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Effect of Aging Treatments on Properties of Zirconia with Different Levels of Translucency

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ABSTRACT

Purpose: The aim of this study was to evaluate the effect of low-temperature degradation(L-TD), and weathering process on translucency, surface roughness, biaxial-flexural strength, and crystalline phase identification of zirconia with different levels of translucency. **Materials and methods:** A total of ninety-six (N= 96) zirconia discs were fabricated in this study. Samples were divided into three groups (n=32 each) according to type of zirconia: IPS EmaxZirCAD of low, medium translucency and Incoris translucent Zirconia of high translucency. Translucency measurements, surface roughness, biaxial flexural strength determination and phase identification were carried out before and after aging. Each group was then sub-divided into 2 subgroups (n=16); (subgroup A): Low-temperature degradation(L-TD) at 134ºC, (subgroup B): Accelerated aging by means of a weathering process (WP). **Results:** Statistical analysis using two-way ANOVA test revealed that there was a significant effect on translucency parameter (TP), and L-TD showed statistically significantly higher mean TP than WP. L-TD showed significantly lower mean roughness than WP. There was no significant difference between biaxial flexural strength values before aging and after L-TD and WP. There was no phase transformation identified before and after aging indicating resistance to different types of artificial aging. **Conclusions:** Low-temperature degradation and weathering process affect translucency parameter and surface roughness of zirconia-based materials. Artificial aging doesn't affect the biaxial flexural strength, and phase identification of different zirconia translucencies.

KEYWORDS

Translucency, surface roughness, zirconia, degradation, weathering process.

INTRODUCTION

Dental ceramic restorations have been used for decades in dental prostheses such as crowns and fixed dental prostheses [(1)], as a consequence of their exceptional cosmetic appearance and

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biocompatibility properties. It has been shown that this kind of dental biomaterial provided the most precise mimicry of color, translucency, and texturing of human dentition [(2)].

A well-known type of dental ceramics is the multi-phase Zirconia, which can occur in three distinct crystallo-graphic phases based on the degree of temperature (i.e., mono-clinic, tetragonal, and cubic). A tetragonal phase structure of zirconia is asserted at room temperature, when yettriaoxide [Y_2O_3] is added as a stabilizing-oxide, despite the fact that the thermodynamically preferred state is the monoclinic one [(3)].

The transformation toughening mechanism is responsible for the higher fracture toughness, hardness, and flexural strength of yettria-stabilized tetragonal zirconia polycrystalline. It has been revealed that during the application of stresses, a four percent positive volumetric change (expansion) occurs as a consequence of phase transformation from the tetragonal form to the monoclinic one resulting in internal compressive stresses that partly guarding the tip of the crack from the external stress and hence, cause prevention of crack propagation [(4)].

Additives, method of processing, and grain size and distribution, are all considered controlling factors of zirconia translucency. Enhancing the grain size and distributing the grains eventually and homogenously, not only can improve the translucency of zirconia through minimizing the interface between grains and decreasing porosity, but also gives exceptional strength [(5)].

There are different grades of 3-YTZP that show some degree of light transmittance. In order to improve the translucency of this material for esthetics the concentration of alumina additive should be decreased, porosity should be eliminated and by increasing the yettria content [(6)].

The most translucent 5-YTZP materials were indicated for anterior crowns and fixed dental prosthesis. It is formed by increasing the yettria content from three mol. % to five mol. %, this will increase the cubic phases that reduce the light scattering defect producing high translucency [(7)].

Artificial accelerated aging or low-temperature degradation (LTD) is a way that can imitate natural conditions allowing the difference in color of ceramic restorations throughout the course of time to be detected. It's worth mentioning that surface roughness compromises the esthetic and biomechanical properties of dental prostheses resulting in accelerated aging [(8)].

Clinical behavior and shortcoming of dental ceramics can be expected from its mechanical properties including strength as a prime feature. As detected by X-Ray Diffraction (XRD), phase transformation from tetragonal to monoclinic phase induced by stresses, greatly improves mechanical properties of ZrO_2-containing ceramics [(9)].

Low-temperature degradation was identified to occur as a result of unprompted phase transformation from the tetragonal to the monoclinic phase, occurring gradually by time at low-thermal and presence of water thus it is triggered on the surface of the ceramics. This type of transformation may reduce flexural strength of certain types of zirconia and induce changes in optical features of high translucent-zirconia after aging treatment[(10)].

The mechanism by which crystalline, mechanical, and optical stability of zirconia-based dental materials work together, can give a prospective guideline for the implementation of translucent zirconia for clinical recommendation [(10)].

The null hypotheses proposed for the present study were that artificial aging will not affect translucency, flexural strength, surface roughness, and crystalline phase identification of yettria stabilized zirconia.

MATERIALS AND METHODS

Sample size and grouping:

Calculating sample size was performed using interventional study (randomized controlled study) taking into consideration the following based on the results of a previous study [(6)], confidence level 0.95%, alpha (α) error 0.05 (5%), beta (β) error 0.1 (10%) i.e. power of the study 0.9 (90%) calculated; the resultant sample size (N) calculated to be (96) samples. The samples were divided to three groups (n=32 each) according to material with different level of translucency:

Group I : IpsEmaxZirCAD of low translucency (n=32).

Group II: IpsEmaxZirCAD of medium translucency(n=32).

Group III : Incoris translucent Zirconia of high translucency(n=32).

Each group was divided into two subgroups (n=16) according to process of aging:

Subgroup A: Low-temperature degradation (L-TD).

Subgroup B: Accelerated aging (weathering process) (WP).

The study design and methodology was approved by the Research Ethics Committee of the Faculty of Dental Medicine for Girls, Al-Azhar University, with the code REC-CR-22-07.

Preparation of samples:

Zirconia blanks were milled in the form of cylinders of 12mm diameter using CAD/CAM Core Tec model 250 in dry milling machine (Eiterfeld, Germany) , according to the design imported (cylinder shape). To compensate for the expected shrinkage during sintering, the blanks were milled with larger dimensions (20-25% larger than the desired final size).

A total of ninety-six (N=96) discs with 12 mm diameter and 1.8 mm thick were cut from the cylinders via a precision cutting device isomet 4000 microsaw (Buehler Germany), under2,500 revolutions per minute through a disc of diamond 0.6 mm thick, under a cooling system. The ratio of water coolant to anticorrosive agent was [30:1].

To compensate for the expected reduction in size that occurred after the sintering process, samples were cut 20:25 % greater than the required final size. it was measured by a digital caliper. Partially sintered disc samples were fully sintered following the manufacturer's guidelines at the center of the TABEO sintering furnace, (TABEO-1/M/ZIRKON-100, Mihmvogot, Germany), high-temperature furnace with program control unit on sintering beads, fig. (1). The digital caliper was also used in determining the dimensions of samples following the sintering process. The planned final dimensions of each sample were 10 mm in diameter and 1.5mm in thickness.

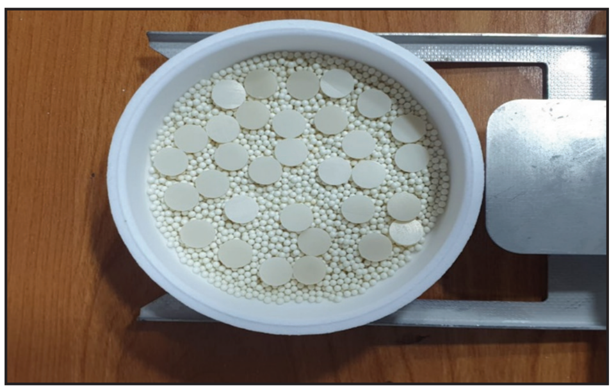

Figure (1) Sintering boats with samples placed over sintering beads

After the samples have been sintered, once it was cooled to room temperature, minimal finishing was carried out according to the manufacturer's recommendations; using diamond finishing stone at low speed using low pressure avoiding local phase transition, twice on each surface in one direction. Polishing was carried out using KENDA finishing and polishing kit (Vaduz, Liechtenstein) without using polishing paste following manufacturer recommendations.

Low-temperature degradation of the samples:

Samples of subgroup (A) were low-temperature degraded using an autoclave (TS-Tau sterile autoclave, FinoMoransco-COMO, Italy). The samples were packed independently in sterilization pack and arranged on autoclave trays and were placed in the autoclave for 1 hour, 134°C, and 0.2 MPa pressure.

Experimental time intervals were calculated after the temperature of the autoclave reached 134^0C. Samples were stored after cooling to room temperature in a glass air-tight container till testing.

Weathering process:

An accelerated weathering tester (Q.U.V Dongguan, Guangdong, China) was used to expose the samples of subgroup (B) to an artificial accelerated aging process through weathering cycles. Samples were put in a chamber that was previously programmed to 25 cycles for 300 hours. A controlled irradiance of ultraviolet lamps that emits light at 340 nm wavelength, was used to subject each sample from only one surface to intermittent cycles of light and darkness (i.e., The test cycle was 8 hours of ultraviolet exposure at 50^0C, 4 hour of condensation exposure at 40^0C plus water spray). The whole weathering procedure was done under 450 KJ/$m^2$of energy.

Testing Procedures:

Samples of the three groups were tested before and after aging:

Translucency Parameter (TP value):

The translucency was determined and recorded through a reflecting spectrophotometer (Model RM200QC, X-Rite, Neu-Isenburg, Germany) with a standard previously fixed aperture size (4 mm). Specimens were placed in the middle of aperture for the two backings.

Surface Roughness procedure:

Specimens were photographed using a USB Digital microscope with a built-in camera (U500X Capture Digital Microscope, Guangdong, China) attached to a computer.

Biaxial flexural strength (MPa):

A test for measuring the biaxial flexural strength by using (uniform compression on a disc) with a ball on a ring fixture was selected [(11)]. The test was utilized at a crosshead speed of one millimeter with a computer-controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA). A five-kilo newton load cell was utilized for test measurements and eventually, all the recorded data was generated and saved via a digital software (Instron® Bluehill Lite Software). Discs were placed on 8-mm diameter circular knife-edge support with a 3.8 mm diameter rounded indenter loaded in the middle.

The unpolished surface of each disc was selected to be the loaded side whereas the polished surface was selected to be the tension side. A thin film of rubber was positioned in between the load applier tip and the sample surface. Higher fracture stress results in more elastic energy being temporarily retained before being released during fracture, producing more fragments.

Crystalline phase identification

The samples were examined to test their amorphous nature using Philips Analytical X-Ray diffraction system, PHILIPS X-Ray Diffractometer (Caerphilly, United Kingdom). The generator tension was 40KV and the generator current was 30mA. Analyses were performed on samples having a fixed surface area dimension of 1.54 Cm^2 both after and before aging. There were using the phases of the Rietveld refinement to index and analyze tetragonal zirconia, monoclinic zirconia, t-ZrO2, cubic zirconia, c-ZrO2 and m-ZrO2.Each detected phase's Y2O3 content was determined using the percentage of tetragonal of the t-ZrO2 in relation to revised lattice parameters phase, quantified by the proportion c/2a.

Statistical analysis:

Mean and standard deviation values were used as representatives for the data and study results. Intergroup evaluations and comparisons were done using Two Way ANOVA test. Boneferroni post-hoc test was used for pairwise comparison when ANOVA test was found to be significant. IBM SPSS software program was used to perform the statistical analysis of the current study (IBM SPSS Statistics for Windows, Ver. 23.0.0, Armonk, NY, IBM Corporation).

RESULTS

Translucency parameter (TP) values before and after aging, table (1), and figure (2):

With low translucency zirconia after L-TD**;** there was a statistically significant increase in mean TP after aging. After WP; there was a statistically significant decrease in mean TP after aging.

With medium translucency zirconia after L-TD**;** there was a statistically significant increase in mean TP after aging. After WP; there was a statistically significant decrease in mean TP after aging.

With high translucency zirconia after L-TD**;** there was a statistically significant increase in mean TP after aging. After WP**;** there was a statistically significant decrease in mean TP after aging.

Table (1) *Repeated measures ANOVA results of comparison of TP values before and after aging.*

Zirconia translucency	Aging process	Before aging		After aging		*p-value*	Effect size
		Mean	SD	Mean	SD		
Low translucency	L-TD	8.43	2.14	10.03	2.53	0.041*	0.049
	WP	7.82	1.94	3.21	0.98	<0.001*	0.299
Medium translucency	L-TD	9.53	2.05	12.45	3.9	<0.001*	0.147
	WP	8.04	1	4.18	1.67	<0.001*	0.23
High translucency	L-TD	11.38	1.77	13.51	3.28	0.007*	0.083
	WP	10.21	1.85	3.9	1.86	<0.001*	0.445

**: Significant at P ≤ 0.05 SD = Standard deviation, P= probability, * non-significant at p>0.05*

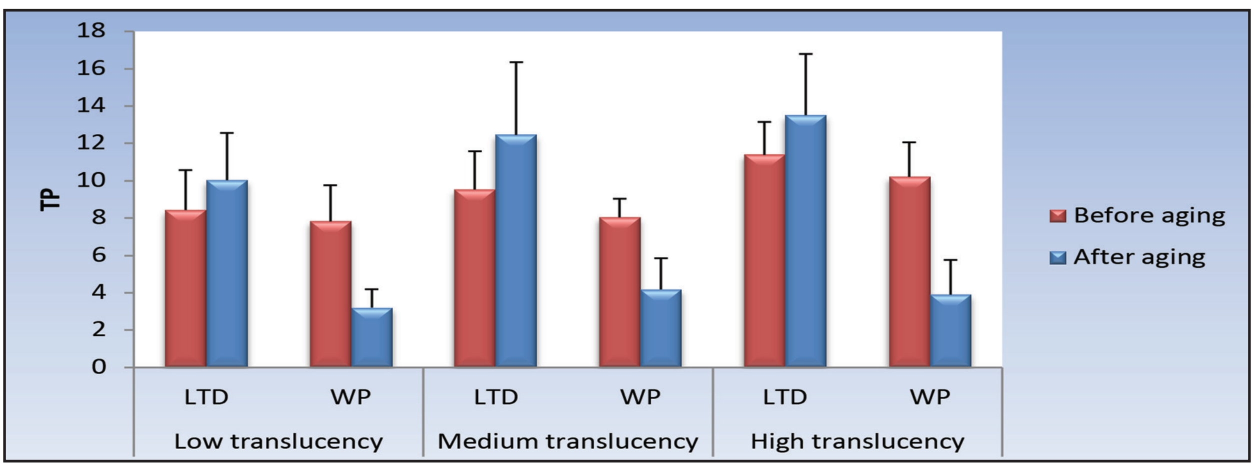

Figure (2): Bar chart illustrating results of comparison of TP measurements represented as mean and standard deviations before and after aging

Surface roughness values before and after aging, table (2), and figure (3):

With low translucency zirconia whether after L-TD or WP**;** there was a statistically significant increase in mean Ra after aging.

With medium translucency zirconia after L-TD**;** there was no statistically significant change in mean Ra after aging. After WP; there was a statistically significant increase in mean Ra after aging.

With high translucency zirconia whether after L-TD or WP; there was no statistically significant change in mean Ra after aging.

Table (2) *Repeated measures ANOVA results of comparison of between Ra (µm) values before and after aging.*

Zirconia translucency	Aging process	Before aging		After aging		*p-value*	Effect size
		Mean	SD	Mean	SD		
Low translucency	L-TD	0.2893	0.0008	0.2924	0.0043	<0.001*	0.144
	WP	0.2897	0.0013	0.2924	0.0026	0.002*	0.11
Medium translucency	L-TD	0.2898	0.0014	0.2907	0.0037	0.303	0.013
	WP	0.2904	0.0019	0.2925	0.0031	0.014*	0.069
High translucency	L-TD	0.289	0.0016	0.2884	0.0023	0.482	0.006
	WP	0.29	0.0012	0.2917	0.0019	0.051	0.044

**: Significant at P ≤ 0.05 SD = Standard deviation, P= probability, * non-significant at p>0.05*

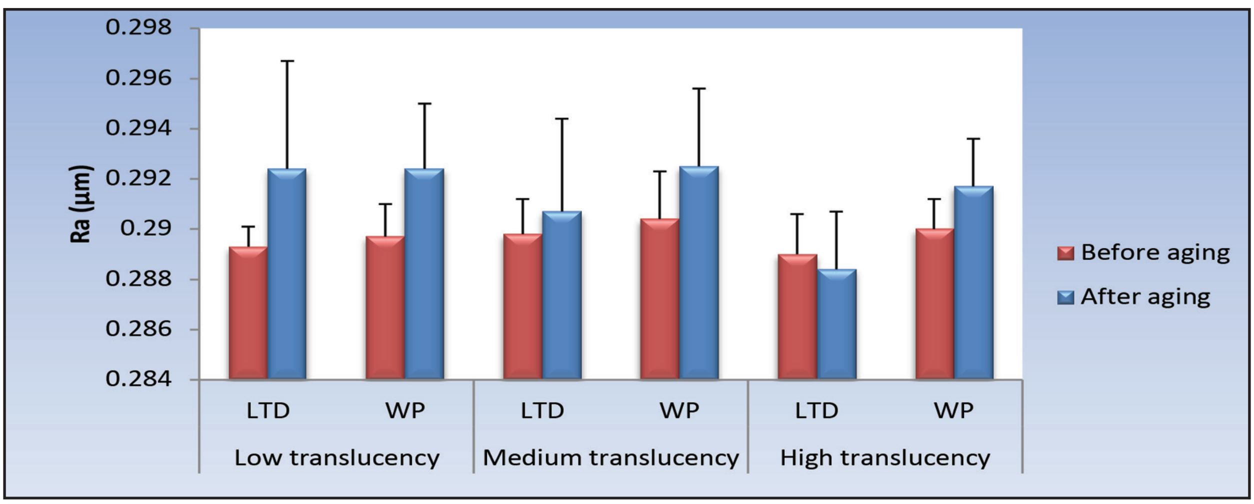

Figure (3): Bar chart illustrating results of comparison of Ra measurements represented as mean and standard deviations before and after aging

Biaxial flexural strength values, table (3), and figure (4):

Whether with low, medium as well as high translucent zirconia**;** there was no statistically significant difference between biaxial flexural strength values without aging, after L-TD as well as after WP.

Crystalline phase identification values, , table (4), and figure (5):

The quantitative phase analysis of translucent zirconia whether low, medium as well as high**;** didn't reveal a measurable phase transformation by XRD after L-TD as well as after WP.

Table (3) *Two way ANOVA results of comparison of between biaxial flexural strength (MPa) values with different interactions of variables*

Aging process	Low translucency		Medium translucency		High translucency		*P*-value	Effect size *(Partial eta squared)*
	Mean	SD	Mean	SD	Mean	SD		
No aging	1318.9	57.8	980.4	66.4	1106.6	212.2	0.074	0.135
L-TD	1080.2	288.8	1168.1	189.6	1106.2	84	0.823	0.011
WP	1086.7	251.2	841.2	157.7	1161.1	449.8	0.082	0.13
***P*-value**	0.184		0.090		0.909			
Effect size *(Partial eta squared)*	0.09		0.125		0.005			

**: Significant at P ≤ 0.05 SD = Standard deviation, P= probability, * non-significant at p>0.05*

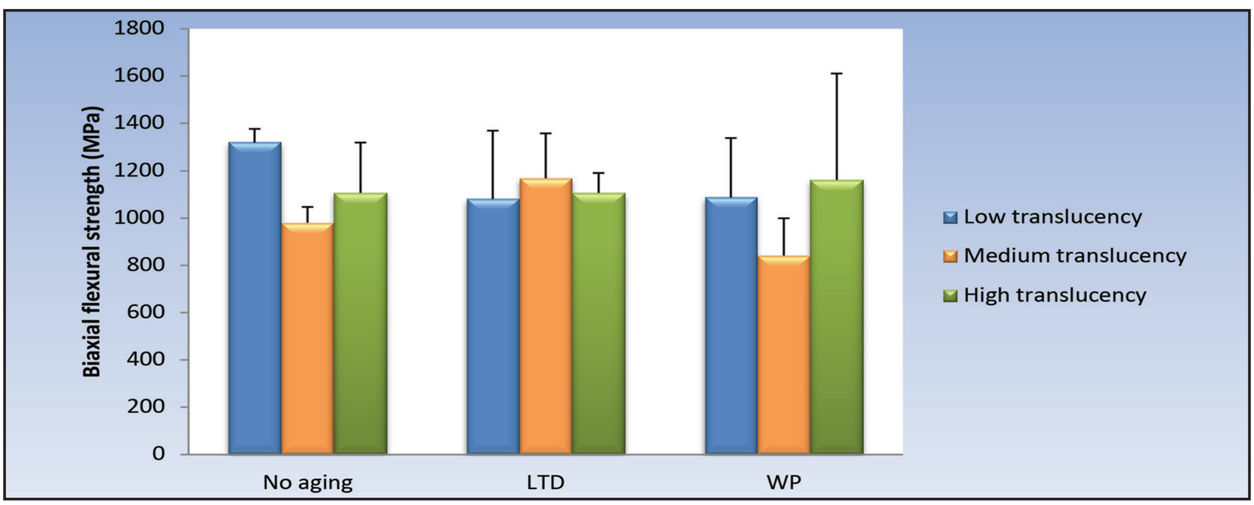

Figure (4): Bar chart illustrating results of comparison of biaxial flexural strength measurements represented as mean and standard deviations before and after aging

Table (4) *Percentage of tetragonal zirconium oxide for with different interactions of variables before and after aging.*

Zirconia translucency	Aging process	Before aging		After aging	
		YO_2 oxide	t-ZrO_2	YO_2	t-ZrO_2
Low translucency	L-TD	67%	33%	65.2%	36%
	WP			69%	31%
Medium translucency	L-TD	72%	25%	73%	27%
	WP			74.2%	24%
High translucency	L-TD	75.9%	28%	78%	27%
	WP			76%	26.2%

**: Percentage% of Yo_2:Yttrium oxide , t-ZrO_2 : tetragonal zirconium oxide*

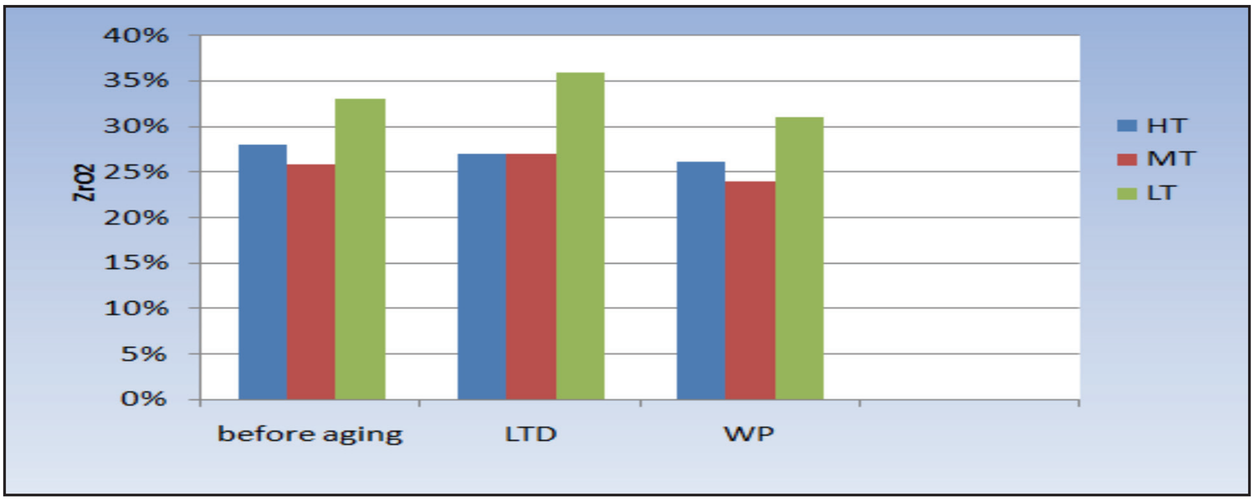

Figure (5): Bar chart representing percentage of zirconium oxide and amount of phase transformation before and after aging

DISCUSSION

Dental prostheses made up of dental ceramics are well known for their inherent esthetics, wear resistance, and biocompatibility [(12)]. Zirconia dental ceramics are used for restoration of posterior teeth, as these oral regions are subjected to high forces of mastication, a condition that highlights the need for an inherently tough and strong dental ceramic like yettria-stabilized tetragonal zirconia (YTZ). However, its opaque nature limits its use in esthetic area due to low optical properties [(13,14)].

Low temperature degradation, which is usually known as aging, is a condition that when occurs under humidity can result in loss of YTZ strength and other advantageous features. It was suggested that detrimental crystal transformation damages the microstructure which enriches YTZ ceramics with the excellent features throughout its fabrication procedures. Hence, it is essential to understand the phenomenon when selecting a dental restorative material [(15)].

Therefore, the present study aimed to evaluate the effect of low-temperature degradation and accelerated aging (weathering process) on translucency, surface roughness, biaxial flexural strength and crystalline phase identification of zirconia-based dental ceramics with various translucency levels.

Zirconia with various translucency levels were used in this study including IpsEmaxZirCAD of low translucency, IpsEmaxZirCAD of medium translucency and Incoris translucent Zirconia of high translucency.

Zirconia blocks were cut using a precision cutting machine (isomet 4000 microsaw). The samples were sectioned 20-25% greater than the required size to compensate for the expected reduction in dimension.

Sintering of samples were done following the manufacturer's instructions at high temperature furnace at 1460-15300C to produce impenetrable structures confirming unity between the zirconia grains and to avoid larger grain formation.

Constructed samples were finished and polished to mimic real clinical conditions because it has been claimed that different finishing techniques have a significant impact on zirconia's mechanical properties [(16)]. Both procedures were carried out according to the manufacturer recommendations; using diamond finishing stone at low speed using low pressure avoiding local phase transition, twice on each surface in one direction.

Several studies have recommended autoclaving as an established method for accelerating the aging of YTZ materials as it causes low-temperature degradation (L-TD)[(17)]. In the present study, aging in an autoclave for 1 hour at 134^{0}C was done for samples in (subgroup A). The aging procedure followed simulate approximately 1year of clinical use [(18)].

Artificial accelerated aging through weathering process is an experimental method used to mimic oral environmental conditions extra-orally[(19)].Samples in (subgroup B) were subjected to 300 hour of artificial aging using a weathering process in an accelerated weathering tester which simulates 1 year clinically[(20)].

In the current study, it was hypothesized that artificial aging will not affect translucency, surface roughness, biaxial flexural strength and crystalline phase identification of yettria stabilized zirconia. However, the null hypothesis was partially rejected as the results showed that artificial aging has a significant effect on translucency, surface roughness but have no effect on biaxial flexural strength and crystalline phase identification of yettria stabilized zirconia.

The results in the present study illustrated that the effect of L-TD aging regardless of zirconia translucency, showed that there was a statistically significant increase in TP mean after aging. Those results were in agreement with a previous study [(21)] in which a statistically significant increase after artificial aging can be attributed to the increase in grain size of the materials therefore increase in the light transmission. Also agreed with another study[(22)] in which translucency increased with increasing aging time for zirconia attributed to reduced dispersion of light from cubic zirconia's grain boundaries.

Another study [(23)] concluded that increasing the aging time results in more translucency of conventional zirconia and more opacity of multilayered one.

These finding were contraindicated with the results reached by a previous study [(24)] in which mean translucent parameter value before aging showed statistically significantly higher value than after aging and contrast ratio before aging showed statistically significantly lower value than after aging. This could be explained due to transformation from tetragonal to monoclinic phase with associated surface roughness which caused an increase in light scattering.

On the other hand, the effect of WP aging regardless of zirconia translucency, showed that there was a statistically significant decrease in TP mean after aging. The results were in agreement with another study [(25)] concluded that light scattering from pores, and the difference in refractive indices between tetragonal and monoclinic zirconia crystals would cause the translucency to decrease.

The results were also in agreement with another study [26], which showed a proportional increase of opacity with the increase in diameter thickness, residual porosities, and grain boundaries. All these factors result in more scattering of incident light and hence the translucency of ceramic is diminished.

On the other hand, these findings were in contradiction with the results reached by another study [27] in which the translucency didn't decrease after aging in the tested type of zirconia which is probably associated with the decrease in the amount of transformation of zirconia and this is followed by decreased superficial irregularity, light scattering, and reflection.

Regarding the surface roughness, there was a statistically significant increase in surface roughness after aging (L-TD& WP) with low and medium translucency as showed in the current study. The finding was in consistent with previous study[28] which found that surface roughness of the zirconia increased after being exposed to hydrothermal aging at low-temperature when aging procedure performed in an autoclave at 122◦C at 2 bars for 8hours.

On the contrary with the present study findings, another study[29]concluded that aging did not promote any relevant surface change. They explained that through the fact that samples were mirror polished, thus after LTD samples might retain its polishing surface. This aging schedule was carried out very quickly as a result to that, it may not lead to pull-out of grain, leading to identical roughness esteems to the non-aged zirconia. However, this finding agreed with the present study in which the low-temperature degradation and weathering process did not affect the surface roughness of high translucent zirconia.

The results were in agreement with a previous study [20] which revealed that artificial aging affects surface roughness of Lava ultimate ceramic but does not affect surface roughness of high translucent zirconia.

The results were also in agreement with another study [23] who stated that the aging process significantly elevated the roughness (Ra) measurements values in all tested groups. However, the Ra values of that study were in the range of 0.003 and 0.01 um which is much lesser than Ra value of 0.2 µm, so it is expected to be acceptable clinically.

On the other hand, results obtained disagree with those of a another previous study [30]which stated that aging doesn't affect surface roughness of YTZ ceramics. The reason may be attributed to the different autoclave durations and different methods of specimen preparation.

Regarding biaxial flexural strength, there was insignificant effect on flexural strength which could be noticed in the results. The finding was in consistent with previous study[31]in which zirconia materials did not show any sign of discernible change in flexural strength after artificial aging although a quantity of greater than 40% of monoclinic phase has been found. This could be clarified by that transformation zone has no extremely sufficient extension within material to impact its main part strength.

On the contrary of the findings in the present study, another study[32] concluded that flexural strength decreased by aging due to presence of internal defects in which the monoclinic phase entered at the surface and phase transformation continued throughout into the majority of the ceramic.

Regarding the crystalline phase identification, various groups of zirconia exhibited distinguishable crystalline form, chemical structure, and microstructural features. All materials have highly polished surface and significant degree of compaction with uniformly distributed boundaries of internal grains together with less than one percentage of remnant porosity [33].

The results in this study didn't reveal any detectable phase transformation suggesting reluctance to hydrothermal degradation. This is in agreement with previous study[30] which revealed a direct proportional relationship between the amount of yettria in dental Zirconia of high translucency and its resistance to accelerated aging.

In agreement with the current study, a previous study [34] which suggested that no monoclinic phase noticed following the process of aging due to high amount of cubic crystal retard the aging degradation in which phase transformation didn't happen.

Besides, results obtained disagree with another study [35] which reported that degradation starts on the surface with a quick transition from a tetragonal to a monoclinic phase and stabilizes after 26 hours. In comparison to highly translucency zirconia, conventional zirconia displayed a greater level of surface deterioration.

CONCLUSIONS

The following can be concluded from the present study:

1. Low-temperature degradation and weathering process have a significant effect on translucency parameter of zirconia with different levels of translucency.
2. Low-temperature degradation and weathering process have a significant effect on surface roughness of low, medium translucent zirconia, and have no effect on surface roughness of high translucent zirconia.
3. Low-temperature degradation, and weathering process didn't affect the flexural strength and phase identification of zirconia-based materials.

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RECOMMENDATIONS

Clinically relevant restorations and different prosthetic designs should be investigated rather than the disc shaped samples

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CONFLICT OF INTEREST

There is no conflict of interest.

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