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Influence of Layer and Sintering Temperature on the Optical Properties of Multi-layered Zirconia Materials

Keywords

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Translucency
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ABSTRACT

Objective: To evaluate the translucency and color difference of the individual layers of two multi-layered zirconia materials at different sintering temperatures and to compare them with lithium disilicate. Methods: Multi-layered zirconia systems with four distinct layers were selected for this study: DD cube ONE ML (4Y-TZP), DD cubeX2 ML (5Y-TZP); and compared with IPS e.max CAD HT (LS$_2$). Plate-shaped A2-shade specimens were obtained from LS$_2$ and individual layers of both zirconia materials. Individual layers were then divided equally into three different sintering temperatures:1300°C, 1450°C, and 1600°C. The TP and ΔE were determined by a spectrophotometer. SEM images were taken. Data was analyzed using SPSS 24.0 software with a p-value <0.05. Results: A significant difference was found in TP and ΔE values amongst all types of ceramic materials. Different sintering temperatures yielded distinct TP and ΔE values when both zirconia materials were tested and compared with LS$_2$. Finally, TP and ΔE values were different amongst the zirconia layers. Conclusion: Sintering temperature, type of ceramic material and different zirconia layers significantly affected the optical properties. Clinical Significance: Multi-layered zirconia materials possess a unique gradient effect that could efficiently enhance the esthetics of monolithic zirconia restorations. However, the sintering condition should be optimized.

INTRODUCTION

In the last decade, monolithic zirconia dental restorations have become a popular choice of restorative material amongst dental clinicians.[1–3] This is mainly due to the many advantages associated with utilizing zirconia dental materials such as the ability to produce zirconia restorations in thinner sections compared to other ceramic materials resulting in less need for extensive preparation designs. Other advantages include biocompatibility, efficient laboratory procedure, excellent mechanical properties and acceptable esthetics.[4,5] However, when restoring esthetically visible zones, clinicians tend to layer zirconia with a glass ceramic material to enhance the esthetic outcome and avoid the opaque appearance accompanied with using monolithic zirconia restorations.[6]

Unfortunately, veneering zirconia cores with glass ceramics led to a higher occurrence of chipping of the veneered layer which in some cases can lead to failure of the restoration.[7–9] This issue is seldom faced when using materials with a high fracture toughness and excellent mechanical properties such as monolithic zirconia restorations.[10] Therefore, recent innovations

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in zirconia dental materials were aimed to increase their indications by enhancing its optical properties. This can open the possibility of replacing layered zirconia cores and perhaps replace lithium disilicate restorations as the first choice of treatment when restoring esthetically visible zones.[11,12]

While the monochromatic zirconia blocks achieved satisfactory esthetic outcome, it still lacked the color-gradient effect apparent in natural dentition.[13] Multi-layered zirconia is uniquely manufactured in a way that mimics the color-gradienct of natural teeth. This is accomplished by adding gradient percentages of metal oxides, such as Fe_2O_3, over several layers.[12] The percentage of the added metal oxide increases gradually as we move from the incisal/ occlusal layer to the cervical layer. The intensity of the chroma increases as the percentage of metal oxide increases therefore giving it the color gradient effect.[11] Usually, the metal oxides are added to the zirconia powder before moulding it to a single disk. Hence, creating a multi-layered color gradient zirconia disk. According to the manufacturer the difference between the layers is the amount of pigmentation incorporated. Therefore, the same yttria content and microstructure is present in all the layers.[14]

Sintering zirconia materials is considered to be the most critical step in manufacturing zirconia dental restorations. Sintering temperatures studied in previous investigations range from 1300°C up to 1600°C.[15–18] It was found that the sintering temperature can affect the optical and mechanical properties of the final restoration.[17–19] This effect was justified by some investigators as a resultant change in the mean grain size, microstructure and pore diameter of the zirconia at different sintering temperatures.[20,21] For example, as the sintering temperature increases, grain size increases leading to a smaller number of grain boundaries and therefore lower chance of light scattering at theses boundaries thus enhancing translucency.[16]

The effect of the different sintering temperatures on the translucency and color difference of translucent monochromatic zirconia materials, including 5mol%- yttria-stabilized tetragonal zirconia polycrystals (5Y-TZP) and 4mol%- yttria-stabilized tetragonal zirconia polycrystals (4Y-TZP), has been investigated. Sen *et al*. investigated the differences in translucency of zirconia with different sintering temperatures (1350°C, 1450°C, 1600°C).[19] The results showed that as temperature increased, TP significantly increased.[19] Another recent study reached a similar conclusion, both 4Y-TZP and 5Y-TZP showed an increase in TP parameters as sintering temperature increased, with 5Y-TZP being the most translucent of the two.[21] Furthermore, it was found that 4Y-TZP showed a significantly better color reproduction compared to 5Y-TZP as sintering temperature raised to 1600°C.[21] On the other hand, Cardoso *et al* found no significant effect of sintering temperature on the translucency of zirconia specimens tested.[18] The effect of sintering temperature on the optical properties of monochromatic zirconia is highly investigated. However, the effect of sintering temperature on the newly developed multi-layered zirconia is still lacking.

The optical behaviour of multi-layered zirconia materials towards changing sintering temperature could be different than the extensively investigated monochromatic translucent zirconia materials. This could be due to its different formulation. The presence of varying percentages of metal oxide concentration in different layers could have an effect on the final optical properties of these new materials. Ramesh *et al*. found that adding MnO_2 to zirconia led to increased pore formation in sintered zirconia.[22] Pores can act as light scattering centers that can increase the opacity of the resultant zirconia restoration. Shih *et al*. on the other hand found that adding CeO in the formulation of zirconia can increase the grain size.[23] Increasing the grain size can enhance the optical properties such as translucency by decreasing the chance of scattering incident light.[16] Hence it can by anticipated that these novel materials could behave differently.

A few studies addressed the effect of sintering temperature on the optical properties of multi-layered zirconia. One study investigated the effect of sintering temperature on TP of multi-layered zirconia.[17] They found that as sintering temperature increased the TP increased.[17] Another recent study investigated the effect of sintering temperature and dwell time on the TP of multi-layered zirconia.[24] They found that as temperature increases and dwell time decreases, TP values decrease.[24] However, both studies didn't investigate the effect of sintering temperature on each individual layer as they may have a different response.[14]

In order to comprehensively evaluate the effect of sintering temperature on a multi-layered zirconia dental material, each layer should be evaluated separately. However up to our knowledge, there are a limited number of studies investigating the effect of sintering temperatures on the optical of each individual layer. Therefore, the objective of this study is to evaluate the effect of sintering temperature on the translucency and color difference of translucent multi-layered, color gradient zirconia dental materials and compare them with lithium disilicate glass ceramics LS_2 to assist in achieving the optimum sintering condition. The null hypotheses include:

- No significant difference in TP and ΔE amongst LS_2 and both zirconia materials at different sintering temperatures and layers.
- No significant difference in TP and ΔE amongst different sintering temperatures.
- No significant difference in TP and ΔE amongst different layers of the same zirconia material.
- No significant correlation between sintering temperature and either TP or ΔE values.

MATERIALS AND METHODS

A total of 108 plate-shaped specimens were fabricated from two types of pre-sintered multi-layered zirconia blanks (4Y-TZP, 5Y-TZP) and pre-crystalized LS_2 blocks as the control.

Details of the material's used in the study is listed in Table 1. The multi-layered presintered zirconia blanks were cut into four sections to obtain samples from each color gradient layer using a water- cooled low speed diamond saw, 0.5 millimeter thickness (Isomet 2000, Buehler, Lake Buff, Illinois, USA). The material was sectioned according to the location of each layer (layer thickness) provided by the manufacturer *(Figure 1*). The first layer corresponds to the enamel layer, the second layer is the intermediate 1, the third is intermediate 2 and the fourth layer is the body /dentin layer *(Figure 1*). Subsequently, plate shaped specimens (n=4) were cut from each layer with a water- cooled low speed diamond saw, 0.5 millimeter thickness (Isomet 2000, Buehler, Lake Buff, Illinois, USA). Specimens from the control group (LS_2) were also obtained by cutting the ceramic pre-crystalized blocks into plate-shaped specimens using the same saw. The size of the fabricated specimens were 25% larger than the required final size to compensate for the shrinkage after sintering/crystallization and for the finishing and polishing process.

The presintered plates fabricated from each layer of both zirconia materials were divided randomly into three subgroups according to the sintering temperature: 1300°C, 1450°C, 1600°C. All the zirconia materials were sintered in the same furnace (inFire HTC, Dentsply Sirona) and with the same holding time (2 hrs). The control group (n=12), lithium disilicate, was crystalized using (Programat P500, Ivoclar Vivadent) according to the manufacturer instructions. The instructions include: 403°C standby temperature, closing time 6 min, heating rate 90°C/min and firing temperature 840°C for 7 minutes.

All the plates were ground flat and polished with a series of silicon carbide sheets (600-, 800- and 1200-grit) at 300 rpm under water cooling until a mirror-like surface was achieved, the finishing and polishing time was standardized. The final size of the plate shaped specimens was 10×10× 1mm ±0.02 mm. The definitive thickness was determined by using a digital calliper (Mitutoyo, Canada Inc., Mississauga, ON, Canada); with an accuracy of ±0.05 mm. All specimens were stored and tested under dry conditions.

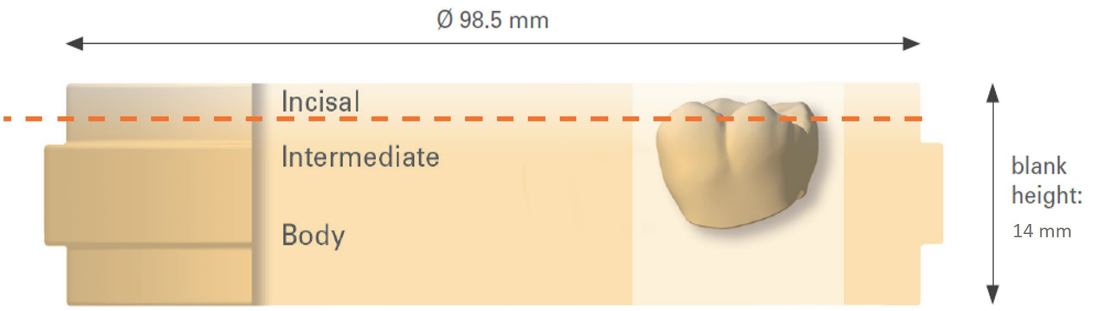
TRANSLUCENCY PARAMETER (TP):

The translucency parameter of the plate-shaped specimens was measured with a spectrophotometer (Labscan XE spectrophotometer, Hunterlab, VA, USA) after different sintering temperatures. The translucency was measured according to the CIELAB color scale relative to the standard illuminant D65 against white background (CIE $L^*=93.26$, $a^*=0.61$, $b^*=2.09$) and black background (CIE $L^*=2.93$, $a^*=0.38$ and $b^*=-0.34$). The spectrophotometer was calibrated before measurements of each group according to the manufacturer's instructions using black and white standard calibrating blocks. Each specimen was measured three times and the average value was determined. The translucency parameter (TP) values were determined by calculating the color difference between readings over the black and white background for the same specimen, using the following formula: $TP = [(L^*_W - L^*_B)^2 + (a^*_W - a^*_B)^2 + (b^*_W - b^*_B)^2]^{1/2}$. Subscripts "B" and "W" refer to the color coordinates over a black and white background, respectively.

Table 1. List of materials.

Material class	Type	Shade	Name	Manufacturer	Size	Chemical Composition
5Y-TZP	Color gradient	A2	DD cubeX2 ML98	Dental Direkt	98.5mm ×14 mm	$ZrO_2 + HfO_2 + Y_2O_3 \geq 99.0$ $Y_2O_3 < 10$ $Al_2O_3 \leq 0.01$ Other oxides < 1
4Y-TZP	Color gradient	A2	DD cube ONE ML98	Dental Direkt	98.5mm ×14 mm	$ZrO_2 + HfO_2 + Y_2O_3 \geq 99.0$ $Y_2O_3 < 8$ $Al_2O_3 \leq 0.15$ Other oxides < 1
Lithium disilicate	Monochrome	A2	IPS e.max CAD HT blocks	Ivoclar Vivadent	B40 L	$SiO_2 = 57.0 - 80.0$ $Li_2O = 11.0 - 19.0$ $K_2O = 0.0 - 13.0$ $P_2O_5 \leq 11.0$ $ZrO_2 \leq 8.0$ $ZnO \leq 8.0$ $Al_2O_3 \leq 5.0$ $MgO \leq 5.0$ Coloring oxides ≤ 8.0

(a) Multi-layer zirconia blank:



Blank height (mm)	Layer 1: Incisal (mm / %)	Layer 2: Intermediate (mm / %)	Layer 3: Intermediate (mm / %)	Layer 4: Body / Dentine (mm / %)
14 mm	3.5 / 24.9	2.1 / 15	2.1 / 15	6.3 / 45.1

(b) Monolayer specimens prepared from each layer:

Layer 1: Incisal

Layer 2: Intermediate 1

Layer 3: Intermediate 2

Layer 4: Body/ Dentin

Figure 1: (a) schematic presentation of the multi-layer zirconia blank provided by the manufacturer (Dental Direkt, Germany), (b) schematic presentation of the monolayer specimens prepared from each layer.

COLOR DIFFERENCE (ΔE):

The same plate-shaped specimens were placed over a black background (CIE $L^*=2.93$, $a^*=0.38$ and $b^*=-0.34$) and the CIELAB coordinates were measured for each specimen using a spectrophotometer (Labscan XE spectrophotometer, Hunterlab, VA, USA). In this mode the color difference (ΔE) was determined between the control (LS_2) and sintered zirconia samples. For each specimen, three measurements were taken at the center of the surface and their average was recorded. The color difference between the LS_2 and zirconia samples after each sintering temperature was calculated by using the following formula: $\Delta E=[(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$. Mean ΔE values below 3.46 was considered "clinically acceptable", while ΔE values above 3.46 was considered "clinically unacceptable".[25]

SEM IMAGING:

Representative samples were selected from both zirconia materials at different sintering temperatures to visualize the effect of dopant and sintering temperature on the microstructure and grain size. The samples were coated with platinum using an auto fine coater (JEC-3000FC, JEOL Ltd., Tokyo, Japan) and examined under scanning electron microscope (SEM) (JSM-IT500HR, JEOL Ltd., Tokyo, Japan) with a magnification of 6000× up to 30,000×. SEM images were taken to compare the structural differences amongst the groups.

STATISTICAL ANALYSIS:

Descriptive statistics was first calculated to report the mean, standard deviation, minimum and maximum values for each subgroup. Before conducting the hypothesis testing, data was assessed for normality using Shapiro-Wilk test. Levene's test was conducted to determine homogeneity of the variances within the groups of each dependent variable. Based on the number of means, either T-test or One-way analysis of variance (ANOVA) was conducted to evaluate the significant differences between the means.

One and two-sample T-tests were also used to compare the mean TP, ΔE of the LS_2 and zirconia materials at different sintering temperatures regardless of the layer. ANOVA was performed to examine the difference in means of the outcomes, which included: ΔE and TP, for the control group (LS_2) and both zirconia materials (4Y-TZP and 5Y-TZP), at different layers and sintering temperatures. If ANOVA deemed at least one group different (p-value <0.05), a post-hoc multiple comparison test (Dunnett T3) was used to determine the significant differences between the compared groups.

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Finally, the correlation between sintering temperature and either TP or ΔE was assessed using Pearson Correlation coefficient. Data was analyzed using SPSS 24.0 software, with a p-value <0.05 (IBM Inc., Chicago, USA).

RESULTS

TRANSLUCENCY PARAMETER (TP):

Results of the T-test comparing the mean TP of the control (LS2) and both zirconia materials sintered at different temperatures is seen in Table 2. The results suggest that the LS_2 has a significantly higher TP value (86.71± 0.5) than both zirconia materials tested at all layers and sintering temperatures ($P<0.001$) (see *Table 2* and *Figure 2*). When comparing the values of both zirconia materials, 5Y-TZP had a significantly higher TP value at both sintering temperatures 1450°C and 1600°C ($P<0.05$) *(Table 2)*. However, both zirconia materials didn't differ significantly at 1300°C sintering temperature ($P>0.05$) *(Table 2)*.

The behaviour of zirconia materials to increasing sintering temperatures was investigated *(Table 3)*. The TP value was at its lowest at sintering temperature 1300°C. The results suggest that as the sintering temperature increases the TP values significantly increases up to 1450°C ($P<0.001$) *(Table 3)*. However, increasing sintering temperature beyond 1450°C doesn't significantly enhance the TP values ($P>0.05$) (see *Table 3*).

The One-way ANOVA was conducted followed by a multiple comparison test (Dunnett T3) to compare the zirconia layers within the same material and sintering temperature *(Table 4)*. It is noticed that in both zirconia materials as we move from the first layer to the fourth, the TP value decreases. However, the decease of TP was not significant amongst all the layers (see *Table 4*). Results showed a significant decrease in TP value between the first and fourth layer of 4Y-TZP at 1450°C and 1600°C sintering temperatures ($P<0.05$). For 5Y-TZP a significant difference was recorded between the first (1.13± 0.22) and second (2.67± 0.69) layer and between the first (1.13± 0.22) and third (2.20±0.38) layer at 1300°C ($P<0.05$). When 5Y-TZP was sintered at 1450°C, the first (66.28± 1.57) and second (66.06± 0.20) layer had a significantly higher TP value compared to the fourth layer (62.74± 0.7) ($P<0.05$). Finally, when the sintering temperature increased to 1600°C, the first layer (72.48±1.43) had a significantly higher TP than all the layers tested ($P=0.001$).

COLOR DIFFERENCE (ΔE):

The results of the T-test comparing the mean ΔE of both zirconia materials at different sintering temperatures is seen in Table 2 and at different layer seen in Figure 3. The results show that the 4Y-TZP had a significantly lower ΔE value than 5Y-TZP at all layers and sintering temperatures except at 1300°C, it had the lowest value ($P<0.001$) *(Table 2, Figure 3)*.

Table 2. Results of the One-sample T-test comparing the mean TP of LS_2 to both zirconia materials and the Independent T-test comparing the mean ΔE of both zirconia materials at different sintering temperatures.

Measurement	Crystalized/ Sintered	Ceramic material	Mean ±SD	Independent T-test	One-sample T- test (compare with control)
TP	crystalized (control)	LS_2	86.71 ±0.50		0.000
	sintered at 1300°C	4Y	1.45 ±0.97	0.06	
		5Y	2.0 ±0.92		
	sintered at 1450°C	4Y	60.31 ±1.18	0.000	
		5Y	64.99 ±1.72		
	sintered at 1600°C	4Y	57.73 ±2.03	0.000	
		5Y	67.53 ±3.19		
ΔE	sintered at 1300°C	4Y	33.64 ±1.9	0.00	
		5Y	29.58 ±1.94		
	sintered at 1450°C	4Y	6.51 ±2.34	0.00	
		5Y	9.97 ±1.81		
	sintered at 1600°C	4Y	3.82 ±2.08	0.00	
		5Y	12.35 ±3.15		

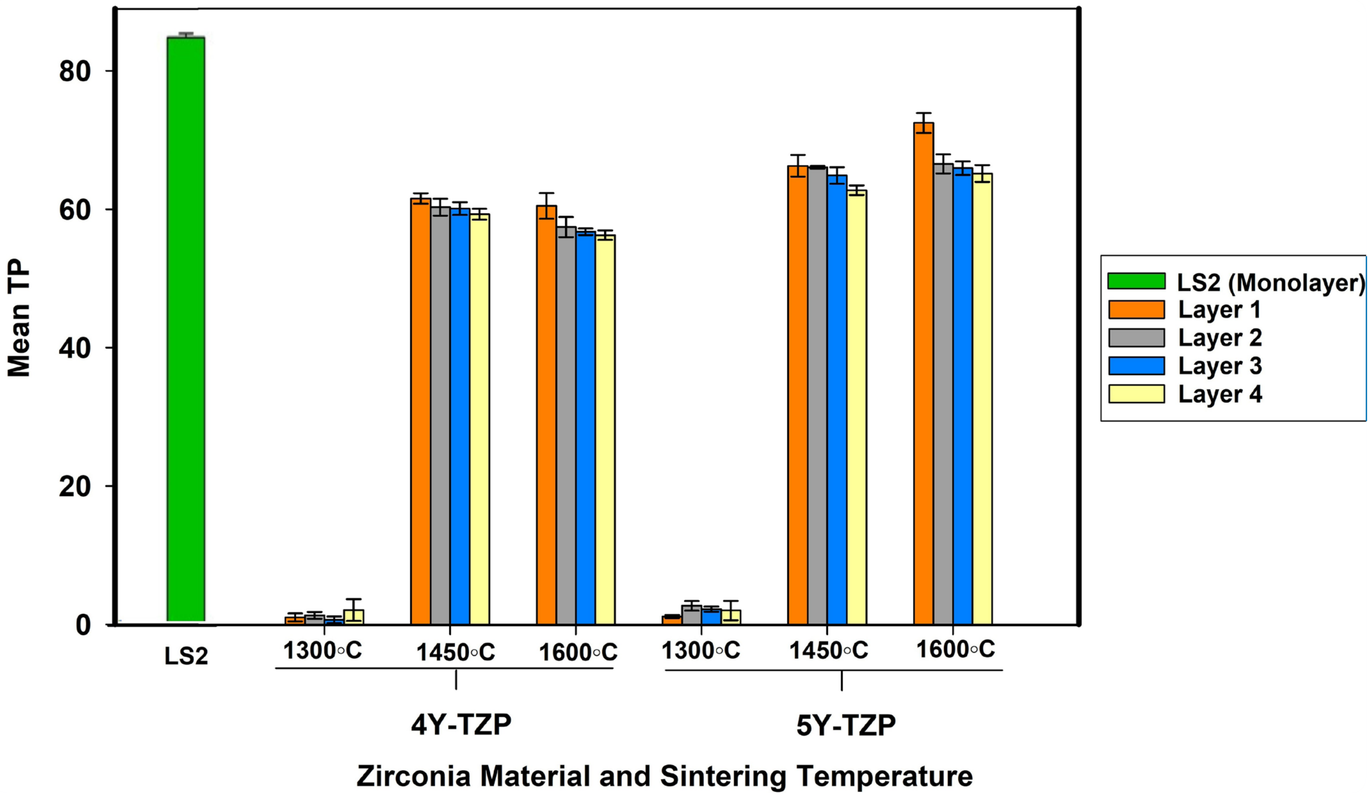

Figure 2: Mean TP value for zirconia materials at different layers and sintering temperatures compared to LS_2.

Table 3. Results of one-way ANOVA comparing the mean TP and ΔE values of zirconia materials at different sintering temperatures followed by multiple comparison test (Dunnett T3).

Measurement	Sintering temperature (°C)	Mean ±SD	P-value
TP	1300	1.62 ±1.0^{a}	0.000
	1450	62.65 ±2.79^{b}	
	1600	62.63 ±5.63^{b}	
ΔE	1300	31.61 ±2.8^{A}	0.000
	1450	8.24 ±2.7^{B}	
	1600	8.08 ±5.06^{B}	

** Different superscript letters indicate a significant difference ($p<0.05$) between the means within the same column.

The mean ΔE of zirconia materials at different sintering temperatures was investigated *(Table 3)*. A significant decrease of ΔE from 1300°C to 1450°C was reported ($P<0.001$). When sintering temperatures increased from 1450°C to 1600°C, no significant change in ΔE was found ($P>0.05$) *(Table 3)*.

The mean ΔE value of the zirconia layers within the same material and sintering temperatures was investigated *(Table 5)*. Results of the one-way ANOVA suggest a significant difference in the ΔE of the tested layers within the same zirconia material at the same sintering temperature ($P<0.05$). The multiple comparison test (Dunnett T3) seen in Table 5 shows a significantly higher ΔE value of the first layer compared to all the other layers when 4Y-TZP is sintered at 1300°C and 1450°C ($P<0.05$). However, no significant difference between the ΔE of the rest of the layers was detected ($P>0.05$). It is also evident that when 4Y-TZP was sintered at 1600°C, the ΔE value of both the third and fourth layers were significantly lower than the first and second layers ($P<0.05$). At sintering temperature 1600°C, the third and fourth layers of 4Y-TZP resulted in a clinically acceptable color difference ($\Delta E \leq 3.46$).[25]

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Table 4. Results of one-way ANOVA comparing the mean TP of zirconia layers within the same sintering temperature and material followed by multiple comparison test (Dunnett T3).

Measurement	Ceramic material	Layer	Mean ±SD at different Sintering temperatures (°C)					
			1300	P-value	1450	P-value	1600	P-value
TP	4Y	1st Layer	1.0 ±0.58^{a}	0.3	61.56 ±0.75^{a}	0.040	60.48 ±1.85^{a}	0.041
		2nd Layer	1.29 ±0.49^{a}		60.30 ±1.21^{ab}		57.43 ±1.45^{ab}	
		3rd Layer	0.64 ±0.49^{a}		60.10 ±0.91^{ab}		56.75 ±0.48^{ab}	
		4th Layer	2.06 ±1.57^{a}		59.27 ±0.78^{b}		56.26 ±0.69^{b}	
	5Y	1st Layer	1.13 ±0.22^{a}	0.011	66.28 ±1.57^{a}	0.002	72.48 ±1.43^{a}	0.001
		2nd Layer	2.67 ±0.69^{b}		66.06 ±0.2^{a}		66.55 ±1.37^{b}	
		3rd Layer	2.20 ±0.38^{b}		64.87 ±1.19^{ab}		65.94 ±0.97^{b}	
		4th Layer	2.0 ±1.4a^{b}		62.74 ±0.7^{b}		65.17 ±1.21^{b}	

**** Different superscript letters indicate a significant difference (p<0.05) between the means within the same column.**

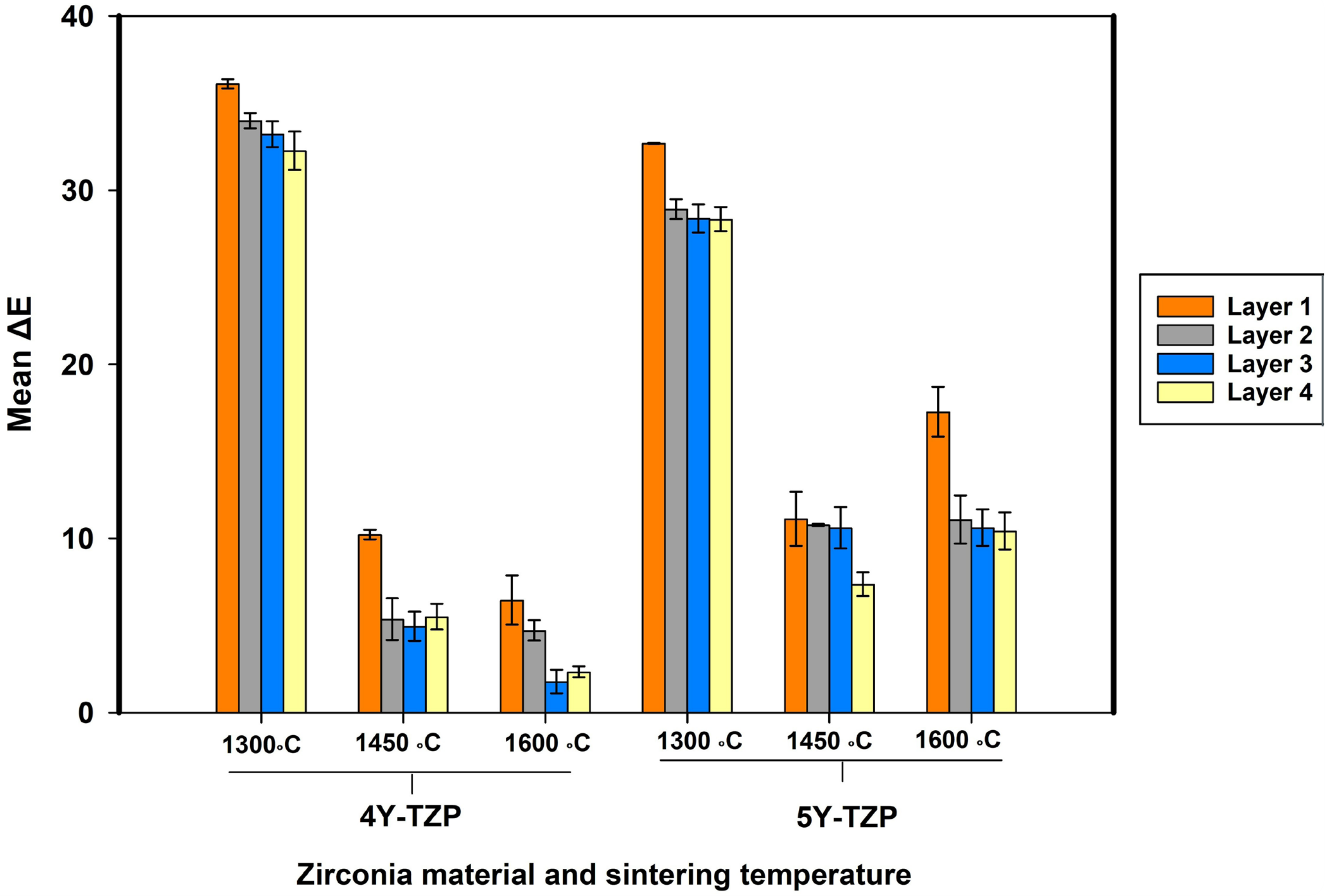

Figure 3: Mean ΔE of zirconia materials at different layers and sintering temperatures.

With regards to the 5Y-TZP, the multiple comparison test (Dunnett T3) seen in Table 5 suggests that at sintering temperatures 1300°C and 1600°C, the first layer recorded the highest ΔE value compared to the rest of the layers (P<0.05). However, at 1450°C, the first, second and third layers were significantly higher than the fourth layer (P<0.05). Unfortunately, all the layers of 5Y-TZP at all temperatures resulted in a clinically unacceptable color difference (ΔE>3.46).[25]

Table 5. Results of one-way ANOVA comparing the mean ΔE of zirconia layers within the same sintering temperature and material followed by multiple comparison test (Dunnett T3).

Measurement	Ceramic material	Layer	Mean ±SD at different Sintering temperatures (°C)					
			1300	P-value	1450	P-value	1600	P-value
ΔE	4Y	1st Layer	36.10 ±0.26^{a}	0.000	10.22 ±0.27^{a}	0.000	6.46 ±1.41^{a}	0.001
		2nd Layer	33.99 ±0.43^{b}		5.36 ±1.19^{b}		4.72 ±0.58^{a}	
		3rd Layer	33.22 ±0.74^{b}		4.96 ±0.84^{b}		1.77 ±0.67^{b}	
		4th Layer	32.26 ±1.1^{b}		5.50 ±0.73^{b}		2.34 ±0.32^{b}	
	5Y	1st Layer	32.69 ±0.02^{a}	0.000	11.12 ±1.54^{a}	0.002	17.28 ±1.43^{a}	0.001
		2nd Layer	28.91 ±0.56^{b}		10.77 ±0.06^{a}		11.08 ±1.38^{b}	
		3rd Layer	28.38 ±0.8^{b}		10.61 ±1.18^{a}		10.61 ±1.05^{b}	
		4th Layer	28.34 ±0.68^{b}		7.37 ±0.68^{b}		10.42 ±1.06^{b}	

** Different superscript letters indicate a significant difference (p<0.05) between the means within the same column.

Sintering temperature had a significant positive correlation with TP (r^2=0.859, P<0.001). On the other hand, sintering temperature a significantly negative correlation with ΔE (r^2=-0.825, P<0.001). Furthermore, the Pearson coefficients for both TP (0.859) and ΔE (-0.825) indicated a very high correlation with the sintering temperature.

SEM IMAGING:

SEM images of both materials after different sintering temperatures is seen in Figure 4 and 5. When comparing the two different types of zirconia materials 4Y-TZP and 5Y-TZP, it is noticed that both materials behaved similarly to increasing sintering temperatures. At 1300°C both 4Y-TZP and 5Y-TZP revealed a microstructure with low-density containing small grains and increased porosity *(Figure 4a-c, Figure 5a-c*). Also, both materials had a significant increase in grain size and density along with a decrease in porosity as the sintering temperature increased from 1300°C to 1450°C and finally 1600°C *(Figure 4c-i, Figure 5c-i*). It is also apparent that the grains reached its largest size at 1600°C for both materials *(Figure 4i, Figure 5i*). However, when comparing the grain size of both types of materials, it is noticed that the grain size of 5Y-TZP is much larger than 4Y-TZP at both sintering temperatures 1450°C and 1600°C *(Figure 4i, Figure 5i*).

DISCUSSION

The results showed significant difference in TP values between LS_2 and both zirconia materials, at all layers and sintering temperatures. Therefore, the null hypothesis is rejected. Furthermore, a significant difference was found between 5Y-TZP and 4Y-TZP at all layers and sintering temperatures except at 1300°C. (P<0.01). The results obtained in this study suggests that LS_2 has highest compared to all the zirconia materials at all sintering temperatures and layers. This result comes in agreement with other similar studies investigating the translucency of LS_2 compared with zirconia. Kwon *et al*. compared the translucency of LS_2 with translucent zirconia and found that LS_2 had significantly superior translucency compared to 5Y-TZP.[26] Multiple studies came to a similar conclusion; LS_2 had significantly increased translucency values compared to translucent zirconia materials (P<0.05).[27–29]

Translucency can be described as the quality of light passing through the material. It is a result of a combination of interactions with the incident light. Interactions include reflection, absorption and transmission.[30] The way the ceramic material interacts with the incident light depends on many factors including grain size, grain boundaries, porosities, crystallographic structure, refractive index and chemical composition.[31]

The significantly high TP values reported in this study for LS_2 can be explained by the minimum difference between the refractive indices of the lithium disilicate crystals (1.55) and glassy matrix (1.5). This leads to a lower chance of hindering the passing light and therefore increased translucency values compared to zirconia materials.[29] In zirconia materials, dopants such as yttria are usually added to the composition of zirconia materials to enhance phase stability. However, addition of oxides can increase the chances of scatterine incident light due to the difference in refractive indices reported betwee, zirconia and added oxides therefore increasing its opacity.[32]

The presence of pores in the tested ceramic material can create a volume of differing refractive index leading to scattering of light and therefore decrease translucency values.[33,34] In zirconia materials, the presence of residual pores is inevitable due to the fabrication process. Gaps in between zirconia grains are

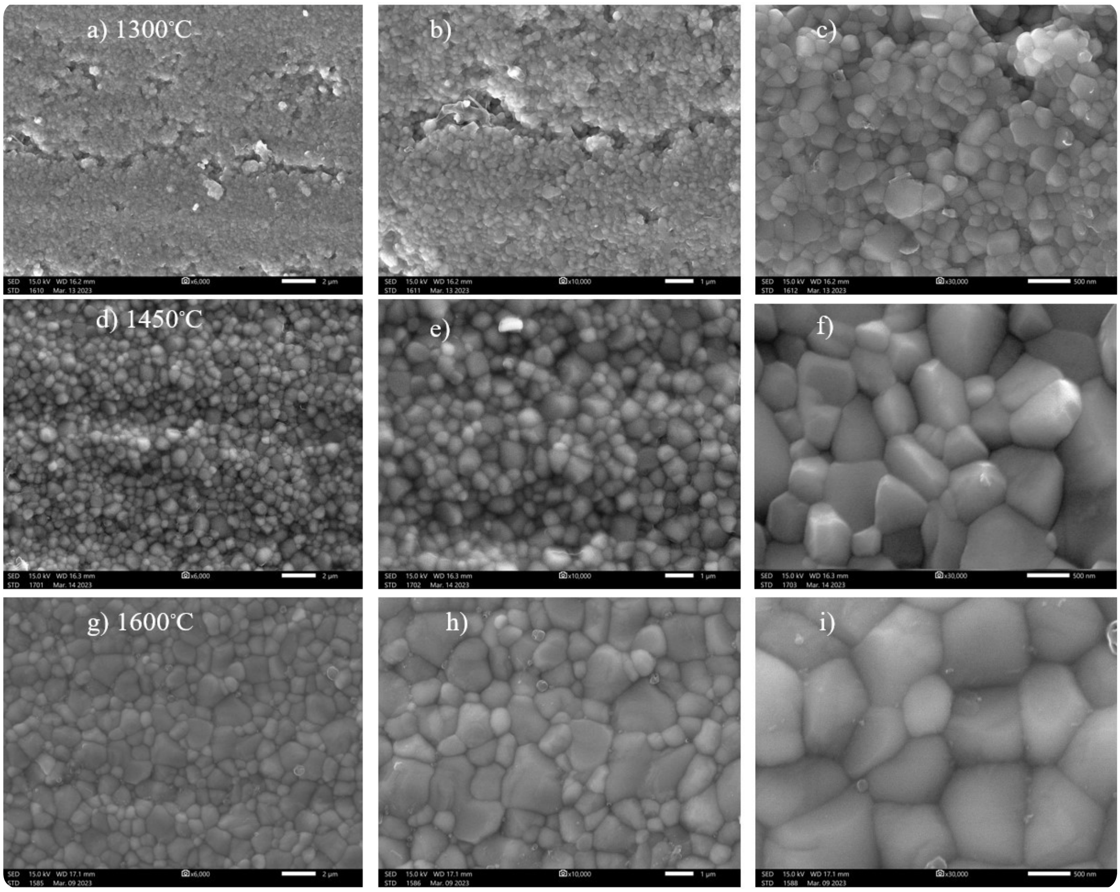

Figure 4: Representative SEM images at different magnifications (6000×, 10,000× and 30,000×) showing the microstructure of DD cube ONE ML (4Y-TZP) after different sintering temperatures: 1300°C (a-c), 1450°C (d-f) and 1600°C (g-i).

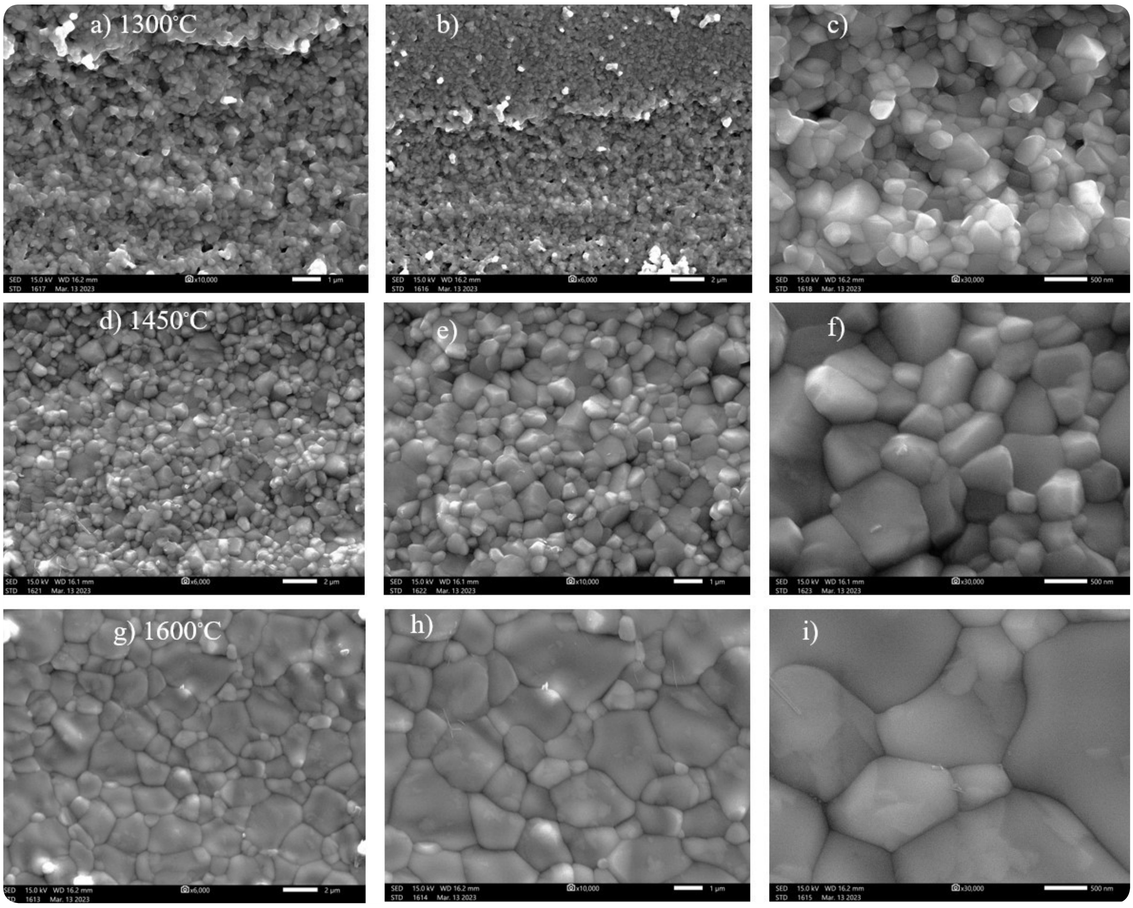

Figure 5: Representative SEM images at different magnifications (6000×, 10,000× and 30,000×) showing the microstructure of DD cubeX^2 ML (5Y-TZP) after different sintering temperatures: 1300°C (a-c), 1450°C (d-f) and 1600°C (g-i).

formed during the moulding of the zirconia powder.[34] Unlike zirconia materials, LS_2 usually have little too no residual pores in its microstructure. Therefore, less chance of scattering light at the site of the pores resulting in higher TP values.[27]

When comparing the TP of both zirconia materials, 5Y-TZP maintained a higher TP score than 4Y-TZP at all layers and sintering temperatures except at 1300°C. Similar results was found in multiple studies comparing the two translucent zirconia materials.[29,35,36] Such high translucency values is expected due to the higher yttria content leading increased amount of cubic phase (>25-50%)[37] in its microstructure as previously seen in SEM images.[29] The cubic crystals are optically isotropic, meaning that index of refraction is the same if measured in all directions. Consequently, incident light can pass in all directions without deflecting leading to increased levels of translucency compared to the other generations.[33] Also, higher translucency values can be attributed to the larger grain size of the highly abundant cubic phase compared to the 4Y-TZP (see *Figure 5*). The larger the grain size, the lower the number of grain boundaries and therefore lower chance of scattering of light at the grain boundaries.[27]

A significant difference in TP values were recorded at different sintering temperatures. Therefore, the null hypothesis is rejected. In addition, results showed that both materials responded similarly to change in sintering temperatures. A similar trend in increase in TP values were recorded for both 4Y-TZP and 5Y-TZP. Both materials had low TP values at 1300°C and were not significantly different. The currently available zirconia materials are sintered at temperatures that range from 1400°C to 1550°C, depending on the manufacturer.[38] Hence, sintering at low temperatures such as 1300°C can create under-fired zirconia specimens, regardless of the type of zirconia. Sintering at low temperatures will result in zirconia with a smaller grain size, larger number of grain boundaries, higher scattering of incident light and therefore much lower TP values.[16] Similar microstructure is seen in our study when zirconia was fired at 1300°C *(Figure 4a-c and Figure 5a-c*).

On the other hand, sintering at higher temperatures, such as the manufacturer recommended temperature (1450°C), creates a significant increase in the TP values of the resulting zirconia specimens for both types of materials. This can be explained by the significant grain growth associated with increasing temperature levels.[21,39–42] Therefore, increasing the grain size increases the TP values as previously explained.[16,19,40,42] Also, the significant increase in translucency can be correlated with the decrease of pore size and quantity through solid-state diffusion when sintering temperature increases.[43] The increase in grain growth and decrease of pores when sintering temperatures rise to 1450°C is also apparent our study *(Figure 4d-f and Figure 5d-f*).

Furthermore, results showed that increasing the sintering temperature beyond 1450°C sintering didn't significantly enhance the TP values for both zirconia materials. Multiple studies reached a similar conclusion in which increasing the sintering temperature beyond 1450°C didn't increase the translucency values of the tested zirconia materials.[17–19] Cardoso *et al*. evaluated the SEM images of zirconia materials sintered at two different sintering temperatures. The images reviled an increase in the grain size as sintering temperature increase forom 1450°C to 1600°C, this is also seen in our study *(Figure 4g-i and Figure 5g-i*). However, this enhancement in size didn't affect the translucency value significantly.[18] This was explained by the fact that translucency is not only affected by the grain size but also by the type and concentration of the phase content. Increasing the temperature from 1450°C to 1600°C doesn't change the composition nor the concentration of the cubic phase leading to no significant change in translucency values.[18,19]

When the TP of different layers within the same type of zirconia at the same sintering temperature was evaluated, a difference in TP was noticed. Therefore, the null hypothesis is rejected. It was found that as we move from first to the fourth layer, the TP decreases and in some samples the difference was significant. Erdelt *et al*. also noted a difference in TP between the different layers of multi-colored zirconia materials.[44] The difference could be due to the incorporation of different percentages of pigmentation in different layers that is visibly evident with the unaided eye (*see Figure 1*). The incorporation of different percentages of pigments in each layer can affect the transmittance of light in different degrees depending on the amount of pigmentation and therefore varying translucency values.

Transmittance is the attenuation of light after it passes through and exits the medium. This involves reflectance, scattering and absorption. While the translucency parameter is related to the reflectance of light through a medium against white and black backgrounds. Supornpun *et al*. reported a difference in transmittance among different shades of zirconia materials. They also found that transmittance was highly dependent on the shade of the tested material.[45] Furthermore, a leaner relationship between TP and transmittance was reported.[45] Their results were consistent with the results of Ueda *et al.*[46] The study evaluated light transmittance of different layers of a multi-colored zirconia blank.[46] They found that different layers recorded a different transmittance coefficient; consequently, translucency. Therefore, it can be concluded that different levels of pigmentation in each layer can effected the TP.[44–46]

When the color difference from LS_2 was measured for both 4Y-TZP and 5Y-TZP, a significant difference was found between both materials at all sintering temperatures. Therefore, the null hypothesis was rejected. A lower ΔE value was recorded for 4Y-TZP at all layers and sintering temperatures except at 1300°C. The results of the present study are consistent with the results of Grambow *et al.*[21] They compared the color difference of two multi-layered zirconia materials, 4Y-TZP and 5Y-TZP, at different sintering temperatures. They concluded that 4Y-TZP performed better at color reproduction than 5Y-TZP as sintering temperatures increased.[21]

The difference in performance of the two types of zirconia materials in color reproduction was explained by Kang *et al*.[47] They found that the type of multi-layered zirconia material effected the final color accuracy due to the difference in translucency levels. When evaluating ΔE, the materials are tested with two backgrounds, white and black. With high translucency levels, such as 5Y-TZP, the higher the chance of the black background to be visible causing more color distortion. However, a material with lower translucency values, such as 4Y-TZP, will have a more stable color due to the lower visibility of the different background.[47]

A significant difference in ΔE values were recorded at different sintering temperatures. Therefore, the null hypothesis is rejected. Results also showed that ΔE of both zirconia materials responded similarly to different sintering temperatures. As sintering temperatures increased from 1300°C to 1450°C, a significant decrease in ΔE was recorded, thus better color reproduction. However, no significant improvement was found when increasing the sintering temperature to 1600°C. These results were in accordance with other similar studies.[15] Ebied *et al*. reported that higher sintering temperatures lead to a significantly enhanced color reproduction.[15] This was mainly due to the increased sintered density of the zirconia specimens leading to a reduction of pores, enhanced crystalline arrangement, also seen in our study *(Figure 4d-f and Figure 5d-f*). This leads to a better light transmission and specular reflection and ultimately better color reproduction.[15] This can also explain the lack of improvement in ΔE beyond sintering temperature 1450°C because the TP, as explained earlier, didn't significantly change beyond this sintering temperature. Therefore, the light transmission and specular reflection would not be significantly affected leading to a similar ΔE value.

When the ΔE of different layers within the same type of zirconia at the same sintering temperature was evaluated, a difference in ΔE was noticed. Therefore, the null hypothesis is rejected. Furthermore, results reveal that the first layer within the same zirconia material and sintering temperature had a significantly higher ΔE value, in most cases, compared to all the other layers. This result was expected due the technique in which the multi-layered zirconia was manufactured. An elemental composition analysis of different layers of a multi-layered zirconia was done by Kolakarnprasert *et al*.[12] They found different concentrations of elements such as Fe and Ti, which are used as pigments, within each layer.[12] The varying concentration of these elements within the layers would lead to the development of color-gradient zirconia mimicking natural teeth. The first layer (incisal) has the least percentage of incorporated metal oxides compared to all the other layers. The percentage of incorporated pigments increase towards the fourth layer leading to an increase in chroma.[12] This difference in color intensity is also visually apparent between the first and remaining layers (*see Figure 1*).

Pearson correlation coefficient between the sintering temperature and either TP or ΔE was significant. Therefore, the null hypothesis was rejected. TP values have a significant positive correlation with sintering temperature. Meaning as sintering temperature increases, these values increase. However, a significantly negative correlation was found between sintering temperature and ΔE. Therefore, as sintering temperature increases ΔE decreases. These results can be explained by the direct effect of sintering temperature on microstructure particularly the grain size. A positive correlation was found between sintering temperature and grain size.[48] Therefore, the higher the temperature, the larger the grain growth therefore high TP values and lower ΔE as explained earlier. However, these results should be applied with caution since some of the properties mentioned can be affected negatively when increasing the sintering temperature beyond a certain limit.

One of the important limitations of this study includes investigating the optical properties of plates from individual layers rather than samples containing the complete color layering. This can affect results of our study since the manufacturer designed the material to be used as a multi-layered material rather than a single layer. Therefore, mindful interpretation of our results is vital. Other limitations of this study include using only two types of A2-shaded, multi-layered zirconia material standardized backgrounds and only altering the sintering temperature. Future studies should include testing samples containing all the layers utilizing different shadent backgrounds at sintering parameters.

CONCLUSION

Within the limitations of this study, it can be concluded that the sintering temperature can significantly affect the translucency and color of multi-layered zirconia materials. As sintering temperature increases to 1450°C, the optical properties reached their peak performance. However, increasing the sintering temperature beyond 1450°C doesn't enhance the optical properties. When comparing the translucency of LS_2 to both zirconia materials, LS_2 maintained the highest performance. From both zirconia types, 4Y-TZP achieved a better color reproduction. The layers of multi-layered zirconia materials tested recorded different TP and ΔE values.

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