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ORIGINAL

Effect of hydrothermal aging on the optical properties of monolithic zirconia ceramics

Efecto del envejecimiento hidrotérmico sobre las propiedades ópticas de las cerámicas monolíticas de zirconio

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Abstract

Background/Purpose: Monolithic zirconia which have good optical properties and excellent mechanical properties such as flexural strength and fracture resistance. This study aimed to investigate the effect of hydrothermal aging on the optical properties of the monolithic zirconia.

Materials and methods: In this *in vitro* study, some groups of all-ceramic restorations were examined, of which monolithic zirconia with high translucency was used as the restorative material and three materials, grade 5 titanium and white and yellow zirconia, as substructure materials. Color standard characteristics, *l*, *a* and *b* values, were measured and recorded by a digital spectrophotometer before and after the aging process. Then, the mean values of data were compared by variance analysis.

Results: The statistical results show that the difference in mean values of color changes (ΔE) between the titanium group and yellow zirconia group was statistically significant. However, no significant difference was observed between titanium and white zirconia groups and between white zirconia and yellow zirconia groups. In all the groups, the differences in the mean ΔE in various thicknesses were significant. The differences between the mean *l*, *a*, and *b* indices were significant in all the groups before and after the aging process.

Conclusion: The color changes associated with the aging process were higher in the yellow zirconia group than in the other two groups. The *l* and ΔE indices decreased, and the *a* and *b* values increased due to the aging process. Restorations with the thickness of 1.5-mm and above exhibited greater color stability after aging.

Key words: Hydrothermal aging, optical properties, zirconia ceramics.

Resumen

Antecedentes/Propósito: La zirconia monolítica que tiene buenas propiedades ópticas y excelentes propiedades mecánicas como la resistencia a la flexión y a la fractura. Este estudio tiene como objetivo investigar el efecto del envejecimiento hidrotérmico en las propiedades ópticas de la zirconia monolítica.

Materiales y métodos: En este estudio in vitro, se examinaron algunos grupos de restauraciones de cerámica sin metal, de los cuales se utilizó circonio monolítico de alta translucidez como material de restauración y tres materiales, titanio de grado 5 y circonio blanco y amarillo, como materiales de subestructura. Se midieron las características de color estándar, los valores l, a y b, y se registraron con un espectrofotómetro digital antes y después del proceso de envejecimiento. A continuación, se compararon los valores medios de los datos mediante un análisis de varianza.

Resultados: Los resultados estadísticos muestran que la diferencia en los valores medios de los cambios de color (ΔE) entre el grupo de titanio y el grupo de circonio amarillo fue estadísticamente significativa. Sin embargo, no se observó ninguna diferencia significativa entre los grupos de titanio y de circonio blanco y entre los grupos de circonio blanco y de circonio amarillo. En todos los grupos, las diferencias en la media de ΔE en varios espesores fueron significativas. Las diferencias entre los índices medios l, a y b fueron significativas en todos los grupos antes y después del proceso de envejecimiento.

Conclusiones: Los cambios de color asociados al proceso de envejecimiento fueron mayores en el grupo de circonio amarillo que en los otros dos grupos. Los índices l y ΔE disminuyeron, y los valores a y b aumentaron debido al proceso de envejecimiento. Las restauraciones con un grosor de 1,5 mm y superior mostraron una mayor estabilidad del color tras el envejecimiento.

Palabras clave: Envejecimiento hidrotérmico, propiedades ópticas, cerámica de circonio.

Introduction

In recent years, dental ceramics have become an increasingly essential materials in tooth restoration[1]. The fabrication of anterior restorations with natural colors and appearances is an essential process in dentistry that is particularly important for patients[2].Metal-ceramic restorations with high fracture strength are successfully used in dentistry[3,4]. Obtaining natural translucency in metal-ceramic restorations is more difficult than that in all-ceramic restorations due to the opaqueness of metal substructure[5,6]. The materials of choice in modern dental ceramic are feldspathic porcelain, leucite-based material, lithium disilicate, and zirconia[7]. Among all-ceramic restorations, zirconia restorations have exhibited favorable physiological and mechanical properties[8,9]. However, despite advances in the esthetics and strength of ceramic materials, the color harmony between ceramics and natural teeth is still not easy to achieve, and it remains a challenge[10].

Manufacturers have been striving to improve the optical properties of all-ceramic materials for better aesthetic outcomes[11]. The color of zirconia can be affected by various factors, substructure color, cement type, zirconia type, zirconia thickness, staining process, sintering conditions, and aging process[11-18]. Although translucent zirconia has been introduced in dentistry, but the color matching of restoration with natural teeth is a problem. In clinical situations, when the tooth of patient is discolored or restored with opaque or costume post-core materials, the restorative material should be able to mask the underlying color of restoration to achieve esthetic results. Therefore, the high translucency of ceramics is not always an advantage[19-20]. The minimum thickness of monolithic zirconia for sufficient fracture resistance is recommended 0.5 mm, but there is no consensus on the minimum thickness of zirconia to provide the esthetic needs[21-24].

Studies have shown that when the surface of zirconia is exposed to water or water vapor, it undergoes a slow, spontaneous, and progressive phase transition. This phenomenon is known as the aging process or low-temperature degradation (LTD). This is one of the disadvantages of zirconia because, after the phase transition from tetragonal to monolithic phase, the surface becomes rough, and a successive displacement of particles with the subsequent micro-crack of cores is observed[25]. One of major research interest is evaluating the mechanical behavior of zirconia after the aging process in a humid environment. Many variables are contributed in the response of zirconia to the aging process, particle size, zirconia properties, and quality, distribution of stabilizing oxides, time, and atmospheric conditions during the sintering process[25]. Clinical and laboratory care are needed to maintain the integrity of the zirconia surface to minimize the effects of the aging process.

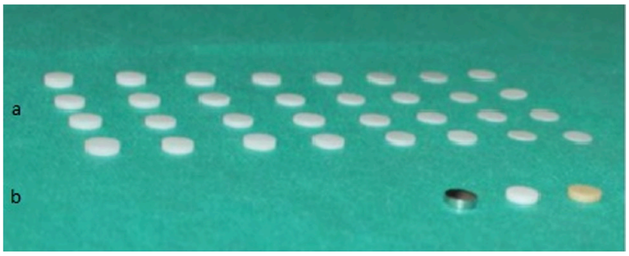
Today, ceramic systems have been introduced to improve the adaptation and color stability of restorations[26-28]. Monolithic zirconia restorations are a good choice in cosmetic dentistry because, in addition to good optical properties, they have excellent mechanical properties such as flexural strength and fracture resistance[29,30-32]. However, achieving the ideal esthetic outcome with these restorations remains a challenge[31-33].

This study aimed to investigate the effect of hydrothermal aging on the optical properties of the all-ceramic restorations. The null hypothesis of the study was that the aging process does not affect the final color of the restorations in different thicknesses of zirconia and on different sub-structures.

Materials and methods

In this experimental study, monolithic zirconia with high translucency (DD cubeX2; Dental Direkt GmbH, Germany) was used as the restorative material, and three materials; grade 5 titanium, white zirconia, and yellow zirconia (IPS e.max ZirCAD; Ivoclar Vivadent AG), were used as the substructure (**Figure 1**).

Fiogure 1: a. Translucent monolithic zirconia disks in different thicknesses, b. Titanium, white zirconia and yellow zirconia disks.



The samples number were considered at n=4 in each group and thickness, based on the study of Jirajariyavej B et al., using G-power software with $\alpha = 0.05$ and 90% test power[34]. High translucency monolithic zirconia was considered for the disks material, and they were fabricated by CAD/CAM method (Coritec 250i; imes-icore GmbH, Eiterfeld, Germany). The disks' diameter was considered 10 mm with the thickness of 0.5, 0.7, 0.9, 1.1, 1.3, 1.5, 1.7, and 1.9 mm, and for each thickness, four disks were prepared.34 Then the samples were immersed for 5 seconds in the paint liquid (DD Bio ZX2 monolithic zero A2 LZDD; Dental Direkt GmbH, Sponge, Germany).

The colored disks were dried with an ordinary bulb for 45 minutes, then the samples were sintered at a temperature of 1.520°C for 12 hours in a sintering furnace (iSINT HT;

imesicore GmbH), and their both sides were equalized by a polishing kit (BruxZir; Glidewell Direct, Irvine, CA, USA). The thickness of the samples was measured by a micrometer with the accuracy of ±0/02 mm (293 MDC-MX Lite; Mitutoyo Corp, Kawasaki, Japan).

A titanium disk with the thickness of 2 mm was prepared by lath as the substructure material, and 2-mm-thick disks were made of white and yellow zirconia, white shade MO 0 as white zirconia and yellow shade MO 2 as yellow zirconia, by the CAD/CAM technique. IPS e.max ZirCAD was crystallized according to the manufacturer's instructions in the furnace (inFire HTC Speed Dentsply Sirona). All the samples were cleaned in an ultrasonic bath (Elmasonic S-30; Dentec, North Shore, Australia), contains 98% ethanol for 20 minutes, and dried in an oil-free air current.

The samples were divided into three groups, all-ceramic restorations on a titanium substructure (Ti), all-ceramic restorations on a white zirconia substructure (W-ZrO_2), and all-ceramic restorations on a yellow zirconia substructure (Y-ZrO_2). The zirconia disks were placed on the three substructures, and a drop of glycerin was used at the interfaces to make proper contact. *l*, *a*, and *b* parameters in CIElab system were determined for zirconia samples in different thicknesses using a digital spectrophotometer (Ultrascan XE; Hunter Associates Laboratory, Inc.), To investigate the optical properties. The total color changes of the samples (ΔE) were calculated based on the changes of *l*, *a*, *b* according to the following formula:

$$\Delta E = [(\Delta a)^2 + (\Delta b)^2 + (\Delta l)^2]^{½}$$

Before the measurement procedure, the spectrophotometer was calibrated according to the manufacturer's instructions. For more accuracy, the color of all the samples was measured three times in the condition that the spectrophotometer tip was placed at the center of the sample.

After the first series of measurements, all the disks underwent the same aging process. All the samples were placed in a thermocycling device (PLC) containing two tanks, cool and hot water baths with temperatures of 5ºC and 55ºC, respectively. All samples were subjected to 10,000 thermal cycles. During each thermal cycle, samples were placed in a cold-water bath for 60 seconds and in a hot water bath for 60 seconds, and the transition time between hot and cold baths was considered 10 seconds. After completion of thermal aging, the samples were cleaned in an ultrasonic cleaner containing distilled water for 5 minutes, and then the second series of color measurements were performed.

Data were analyzed using SPSS software through paired t-test, Tukey Post Hoc test, and ANOVA.

Results

The experimental results show that the mean ΔE index was 1.11±0.52 in the Ti group, 1.49±0.53 in the Y-ZrO_2 group, and 1.35±0.51 in the W-ZrO_2 group; the differences among all groups were significant (P=0.014). Pairwise comparisons of the groups showed that the difference in the mean ΔE between the Ti group and the Y-ZrO_2 group was significant (P=0.004) but, no significant differences were observed between W-ZrO_2 and Ti groups (P=0.063) and also between W-ZrO_2 and Y-ZrO_2 groups (P=0.28). In all the groups, the differences in the mean ΔE in the different thicknesses were significant (**Table I**).

The differences in the mean *l*, *a*, and *b* indexes in all the groups before and after the aging process were significant (**Tables II** and **III**).

Values greater than 5.5 for the ΔE index are compared as clinically acceptable and more than 2.6 as the perceived limit of integrated zirconia color changes against thicknesses in different substructures due to the aging process in **Table IV.**

Table I: Mean color changes of ΔE in different thicknesses.

Group	Thickness	Mean	SD	P_Value
Ti	0.5	1.78	0.29	<0.001
	0.7	1.56	0.54	
	0.9	1.28	0.11	
	1.1	1.30	0.28	
	1.3	1.17	.22	
	1.5	0.72	0.25	
	1.7	0.61	0.22	
	1.9	0.42	0.27	
Y-ZrO_2	0.5	2.06	0.08	<0.001
	0.7	1.84	0.52	
	0.9	1.90	0.43	
	1.1	1.66	0.14	
	1.3	1.26	0.36	
	1.5	1.47	0.50	
	1.7	0.98	0.36	
	1.9	0.78	0.08	
W-ZrO_2	0.5	1.94	0.48	<0.001
	0.7	1.39	0.13	
	0.9	1.71	0.27	
	1.1	1.42	0.23	
	1.3	1.84	0.15	
	1.5	1.11	0.40	
	1.7	0.80	0.11	
	1.9	0.61	0.05	

Table II: Mean differences of *l*, *a*, and *b* indexes before and after hydrothermal aging.

Parameter	Group	Before	After	The difference	P_ Value
l	**Ti**	79.59±0.60	78.79±0.86	-0.77	<0.001
	Y-ZrO_2	79.76±0.39	79.20±0.76	-0.56	<0.001
	W-ZrO_2	81.22±0.66	80.60±0.47	-0.62	<0.001
a	**Ti**	-0.27±07	-0.16±.83	0.11	<0.001
	Y-ZrO^2	0.97±0.26	1.08±0.38	0.11	<0.001
	W-ZrO_2	0.13±.50	0.19±0.46	0.06	<0.001
b	**Ti**	17.26±0.81	17.92±0.45	0.66	<0.001
	Y-ZrO_2	19.33±0.74	20.53±1.05	1.2	<0.001
	W-ZrO_2	17.31±0.98	18.27±0.80	0.96	<0.001

Table III: Changes in *l*, *a*, and *b* in different thicknesses.

Variable	Thickness	Ti			$Y\text{-}ZrO_2$			$W\text{-}ZrO_2$		
		Mean	S.D	P_Value	Mean	S.D	P_Value	Mean	S.D	P_Value
Δl	0.5	-1.04	0.33	0.004	-1.06	0.36	<0.001	-0.94	0.42	0.002
	0.7	-0.98	0.25		-0.86	0.31		-0.94	0.09	
	0.9	-0.94	0.18		-0.77	0.44		-0.76	0.21	
	1.1	-1.10	0.35		-0.77	0.27		-0.65	0.32	
	1.3	-0.95	0.32		-0.30	0.21		-0.44	0.20	
	1.5	-0.57	0.33		-0.11	0.15		-0.52	0.11	
	1.7	-0.49	0.25		-0.17	0.09		-0.36	0.13	
	1.9	-0.30	0.25		-0.36	0.15		-0.30	0.13	
	Total	-0.80	0.38		-0.55	0.41		-0.61	0.31	
Δa	0.5	0.26	0.15	<0.001	0.43	0.07	<0.001	0.15	0.06	<0.001
	0.7	0.26	0.07		0.15	0.04		0.12	0.04	
	0.9	0.16	0.04		0.08	0.03		0.05	0.03	
	1.1	0.06	0.10		0.06	0.03		0.04	0.01	
	1.3	0.09	0.04		0.05	0.01		0.01	0.01	
	1.5	-0.02	0.04		0.03	0.01		0.01	0.012	
	1.7	0.06	0.05		0.01	0.01		0.07	0.009	
	1.9	-0.02	0.02		0.01	0.01		0.01	0.009	
	Total	0.10	0.12		0.10	0.13		0.05	0.06	
Δb	0.5	1.42	0.22	<0.001	1.66	0.33	0.014	1.66	0.39	0.027
	0.7	1.14	0.68		1.59	0.53		1.02	0.10	
	0.9	0.84	0.20		1.34	0.59		0.89	0.41	
	1.1	0.66	0.07		1.24	0.19		0.93	0.33	
	1.3	0.64	0.11		1.07	0.32		0.92	0.75	
	1.5	0.32	0.12		1.01	0.14		0.96	0.45	
	1.7	0.21	0.02		0.96	0.37		0.71	0.07	
	1.9	0.06	0.07		0.68	0.08		0.52	0.08	
	Total	0.66	0.50		1.19	0.45		0.95	0.46	

Table IV: Comparison of the color change of monolithic zirconia thicknesses on different substructures due to aging process with the 2.6 and 5.5 values.

Thickness	Mean ΔE	P-value (Perception limit, 2.6)	P-value (Perception limit, 5.5)
<1.5	1.61	<0.001	<0.001
>1.5	0.83	<0.001	<0.001

Discussion

The results showed that the differences in *l*, *a*, *b* and ΔE parameters of zirconia before and after aging were significant in association with thickness and substructure type. The null hypothesis of this study, the aging process no effect the final color of restorations in different zirconia thicknesses and substructure, was rejected. Therefore, the aging process could affect the color of zirconia in all the groups, especially in thicknesses less than 1.5 mm. In all the analyses, the effect of thickness on zirconia color changes concerning aging was significant. The substructure color affected the *a*, *l*, and *b* values in the translucent zirconia. The greatest changes in the index l, *a*, *b*, and *ΔE* due to aging process in all the groups were related to zirconia with a thickness of 0.5 mm and the lowest changes were related to the thickness of 1.9 mm (**Table III**). The *l* and *ΔE* indices decreased and a and b indexes increased due to aging (**Table II**).

The results showed that the difference in mean ΔE was significant due to the thickness of zirconia in all groups. Therefore, in all the groups (Ti, $Y\text{-}ZrO_2$, and $W\text{-}ZrO_2$) the color change decreased significantly with increasing the ceramic thickness. In the Ti group, the color changes at thicknesses less than 1.1 mm were significantly higher than those at thicknesses more than 1.3 mm. In the $Y\text{-}ZrO_2$ group, color changes at thicknesses less than 1.5 mm were significantly greater compared to thickness more than 1.7 mm and in the $W\text{-}ZrO_2$ group, color changes at thicknesses of less than 1.3 mm were significantly higher compared to the thicknesses of more than 1.5 mm (**Table III**). Therefore, the maximum change was concerned with the minimum zirconia thickness and vice versa, which is supported by the results of y Volpato *et al*[25]. In fact, the color changes in the samples after aging process can be attributed to changes in the microstructure of the zirconia surface due to the long aging process. Monolithic particles are larger than tetragonal particles and increase the roughness of zirconia surface, exposing the surface to more color changes. Dikicier *et al*, showed that the effect of aging process on color parameters, regardless of thickness was significant and for all the systems, a value increased and l and b values decreased with increasing thickness. They also indicated that the thickness of restoration significantly affected its color. Their results regarding changes in a and l values are consistent with this study[35]. Stevenson et al reported that the color of ceramic restorations are affected by tooth color, ceramic thickness, and the opacity of materials[36]. The present study showed that the mean ΔE in the $Y\text{-}ZrO_2$ group was higher than that in the $W\text{-}ZrO_2$ and Ti groups, which was significant.

These findings indicated that the use of $Y\text{-}ZrO_2$ as a substructure resulted in more color changes than $W\text{-}ZrO_2$ and Ti.

The color changes in zirconia samples after the aging process were significantly less at thicknesses more than 1.5 mm than 2.6 value, suggesting that this color change is imperceptible by the naked eye, and t than 2.6 value, suggesting that this color change is imperceptible by the naked eye this color change is imperceptible by the naked eye. Therefore, it is not important. On the other hand, the color change in zirconia samples with thicknesses less than 1.5 mm due to the aging process was significantly greater than the 2.6 value and significantly lower than the 5.5 value. Therefore, the color change in these samples is perceptible but clinically acceptable.

Zirconia has an internal chemical structure with high density, so that its porosity is less than 0.05%. 37,38 This property of zirconia explains its slight changes in its appearance due to the aging process. Changes probably occur only on the zirconia surface; therefore, creating a more uniform and homogeneous surface may reduce the aging effect. However, it should be noted that several factors can directly affect the transition from the tetragonal phase to the monolithic, including defects or the absence of oxygen, size, shape, and location of ZrO_2 particles as well as type and amount of stabilizing oxide, and presence of residual stress that are not clinically controllable[39-42]. Some laboratory and clinical factors that can be controlled include thermal changes, surface treatments, finishing line type, and polishing[44-47].

This study examined only one brand of monolithic zirconia in one shade. There may be additional value in future studies looking at other zirconia brands, other shades, and the effects of different cement types.

Conclusion

Within the limitation of this study, it could be concluded that the mean ΔE changes were significantly different among all groups (Ti, Y-ZrO_2, and W-ZrO_2). The color changes due to the aging process in the Y-ZrO_2 group were greater than the other two groups. The *l* and *ΔE* indexes decreased and *b* and *a* indexes increased due to the aging process. In all the groups, thicknesses more than 1.5 mm exhibited greater color stability after the aging process.

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