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THE EFFECT OF MULTIPLE FIRING ON THE COLOR, TRANSLUCENCY, AND FLEXURAL STRENGTH OF NEWLY INTRODUCED LITHIUM DISILICATE CERAMICS

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ABSTRACT

Statement of problem: Ceramic is subjected to multiple firing which exposed it to more heat treatments. The effect of these heat treatments on optical and mechanical properties of lithium disilicate glass-ceramics is not fully studied.

Objective: To evaluate the Impact of multiple firings cycles on color change, translucency, and flexural strength of lithium disilicate ceramic fabricated by press and milled technology.

Material and Methods: Lithium disilicate ceramics fabricated by two methods were used. Sixty disk-shaped samples (10x 1mm) were prepared from ceramics thirty for each group (Amber press (P) and Amber mill (M)) (n=30). Then each group (P and M) was divided into three subgroups according to the number of firing cycles one, three, and five times (n=10). After finishing firing cycles for each group**,** color change (ΔE) and translucency measurement (TP) were done using Vis-NIR spectrophotometer. The biaxial flexure strength was measured for all samples. Two-way Analysis of Variance (ANOVA) was made to analyze the effect of ceramic type, firing cycles and their interaction on mean TP, ΔE and biaxial flexural strength. When ANOVA test is significant so Bonferroni's post-hoc test was used for pair-wise comparisons. The significance level was set at $P \le 0.05$.

Results: Statistical analysis showed that ceramic type and firing cycles had a statistically significant effect on mean colour change (ΔE), translucency (TP) and biaxial flexural strength. The interaction between the two independent variables had a statistically significant effect on mean ΔE.

Conclusions: Multiple firings affect the color change and translucency of lithium disilicate ceramics. More firing cycles led to more reduction in flexural strength of Amber milled compared to that of Amber press. Amber mill has better translucency and biaxial flexural strength than the Amber Press.

KEYWORDS: Ceramic, Lithium disilicate, Color, Firings, Translucency.

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INTRODUCTION

Natural appearance of teeth and a beautiful smile is in high demand these days which leads to the development of more aesthetically appealing restorations. In the past, metal ceramic restorations were used for their excellent fracture resistance, although color reproduction in metal ceramics is difficult due to their metal substructure which increases light reflectivity especially in the anterior zone[1]. Because of the high demand of aesthetics, all ceramic systems were introduced to dentistry. They are highly aesthetic restorations with excellent optical properties which do not deteriorate with time. Also, they are chemically stable, biocompatible, have a high compressive strength and a similar co-efficiency of thermal expansion to the tooth structure. However, under tensile strength they are fragile [2].

Lithium disilicate glass-ceramics are a type of dental ceramics that are highly functional in restorative dentistry due to their greater aesthetic properties, and beside that their hardness matches to natural teeth [3]. It has a major crystalline phase of $Li_2Si_2O_5$, which shows a microstructure with elongated crystals form an interlocking pattern [4]. Due to its high strength and toughness, restorations made from this material can be cemented by conventional or adhesive cement [3]. Lithium disilicate is a material that can be used in all areas of the mouth when specific considerations are considered. Besides that, due to it high aesthetic and strength properties allows it to be a core for a veneered restoration or for full contour restoration fabricated from one high-strength ceramic[5]. For laboratory ceramists, the versatility and performance of lithium disilicate enable them to optimize their productivity, since it can be fabricated by either heat pressing or CAD/CAM milling techniques[6]. lithium disilicate "Ingot-type" has been used in the lost wax technique for fabrication of the conventional dental fixed-restoration. Recently, a lithium disilicate block has been introduced to be used in milling procedures due to the advance of CAD/CAM (computer-aided design and computer-aided manufacturing) technology. The lithium disilicate block is used in the intermediate state of crystallization ($Li_2Si_2O_5$) for an easy milling procedure, increased cutting efficiency, and minimal waste of milling tools, , and it is necessary to have additional thermal refinement processes after milling for crystallization enrichment [7].

Dental ceramics are fired several times for many reasons during the fabrication of fixed prosthodontics. Firing is needed after grinding and polishing to release stresses. Also, it is needed for the layering and staining of restoration. Besides that, achieving the proper color and optical effects of the final restoration need multiple firings. All these procedures may cause stresses concentration on the ceramic surface and bring about alternations of the physical and mechanical properties of the ceramics [8].

Amber mill and Amber press are among the newly introduced lithium disilicate ceramics, they are characterized by their aesthetic values, structural stability, and edge stability. Amber Mill utilizes NLD (nano lithium disilicate) technology that allows the translucency to be controlled by firing temperature. While Amber Press ingots offer an acid-free workflow. Due to its nearly imperceptible reactionary layer [9,10]. However, the research on these materials is limited, and therefore, their physical and material properties need to be appropriately determined for better understand their quality and limitations when exposed to numerous firings. Therefore, this in vitro study was aimed to investigate the effects of multiple firing on color, translucency, and flexure strength of Amber lithium disilicate ceramic material fabricated with two different techniques. The null hypothesis was that the materials' color, translucency, and flexure strength would not be affected as the number of firings increased.

MATERIALS AND METHODS

Sample grouping

Based upon the results of previous study[8], the effect sizes were f = 1.93 and 1.57 for the two factors (Ceramic type, firing cycles), respectively assuming that SD within the cell is 0.5. Using alpha (α) level of (5%) and Beta (β) level of (20%) i.e., power = 80%; the study should include 7 samples per cell. A total of 60 ceramic discs were constructed and prepared in this study. Discs were divided into two main groups according to technique of fabrication; Group P: ceramic discs of Amber press (30 discs) and Group M*:* ceramic discs of Amber mill (30 discs). According to the number of firing cycle each group was divided into three subgroups: subgroup (1): 1 firing cycle (10 discs), subgroup (3): 3 firing cycles (10 discs) and subgroup (5): 5 firing cycles (10 discs).

Pressable Ceramic Samples Fabrication

Digital designing of discs was done by software 3D builder. The disc specimen is designed with dimensions 10mm diameter 1mm thickness. Then wax cad material was used to mill the designed discs using VHF CAM5-S1. The dimension accuracy of the milled wax pattern was achieved by using digital caliper to check the wax pattern dimensions (10 mm diameter and 1 mm thickness). Round wax sprues of 3mm in length and 2.5mm in diameter were attached to the disc axially 45 degrees. Then settled to sprued wax discs on ring base. Investing was carried out following the manufacturer's instructions. A ring was prepared and a wetting agent was added. The investment material was poured in ring and left to set for 30 minutes. After complete setting of the investment, it was inserted in the burnout furnace (BEXCO, India.) and left of 60 mins at 850°C then removed. An Ingot of Amber SP A2 was placed in the investment ring and the plunger was positioned and was placed in the center of EP3000 press furnace (Ivoclar Vivodent, Germany), and the firing program was selected and activated (table 1). The investment ring was removed and allowed to be cooled to room temperature then devesting was done followed with roloblast was used to remove the first step of the investment material with Renfert Sandblaster (Renfert, Germany). The discs were added to Invex liquid and left for 15 mins in an ultrasonic machine (Effica, L&R, USA). The discs were then cut from the sprue. After that, they were polished using a white diamond polisher and sandpaper.

Fabrication of CAD/CAM Ceramic Samples

Blocks of Amber mill during the non-crystalized stage were rounded with a lathe machine (Semi-Automatic EKL-1700) vertically to obtain a cylindrical block with a diameter of 10mm. Each block was then sectioned horizontally by an Isomet (Buehler IsoMet 4000 Precision Cutter, Illinois, USA) in order to obtain discs of thickness 1mm so a disc of dimension 1mm thickness and 10mm diameter was made. Discs were crystallized to achieve full sintered stage (Table 2).

After fabricating the 60 discs of Amber mill and Amber Press, 10 discs of each material were fired one cycle (glazing), 10 discs were fired three cycles between glazing and correction, and 10 discs were fired five cycles between glazing and correction (table 3)

TABLE (1): firing program of Amber press.

Start Temp (B,C°)	Heating Rate (°C/ min)	Max Temp. (°C)	Holding Time (Min.)	Vacuum On (°C)	Vacuum Off (°C)
700	60	915	15/20	700	900

TABLE (2): Initial firing for crystallization of Amber mill.

Start Temp (B,C°)	Heating Rate (°C/ min)	Max Temp. (°C)	Holding Time (Min.)	Vacuum On (°C)	Vacuum Off (°C)
400	60	815	15:00	430	739

TABLE (3): Glazing and correction program cycles.

Glaze	Start Temp (B,C°)	Heating Rate (°C/ min)	Max Temp. (°C)	Holding Time (Min.)	Vacuum On (°C)	Vacuum Off (°C)
	403/757	50/90	740/1364	1:00	450/842	739/1362
Correction	Start Temp (B,C°)	Heating Rate (°C/ min)	Max Temp. (°C)	Holding Time (Min.)	Vacuum On (°C)	Vacuum Off (°C)
	403/757	50/90	770/1418	1:00	450/842	769/1416

Color and translucency measurements

The Vis-NIR spectrophotometers (Agilent Cary 5000 spectrophotometer, Agilent, USA.) was used to measure color and translucency for each group after finishing firing cycles according to a quality management system certified to ISO 9001.

The degree of color difference between the compared colors is expressed in ΔE units. The total color difference, according to L*, a*, b* coordinates, is calculated as shown in the equation:

$$\Delta E_{ab}{}^{*} = \sqrt{(\Delta L^{*})^2 + (\Delta a^{*})^2 + (\Delta b^{*})^2}$$

The color difference between the samples placed on an ideal black background and on an ideal white background were calculated and established as a translucency parameter (TP) =

$$(L*_B - L*_W)^2 + (a*_B - a*_W)^2 + (b*_B - b*_W)^2]^2.$$

Where L* = Luminance Reflectance, a*= red-green color coordinate, b*= yellow-blue color coordinates over the black background and the subscript b refers to the color coordinates over the black background and w subscript refers to those over white background.

Biaxial Flexural Strength Testing:

The piston on three balls technique in an Instron testing machine model 3345 (Instron 3345 biaxial flexural strength testing machine, USA.) was used to measure the Flexural strength according to ISO 6872 specification for testing ceramic materials then the data recorded using computer software Bluehill version 3.3. The fracture load for each specimen was recorded and the biaxial flexure strength was calculated using the following equation: $\sigma = [0.2387P (X - Y)]/b^2$. where: σ = Maximum center tensile stress (MPa), P = Maximum load (N), b = Specimen thickness at fracture origin (1mm).

Microstructural analysis

The microstructures were then analyzed by the SEM observations at magnifications 20X and 40X.

Two-way Analysis of Variance (ANOVA) was used to study the effect of ceramic type, firing cycles and their interaction on mean TP, color change (ΔE) and biaxial flexural strength. When ANOVA test is significant so Bonferroni's post-hoc test was used for pair-wise comparisons The significance level was set at $P \leq 0.05$.

RESULTS:

Means ± Standard Deviations are presented in table 4. Two-Way ANOVA showed that ceramic type and firing cycles had significant effect on mean ΔE. The interaction between the two variables had also a significant effect on mean ΔE. Bonferroni's post-hoc test (table 4) showed that either with one, three or five firing cycles; Amber mill had statistically significantly higher mean ΔE than Amber press. In addition, either with Amber mill or Amber press the statistically significantly highest mean ΔE was found after five cycles. The statistically significantly lowest mean ΔE was found after three cycles.

Means ± Standard Deviations of TP values are presented in Table 5. Two-Way ANOVA showed that ceramic type and firing cycles had a statistically significant effect on mean TP. The interaction between the two variables had no statistically significant effect on mean TP. Bonferroni's post-hoc test (table 5) showed either with one, three or five firing cycles; Amber mill had significant higher mean TP than Amber press. In addition, either with Amber mill or Amber press**;** there was no statistically significant difference between three and five firing cycles; both showed statistically significant higher mean TP than one firing cycle.

Means ± Standard Deviations of biaxial flexural strength values are presented in Table 6. Two-Way ANOVA showed the results showed that ceramic type and firing cycles had a statistically significant effect on mean biaxial flexural strength. The interaction between the two variables had no statistically significant effect on mean biaxial flexural strength. Bonferroni's post-hoc test (table 6) showed with one firing cycle; Amber mill showed statistically significantly higher mean biaxial flexural strength than Amber press. Either with three or five firing cycles, there was no statistically significant difference between mean biaxial flexural strength of the two ceramic types. In addition, with Amber mill the one firing cycles recorded the highest mean biaxial flexural strength with non-statistically significant difference from three firing cycles but a statistically significantly higher value than five firing cycles which showed the lowest mean biaxial flexural strength. While with Amber press; there was no statistically significant difference between the different firing cycles.

The SEM images disclosed different crystalline structure in the two lithium disilicate ceramics. The Amber mill showed at the rod-shaped lithium disilicate crystals embedded in a glass matrix, that are interlocked and haphazardly organized (figure 1A). While the Amber press showed long rod-shaped lithium disilicate crystals with large sized grains (figure 2A). Both with increasing firings, the crystal sized decreased and became more

TABLE (4): Mean ± standard deviation (SD) for ΔE values with numbers of firing cycles.

Firing cycles	**Amber mill**	**Amber press**	***P*-value (Between ceramic types)**	**Effect size *(Partial eta squared)***
	Mean±SD	**Mean±SD**		
One	2.16^{B} ±0.13	1.39^{B} ±0.08	<0.001*	0.97
Three	0.88^{C} ±0.14	0.78^{C} ±0.15	0.040*	0.15
Five	3.04^{A} ±0.07	2.09^{A} ±0.15	<0.001*	0.89
P-value (Between firing cycles)	<0.001*	<0.001*		
Effect size *(Partial eta squared)*	0.98	0.95		

: Significant at P ≤ 0.05, Means with different superscript letters within the same horizontal column show statistically significant.

TABLE (5): The mean, standard deviation (SD) values for TP values with interaction between variables.

Firing cycles	Amber mill	Amber press	*P*-value (Between ceramic types)	Effect size *(Partial eta squared)*
	Mean±SD	Mean±SD		
One	17.5 ^{B}±0.4	12.4 ^{B}±0.5	<0.001*	0.96
Three	19.3 ^{A}±0.5	14 ^{A}±0.5	<0.001*	0.97
Five	19.8 ^{A}±0.5	14.2 ^{A}±0.6	<0.001*	0.98
P-value (Between firing cycles)	<0.001*	<0.001*		
Effect size *(Partial eta squared)*	0.664	0.453		

: Significant at P ≤ 0.05, Different superscripts letters in the same column show are statistically significantly different.

TABLE (6): The mean, standard deviation (SD) values of biaxial flexural strength values with interactions between variables.

Firing cycles	Amber mill	Amber press	*P*-value (Between ceramic types)	Effect size *(Partial eta squared)*
	Mean±SD	Mean±SD		
One	300 ^{A}±44.7	188.6±39.3	0.001*	0.270
Three	287.5^{A}±50.3	220.4±55.9	0.018	0.052
Five	207 ^{B}±22.30	200.6±55	0.025	0.001
P-value (Between firing cycles)	0.001*	0. 17		
Effect size *(Partial eta squared)*	0.021	0.071		

: Significant at P ≤ 0.05, Different superscripts letter in the same column show statistically significantly different.

condensed (figure 1,2 (B, C)). At 40X magnification the crystals of Amber milled in the first firing cycle had an average length of 0.97 µm (Fig. 1A) while after 3 firings the crystals size decreased to an average of 0.83 µm (Fig. 1B) and after 5 firings their size decreased to 0.78µm (Fig. 1C). At 40X magnification the crystals of Amber press in the first firing cycle had an average length of 3.42 µm (Fig. 2A), after 3 firings the crystals size decreased to an average of 1.73 µm (Fig.2B) and after 5 firings their size decreased to 1.09µm (Fig. 2C).

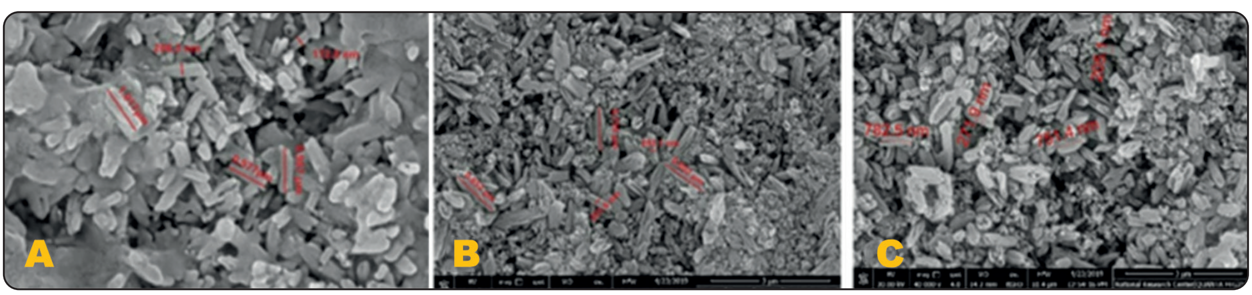

Fig. (1): Representative SEM photos of Amber mill showing, A: First firing, B: Third firings, C: Fifth firings.

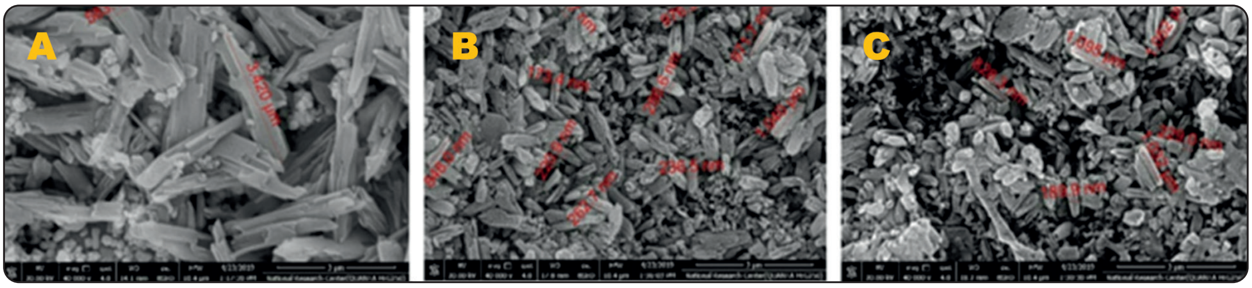

Fig. (2): Representative SEM photos SEM photos of Amber press, A: First firing, B: Third firings, C: Fifth firings.

DISCUSSION

All ceramic restorations are widely accepted as an ultimate esthetic treatment option as they offer superior aesthetics and allow transmission of light a as good as enamel and dentin[11]. The fabrication of a highly aesthetic restoration is a complicated process that requires many steps that expose the process to a great liability of failure[12]. The literature has approved that multiple all-ceramic materials and systems are presently available for different clinical use and there isn't aparticular universal material or system for all clinical situations[11]. Amongst all ceramic materials available in the market is lithium disilicate. Lithium disilicate is one of the best and most commonly used veneer materials in dentistry today[13]. In addition, it has superior biocompatibility, plaque resistance and natural appearance compared to other all ceramic systems[14,15].

While constructing the ceramic restoration, it is fired several times. The first firing after sintering serves to eliminate microcracks and release the stresses associated with the grinding and polishing procedures, as recommended by the manufacturers[16]. It is thought to be necessary to do second and third firings for the making of a restoration using the staining or layering technique, before placement in the mouth. The fourth and subsequent firings are required during shape and colour corrections. It was assumed that after the third firing, an all-ceramic restoration is ready for placement in the mouth by the dentist. It was assumed that any firing after third firings to be necessary only if the dentist needed further shaping and colour corrections of the ceramic restoration[8].

The aesthetics and mechanical strength of a restoration are important requirements for the clinical success of any restoration. Several factors can result in their failure. Many of these failures are related to the restoration fabrication procedure, as the number of times that ceramics are fired. It is prevailing that distribution of the sintering, glass, and crystal phases in the microstructure of the porcelain was affected by the firing cycle and temperature affect, so the optimum strength of all-ceramic restorations could be overwhelmed [16]. Besides that, multiple firing may be the causing factor of the difference between the target colour and the actual colour reached in the definitive restoration. According to the magnitude of this colour difference, the restoration maybe considered unsatisfactory and require replacement[17].

In this study, discs were used for the sample design to ensures standardized size and surface quality specifically when optical properties of a material were tested. Besides that, discs also enhance a flat surface which eliminate the edge loss effect[18]. Color assessment is a complex pyscho-physiologists process, which is subjected to many variables. Due to the inconsistencies in a person's ability to select color matches reliably as well as the subjective nature of color perception, instrumental colorimetric techniques have been used to achieve quantitative evaluation of the color differences[19]. Spectrophotometers were used for color measurements in this study as they have been reported to be the preferred instrument for measuring color as they are accurate and evaluate metamerism in contrast to colorimeters[20,21]. The TP formulation was used to calculate the translucency changes in our study TP because it became the most commonly preferred parameter by researchers in translucency measurements since it is calculated with a formulation similar to color change formulation, and it is revealed that it produces a mathematical result supporting the clinical observations of the conducted studies[22,23]. The biaxial flexural strength was measured using piston on three ball tests in this study as it has been reported to have excellent results when used on brittle materials as dental ceramics and the International Organization for Standardization selected piston on three ball tests to establish Iso 6782 for dentistry-ceramic materials[24,25].

According to the result of the study ceramic material and multiple firing had significant effect on color change, translucency, and biaxial strength. Thus, the null hypothesis was rejected. The result of the present study showed that ceramic material had significant effect on the color change as Amber mill ceramic showed higher color change than Amber press ceramic. The difference in the composition and processing of the two production can narrative for this outcome[26]. Ceramic press specimens are manufactured by controlled crystallization and CAD/CAM consists of two crystallization processes. The pressed ceramics industrially treated and yield the materials from the metasilicate phase (Li_2SiO_3) to lithium disilicate while the CAD/CAM ceramics are completed by heat treatment, which done in the clinic or in the laboratory to dissolve metasilicate phase and lithium disilicate crystallizes. The nucleating agents control this reaction[27]. Beside that it has been reported that the glass phase was not completely depleted of crystallizable material and crystalline growth could still occur. This might be the cause that the Amber Mill is less color stable than Amber Press as pressable ceramics are completely crystallized after pressing while CAD/CAM ceramics my continue to crystalize after sintering so they may not reach full crystallization after sintering[16].

The results of the study proved that the firing cycles also had a significant effect on the final color received. The color differences ΔE obtained after repeated firing presented as a composite of changes in individual color coordinates and clear alternation in color parameters [28]. Repeated firings might result in microstructural changes within the ceramic materials[28]. This was shown in SEM of samples as the grain sized decreased and became more condensed. In addition, color change after multiple firing can also be correlated to the color instability of metal oxides during firings which can alter the final color of the ceramic[12, 29,30]. Furthermore, the color change may be due to the adverse effect of high firing temperatures of all ceramics containing leucite and lithium disilicate[29]. The results of this study were in agreement with Pires-de-Souza Fde et al 2009 [31] and Emam et al 2020[28] who investigated the effect of repeated firing on color stability of ceramics after repeated firing and concluded that color stability is affected by repeated firing for both tested materials. It is agreed that a color difference of $\Delta E \geq 3.7$ is considered to be a poor match and indicates a visually perceivable color difference which is clinically unacceptable[29]. According to measurements of color change of both Amber Mill and Amber Press were implemented after the 1st, 3rd and 5th firing cycle were below 3.7 so they were considered clinically accepted.

In this study, a two-way ANOVA showed significant difference between the translucencies of Amber milled and Amber press as Amber milled had higher translucency. Translucency of dental ceramics depends on many factors such as the ratio of the crystalline to glass phase and difference in the refractive index between these phases, the morphology of crystals, grain size and boundaries, size and distribution of pores, second phase component, additives, and light scattering from the surface[32,33]. The increase in the crystal size will affect the matching of the optics of the glass phase and crystal, resulting in increased opacity of the material[34]. There is a morphological difference/a difference in the morphology between the milled and press Amber as smaller grain size of milled Amber may be increase its translucency. Although the results in our study showed significant increase in translucency due to repeated firings in both Amber Mill and Amber Press samples, it was reported that an additional firing resulted in a significant difference in translucency grade of the ceramic systems and a decrease in the opacity of all veneered materials, thus altering the readings[35,36]. These statements support with the results obtained from the SEM pictures of this study as Amber Mill had smaller sized crystals with higher translucency than the Amber Press. In addition, the increase in the firing cycles caused the crystal size to decrease resulting in increased translucency. Also supporting the results of this study is Li et al. [37], who investigated the effect of firings on a ceramic material after firing 1, 3, and 5 times on the translucency parameter (TP) and color. They founded that TP, ΔL increased with increasing the number of firing cycles. Also, the numbers of firing cycles increased, the porosity volume decreased while color change and translucency increased[37].

The glass–ceramic structure formed after thermal processing models a structural order featuring the macro-scale voids between the glass–crystal interfaces, the micro-scale shape and size of the crystals, and the nano-scale defects in the crystalline lattice[38,39]. It was stated that the strength of a glass–ceramics with a high crystalline volume fraction affected with the crystal size [40]. The result of this study revealed that Amber mill showed statistically significantly higher mean biaxial flexural strength than Amber press. It was reported that lithium disilicates with larger crystal had both interlocking effect and residual stress effect, and the latter could counteract the intensive "interlocking effect" of the crystals, causing the degradation of strength[41]. This could be the reason why during the first firing cycles the Amber Mill had the highest flexural strength and was significantly higher than the Amber Press. This was supported with SEM pictures which revealed that the Amber Mill had smaller grain size than the Amber Press. the results of this study were in disagreement with Fabian Fonzar et al,[42] who reported no difference between the flexural strength of lithium disilicate press and CAD ceramics. These disagreements could be attributed to different in the measurement of the flexural strength and different types of material.

The flexural strength of the Amber Mill samples was significantly decreased after 5 firing cycles, although the flexural strength of the Amber press decreased after multiple firing cycles but was statistically insignificant this may be due decrease in crystal size with increasing firings. The SEM of Amber milled showed to smaller crystal size after 5 firing cycles 0.78 µm. This was agreement with Zhang et al[43] who found the decrease in strength with decreasing crystal size within a certain range. On the other hand, Ozdogan et al[44] found that the repeat firing processes did not affect the flexural strength of pressed and milled lithium disilicate ceramics. These differences could be attributed to different materials and samples shape.

Limitations of the present study included the in vitro design that might not properly reflect the clinical conditions. So in vivo studies are needed to reach a specific conclusion regarding the durability of the materials tested. Future studies should evaluate the effects of firing cycles on the surface roughness, microstructure, and thermal changes of the materials.

CONCLUSION

Under the limitation of this study, several conclusions could be drawn:

1- Multiple firings increased the color change and translucency of lithium disilicate ceramics.

2- More firing cycles led to more reduction in flexural strength of Amber milled compared to that of Amber press.

3- Amber mill has better translucency and biaxial flexural strength than the Amber Press.

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