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The Effect of Hydrothermal Aging on The Color Stability of Gradient Zirconia. In Vitro Study

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Aim: The study aims to assess the influence of hydrothermal aging on gradient zirconia's microstructure and color stability.
Materials and methods: Ten discs were constructed from gradient zirconia blanks (3Y-TZP-5Y-TZP) using CAD\CAM technology. Hydrothermal aging was done in an autoclave. Color parameters (a*, b*, L*) for each disc was measured using a spectrophotometer. In order to investigate the zirconia specimens' crystalline structure, X-ray diffraction (XRD) was performed. Color stability and microstructural changes were examined before and after the aging process.
Results: Following hydrothermal aging, a statistically significant decrease in the mean (L*) value was found. However, following hydrothermal aging, a statistically significant increase in the mean (a*) and (b*) values was reported. Regarding the microstructure, hydrothermal aging did not result in any significant changes.
Conclusion: Artificial aging has led to color changes in gradient zirconia that were in the clinical acceptability range but has no impact on the microstructure of gradient zirconia.

Keywords: Artificial accelerated aging, Color stability, Gradient zirconia, Microstructure, Monolithic restorations

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Introduction

Zirconia restorations are now often utilized in dental practices. Because of its tremendous strength, which is comparable to metal's strength, it gradually substituted porcelain fused to metal restorations. Zirconia's first generation is 3 mol% yttria-stabilized tetragonal zirconia polycrystalline ceramic (3Y-TZP). (3Y-TZP) has superior strength and fracture toughness.[1] On the other hand, one of the disadvantages of (3Y-TZP) is the optical properties as it is opaque. For enhancing the optical characteristics, (3Y-TZP) was used as coping and then veneered with translucent porcelain. The main disadvantage of bi-layered zirconia restorations is chipping of the brittle veneering ceramic.[2,3]

For that reason, monolithic zirconia restorations have drawn interest as a potential solution to the chipping problem. Furthermore, the avoidance of significant tooth preparation is another benefit of monolithic restorations. Several modifications have been made to use monolithic restorations with proper esthetics. The increase of yttria from 3 mol% to 4 and 5 mol% accomplishes this. Translucency has been successfully increased. On the other hand, mechanical characteristics deteriorated due to the lack of transformation toughening. Translucent zirconia should therefore only be used in areas of the oral cavity which undergo less stress. Subsequently to allow the use of 5Y-PSZ materials successfully, their strength should be increased.[4]

Recently, Gradient zirconia has been introduced to the market. To benefit from both, it integrates zirconia from generations 3Y-TZP and 5Y-TZP into a single blank. The higher yttrium content in zirconia, the more translucency and less flexural strength. As a result, 3Y-TZP is utilized on the body part that requires greater strength. However, because 5Y-TZP is more translucent, it is employed in the incisal area for improved aesthetics.[5]

Generally, the color of zirconia is affected by many factors. Among these factors are the number of firings, ceramic thickness, cement shade, abutment color and sintering parameters.[1] To ensure the success of ceramic restorations over many years, the color stability should be granted and maintained. Nonetheless, a variety of factors affect their optical characteristics. For instance, finishing and polishing, surface treatments and aging. The optical characteristics of zirconia are negatively affected after aging due to changes in the crystalline structure.[6]

As a polymorphic material, zirconia can exist in three phases: cubic, tetragonal, and monoclinic. Between the room temperature and 1170°C, monoclinic phase (m) is stable. At this temperature, the tetragonal phase (t), which is stable between 1170°C and 2370°C, transforms from the monoclinic phase. It then changes into the cubic phase (c), which is stable above 2370°C, after this.[7] At room temperature, monoclinic is the only stable phase. The primary issue is that the monoclinic phase's mechanical and optical properties are insufficient to be used in the oral cavity. Stabilizing the cubic and tetragonal phases is crucial because of this. Yttrium oxide (Y2O3) and other stabilizing oxides can be utilized for this stabilization. When zirconia is subjected to stress, tetragonal→monoclinic (t→m) phase transformation occurs. A volumetric expansion occurs concurrently with this phase transformation. This expansion may prevent a crack from propagation.[8]

Under moist conditions, t→m phase transformation can occur. This is known as low temperature degradation (LTD). LTD occurs spontaneously and the presence of water at room temperature will lead to it's aggravation. The transformation to

monoclinic phase will negatively affect optical characteristics as monoclinic are bulkier than tetragonal. This will eventually result in an increase in the restoration's surface roughness thus the color stability will be affected.[6] Several attempts were done to overcome the LTD phenomenon in zirconia. These include adding aluminum oxide, adding more stabilizing oxides, or reducing the particle size. Also, less aging susceptibility can be achieved by increasing the cubic phase. Since this phase is stable, it does not undergo transformation.[9]

Numerous investigations were conducted to assess how various aging methods influenced the zirconia's optical characteristics of zirconia. Alghazzawi[10] assessed the impact of LTD on the optical characteristics. It included seven different zirconia brands (containing 3% and 5% yttrium oxide). Where the control group was lithium disilicate. Ten samples from each brand were subjected to aging using autoclave. A 2.1 bar pressure at 134°C was applied for 100 hours at various intervals. They found that aging had an impact on the optical characteristics of both zirconia and e-max, and that the changes differed between the various zirconia brands.

Other researchers[11] assessed how aging and varying core thicknesses affected the color stability of several ceramic materials. The aging was conducted in weathering machine for 200 hours. After aging, changes in color were noticed. Other aging methods such as ultraviolet and gamma irradiation were evaluated and a change of zirconia color was found.[12]

Several studies assessed how hydrothermal aging affected the optical characteristics of various generations of zirconia. Gradient zirconia's color stability after exposure to aging has not yet been assessed, nevertheless. So, this study aims to assess the influence of hydrothermal aging on gradient zirconia's microstructure and color stability. The study's null hypothesis states that there will be no difference in gradient zirconia's color and microstructure before and after hydrothermal aging.

Materials and Methods

sample size calculation

This power analysis used color change (ΔE) before and after aging as the primary outcome. Based upon the results of Abdel Hamid T et al[13]; the effect size for paired t-test was dz = 2.98. Using alpha (α) level of (5%) and Beta (β) level of (20%) i.e., power = 80%; the minimum sample size was four specimens. Sample size was increased to 5 specimens per group to compensate for the use of non-parametric tests. For reliability we will increase the sample size into 10 specimens. Sample size calculation was performed using G*Power Version3.1.9.2.

Sample Fabrication

Construction of Gradient zirconia discs:

Ten Gradient zirconia disc specimens were milled from IPS e.max ZirCAD Prime block (Ivoclar Vivadent, Liechtenstein) using 5-axis milling machine (Roland DWX 50, California, USA). VHF 0.6mm, 1mm and 2mm burs were used for dry milling. After milling of the discs, a bur mounted on straight handpiece was used to detach the discs from the blank remaining and smooth surfaces were obtained.

Zirconia sintering:

All zirconia disc specimens (n=10) were sintered following the manufacturer's recommendations which provide sintering chart instructions using Zubler sintering oven (VARIO S400 Zubler sintering oven, USA). In the sintering oven, the increase in temperature from 25°C to 1200°C was set to occur at a rate of 15°C per minute, where the temperature remains constant for 60 minutes.

Then raising the temperature to 1300°C at 2°C /minute rate was done and raising to 1530°C at a rate of 10°C /minute was done

again where it remained constant for 150 minutes.

A long cooling cycle was employed, which allowed the temperature to drop from 1530°C to 155°C at a rate of 15°C /minute. After being taken out of the furnace, the disc specimens were left to bench cool until they reached room temperature.

Zirconia polishing

In order to standardize the pressure and direction of polishing, a straight hand piece placed on a surveyor was used. Polishing of the discs was carried out using 2 instruments from the polishing kit (Eve Diacera Diasynt plus zirconia polishers eva ernst vetter Gmbh, pforzheim, Germany). Two steps were performed, first of which a pre-polishing disc was used by using a handpiece for 60 seconds at speed of 10,000 rpm. The second step was done using H8DC at 90 degrees angle for a total of 2 minutes, 1 minute for each polisher.

Zirconia glazing

The discs surfaces were coated by a thin layer of clear glaze CERABIEN™ ZR External Stain (clear glaze kuraray noritake, Tokyo, Japan). Discs were then placed in SUMMIT's Porcelain furnace (IBEX Dental technologies, Texas, USA). The manufacturer's recommended firing parameters were followed. The temperature was adjusted to 600°C for five minutes, with a heating rate of 42°C per minute, and it was kept at 840°C for four minutes.

Accelerated Hydrothermal aging:

The disc specimens were numbered and individually placed in sterilization pouches and then the pouches were sealed and placed in a steam autoclave (SUN Class B Autoclave, LyncMed Medical Technology Beijing, China). Aging was carried out at 134ºC, 2 bar pressure for 5 hours according to ISO standards 13356:2015. Where it was reported that zirconia aging in an autoclave at 134 °C for 1 h simulates 3 to 4 years of zirconia aging in the oral cavity. [14]

Testing procedures:

All samples were tested twice; the first time before aging and the second time after aging. They were subjected to the following tests:

- Color stability measurements (ΔE) using a spectrophotometer.
- Microstructural changes using X-ray diffraction analysis.

Color Stability Measurement:

Using an Agilent Cary 5000 UV-Vis-NIR USA spectrophotometer, the samples' initial colors were assessed. The samples were inserted into the device at an angle of 10 degrees after the aperture size was adjusted to 5 mm. According to the CIE L*a*b* color system, the measurements of color were done.

After the samples being artificially aged in an autoclave for 5h, color measurements were taken again by the same spectrophotometer Cary 5000©. In each measurement, recording the values of CIE L*a*b* was done. ΔE units is used to represent the amount of difference in color between the colors being compared. The following formula is used to get the total color difference using the coordinates L*, a*, and b*:[15,16]

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

Where the difference in the parameters of color of the two colors before and after hydrothermal aging is represented by ΔL*, Δa*, and Δb* coordinates.[16,17]

X- ray diffraction analysis (XRD):

Before and after aging, XRD was used to identify the crystalline phases present in gradient Zirconia disc specimens. The diffractometer (Xpert pro, PANalytical, Malvern, Worcestershire, UK) used for the XRD analysis was set up with the following settings: scan range 0–90 (2h), step size 0.02, scan time per step 0.6s, and Cr Ka radiation (40 mA, 40 kV). By utilizing the Gravie-Nicholson equation, the percentage of t-m transformation was determined.[18]

$$Xm = Im(-1,1,1) + Im(1,1,1) / Im(-1,1,1) + Im(1,1,1) + It(1,0,1)$$

Results

Regarding color stability:

Following hydrothermal aging, there was a statistically significant decrease in the mean (L*) value. Table (1) displays the mean, standard deviation (SD) values, and the outcomes of the paired t-test used to compare the (L*) values before and after hydrothermal aging. While there was a statistically significant increase in mean (a*) and in mean (b*) values after hydrothermal aging Table (2) & (3) respectively.

The values of the mean and standard deviation (SD) for color change (ΔE) were 2.97 (0.17) with a minimum of 2.59 and a maximum of 3.19.

Table 1 - Mean, standard deviation (SD) values and outcomes of paired t-test used to compare (L*) values before and after hydrothermal aging.

Before hydrothermal aging (n =10)		After hydrothermal aging (n =10)		Change (ΔL)		*P*-value	Effect size (d)
Mean	SD	Mean	SD	Mean	SD		
74.78	0.31	73.1	0.57	-1.68	0.31	<0.001*	1.965

***: Significant at P ≤ 0.05**

Table 2 - Mean, standard deviation (SD) values and outcomes of paired t-test used to compare (a*) values before and after hydrothermal aging.

Before hydrothermal aging (n =10)		After hydrothermal aging (n =10)		Change (Δa)		*P*-value	*Effect size (d)*
Mean	SD	Mean	SD	Mean	SD		
0.9	0.1	1.76	0.12	0.86	0.05	<0.001*	7.339

***: Significant at P ≤ 0.05**

Table 3 - Mean, standard deviation (SD) values and outcomes of paired t-test used to compare (b*) values before and after hydrothermal aging.

Before hydrothermal aging (n =10)		After hydrothermal aging (n =10)		Change (Δa)		*P*-value	*Effect size (d)*
Mean	SD	Mean	SD	Mean	SD		
13.53	0.18	15.81	0.28	2.28	0.15	<0.001*	7.79

***: Significant at P ≤ 0.05**

Regarding the microstructure:

The value of the microstructure after hydrothermal aging showed no statistically significant change. Table (4) displays the values of the mean, standard deviation (SD) and the outcomes of paired t-test used to compare the values of the microstructure (Xm) before and after hydrothermal aging.

Table 4 - Mean, standard deviation (SD) values and outcomes of paired t-test used to compare microstructure values (Xm) before and after hydrothermal aging.

Before hydrothermal aging (n =10)		After hydrothermal aging (n =10)		Change		*P*-value	*Effect size (d)*
Mean	SD	Mean	SD	Mean	SD		
0.558	0.09	0.578	0.065	0.02	0.118	0.613	0.217

***: Significant at P ≤ 0.05**

XRD Analysis

At 2θ angle of 30.3, tetragonal peak was recorded. No monoclinic peaks were recorded before aging Figure (1). While after aging, a small monoclinic peak was recorded at 2θ angle of 28.8 indicating minimal transformation due to aging. The quantity of every phase was measured by counts recorded for this phase Figure (2).

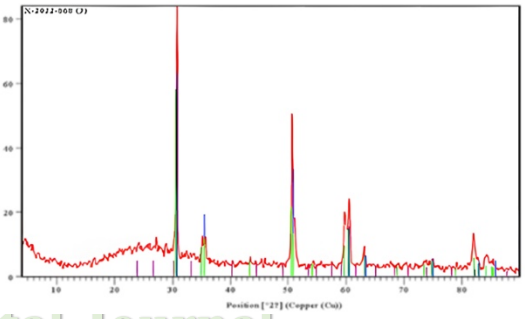

Figure 1 - XRD analysis before aging. At 2θ angle of 30.3, tetragonal Zirconia peak was recorded.

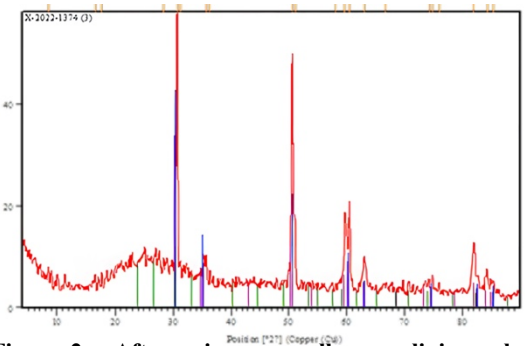

Figure 2 - After aging, a small monoclinic peak was recorded at 2θ angle of 28.8 indicating minimal transformation due to aging.

Discussion

Gradient zirconia was introduced into the market to improve the esthetics of traditional opaque zirconia so that monolithic restorations can be done with it. Oral cavity restorations are exposed to a variety of environmental changes, including variations in temperature, pH, and mechanical stresses that cause t-m phase transformation. This phase transformation may have an influence on the long-term success of the restoration in the oral cavity.[19]

Accelerated hydrothermal aging of gradient zirconia disc specimens was done in an autoclave, as autoclave is considered the standard method for artificial aging. Aging was done for 5 hours at 134ºC under 2 bar pressure. This aging protocol was done according to the approved protocol for aging stated by the ISO (ISO 13356:2015).[20] Chevalier et al.[21] reported that zirconia aging in an autoclave at 134 °C for 1 h simulates 3 to 4 years of zirconia aging in the oral cavity.

In the current study, gradient zirconia's color was measured instrumentally using a Cary 5000 spectrophotometer, both before and after aging. Visual color measurement was not used as it is a subjective and unreliable method and errors may occur.[22] Since spectrophotometers are thought to be the most flexible and sophisticated tools for shade matching, they were chosen over colorimeters. Also, they provide accurate results.[23]

Quantitative changes in microstructure were analyzed by PAN analytical diffractometer using Garvie Nicholson equation which determines the percentage of transformation of tetragonal to monoclinic phases. XRD was used as it is considered a non-destructive reliable method in evaluating the surface structural changes and characterization resulting from aging in zirconia.[24]

As gradient zirconia is a new material and by reviewing the literature, many studies have assessed the impact of aging on the optical characteristics of various zirconia types. However, the stability of color of gradient zirconia after exposure to aging in an autoclave is not yet evaluated.[25] Thus, this study aims to assess the influence of aging on gradient zirconia's microstructure and color stability.

In accordance with the findings of the present study, the null hypothesis was partially rejected as the color of gradient zirconia showed a statistically significant difference before and after hydrothermal aging, while the zirconia's microstructure both before and after hydrothermal aging did not differ significantly.

Regarding the clinically acceptable values of color changes, there is some controversy in literature.[26,27] In the present study, the mean values of the differences in color (ΔE) of gradient zirconia discs were calculated and compared to many studies. In which, the clinically acceptable perceivable threshold of ΔE values range between 1 and 3.33 units and unacceptable threshold of ΔE more than 3.33 units.[27]

Based on the results of the present study, the color change (ΔE) of the gradient zirconia was 2.97 which is clinically acceptable (ΔE < 3.33) and within the clinically perceivable threshold.[26] In the current study, (L*) value decreased after aging in autoclave, which indicates loss of brightness. While (a*) value increased, which indicates that gradient zirconia specimens became more reddish. Also, (b*) value increased, which indicates gradient zirconia specimens became more yellowish. These results agreed with Douglas et al.[28] who found that after aging, all tested materials became darker and more red and yellow. The reason behind this color change was clarified by Kurt et al.[29] They concluded that there is a direct relation between aging process and changes in color of monolithic zirconia. Their explanation for this color change was due to

the direct exposure of the surface of monolithic zirconia to water i.e. saliva which lead to LTD of the surface crystals of the material. This is followed by expansion of the grains of the monoclinic phase thus resulting in microcracks and increased surface roughness and subsequently color change.

In the intraoral environment, monolithic restorations are in a direct exposure to any fluctuations as they are not protected by a veneering ceramic layer. Thus, the exposure to any changes in the intraoral environment will directly trigger the LTD phenomenon to occur. Also, in the monolithic zirconia, the amount of the alumina content is reduced to render more translucency in the restorations. However, the alumina plays an important role in the resistance of the LTD phenomenon. For that reason, monolithic zirconia restorations may be more affected by LTD.[29]

The findings of this study were consistent with those of earlier investigations.; Alghazzawi et al.[30] Concluded that artificial aging of zirconia affects its optical characteristics, when the time increased the effects also increased and the amount of change was affected among the different brands of zirconia. They explained this color change as the result of the coloring metallic oxides burning or the pigment breaking down after repeated heat exposure. Furthermore, Zhang et al.[32] discovered that between the aged and non-aged zirconia, the a∗ values differed significantly. They found increased in the a∗ values (more reddish) after aging Y-TZP ceramics. The authors attributed the color instability of the samples due to pigment breakdown of the coloring pigments in Y-TZP ceramics due to thermal conditions.

The current study's findings were in disagreement with those of Hamza et al.[17] In which evaluation of the color stability of three CAD/CAM materials after aging was done. It was claimed that aging did not have an impact on the color of the three tested groups. The stability in color of the zirconia specimens may be resulted from differences in the design of the study, measurement methods or equipment from the current study. In their study aging was done for 300h in a Weather-O-meter, while in the current study aging was done in a stem autoclave for 5 h. Other studies[31,32] have reported that after artificial accelerated aging Y-TZP ceramics are stable in color. Also, these results differed from the current study as aging in these studies was done by thermocycling or placing the specimens in a dry oven for 5 to 40 h at 134°C temperature and 2 bar pressure.

The microstructure of Zirconia was quantitatively assessed by XRD. Before aging the XRD pattern showed no monoclinic peaks. While after aging minimal peaks of monoclinic phase appeared. The results agreed with Elsheemy et al.[33] and Amarante et al.[34] They reported that the XRD pattern did not exhibit a monoclinic phase after aging. They suggested that this could be because the zirconia specimen's examined part was not yet affected by LTD, or it could be because the t-m transformation was insufficient to show up in the XRD.

The findings of this study disagreed with previous study done by Borchers et al.[35] They found significant t-m phase transformation occurred. This may be due to difference in the aging conditions between the two studies. In their study, aging condition was carried out in water for 64 days at 80ºC and 8h in autoclave at 134 ºC. While, in the present study aging condition was carried out at 134ºC, 2 bar pressure for 5 hours. Which suggests that significant t-m phase transformation may have occurred due to aging in an autoclave using higher temperatures or prolonged aging time.[36]

Another explanation for the absence of significant t-m phase transformation after aging in the present study is the dry milling system used for gradient zirconia. According

to Kamel et al.[18] They found that after aging procedure in wet milling system, peaks of monoclinic phase appeared significantly. While, minimal peaks of monoclinic phase appeared in dry milling system. They attributed this finding due to absence of water during milling as they assumed that the machining of the pre-sintered Y-TZP blocks by CAD/CAM induces voids at the surface of zirconia. These voids cannot be removed completely by the process of sintering. The voids act as sites for stress concentration in the presence of water and by the migration of water into the lattice of zirconia, these voids will grow thus will start the tetragonal to monoclinic phase transformation.[37]

The limitations of this study include that not all the intraoral aging conditions could be replicated. Further clinical and laboratory studies are recommended to test the stability in color and microstructural changes of gradient zirconia using different aging methods (e.g. weathering machine or mechanical aging) or aging in autoclave for longer duration.

Conclusion

Within the limitations of this study, the ensuing conclusions were drawn:

1. Gradient zirconia showed clinically acceptable changes in color after aging (ΔE <3.33).
2. Artificial accelerated aging has no impact on the microstructure of gradient zirconia.

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