Optical properties, biaxial flexural strength, and reliability of new-generation lithium disilicate glass-ceramics after thermal cycling

Running title: PROPERTIES OF LITHIUM DISILICATE CERAMICS AFTER THERMAL CYCLING

Authors

Almira Ada Diken Türksayar, DDS,[1] Münir Demirel DDS, PhD,[2] Mustafa Borga Donmez, DDS, PhD[3,4]

[1]Assistant Professor, Department of Prosthodontics, Faculty of Dentistry, Biruni University, İstanbul, Turkey

[2]Lecturer, Oral and Dental Health, Vocational School, Biruni University, İstanbul, Turkey

[3]Assistant Professor, Department of Prosthodontics, Faculty of Dentistry, Istinye University, İstanbul, Turkey

[4]Visiting Researcher, Department of Reconstructive Dentistry and Gerodontology, School of Dental Medicine, University of Bern, Bern, Switzerland

Corresponding author:

Dr. Mustafa Borga Dönmez, Department of Reconstructive Dentistry and Gerodontology, School of Dental Medicine, University of Bern, Freiburgstrasse 7, 3010, Bern, Switzerland

Email: mustafa-borga.doenmez@unibe.ch

Conflict of Interest

The authors declare no conflict of interest. The authors do not have any financial interest in the companies whose materials are included in this article.

Received September 16, 2022

Accepted December 16, 2022

ABSTRACT

Purpose: To investigate the color stability, translucency, biaxial flexural strength (BFS), and reliability of nano-lithium disilicate and a fully crystallized lithium disilicate after thermal cycling and to compare with those of a commonly-used lithium disilicate.

Material and Methods: Three lithium disilicate glass-ceramics were used to prepare disk-shaped specimens (ø:12 mm, thickness: 1.2 mm) from 3 A2 shaded HT lithium disilicate glass-ceramics (Amber Mill, AM; Initial LiSi Block, IN; IPS e.max CAD, EX). AM and EX specimens were crystallized and all specimens were polished with a polishing paste (Diamond Polish Mint). A spectrophotometer (CM-26d) was used to measure color coordinates before and after thermal cycling. BFS test was performed after thermal cycling. Color differences (ΔE_{00}) and relative translucency parameter (RTP) values were calculated. One-way analysis of variance (ANOVA) (ΔE_{00} and BFS), 2-way ANOVA followed by Tukey HSD tests (RTP), and chi-square tests (Weibull modulus and characteristic strength) were used for the statistical analyses (α=.05).

Results: No significant differences were observed among the ΔE_{00} values of tested materials (df=2, F=2.933, *P*=.070). RTP values were only affected by material type (*P*<.001) as AM had the highest RTP (*P*<.001), while IN and EX had similar values (*P*≥.165). BFS values varied among tested materials (df=2, F=21.341, *P*<.001). AM and EX had similar BFS values (*P*=.067) that were higher than that of IN (*P*≤.001). Weibull moduli of the materials were similar (*P*=.305). whereas EX had the highest and IN had the lowest characteristic strength values (*P*<.001)

Conclusions: While nano-lithium disilicate had the highest translucency, all materials had imperceptible color and translucency changes after thermal cycling when reported threshold values were considered. Newly introduced lithium disilicate glass-ceramics had adequate flexural strength as compared to the precursor material.

Keywords: Biaxial flexural strength, color stability, lithium disilicate, translucency, Weibull analysis

Computer-aided design and computer-aided manufacturing (CAD-CAM) technologies have facilitated the use of various restorative materials, including glass-ceramics.[1] Glass-ceramics are widely preferred due to their biocompatibility, high translucency, and pleasing esthetics.[2] In addition, the inherent brittleness of glass-ceramics is improved with the addition of various reinforcing particles such as lithium disilicate.[1]

Ever since the introduction of IPS e.max CAD (Ivoclar) in 2006,[1] lithium disilicate glass-ceramics have been the most preferred restorative material among glass-ceramics[3,4] due to their mechanical, optical, and chemical properties.[5,6] However, the dental market is constantly evolving and new lithium disilicate glass-ceramics are being rapidly introduced.[7,8] Nano-lithium disilicate glass-ceramic (Amber Mill, HassBio Corp) is one of the recently marketed products,[9] which has a unique feature that allows it to adjust its translucency depending on the crystallization process.[10] Another novel material is fully crystallized lithium disilicate glass-ceramic (Initial LiSi Block; GC Corp), which does not require any firing (crystallization or glazing) process and can be delivered after polishing.[6] Given the increased number of lithium disilicate glass-ceramics available in the dental market, studies on the comparison of the optical and mechanical properties of these materials would be beneficial for researchers and clinicians.

Studies on nano-lithium disilicate[6,9-15] and fully crystallized lithium disilicate[6,16] glass-ceramics are scarce. To the authors' knowledge optical properties of fully crystallized lithium disilicate have not been investigated, while only 3 studies on nano-lithium disilicate[9,10,12] have focused on this aspect. Therefore, the present study aimed to compare the color stability, translucency, and biaxial flexural strength of 2 newly introduced lithium disilicate glass-ceramics (nano-lithium disilicate and fully crystallized lithium disilicate) with those of a commonly used lithium disilicate glass-ceramic. In addition, given that restorative materials are constantly subjected to intraoral thermal changes,[17] the present study also aimed to evaluate the effect of thermal cycling on translucency. The null hypotheses were that 1) material type would not affect color stability, after

thermal cycling, 2) material type and thermal cycling would not affect translucency, and 3) material type would not affect biaxial flexural strength, after thermal cycling.

Materials and methods

Detailed information on the lithium disilicate glass-ceramics tested in the present study are presented in Table 1. A dental design software (exocad DentalCAD; exocad GmbH) was used to generate a disk-shaped (ø:12 mm, thickness: 1.2 mm) standard tessellation language (STL) file. Thirty specimens were fabricated from 3 different A2 shaded CAD-CAM lithium disilicate ceramics (Amber Mill (AM); HassBio Corp, Initial LiSi Block (IN); GC Corp, and IPS e.max CAD (EX); Ivoclar) by using this STL file and a milling unit (inLab MC XL; Dentsply Sirona) (n=10). The number of specimens was determined based on a priori power analysis (f=0.78, 1-β=95%, α= 0.05). While IN and EX specimens were fabricated from highly translucent blocks, AM specimens were crystallized in a porcelain furnace to achieve high translucency (Programat P310; Ivoclar).[11] EX specimens were also crystallized in the same furnace. Table 2 summarizes the firing parameters of AM and EX. All specimens were wet-ground using silicon carbide abrasive papers (#600, #800, and #1000) to achieve a uniform surface and thicknesses were controlled with a digital caliper (Absolute Digimatic; Mitutoyo). Finally, all specimens were polished using a low-speed handpiece, a 3-stage polishing assortment (OptraFine Assortment; Ivoclar), and a diamond polishing paste (OptraFine HP Polishing Paste; Ivoclar). Light blue and dark blue instruments (surface finishing) of the polishing assortment were used at 10000 rpm, while the polishing paste was applied by using a brush at 8000 rpm (high gloss polishing).[18]

Baseline color coordinate (L*, a*, and b*) measurements were performed using a spectrophotometer (CM-26d; Konica Minolta),[8,9] which has a diffused illumination integrating sphere system (8° viewing) and two specular component modes (specular component included and excluded). It allows either medium (12 mm/8 mm illumination/measurement area) or small area view

(6 mm/3 mm illumination/measurement area) and uses the CIE Standard 2° or 10° human observer characteristics along with a number of different illuminants in its color estimations. In the present study, the parameters of the spectrophotometer were set to small area view, 2° human observer characteristics, CIE D65 illumination, and the specular component was excluded.[9] One experienced practitioner (MD) performed measurements on white (L*: 91.90, a*: 1.42, and b*: 8.29), gray (L*: 51.73, a*: 1.01, and b*: 3.14), and black (L*: 8.52, a*: 0.32, and b*: 0.65) backings for each specimen 3 times, and these values were averaged. Before each measurement, the spectrophotometer was calibrated in line with manufacturer's recommendations and a saturated sucrose solution was used for the optical contact.

After baseline measurements, specimens were subjected to thermal cycling of 10000 cycles between 5 °C-55 °C and a dwell time of 30 seconds (THE 1100; SD Mechatronik), which corresponds approximately 1 year of intraoral use.[19] Color coordinate measurements were repeated using the same method. The color difference (ΔE_{00}) between the initial and after thermal cycling state of the specimens was calculated using the measurements performed on a gray background, while the measurements performed on black and white backgrounds were used to calculate the relative translucency parameter (RTP) of the specimens before and after thermal cycling using CIEDE2000 formula:

$$\text{CIEDE2000}=[(\Delta L'/k_L S_L)^2 + (\Delta C'/k_C S_C)^2 + (\Delta H'/k_H S_H)^2 + R_T(\Delta C'/k_C S_C)(\Delta H'/k_H S_H)]^{1/2}$$

Differences in the lightness, chroma, and hue of a specimen are represented by $\Delta L'$, $\Delta C'$, and $\Delta H'$, while R_T refers to the interaction between the chroma and hue differences in the blue region. Weighting functions of S_L, S_C, and S_H are used to adjust the total color difference. The parametric factors (kL, kC, and kH) were considered as 1.[3,18-20]

Biaxial flexural strength (BFS) test was performed according to International Organization for Standardization standard 6872:2015 (n=10).[21] Three stainless steel balls (ø: 3.2 mm) were positioned 120-degrees apart from each other on a circle (ø: 10 mm) to place the specimens. A universal testing

machine (Lloyd LRX; Lloyd Instruments) with an attached piston (ø: 1.6 mm) was used to apply 1 mm/min force to the center of the specimens until failure and the force at the moment of fracture was recorded.[22] The following formula was used to calculate the BFS values:[11,22,23]

$$\sigma = -0.2387\ P\ (X-Y)/d^2$$

$$X = (1+v)\ \mathrm{In}(r_2/r_3)^2 + [(1-v)/2]\ (r_2/r_3)^2$$

$$Y = (1+v)\ [1+\mathrm{In}(r_1/r_3)^2] + (1-v)\ (r_1/r_3)^2$$

with σ= BFS (MPa), P= force at failure (N), d= thickness of the specimen (mm), v= Poisson's ratio (0.187 for AM, 0.198 for IN, and 0.216 for EX),[6] r_1= radius of the support circle (mm), $r_{2=}$ radius of the loaded area (mm), and r_3= radius of the specimen (mm).

Kolmogorov Smirnov tests were used to analyze the normality of data. Due to normal distribution, 2-way analysis of variance (ANOVA) (RTP), 1-way ANOVA (ΔE_{00} and BFS), and Tukey's significantly honest difference tests were used to evaluate the differences among materials. Maximum likelihood estimation method was used to perform Weibull analysis (Minitab Software V.17; Minitab). Weibull moduli and characteristic strength values were further analyzed by using chi-square tests. All statistical analyses were performed using an analysis software (SPSS v23; IBM Corp) at a significance level at α=.05. Perceptibility and acceptability of ΔE_{00} values (perceptibility: 0.8 units, acceptability: 1.8 units)[24] and the differences in RTP values (ΔRTP) after thermal cycling (perceptibility: 0.62 units, acceptability: 2.62 units)[25] were evaluated based on the thresholds set by previous studies.

RESULTS

One-way ANOVA revealed that material type did not affect ΔE_{00} values (df=2, F=2.933, *P*=.070), while none of the materials had mean ΔE_{00} values that were greater than 0.8 units (ΔE_{00}= 0.57 for AM, ΔE_{00}= 0.42 for IN, ΔE_{00}= 0.43 for EX). Material type significantly affected the RTP values (*P*<.001), whereas the effect of thermal cycling (*P*=.886) and the interaction between main factors

(*P*=.887) were nonsignificant. AM had the highest RTP values before (*P*<.001) and after thermal cycling (*P*<.001). However, the differences between IN and EX were nonsignificant (*P*≥.165) (Table 3). When the mean ΔRTP values (ΔRTP= 0.4 for AM, ΔRTP= 0.59 for IN, and ΔRTP= 0.05 for EX) were considered, none of the materials had values greater than 0.62 units.

BFS of tested material showed significant differences (df=2, F=21.341, *P*<.001). IN had the lowest BFS values (*P*≤.001), while the difference between AM and EX was nonsignificant (*P*=.067). No significant differences were observed among the Weibull moduli of the specimens (*P*=.305) even though IN had higher Weibull modulus than the other specimens, while AM and EX had values that were closer to each other. As for the characteristic strength, EX had the highest and IN had the lowest values (*P*<.001) (Table 4). Figure 1 illustrates the survival probability of each material.

DISCUSSION

Tested lithium disilicate glass-ceramics had similar ΔE_{00} values after thermal cycling, which led to the acceptance of the first null hypothesis. In addition, all materials had ΔE_{00} values that were lower than previously described clinical perceptibility threshold of 0.8 units.[24] Even though this finding may indicate high color stability for tested materials even after a year-long intraoral use,[17] this interpretation should be made carefully as other mechanical and chemical factors may also affect a material's color stability. In addition, there are no universally accepted thresholds for perceptibility and acceptability, and other threshold values have also been used.[20]

A previous study investigated the color stability of AM when prepared in different thicknesses (0.7 mm and 1.5 mm) and subjected to coffee thermal cycling.[9] The authors[9] concluded that AM had similar ΔE_{00} values to those of EX, while 0.7 mm-thick specimens had perceptible color changes. Jurado et al[10] reported that EX had higher color stability than AM when subjected to additional crystallization firings. In another study, Liebermann et al[12] concluded that AM and EX had similar ΔE_{00} values when bonded with a light-shaded resin cement, while AM had lower ΔE_{00} values when a dark-shaded resin cement was used. A direct comparison between the present study and those

studies[9,10,12] could be misleading, given the differences in testing methods. In addition, to the authors' knowledge this is the first study on the optical properties of IN; thus, comparisons with previous studies were not possible.

Even though thermal cycling did not affect the RTP values of the specimens, material type had a significant effect on these values. Therefore, the second null hypothesis was rejected. AM had higher RTP values than those of other groups, regardless of thermal cycling. Chemical properties and crystalline structure of a ceramic are effective on its translucency.[19] Therefore, this difference may be related to the differences in crystal sizes of tested materials as AM (0.2 μm)[26] had the smallest crystals, followed by IN (0.3 μm)[16] and EX (1-1.5 μm).[27] However, another study reported similar RTP values for AM and EX.[9] When ΔRTP values were considered, all materials had imperceptible changes according to the thresholds set by Salas et al.[24] Based on these results and the fact that IN had similar RTP values to EX in the present study, it can be speculated that IN and AM may have clinically pleasing esthetics similar to that of EX. However, given the limited knowledge on the optical properties of these newly introduced lithium disilicate glass-ceramics, this hypothesis needs in vivo support.

AM and EX had similar BFS values that were significantly higher than that of IN. Therefore, the third null hypothesis was rejected. The size and the number of crystals in a restorative material affect its mechanical properties.[4] A previous study reported that crystals that are greater than 1 μm increased the fracture toughness of ceramics,[6] which may be associated with the difference between EX and IN. Even though AM has the smallest crystals, a recent study on lithium silicate glass-ceramics reported that both AM and EX had higher lithium disilicate content compared with IN.[6] In addition, given that AM and EX specimens were crystallized, microcracks that are inherently present in blocks or that may have occurred during milling might have disappeared after heat treatment. Nevertheless, all materials had BFS values that were higher than 300 MPa, which is the threshold value for a material to be used monolithically for single crowns or 3-unit fixed partial dentures that do not involve molar teeth according to ISO 6872:2015.[21] In addition, IN had a similar trend in terms of

characteristic strength and BFS as it had the lowest value among tested materials. Even though no significant difference was observed among tested materials, higher Weibull modulus of IN may be associated with the fact that it was the only pre-crystallized block and additional crystallization process may be prone to human error. It can also be speculated that mechanical properties do not necessarily reflect reliability.[11]

Stawarczyk et al[11] compared the initial and after hydrothermal aging BFS values of AM and EX, and reported that EX had higher initial values. This finding was corroborated by a recent exploratory study on lithium silicate glass-ceramics, which concluded that EX had higher fracture toughness than IN and AM.[6] However, Stawarczyk et al[11] also showed that EX and AM had similar BFS values after hydrothermal aging, which is in line with the present study. Other studies on the mechanical properties of tested materials have reported similar fracture resistance values while comparing AM[9] and IN[16] with EX after thermomechanical aging.

All specimens were subjected to a single polishing process after fabrication for standardization. However, this may be a limitation considering that previous studies have shown the significant effect of surface treatments on CAD-CAM materials.[3,14,18] In the present study, a standardized material thickness that was in line with ISO 6872:2015[21] was preferred as the same specimens were used to evaluate all parameters. However, different thicknesses may lead to different ΔE_{00} and RTP values.[19] In addition, other factors that may affect the tested parameters such as resin cement, background tooth layer, staining solutions, brushing, and mastication were not included. Another limitation was that a single device was used for the color measurements, and different devices, as well as light sources, may lead to different results. The fact that only one mechanical test was performed in the present study is a limitation. BFS test has the advantage of maintaining the contact between the specimen and the loading tip throughout the procedure.[13] However, this test and flexural strength parameter alone cannot be used to estimate the clinical applicability of a material. A fractographic analysis was not performed in the present study, which may have elaborated the findings of the BFS test. The authors believe that the results of the present study should be interpreted

as preliminary, particularly given that fully crystallized lithium disilicate is a relatively new material on the dental market and the specimens tested in the present study were not shaped as actual prostheses. Future in vivo and in vitro studies should investigate other properties of nano-lithium disilicate and fully crystallized lithium disilicate glass-ceramics such as biocompatibility, fracture resistance, fracture toughness, stainability, fabrication trueness, and internal adaptation to corroborate the findings of the present study and to elaborate the clinical applicability of these materials.

CONCLUSIONS

Nano-lithium disilicate glass-ceramic had the highest translucency among tested materials, regardless of thermal cycling, while all materials had imperceptible color and translucency changes after thermal cycling when reported thresholds were considered. Fully crystallized lithium disilicate glass-ceramic had the lowest flexural strength values; however, newly introduced lithium disilicate glass-ceramics had adequate flexural strength to be used for restorations that are indicated for the precursor material. In addition, non-significantly higher Weibull modulus of fully crystallized lithium disilicate glass-ceramic may indicate its reliability compared with a pre-crystallized CAD-CAM blocks of similar chemical composition.

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TABLES

Table 1. List of CAD-CAM lithium disilicate glass-ceramics used

Material	Chemical Composition (wt%)	Manufacturer
Amber Mill, A2, HT (Nano-lithium disilicate glass-ceramic, AM)	SiO_2: <78% Li_2O: <12% Coloring oxides: <12%	HASSBio, Kangneung, Korea
IPS e.max CAD, A2, HT (Lithium disilicate glass-ceramic, EX)	SiO_2: 57-80% Li_2O: 11-19% K_2O: 0-13% P_2O_5: 0-11% ZrO2: 0-8% ZnO: 0-8% Coloring oxides: 0-8%	Ivoclar, Schaan, Lichtenstein
Initial LiSi Block, A2, HT (Fully crystallized lithium disilicate glass-ceramic, IN)	SiO_2: 81% P_2O_5: 8.1% K_2O: 5.9% Al_2O_3: 3.8% TiO_2: 0.5% CeO_2: 0.6%	GC Corp, Tokyo, Japan

Table 2. Crystallization parameters

	B [°C]	S [min]	t1/t2 [°C/min]	T1/T2 [°C]	H1/H2 [min]	Vac. 1 [°C] / Vac. 2 [°C]	L [°C]	tL *
AM	400 °C	3 min	60 °C/min	815 °C	15 min	550/815 °C	690 °C	0
EX	403 °C	6 min	90/34 °C/min	830/850 °C	10 s/7 min	550-830/830-850 °C	710 °C	0

Table 3. Mean ±standard deviation ΔE_{00} and RTP values (95% CI)

Material	ΔE_{00}	RTP before thermal cycling	RTP after thermal cycling
AM	0.57 ±0.18[a] (0.44-0.70)	23.90 ±1.02[b] (23.17-24.62)	23.50 ±1.31[b] (22.56-24.44)
IN	0.42 ±0.14[a] (0.32-0.52)	21.32 ±1.77[a] (20.05-22.59)	20.73 ±1.46[a] (19.69-21.77)
EX	0.43 ±0.15[a] (0.32-0.54)	20.33 ±0.15[a] (20.22-20.44)	20.28 ±0.18[a] (20.15-24.41)

*Different superscript lowercase letters indicate significant differences in columns (P<.05)

Table 4. Mean ±standard deviation biaxial flexural strength (BFS in MPa), Weibull modulus, and characteristic strength (MPa) values (95% CI)

Material	BFS	Weibull modulus	Characteristic strength
AM	514.08 ±33.03[b]	19.69[a]	528.11[b]
	(490.45-531.29)	(12.17-31.85)	(510.87-545.94)
IN	458.50 ±16.09[a]	27.76[a]	466.25[a]
	(446.98-470.01)	(17.98-42.87)	(455.30-477.46)
EX	560.56 ±48.17[b]	16.56[a]	580.55[c]
	(526.10-595.01)	(9.71-28.26)	(558.23-603.77)

*Different superscript letters indicate significant differences in columns (P<.05)

FIGURES

Figure 1. Survival probability versus biaxial flexural strength (BFS) curves for tested materials