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EFFECT OF ACCELERATED AGING ON THE TRANSLUCENCY OF MONOLITHIC ZIRCONIA AT MINIMAL THICKNESS

Ahmed M. Alamledin,* Tamer E. Shokry,** Ahmed Y. El Kouedi***

ABSTRACT

Objective: The objective of this study was to evaluate the effect of accelerated aging on the translucency of two monolithic zirconia ceramics used at their minimal thickness. **Materials and methods:** A total of 20 disc-shaped specimens (10x10x0.5 mm) were prepared from two zirconia blanks, high-translucent partially-stabilized tetragonal zirconia (Zolid ht+) and ultra-translucent fully-stabilized cubic zirconia (Zolid fx), and divided into two groups (n=10). An integrating sphere reflective light recording spectrophotometer was used to assess the color parameters L*, a*, and b* before and after artificial accelerated aging for 5 hours in an autoclave at 134°C with 2 bar steam pressure. The Translucency Parameter (TP) was calculated based on $TP=[(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{1/2}$. The tests of homogeneity of variance, normality, and 1-way ANOVA statistical analysis of variance were performed by Graph Pad InStat software for windows ($\alpha=.05$). **Results:** A statistically significant higher mean TP value was recorded with fully-stabilized cubic zirconia group compared to that of partially-stabilized tetragonal zirconia, both before ($P=.003^*<.05$) and after ($P<.0001^*<.05$) accelerated aging. Artificial accelerated aging had a significant effect on the translucency of both fully-stabilized cubic zirconia ($P=.0227^*<.05$) and partially-stabilized tetragonal zirconia ($P=.023^*<.05$) increasing the mean TP value of both materials. **Conclusions:** Within the limitations of this in vitro study, it was found that accelerated aging had a significant effect on the translucency of both fully-stabilized cubic and partially-stabilized tetragonal monolithic zirconia, causing an increase in the mean TP value of both materials.

KEY WORDS: Translucency, monolithic zirconia, accelerated aging,

INTRODUCTION

The success of ceramic restorations over the past decade has rendered these esthetic restorations a successful replacement to their older metal-ceramic predecessor. Metal-ceramic restorations have been considered the “gold standard” for a long time (1). Newly-developed materials and technologies, in addition to the increased patients’ demand for higher esthetics, has made for the greatest drive toward these more esthetic alternatives (2). The use of zirconia, which possesses both superior mechanical properties and adequate esthetics, has increased dramatically over the past decade (3). However, many concerns remain with the use of zirconia as a biomedical dental ceramic. The first of these was its lack of acceptable translucency for the earlier generations of zirconia, which necessitated the veneering of zirconia with a more translucent glass-containing porcelain, forming a bilayered structure(4). Nonetheless, chipping of the veneering porcelain arose as one of the major failures of these bilayered zirconia restorations (5). This has led the quest to the development of more translucent generations of zirconia, which could be used on its own without the need for a veneering porcelain; in other words, monolithic zirconia (6).

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* Assistant Lecturer and PhD student, Department of Fixed Prosthodontics, Faculty of Dental Medicine, Al-Azhar University, Cairo, Egypt.

** Professor, Department of Fixed Prosthodontics, Faculty of Dental Medicine, Al-Azhar University, Cairo, Egypt.

*** Assistant Professor, Department of Fixed Prosthodontics, Faculty of Dental Medicine, Al-Azhar University, Cairo, Egypt.

Partially-stabilized tetragonal zirconia (PSZ) is one of the most heavily-used restorative materials for the fabrication of fixed dental prostheses. Although the more recent generations developed of this class of dental zirconia possessed enough translucency to be used in a monolithic form, it was far below the translucency obtained by its more translucency competitor glass ceramics (7). Fully-stabilized cubic zirconia (FSZ) has been developed to address the concern of the translucency challenge observed with older generations of monolithic zirconia (8). Unlike the metastable nature of tetragonal zirconia, increasing dopant concentrations to a critical limit has allowed for making the cubic phase prevalent in a whole-new generation of dental zirconia, known as cubic or fully-stabilized zirconia. The cubic nature FSZ possesses better translucency and is believed to be able to produce a more life-like natural fixed dental restorations (9). In the meantime, because the cubic phase in this class of zirconia is fully-stable, this material does not possess the transformation toughening mechanism, which its predecessor, partially-stabilized tetragonal zirconia, was famous for (10).

Thickness is a strong determinant of success in all ceramic restorations. Its impact goes beyond the structural success of these restorations, to areas like the esthetic success of these systems (11). The thickness required for monolithic zirconia has, unsurprisingly, been decreased when the space needed for the late veneering porcelain was eliminated. Monolithic zirconia has the ability to resist high occlusal loads with as low as 0.5 mm minimal axial and occlusal thickness (12), even when used as a single tooth full coverage fixed dental restoration –i.e. crown- in the molar region (13). This decrease in overall thickness of zirconia restorations, with the subsequent added advantage of conservation of tooth structure, had some other positive consequences (14). This thickness decrease has its sequential effects on the translucency of both the partially-translucent tetragonal zirconia and the more translucent cubic zirconia (15). A decrease in thickness shortens the distance of light pathway through a translucent ceramic, leading to less light/material interference, and more enhanced translucency (16). However, in clinical practice, thickness control is multifactorial, and needs a compromise on many levels. Thus, understanding the effects of thickness variation on the overall translucency of newly-developed all ceramic materials is paramount (17).

The role of translucency in the final overall esthetic outcome of all ceramic restorations is, unquestionably, enormous. An all ceramic restoration with a translucency match close to that of adjacent natural teeth can be conceived to the eye as "natural-looking" (18). The advent of enhanced and augmented translucency in newer generations of monolithic zirconia, namely cubic zirconia, has made deciding the degree of translucency anticipated from a zirconia restoration justifiable. Zirconia ceramics are polycrystalline. Like all polycrystalline materials, their optical properties depend, to a large degree, on their microstructure (19). As light hits the surface of zirconia, a fraction of the incident light is reflected, but most of the light is scattered at the grain boundaries and within internal flaws such as pores. These pores have a different refractive index than zirconia grains and influence the light passage through the material, adding to its opacity (20). A quantitative representation of color by the CIELab system has fueled the extensive application in dentistry to study esthetic and translucent materials, as well as study materials' optical behavior in an objective way (21). Spectrophotometers have been used for several decades to determine translucency instrumentally with numeric expression of the CIELab coordinates by converting measured spectral reflectance values into CIELab color coordinates (22). According to the CIELab system, the translucency of dental restorative materials has been extensively evaluated using the Translucency Parameter (TP) (23). TP is defined as “the color difference of a material of a uniform thickness in optical contact with ideal white and black backgrounds”. Translucency parameter (TP) is a direct measure of relative translucency (24).

Notwithstanding, as monolithic zirconia has become at a constant exposure to the tough oral environment, with all the humidity and temperature fluctuations anticipated, the ability of monolithic zirconia to survive these environmental conditions arose at the center of studying biomedical zirconia as a popular dental restorative material (25). The degradation of zirconia when exposed to humidity at low temperatures is well documented. This “aging” of zirconia has been studied as a phenomenon known as Low Temperature Degradation (LTD) (26). This LTD phenomenon can occur in temperatures as low as the room temperature and up to 400ºC (10) leading to spontaneous transformation of zirconia from its metastable tetragonal phase into the monoclinic one without the application external loads. This spontaneous phase transformation is progressive and known to be time dependent (27). The dilemma facing manufacturers of Y-TZP is that compositions best-known for their fracture toughness, due to their desirable transformation toughening mechanism, are the most prone to the undesirable low temperature degradation (28). Artificial Accelerated Aging (AAA) is a method claimed to simulate the effects of intraoral conditions. It is believed to initiate a cascade of material changes within biomedical monolithic zirconia similar to that produced by the LTD (28). Different studies have used a proposed autoclaving protocol to test the effect of zirconia aging on its properties. It has been proposed that steam sterilization of zirconia in an autoclave at 134ºC and 2 bar steam pressure for 5 hours simulates 15-20 years at 37ºC (29). The effect of zirconia aging on its translucency are numerous. Surface grain pullout, slow crack propagation, surface roughening, surface uplifts, and enhanced wear rates can be possible sequels to this phenomenon (30). Deterioration of optical properties and translucency of zirconia can be a consequence to LTD (9). Little is known about the effect of LTD and aging of zirconia on its optical properties, making further research into the effect of aging on the translucency of monolithic zirconia valuable (31). The hypothesis of this study was that accelerated aging will have a significant effect on the translucency of the two types of monolithic zirconia at their minimal thickness.

MATERIALS AND METHODS

Two highly translucent monolithic zirconia materials were used in the study, categorized based on the degree of their stability into: Partially-stabilized tetragonal zirconia PSZ (Ceramill® Zolid ht+) (HT group) and Fully-stabilized cubic zirconia FSZ (Ceramill® Zolid fx) (FX group). A total of 20 rectangular disc-shaped specimens (10x10x0.5 mm) were fabricated from a white zirconia blank of each of the two materials (n=10) in their presintered state. Cuts were made into the presintered blanks using a linear high precision microtome (Isomet 4000; Buehler) with a diamond disc 20 cm in diameter and 0.6 mm-thick, running at a speed of 2500 rpm under a continuous water coolant. The integrated cooling system flooded specimens from both sides of the cutting blade. All specimens were cut 20% larger than the desired dimensions to compensate for the sintering volumetric shrinkage. All discs were 12×12 mm in the pre-sintered state with an initial thickness of 0.6 mm; which made the discs have a 10×10 mm final dimensions with a final thickness of 0.5 mm. Specimens’ dimensions –width and thickness- were confirmed using a digital caliper (Fisher Scientific Traceable Caliper) immediately after sectioning in their presintered state, and rechecked after the sintering process to confirm that the desired thickness was obtained.

White presintered zirconia disc-shaped specimens were colored before the sintering stage by dipping into an A2 coloring liquid corresponding to each of the two types of zirconia material used;

Ceramill® Coloring Liquid New Formula for PSZ, and Ceramill® fx Coloring Liquid for FSZ (Amann Girrbach). Specimens were sintered in a zirconia sintering furnace (Ceramill® Therm 3; Amann Girrbach) at a temperature of 1450ºC in the Sinter Program 2. In this program, which is recommended by the manufacturer for a single tooth restoration, the sintering process starts at room temperature with a constant heating rate of 720ºC/h (12ºC/min) until it reaches a final sintering temperature of 1450 ºC at which holding for 1 hour is maintained. Then, cooling at a rate of 20 ºC/min ensures the temperature to go down to 200 ºC at the end of the sintering cycle. After sintering, all discs were polished by the same operator on one side using a soft Robinson's brush (Abbott-Robinson's soft brush; Keystone Industries) and a zirconia polishing paste (Pearl Surface Z; Kuraray Noritake Dental Inc.). The brush was attached to a low-speed handpiece run by an electrically-powered motor at a standardized speed of 2000 rpm. Each specimen was fixed to a holder to be stable and made in contact with the fixed brush. Once contact was insured, the motor was turned on and a back-and-forth movement was made on the surface for three strokes, then, the motor was stopped, and another specimen was fixed to be polished in the same way. After polishing was done, all specimens were ultrasonically cleaned in an ultrasonic cleaner (VS350; Silfradent) in an ethyl alcohol bath for 10 minutes, air dried, packed and stored for translucency measurement.

An integrating-sphere reflective light-recording spectrophotometer (RM200QC; X-Rite, Neu-Isenburg) was used to assess the color parameters L*, a*, and b* on the polished side of each specimen before hydrothermal aging. Measurements were performed at the center of each specimen over a standardized white (CIE L*= 88.81, a*= -4.98, b*= 6.09) and black (CIE L*= 7.61, a*= 0.45, b*= 2.42) backgrounds relative to the CIE standard illuminant D65. Specimens were placed at the center of the measuring port and kept in the same position for the two backgrounds. Each specimen was measured three times on each of the two background, and an average of the three measurements was calculated to obtain one value for each of the color coordinates, for each specimen, on each of the two backgrounds. Translucency was assessed using the Translucency Parameter (TP) calculated based on the following equation: $TP=[(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{1/2}$. In this equation, $\Delta L=(L^*_B-L^*_W)$, $\Delta a^*=(a^*_B-a^*_W)$, and $\Delta b^*=(b^*_B-b^*_W)$, Where "B" and "W" are for the black and white backgrounds, respectively. Specimens were then cleaned again in the same ultrasonic cleaner using the same protocol, repacked with the same number initially assigned to each specimen, and were ready for the aging process. Hydrothermal aging was carried out in an autoclave (CBM, Model 4223; Castello, Torre de' Picenardi) at 134ºC, 2 bar steam pressure for 5 hours according to ISO standards 13356:2015 (45). Final translucency measurements for all specimens were done using the same spectrophotometer, on the same black and white backgrounds, using the exact same measuring protocol used before the hydrothermal aging process. Translucency of each specimen was assessed using the Translucency Parameter (TP) calculated from the same equation used in the pre-aging calculations. Now, each specimen had its TP values before and after hydrothermal aging recorded and tabulated on a computer software (Microsoft Excel spreadsheets, Microsoft), and became ready for statistical analysis.

Statistical analysis was done using software (Instat software for windows; Graph Pad). A P value of 5% was the limit of statistical significance ($P<0.05$). Continuous variables were expressed as mean +/- Standard Deviation (SD) at a 95% Confidence interval. After homogeneity of variance and normal distribution of errors have been confirmed using Kolmogorov-Smirnoff test, One-way analysis of variance (ANOVA) was performed to test statistical significance, followed by Tukey's pairwise test if showed significant. Student t-test was used within material type to analyze the effect of hydrothermal aging on each of the two materials. Power analysis was used to calculate sample size (n=10), which was proved to be statistically large enough to detect large effect sizes for main effects and pairwise comparisons, with the satisfactory level of power set at 80% and a 95% confidence level.

RESULTS

The effect of accelerated aging on each of the two materials was statistically analyzed using student t-test. For the fully-stabilized cubic zirconia, it was found that after aging, FX group recorded statistically significant higher mean TP value (17.39±1.32) than before aging which recorded a mean TP value of (15.51±1.99) as demonstrated by student t-test (p=0.0227< 0.05). For the partially-stabilized tetragonal zirconia, it was found that after aging, HT group recorded statistically significant higher mean TP value (14.24±1.6) than before aging which recorded a mean TP value of (12.82±1.46) as demonstrated by student t-test (p=0.023< 0.05) and presented in table (1) and figure (1).

Inter-group comparisons were made to analyze the translucency parameter of the two tested materials both before and after artificial accelerated aging. It was found that, before aging fully-stabilized cubic zirconia (FX group) recorded statistically significant higher mean TP value (15.51±1.99) than partially-stabilized tetragonal zirconia (HT group) which recorded a mean TP value of (12.82 ±1.46) as demonstrated by student t-test (p=0.003<0.05). After aging, the FX group recorded a statistically significant higher mean TP value (17.39±1.32) than HT group which recorded a mean TP value of (1 4.24±1.6) as demonstrated by student t-test (p=<0.0001< 0.05) and presented in table (1) and figure (1).

TABLE (1): Comparison of translucency parameter (TP) results (Mean±SD) between both groups before and after artificial aging.

Variable		***Before aging***	***After aging***
FX	*Mean±SD*	15.51^{Aa}±1.99	17.39^{Ab}±1.32
HT	*Mean±SD*	12.82^{Ba}±1.46	14.24^{Bb}±1.6
Statistics	***P value***	0.003*	<0.0001*

**; statistically significant (p < 0.05) Different uppercase letters in columns and lowercase letters in rows denote statistically significant difference.*

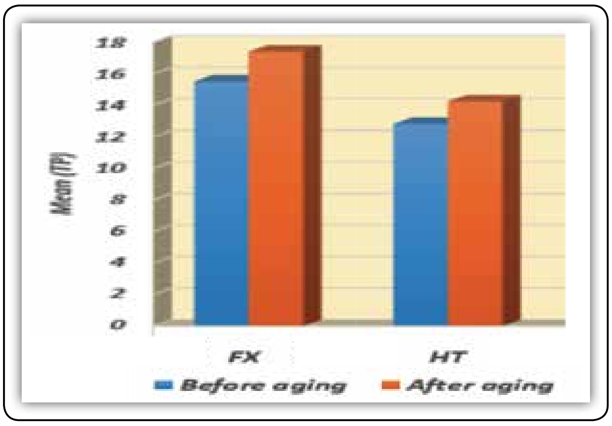

FIG (1) Mean TP values before and after accelerated aging for fully- (Zolid fx) and partially-stabilized (Zolid ht+) zirconia ceramics.

DISCUSSION

The ability of dental zirconia to withstand the oral environment changes and to maintain its properties arose as an issue of concern. In this study, the effects of accelerated aging of monolithic zirconia was investigated using an artificial accelerated aging process by autoclaving. The lower translucency anticipated from partially-stabilized tetragonal zirconia, compared to the more recent fully-stabilized cubic zirconia, is not the only concern regarding this type of zirconia ceramics. Another concern in the dental literature regarding the partially-stabilized tetragonal zirconia is its hydrothermal aging susceptibility and the ability of this zirconia to maintain its properties over time when serving in the tough oral conditions. The second material used in this study was a fully-stabilized cubic zirconia –i.e. Zolid fx-, which is usually used for the fabrication of more esthetically-demanding fixed dental restorations. It was of interest to shed a light on the difference in one of the most important optical determinants of success for dental restorations, i.e. translucency. Hence, the study of initial translucency of these two materials before they underwent a change that could be done by artificial accelerated aging is of interest. Each material was colored using its specific coloring liquid recommended by the manufacturer; Ceramill ® Liquid Cubical Zirconia for the FSZ Zolid fx, and Ceramill ® Liquid New Formula for the PSZ Zolid ht+. Both liquids were chosen to have the same A2 final shade. The A2 shade was selected as the nominal shade because it is a universal shade and the most commonly selected shade (11).

The hypothesis of the study that artificial accelerated aging will have a significant effect on the translucency parameter of the two types of monolithic zirconia at their minimal recommended thickness was accepted. The polycrystalline nature of zirconia can be a main cause in this result. When a light beam passes through a material, crystals within the microstructure of the substance interfere with light passage, deflecting the light beam and leading to increased scattering. This increased scattering and decreased light penetration leads to the less translucent appearance of a more crystalline structure. With the thickness of a zirconia being decreased to 0.5 mm, a favorable decrease in the number of crystals interfering with the passage of light through that restoration can be achieved, with an eventual increase in and enhancement of its translucency. This decreased thickness may have accounted for the detection of the slightest of changes within the zirconia structure after aging.

This result can be attributed to the structural differences and the difference in chemical composition between the two materials. The HT group represents a partially-stabilized tetragonal zirconia, whilst the FX group represents a fully-stabilized cubic zirconia. The FSZ FX group has a higher concentration of yttria (17) as a stabilizing oxide (between 9.15% and 9.55%) than the PSZ HT group. This increased yttria content has led to the development of FSZ in which the cubic lattice is the prevalent phase in a zirconia structure (32). The isotropic arrangement and the non-birefringent nature of cubic crystals makes for a more favorable light pathway through FSZ compared to PSZ (32). This means that the refractive index of a material changes as the direction of light propagation/transmission through that material changes. Both fully-stabilized cubic zirconia (FX group) and partially-stabilized tetragonal zirconia (HT group) showed a statistically significant higher mean TP value (17.39±1.32 for FX group, and 14.24±1.6 for the HT group) after aging than that of the same group before aging (15.51±1.99 for FX group, and 12.82±1.46 for the HT group). A possible scenario for this change might be due material structural changes due to degradation of the metal salts within the coloring liquid. After aging of zirconia, the breakdown of metal oxides in the coloring liquid could eliminate the influence of the coloring liquid (33). With the PSZ (HT group), spontaneous tetragonal to monoclinic transformation can be a possible cause for the material's change after artificial aging. The larger grains of the monoclinic lattice may cause less light interactions, which with this decreased thickness (i.e. 0.5 mm) could finally make this influence evident.

CONCLUSIONS

Within the limitation of the current study it can be concluded that: higher translucency can be obtained from fully-stabilized cubic zirconia compared to partially-stabilized tetragonal zirconia. Artificial aging affects the translucency of monolithic zirconia.

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