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ORIGINAL ARTICLE

Impact of thermocycling on surface roughness, microhardness and optical properties of three different lithium disilicate ceramics

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KEYWORDS

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Abstract *Objectives:* This investigation was carried out to examine the influence of thermocycling aging on the surface roughness (R_a, μm), color parameters (L*, a*, b*), lightness change (ΔL*), chroma change (ΔC^*_{ab}), color change (ΔE), and microhardness (VH) of three lithium disilicate ceramics.

Materials and methods: Forty-five specimens were prepared from three lithium disilicate materials (n = 15)—e.max CAD (EC), e.max Press (EP), and GC Initial LiSi Press (LP) ceramics—with dimensions of 6.0 × 1.2 × 16.0 ± 0.2 mm following the manufacturers' instructions. All specimens were exposed to 5000 thermal cycles with bath temperatures ranging between 5 °C and 55 °C. Data of surface roughness, color parameters, and microhardness were obtained using an optical profiler, a spectrophotometer, and a Vickers hardness tester, respectively. One-way ANOVA, a post-hoc Tukey's test, and a paired sample *t*-test were implemented for statistical analysis ($p \leq 0.05$).

Results: For surface roughness, insignificant differences were reported between the materials

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either before or after thermocycling ($p > 0.05$) while each material displayed a significant increase after being subjected to thermocycling ($p < 0.05$). For color parameters, LP showed significantly lower L* and b* after thermocycling while EC presented a significant reduction in a* in comparison with other materials ($p < 0.05$). EP showed a significant decrease in ΔL^*, ΔC^*_{ab} and ΔE compared with other materials ($p < 0.05$). Regarding microhardness, LP showed significantly increase value in comparison with other materials ($p < 0.05$).

Conclusions: Thermocycling had a major impact on the surface roughness, microhardness and optical characteristics of the tested materials. E.max Press displayed less changes in (ΔL^*), (ΔC^*_{ab}) and (ΔE), while GC LiSi Press had better behavior in terms of microhardness.

1. Introduction

Lithium disilicate is a popular material used in dental ceramics. This material consists mainly of randomly oriented, needle-shaped lithium disilicate crystals imbedded within the glossy matrix (Plengsombut et al., 2009). The role of these crystals is to arrest crack propagation and accordingly enhance the material's strength (Holand et al., 2000; Huang et al., 2017).

Restorations of lithium disilicate can be constructed using heat-pressed and computer-aided design (CAD), as well as computer-aided manufacturing (CAM) technology. For the heat-pressed technique, lithium disilicate is usually manufactured in the sort of ingots, which are fully crystalized; during heating and pressure, these ingots become viscous and pressable, thus formulating the required restoration. For the CAD/CAM fabrication techniques, the lithium disilicate is usually offered in the form of blocks, where the material is partially crystalized and requires milling, and then complete crystallization for the final restoration (Bischoff et al., 2011; von Clausbruch et al., 2000; Wen et al., 2007). During crystallization, the final shape, content, and size of the crystals are defined (Anusavice and Zhang, 2005; HASSELMAN and FULRATH, 1966). Oxides in different proportions could be added to define the final color and translucency of the restorations (Anusavice et al., 1994a, 1994b).

Recently, a new mean of generating lithium disilicate ceramics has been promoted to enhance the ceramic esthetic outcomes and mechanical characteristics. It has been proven to have high flexural strength and elastic modulus (Al-Thobity and Alsalman, 2021). Nevertheless, the impacts of thermocycling on the surface roughness (R_a, μm), color change (ΔE), and microhardness (VH) have not been well evaluated. Thus, this investigation aimed to explore the influence of thermocycling on the surface roughness, lightness change (ΔL^*), chroma change (ΔC^*_{ab}), color change (ΔE), and microhardness of three lithium disilicate materials. The null hypothesis was set as that insignificant differences would be reported between the lithium disilicate materials after being subjected to thermocycling regarding surface roughness, color changes, and microhardness.

2. Materials & methods

2.1. Study group and specimen preparation

Forty-five specimens (n = 15, each group) of three types of lithium disilicate materials were fabricated; two types were heat pressed, while the third type was manufactured using CAD/CAM technology. Material composition and processing protocols were reported in a previous study (Al-Thobity and Alsalman, 2021). To prepare the heat-pressed specimens, 30 polymethyl methacrylate CAD/CAM blocks (AcrylCAD, Ivoclar, Liechtenstein) were employed and cut using a diamond saw (ISOMET 1000, Buehler, IL, USA) at low speed to fabricate 30 specimens with dimensions of 6.0 × 1.2 × 16.0 ± 0.2 mm. After that, the specimens' distribution was randomly made into two groups (n = 15), where EP group comprised of e.max Press specimens (IPS e.max Press HT/A1, Ivoclar, Liechtenstein) and LP group contained LiSi Press specimens (GC Initial LiSi Press HT/E58, GC America, Tokyo, Japan). Sprueing of the polymer specimens was performed using a wax wire (BEGO wax wire, BEGO GmbH, Bremen, Germany) linked to an investment ring (IPS Investment, Ivoclar, Liechtenstein) and then invested using a press ceramic investment (IPS PressVest, Ivoclar, Liechtenstein). Preheating and subsequent pressing of the specimens were conducted using a press furnace (Programat EP5010, Ivoclar, Liechtenstein) following the manufacturers' instructions; the firing temperature was 910 °C with 15 min holding time for the EP specimens and 897 °C with 20 min holding time for the LP specimens.

For the CAD/CAM lithium disilicate material (EC; Group 3), specimens (n = 15) were prepared from lithium disilicate blocks (IPS e.max CAD HT/A1, Ivoclar, Liechtenstein). Specimen cutting was executed using a diamond saw (ISOMET 1000, Buehler, IL, USA) at low speed, resulting in specimens with dimensions of 6.0 × 1.2 × 16.0 ± 0.2 mm. The specimens' crystallization was conducted using a furnace (Programat EP5010, Ivoclar, Liechtenstein) following the manufacturer's directions, where the firing temperature was 850 °C and the holding time was 7 min.

All specimens were finished and polished with #600, #1200, and #2400 grit silica papers until the required dimensions were obtained, which was confirmed using a digital caliper (extra-large LCD screen digital caliper, Neiko Tools US, LaPorte, IN). After this, all specimens were kept for 48 h at room temperature.

2.2. Specimen holder specifications

A custom holder (Aquasil Putty, Dentsply Sirona, North Carolina, USA) was fabricated to keep specimens in place during testing. To unify the surface roughness and color change recordings, permanent points were placed on each aspect of the holder. Marks were positioned on the holder toward the

center of each long side of the specimen, 4 mm from the specimen's left side away from the center and 4 mm from the right side. In addition, one point of recording was positioned at the center of the holder's short side. Thus, three points of recording were measured for each specimen, and the average was subsequently determined.

2.3. Surface roughness and color change baseline measurements

All specimens for the three groups were subjected to baseline surface roughness and color change measurements before aging. Surface roughness (R_a, μm) readings were recorded at three points for each specimen using an Optical Profiler (Contour GT-K1 optical profiler; Bruker Nano, AZ, USA). All three scans were performed on each specimen at 20× magnification. Data analysis of the acquired images was then done using Vision64 software (Vision64 Map; Bruker Nano) (Fig. 1).

Measurements of color change were performed using a spectrophotometer (Color-Eye 7000A; X-rite, Grand Rapids, Michigan, USA). Following the manufacturer's directions, black and white ceramic tiles were employed to calibrate the spectrophotometer. The color values were secured using the CIE L*a*b* color system, where the measurements of the color variables (L*, a*, b*) were performed on each side and at the specimen's center.

2.4. Thermocycling protocol

All specimens were exposed to the aging process wherein thermocycling was performed. The specimens were placed in the thermal cycling machine (ProtoTech, El Segundo, California, USA) and exposed to 5000 cycles. The range of the bath temperature was from 5 °C to 55 °C, with 30 s of a dwell time for each bath and 10 s of transfer time between the different baths.

2.5. Surface roughness and color changes post-aging measurements

Next, all specimens were conveyed to perform post-aging surface roughness (R_a, μm) and color variable (L*, a*, b*) measurements. Changes in lightness (ΔL*) and chroma (ΔC^*_{ab}) were identified comparing the baseline and post-aging color variables using the following formulas; $\Delta L^* = (L^*1 - L^*0)$, $\Delta C^*_{ab} = (\Delta a^{*2} + \Delta b^{*2})^{1/2}$ (Burns, 1997; Cho et al., 2006). In addition, color change (ΔE) was determined by using the following formula (Yuan et al., 2018): $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

2.6. Microhardness measurements

To assess the impact of thermocycling on the hardness of the materials, all specimens were positioned in a microhardness

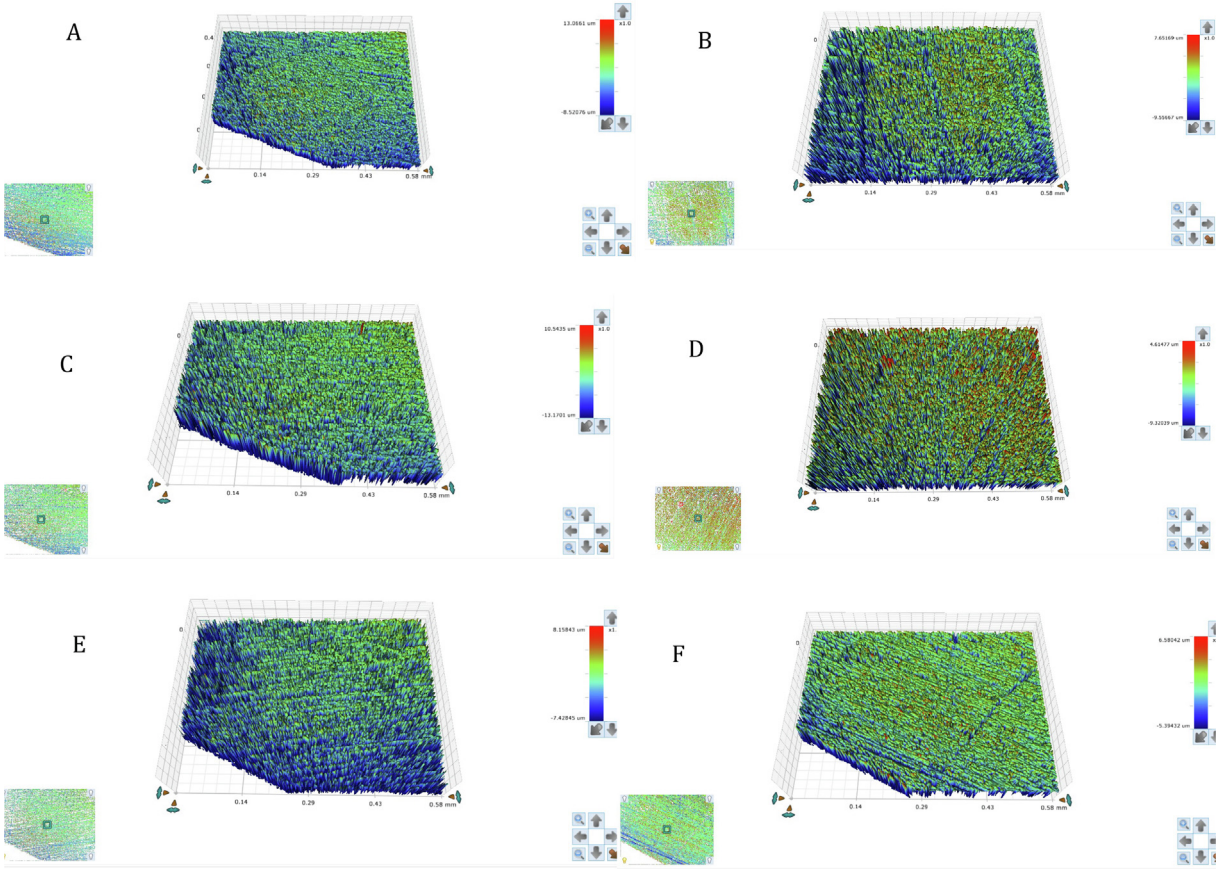

Fig. 1 Images represent surface roughness of the ceramic materials. A: EC before thermocycing. B: EC after thermocycling. C. EP before thermocycing. D: EP after thermocycling. E. LP before thermocycing. F: LP after thermocycling.

testing machine using a Vickers hardness indenter (Wilson Hardness, IL, USA). Each specimen was undergone to three indentations of a 1-kg load with 15 s dwell time almost at the specimen's center, as well as 5 mm away from the center on each side. The microhardness value for each specimen was determined based on the average of the three indentations and reported as Vickers hardness (VH) units.

2.7. Statistical analysis

The analysis was executed using SPSS-20.0 (IBM product, Chicago, Illinois, USA). Numerical data of recordings of surface roughness, color parameters, color changes, and microhardness were exhibited as means and standard deviations (SDs). These numeric variables were assessed for test of normality using the Kolmogorov–Smirnov test for each material that revealed a normal distribution. One-way ANOVA and post-hoc Tukey's test were executed to assess surface roughness, color parameters, color change, and microhardness among the materials. A paired sample *t*-test was used to evaluate mean differences before and after thermocycling. A *p*-value ≤ 0.05 was counted as a statistically substantial difference of means.

3. Results

Comparison of surface roughness values between the ceramics revealed insignificant differences before thermocycling ($F = 0.352$, $p = 0.707$) and after thermocycling ($F = 1.89$, $p = 0.171$). However, the paired differences of means within each material displayed a significant increase in each group after the thermocycling ($p < 0.05$). The highest surface roughness was reported in the EC group (0.592 μm) after thermocycling, while the lowest value of surface roughness was reported in the EP group (0.403 μm) before thermocycling (Table 1).

Color parameters (L*, a*, b*) among the materials were analyzed using one-way ANOVA and post-hoc Tukey's test (Table 2). LP had a significant lower L* in comparison with other groups either before or after thermocycling. However, EP presented significantly lower lightness change (ΔL*) compared to other groups. Paired differences of means within each material using a paired sample *t*-test exhibited a significant increase in L* after thermocycling ($p < 0.001$). There was insignificant difference of a* color variable between materials before thermocycling ($F = 0.213$, $p = 0.809$). However, EC showed a significant decrease in a* after the thermocycling. The results of the b* color variable among materials before thermocycling ($F = 140.9$, $p < 0.001$) and after thermocycling ($F = 202.9$, $p < 0.001$) were found to be highly significant. The paired differences of means of b* within each material before and after thermocycling were also highly significant (Table 2). The LP group had significantly lower b* value compared to other groups either before or after thermocycling. Regarding changes in chroma ΔC^*_{ab}, EP group displayed a significant lower ΔC^*_{ab} as compared with other groups (Table 3).

ANOVA showed substantial differences in color change (ΔE) among the three materials after thermocycling ($F = 13.263$, $p < 0.001$) (Table 3). The EP group exhibited a significant lower ΔE compared with other groups. Regarding the microhardness test (VH), significant differences were reported between the materials ($F = 6.235$, $p = 0.004$). LP group significantly increased in the microhardness in comparison with other groups (Table 3).

4. Discussion

This investigation was designed to test the surface roughness and the color change of three lithium disilicate ceramics before and after being exposed to the thermocycling aging process and also to compare the microhardness values between the groups after the thermocycling. The results indicated that there was insignificant difference among the materials in terms of surface roughness either before or after thermocycling. However, a significant difference was noticed within each material after being subjected to the thermocycling. Regarding the color variables, there were significant differences within and among the materials in terms of the three variables (L*, a*, b*), except a*, which showed insignificant difference among the materials before thermocycling. The results of the color change (ΔE) lightness change (ΔL*), chroma change (ΔC^*_{ab}) and the microhardness exhibited significant difference among the lithium disilicate materials. Thus, the null hypothesis was determined to be partially rejected.

The smooth surfaces of ceramic materials maintain high esthetic appearance, patient satisfaction, and favorable biological and mechanical merits. Rougher surfaces could lead to plaque accumulation, staining of the ceramic restorations, and consequently soft tissue inflammation (Al-Thobity et al., 2021; Mahrous et al., 2021). The outcomes of the current study exhibited that there was insignificant difference in the surface roughness among the three lithium disilicate materials either before or after thermocycling. However, the thermocycling had a significant effect on each tested material compared to the surface roughness values before thermocycling. These findings were in harmony with previous studies that showed the remarkable effects of thermocycling (Vasiliu et al., 2020; Yuan et al., 2018). Yuan et al. (Yuan et al., 2018) reported that CAD/CAM lithium disilicate displayed significantly rougher surfaces with 6000, 12,000, and 18,000 thermocycles. Similarly, Vasiliu et al. (Vasiliu et al., 2020) found that 10,000 thermocy-

Table 1 Comparison of surface roughness values within and between the lithium disilicate materials.

Surface roughness (R_a, μm)	EC (M ± SD)	EP (M ± SD)	LP (M ± SD)	[+]P-value
Before	0.436 ± 0.112	0.403 ± 0.076	0.407 ± 0.094	0.707
After	0.592 ± 0.093	0.531 ± 0.078	0.546 ± 0.035	0.171
Difference	0.156 ± 0.14	0.127 ± 0.104	0.138 ± 0.108	
[++]P-value	0.007	0.004	0.003	

*The significant level is set at $p \leq 0.05$.
[+] One-way ANOVA test for comparison between the materials.
[++] Paired sample *t*-test for comparison between before and after thermocycling results.

Table 2 Color parameters (L*, a*, b*) analysis of the lithium disilicate materials.

Color parameters		EC (M ± SD)	EP (M ± SD)	LP (M ± SD)	[+]P-value
L*	Before	63.38 ± 1.51[b]	66.90 ± 1.32[c]	61.44 ± 3.26[b,c]	< 0.001
	After	72.59 ± 0.75[b]	72.73 ± 1.14[c]	70.38 ± 1.77[b,c]	< 0.001
	Difference	9.22 ± 1.94	5.83 ± 1.72	8.94 ± 2.42	
	[++]P-value	< 0.001	< 0.001	< 0.001	
a*	Before	−2.13 ± 0.10	−2.10 ± 0.08	−2.00 ± 1.01	0.809
	After	−1.20 ± 0.13[a,b]	−1.61 ± 0.05[a]	−1.45 ± 0.39[b]	< 0.001
	Difference	0.93 ± 0.18	0.49 ± 0.07	0.54 ± 0.68	
	[++]P-value	< 0.001	< 0.001	0.008	
b*	Before	3.07 ± 0.74[b]	3.07 ± 0.49[c]	−2.99 ± 1.77[b,c]	< 0.001
	After	9.44 ± 1.04[a,b]	7.01 ± 0.72[a,c]	3.14 ± 0.80[b,c]	< 0.001
	Difference	6.36 ± 1.19	3.94 ± 0.80	6.13 ± 1.49	
	[++]P-value	< 0.001	< 0.001	< 0.001	

^The significant level is set at $p \leq 0.05$.
[a] The mean difference is significant between EC vs EP at $p \leq 0.05$ level.
[b] The mean difference is significant between EC vs LP at $p \leq 0.05$ level.
[c] The mean difference is significant between EP vs LP at $p \leq 0.05$ level.
[+] One-way ANOVA test for comparison between the materials and post-hoc Tukey's test.
[++] Paired sample *t*-test for comparison between before and after thermocycling results.

Table 3 Comparison of color change, microhardness values and NBS.

Factors	EC (M ± SD)	EP (M ± SD)	LP (M ± SD)	[+]P-value
ΔL*	9.22 ± 1.94[a]	5.83 ± 1.72 [a,c]	8.94 ± 2.42[c]	< 0.001
Chroma Change (ΔC^*_{ab})	6.43 ± 1.2[a]	3.97 ± 0.81[a,c]	6.15 ± 1.64[c]	< 0.001
Color Change (ΔE)	16.51 ± 3.3[a]	10.26 ± 2.59[a,c]	15.61 ± 4.59[c]	< 0.001
Microhardness (VH)	2226.23 ± 226.38[b]	2242.83 ± 141.21[c]	2434.65 ± 160.17[b,c]	0.004

[*] The significant level is set at $p \leq 0.05$.
[+] One-way ANOVA test for comparison between the materials and post-hoc Tukey's test.
[a] The mean difference is significant between EC vs EP at $p \leq 0.05$ level.
[b] The mean difference is significant between EC vs LP at $p \leq 0.05$ level.
[c] The mean difference is significant between EP vs LP at $p \leq 0.05$ level.

cles had a substantial impact on the surface roughness of the e.max Press lithium disilicate material.

The color stability of ceramic materials is an essential property to ensure the success of dental restorations in the clinical setting (Gawriołek et al., 2012; Nogueira and Della Bona, 2013). Various reports have assessed the influence of thermocycling on color changes of ceramic materials (Gürdal et al., 2018; Haralur et al., 2019; Palla et al., 2018; Seyidaliyeva et al., 2020; Yuan et al., 2018). Gürdal et al. (2018) noticed that the color change (ΔE) values following 5000 cycles of thermocycling for CAD/CAM lithium disilicate ranged between 3.13 and 8.33. Yuan et al. (Yuan et al., 2018) reported that the mean color change of CAD/CAM lithium disilicate was below 2.6 ΔE. The results obtained from the current study exhibited that the color change for all tested ceramic materials were between 10 and 15 ΔE, where the e.max Press was significantly higher than other groups. The ΔE values of the tested materials were higher than the clinically perceptible value (ΔE = 2–3.5) (Gürdal et al., 2018; O'Brien et al., 1991).The variation in the results between the current and other studies might be referred to difference in the specimen preparation protocol, variations in the thermocycling process and measuring tools.

The outcomes of the current study showed that the least changes in lightness and chroma after thermocycling were noticed in e.max Press which could be referred mainly to the difference in the microstructure and the construction technique between the materials. Bagis and Turgut (2013) reported that the e.max Press had insignificant difference in the chroma value when compared to e.max CAD. Chen et al. (2015) reported that there were significant changes in chroma and lightness values of e.max Press under different substrates. These differences could be a result of the variations in the protocol of specimen's preparation and the measuring tools.

The hardness of lithium disilicate materials has a major influence on the materials' ability to resist occlusal wear (Vasiliu et al., 2020). This study presented that the microhardness mean value of GC LiSi lithium disilicate was substantially higher than that of e.max CAD and e.max Press after being

exposed to 5000 cycles of thermocycling. This variation may have been caused by the differences in processing technique and the composition of the three materials (Zheng et al., 2008).

For future studies, it is recommended that the physical properties of the three materials be assessed at higher levels of thermocycling, as well as by adding cyclic loading protocols to mimic the occlusal forces. Furthermore, impacts of the materials' different thicknesses on the mechanical properties could also be assessed. Although this study encountered variations in intraoral temperatures using thermocycling, it also has some limitations, including using only three ceramic materials and not considering various intraoral factors, such as simulation of the occlusal forces and the acidity of drinks and food.

5. Conclusions

- Regarding surface roughness, there was insignificant difference among the three lithium disilicate materials either before or after thermocycling. However, each material had significantly rougher surface after being subjected to thermocycling.
- GC Initial LiSi Press displayed significantly lower L* and b* values than e.max CAD and e.max Press.
- E.max Press exhibited a significant decrease in color change (ΔE) lightness change (ΔL*), chroma change (ΔC^*_{ab}).
- LiSi Press showed significantly increase microhardness value after being exposed to thermocycling.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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