		F	P
			Lower Bound	Upper Bound		
Control	.254[b]	.001	.252	.257	9.560	.000*
20 sec etch	.251[c]	.001	.249	.254		
40 sec etch	.256[b]	.001	.253	.258		
60 sec etch	.261[a]	.001	.258	.263		

*Significance level P<0.05, *Significant*

Tukey's post hoc test: means sharing the same superscript letter are not significantly different
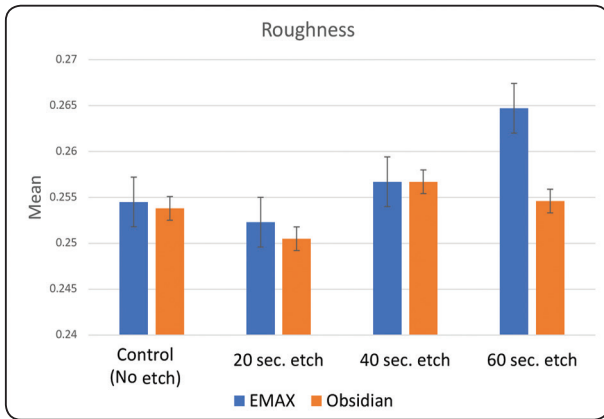
Fig. (1) Bar chart showing mean values of roughness and effect of etching time in both groups

b) Comparison between groups

In control subgroups, a higher mean value was recorded in E-max, with no significant difference (p=0.516), (Table 2, Fig. 1)

In 20 sec etch subgroups, a higher mean value was recorded in E-max, with no significant difference (p=0.608), (Table 2, Fig. 1)

In 40 sec etch subgroups, the same mean value was recorded in both groups, with no statistically significant difference (p=1), (Table 2, Fig. 1)

In 60 sec etch subgroups, a higher mean value was recorded in E-max, with a significant difference (p=0.00), (Table 2, Fig. 1)

C) Interaction of variables

Two ways ANOVA test was used to study the interaction of both etching time and group variables. **Regarding the etching time variable**, the highest mean value was recorded in 60 second etch, followed by 40 sec etch, then control (no etch); with the least value recorded in 20 second etch. Two ways ANOVA test revealed that the difference between etching time was statistically significant (p<0.0001). Tukey's post hoc test revealed a significant difference between each 2 etching times (Table 3a)

Regarding the group variable, a higher mean value was recorded in E-max with statistically significant difference (p=0.014), (Table 3b)

Two ways ANOVA test revealed that the effect of interaction of both variables was not statistically significant (p=0.176), (Table 3c)

II- Effect of etching time on color

TABLE (3b) Comparison of Values of roughness in different groups (2 ways ANOVA)

Group	Mean	Std. Error	95% Confidence Interval		F	P
			Lower Bound	Upper Bound		
E max	.257	.001	.255	.259	6.247	.014*
Obsidian	.254	.001	.252	.256		

Significance level P<0.05, *Significant

TABLE (3c) Results of 2 ways ANOVA test for roughness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
-Etch_time	.001	3	.000	9.560	.000*
-Group	.000	1	.000	6.247	.014*
Etch time * Group	.000	3	.000	1.679	.176NS

Significance level P<0.05, *Significant, NS=non-significant

a- Comparison within the same group

In E-max group, the highest mean value was recorded in 60 second etch, followed by 40 sec etch then 20 second etch; with the least value recorded in control (no etch). ANOVA test revealed that the difference between etching time was statistically significant (p<0.0001). Tukey's post hoc test revealed a significant difference between each 2 etching subgroups (Table 4, Fig. 2)

In Obsidian group, the highest mean value was recorded in 60 second etch, followed by 40 sec etch then 20 second etch,; with the least value recorded in control (no etch). ANOVA test revealed that the difference between etching subgroups was statistically significant (p<0.0001). Tukey's post hoc test revealed a significant difference between each 2 etching subgroups (Table 4, Fig. 2)

b- Comparison between groups

In control subgroups, a higher mean value was recorded in E-max group, with statistically significant difference (p<0.0001), (Table 5, Fig. 2)

In 20 sec etch subgroups, a higher mean value was recorded in E-max, with statistically significant difference (p<0.0001), (Table 5, Fig. 2)

In 40 sec etch subgroups, a higher mean value was recorded in E-max, with statistically significant difference (p<0.0001), (Table 5, Fig. 2)

In 60 sec etch subgroups, a higher mean value was recorded in E-max, with statistically significant difference (p<0.0001), (Table 5, Fig. 2)

C- Interaction of variables

Two ways ANOVA test was used to study the interaction of both etching time and group variables. **Regarding the etching time variable**, the highest mean value was recorded in 60 second etch, followed by 40 sec etch, then 20 second etch; with the least value recorded in control (no etch). Two ways ANOVA test revealed that the difference between etching time was statistically significant (p<0.0001). Tukey's post hoc test revealed a significant difference between each 2 etching times (Table 6a)

Regarding the group variable, a higher mean value was recorded in E-max with statistically significant difference (p<0.0001), (Table 6b)

Two ways ANOVA test revealed that the effect of interaction of both variables was statistically significant (p<0.0001), (Table 6c)

TABLE (4) Values of colour difference   and effect of etching time within the same group (ANOVA test)

| | | Mean | Std. Dev | Std. Error | 95% Confidence Interval for Mean | | Min | Max | F | P | | |
					Lower Bound	Upper Bound						
E_max	Control (No etch)	1.4733[d]	.03	.01	1.46	1.49	1.45	1.51	641.883	.000*		
	20 sec. etch	2.4267[c]	.14	.04	2.35	2.50	2.25	2.57				
	40 sec. etch	3.2128[b]	.13	.03	3.14	3.28	3.12	3.38				
	60 sec. etch	4.1976[a]	.54	.14	3.90	4.50	3.49	4.75				
Obsidian	Control (No etch)	1.0809[d]	.04	.01	1.06	1.10	1.04	1.14	327.489	.000*		
	20 sec. etch	1.3588[c]	.25	.06	1.22	1.50	1.04	1.61				
	40 sec. etch	2.5569[b]	.10	.03	2.50	2.61	2.48	2.69				
	60 sec. etch	3.3295[a]	.18	.05	3.23	3.43	3.15	3.57				

*Significance level P<0.05, *Significant*

Tukey's post hoc test: Within the same comparison, means sharing the same superscript letter are not significantly different

TABLE (5) Comparison of values of color difference between E-max and Obsidian (independent t test)

Etching	Group	Mean	SD	t	P
Control	E max	1.4733	.03	-30.934	0.00*
	Obsidian	1.0809	.04		
20 sec. etch	E max	2.4267	.14	-14.632	0.00*
	Obsidian	1.3588	.25		
40 sec. etch	E max	3.2128	.13	5.632	0.00*
	Obsidian	2.5569	.10		
60 sec etch	E max	4.1976	.54	6.61	0.00*
	Obsidian	3.3295	.18		

*Significance level P<0.05, *Significant*
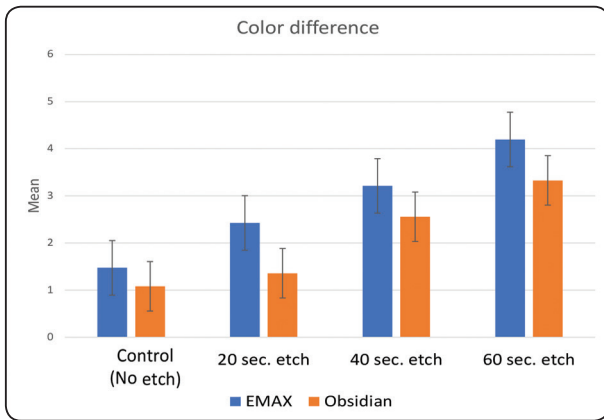
Fig. (2) Bar chart showing mean values of color difference and effect of etching time in both groups

Correlation between roughness and color (Table 7, Fig. 3)

Pearson correlation test revealed a statistically significant strong positive correlation between color difference and roughness in E-max group (p<0.0001)

Pearson correlation test revealed a non-significant weak positive correlation between color difference and roughness in Obsidian group (p=0.446)

Pearson correlation test revealed a statistically significant moderate positive correlation between color difference and roughness in both groups (all together) (p<0.0001)

TABLE (6A) Comparison of Values of color difference in different etch time (2 ways ANOVA)

Etch_time	Mean	Std. Error	95% Confidence Interval		F	P
			Lower Bound	Upper Bound		
Control	1.277[d]	.043	1.193	1.362	640.808	.000*
20 sec etch	1.893[c]	.043	1.808	1.977		
40 sec etch	2.943[b]	.043	2.859	3.028		
60 sec etch	3.705[a]	.043	3.621	3.790		

*Significance level P<0.05, *Significant*

Tukey's post hoc test: means sharing the same superscript letter are not significantly different

TABLE (6B) Comparison of Values of color difference in different groups (2 ways ANOVA)

Group	Mean	Std. Error	95% Confidence Interval		F	P
			Lower Bound	Upper Bound		
E max	2.611	.030	2.551	2.670	53.394	.000*
Obsidian	2.299	.030	2.239	2.358		

Significance level P<0.05, *Significant

TABLE (6C) Results of 2 ways ANOVA test for color difference

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
-Etch_time	105.144	3	35.048	640.808	.000*
-Group	2.920	1	2.920	53.394	.000*
-Etch time * Group	18.537	3	6.179	112.975	.000*

*Significance level P<0.05, *Significant*
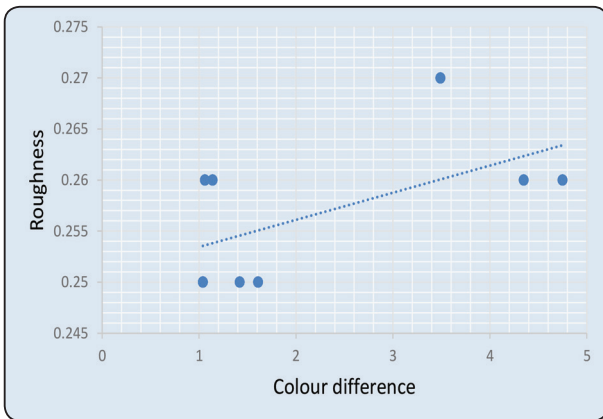
Fig. (3a) Scatter plot showing strong positive correlation between color difference and roughness in E-max group
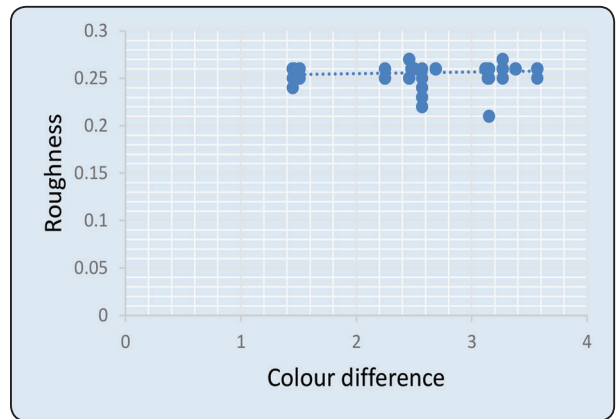
Fig. (3b) Scatter plot showing weak positive correlation between color difference and roughness in obsidian group
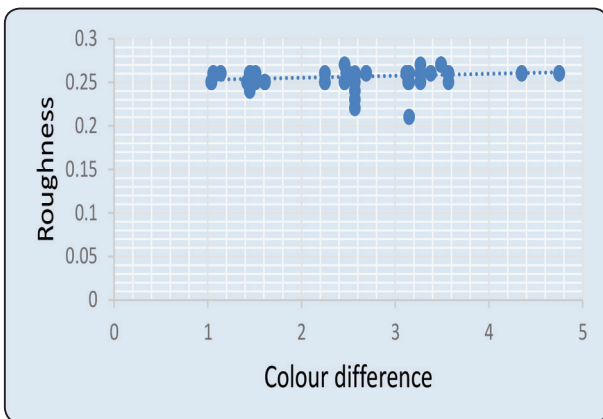
Fig. (3c) Scatter plot showing moderate positive correlation between color difference and roughness

TABLE (7) Results of Pearson's correlation test

		Correlation
	Pearson Correlation	.820
E max	P value	.000*
	significance	Strong positive
	Pearson Correlation	.100
Obsidian	P value	.446 NS
	significance	Weak positive
Overall (Both groups)	Pearson Correlation	.335
	P value	.000*
	significance	Moderate positive

*Significance level P<0.05, *Significant, NS=non-significant*

ESM

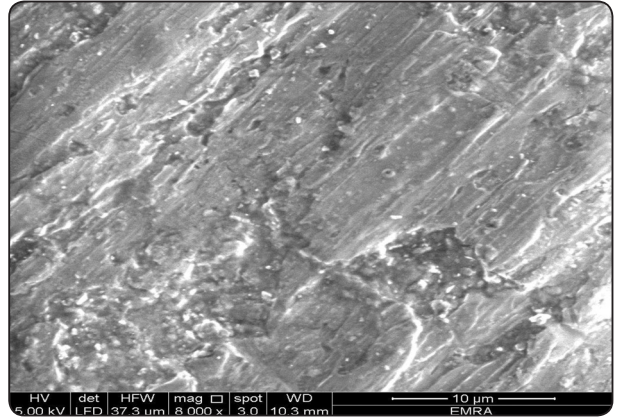Fig. 4 (a) electron microscopic scanning of Control group (E max)

Fig. 4 (b) electron microscopic scanning of Control group (obsidian)
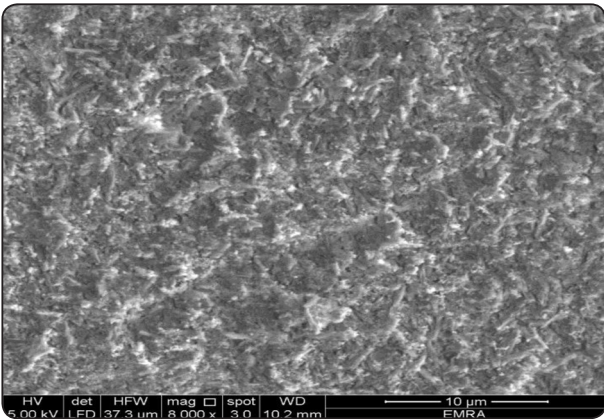

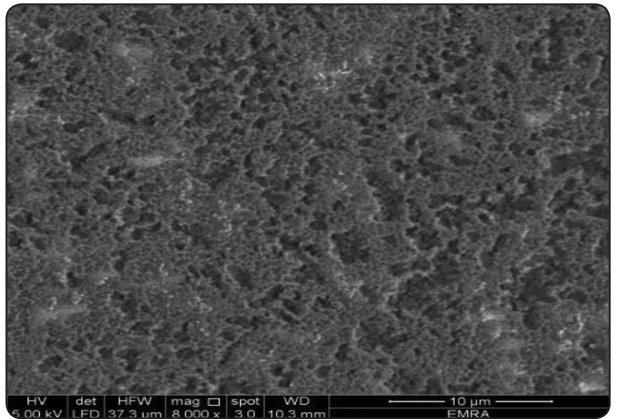Fig. 5 (a) Electron microscopic scanning of 20 second treatment (E max)

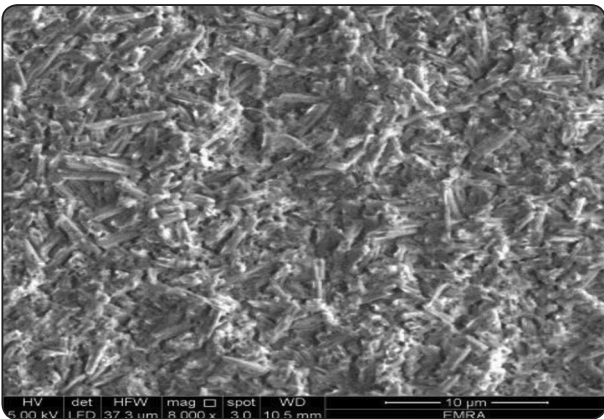Fig. 5 (b) Electron microscopic scanning of 20 second treatment(obsidian)

Fig. 6 (a) Electron microscopic scanning of 40 second treatment (E max)
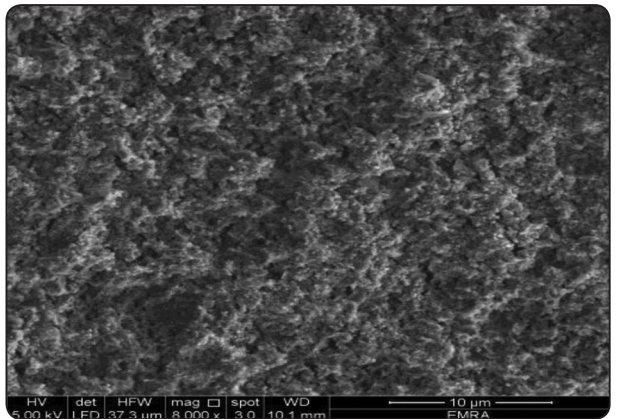

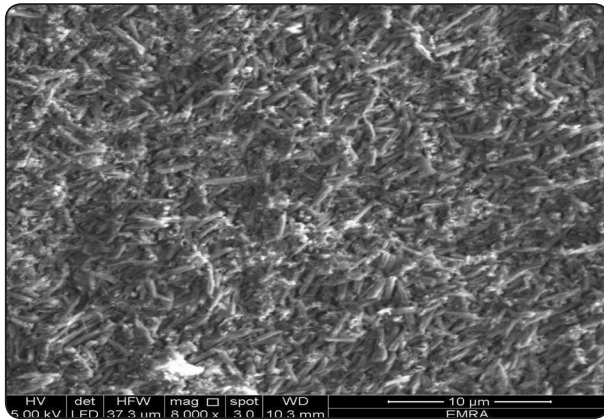Fig. 6 (b) electron microscopic scanning of 40 second treatment (obsidian)

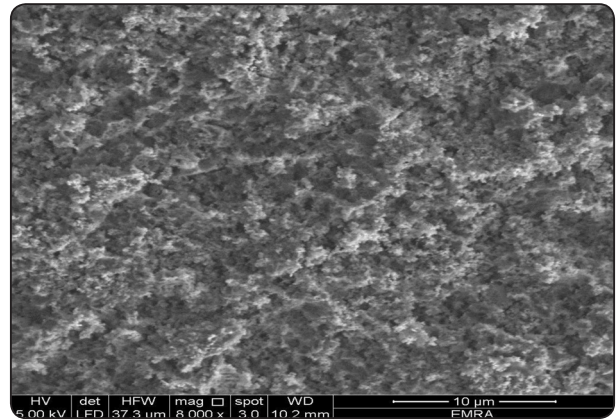Fig. 7 (a) electron microscopic scanning of 60 second treatment (E max)

Fig. 7 (b) electron microscopic scanning of 60 second treatment (obsidian)

DISCUSSION

Improvements in CAD/CAM technology have led to development of metal free restorative materials with different mechanical and optical properties. Regardless of the microstructure of the restorative material, adhesive luting protocol requires conditioning of the intaglio surface of the restoration to enhance the bond strength of restorative material to resin cement. It has been reported that surface treatment affects not only bond strength but also mechanical and optical properties of ceramics. Therefore, this in vitro study investigated the effect of different surface treatments on color of current CAD/CAM restorative materials.

In this study; etching was done with HF acid with four different etching times (0, 20, 40, and 60). It is known that HF etching of porcelain provides the necessary surface roughness to mechanical interlocking but over etching could have a weakening effect on the porcelain. Therefore, it is important to know the adequate HF etching time for micromechanical retention without weakening the ceramic. This is the reason why the present study investigated the adequate etching protocol for a lithium disilicate-based glass ceramic.

Numerous studies have been conducted comparing the effects of different etchants on the microstructure of glass-ceramics [18, 19, 22,23and 24]. previous study found that HF produced the most aggressive etching pattern with the most prominent topographic pattern on all dental ceramics examined due to the high roughness values obtained (Ra = 1.4 μm, Rq = 2.1 μm, and Rt = 39.8 μm) compared with acidulated phosphate fluoride.

Regarding the etching time, many studies have been done with different kinds of ceramics and HF etchants. Chen et al [25]. evaluated two HF etchants (2.5 and 5%) and seven different etching times (0, 30, 60, 90, 120, 150, and 180 sec). Wolf et al [6] evaluated the surface roughness of feldspathic porcelain etched with 9.5% HF for 30, 60, 150, and 300 sec and the tensile bond strength to resin. The authors found a positive correlation between ceramic surface roughness and increasing HF etching time [26, 27, 28], which agreed with other studies and with the present study.

In the current study, the hydrofluoric acid treatment of ceramic surfaces for 60 seconds showed the highest roughness value with significant difference with the other groups. This may attributed to the further loss of the glassy phase around the crystals, which exposed the crystal structure.

The emax samples showed a significant surface roughness more than the obsidian samples; this may

attributed to the morphological difference between the two types of crystals. The lithium disilicate crystals in e max material process a needle like or tetragonal shape. While the lithium silicate crystals in obsidian material process a spherical or monoclinic shape. So that after removal of glassy matrix the needle like crystals will project sharper [29, 30] and more than the other spherical lithium silicate crystals.

In case of short duration HF etching, it acts on localized areas, promoting dissolution of small amount of the glass matrix and impurities [31, 32]. Increasing duration leads to more homogeneous surface, due to extensive loss of the glassy matrix and pullout of lithium silicate/zirconia grains,

In the current study, the highest color difference value ΔE was found in samples treated with 60 second hydrofluoric acid etching for both groups' e max as well as obsidian. Increasing itching time leads to further loss of glassy matrix [33]. So in the highest etching time (60 second) the largest amount of glassy matrix removed. which agreed with other studies[34, 35, 36, 37]

This glassy matrix is responsible for the translucency of ceramic. So that the maximum opacity was found in 60 second etching time group as it processes the maximum amount of crystals and the least amount of glassy matrix.

The higher mean value of color difference was in e-max groups than obsidian with statistically significant difference. This will be attributed to the higher surface roughness of e-max that leads to increasing the obesity of the material.

In the present study, SEM images of the etched and unetched ceramic surfaces noticeably represented the effect of the different etching durations on the microstructure of the glass ceramic. SEM images revealed numerous irregularities and voids in the etched ceramic surfaces as well as elongated lithium silicate crystals in comparison with the unetched ceramic surfaces, which displayed

homogenous patterns. This is explained by the selective removal of the glassy matrix in the treated specimens 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 specimens etched for 60 seconds versus those etched for 10 seconds, which demonstrated fewer microstructure alterations.

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