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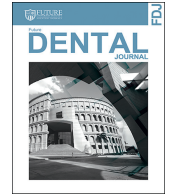
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### Cover Page Footnote

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## Effect of accelerated aging on translucency of monolithic zirconia



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### ABSTRACT

**Purpose:** The objective of the study was to evaluate the translucency of different thickness of a translucent zirconia before and after accelerated aging.

**Materials and methods:** Sixty slices of translucent zirconia were obtained by cutting InCoris TZI blocks into slices using Micracut precision cutting machine. The slices were divide into four groups ( $n = 15$ ) according to their thickness (0.5 mm, 0.8 mm, 1 mm and 1.2 mm). CIE lab coordinates were measured for each slice against black and white backgrounds using vita easy shade and TP was calculated. All specimens were subjected to accelerated aging using autoclave (134 °C, 0.2 MPa) and TP was calculated after accelerated aging. One way analysis of variance combined with a Tukey-post hoc test was used to analyze the data obtained ( $P = 0.05$ ).

**Results:** Results of the present study showed that thickness of zirconia has determinal effect on its translucency as there was no statistically significant difference in TP between 0.5 mm and 0.8 mm thicknesses while there were statistically significant differences in TP between 0.8 mm, 1.0 mm, 1.2 mm. Effect of aging was significant on 0.5 mm thickness.

**Conclusion:** Thickness of zirconia has significant effect on translucency. Aging has significant effect on thinner sections of zirconia. More research is required on zirconia towards making the material more translucent for its potential use as esthetic monolithic restoration.

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### 1. Introduction

The apparent color of natural teeth is the result of the reflectance from dentin modified by absorption, scattering and thickness of enamel. Therefore, understanding optical properties of teeth is imperative for accurate and consistent color reproduction [1]. Color and its elements such as Hue, Chroma, value, opacity, translucency, light transmission, scattering, metamerism and fluorescence influence the esthetics of a dental restoration. The eye is able to distinguish between artificial and natural teeth, based on minute differences in color and translucency [1,2].

The translucency is the quantity of light passing through a material and it is essential to the esthetic feature of dental restorations. Translucency occurs when a light beam passes through an

object, is partly reflected, scattered, and transmitted through the object, the more the amount of light passing through the object, the higher the translucency. Translucency is inversely related to the thickness of the ceramic restoration and the amount of light scattering that occurs within the body of the material. Scattering of light is affected by many factors such as different refractive indices among ceramic phases, voids, high crystalline content, and crystal number and size. Particle size has a marked effect on the translucency of ceramic materials, especially when particles forming the material are smaller than wave length of visible light [2–4].

The translucency of ceramic materials has been emphasized as one of the primary factors in governing the esthetic outcome of ceramic restorations. In clinical situations, ceramic restorations with various thicknesses are required. Therefore, a precise knowledge of the relationship between the translucency and thickness of restorative materials is essential to improving the esthetic outcome of dental restorations.

There are two methods to evaluate translucency and opacity of esthetic restorative materials; Absolute translucency by direct

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transmittance of light, and relative translucency using either the contrast ratio (CR) or the translucency parameter (TP) [4,5].

Translucency of a material is also described using the **translucency parameter (TP)**, which was developed to relate human visual perception to the translucency, since TP is defined as the color difference of a material of a given thickness over white and black backgrounds [5].

3Y-stabilized zirconia which has been shown to possess excellent mechanical properties, may not meet the esthetic requirements because of its poor translucency. This material has traditionally been opaque with a white to grayish appearance in its polycrystalline form [6].

The translucency of zirconia is controlled by several properties including grain size, distribution of grains, processing method, and additives. To obtain translucent zirconia, the size of crystals must be optimized along with a homogenous distribution grain size, for both excellent strength and minimal grain interfaces and minimal porosity which can reflect light and cause opacity. To ensure the highest translucency and esthetics, highly pure initial zirconia powder with minimal impurities must be used to allow for a higher relative light transmission [7].

Kim et al. [8] studied the effect of thickness reduction on color and translucency of dental monolithic zirconia ceramics using one hundred sixty five monolithic zirconia specimens divided into 5 groups according to the number of coloring liquid application. Each group was then subdivided into 11 subgroups by reducing the thickness up to 1.0 mm in 0.1 mm increments. The results showed the translucency generally increased as thickness reduction increased in all groups.

Little is known about color changes in ceramic restorations over time, as investigators focused on the mechanical properties of the material. However, the adverse conditions of the oral environment may cause changes in the physical properties of the material. Artificial accelerated aging is a method that simulates the clinical conditions to which materials are subjected, and allows the color difference of ceramic restorations over the course of time to be determined [9].

In a critical review Lughy and Sergio 2010 [10] defined Low temperature degradation (LTD) or aging as the spontaneous tetragonal to monoclinic (t–m) phase transformation occurring over time at low temperatures, when the t–m transformation is not triggered by the stresses produced at the tip of an advancing crack. LTD is a spontaneous tetragonal (T) to monoclinic (M) transformation taking place over time at low temperatures, this type of transformation is different from stress induced T-m transformation that occurs at the tip of a propagating crack within the zirconia, the former occurs at stress free sites while the latter takes place at stressed concentration sites. It is clear that, once transformed to the m-polymorph, zirconia cannot show phase transformation toughening (PTT), just like a used match cannot be lit again. Overall PTT and LTD are based upon the same phenomenon, the t–m transformation of zirconia, and, to date, we cannot exclude the remarkable possibility of the former without exposing ourselves to the risks of the latter.

Reported drawbacks of the LTD were enhanced wear rates [11] with loss of small surface zirconia grains in the surrounding environment causing an increase in surface roughness and surface uplifts with both mechanical [12] and esthetic worsening [10].

Although hundreds of laboratory researches dealt with the effect of aging and its theoretically assumed mechanisms on the mechanical outcome and serviceability of the material [10] no studies to our knowledge have discussed the esthetic and color changes upon such phenomenon. The clear realization that the same microstructure that causes certain mechanical properties to manifest in zirconia is also responsible for its optical properties.

Thus it's fair to conclude that aging related changes in the microstructure that leads to changes in the mechanical properties would also lead to optical changes. However through studying of the factors controlling LTD and its effects on the material microstructure and surface, esthetic outcome and stability of the material with its variable products and processing techniques may to a great extent be affected.

The purpose of this study was to investigate the effect of different thicknesses and accelerated aging on translucency of monolithic zirconia. The null hypotheses tested were 1) differences in thickness have no significant effect on translucency of monolithic zirconia. 2) Accelerated aging has no detrimental effect on translucency of monolithic zirconia.

## 2. Materials and methods

Sixty translucent zirconia specimens were divided into four groups (n = 15) according to their thickness (Group A 0.5 mm, Group B 0.8 mm, Group C 1 mm and Group D 1.2 mm). Translucent zirconia specimens were obtained by cutting four InCoris<sup>1</sup> TZI blocks (40 × 19 × 15 mm), using IsoMet 30HC precision cutting blade<sup>2</sup> (Diameter 127 mm, thickness 0.76 mm) mounted on MICRACUT 150/201 precision cutter.<sup>3</sup> The machine was adjusted using its built in micrometer to cut the InCoris<sup>1</sup> TZI block under water coolant into slices of approximately 0.6 mm, 1 mm, 1.2 mm, and 1.5 mm thickness, 20% larger than the desired final size to compensate for sintering shrinkage caused during the sintering stage. The other dimensions of the slice were 19 mm length and 15 mm width.

The thickness of each slice was checked with a digital caliper<sup>4</sup> and minor corrections were done using Wetordry<sup>5</sup> Sandpaper sheet of grit 400, 600, and 1200 in the presence of water to ensure flat surface and desired thicknesses.

The slices were then ultrasonically cleaned for 10 min in distilled water then air dried with oil free compressed air and left for 24 h for complete dryness.

Prior to sintering the InCoris TZI slices were fully submerged in a plastic vessel containing A2 InCoris<sup>1</sup> TZI Sirona coloring liquid<sup>1</sup> allowing the slice disc to absorb the coloring solution for 5 min then the discs were removed from the plastic vessel using a pair of plastic tweezers. The recently colored slices were then placed on a glass slab for 2 h for drying.

The InCoris<sup>1</sup> TZI slices were placed on sintering boats filled with the sintering beads, at least 1 cm apart from each other, then sintered in Sirona inFire HTC<sup>1</sup> speed with the pre-programmed InCoris TZI programs. The program was started to run automatically for 90 min and 1540 °C sintering temperature. After sintering, the thickness of each slice was checked with a digital caliper. The final thicknesses of the slices were 0.5 mm, 0.8 mm, 1.0 mm, and 1.2 mm.

Polishing was carried out by low speed hand piece and an electric motor with a rate of 7000–10000 rpm under constant water coolant using polishing kit (K0262 Dialite ZR Intra-Oral Adjustment finishing and polishing system<sup>6</sup>) and polishing paste ZI-Polish in the following sequence Green course grinder, Green medium polishing points for pre-polishing, Orange fine polishing points for high luster and finally (ZI-Polish<sup>2</sup>) diamond polishing

<sup>1</sup> Sirona, Bensheim, Germany.

<sup>2</sup> Buehler, Illinois, USA.

<sup>3</sup> METKON Precision Cutting Machines, Turkey.

<sup>4</sup> 8" vernier caliper (Mitutoyo, Tokyo, Japan).

<sup>5</sup> 3M, St. Paul, USA.

<sup>6</sup> Brasseler (Br)USA.

**Table 1**  
Tests of between-subjects effects.

Source	Type III sum of squares	df	Mean square	F	Sig.
Corrected model	356.321	7	50.903	13.649	0.000
Intercept	14778.760	1	14778.760	3962.753	0.000
Thickness	259.693	3	86.564	23.211	0.000
Aging	60.027	1	60.027	16.096	0.000
Thickness × Aging	47.700	3	15.900	4.263	0.007
Error	372.942	100	3.729		
Total	16942.537	108			
Corrected Total	729.263	107			

df: degrees of freedom = (n – 1), Significant at P ≤ 0.05.

paste was applied with a special brush (Wheel Brush Rodeo<sup>7</sup>) that is double loaded with natural horse hair for best results of finalizing the surfaces.

All the specimens were tested for degree of translucency using portable intraoral digital spectrophotometer (Vita EasyShade<sup>8</sup>).

Vita easy shade in “tooth single” mode was used to determine the values of CIELab coordinates from specimen placed on white and black background. Three measurements were taken for each specimen on white and black backgrounds and the average of each parameter (L\*, a\* and b\*) was recorded. The values were used to calculate the translucency parameter (TP) according to the following formula: [13].

$$TP = [(L_b - L_w)^2 + (a_b - a_w)^2 + (b_b - b_w)^2]^{1/2}$$

where b is for black and w for white.

All 60 Zirconia slices were subjected to artificial accelerated aging using autoclave [14] at 134 °C, 0.2 MPa for 5 h according to ISO standards 13356.

Each slice was placed in a sterilization pack labeled with the ceramic material type, thickness, and specimen number, after sealing of the sterilization packs the specimens were placed in the autoclave.

Using the same intraoral spectrophotometer the values of CIE-Lab coordinates from each specimen were recorded and translucency parameter (TP) was calculated after artificial aging using the same formulas as before aging.

### 3. Statistical analysis

Data were explored for normality by checking data distribution, histograms, calculating mean and median values and finally using Kolmogorov-Smirnov and Shapiro-Wilk tests. All data showed parametric (normal) distribution and were presented as mean and standard deviation values.

The significance level was set at P ≤ 0.05. Statistical analysis was performed with IBM<sup>9</sup> SPSS<sup>10</sup> Statistics Version 20 for Windows.

### 4. Results

The results showed that material thickness, aging and the interaction between the two variables had a statistically significant effect on mean TP. Since the interaction between the three variables were statistically significant, so the variables were dependent upon each other and we compared between different levels within each variable (See Table 1).

#### 4.1. TP measurements before and after aging

Results of the present study showed that there was no statistically significant difference in TP between 0.5 mm and 0.8 mm thicknesses (16.25 ± 0.75 and 13.67 ± 1.21 respectively) while there were statistically significant differences in TP between 0.8 mm, 1 mm and 1.2 mm (13.67 ± 1.21, 11.49 ± 0.95 and 9.25 ± 1.45 respectively) (Table 2). (Fig. 1).

After aging only the 0.5 mm specimens showed statistically significant difference than before aging (12.56 ± 1.36), while 0.8, 1, and 1.2 mm thicknesses there were statistically insignificant after aging (13.24 ± 2.42, 11.08 ± 1.33 and 9.74 ± 1.31 respectively). (Table 2).

### 5. Discussion

Translucency is an important parameter in matching the appearance of the natural tooth and was identified as one of the primary factor in controlling esthetics and a critical consideration in the selection of materials [15].

Translucency is generally measured with contrast ratio (CR) or translucency parameter TP. CR is the ratio of the reflectance of a specimen over a black background to that over a white background of a known reflectance, and is an estimate of opacity [16]. CR ranges from 0 to 1, with 0 representing transparency (total translucency) and 1 representing to total opacity (no translucency). The TP is the ΔE (difference in color) between a uniform thickness of a material measured over white and black backgrounds. Although CR and TP for translucency measurement of dental ceramics is matter of discussion, at present they still represent a well-established parameter [13].

The Lambert's law  $T = e^{-ax}$  identifies that the transmittance (T, a physical parameter representing the ability of light to pass through certain medium) decreases exponentially with increasing thickness (x). Therefore, thickness is a key factor relating to light transmittance.

The results of the present study showed that the material thickness had a significant impact on TP; the translucency was significantly increased as a result of decrease in thickness for translucent zirconia, 0.5 mm thickness showed the highest mean TP (16.12 ± 0.75) and the 1.2 mm thickness showed lowest mean TP (9.25 ± 1.45). There was no significant difference in TP between 0.5 mm and 0.8 mm (16.25 ± 0.75 and 13.67 ± 1.21 respectively) which is most probably attributed to the small difference in thickness (Table 2).

These results were in accordance with several studies which investigated the relationship between the thickness and translucency of ceramics. **Antonson and Anusavice**, [17] suggested that there was a linear relationship between contrast ratio of dental ceramics to their thickness. **Rasetto et al** [18] found a significant decrease in light transmission with increase in thickness of ceramics.

<sup>7</sup> Bredent, GmbH & Co.KG.

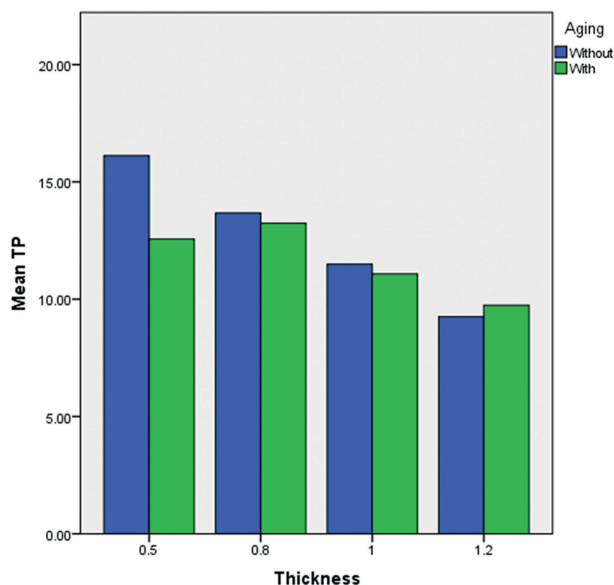
<sup>8</sup> VITA Zahnfabrik, Germany.

<sup>9</sup> IBM Corporation, NY, USA.

<sup>10</sup> SPSS, Inc., an IBM Company.

**Table 2**  
Effect of aging within each thickness on TP. Same letters show no statistically significant difference.

TP	Thickness 0.5				Thickness 0.8				Thickness 1				Thickness 1.2			
	Aging without		Aging with		Aging without		Aging with		Aging without		Aging with		Aging without		Aging with	
	Mean	std	Mean	std	Mean	std	Mean	std	Mean	std	Mean	std	Mean	std	Mean	std
	16.12 <sub>a</sub>	0.75	12.56 <sub>b</sub>	1.36	13.67 <sub>a</sub>	1.21	13.24 <sub>a</sub>	2.42	11.49 <sub>c</sub>	0.95	11.08 <sub>c</sub>	1.33	9.25 <sub>d</sub>	1.45	9.74 <sub>d</sub>	1.31



**Fig. 1.** Bar chart representing mean TP for each thickness before and after aging.

Wang et al. [19] showed that the translucency of dental ceramics was significantly influenced by both material type and thickness; there was an increase in translucency parameter with decrease in thickness. Cekis-Nagas et al [20] indicated that greater thickness of zirconia results in lower light transmission. Awad et al [21] found that doubling the thickness of ceramic material results in large decrease in translucency irrespective of the type of material. Moreover the total refractive index of a ceramic material is directly related to the thickness of the material [15]. Therefore, it is evident that thickness is a coverable of light transmission through ceramic specimens ranging from 0.5 mm to 1.2 mm.

These findings can be explained by increased light absorption, scattering, and decreased transmission of light with increased ceramic thickness as thinner ceramic permits more light transmission, simply due to fewer light/grain-boundary interaction [22].

A large role in the transparency of zirconia is played by density (porosity). Pores larger than 50 nm cause significant scattering and thus reduction of light transmission (“pore scattering”) [23]. Using the spark plasma sintering technique, Alaniz et al. [24] used nanocrystalline 8 mol% yttria-stabilized zirconia powder with a grain size of 50 nm to produce transparent specimens of different colors, according to the holding time at the final pressure and temperature. Specimens of amber brown, dark orange, and ruby-red colors were obtained, each one with high optical resolution [24]. The authors in the same paper proposed in fact that in high-density nanocrystalline zirconia, it is unlikely that pore diameter would be greater than grain size, and the use of <50-nm grain zirconia reduces the problem of light scattering [24]. The suggestion that pores can be the main factor that affects the translucency

was also proposed by Jiang et al. [25] The authors demonstrated that using 40-nm powder instead of 90-nm powder reduces pores, improves the sintered density and reduces scattering. Sintering temperature also plays a primary role in determination of sintered density [26]. The authors have shown that the transmittance increases with sintering temperature (from 1350 to 1500 °C), and at the same temperature the transmittance of 40-nm powder was higher. With the increasing temperature, the zirconia crystal structure becomes more compact, whereas porosity, defect, and flaws decrease [25].

When comparing the translucency of different materials, data should not be evaluated only from a statistically viewpoint, as the statistically significance parameter alone could not be representative of the clinical perception of translucency. Concerning CR, the mean minimal difference perceivable by the human eye was defined as the translucency perception threshold (TPT) by Liu et al. [27] and it was found to be 0.07, but with significant variations according to the skill of the observer. Clinician having 10 years of shade-matching experience can have a TPT of 0.04, whereas dental students were in the range of 0.09. Besides the CR, the TP is also used to identify the translucency of dental materials [28,29]. Recently, Barizon et al. [30] found a correlation between CR and TP, concluding that either CR or TP can be used to evaluate the relative translucency of ceramic systems. Conversely for CR, for TP the limit of clinical acceptability has not yet been identified, and it is uncertain whether the differences reported could be clinically relevant, even if the correlation between CT and TP tentatively supports the same conclusions [15].

From a clinical viewpoint, translucency is a key factor in material selection, but its evaluation should be flanked by flexural resistance evaluation. Lowering the thickness of the restoration would allow more translucency of the material, thus more aesthetic, but at the same time less resistant to fracture. On the contrary, by increasing the thickness, the fracture resistance of the material will be increased but the translucency will be decreased, limiting its esthetic performances. Concerning monolithic zirconia, the use of which shows a fast-growing interest by clinicians, the minimal feasible thickness for clinical use, taking into account the clinical masticatory forces in terms of flexural resistance, could be considered at about 0.5 mm [26]. At the same time, with conventional zirconia, the aesthetic outcome is limited by the opacity of the material beyond about 0.75-mm thickness [31], being this the thickness at which a traditional zirconia matches the opacity of dentin [31,32]. Thus, with zirconia being liable to break below 0.5 mm and too opaque over about 0.75 mm, the interval 0.5 < ZrO2 < 0.75 mm could be considered a clinical range for the use of monolithic traditional zirconia combining resistance and aesthetic [15].

Aging had significantly affected the TP of zirconia specimens at 0.5 mm thickness (12.56 ± 1.36 after aging compared to 16.12 ± 0.75 before aging) while 0.8 mm, 1 mm and 1.2 mm thicknesses were not significantly affected (13.24 ± 2.42, 11.08 ± 1.33 and 9.74 ± 1.31 after aging compared to 13.67 ± 1.21, 11.49 ± 0.95 and 9.25 ± 1.45



before aging respectively) Table 2, which could be explained by the fact that thicker specimens contains more zirconia grains per unit volume than thin specimens, higher percentage of zirconia grains that were affected by LTD in 0.5 mm thickness compared to the percentage of grains affected in thicker specimens in other words LTD always starts at the surface of the specimens and the surface of the 0.5 mm specimens has a greater percentage of the total volume of the specimen compared to thicker sections. so changes in microstructure caused by LTD such as; release of small zirconia grains, surface uplifts, grains pull-out and surface roughening [10] have more pronounced effect on thin sections. This may be the most accepted reason behind decreased transmittance and increased scattering centers.

## 6. Limitation of the study

To better understand the clinical appearance of monolithic zirconia the effect of cement and long term color stability should be investigated in addition, further analysis with advanced imaging and microstructure analysis may be beneficial to precisely demonstrate the aging dynamics.

## 7. Conclusions

Within the limitations of this study the following can be concluded:

- Thickness of the restorations has determinial effect on its translucency, as the thickness increases translucency decreases.
- Aging has significant effect on translucency of zirconia in thin sections.

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