Original Article

Effects of Aging on Mechanical and Optical Properties of Translucent and Opaque Zirconia

Sima Abbasimoghaddam[1], Seyed Mohammad Reza Hakimaneh[2], Tabassom Hooshmand[3], Sayed Shojaedin Shayegh[4]

[1] Dental Student, Faculty of Dentistry, Shahed University, Tehran, Iran
[2] Assistant Professor, Department of prosthodontics, Faculty of Dentistry, Shahed University, Tehran, Iran
[3] Professor, Department of Dental Biomaterials, School of Dentistry/Research Center for Science and Technology in Medicine, Tehran University of Medical Sciences, Tehran, Iran
[4] Associate Professor, Department of prosthodontics, Faculty of Dentistry, Shahed University, Tehran, Iran

Abstract

Background and Aim: This study evaluated the effect of hydrothermal aging on optical and mechanical properties of opaque and translucent zirconia.
Materials and Methods: For the biaxial flexural strength (BFS) test, 40 sintered opaque and translucent disc-shaped zirconia, and for the assessment of surface roughness and optical properties, 20 specimens were fabricated and artificially aged in an autoclave for 10 h. The CIE L*a*b* color parameters were measured. The translucency parameter (TP), contrast ratio (CR), opalescence (OP), and color difference against a white (ΔEW) and a black (ΔEB) background were all calculated. The surface roughness (Ra and Rz) was analyzed using a profilometer and the BFS test was performed at a crosshead speed of 1 mm/min. Data were analyzed using two-way ANOVA, t-test, and Mann-Whitney test ($\alpha=0.05$).
Results: The mean Ra (P=0.023) and Rz (P=0.011) significantly decreased after aging. Aging had no significant effect on TP (P=0.384), CR (P=0.261), or BFS (P=0.912). The mean TP values of translucent specimens were significantly higher than those of opaque specimens (P=0.046). For translucent specimens, the mean OP did not significantly change after aging (P=0.685) but aging significantly increased the OP of opaque specimens (P=0.007). The mean ΔEW and ΔEB of translucent specimens were higher than those of opaque specimens (P=0.044 and P=0.019, respectively).
Conclusion: Although aging affected the OP parameter, other optical properties and also BFS of both zirconia types were not affected by hydrothermal aging. However, the surface roughness of both zirconia types significantly decreased as a result of aging.
Key Words: Zirconium Oxide, Aging; Surface Properties, Flexural Strength

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Corresponding author:
Sayed Shojaedin Shayegh, Associate Professor, Department of prosthodontics, Faculty of Dentistry, Shahed University, Tehran, Iran

shayegh.hakim@gmail.com

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Introduction

The use of tooth-colored restorations has greatly increased in cosmetic dentistry. Different types of full-coverage restorations are now available, such as metal-ceramic and all-ceramic restorations. Among them, all-ceramic restorations are becoming more popular due to their excellent esthetics. (1) Zirconia is an esthetic material that is tougher and more biocompatible than other ceramic materials, and has good mechanical properties. (2,3) Zirconia has three structural types. The monoclinic crystal structure exists at temperatures below 1170°C. The tetragonal crystal structure exists at 1170°C to 2370°C, and the cubic crystal structure exists at temperatures above 2370°C. (4,5)

Optical property of a material is its reaction to electromagnetic radiation in the visual spectrum. (6) The esthetic properties of zirconia restorations differ according to the optical properties of the material including its opalescence, contrast ratio (CR), translucency, direct transmittance of light, and color. (7)

Opacity is an important characteristic affecting the esthetic properties of dental restorations. (8) The CR, which is used to quantify opacity, depends on the material thickness and background reflectance of restorations. (8) The state between transparency and opacity is depicted as translucency. A dental ceramic system's translucency depends on the grain size, thickness, coefficients of scattering and absorption, and pigmentations. (8) In addition, the ceramic source, amount and type of additive materials, number of pores, ceramic shade, sintering temperature and time, presence of a cubic phase, refraction index, surface roughness, atmospheric conditions while sintering, CR, and staining technique can affect the translucency of zirconia restorations. (7) The translucency of a material is measured using the translucency parameter (TP). The definition of TP is the difference in the color of a material of a given thickness against white and black backgrounds. (2) To calculate the TP, the color difference of the same sample against the black and white backgrounds is measured. (9) From an esthetic viewpoint, a ceramic material should allow light passage such that it displays the optical properties of natural teeth. (10)

The surface roughness of a material is recorded on the basis of the deviations from the normal vector of an ideally smooth surface. If these deviations are large, the surface is rough and if they are small, the surface is smooth. (11) Coarse surfaces cause the adjacent and opposing teeth to wear and result in their staining. Therefore, the surface roughness of the coating materials should be minimized to ensure optimal comfort, esthetics, and oral hygiene. (12)

The processing factors that can affect the zirconia's optical properties include the aging, baking temperature, and particle size. (1,13) Aging, i.e., low-temperature degradation (LTD), can also affect the zirconia surface roughness. LTD can affect the surface microstructure and flexural strength of zirconia. (14) LTD involves the spontaneous tetragonal-to-monoclinic phase transformation at low temperatures occurring over time. As aging progresses, the flexural strength decreases and surface roughness increases.[14] Presence of the cubic phase in the zirconia structure and particle sizes < 1 μm make the zirconia’s internal structure more susceptible to aging. (15) The tetragonal-to-monoclinic phase transformation can also decrease the translucency of zirconia restorations and has a negative effect on the optical properties of the restoration. (7) To simulate the aging of zirconia, various methods such as hydrothermal aging and accelerated aging have been employed. (16-18) If the transformation is induced in presence of water, it is called hydrothermal aging.(9) In this process, samples are placed in an autoclave at 134 °C and 2.3 bar pressure for a specific duration. One hour of this process is equal to 4 years of usage in clinical conditions. (19) The all-ceramic zirconia restorations are expected to last for 15-20 years on average. (20)

Fischer et al. (21) introduced flexural strength as an important factor affecting the durability of ceramic restorations. In addition, according to Pittayachawan et al, (20) the flexural strength is an important and reliable factor to evaluate the brittleness of materials. Flexural strength depends on many different factors such as the specimen thickness and grain size. (22) According to Takano et al, (23) the number of firings and aging duration influence the flexural strength. The flexural strength of dental restorative materials is measured by three different methods: three-point, four-point, and biaxial flexural strength (BFS) tests. In all three tests, a constant force is applied until the material breaks. In the three-point test, a non-uniform stress is employed, while in the four-point test, a uniform stress is applied between two force-applying pistons. In the BFS test, the force is applied to the center of a disc; thus, the possibility of breakage decreases at the edges. (24) Therefore, the BFS test was used in this study to measure the flexural strength of zirconia specimens.

Information on how aging could affect different properties of translucent and opaque zirconia is lacking. Therefore, the present study aimed to evaluate the effect of aging on the optical properties, flexural strength and surface roughness of translucent and opaque yttria-stabilized zirconia ceramics. The null hypothesis was that aging would have no significant effect on the optical and mechanical properties of the two types of zirconia.

Materials and Methods

Specimen preparation:

In this in vitro experimental study, two types of specimens were designed and milled from the pre-sintered opaque and translucent yttria-stabilized zirconia blanks (Prattaue anterior Zirkonzahn, Bari, Italy) using a computer-aided design/computer aided manufacturing system (5X200, Arum 3D Dental Solutions, Doowon, Korea). For the BFS test, disc-shaped specimens were milled according to the ISO standard 6872 (25) with the final diameter and thickness of 12 and 1.2 mm, respectively (n=20 for each type of zirconia). For evaluation of the optical properties and surface roughness, the disc-shaped samples were milled to reduce the thickness to 0.5 (the diameter remained 12 mm; n=10 from each type of zirconia). The specimens were sintered according to the manufacturer's instructions (1450°C for 8 h) in a furnace (P300; Ivoclar-Vivadent, Schaan, Liechtenstein). The samples were polished with 180-, 600-, and 1200-grit silicon carbide papers (each paper was used for 5 min under water coolant). The specimens were then cleaned ultrasonically (Dentine XL, Faraz Mehr, Iran) in ethanol for 10 min. The diameter and thickness of each specimen were measured with a digital caliper for standardization (CD-6 ASX; Mitutoyo, Tokyo, Japan). Each sample was glazed using e.max Ceram glaze powder (B601380; Ivoclar-Vivadent, Schaan, Liechtenstein) and fired in a furnace at 900°C according to the manufacturer's instructions.

Aging procedure:

The specimens were placed in autoclave-safe bags and placed in an autoclave (AC-300DD; SAZGAR Co., Tehran, Iran) at 134°C and 2-2.3 bar pressure for 10 h according to Hallman et al. (19)

BFS test:

The test was performed on specimens according to ISO 6872. (25) The specimens were positioned concentrically on supporting steel balls that were 120° apart and equidistant from each other in a triangular position. The load was applied by a flat rounded tungsten piston at a cross-head speed of 1 mm/min until catastrophic failure occurred. The failure loads were recorded, and the BFS was calculated and reported in megapascals (MPa). The BFS test was performed before and after aging. BFS was calculated according to the formula:

$$S = -0.2387 \frac{P(X-Y)}{d^2}$$

Where S is the maximum tensile stress in pascals, P is the total load causing fracture in Newtons (N), and d is the specimen thickness at the fracture site in millimeters. X and Y were determined as follows:

$$X = (1+v)\ln\left({}^{r2}/_{r3}\right)^2 + \left(\frac{1-v}{2}\right)\left({}^{r2}/_{r3}\right)^2$$

$$Y = (1+v)\left[1+\ln\left({}^{r1}/_{r3}\right)^2\right] + (1-v)\left({}^{r1}/_{r3}\right)^2$$

in which v is the Poisson's ratio. If the value for the respective ceramic is not known, a Poisson's ratio=0.25 is used; r1 is the radius of the support circle in millimeters; r2 is the radius of the loaded area in millimeters; r3 is the radius of specimen in millimeters; d is the specimen thickness at the fracture site in millimetres (20).

Surface roughness:

The surface roughness was evaluated according to ISO 1997. (26) The Ra and Rz parameters were recorded. Three measurements were performed for each specimen before and after aging with a calibrated profilometer (TR200;

SaluTron Messtechnik GmbH, Frechen, Germany), and the mean *Ra* and *Rz* values were calculated.

Measurement of optical properties:

A spectrophotometer (X-RITE Ci6X) was used to measure the CIE L^*, a^*, b^* coordinates for each sample against black and white backgrounds at three different points. The measurements were made before and after aging. In the present study, white and black materials used as background were glazed papers. Next, CR, TP, and opalescence parameter (OP) were calculated.

Spectrophotometry:

The same spectrophotometer was used to measure the CIE $L^*a^*b^*$ values for the background materials and all tested specimens. The CIE $L^*a^*b^*$ coordinates of the background materials were measured and found to be as follows: white background (CIE $L^* = 87.94$, $a^* = -0.55$, and $b^* = 3.62$) and black background (CIE $L^* = 25.68$, $a^* = -0.10$, and $b^* = -0.41$).

TP:

For each specimen, the TP was calculated according to the following equation: (15)

$$TP=\sqrt{(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2}$$

where the subscripted B and W indicate the color coordinates of the sample against the black and white backgrounds, respectively.

CR:

Y (luminance from Tristimulus Color Space/ XYZ) was calculated using the L^* values (15) and was recorded against the black and white backgrounds, respectively. The CR values were calculated according to the following formula: (15)

$$CR=\frac{Y_b}{Y_w}$$

OP:

OP was calculated using the a* and b* coordinates of each sample against the black (subscripted "B") and white (subscripted "W") backgrounds, according to the following equation: (15)

$$OP=\sqrt{(a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2}$$

Color change before and after aging:

To calculate the color change (ΔE) after aging, ΔE values of non-aged and aged samples were calculated according to the following formula: (15)

$$\Delta E_{ab}^* = \sqrt{(L_N^* - L_A^*)^2 + (a_N^* - a_A^*)^2 + (b_N^* - b_A^*)^2}$$

Where the subscripted N and A refer to the non-aged and aged samples, respectively. The threshold of perceptible ΔE is 1.2, and the threshold of acceptable ΔE is 2.7. (15)

Statistical analysis:

We used descriptive statistics, including tables, charts, and central measures of dispersion to present the data. The normality of data distribution was assessed by performing the one-sample Kolmogorov-Smirnov test. To analyze the effect of aging and zirconia type on BFA, two-way ANOVA was used. To analyze the effect of aging and zirconia type on Ra, Rz, CR, and optical properties, two-way repeated measures ANOVA was applied with aging as the within-subject factor and the type of zirconia specimen as the between-subject factor. To compare the color difference against the white background (ΔEW) between the two zirconia types considering the normal distribution of data in the two groups, independent t-test was used, and for comparison of the color difference against the black background (ΔEB) between the two groups, the Mann-Whitney test was applied. Within-group comparisons were done using the paired-sample t-test. SPSS version 22 was used to perform the analyses. The type one error of the tests was set at $\alpha=0.05$.

Results

Surface roughness (Ra and Rz):

Table 1 shows the Ra and Rz values for the two types of zirconia specimens before and after aging. The results of two-way repeated measures ANOVA showed that aging had significant effects on Ra and Rz, and mean values of Ra and Rz significantly decreased after aging ($P=0.023$ and $P=0.011$, respectively), but the difference between the two types of zirconia specimens was not statistically significant ($P=0.339$ and $P=0.522$, respectively). Also, the

Table 1: Ra and Rz values in two types of zirconia specimens before and after aging

Zirconia		Ra before aging	Rz before aging	Ra after aging	Rz after aging
Opaque	Mean	1.0508	4.9379	.6428	2.9440
	Std. deviation	.15667	.83674	.08235	.48120
	Minimum	.46	2.29	.33	1.52
	Maximum	1.98	10.52	1.03	5.87
Translucent	Mean	.8186	4.2502	.6752	2.9745
	Std. deviation	.07998	.40527	.08706	.26773
	Minimum	.47	2.64	.24	1.58
	Maximum	1.20	6.63	1.17	4.03

pattern of decrease in *Ra* and *Rz* was similar in both types of zirconia (P=0.248 and P=0.540, respectively). In other words, the interaction effect of aging and type of zirconia was not significant (P>0.05).

TP:

Table 2 shows the TP values of the two types of zirconia before and after aging. The results of two-way repeated measures ANOVA showed that aging had no statistically significant effect on TP (P=0.384), but the two types of zirconia had significantly different mean TP values (P=0.046). The translucent zirconia specimens had greater mean TP values than the opaque zirconia specimens. The interaction effect of aging and type of zirconia on this variable was not significant (P=0.199).

CR:

Table 3 presents the CR of the two types of zirconia before and after aging. Repeated measures ANOVA showed that aging had no statistically significant effect on CR (P=0.261). The CR values were not significantly different between the two types of zirconia (P=0.055), and the interaction effect of aging and type of zirconia was not significant either (P=0.255).

OP:

Table 4 presents the OP of the two types of zirconia specimens before and after aging. Two-way repeated measures ANOVA followed by paired-sample t-test showed that the mean OP of translucent zirconia was not affected by aging (P=0.685), but aging significantly increased the OP of opaque zirconia (P=0.007).

Color change (ΔEW and ΔEB):

Table 5 shows the ΔE values of the two types of zirconia specimens after aging. T-test showed higher mean ΔEW in the translucent zirconia than opaque zirconia (P=0.044). Moreover, nonparametric Mann-Whitney test showed that the translucent zirconia had higher mean ΔEB than the opaque zirconia (P=0.019).

BFS:

Table 6 shows the BFS according to the aging status of the two types of zirconia. The results showed that aging had no statistically significant effect on BFS (P=0.912), while the BFS was significantly different between the two types of zirconia. The mean BFS values of the opaque zirconia specimens were higher than those of the translucent zirconia specimens (P<0.001). The interaction effect of aging and type of zirconia was not significant (P=0.495).

Discussion

The results of the present study showed that aging had significant effects on ΔE and OP. But other optical parameters and flexural strength were not affected. The surface roughness of both types of zirconia significantly decreased by

Table 2: TP of different types of zirconia specimens before and after aging

Zirconia		TP before aging	TP after aging
Opaque	Mean	11.6916	12.0582
	Std. deviation	.64355	.52522
	Minimum	8.09	10.37
	Maximum	14.78	14.54
Translucent	Mean	14.8643	13.2740
	Std. deviation	.11210	1.49088
	Minimum	14.36	.02
	Maximum	15.32	15.50

Table 3: CR of the two types of zirconia specimens before and after aging

Zirconia		CR before aging	CR after aging
Opaque	Mean	.7135	.7132
	Std. deviation	.01298	.01369
	Minimum	.65	.65
	Maximum	.75	.75
Translucent	Mean	.6438	.6831
	Std. deviation	.00223	.03562
	Minimum	.64	.63
	Maximum	.65	1.00

Table 4: OP of the two types of zirconia specimens before and after aging

Zirconia		OP before aging	OP after aging
Opaque	Mean	4.4935	4.6655
	Std. deviation	.09705	.05913
	Minimum	4.29	4.46
	Maximum	5.17	4.98
Translucent	Mean	4.9989	4.8280
	Std. deviation	.08465	.54370
	Minimum	4.68	.00
	Maximum	5.52	5.75

Table 5: Statistical indicators of ΔEB and ΔEW for two types of zirconia

Zirconia		ΔEB after	ΔEW after
Opaque	Mean	.3410	.6810
	Std. Error of Mean	.06742	.09554
	Minimum	.11	.38
	Maximum	.84	1.27
Translucent	Mean	2.7030	1.1090
	Std. Error of Mean	1.54049	.17294
	Minimum	.15	.49
	Maximum	14.95	1.94

ΔEB: Color change against a black background
ΔEW: Color change against a white background

Table 6: BFS of the two types of zirconia specimens before and after aging

Zirconia	Aging	Statistical features	BFS
Opaque	Before	Mean	660.9790
		Std. Error of Mean	37.37926
		Minimum	454.67
		Maximum	805.15
	After	Mean	688.3180
		Std. Error of Mean	. 46.67568
		Minimum	487.99
		Maximum	918.82
Translucent	Before	Mean	492.6890
		Std. Error of Mean	29.43332
		Minimum	354.91
		Maximum	690.23
	After	Mean	472.8590
		Std. Error of Mean	15.81544
		Minimum	396.43
		Maximum	537.46

aging. Therefore, the null hypothesis was partially rejected.

Aging did not affect the TP of the translucent or opaque zirconia. According to Kim et al, (28) aging for 10 h had no significant effect on TP, which is in agreement with the findings of the present study. In contrast, the results of Fathy et al. (1) showed that the translucency of zirconia decreased as a result of aging. They suggested that pore formation and the difference in the refractive index of the monolithic and tetragonal zirconia crystals could induce increased light scattering and thus, lead to a decrease in translucency. Since 1 h of aging is equivalent to 4 years of clinical use, (19) and increasing the aging time affects the translucency of zirconia, (27) the difference in the results between the studies could be due to the longer aging time in the study by Fathy et al. (1) Furthermore, Walczak et al. (30) analyzed the translucency of zirconia before and after aging. In contrast to the findings of the present study, their results showed that the translucency of zirconia decreased after aging. This discrepancy might be because they used a different brand of zirconia. Different zirconia materials require different sintering protocols. It has been hypothesized that different sintering conditions could affect the grain size, porosity, and other characteristics of zirconia materials, and consequently affect the translucency. (30-32) Also, Al_2O_3 doping has been shown to have a negative effect on the translucency of zirconia materials, (30,33) and different amounts of Al_2O_3 could cause different results between different zirconia materials. (39) Furthermore, the glazing performed in this study might contribute to insignificant changes in TP after aging. Manziuc et al. (34) evaluated the effect of glazing on the translucency of zirconia and demonstrated that the translucency of zirconia specimens remained unchanged after glazing. This could further explain the differences in the results between different studies, but further investigations are necessary to assess the effects of glazing on TP.

The results of the present study showed significant color change in both types of zirconia after aging. However, the color of translucent zirconia specimens changed to a greater extent than that of opaque zirconia. Both types of zirconia showed ΔE values lower than the maximum clinically acceptable value (2.7) (15).

However, ΔEB of the translucent zirconia was nearly 2.7, i.e., almost the same as the maximum clinically acceptable color change. Alghazzawi (15) reported that ΔE of zirconia increased with aging, which is in agreement with the findings of the present study. Pigment breakdown, burning of metal oxides, and phase transformation have been suggested as reasons for the increase in ΔE with aging. (20,35) Kim et al. (28) reported that changes in the color (especially changes in the b∗ value) occurred within the first hour of aging, after which color parameters became stable. In addition, they suggested that the colorants could affect the movements of oxygen vacancies, and consequently affect the zirconia color.

According to the findings of the present study, aging could not affect the CR. On the other hand, in the study by Alghazzawi, (15) aging decreased the CR of zirconia, i.e., the material became more opaque as the aging duration increased. However, the aging duration in their study was longer than that in the present study, which could account for the different results. As we used 10 h of aging, further investigations with a longer aging duration is necessary to confirm these results. Furthermore, the results of the present study showed that aging increased the OP only in opaque zirconia and had no effect on the translucent zirconia. However, according to Alghazzawi, (15) OP decreased due to aging, which is in contrast to the results of the present study. It should be noted that he used colored zirconia samples in his investigation, and this might have affected the optical properties of zirconia samples.

The results of the present study revealed that aging did not have a significant effect on the flexural strength of translucent and opaque zirconia. Similarly, several previous studies also reported that aging did not induce a significant change in the flexural strength of zirconia. (15,29,30) However, Tang et al. (24) reported an increase in flexural strength of zirconia frameworks after aging. Flinn et al. (9) reported that the flexural strength of zirconia decreased as a result of aging, which is attributed to an increase in the monoclinic phase volume and in the phase-transformation layer thickness. Such contradictory findings could be because of the use of zirconia specimens from different brands (9) and the adoption of different aging protocols. (36) Flinn et al, (9) also found that in the first 5 h of aging, the flexural strength did not change significantly. Thus, considering the aging time in the present study, this result is somewhat in agreement with the results of the present study. Furthermore, Camposilvan et al. (22) found that glazing decreased the effect of aging on zirconia. In other words, it protected the zirconia against the adverse effects of aging. Since all zirconia samples in the present study were glazed, it could be speculated that glazing protected the zirconia and thus, prevented significant change in the flexural strength. Moreover, in the present study, the flexural strength of opaque zirconia was found to be significantly higher than that of translucent zirconia. Therefore, it might be concluded that opaque zirconia exhibits greater clinical durability.

The findings of this study showed that aging decreased the surface roughness in both translucent and opaque zirconia. Tang et al, (24) also reported similar results. They suggested that Ra was influenced by the ceramic density and porosity, and that aging could increase the ceramic density and decrease the porosity, leading to a reduction in surface roughness. This decrease in the surface roughness of dental restorations in the oral cavity has positive effects such as prevention of plaque accumulation and bacterial contamination, and a reduction in wear and chipping of opposite teeth. (37) On the other hand, Alghazazawi (15) showed that the surface roughness increased with aging due to the phase transformation from the tetragonal to the monoclinic phase. They also suggested that the absence of cubic phase could increase the susceptibility to aging. In contrast, Yang et al. (36) reported that aging did not have a significant effect on surface roughness. Different aging protocols and measuring methods might be the reason for this difference in the results among the studies. According to the studies done by Manziuc et al, (34) and Haralur et al, (39) glazing decreased the surface roughness of dental ceramics, and consequently decreased plaque accumulation and improved biocompatibility. Alghazzawi (15) used unglazed samples, so glazing could be one of the factors that produced different results.

It seems that because of the decrease in surface roughness after aging, the flexural strength changes were not significant in this study. Hjerppe et al. (40) reported that surface roughness changes influenced the flexural strength. According to Tang et al, (24) an increase in surface roughness could help in distributing the stresses concentrated on samples and thus, increasing the flexural strength. This is in agreement with our hypothesis that a change in surface roughness could affect the flexural strength. However, further investigations are needed to prove this hypothesis.

Manziuc et al. (34) suggested that glazing might cause significant color change in zirconia restorations. However, as the optical properties were not analyzed before glazing, the corresponding effect of aging and glazing on the color change could not be identified in the present study and further research in this field is necessary. Also, in the present study, X-ray diffraction test was not performed and future studies should evaluate the structural differences between the translucent and opaque zirconia.

Conclusion

Within the limitations of this study, it can be concluded that:

1- Aging decreased the surface roughness of both translucent and opaque zirconia.

2- Aging increased the OP of opaque zirconia.

3- Aging caused color change in translucent and opaque zirconia. The color of the translucent zirconia changed by a greater extent than that of opaque zirconia.

4- Aging had no significant effect on the flexural strength of translucent or opaque zirconia.

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