European Journal of Prosthodontics and Restorative Dentistry (2024) **32**, 377–383

Effect of Repeated Firings on Color Stability of Zirconia-Reinforced Lithium Silicate and Lithium Disilicate Ceramics

Keywords

Lithium Disilicate
Zirconium Oxide
Computer-Aided Design
Color

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Received: 04.10.2024
Accepted: 03.11.2024

doi: 10.1922/EJPRD_2466Habibkhodaei07

ABSTRACT

*Purpose: The purpose of this invitro study was to assess the changes in the color attributes of high-translucency (HT) lithium disilicate and zirconia-reinforced lithium silicate in BL3/OM1, A1, A2, and A3 shades during first, second, and third firing cycles. Materials and Methods: Eighty specimens of two ceramic systems (IPS e.max CAD and Vita Suprinity) in BL3/OM1, A1, A2, and A3 shades, were prepared in thickness of 1 mm (n=10). Each group was subjected to 3 firing cycles and the CIE L*a*b* color parameters of specimens were measured after each firing cycle by a reflectance spectrophotometer. The color change (ΔE) between each two firing cycles was calculated by the relevant equation. Data were analyzed by one-way ANOVA and Tamhane post hoc test. Results: All ΔE values were significant ($P<0.001$) except for ΔE2-1 (color difference between second and first firing) and ΔE3-2 (color difference between third and second firing) in A2 shade of IPS e.max ($P=0.436$) and ΔE2-1 and ΔE3-2 in OM1 shade of Suprinity ($P=0.345$). Conclusion: Repeated firing resulted in generally higher ΔE in ZLS than LDS, and OM1 Suprinity experienced maximum ΔE after repeating firing.*

INTRODUCTION

Dental ceramics have gained increasing popularity considering the longer clinical service of indirect restorations, and increased demands and expectations of patients from cosmetic dental restorations. The primary all-ceramic restorations had shortcomings such as requiring greater tooth preparation and removal of tooth structure, chipping of the veneering porcelain, and inability to mask the underlying color[1-3].

By the advances in dental science and technology, fabrication of dental restorations also improved. Use of digital technology in dentistry increased precision and decreased the chair time. The advent of computer-aided design/computer-aided manufacturing (CAD/CAM) systems enabled treatment planning and restoration delivery all in one session[4,5]. Several dental materials are currently available in the market for the fabrication of CAD/CAM restorations, each having their own unique optical and mechanical properties[6-8].

Favorable optical properties are imperative for a successful restoration. Aside from the type of material used for the fabrication of restoration, its color match with the adjacent teeth is among the most important parameters that needs to be taken into account in fabrication of restoration[9,10].

Several factors affect the color match and optical properties of restorations such as the underlying tooth color[6,7,11,12], ceramic color[6,11], type of material[11,12], ceramic thickness[6,12,13], number of firing cycles[14-18], and color and thickness of luting cement[13,19]. Considering the complex interactions of these parameters, estimation of the final color of restoration would be difficult, and requires high attention to details in the manufacturing process.

The effect of the abovementioned parameters on optical properties of all-ceramic restorations has been previously evaluated. However, studies on the effect of repeated firing on color of lithium disilicate (LDS) and zirconia-reinforced lithium silicate (ZLS) ceramics are limited. Thus, this study aimed to assess the effect of repeating firing on color of high-translucency (HT) LDS and ZLS ceramics. Moreover, since the majority of previous studies on this topic only evaluated one shade (often A2) of ceramic, this study evaluated four shades of A1, A2, A3 and BL3/0M1 of ZLS and LDS to assess the effect of color shade on color stability as well. The first null hypothesis was that repeated firing would have no significant effect on color stability of CAD/CAM ZLS and LDS ceramics. The second null hypothesis was that no significant difference would be found among different shades of each type of ceramic regarding color change after repeated firing.

Color stability of dental ceramics is crucial for the esthetic success of restorations. In clinical practice, repeated firing cycles are often necessary for adjustments, corrections, and glazing, which may influence the optical properties of ceramics. Previous research has provided limited information on how repeated firing affects the color stability of lithium disilicate (LDS) and zirconia-reinforced lithium silicate (ZLS) ceramics, especially across different shades. Therefore, this study aims to evaluate the effects of repeated firing on the color attributes of these ceramics to provide insights that can help clinicians maintain the esthetic integrity of restorations.

MATERIALS AND METHODS

Eighty ceramic specimens were prepared from two CAD/CAM materials: IPS e.max CAD (HT, shades BL3, A1, A2, A3; Ivoclar Vivadent AG, Schaan, Liechtenstein) and Vita Suprinity PC (HT, shades 0M1, A1, A2, A3; Vita Zahnfabrik, Bad Säckingen, Germany). These materials were selected because lithium disilicate (LDS) and zirconia-reinforced lithium silicate (ZLS) ceramics are among the most commonly used for CAD/CAM dental restorations. Understanding how repeated firing cycles affect their color stability is important for the esthetic outcomes of restorations.

The sample size of 10 specimens per group was determined based on previous studies that investigated the effect of surface treatments and firing cycles on the color and optical properties of dental ceramics[12,20-22]. These studies demonstrated that a sample size of 10 is sufficient to observe statistically significant differences.

Each specimen was milled from CAD/CAM blocks measuring 12 × 14 × 18 mm using a precision CNC cutting machine (Model XYZ; Nemofanavaran Pars, Mashhad, Iran) equipped with a diamond disc operating at low speed under water coolant, following the manufacturers' recommendations. To ensure sufficient thickness for polishing, each specimen was sectioned to an initial thickness of 1 ± 0.05 mm plus an additional 0.05 mm, resulting in dimensions of 1 × 14 × 12 mm. After sectioning, one surface of each specimen was manually polished under running water using water-resistant silicon carbide abrasive papers of 600, 1200, and 2000 grit (3M ESPE, St. Paul, MN, USA) to achieve a completely smooth surface. The final thickness of each specimen was verified using a digital caliper (Model Y-100624; Guilin Guanglu Measuring Instrument Co., Guilin, China) to ensure consistency at 1 ± 0.01 mm. Ten specimens were prepared for each shade, resulting in a total of 80 specimens.

The specimens were cleaned in an ultrasonic bath containing distilled water for 15 minutes and then dried with absorbent paper. Each of the eight groups underwent three firing cycles in a calibrated ceramic furnace (Programat P500; Ivoclar Vivadent AG) according to the manufacturers' protocols.

Color measurements were performed after each firing cycle using a calibrated reflectance spectrophotometer (Model X; DeguDent GmbH, Hanau-Wolfgang, Germany) under standardized lighting conditions (CIE standard illuminant D65) and a 45°/0° geometry observer angle. To ensure reproducible positioning during spectrophotometric assessment, a mounting jig was fabricated using silicone putty impression material with a depression at the center for correct placement of specimens. The tip of the spectrophotometer was adjusted over the putty to prevent environmental light interference. The spectrophotometer was calibrated prior to each measurement using the manufacturer's calibration tiles.

The L*, a*, and b* color parameters of each specimen were measured according to the CIE Lab* color space. Measurements were taken at four different points on each specimen, and the mean values were calculated and reported for each color parameter after each firing cycle. To calculate the color change (ΔE) between firing cycles, the following formula was used:

$$\Delta Eab = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{½}$$

Color differences were calculated between the first and second firing cycles (ΔE2–1), the second and third firing cycles (ΔE3–2), and the first and third firing cycles (ΔE3–1). The ΔEab formula was chosen due to its widespread use in dental research and to facilitate direct comparison with existing data, despite the CIEDE2000 formula offering a more perceptually uniform metric[23].

It should be noted that baseline color measurements prior to any firing were not obtained. This is because the ceramic materials used are supplied in a pre-crystallized (blue) state, which does not reflect their final optical properties. According to the manufacturers' instructions, these materials require at least one firing cycle to achieve their intended crystalline structure and translucency. Therefore, the first firing cycle was considered the baseline for all subsequent color measurements.

Data were analyzed using SPSS version 24. Normality of data distribution was assessed using the Shapiro-Wilk test, and homogeneity of variances was evaluated with Levene's test. Since the assumption of homogeneity of variances was violated (Levene's test, $p<0.05$), we used one-way ANOVA with Tamhane's T2 post-hoc test for multiple group comparisons. Paired t-tests were employed for within-group comparisons across different firing cycles due to the repeated measures design. A significance level of 0.05 was set for all statistical tests.

RESULTS

The assumption of homogeneity of variances was not met by the Levene's test. Thus, since the interaction effect of frequency of firing cycles and shade on color stability was significant ($P<0.001$), data were analyzed by one-way ANOVA and Tamhane's post-hoc test.

Comparison of 8 groups regarding ΔE2-1, ΔE3-2, and ΔE3-1 by one-way ANOVA revealed significant differences in all three parameters ($P<0.001$). Table 1 presents pairwise comparisons of the eight groups regarding ΔE2-1, ΔE3-2, and ΔE3-1.

Table 2 shows pairwise comparisons of ΔE2-1, ΔE3-2, and ΔE3-1 separately for each ceramic type and shade using paired t-test. Significant differences were found between all ΔE values in B13 shade of e.max ($P<0.001$), A1 shade of e.max ($P<0.001$), A3 shade of e.max ($P<0.001$), A1 shade of Suprinity ($P<0.001$), A2 shade of Suprinity ($P<0.001$), and A3 shade of Suprinity ($P<0.001$). However, in A2 shade of e.max, and 0M1 shade of Suprinity, the difference between ΔE2-1 and ΔE3-2 was not significant; while other differences were all statistically significant ($P<0.001$).

Table 3 presents pairwise comparisons of different shades of each ceramic regarding ΔE values. As shown, in e.max group, significant differences were noted between all shades in ΔE3-2 ($P<0.001$) except for the difference between A1 and BL3 shades ($P=1.00$). Also, the difference in ΔE3-1 was significant between all shades except for A1 and BL3 shades ($P=0.26$), and A1 and A3 shades ($P=0.27$).

In the Suprinity group, the difference between all shades was significant in ΔE2-1 ($P<0.001$). Also, the difference between all shades was significant in ΔE3-2 except for the difference between A1 and A2 shades ($P=0.792$) and A1 and A3 shades ($P=0.262$). The difference between all color shades was significant in ΔE3-1 ($P<0.001$). No other significant differences were noted ($P>0.05$).

Comparison of L1, L2 and L3 parameters *(Figure 1)* among the 8 groups by repeated measures one-way ANOVA revealed a significant difference in all ceramic shades ($P=0.000$) except for A2 shade of e.max ($P=0.148$). Pairwise comparisons of the values in each ceramic type/shade by t-test showed significant differences in all comparisons ($P=0.00$) except for the difference between L1 and L3 in 0M1 shade of Suprinity which was not significant ($P=0.140$).

Table 1. Pairwise comparisons of the eight groups regarding ΔE2-1, ΔE3-2, and ΔE3-1.

Ceramic type	Ceramic shade	ΔE2-1	ΔE3-2	ΔE3-1
E. max	BL3	.38 ± .13	.82 ± .11	1.10 ± .12
	A1	.62 ± .19	.80 ± .07	1.30 ± .20
	A2	.47 ± .16	.53 ± .14	.76 ± .08
	A3	.51 ± .14	1.23 ± .12	1.56 ± .10
Suprinity	0M1	8.89 ± .21	8.84 ± .12	.30 ± .11
	A1	5.36 ± .31	.49 ± .14	5.01 ± .24
	A2	2.23 ± .09	.36 ± .14	2.10 ± .13
	A3	4.71 ± .24	.71 ± .19	4.16 ± .15

Table 2. Pairwise comparisons of E2-1, ΔE3-2, and ΔE3-1 separately for each ceramic type and shade using paired t-test.

Material/ Comparison	E.max Bl3	E.max A1	E.max A2	E.max A3	Suprinity 0M1	Suprinity A1	Suprinity A2	Suprinity A3
ΔE21 vs. ΔE 32	.000	.022	.436	.000	.345	.000	.000	.000
ΔE21 vs. ΔE31	.000	.000	.001	.000	.000	.000	.002	.000
ΔE32 vs. ΔE31	.000	.000	.001	.000	.000	.000	.000	.000

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Table 3. Pairwise comparisons of different shades of each ceramic regarding ΔE values.

Ceramic type	Color shade	ΔE21	ΔE32	ΔE31
E.max	BL3 – A1	.11	1.00	.26
	BL3 – A2	.99	.003	.000
	BL3 – A3	.76	.000	.000
	A1 – A2	.86	.003	.000
	A1 – A3	.98	.000	.27
	A2 – A3	1.00	.000	.000
Suprinity	BL3 – A1	.000	.000	.000
	BL3 – A2	.000	.000	.000
	BL3 – A3	.000	.000	.000
	A1 – A2	.000	.792	.000
	A1 – A3	.003	.262	.000
	A2 – A3	.000	.007	.000

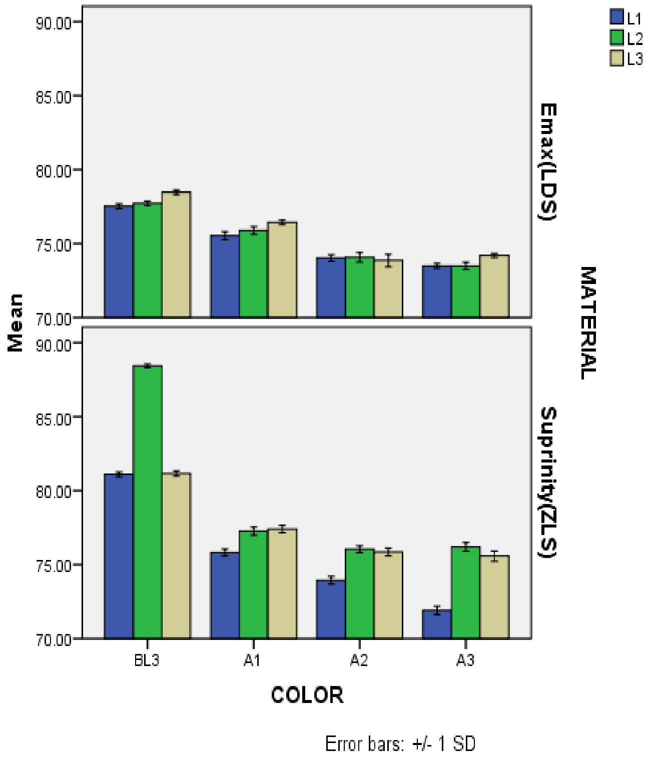

Figure 1: Mean L1, L2 and L3 of different ceramic shades

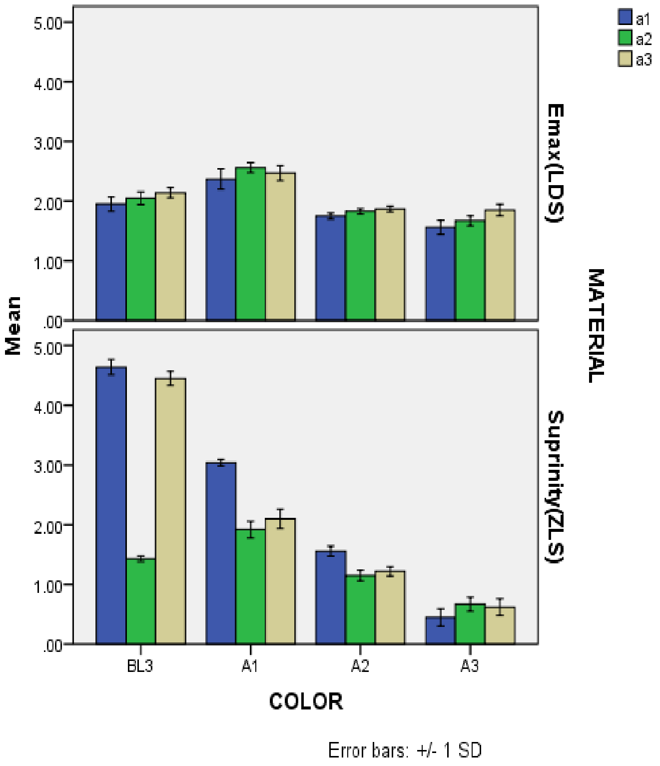

Figure 2: Mean a1, a2 and a3 of different ceramic shades.

Comparison of a1, a2 and a3 parameters (*Figure 2*) among the 8 groups by repeated measures one-way ANOVA revealed a significant difference in all ceramic shades (P=0.00). Pairwise comparisons of the values in each ceramic type/shade by t-test showed significant differences in all comparisons (P<0.001) except for the difference between a1 and a2 in A3 shade of e.max (P=0.06), a1 and a3 in A1 shade of e.max (P=0.128), and a2 and a3 in A3 shade of Suprinity (P=0.177).

Comparison of b1, b2 and b3 parameters (*Figure 3*) among the 8 groups by repeated measures one-way ANOVA revealed a significant difference in all ceramic shades (P=0.00). Pairwise comparisons of the values in each ceramic type/shade by t-test showed significant differences in all comparisons (P=0.00).

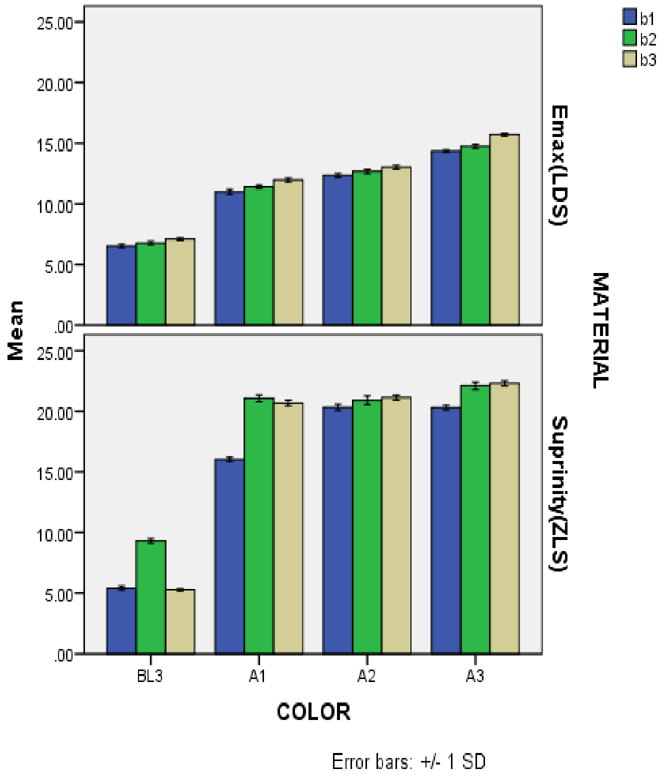

Figure 3: Mean b1, b2 and b3 of different ceramic shades.

DISCUSSION

This study assessed the effect of repeated firing on color stability of HT LDS and ZLS ceramics. The first null hypothesis was that repeated firing would have no significant effect on color stability of CAD/CAM ZLS and LDS ceramics. The second null hypothesis was that no significant difference would be found among different shades of each type of ceramic regarding color change after repeated firing. The first null hypothesis was rejected since the difference in ΔE values was significant in all color shades of e.max LDS ceramic except for A2 shade in comparison of ΔE2-1 and ΔE3-2 (P=0.436). For the Suprinity ZLS ceramic, the difference in ΔE values was significant in all color shades except in 0M1shade in comparison of ΔE2-1 and ΔE3-2 (P=0.345). The second null hypothesis was also rejected considering the presence of significant differences between different shades of both ceramic types.

Repeated firing cycles can influence the microstructure and optical properties of dental ceramics. Literature indicates that multiple firings may lead to changes in crystalline phases, oxidation of coloring agents, and alterations in the glass matrix, all of which can contribute to color shifts[24]. Clinically, such color changes can be perceptible to patients and may compromise the esthetic outcome of restorations, leading to dissatisfaction and the need for remakes.

By investigating how repeated firing affects the color stability of LDS and ZLS ceramics, this study provides insights that are directly applicable to clinical practice. Understanding these effects allows clinicians to minimize undesirable color changes by limiting the number of firings or selecting materials less susceptible to color shifts.

ΔE is often calculated to find out whether the color change is perceivable by the human eye or not. Evidence shows that in cases with ΔE > 1, the color difference between the two specimens would be detectable by 50% of the observers. In cases with ΔE > 2, the color difference wound be detectable by 100% of the observers. ΔE between 1 and 2 is often perceptible, but ΔE<3.7 is generally considered to be clinically acceptable[25].

In the present study, all ΔE2-1, ΔE3-2 and ΔE3-1 values were < 3.7 in e.max, and therefore clinically acceptable. However, ΔE3-1 in BL3, A1 and A3 shades, and ΔE3-2 in A3 shade were all > 1, indicating that the color change would be clinically detectable by 50% of the observers. These findings indicate that increasing the firing cycles for e.max ceramic increases the possibility of detection of color change, although this change is within the clinically acceptable range.

In Suprinity specimens, ΔE2-1 in 0M1, A1, and A3 shades, ΔE3-2 in 0M1, and ΔE3-1 in A1 and A3 shades were higher than 3.7, and were therefore, clinically unacceptable, and detectable by all observers. Moreover, ΔE2-1 and ΔE3-2 in 0M1 shade of Suprinity ceramic were the maximum among all (approximately 8.8) and detectable by all observers. In this study, the numerical values of ΔE in the majority of Suprinity specimens were higher than the corresponding values in e.max, indicating greater effect of repeated firing on Suprinity ceramic, compared with e.max. According to Pires-de *et al*,[26] color change of ceramics may be due to the presence of metal oxides that are added to ceramics to obtain the desired color shades. The bond of these metal oxides can be easily broken at high temperatures or under ultraviolet radiation, and lead to release of peroxide compounds, which can cause porcelain discoloration. Thus, lower glass content and presence of higher amounts of fillers such as metal oxides in the structure of ZLS can explain poor color stability of these restorations in repeated firing cycles[27], which calls for further attention in fabrication of such restorations or adjusting their shape and contour. For instance, in e.max ceramic specimens, the change in L parameter was significant following repeated firing cycles, except for A2 shade. The L* parameter significantly increased due to repeated firing cycles in most shades. The increase in a* parameter was also significant in most shades due to repeated firing. The increase in b* parameter was significant in all specimens as well. In Suprinity specimens, the change in L* parameter was also significant in most shades. It increased after second firing and decreased after the third firing. The change in a* parameter was significant in all shades except one (a2 and a3 in A3 shade) such that it significantly decreased after second firing and then increased again after the third firing. However, the final a* value after third firing was significantly lower than that after the first firing (except in A3 shade). In all specimens, the b* parameter significantly increased after each firing cycle.

To the best of the authors' knowledge, studies on the effects of repeated firing on color stability of e.max and Suprinity ceramics are limited, and none of the available studies compared different color shades of ceramics in this respect. Cui *et al.*[28] assessed the effect of repeated firing and thickness of ceramic on color stability of A2 shade of LDS ceramic. They found that repeated firing decreased the L* parameter and caused further compression and interlocking of LDS crystalline microstructure, and led to subsequent color change. In the present study, the change in L* parameter due to repeated firing cycles was not significant in the A2 shade of LDS ceramic but it increased in other color shades of this ceramic. De Morais *et al.*[29] evaluated the effect of repeated firing on optical and mechanical properties of LDS and ZLS ceramics. The specimens underwent 2, 5, and 7 firing cycles. ZLS ceramic showed ΔE > 1 after 5 and 7 firing cycles compared with the control group (2 firing cycles). LDS ceramic showed ΔE < 1 after 5 and 7 firing cycles compared with the control group. The ΔE of LDS ceramic was significantly lower than that of ZLS ceramic, irrespective of the frequency of firing cycles. Also, after 7 firing cycles, a significant increase in b* and L* parameters was noted. They showed higher color stability of LDS compared with ZLS ceramic. Also, maximum ΔE was recorded after 7 firing cycles in ZLS ceramic specimens (ΔE=1.34), which was still within the clinically acceptable range. De Morais *et al.*[29] did not mention the color shade of ceramic specimens used in their study but since A2 shade is most commonly used, it might have been A2. In the present study, A2 shade of ZLS ceramic showed ΔE > 1 after 2 and 3 firing cycles (compared with the control group with 1 firing cycle), and A2 shade of LDS ceramic showed ΔE < 1 after 2 and 3 firing cycles (compared with the control group with 1 firing cycle). Furthermore, ZLS specimens showed a higher ΔE compared with the corresponding shades of LDS, and maximum ΔE was noted in ZLS ceramic, which was in line with the results of de Morais *et al*[29]. Miranda *et al.*[30] assessed the effect of staining and repeated firing cycles on surface properties and optical characteristics of CAD/CAM LDS ceramic and found that increasing the firing cycles caused significant color change of specimens. The ΔE values observed in our study indicate that repeated firing cycles can lead to color changes that are clinically perceptible. Specifically, in the ZLS ceramic group, certain shades exhibited ΔE values exceeding the acceptability threshold of 3.7, suggesting that these changes could be noticeable to patients and affect the esthetic outcome of restorations. Clinicians should be aware of these potential changes when performing multiple firing cycles and consider minimizing additional firings to maintain the desired color match.

Many studies have assessed the effect of repeated firing cycles on LDS ceramic fabricated by the Press technique. Dong-Dong *et al.*[31] showed that repeated firing caused color change of ceramic, although it was within the clinically acceptable range. Fehmi *et al.*[32] assessed the effect of repeated firing on A2 shade of LDS Press and zirconia. They showed that multiple firing cycles caused a significant increase in L* and a significant reduction in a* parameter in LDS ceramic, but had no significant effect on b* parameter. Such changes were noted after 3 and 5 firing cycles. Except for ΔE after 1 and 5 firing cycles, which was within the clinically acceptable threshold (ΔE=1.8), it was higher in other specimens. In the present study, ΔE2-1 and ΔE3-1 in A2 shade of e.max were above the clinically acceptable threshold reported by Fehmi *et al*,[32] but ΔE3-2 was acceptable. In A2 shade of Suprinity, an increase in L* and a reduction in a* were noted after the third firing cycle, compared with the control group.

The present results showed the significant effect of repeated firing on ΔE in both ceramic types. Generally, the numerical value of color change was higher in ZLS compared with LDS ceramic, explained by its lower glass filler and higher metal oxide content. The maximum ΔE noted in 0M1 shade of Suprinity ceramic calls for further attention by the clinicians.

Evaluation of the effect of repeated firing on different color shades of two types of commonly used ceramics was a strength of this study, which had not been addressed before. Not assessing the effect of ceramic thickness, evaluation of only two ceramic types, and rectangular shape of specimens were among the limitations of this study, which should be addressed in future studies.

A limitation of this study is the absence of baseline color measurements before any firing cycles. The pre-crystallized state of both lithium disilicate (LDS) and zirconia-reinforced lithium silicate (ZLS) ceramics presents a significantly different appearance compared to their final fired state, rendering initial color measurements irrelevant for clinical comparisons. Consequently, we were unable to assess the color changes that occur during the very first firing cycle. This limitation is inherent to the nature of the materials, as their optical properties are only fully developed after the initial crystallization firing. Future studies might explore alternative materials that allow for pre-firing color measurements or investigate methods to estimate the initial color properties before firing. The study is limited to two types of ceramics, future studies could expand on this research by including additional ceramic materials to provide a more comprehensive understanding of how repeated firing affects various ceramic systems. We acknowledge that using the CIEDE2000 formula could offer a more accurate representation of perceived color differences. Future studies may consider employing both formulas to provide a comprehensive analysis.

CONCLUSION

Within the limitations of this study—including the absence of pre-firing baseline color measurements due to material properties—we found that repeated firing cycles significantly affect the color stability of both LDS and ZLS ceramics. The effect was more pronounced in ZLS ceramics, particularly in the 0M1 shade, where color changes exceeded clinically acceptable thresholds. Clinicians should consider the potential for color alteration when multiple firing cycles are necessary and aim to limit firings to preserve the esthetic integrity of ceramic restorations.

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