

Effect of Zirconia Pigmentation on Translucency

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ABSTRACT

Purpose: the aim of this study was to investigate the effect of shading on translucency and mean grain size of three Zirconia systems (Lava Frame, Ice Zirkon Translucent and Prettau Zirkon). *Materials and Methods:* specimens pigmented in A1, A4 and a non-pigmented specimen was evaluated with Scanning Electron Microscopy and spectrophotometry. *Results:* the transmittance ranged between 0.18 and 0.52, in Ice Zirkon Translucent A4 and Prettau Zirkon non-pigmented, respectively. The pigmentation A1 and A4 significantly reduced the light transmission in all the systems evaluated ($p < 0.05$), with the exception of Lava A1. The light transmittance of each ceramic system is different from each other in the three pigmentations, except for the Prettau Zirkon A1, which did not show a significant difference compared with Lava A1 ($p > 0.05$). The mean grain size increased with pigmentation. The mean grain size ranged between 0.43 μm , for Prettau Zirkon non-pigmented, and 0.82 μm for Prettau Zirkon A4. *Conclusion:* Light transmission and mean grain size was affected by pigmentation of dental zirconia ceramics.

INTRODUCTION

Due to mechanical properties, yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics can be used as an alternative for conventional metal frameworks.¹ While it's the strongest ceramic ever used, its translucency remains a disadvantage.² The translucency of zirconia becomes a problem, especially when placing a crown in the presence of other anterior natural teeth. In order to have a biomimetic result, space for a porcelain veneer thick enough to cover the zirconia core means a substantial reduction of tooth structure, which is not always possible. Besides that, veneering undergo chipping and to prevent veneer chipping, monolithic zirconia is often used in full arch restorations, posterior crowns and fixed dental prostheses.^{3,4} In monolithic, wherein the thickness is bigger, the opacity of zirconia can pose a problem.⁵

Zirconia is more opaque than other dental ceramic materials as a consequence of its high crystalline content and is white in color.^{2,6,7} To achieve more natural looking properties for the veneering of fixed partial dentures and crowns, the zirconia frameworks can be shaded before sintering.¹ Some colorant oxides of Fe, Cu, Co, Mn and opacifying oxides of Sn, Zn, Al, Zr, and Ti are added allowing, a more natural appearance, similar to the yellow dentin overlaid by translucent enamel.⁸ It was shown that this addition of metal oxides has the potential to lead to crystallographic and microstructural changes that could affect the mechanical and optical properties.^{9,10}

In addition to all these aesthetic considerations, the success of a ceramic restoration depends primarily on the durability of the bond between ceramic and cement, and respectively between cement, enamel and dentin.

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Adhesive cementation of zirconia restorations is possible when used resin cement systems with acidic group (phosphate, carboxylate)^{11,12} and have several advantages including reduced marginal microleakage and reduced occurrence of secondary caries.¹³ Dual resin cements would exhibit increased polymerization if a zirconia with superior light transmission was used and this probably affects the longevity of the rehabilitation.

Translucency of zirconia is determined by a complex combination of different parameters, including residual porosity,^{5,7} grain size,⁶ crystalline structure,⁷ number of ceramic firings and repeated ceramic staining cycles,¹⁴ additives such as alumina⁵ or other oxides such as cerium and hafnium are known to reduce translucency in zirconia, sintering conditions¹⁵ and thickness.²

Since the literature report that shading of zirconia has the potential to lead to crystallographic and microstructural changes, it is relevant to evaluate if translucency is affected.^{9,10}

The aim of this study was to determine whether the coloring affected the translucency of zirconia. The two null hypothesis tested were that the coloring and the type of zirconia used, would not affect the translucency. Additionally, we analyzed the influence of coloring in the microstructure of different zirconia systems.

MATERIALS AND METHODS

SPECIMEN PREPARATION

Yttrium partially stabilized green-stage zirconium dioxide blocks (ICE Zirkon Translucent, Prettau Zirkon and Lava Frame) (Table 1) were cut into rectangular-shaped specimens (10×20 mm) of 0.8 mm thick with a low-speed diamond saw (ISOMET, BUEHLER, Illinois, EUA) under a constant flow of water. The specimens were then machine-polished on both sides sequentially with 200, 400 and 600 wet silicon carbide grinding papers until the thicknesses were reduced to 0.75

mm measured using a digital caliper (Mitutoyo Digimatic, Tokyo, Japan).

Subsequently, the specimens of each zirconia system were randomly divided according to pigmentation (non pigmented, pigmented A1 and pigmented A4) (Table 1). Therefore, 9 experimental groups were set to (n=5).

The sample size (n) was determined, taking into account the results of a pilot study conducted previously, establishing $\alpha=0.05$ and the power test (β) of 0.8.¹⁶

Lava specimens pigmented A1 and A4 were immersed for 10 seconds in the SF1 and SF4 solutions, respectively, and then sintered, according to the manufacturer's instructions, for 8 hours and 30 minutes with a final temperature of 1500°C.

Pigmented A1 and A4 Ice Zirkon Translucent specimens were immersed for 10 seconds in the color liquid A1 and A4, respectively, and then dried under a warming lamp for 45 minutes (Zirkonlampe 250, Zirkon zahn, Gais, Italy). Sintering was done, according to the manufacturer's instructions, for 3 hours at a heating rate of 8°C/min with a final temperature of 1500°C remaining for 2 hours before cooling.

Prettau Zirkon specimens pigmented A1 and A4 were immersed for 10 seconds in the color liquid A1 and A4, respectively, and then dried for 45 minutes with infrared drying lamp (Zirkon lampe 250, Zirkonzahn, Gais, Italy). Sintering was done, according to the manufacturer's instructions, for 3 hours at a heating rate of 8°C/min with a final temperature of 1600°C remaining for 2 hours. At the end of the sintering cycle, all the specimens were furnace-cooled to room temperature.

Specimens not shaded were sintered in the same conditions. The sintering process resulted in approximately 20% shrinkage, being the final thickness of the specimen's 0.6 mm ± 0.03 mm.

Before measurement of transmittance, specimens were ultrasonically cleaned in distilled water for 10 minutes and then dried with compressed air.

Table 1. Overview of the experimental groups

Product	Shade (Color liquid)	Batch number	Manufacturer
Lava Frame	Non pigmented A1 (SF1) A4 (SF4)	392508	3M ESPE, St. Paul, MN, USA
Ice Zirkon Translucent	Non pigmented A1 (color liquid A1) A4 (color liquid A4)	ZA9112E	Zirkonzahn, Zirkonzahn, Gais, Italy
Prettau Zirkon	Non pigmented A1 (Aquarell A1) A4 (Aquarell A4)	ZA849A	Zirkonzahn, Zirkonzahn, Gais, Italy

TRANSLUCENCY TEST

Direct light transmittance measurements were performed in the 300 to 700 nm wavelength range using a UV-Vis (ultraviolet-visible) spectrophotometer (Helios Alpha, Thermo Electron Corporation, USA). Each specimen was positioned in the spectrophotometer and a black rectangular cardboard segment (4 x 4 cm) with a central orifice of 10 mm in diameter was used to position the specimens in front of the sphere holder. Data were recorded with a computer connected to the spectrophotometer, and a graph of light transmittance percentage per nanometer was obtained for each specimen by using the software (Vision Pro, Cognex Corporation, Natick, USA). Transmittances at a wavelength of 468 nm – corresponding to the maximum absorption of the photoinitiator camphorquinone – of each group were analyzed.

MICROSTRUCTURAL ANALYSIS

After translucency tests, one specimen for each group was randomly selected and analyzed with a scanning electron microscope (JSM 7001, JEOL, Tokyo, Japan) to microstructural characterization. The specimens were ultrasonically cleaned (Elmasonic One, Elma, Singen, Germany) for 5 min in 80% ethanol and then air-dried. Subsequently, each specimen was gold sputtered for 80s (JFC 1100, JEOL, Tokyo, Japan). The analysis was carried out using an acceleration voltage of 20 kV and a magnification of 200-10000x. The mean grain size was measured according to the linear intercept method of ASTM Standard E112.¹⁷ For each specimen, 8 line segments were taken into account in one image analysis on SEM photomicrographs with 10000 x magnification using Matlab (Mathworks 7, Natick, USA).

STATISTICAL ANALYSIS

Data were statistically analyzed with the software (IBM SPSS Statistics 20; SPSS Inc, Chicago, USA). The results of transmittance (0.0–1.0) at a wavelength of 468 nm were submitted to two-way analysis of variance (ANOVA) followed by one-way ANOVA and Tukey's HSD multiple comparison tests, according to pigmentation and ceramic system, at a significance level of $p < 0.05$.

RESULTS

The result of the mean transmittance for 468 nm of each group is summarized in Table 2.

There was a statistically significant influence of Zirconia systems ($p < 0.001$) and pigment ($p < 0.001$) for the transmittance (Table 3). It was also observed that there was a statistically significant interaction between these two factors ($p < 0.001$) (Table 3).

All ceramic systems are different ($p < 0.05$) from each other in the three pigmentations, except for the Prettau Zirkon A1, which did not show statistically different from Lava Frame A1 ($p = 0.687$) (Figure 1).

For all the ceramic systems studied, the A4 pigmented specimens had a lower transmittance compared with specimens non-pigmented and A1 pigmented ($p < 0.05$). A1 pigmentation specimens had a lower transmittance compared with non-pigmented specimens, except for those manufactured in Lava Frame, which did not show a statistically significant difference ($p = 0.091$).

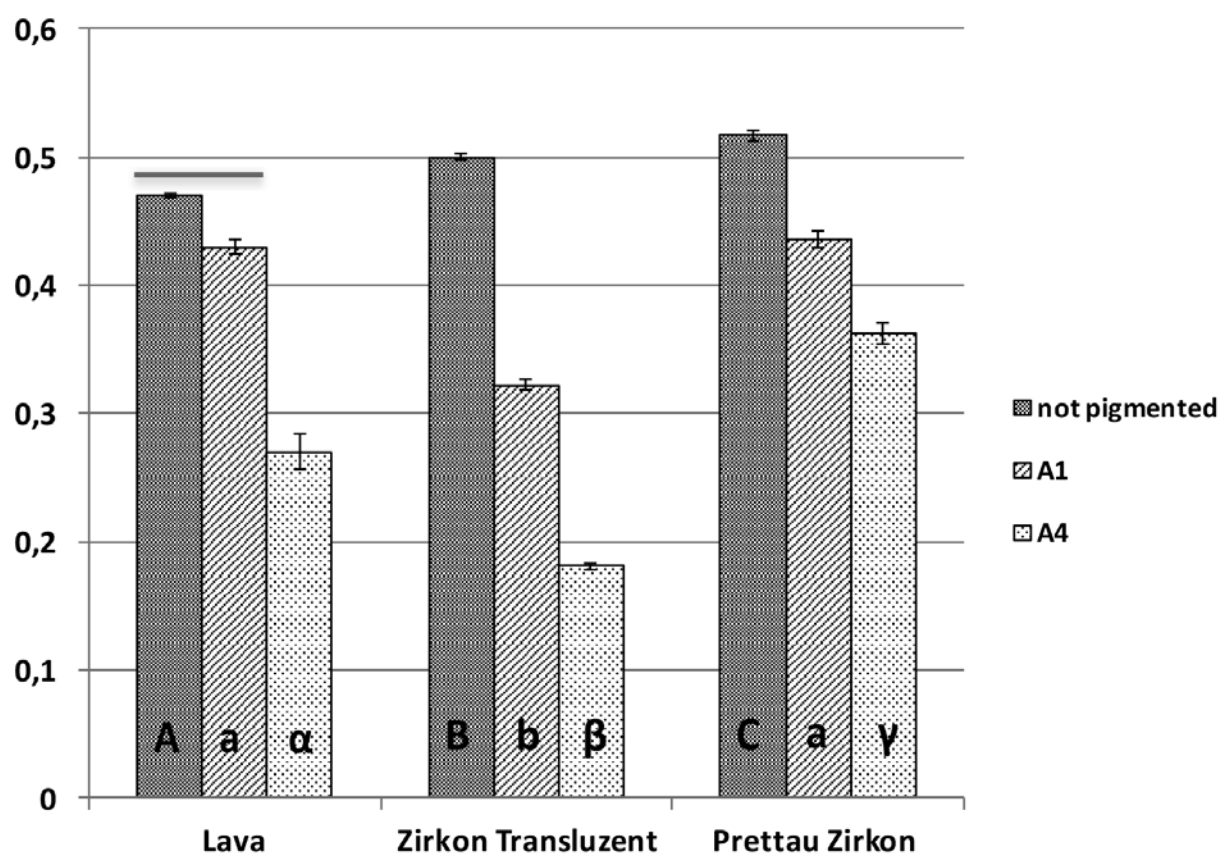
The mean grain size ranged from 0.43 μm for non-pigmented Prettau Zirkon to 0.82 μm for Prettau Zirkon A4. The mean grain size for non-pigmented was 0.52, for A1 was 0.58 and for A4 was 0.74 (Figure 2). Significantly negative Spearman correlations were observed between transmittance and grain size $r = -0.716$ ($p < 0.05$) (Figure 3).

Table 2. Transmittance and standard deviation values obtained for 468 nm wavelength

Group	Mean Transmittance 468 nm (standard deviations)	Mean grain size (μm) (standard deviations)
Lava (non-pigmented)	0.47 (± 0.003)	0.64 (± 0.001)
Lava A1	0.43 (± 0.010)	0.53 (± 0.003)
Lava A4	0.27 (± 0.027)	0.69 (± 0.007)
Ice Zirkon Translucent (non-pigmented)	0.50 (± 0.005)	0.50 (± 0.001)
Ice Zirkon Translucent A1	0.32 (± 0.007)	0.60 (± 0.001)
Ice Zirkon Translucent A4	0.18 (± 0.004)	0.72 (± 0.003)
Prettau Zirkon (non-pigmented)	0.52 (± 0.007)	0.43 (± 0.002)
Prettau Zirkon A1	0.44 (± 0.014)	0.62 (± 0.001)
Prettau Zirkon A4	0.36 (± 0.016)	0.82 (± 0.001)

Table 3. Two-way ANOVA for light transmittance of specimen. (Values with * are significantly different, $P < 0.05$)

ANOVA table	Sum of square	Df	Mean square	F value	P value*
Pigmentation	3.177	2	1.588	932.341	<0.001*
Zirconia system	0.897	2	0.448	263.232	<0.001*
Pigmentation/ zirconia system	0.637	4	0.159	93.469	<0.001*
Residual	0.061	36	0.002		
Total	49.277	45			

**Figure 1:** Transmittance of the several experimental conditions. (For each ceramic system, pigmentations under the same horizontal line are statistically similar; for each pigmentation, ceramic systems with the same letter are not statistically different).

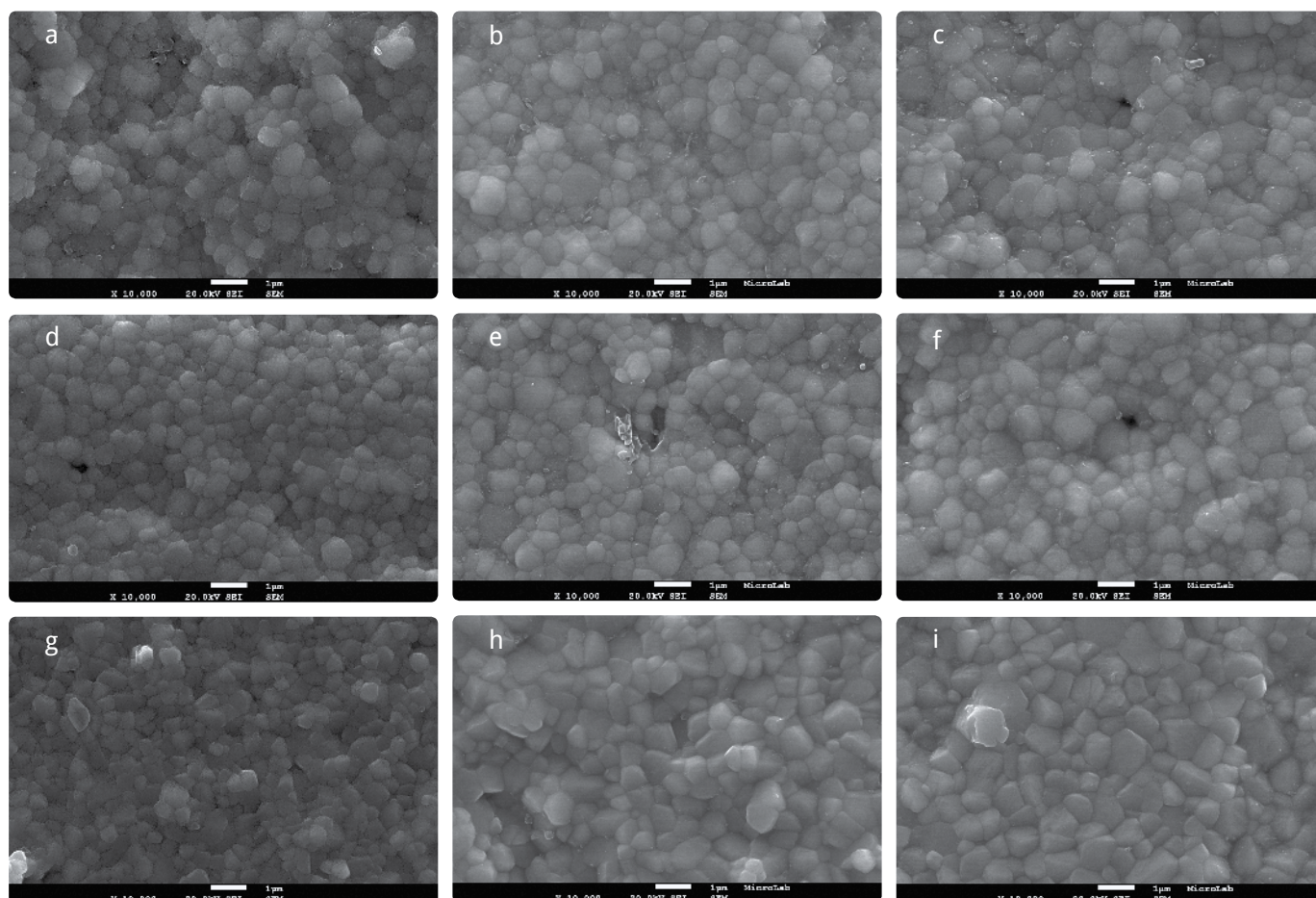


Figure 2: Scanning electron microscope images of zirconia. (A) Lava, (B) Lava A1, (C) Lava A4, (D) Ice Zirkon Translucent, (E) Ice Zirkon Translucent A1, (F) Ice Zirkon Translucent A4, (G) Prettau Zirkon, (H) Prettau Zirkon A1, (I) Prettau Zirkon A4.

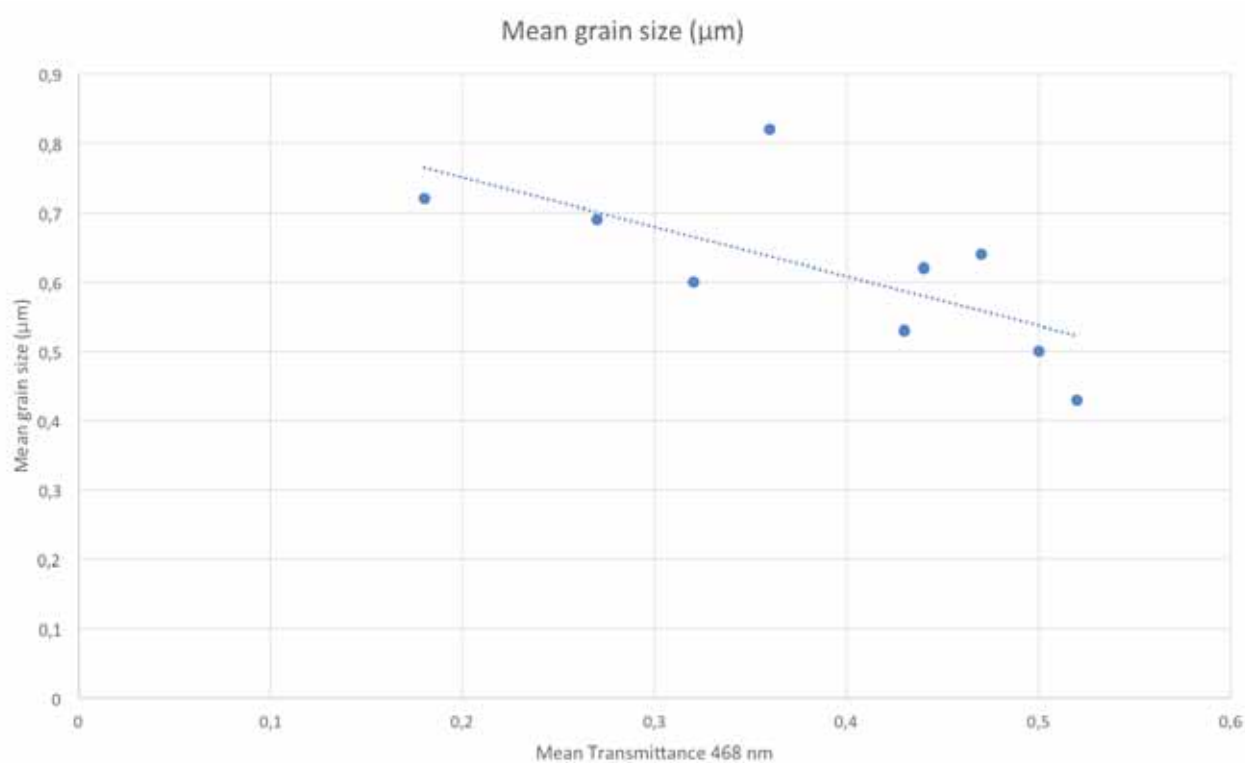


Figure 3: Scatter plots for Spearman correlation between the transmittance and grain size.

DISCUSSION

The data obtained in this study support rejection of the null hypothesis, as the translucency of zirconia is affected by pigmentation and zirconia system.

Differences in translucency can be explained by differences in structure and chemistry that influence the interaction with light.^{5,18} The amount of light that is absorbed, reflected and transmitted depends on the quantity of crystals within the core matrix, and the size of the particles compared to the incident light wavelength.^{19,20} Materials composed of small particles (approximately 0.1 μm in diameter) are less opaque when visible light passes through it.⁵ For maximal scattering and opacity, dispersed particles slightly greater in size than the wavelength of light, are required.^{7,20} According to this, it was found that the groups with major differences between the light wavelength and size of particles (Lava Frame A4, Ice Zirkon Translucent A4 and Prettau Zirkon A4) had smaller transmittance.

The mean grain size of the current commercial Zirconia varies from 0.2 to 0.8 μm , and the present results are in agreement with literature.⁵ Grain size could be modified by varying sintering conditions, like pressure, temperature, and time. Higher temperatures and longer sintering times produce larger grain sizes.^{21,22} Besides sintering conditions, the immersion in coloring solutions, has been cited to increased grain size^{19,10} and this was observed in the present study. As explained above, mean grain size affects translucency and was observed a negative correlation between grain size and transmittance, previously reported.^{5,23} We observed that higher transmittance was obtained in groups with smaller grain size numbers and also a diminished translucency with pigmentation of all the brands evaluated was observed. This can be the consequence of increased mean grain size and the presence of elements like oxides of Fe, Cu, Co, Mn and opacifying oxides of Sn, Zn, Al, Zr, and Ti (added to obtain a yellow to brown shade that matches that of the natural teeth) with different refractive index than zirconia cause scattering of light and consequently decreases translucency.^{5,23,24}

Although pigmentation reduces the translucency in all brands availed, even the shaded zirconia allowed the light to pass through the material, in contrast to the results of early studies on this matter.^{6,7,24} Previous research found that the first zirconia systems were totally opaque, with values equal to those of metal-ceramics.^{6,25} However, it is known that the most significant sources that diminish the translucency of 3Y-TZP are light scattering at the grain boundaries, pores, and secondary phases.⁵ Measures have been taken by the dental industry to reduce the porosity and to eliminate sintering additives, such as alumina. As a result, more recent studies showed that modern zirconia allowed the light to pass through it.^{5,18,26}

The present results show a significant attenuation transmittance through almost all shaded zirconia and all zirconia systems evaluated have dissimilar optical properties. In a clinical situation, this may account for lower light energy reaching the cement layer during luting procedures of shaded zirco-

nia, potentially resulting in poor polymerization.^{27,28} This can lead to poor mechanical properties of the cement, which may result in debonding of ceramic restoration over time.²⁹⁻³¹ An opportune future study would evaluate the effect of these variables in the polymerization of light cured resin cement. Yet, according to the results, some zirconia system may be more appropriate for anterior rehabilitation for allowing better biomimetic effect.¹⁸

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

- Light transmission was significantly affected by pigmentation.
- Except for Lava system, pigmentation A1 and A4 decrease significantly with light transmission.
- Prettau Zirkon system showed highest translucency except for the A1 pigmentation.
- Ice Zirkon Translucet system showed minor translucency in all pigmentations.
- Mean grain size is affected by pigmentation, with exception of Lava system, increased grain with pigmentation was observed.

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