Effect of Repeated Firing on the Translucency of CAD/CAM Lithium Disilicate Ceramics

Nutthaporn Lerttanpan[1,*], Natthapong Itthipongsatorn[1], Puliwan Gorwong[2], and Pasiree Thongthai[3]

[1]Department of Esthetic Restorative and Implant Dentistry, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand
[2]Dental Materials Research and Development Center, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand
[3]Department of Operative Dentistry, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand
*Corresponding author, E-mail: Nutthaporn.peanut@gmail.com

Abstract

The advancement of computer-aided design and computer-aided manufacturing (CAD/CAM) systems has enhanced the fabrication of chairside dental restorations using lithium disilicate ceramics. However, repeated firing cycles for shade correction and surface characterization may alter their optical properties, particularly translucency. This in vitro study evaluated the effect of repeated firings on the translucency of two CAD/CAM lithium disilicate ceramics, IPS e.max CAD (EM) and Amber Mill (AM). A total of 20 rectangular specimens (12×14×1 mm) were fabricated from high-translucency, A2-shade ceramic blocks (10 specimens per material). All specimens underwent normal firing, including crystallization and glaze firing, followed by 1, 3, and 5 repeated glaze firings. Translucency was measured using a spectrophotometer, and the translucency parameter (TP_{00}) was calculated from color differences against black and white backgrounds. Two-way repeated measures ANOVA and Bonferroni post-hoc tests (α=.05) were used to analyze the effects of material type and firing cycles. Results indicated a significant reduction in translucency with repeated firings for both ceramics (p<.05), with AM consistently exhibiting higher TP_{00} values than EM across all firing cycles, demonstrating a significant interaction between material type and firing cycles. The study concluded that repeated firings reduced the translucency of both CAD/CAM lithium disilicate ceramics.

Keywords: *CAD/CAM ceramic, lithium disilicate, repeated firing, translucency*

1. Introduction

The advancement of computer-aided design and computer-aided manufacturing (CAD/CAM) ceramic systems has greatly enhanced restorative dentistry. Compared to traditional ceramic fabrication methods, CAD/CAM technology provides several benefits, including consistent manufacturing quality, reduced production time, minimized human error, and greater cost efficiency (Li et al., 2014; Miyazaki et al., 2009). Among the various CAD/CAM materials available, lithium disilicate ceramics (LDCs) have become widely used in modern restorative procedures due to their excellent aesthetic properties, favorable mechanical strength, chemical stability, and broad clinical applications (Phark, & Duarte Jr, 2022; Spitznagel et al., 2018).

CAD/CAM LDCs exhibit variations in chemical composition, microstructure, crystallization stage, and fabrication processes (Lubauer et al., 2022; Phark, & Duarte Jr, 2022). IPS e.max CAD (Ivoclar Vivadent) is widely used in clinical dentistry and has demonstrated long-term success in dental restorations (Souza et al., 2021). While Amber Mill (HASSBio) offers a unique feature of customizable translucency, which can be adjusted by modifying the crystallization temperature, as specified in the manufacturer's instructions. Both materials are supplied in a partially crystallized state and require a laboratory-controlled crystallization process. During this phase, their initial coloration—blue for IPS e.max CAD and amber-like for Amber Mill—undergoes a crystallization, resulting in a final shade that closely resembles natural tooth.

As CAD/CAM LDC blocks are monochromatic, they may not entirely mimic the complex characteristics of natural teeth. To enhance their esthetic properties, dental professionals commonly use surface characterization techniques, including the application of stain and glaze (Saint-Jean, 2014). Multiple firing cycles are often required to achieve the desired shade, translucency, and surface texture (Campanelli de Morais et al., 2021; Miranda et al., 2020). Maintaining translucency throughout these firings is essential for the esthetic success of ceramic restorations (Ozdogan, & Tosun, 2024).

The optical properties, particularly translucency, of restorative materials are crucial for achieving a favorable esthetic outcome and must be carefully considered (Della Bona et al., 2014). Translucency is defined as a condition between transparency and opacity (Kim et al., 2009). It is a material property that occurs when light passes through, being reflected, scattered, and transmitted within the material (Della Bona et al., 2014; Kim et al., 2009). A higher degree of light transmission indicates greater translucency (Awad et al., 2015; Nogueira, & Della Bona, 2013). The translucency of ceramic materials can be measured using the translucency parameter (TP_{00}) (Reid et al., 2023; Vichi et al., 2023). This parameter is determined by assessing the color difference of the material against black and white backgrounds (Reid et al., 2023; Vichi et al., 2023).

Previous studies have reported that the TP_{00} values of lithium disilicate ceramics are influenced by factors such as material type, thickness, and translucency (Czigola et al., 2019; Johnston, 2014). Dental ceramics such as zirconia-reinforced lithium silicate and conventional lithium disilicate also have been studied, highlighting the influence of shade and thickness on TP_{00} values (Pop-Ciutrila et al., 2021). Regarding the impact of repeated firings, increased translucency has been observed in CAD/CAM lithium disilicate ceramics (Nejatidanesh et al., 2020; Zaghloul et al., 2022). Conversely, other studies have shown a reduction in translucency following multiple firings of lithium silicate-based ceramics (Lubauer et al., 2022; Ozdogan, & Ozdemir, 2021). Given these conflicting findings, this study aimed to answer the following research question: How do repeated firings affect the translucency of CAD/CAM lithium disilicate ceramics? The null hypotheses were that: 1) repeated firings would not alter the translucency of CAD/CAM LDCs, and 2) there is no significant difference in translucency between the two CAD/CAM lithium disilicate ceramics after repeated firings.

2. Objectives

To evaluate the effect of repeated firings on the translucency of CAD/CAM LDCs.

3. Materials and Methods

This in vitro study evaluated the effects of repeated firings on the translucency of CAD/CAM lithium disilicate ceramics. Standardized specimens were fabricated from two lithium disilicate ceramic materials: IPS e.max CAD (Ivoclar, Schaan, Liechtenstein; EM) and Amber Mill (HASSBio, Kangneung, Korea; AM), both obtained in high translucency (HT) and A2 shade (Table 1).

Since no previous studies have evaluated the effects of repeated firings on these specific materials under similar conditions, a pilot study was conducted. Sample size calculation was performed using G*Power software (version 3.1) based on the pilot study results (n = 3 specimens per group) to achieve 80% power (α=.05) with a calculated effect size of 0.77. The required sample size per group was initially determined as three specimens, but this was increased to 10 specimens per group to account for potential laboratory processing errors and specimen loss.

A total of 20 rectangular specimens ($14\times12\times1$ mm^3) were sectioned from $14\times12\times15$ mm^3 CAD/CAM ceramic blocks using a low-speed precision saw (Isomet 1000 precision saw, Buehler) into 1.0 mm thick plates (Figure 1 and 2), with 10 pieces per test group. The surfaces of the ceramic specimens were smoothed and standardized using 400-, 600-, 1200-, and 2000-grit abrasive papers under water cooling (Aurélio et al., 2017). The thickness control of the specimens was assessed at the center of each specimen with a digital caliper (Digital Micrometer, Mitutoyo).

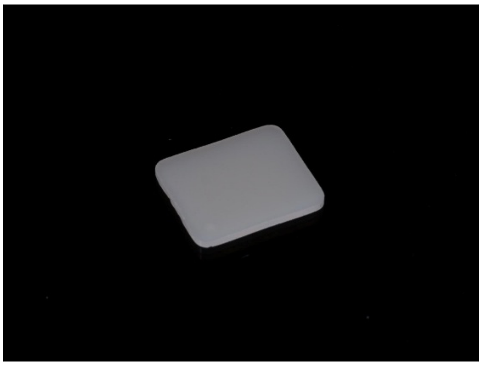


Figure 1 Specimen sectioned from IPS e.max CAD block

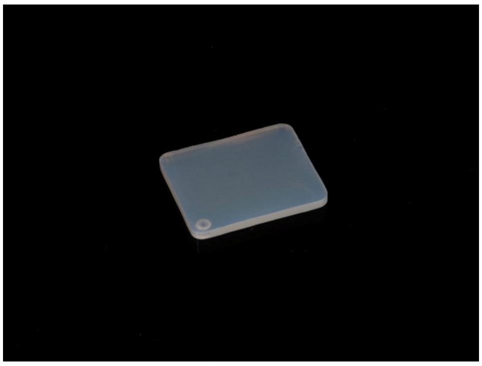


Figure 2 Specimen sectioned from Amber Mill block

Table 1 The composition of materials used in this study

Material	Manufacture	Code	Chemical composition	Lot number
IPS. e.max CAD	Ivoclar, Schann, Liechtenstein	EM	SiO_2: 57–80%, Li_2O: 11–19%, K_2O: 0–13%, P_2O_5: 0–11%, ZrO_2: 0–8%, ZnO: 0–8%, Coloring oxides: 0–12%	YB54GB
Amber Mill	HASSBio, Kangneung, Korea	AM	SiO_2: <78%, Li_2O: <12%, Coloring oxides: <12%	EBE06OG1201

All specimens underwent normal firing (NF), consisting of crystallization and glaze firing, using a ceramic furnace (Programat P700, Ivoclar). According to the manufacturer's instructions, crystallization for EM was conducted at 850°C, with a standby temperature of 403°C, a 6-minute closing time, and a heating rate of 60°C/min. The vacuum was activated at 770°C and deactivated at 850°C. For AM, crystallization reached 815°C, starting from a standby temperature of 400°C, with a 3-minute closing time and the same heating rate. The vacuum was applied at 550°C and deactivated at 815°C. Glaze firing for both materials followed the same protocol: starting at 403°C with a 6-minute closing time, heating at 60°C/min to 710°C, with vacuum activation at 450°C and deactivation at 709°C. A holding time of 1 minute was applied at the final temperature. After completing normal firing process, all specimens underwent up to five repeated firing cycles at glaze firing temperature. After each cycle, the specimens were slowly cooled to room temperature (25°C) for 10 minutes between each firing cycle to simulate a chairside laboratory workflow.

The color of each specimen was assessed after different numbers of firings: normal firing (NF), one repeated firing (1RF), three repeated firings (3RF), and five repeated firings (5RF). Color measurements were performed using a spectrophotometer (Ultrascan Pro, Hunter Lab), which was calibrated before each measurement following the manufacturer's standard procedure. Data were collected using a 7 mm reflection port, with specimens centrally aligned using a tape guide to ensure consistent positioning. Each specimen was measured three times on both black and white backgrounds, and the average value was used for analysis. The translucency parameter (TP00) was calculated based on the color difference between the specimen on black (B) and white (W) backgrounds.

The following formula was used to compute TP_{00} (Reid et al., 2023; Vichi et al., 2023):

$$TP_{00} = \left[\left(\frac{L'_B - L'_W}{k_L S_L}\right)^2 + \left(\frac{C'_B - C'_W}{k_C S_C}\right)^2 + \left(\frac{H'_B - H'_W}{K_H S_H}\right)^2 + R_T\left(\frac{C'_B - C'_W}{k_C S_C}\right)\left(\frac{H'_B - H'_W}{k_H S_H}\right)\right]^{1/2}$$

In this formula, L, C, and H represent lightness, chroma, and hue, respectively. RT is a rotation factor that accounts for the interaction between chroma and hue in the blue region. SL, SC, and SH adjust the color difference based on variations in the L*ab** color system, while KL, KC, and KH represent experimental conditions (for this study, KL=KC=KH=1).

Extra specimens from AM and EM were prepared for scanning electron microscopy (SEM) analysis following normal firing (NF) and five repeated firings (5RF). The specimens were etched with 5% hydrofluoric acid (HF) as per the manufacturer's instructions, then cleaned and coated with gold. SEM imaging was performed using a JSM-IT700HR (JEOL) at 10,000× magnification.

The distribution of variables was evaluated using the Shapiro-Wilk test. A two-way repeated measures ANOVA was performed to assess the effects of material type, number of firings, and their interaction on the mean translucency parameter (TP_{00}). For significant results, Bonferroni post-hoc analyses were performed ($\alpha = 0.05$). All statistical analyses were performed using IBM SPSS Statistics v29.0 (SPSS Inc.).

4. Results and Discussion

4.1 Results

Table 2 Two-way repeated-measures ANOVA for translucency parameter (TP_{00}) after repeated firings

Source	Sum of Squares	df	Mean square	F	Sig
Firing times	29.840	3	9.947	59.025	<.001
Material	3179.826	1	3179.826	1781.894	<.001
Firing times * Material	2.652	3	.884	5.246	.003

The Two-way repeated measures ANOVA indicated a significant effect of firing times and material on the translucency parameter ($p<.05$). Furthermore, a significant interaction was observed between firing times and material in relation to the translucency parameter ($p<.05$), see Table 2.

Table 3 Mean and SD of TP_{00} values

Material	NF	1RF	3RF	5RF
IPS. e.max CAD (EM)	15.07 ±.49^{Aa}	14.26 ±.36^{Ba}	14.19 ±.32^{Ba}	13.65 ±.46^{Ca}
Amber Mill (AM)	28.10 ±.93^{Ab}	26.94 ±.92^{Bb}	26.21 ±1.09^{Cb}	26.35 ±.99^{Cb}

Uppercase letters indicate the differences in TP_{00} values across the firing times for each material and lowercase letters refer to the differences in TP_{00} values between the lithium disilicate ceramic materials within each firing time, analyses by two-way repeated measures ANOVA followed by Bonferroni post-hoc analysis (p<0.05).

Table 3 shows the mean ±SD of TP_{00} values. When considering the material, EM exhibited significantly lower TP_{00} values across all firing times compared to AM. The translucency parameter measurements indicated a consistent reduction in TP_{00} values after repeated firings for both materials. For EM, TP_{00} decreased from 15.07 (NF) to 13.65 (RF5), while for AM, it decreased from 28.10 (NF) to 26.35 (RF5). Significant differences were found between the firing times for each material (p<.05).

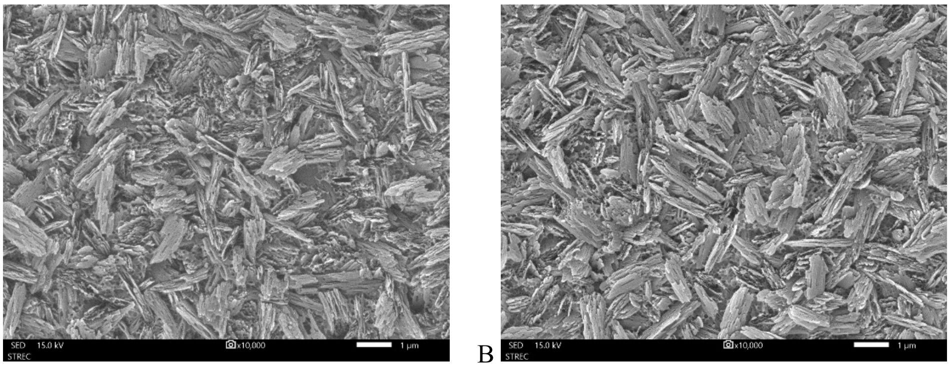
A  B

Figure 3 Scanning electron microscope images of specimens from IPS e.max CAD with magnification ×10000 (A: after normal firing, B: after 5 repeated firings)

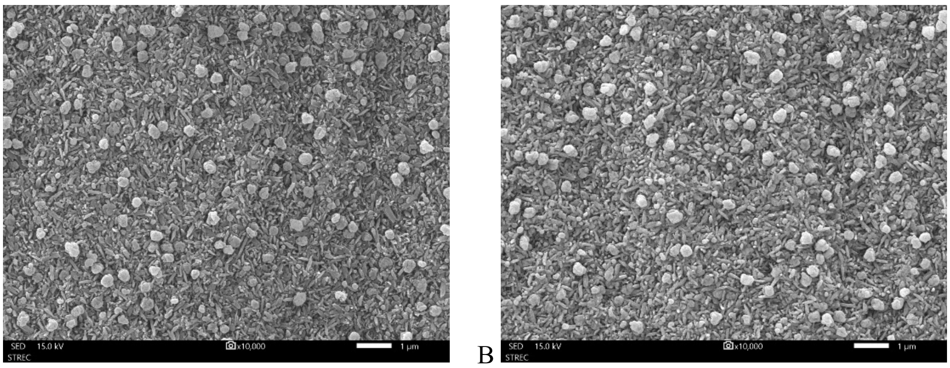
A  B

Figure 4 Scanning electron microscope images of specimens from Amber Mill with magnification ×10000 (A: after normal firing, B: after 5 repeated firings)

SEM analysis identified microstructural variations among two materials are shown in Figures 3 and 4. EM displayed densely packed lithium disilicate crystals measuring 1–1.5 µm, forming a layered rod-like structure. In contrast, AM exhibited smaller rod-shaped lithium disilicate crystals (0.3 µm) embedded within a loosely arranged matrix, along with an unetched glassy phase. SEM analysis showed no significant changes in crystal size for either material after normal firing (NF) and five repeated firings (RF5).

4.2 Discussion

This in vitro study examined the impact of repeated firings on the translucency of two CAD/CAM lithium disilicate ceramics. Firing cycles at 1, 3, and 5 firings were employed to assess the changes in translucency across multiple firings. It represents a possible scenario of surface characterization of the restorations to mimic natural teeth, especially in the esthetic area (Saint-Jean, 2014). Analyzing these trends offers critical insights into the behavior of ceramics, facilitating more informed decision-making. Glaze firing temperatures were selected to replicate clinical conditions in which dentists apply stains and glazing to refine the aesthetic characteristics of restorations (Miranda et al., 2020).

The results led to the rejection of the null hypotheses. The first null hypothesis, stating that repeated firings would not affect the translucency of CAD/CAM LDCs, was rejected, as translucency decreased with each firing cycle. Additionally, the second null hypothesis, which assumed similar translucency parameter for both materials, was also rejected, as EM and AM exhibited different TP_{00} values across all firing cycles.

Translucency can be assessed through various methods, with the translucency parameter (TP) and contrast ratio (CR) being commonly used (Johnston, 2014; Reid et al., 2023; Skyllouriotis et al., 2017; Vichi et al., 2023). TP quantifies the color difference between specimens over white and black backgrounds (Czigola et al., 2019; Johnston, 2014; Skyllouriotis et al., 2017), while CR measures opacity by comparing the reflectance of a specimen placed over black and white backgrounds (Skyllouriotis et al., 2017). A previous study (Nogueira, & Della Bona, 2013) observed a correlation between TP and CR, but no consensus exists regarding the optimal method for quantifying translucency. TP is frequently utilized in dental research and is calculated using the CIELAB color difference formula (Czigola et al., 2019; Johnston, 2014; Vichi et al., 2023). To improve the correlation with visual perception, the CIE developed the CIEDE2000 system, which has been shown to better represent color differences compared to the CIELAB (Gómez-Polo et al., 2016). The CIEDE2000 system has also been applied in calculating the translucency parameter (TP_{00}) (Reid et al., 2023; Vichi et al., 2023).

In the present study, both EM and AM specimens were high-translucency blocks; however, significant differences in translucency were observed across all firing cycles, including after normal firing. The translucency of lithium silicate ceramics primarily depends on their microstructure, especially the size and distribution of lithium disilicate ($Li_2Si_2O_5$) crystals (Lubauer et al., 2022). Ceramics containing larger crystals tend to reduce translucency due to increased light scattering (Zaghloul et al., 2022). AM consistently exhibited higher translucency than EM, even after multiple firing cycles, likely due to its lower lithium disilicate crystalline content and smaller crystal size compared to EM (Lubauer et al., 2022). Additionally, the higher proportion of amorphous glassy phase in AM could further enhance its optical transparency compared to EM, aligning with previous research indicating that increased crystalline content enhances mechanical properties but reduces translucency (Willard, & Chu, 2018). However, the findings from this study contrast with some previous studies, which reported increased translucency following repeated firings. This discrepancy might result from methodological differences, particularly regarding firing protocols (Nejatidanesh et al., 2020; Zaghloul et al., 2022).

The progressive reduction in translucency observed in both materials after repeated firings may be due to structural degradation and increased porosity (Ozdogan, & Ozdemir, 2021). Moreover, multiple heat treatments can promote further crystallization and alter the residual glass composition, potentially contributing to decreased translucency (Lubauer et al., 2022). Although previous SEM analyses (Ozdogan, & Ozdemir, 2021) reported visible structural modifications after multiple firings, no obvious crystal size alterations were observed in SEM evaluation of this study. Thus, advanced microstructural characterization techniques such as X-ray diffraction (XRD) are recommended in future studies to identify subtle changes potentially influencing translucency.

Since repeated firings at glaze temperature resulted in a reduction in translucency, it could impact shade selection and the final esthetic outcome of restorations. Clinicians should minimize unnecessary firing cycles to preserve the esthetic properties of restorations and consider how different materials respond to repeated firings, as excessive firings could lead to increased opacity, which may compromise the final esthetic outcome. Additionally, the significant differences in translucency between EM and AM suggest that material selection plays a crucial role in achieving the desired optical properties. These findings emphasize the importance of understanding the behavior of different lithium disilicate ceramics under clinical processing conditions to optimize restorative outcomes. AM, with its consistently higher translucency, may be more suitable for cases requiring enhanced light transmission, such as thin veneers or restorations in highly esthetic zones. Conversely, EM, which exhibited lower translucency, might be preferable in situations where more masking of underlying tooth structure or restorative materials is needed.

The limitations of this study included the use of plate-shaped specimens rather than anatomically accurate forms, which may not adequately represent clinical restorations. Furthermore, the standardized 1-mm thickness may not fully capture the variability of various restoration thicknesses in clinical practice. Future research should explore the impact of repeated firings on the microstructural level while incorporating different material types and thicknesses.

5. Conclusion

Repeated firings significantly affected the translucency parameter (TP_{00}) of both EM and AM ceramics. Although both materials were classified as high translucency blocks, they exhibited distinct TP_{00} values, with AM consistently maintaining higher translucency than EM. These findings highlight the influence of material composition and firing times on the translucency of CAD/CAM ceramics.

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