Clinical and Experimental
Health Sciences

Optical Properties of Newly Developed Monolithic CAD/CAM Materials After Aging

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Received: 07.10.2022 **Accepted:** 17.01.2023

ABSTRACT

Objective: With the widespread use of CAD/CAM (computer aided design and manufacture) systems in dentistry, many restorative materials have been produced. In our study, we aimed to evaluate the change in optical properties of newly developed translucent monolithic zirconia (TMZ) (Cercon HT Dentsply, Sirona, USA), zirconia-reinforced lithium silicate (ZLS) (Celtra Duo, Dentsply, Germany) and lithium disilicate (LS2) (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) materials with aging.

Methods:10 discs of 12mm diameter and 1.2 mm thickness were produced from high translucent A2 color of each material. The aging process was applied in an autoclave under 134ºC, 0.2MPa pressure. For optical evaluation, L*, a*, b* values of the samples were measured by spectrophotometry before aging, after 3 hours and 6 hours aging. ΔE00 values and translucency parameters (TP) were calculated. The data were evaluated statistically.

Results: In the present study, all the materials had undergone color change as a result of aging, but this change was within acceptable limits (ΔE00 <1.8). The ΔE00 value of the ZLS was above the perceptibility threshold (ΔE00 >0.8). There was a decrease in the translucency of all materials used in the study, but this decrease was not statistically significant.

Conclusion: In the light of the findings obtained from this in vitro study, aging did not cause color and translucency changes in TMZ, ZLS, LS2 monolithic CAD/CAM materials.

Keywords: CAD/CAM, zirconia, translucency, CIE Lab, optic

1. INTRODUCTION

With the increase in aesthetic expectations of patients and the use of CAD/CAM (computer aided design and manufacture) technologies in dentistry, many new restorative materials with superior properties in terms of biocompatibility, aesthetics and function have been produced (1).

Lithium disilicate reinforced glass-ceramic (LS_2) restorations are preferred especially in anterior restorations due to their optical properties, however the most important shortcoming of these restorations is their low fracture resistance. In 2005, precrystalline IPS e.max CAD blocks containing 40% lithium metasilicate ($Li_2Si_2O_3$) and lithium disilicate ($Li_2Si_2O_5$) crystal cores were produced. Blocks with an initial blue color have a medium hardness and strength (130MPa), so they are easier to mill (2). After milling, the restoration is heated to 850°C, during this process flexural strength increases to 262±88MPa and restoration changes to the selected tooth color (3). Crystallized material contains approximately 1.5mm in size fine grainy needle-shaped lithium disilicate crystals integrated into a glass matrix at a rate of 70% by volume (4).

Zirconia is one of the materials with superior biological and mechanical properties, which contains 3 different crystal forms; monoclinic, tetragonal and cubic (5). Zirconia is a ‘metastable’ material that can transform from the tetragonal phase to the monoclinic phase under certain conditions. As a result of mechanical forces such as sandblasting, abrasion and heat treatments, some of the tetragonal particles turn into monoclinic particles that are larger in volume (6). This increse in volume between the monolithic crystals causes the particles to separate from the surface. (7). As a result of this separation, the surface roughness increases and microcracks are formed (8). Kobayashi et al. observed that spontaneous transformations from tetragonal phase to monoclinic phase

ISSN:2459-1459

DOI: 10.33808/clinexphealthsci.1185717

occur in zirconia exposed to high humidity environment and low temperature. This phenomenon is known as 'Low temperature degradation (LTD)' or 'Hydrothermal degradation' (9). Although LTD often occurs at 200-400°C, prolonged exposure of the material to heat and moisture in the oral cavity can also cause LTD (10).

Due to zirconia's opaque appearance, it must be veneered with feldspathic porcelain to provide an acceptable aesthetic. This layered structure combines the strength of zirconia with the aesthetics of ceramic (11). However, due to ceramic chipping in veneered zirconia systems (12), more translucent monolithic zirconia (TMZ) restorations have been developed by modifying the microstructure (13). Increasing the yttria concentration is the most preferred method used for this purpose. In this way, the cubic phase amount and translucency of the material are increased. The increase in yttria content causes an increase in the amount of optically isotropic cubic phase and hence its translucency (14). A yttria content of 5% moles is defined as 'high translucent zirconia' and 8% moles as 'ultratranslucent zirconia' (15).

Zirconia-reinforced lithium silicate glass ceramics (ZLS) are new materials that can be used with CAD/CAM technology produced to combine the positive mechanical properties of zirconia with the aesthetic appearance of glass ceramics. It is obtained by strengthening the glass phase with 10% by weight of ZrO_2. After crystallization, lithium metasilicate and lithium disilicate crystals exist as a binary microstructure in the glassy phase containing 10% zirconium oxide. The crystals are 0.5-0.7 µm in size and are 4-8 times smaller in size than lithium disilicate crystals (16).

The aim of this study was to evaluate the effect of artificial aging on color and translucency of newly developed TMZ, ZLS and LS_2 CAD/CAM materials.

The null hypothesis was that no significant differences would be found in the color and transluceny of monolithic CAD/CAM materials with increasing aging time.

2. METHODS

According to the results of the power analysis performed to determine the number of samples required for this study, at least 8 samples should be taken for each of the groups with 95% confidence and 95.6% test power.

Three types of ceramic materials were used in this study; translucent monolithic zirconia (TMZ) (Cercon HT, Dentsply, Sirona, USA), zirconia reinforced lithium silicate (ZLS) (Celtra Duo, Dentsply, Hanau-Wolfgang, Germany) and lithium disilicate (LS_2) (IPS emax CAD, Ivoclar Vivadent, Schaan, Leichtenstein) (Table 1).

Table 1. *Materials used in the study and manufacturer*

	Material Type	Composition	Manufacturer	Lot No
IPS emax CAD	Lithium disilicate	%57-80 SiO_2, %11-19 Li_2O, K_2O_3, P_2O_5, ZrO_2, Al_2O_3	Ivoclar Vivadent, Schaan, Leichtenstein	Y18855 Y33214 X42738
Celtra Duo	Zirconia reinforced lithium silicate	%58 SiO_2, %18 Li_2O, %10 ZrO_2, P_2O_5, Al_2O_3	Dentsply,Hanau-Wolfgang,Germany	16002157 16004027
Cercon HT	Translucent zirconia	Zirkonyum oksit, %5 litrium oksit, hafnium oksit <%3,Al_2O_3,SiO_2<%1	Dentsply, Sirona, USA	18033377 18034109

10 disc shaped specimens (12X1.2 mm) from HT (high translucent), A2 discs and blocks of materials were prepared for each group with CAD/CAM technology. For LS_2 and ZLS discs, partially crystallized blocks were milled to cylindrical form using Cerec system (InLab MC X5 Sirona, Bensheim, Germany). Then the cylinders were sliced into discs with a low-speed water-cooled diamond saw to the designed thickness (IsoMet 1000, Buehler, Illinois, USA). After milling, the specimens were crystallized according to the manufacturer's instructions. For TMZ, specimens with the final diameter of 12 mm and the final thickness of 1.2 mm were milled (InLab MC X5 Sirona, Bensheim, Germany) and sintered in a sintering oven (MIHM-VOGT GmbH & Co. KG, Stutensee-Blankenloch, Germany) according to the manufacturer's instructions. All sample sizes were checked with a digital caliper (Insize Mini Digital Caliper Series 1111, China). Finally one side of the discs were polished with coarse, medium coarse and super fine grit ceramic polishing rubbers (Diapol HP,Eve, Ernst Vetter GmbH, Germany) and polishing paste (Renfert Polish all in one Diamond Polish, Renfert GMBH, Hilzingen, Germany).

The specimens were artificially aged in an autoclave (Goldberg, Eryiğit Otoklav A.Ş., Türkiye) at 134 °C, under 0.2 MPa for 3 and 6 hours according to ISO standard 13356 (17).

According to the CIE Lab color system developed by the Commission Internationale de l'Eclairage, international color commission; each color is expressed in terms of three components; referred to by the abbreviations L^*(brightness), a^*(red-green), and b^*(yellow-blue) (18). The vertical axis L represents the brightness or lightness coordinates of the object between white and black, the horizontal axis a represents the chroma coordinates of the object between red and green, the horizontal axis b represents the chroma coordinates of the object between yellow and blue. The intersection of these three coordinates gives the value of that color (19). The amount of color change (ΔE_{00}) that occurs in any object after a certain period of time or as a result of an applied treatment can be calculated using L^*, a^* and b^* values.

For optical evaluation, measurements were made on the polished surfaces of the samples on white (L=92,98, a=-1,42, b=4,34) and black (L=26,38, a= 0,24, b= – 0,08) backgrounds with a spectrophotometer (CM-26d, Konica Minolta, Tokyo, Japan) before aging (baseline), after aging for 3 hours and after aging for 6 hours. L^*, a^* and b^* values were obtained. The color change (DE_{00}) of the samples was calculated with the CIEDE2000 equation. In this study, the CIEDE (1:1:1) system, in which kL, kC and kH parametric values were taken as '1', was used.

$$DE_{00}=[(DL'/k_LS_L)^2 + (DC'/k_CS_C)^2+ (DH'/k_HS_H)^2 +R_T (DC'/k_CS_C)(DH'/k_HS_H)]^{1/2}$$

In the CIE Lab system, the translucency value of an object is expressed by the translucency parameter (TP)(20). Translucency parameters were calculated with the following formula using the color difference of L^*, a^* and b^* values measured from samples on black and white backgrounds.

$$TP=[(L_{B*} – L_{W*})^2 + (a_{B*} – a_{W*})^2+ (b_{B*} – b_{W*})^2]^{1/2}$$

For statistical analysis the data were expressed as mean and standard deviation. The distribution normality of the variables were tested with Kolmogorov-Smirnov test. According to Kolmogorov Smirnov test, the data were not normally distributed so non-parametric tests were used for analyses. For all tests, SPSS 27.0 Statistical Software Package (IBM Corporation,New York,USA) was used and the level of significance was set at p<0.05. Kruskal-Wallis and Mann-Whitney U tests were used in the analysis of quantitative independent data, Friedman test and Wilcoxon test were used in the analysis of dependent quantitative data.

3. RESULTS

The means and the standard deviations of the L^*, a^*, b^* values are shown in Tables 2,3 and 4. No significant difference was observed between the baseline, after 3 and 6 hours of aging L^* and b^* values in TMZ, ZLS and LS_2 groups (Table 2,Table 4). In the TMZ group, no significant difference was observed between the baseline and after 3 and 6 hours of aging a^* values. In the ZLS group, 6 hours aging a^* value was significantly higher than the 3 hours aging and baseline a^* values. No significant difference was observed between the baseline and 3 hours of aging a^* values. In the LS_2 group, the 6 hours of aging a^* value was significantly higher than the baseline a^* value but 3 hours of aging a^* value did not differ significantly from baseline and 6 hours of aging a^* value (Table 3).

When we compared the ΔE_{00} values of the materials at different aging intervals; ΔE_{00} values of all groups were below acceptability threshold after aging of 3 hours and 6 hours (ΔE_{00} < 1.8). Only the ΔE_{00} values of ZLS were above the perceptability threshold (ΔE_{00} > 0.8). There was no significant difference between the baseline-3hours, baseline-6hours and 3 hours-6 hours ΔE_{00} values of all materials (p>0.05) (Table 5).

When we compared the TP values between groups; baseline, 3 hours and 6 hours of aging TP values were significantly higher in the LS_2 group than in the TMZ and ZLS groups before and after aging. The TP values of the ZLS group were also significantly higher than the TMZ group. No significant difference was observed between the baseline, 3 hours and 6 hours of aging TP values of TMZ, ZLS and LS_2 groups (p>0.05) (Table 6).

***Table 2.** L* values of materials according to different aging times*

		Min-Max	Median	Mean ± SD	p
TMZ	Baseline L	76,1-77,2	76,5	76,5 ± 0,4	0,452 F
	3 hours L	76,3-77,2	76,8	76,4 ± 0,2	
	6 hours L	76,5-77,3	76,8	76,8 ± 0,3	
ZLS	Baseline L	72,4-74,7	73,5	73,5 ± 0,7	0,273 F
	3 hours L	72,1-74,4	73,4	73,4 ± 0,7	
	6 hours L	72,0-74,1	73,0	73,0 ± 0,6	
LS2	Baseline L	74,4-75,6	74,9	75,0 ± 0,4	0,061 F
	3 hours L	73,8-75,6	74,6	74,7 ± 0,5	
	6 hours L	73,3-76,3	74,2	74,3 ± 0,9	

[F] Friedman

***Table 3.** a* values of materials according to different aging times*

		Min-Max	Median	Mean± SD	p
TMZ	Baseline a	2,66-3,30	2,93	2,96 ± 0,19	0,452 F
	3 hours a	2,70- 3,19	2,90	2,93 ± 0,14	
	6 hours a	2,84- 3,37	2,95	2,99 ± 0,16	
ZLS	Baseline a	0,69-1,82	1,53	1,42 ± 0,32	0,003 F
	3 hours a	1,41-1,96	1,74	1,73 ± 0,17	
	6 hours a	1,72-2,17	1,90	1,91 ± 0,15	
LS2	Baseline a	0,30- 0 53	0,38	0,40 ± 0,08	0,045 F
	3 hours a	0,10- 0,69	0,51	0,46 ± 0,17	
	6 hours a	0,39- 0,71	0,54	0,55 ± 0,13	

[F] Friedman

***Table 4.** b* values of materials according to different aging times*

		Min-Max	Median	Mean± SD	p
TMZ	Baseline b	15,7-17,2	16,4	16,4± 0,4	0,067 F
	3 hours b	15,4-17,2	15,9	16,0 ± 0,5	
	6 hours b	15,7-16,8	15,9	16,0 ± 0,3	
ZLS	Baseline b	14,1-16,2	15,3	15,3 ± 0,6	0,150 F
	3 hours b	15,2-16,0	16,0	15,8 ± 0,3	
	6 hours b	15,0-16,3	15,7	15,7 ± 0,5	
LS2	Baseline b	13,4-14,1	14,0	13,9 ± 0,2	0,082 F
	3 hours b	13,5-14,5	14,3	14,2 ± 0,3	
	6 hours b	13,8-14,6	14,0	14,2 ± 0,3	

[F] Friedman

Table 5. *The ΔE_{00} values of the materials at different aging times*

		Min-Max	Median	Mean ± sd	p
TMZ	B- 3h	0,12- 0,99	0,56	0,51 ± 0,27	0,741 F
	B-6h	0,17- 1,07	0,55	0,57 ± 0,27	
	3h-6h	0,20- 1,03	0,31	0,44 ± 0,28	
ZLS	B- 3h	0,30- 1,82	0,86	0,95 ± 0,54	0,497 F
	B-6h	0,27- 2,29	0,69	0,89 ± 0,61	
	3h-6h	0,25-1,86	0,73	0,81 ± 0,46	
LS2	B- 3h	0,11-1,15	0,60	0,61 ± 0,31	0,741 F
	B-6h	0,21-1,51	0,62	0,68 ± 0,38	
	3h-6h	0,11- 1,47	0,83	0,83 ± 0,44	

F Friedman

Table 6. *TP values of materials at the end of different aging times*

		Min-Max	Median	Mean ± sd	p
TMZ	Baseline TP	6,9- 7,8	7,3	7,3 ± 0,3	0,273 F
	3 hours TP	7,2- 7,7	7,5	7,4 ± 0,2	
	6 hours TP	7,2- 7,5	7,3	7,3 ± 0,1	
ZLS	Baseline TP	14,3- 16,7	16,3	16,0 ± 0,7	0,150 F
	3 hours TP	14,4- 17,0	15,8	15,9 ± 0,8	
	6 hours TP	14,6- 16,6	15,5	15,5 ± 0,7	
LS2	Baseline TP	15,6- 17,0	16,4	16,4 ± 0,4	0, 301 F
	3 hours TP	15,2- 17,0	16,4	16,3 ± 0,5	
	6 hours TP	15,3- 18,0	16,0	16,3 ± 0,8	

F Friedman

4. DISCUSSION

The null hypothesis that aging would not affect the color and translucency of monolithic CAD/CAM materials (TMZ, ZLS and LS_2) used in the present study was accepted.

The thickness of the material used in monolithic restorations effects the optical and mechanical properties (21). Kanchanavasita et al. reported that material thickness has a significant effect on translucency, and translucency increases with decrease in thickness (22). For all-ceramic restorations, a thickness of 1-1.5mm is generally required (23). In our study the samples were prepared in a thickness of 1.2 mm in order to be similar to the crown thicknesses used in the clinic.

In addition to thickness, surface finishing processes are also effective in the translucency of ceramics (24). It has been reported that in glazed monolithic zirconia, the glaze layer wears off over time and the restoration causes wear on the opposing tooth or restoration (25). For this reason, it has been stated that polishing is a more suitable surface finishing process than glazing for zirconia restorations (26). Likewise, polishing the ZLS and LS_2 restoration surfaces is reported to be sufficient in terms of wear and plaque retention (27). In this study, mechanical polishing was applied to one surface of each sample in order to ensure standardization between the different materials used.

Materials to be used in restorative dentistry age by exposure to temperature changes, chewing forces and moisture in the oral environment, so it is very important for these materials to be aesthetically and mechanically stable as well as biocompatibility. There are many aging methods that can simulate long-term clinical conditions in a short time. One of this methods is accelerated aging in autoclave (17). Aging in an autoclave at 134°C for 1 hour is an effective method for predicting the long-term performance of materials since it is equivalent to 3-4 years of in vivo use (28). Therefore, in our study, autoclave aging was applied for 3 hours and 6 hours, reflecting the clinical use of approximately 10-20 years.

For long-term clinical success of aesthetic restorations, the color must be stable, but the color and translucency of dental ceramics is affected by hydrothermal aging (29,30). The amount of color change is interpreted according to two different threshold values: the color change value that can be noticed by 50% of the observers is defined as the 'perceptibility threshold' and the acceptable color change by 50% of the observers is defined as the 'acceptability threshold' value. Paravina et al. reported the perceptability threshold as 0.8 and the acceptability threshold as 1.8 for ΔE_{00} (31).

There are various studies evaluating the color change of all ceramics after aging. Different outcomes were reported in these studies, resulting in L^*, a^*, b^* values that increased or decreased and ΔE_{00} values that exceeded or did not exceed the acceptability threshold (32,33,34). The materials used in the studies, the aging protocols, the various color measurement devices and the accepted threshold values could all be contributing factors to the wide range of results. In our study, after 3 hours and 6 hours of aging, L^*, a^*, b^* values were affected. In general, translucent zirconia samples became lighter, more reddish, and more bluish, while ZLS and LS_2 samples became darker, more reddish and more yellowish. Zhang et al. reported in their meta-analysis study that aging for more than 20 hours caused color change in translucent zirconias above the acceptability threshold (35). Similarly, in this study, aging period was less than 20 hours; color changes in all groups were within the clinically acceptable range. However, ΔE_{00} values of ZLS samples were above the perceptability threshold. According to Gonuldas et al. thermal conditions may cause pigment destruction in dental ceramics, resulting in color instability (36).

An aesthetic restoration depends not only on the color match but also on the harmony of translucency (37). Translucency is a substance's ability to reflect some light while also transmitting some of it (38). A fully opaque material's TP value is zero. As the translucency of the material increases, the TP value also increases (20).

The TP value of LS_2 was found to be higher than TMZ which is consistent with the studies of Nassary Zadeh and Harada. (39,40). However, unlike the studies of Sen and Us, LS_2 baseline TP values were significantly higher than ZLS baseline TP values. The literature contains a wide range of results from studies on the effect of aging on translucency (33,41,42,43). According to the findings of the meta-analysis study of Zhang et al., there is a significant change in TP values when the aging time in autoclave exceeds 20 hours (35). In the present study consistent with this literature, aging for 3 hours and 6 hours caused a decrease in TP values of translucent zirconia samples, but this change was not statistically significant.

Since there is no study in the literature in which ZLS is aged in an autoclave, we compared our data with studies using similar thermal aging methods. Alp et al. subjected the ZLS and LS_2 samples to 5000 thermal cycles with coffee. They reported that LS_2 was more translucent than ZLS before and after the thermal cycle, and the thermal cycle reduced the translucency of both materials (44). Porojan et al. applied 10000 thermal cycles to ZLS and LS_2 samples. They reported that aging caused a significant decrease in TP in all samples (45). In our study, unlike these studies, aging caused a slight decrease in TP values of ZLS and LS_2 samples, however, this change was not significant. The reason why our findings were different from literature may be the different aging methods applied.

Tong et al. reported that porosity is one of the factors affecting the translucency of ceramics (46). During clinical use, a dissolution occurs in glass ceramics. The difference between the dissolution rates of the crystal and glass phases causes a rough surface to form. In zirconia ceramics, on the other hand, surface roughness may occur with the change of the volume of the crystals in the phase change regions and affect the translucency (6,29). Pereira et al. state that a smoother surface reduces hydrothermal aging by decreasing the areas that interact with water (47). In the present study, mechanical polishing was applied to TMZ, ZLS and LS_2 samples, and it was determined that there was no significant change in translucency as a result of aging, and the color difference remained at a clinically acceptable level. Based on this result, it can be concluded that the mechanical polishing of zirconia and glass ceramic materials is effective in protecting the materials from the negative effects of hydrothermal aging.

5. CONCLUSION

All the materials used in this study have undergone color change as a result of aging, but this change was within acceptable limits. Only the ΔE_{00} value of ZLS was above the perceptability threshold. There was no statistically significant difference in the amount of color change between aged TMZ, ZLS and LS_2 samples. After aging the highest TP value was found in the LS_2 group, and the lowest TP value was found in the TMZ group. The difference between groups was significant. There was a decrease in the translucency of all materials used in the study, but this decrease was not statistically significant.

In the light of the findings obtained from this in vitro study, translucent zirconia, zirconica reinforced lithium silicate and lithium disilicate monolithic ceramic materials that were used in this study were found to be optically sufficient for approximately 15 years of clinical use. However, further clinical studies are needed to support the results of our study.

Funding: *The present work was supported by the Research Fund of Istanbul University. Project No: 36863*

Conflicts of interest: *The authors declare that they have no conflict of interest.*

Peer-review: *Externally peer-reviewed.*

Author Contributions:

Research idea: HBÖ

Design of the study: ES, HBÖ

Acquisition of data for the study: HBÖ

Analysis of data for the study: HBÖ

Interpretation of data for the study: ES, YUA

Drafting the manuscript: HBÖ

Revising it critically for important intellectual content: YUA, ES

Final approval of the version to be published: HBÖ, YUA, ES

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How to cite this article: Uysal Özel HB, Aslan YU, Sancaklı E. Optical Properties of Newly Developed Monolithic CAD/CAM Materials After Aging. Clin Exp Health Sci 2023; 13: 645-651. DOI: 10.33808/clinexphealthsci.1185717