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EFFECT OF SINTERING TECHNIQUE ON THE COLOR COORDINATES OF A TRANSLUCENT ZIRCONIUM CERAMIC MATERIAL

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ABSTRACT

INTRODUCTION: Monolithic zirconium restoration is widely used in dental practice. Several sintering processes have been employed and it may affect the color coordinates of monolithic zirconium restorations which is crucial for a successful esthetic restoration.
Purpose of the study: to evaluate the effect of different sintering techniques on the color parameters of zirconium ceramic restorations.
MATERIALS & METHODS: Thirty specimens were milled from Zenostar Translucent zirconia, shade T2 using Kavo Everest engine milling machine (KaVo Dental, Charlotte, NC, USA). The final dimensions of the discs were 10 mm diameter x 2 mm thickness. They were divided into three groups according to the sintering process, ten discs each. Group I: Microwave Sintering (MS), Group II: Speed Sintering (SS), & Group III conventional Sintering (CS). The spectrophotometer (Easyshade V, VITA Zahnfabrik GmbH, Bad Sackigen, Germany) was recalibrated according to the manufacturer's recommendation. During the color measurements, the specimens were placed on a gray background and the L, a, and b values were recorded. The difference in color (ΔE) was calculated through differences in the color parameters using the following equation: =[(L1-L0*)2+(a1*-a0*)2+(b1*-b0*)2]1/2
RESULTS: there was a statistically significant difference in ΔE between the tested groups. The lowest value was recorded for CS and MS groups. On comparing to the classical vita shade tabs, ΔE values for the studied groups was in the acceptable value except when compared to C1 and B2 shade tabs.
CONCLUSIONS: The sintering process affected the optical and color parameters of zirconium specimens especially for SS group.
RUNNING TITLE: Effect of Zirconia Sintering Techniques on Cielab Color Coordinates.

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INTRODUCTION

Polycrystalline Zirconium (Y-TZP) have become increasingly common in the fabrication of fixed prostheses in dentistry due to their biocompatibility and their excellent mechanical properties achieved by transformation toughening which results in a material strength of up to 1200 MPa. These restorations can be used in stress bearing areas and massive tooth loss. However, one of the main drawbacks with such restorations was their opaque properties resulting in unesthetic and dull restorations. That's why they are being used in unesthetic areas or posterior tooth replacements in the oral cavity and/or to be used as a core material.(1)
To attain sufficient space for the veneering porcelain to hide the opacity of zirconia and to match the optical properties of adjacent teeth, a substantial reduction in tooth structure is needed.(1) Even though the material had sufficient mechanical properties, a common problem with these restorations was their liability for chipping or delamination (loss of veneering material) in single crowns around 6% to 10% in 3 to 5 years respectively, and between 20% to 32% respectively in 5 to 10 years in FPDs.(2–4) A call for monolithic zirconia started gaining ground to overcome the problems with veneered zirconia restorations leading to the introduction of more translucent zirconia materials for the fabrication of restorations that match the patient's esthetic requirements without compromising strength.(5–9)
The main commercial method of increasing the translucency of Y- TZP is by reducing porosity of the material, decreasing grain size, and eliminating any alumina used as a sintering aid(1) Y-TZP is comprised of stabilized tetragonal zirconia, which is birefringent

with an anisotropic refractive index. This makes the material reflect and refract light at the grain boundaries, which reduces light transmittance though the material making it opaque. In theory, to make zirconia substantially translucent without compromising strength, particle grain size has to be decreased below 100-nm so light may penetrate the material without scattering at different thicknesses of material. The smaller the grain size the better the translucency. (10)

Currently, the most popular method of fabricating zirconia restorations is using CAD/CAM technology, however, after milling, the zirconia still needs to be sintered to reach the highest particle density of the material and maximum strength. The most common sintering method for zirconia uses conventional furnaces with holding temperatures between 1350 - 1400ºC which is maintained for 2 – 4 hours. (11) This process usually results in a sintering cycle of around 10 hours, which prompted the introduction of an alternative shorter cycle, speed sintering, to help save time and be more economical. Speed sintering relies on zirconia being sintered at a temperature of 1500 – 1600 ºC, held for 30 minutes. (12)

Microwave sintering has been proven successful when used for a variety of oxide and non-oxide ceramics, with specific benefits including improved mechanical properties derived from a fine grain microstructure produced through the volumetric heating and shorter processing time involved in the procedure. (13) Contrary to conventional firing in which the radiant heat starts from the surface of zirconia reaching the core by thermal conduction, in microwave sintering, each constituent unit of the crystal structure is excited individually, resulting in uniform distribution of heat.(14)

Multiple studies found that the changes in the sintering temperatures had a direct effect on the grain size of zirconia (15), its color change, translucency, and contrast ratio (16–18). The aim of this study is to evaluate the color difference of zirconia after sintering using three different sintering cycles: conventional sintering, speed sintering and microwave sintering.

The null hypothesis is that there is no difference in color between the three tested groups.

MATERIALS AND METHODS

Thirty zirconia disc specimens were milled from Zenostar Zr Translucent zirconia, shade T2 (Wieland Dental, Pforzheim, Germany) using Kavo Everest engine milling machine (KaVo Dental, Charlotte, NC, USA).

After zirconia blocks were milled, they were separated from the remaining of the blank using a diamond bur mounted on straight hand piece and placed under infrared lamp to dry. The final dimensions of the discs were 10 mm diameter x 2 mm thickness.

Grouping of the specimens

The thirty CAD/CAM pre-sintered zirconia discs were randomly divided into equal groups according to the sintering technique used in this study (n=10);

Group I: Microwave sintering (MS)

Ten zirconia discs were placed in the ceramic holder and placed in the middle of the microwave assisted furnace. The temperature was raised at a rate of 6°C/minute, until it reached the coupling temperature for zirconia (800°C) then the microwave was turned on at 100% power. The temperature continued to rise until it reached the target temperature (1500°C), and then held at this temperature for 2 hours. The furnace was then allowed to cool using the furnace cooling fans to room temperature at its own rate.

Group II: Speed sintering (SS)

Ten zirconia discs were sintered in Sirona Infire HTC Speed furnace (Sirona Dental Systems Inc, NY, USA) using speed sintering cycle according to the parameters recommended by the manufacturer. The temperature was raised until it reached the target temperature (1550 °C) and was held at this temperature for 30 min.

Group III: Conventional sintering (CS)

Ten zirconia discs were sintered in Sirona Infire HTC Speed furnace (Sirona Dental Systems Inc, NY, USA) using conventional sintering cycle according to the parameters recommended by the manufacturer. The temperature was raised until it reached the target temperature (1500 °C) and was held at this temperature for 2 hours.

All sintering parameters are shown in table (1).

Color Measurement

The spectrophotometer Easyshade V (VITA Zahnfabrik GmbH, Bad Sackigen, Germany) was adjusted in accordance with the manufacturer's recommendations prior to each color measurement. The operator's eyes were closed to which groups the specimens belonged. Using the values of the Commission Internationale de l'Eclairge L*a*b (CILAB), the color of each specimen has been recorded. (19). During the color measurements, the specimens were placed on a gray background and the L, a, and b values were recorded. The difference in color (ΔE) was calculated through differences in the color parameters using the following equation:

$$\Delta E^{*}=[(L1^{*}-L0^{*})^{2}+(a1^{*}-a0^{*})^{2}+(b1^{*}-b0^{*})^{2}]^{1/2}$$

Statistical Analysis

Data was collected and analyzed using IBM SPSS for Windows (Version 23.0) and significance was inferred at p value <0.05. Normality was checked for all variables using descriptive statistics, plots (histogram, boxplots, and Q-Q plots), and normality tests. All variables showed normal distribution, so means and standard deviation (SD) were calculated, and parametric analysis was adopted. Comparisons of color coordinates and color change/difference (ΔE) between the three study groups were done using One-way ANOVA, followed by multiple pairwise comparisons using Bonferroni adjusted significance level. Two-way ANOVA was performed to assess the influence of both heating technique and choice of shade tabs on color change (ΔE) with calculation of adjusted means, standard error (SE), and 95% confidence intervals (CI).

RESULTS

The mean color coordinates of the tested groups are listed in Table 2. The One-way ANOVA test showed significance between the study groups regarding each color coordinate (p,.05). In the post-hoc test, group SS showed the highest values in L and h values, and the lowest values for b and c, which were statistically significant from the other 2 groups (p<.05). All post-hoc pairwise comparisons for the color coordinates are listed in Table 3.

Table 1: Sintering parameters for the 3 study groups

	Temp.1 (°C)	Temp.2 (°C)	Heating rate (°C/ min)	Holding time (mins)
Microwave Sintering				
Heating phase	-	800	6	-
Heating phase*	800	1500	6	-
Holding phase	1500	1500	-	120
Cooling phase	1500	Room temperature	-	-
Speed Sintering				
Heating phase	-	1550	25	-
Holding phase	1550	1550	-	30
Cooling phase	1550	300	13.3	-
Conventional Sintering				
Heating phase	-	900	10	-
Holding phase	900	900	-	30
Heating phase	900	1500	3.33	-
Holding phase	1500	1500	-	120
Cooling phase	1500	900	10	-
Cooling phase	900	300	8.33	-

*Microwave assisted sintering

Table 2: Color coordinates (L, a, b, c, h) in the three study groups

	Microwave (n=10)	Conventional (n=10)	Superspeed (n=10)	P value
	Mean ± SD			
L	83.41 ± 0.64 **a**	84.94 ± 0.61 **b**	86.03 ± 0.66 **c**	**<0.001***
a	-0.38 ± 0.14 **a**	-0.07 ± 0.15 **b**	-0.52 ± 0.11 **a**	**<0.001***
b	15.74 ± 0.31 **a**	14.64 ± 0.40 **b**	13.14 ± 0.46 **c**	**<0.001***
c	15.74 ± 0.31 **a**	14.64 ± 0.40 **b**	13.25 ± 0.64 **c**	**<0.001***
h	91.35 ± 0.46 **a**	90.26 ± 0.62 **b**	92.33 ± 0.54 **c**	**<0.001***

One-Way ANOVA was used
*statistically significant at p value <0.05
a,b,c: different letters denote statistically significant differences between groups using Bonferroni adjusted significance level

Table 3: Post-hoc pairwise comparisons of color coordinates between the three study groups using Bonferroni adjusted significance levels

Measure	Group	Compared to	Mean difference (SE)	P value
L	Microwave	Conventional	-1.53 (0.28)	**<0.001***
		Superspeed	-2.62 (0.28)	**<0.001***
	Conventional	Superspeed	-1.09 (0.28)	**0.002***
a	Microwave	Conventional	-0.31 (0.06)	**<0.001***
		Superspeed	0.14 (0.06)	0.09
	Conventional	Superspeed	0.45 (0.06)	**<0.001***
b	Microwave	Conventional	1.10 (0.18)	**<0.001***
		Superspeed	2.60 (0.18)	**<0.001***
	Conventional	Superspeed	1.50 (0.18)	**<0.001***
c	Microwave	Conventional	1.10 (0.21)	**<0.001***
		Superspeed	2.49 (0.21)	**<0.001***
	Conventional	Superspeed	1.39 (0.21)	**<0.001***
h	Microwave	Conventional	1.09 (0.24)	**<0.001***
		Superspeed	-0.98 (0.24)	**0.001***
	Conventional	Superspeed	-2.07 (0.24)	**<0.001***

SE: Standard Error
*statistically significant using Bonferroni adjusted significance level

When comparing each group sample to known shade tabs (A1, A2, B1, B2, C1), one-way ANOVA showed significant difference in ΔE between the study groups when compared to all the shade tabs. The lowest mean ΔE value was recorded for MS (2.50 ± 0.31) when compared with shade tab A1, however, there was no significant difference (p>0.5) when compared with group CS (2.75 ± 0.50). The highest mean ΔE values were recorded between the study groups and shade tab C1, 7.35 ± 0.56, 8.56 ± 0.59 and 9.55 ± 0.67 for groups MS, CS and SS respectively. Comparisons between the groups and shade tabs are listed in Tables 4 and 5.

Two-way ANOVA test showed that the sintering technique and choice of shade tab had a significant difference (p<.05) on color change (ΔE) as shown in Table 6.

When comparing the study groups to each other, the highest ΔE (3.72 ± 0.75) was found between speed sintering and microwave sintering, however this difference is considered within the clinical accepted range (ΔE 3-5) as shown in Table 7.

Table 4: Color change (ΔE) in the three study groups against different shade tabs

ΔE	Microwave (n=10)	Conventional (n=10)	Superspeed (n=10)	P value
	Mean ± SD			
Against A1	2.50 ± 0.31 **a**	2.75 ± 0.50 **a**	3.49 ± 0.66 **b**	**0.001***
Against A2	4.04 ± 0.64 **a**	5.85 ± 0.59 **b**	7.67 ± 0.68 **c**	**<0.001***
Against B1	5.22 ± 0.35 **a**	5.65 ± 0.49 **a,b**	5.97 ± 0.58 **b**	**0.006***
Against B2	4.21 ± 0.66 **a**	6.09 ± 0.59 **b**	7.78 ± 0.58 **c**	**<0.001***
Against C1	7.35 ± 0.56 **a**	8.56 ± 0.59 **b**	9.55 ± 0.67 **c**	**<0.001***
Average	4.66 ± 1.69 **a**	5.78 ± 1.94 **b**	6.89 ± 2.13 **c**	**<0.001***

One-Way ANOVA was used
*statistically significant at p value <0.05
a,b,c: different letters denote statistically significant differences between groups using Bonferroni adjusted significance level

Table 5: Post-hoc pairwise comparisons of color change (ΔE) between the three study groups using Bonferroni adjusted significance levels

Measure	Group	Compared to	Mean difference (SE)	P value
ΔE against A1	**Microwave**	**Conventional**	-0.25 (0.23)	0.83
		Superspeed	-0.99 (0.23)	**0.001***
	Conventional	**Superspeed**	-0.74 (0.23)	**0.01***
ΔE against A2	**Microwave**	**Conventional**	-1.82 (0.28)	**<0.001***
		Superspeed	-3.63 (0.28)	**<0.001***
	Conventional	**Superspeed**	-1.82 (0.28)	**<0.001***
ΔE against B1	**Microwave**	**Conventional**	-0.43 (0.22)	0.16
		Superspeed	-0.75 (0.22)	**0.005***
	Conventional	**Superspeed**	-0.32 (0.22)	0.46
ΔE against B2	**Microwave**	**Conventional**	-1.88 (0.29)	**<0.001***
		Superspeed	-3.57 (0.29)	**<0.001***
	Conventional	**Superspeed**	-1.69 (0.29)	**<0.001***
ΔE against C1	**Microwave**	**Conventional**	-1.21 (0.27)	**<0.001***
		Superspeed	-2.20 (0.27)	**<0.001***
	Conventional	**Superspeed**	-0.99 (0.27)	**0.003***
Average	**Microwave**	**Conventional**	-1.12 (0.39)	**0.01***
		Superspeed	-2.23 (0.39)	**<0.001***
	Conventional	**Superspeed**	-1.11 (0.39)	**0.01***

*statistically significant using Bonferroni adjusted significance level

Table 6: Two-way ANOVA assessing the influence of sintering technique and shade tab on color change (ΔE)

		Adjusted mean (SE)	95% CI	P value
Group	**Microwave**	4.66 (0.11) **a**	4.45, 4.88	**<0.001***
	Conventional	5.78 (0.11) **b**	5.57, 5.99	
	Superspeed	6.89 (0.11) **c**	6.68, 7.11	
Shade tab	**A1**	2.91 (0.14) **a**	2.64, 3.19	**<0.001***
	A2	5.85 (0.14) **b**	5.58, 6.13	
	B1	5.61 (0.14) **b**	5.34, 5.89	
	B2	6.03 (0.14) **b**	5.75, 6.30	
	C1	8.49 (0.14) **c**	8.21, 8.76	

SE: Standard Error, CI: Confidence Interval
Model F: 169.27, p value <0.001*
a,b,c: different letters denote statistically significant differences between groups using Bonferroni adjusted significance level

Table 7: Comparison of color difference (ΔE) of microwave and superspeed groups against conventional group and against each other

	Mean ± SD	One-way ANOVA P value
Microwave against conventional	2.01 ± 0.88 a	<0.001*
Microwave against superspeed	3.72 ± 0.75 b	
Superspeed against conventional	2.18 ± 0.53 a	

*statistically significant at p value <0.05

a,b: different letters denote statistically significant differences between groups using Bonferroni adjusted significance level

DISCUSSION

Tetragonal zirconia polycrystals (Y-TZP) became the first alternatives used as a substructure in fixed prosthetic restorations, thanks to their white color, biocompatibility, and good mechanical properties.(20,21) The traditional method was fabricating a zirconium core to be veneered with ceramics, (22)but the thermal stresses generated at the interface may lead to chipping fractures, which is considered to be the most common cause for clinical failure.(23)

Monolithic zirconia is sintered from 1300C to 1650C. changing the firing conditions, such as the final sintering temperature or the heating mode, may be an alternative to fabricate full anatomic monolayer restoration without veneering ceramic in attempt for amore translucent esthetic final restoration.(11,22)

This study attempted to evaluate the effect of sintering on the color coordinates of monolithic zirconium by altering the sintering process. The results in this study showed that altering the processing technique affect the color parameter of translucent zirconium restoration, hence the null hypothesis is rejected as a significant difference in the optical properties were noticed between the studied groups (21).

Speed and Microwave sintering cycle was chosen in this study as new sintering techniques that can accommodate shorter sintering cycles. These short-time or speed sintering cycles embrace new prospects since a restoration can be milled and sintered in only one day without affecting both mechanical and optical properties.(24)

The clinical advantage was that changing the sintering parameters without affecting the optical properties of the zirconia restoration would help in a fast delivery for the patient without affecting its mechanical properties.

A Vita easy shade spectrophotometer is a reliable and valid digital instrument used for color measurement. That's why it is used in the present study.(25)

Since the human perception of color appears to be subjective, the color appearance of ceramics is primarily determined by the spectral reflectance generated from light scattering at the surface. This can be quantified scientifically using the Commission Internationale de l'Eclairage (CIE) system (21). The quantitative difference of color appearance (ΔE_{diff}) that indicated "clinically imperceptible" as $\Delta E_{diff} <3$, "clinically acceptable" as ΔE_{diff} 3-5, and "clinically unacceptable" as $\Delta E_{diff}>5$ seems practical for clinical practice (21,26).

In the present study, a significant difference was noticed between the three studied groups, owing to the increase in the holding time and the heating rate which had been reported to affect the grain size, microstructure, and affecting the optical properties zirconia (27). As the grain size enlarges, zirconia may spontaneously turn into vulnerable t- to m- phase transformations, induce alteration in optical appearance (28,29).

Although ΔE was significantly changed among different sintering techniques, the amount of changing was within clinically acceptable limit (ΔE diff=3-5) compared to standard clinical color appearance, 2.01,2.18 & 3.72. With the highest difference recorded for speed sintering group which clarify the effect of shortening the holding time and the sintering duration on decreasing the optical properties of translucent zirconia, like previous studies as Ebeid et al 2014, Juntavee et al 2018 (19,21).

The results in the present study agreed and was consistent with Salah et al 2023(30), Ebeid etal 2014(18). ΔE will decrease by increasing the firing temperature. This can be justified as the material density increase with longer heat treatment cycle.(31,32) Increased sintering time leads to the densification of zirconia particles, thus decreasing porosities between the grain boundaries by solid-state diffusion, which enhances densification and consequently affect its optical properties.(21)The increased zirconia density will lead to reduced light scattering at the interface between adjoining crystals, enhancing spectral reflection and optical transmission with less refraction, leading to improved color.(19)

For the L a b Values for the three tested groups, all the tested groups showed increased translucency and lightness values ranging from 83 to 86 which showed that all have similar lightness values. Upon raising the sintering temperature, the zirconia particles could join, causing decrease in the porosity between the grain boundaries during solid-state diffusion phase, and leading to increase in the material density and better lightness parameters. For the b value, which represent yellow-blue scale, all the tested groups showed a positive value representing a yellow scale, which decreased significantly in the SS group which showed the lowest value, owing to the short treatment cycle time which affected the crystal orientation and maturation. the long sintering time tend to achieve higher brightness than short sintering duration, which was supported by other studies (16,18,27,28).

On comparing the studied groups to Vita classical shade guide (Vita Zahnfabrik, Säckingen, Germany), it was noticed that ΔE difference was least upon comparing to A1 shade tab with ΔE = MS 2.50 ± (0.31), CS 2.75 ± (0.50) , SSS 3.49 ± (0.66), which confirm that upon decreasing the holding time and heating rate, a more color change can be detected as reported in previous studies (19,21) so based on color perception, the study can assure clinician's confidence to have a monolithic zirconium restoration from the dental laboratory without detectable color difference from the selected standard shade guide even if the sintering process was altered.

On comparing the MS, CS, & SS groups, a significant increase in the ΔE was noticed, this could be attributed that the treatment process decreases the pores between the grains and increases zirconium density, so less scattering of light and more transmission occur to achieve better translucency, brightness, and optical characteristics(19,33). This agreed with our results as increasing the sintering temperature and time led to better specular reflection, light transmission, and a better perception of color.

On comparing with other vita classical shade tabs, a more color difference was noted owing to the darker and less bright shades in comparison to the translucent nature of the tested zirconium blank, so the difference was more obvious and easily detected especially regarding B2 & C1 shade tabs as the change became unacceptable clinically above 6 and more. These changes were noted clearly owing to superspeed sintering group which emphasized that shortening the sintering cycle will affect the crystal orientation and its maturation and so more obvious changes can be detected easily.

A limitation of this study is that only one zirconium type was evaluated. The results may not apply for other brands of monolithic zirconia thus other brands may require further investigations. In addition, the effect of aging was not investigated in our study and therefore also requires further investigation.

CONCLUSIONS

1- Increasing the sintering time and temperature will improve the optical properties of translucent zirconium.
2- Speed sintering showed the most obvious color changes.
3- Significant color change ΔE for C1 shade tab in all the tested groups.

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