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Effect of Various Sintering Protocols on The Translucency of Highly Translucent Cubic Zirconia

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ABSTRACT

Aim of the study: This study was carried out to evaluate the effect of various sintering protocols on the translucency of highly translucent cubic zirconia. Materials and methods: A total of forty discs of two types of zirconia ceramics were used in this study. The specimens were divided into two main groups according to the type of zirconia; Group 1 (n=20): Cubic zirconia (DD Cube X2 98color) and Group 2 (n=20): Tetragonal zirconia (DD Bio ZX2 color). Each group was subdivided into two subgroups, where 10 discs were per subgroup according to the sintering protocol. Cubic and tetragonal zirconia blanks of dimensions (98 mm diameter \times 25 mm thickness) were CAD/CAM milled into cylindrical-shaped blocks of dimensions (15 mm diameter \times 12 mm thickness) to compensate for the approximately 23% shrinkage of the material during sintering, so as the final dimentions would be (12 mm diameter \times 1 mm thickness). Discs were dried under a heating lamp and then conventional sintering showed statistically significantly lower mean CR and higher mean TP than speed sintering (P-value <0.001). Conclusion: Different sintering protocols showed a significant effect on the translucency of cubic and tetragonal zirconia

1. INTRODUCTION

The use of zirconium dioxide-based ceramics in dental field has grown due to their high mechanical properties. However, opacity remains their main drawback. In an attempt to overcome this problem, ceramic veneering of zirconia has been advocated.^(1,2) However, veneer chipping remains the most frequent failure.^(3,4) The evolution of monolithic zirconia restorations has served as a solution to avoid veneering drawbacks.⁽⁵⁻⁷⁾

In an attempt to improve the translucency of zirconia, changes in microstructure and sintering process have been made that led to the development of translucent zirconia.⁽⁸⁻¹⁰⁾ Despite being suitable for fabrication of monolithic posterior restorations, it lacks sufficient translucency for use in anterior zone.⁽¹¹⁾ This eventually led to the development of highly translucent cubic zirconia, that most manufacturers claim that it has sufficient translucency, strength, and color matching with natural teeth to be used for single restorations anywhere in the mouth.^(12,13)

Milling technology is used in fabrication of dental zirconia prosthesis.⁽¹⁴⁾ Zirconia milling processes are either hard or soft. Milling of fully sintered zirconia is denoted as hard milling, and may cause wear of the milling bur. While milling of partially sintered zirconia is denoted as soft milling, as subsequent sintering is needed to achieve fully dense pieces.^(15,16)

Soft milled dental zirconia is conventionally sintered where the powder compact is heated to the sintering temperature for a given time in a furnace. ^(17,18) However, conventional sintering is a process that consumes high energy and also time, as involves a slow rate of heating and cooling coupled with a prolonged holding time (sintering could be up to 12 hours); thus prohibits chair-side treatment.⁽¹⁹⁻²¹⁾

Therefore, manufacturers introduced rapid sintering cycles that involves an increased rate of heating and/or decreased holding time at peak temperature.⁽²²⁾ The change in the parameters of sintering can influence zirconia's microstructure and consequently, reflects on its optical properties.⁽²³⁾ This aim of this study was to evaluate the translucency of commercially available dental zirconia ceramics of different generations, after conventional and speed sintering. The null hypothesis of this study was that the translucency of both cubic and tetragonal zirconia would not be affected by the various sintering protocols.

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2. MATERIALS AND METHODS

Materials

The materials used, their specifications, compositions, and manufacturer are listed in table (1).

Table 1.

Brand names, composition, and manufacturer of zirconia used in this study.

Materials	Brand names	Composition	Manufacturer	Shade	Batch Number
Cubic zirconia	DD CubeX ^{*®} • • •	$\begin{array}{l} ZrO_2 + HfO_2 + Y_2O_3 \geq 99.0 \\ Y_2O_3 < 10 \ (5 \ mol\%; \ 5Y\text{-}PSZ) \\ Al_2O_3 \leq 0.01 \\ Other \ oxides \leq 1 \end{array}$	Dental Direkt materials Germany	A3.5	8161706005
Tetragonal Zirconia	BioZX ²	$\begin{array}{l} ZrO_2 + HfO_2 + Y_2O_3 \geq 99.0 \\ Y_2O_3 < 6 \\ Al_2O_3 \leq 0.15 \\ Other \ oxides < 1 \end{array}$	Dental Direkt materials Germany	A3.5	6161832007

Study design

Forty zirconia specimens were constructed and divided into two groups according to the type of zirconia. Group (1): (n= 20) cubic zirconia DD CubeX^{*®}. Group (2): (n= 20) tetragonal zirconia DD Bio ZX². Each group was subdivided into two subgroups according to the sintering protocol, 10 discs each. Subgroup (A): (n= 10) specimens were subjected to conventional sintering protocol. Subgroup (B): (n= 10) specimens were subjected to speed sintering protocol.

Specimens Preparation

DD CubeX³⁰ and DD Bio ZX² zirconia blanks of dimensions (98 mm diameter \times 25 mm thickness) were CAD/CAM milled by a milling machine^{*} into cylindrical-shaped blocks of dimensions (15 mm diameter \times 25 mm thickness). Then disc-shaped specimens of dimensions (15 mm diameter \times 1.2 mm thickness), to compensate for the approximately 23% shrinkage, were obtained by sectioning the cylindrical blocks using Isomet diamond cutting saw^{**}, so as the final dimensions of the specimens after sintering would be (12 mm diameter \times 1 mm thickness).

Drying of zirconia specimens

Specimens were dried under a heat radiating infrared lamp^{***} for 30 minutes, as according to the sintering furnace manufacturer's instructions, any residual moisture is very likely to cause damage to the specimens.

Sintering of zirconia specimens

Conventional sintering protocol, subgroup (A): The specimens were sintered in a sintering furnace^{****} according to the manufacturer's sintering protocol, (Table 2). DD CubeX^{2®} and DD BioZX² subgroups were sintered simultaneously, each in a single sintering cycle.

** (ISOMET 4000, Buehler, Lakebluff, U.S.A).

Table 2

Conventional sintering protocol for DD ${\rm Cube}X^2 \ensuremath{\mathbb{R}}$ and DD Bio ZX^2 according to the manufacturer.

	Temp. 1 [°C]	Temp. 2 [°C]	Heating rate [°C/min]	Dwell time [min]	Time [min]
Heating	20	900	8	-	110
Dwell	900	900	-	30	30
Heating	900	1450	3	-	183
Dwell	1450	1450	-	120	120
Cooling	1450	200	10	-	125
					Total time: 568 min.

Speed sintering protocol, Subgroup (B): The specimens were sintered according to the manufacturer's sintering protocol illustrated in table (3), following the same procedures used for sintering specimens of subgroup (A).

Table 3:

Speed sintering protocol for DD CubeX2® and DD Bio ZX2 according to the manufacturer.

	Temp. 1 [°C]	Temp. 2 [°C]	Heating rate [°C/min]	Dwell time [min]	Time [min]
	20	990	60	-	16
Heating	990	1350	10	-	36
	1350	1450	15	-	7
Dwell	-	1450	-	50	50
	1450	1350	10	-	10
Cooling	1350	990	40	-	9
					Total time: 128 min.

^{* (}Roland, California, USA).

^{*** (}Bredent, Senden, Germany).

^{**** (}In Fire HTC speed furnace; Sirona, Long Island City, NY, USA).

Checking final dimensions of specimens

After sintering, Specimens' dimensions were confirmed to be accurate within 0.1 mm with a digital caliper^{*}. If discrepancy more than 0.1 mm was recorded, sample was discarded and the previous procedures were repeated.

Testing procedure

Quantitative measurement of translucency was obtained by measuring the Y tristimulus values and CIELAB coordinates of the specimens after backing with a black (L* = 2.06, a* = -0.46, b* = 1.10, Y= 0.1810) and white (L* = 99.85, a* = -0.01, b* = -0.15, Y= 99.6120) background using the spectrophotometer.**

Translucency evaluation

Translucency parameter (TP)

The translucency parameter was calculated from the color difference of each specimen when analyzed against the black and white background, according to the formula: $\text{TP} = [(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2]^{1/2(24)}$ TP is expressed as a value ranging from 0 to 100, as greater values correspond to higher levels of translucency.

Contrast ratio (CR)

The contrast ratio was obtained using the CIE XYZ system, and was calculated using the equation: $CR = Y_b/Y_w$.⁽²⁵⁾ In all calculations "0" is fully transparent and "1" is fully opaque.

Statistical Analysis

Data were explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests, data showed parametric (normal) distribution. Two-way ANOVA was used to study the effect of zirconia type, sintering and their interaction on mean TP, and CR. Bonferroni's post-hoc test was used for pairwise comparisons when ANOVA test is significant. The significance level was set at $P \le 0.05$. Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY: IBM Corp.

3. RESULTS

Effect of sintering protocols on translucency parameter

Regardless of sintering, zirconia type had a statistically significant effect on mean TP. Regardless of zirconia type, sintering had a statistically significant effect on mean TP. The interaction between the variables had no statistically significant effect on mean TP, and thus, the variables are independent from each other. Data are presented numerically in table 4.

Comparison between sintering protocols

For both cubic or tetragonal zirconia, conventional sintering showed statistically significantly higher mean TP than speed sintering (*P*-value <0.001). Data are presented numerically in table (5) and graphically in figure (1).

* (Mitutoyo, Tokyo, Japan).

Table 4:

Two-way ANOVA results for the effect of different variables on mean TP.

Source of variation	Type III Sum of Squares	df	Mean Square	F-value	P-value	Effect size (Partial eta squared)
Zirconia type	131.515	1	131.515	1679.993	<0.001*	0.979
Sintering	37.617	1	37.617	480.52	<0.001*	0.93
Zirconia type x Sintering interaction	0.15	1	0.15	1.917	0.175	0.051

df: degrees of freedom = (n-1), *: Significant level $P \le 0.05$.

Table 5:

The mean, standard deviation (SD) values and results of two-way ANOVA test for comparison between TP values with different interactions of variables.

o: , ;	Cul	oic	Tetrag	gonal	<i>P</i> -value	Effect size	
Sintering	Mean	SD	Mean	Mean SD zirconia type		(Partial Eta Squared)	
Conventional sintering	13.32	0.19	9.82	0.25	<0.001*	0.944	
Speed sintering	11.5	0.23	7.76	0.4	<0.001*	0.951	
<i>P</i> -value (Effect of sintering)	<0.001*		<0.001*				
Effect size (Partial Eta Squared)	0.854		0.8	83			

*: Significant level $P \leq 0.05$.

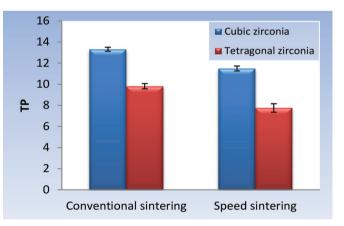


Figure (1) — Bar chart representing mean and standard deviation values for TP with different interactions of variables.

Effect of sintering protocols on Contrast ratio

Regardless of sintering, zirconia type had a statistically significant effect on mean CR. Regardless of zirconia type, sintering had a statistically significant effect on mean CR. The interaction between the variables had no statistically significant effect on mean CR, and thus, the variables are independent from each other. Data are presented numerically in table 6.

^{** (}Cary 5000 UV-Vis-NIR Spectrophotometer; Agilent Technologies)

Table 6:

Two-way ANOVA results for the effect of different variables on mean CR.

Source of variation	Type III Sum of Squares	df	Mean Square	F-value	P-value	Effect size (Partial eta squared)
Zirconia type	0.086	1	0.086	2412.815	<0.001*	0.985
Sintering	0.027	1	0.027	758.231	< 0.001*	0.955
Zirconia type x Sintering interaction	0.00001	1	0.00001	0.37	0.547	0.01

df: degrees of freedom = (n-1), *: Significant level $P \le 0.05$.

Comparison between sintering protocols

For both cubic or tetragonal zirconia, conventional sintering showed statistically significantly lower mean CR than speed sintering (*P*-value <0.001). Data are presented numerically in table (7) and graphically in figure (2).

Table 7:

The mean, standard deviation (SD) values and results of two-way ANOVA test for comparison between CR values with different interactions of variables.

	Cu	Cubic		gonal	P-value (Effect of	Effect size
Sintering	Mean	Iean SD Mean SD		zirconia type)	(Partial Eta Squared)	
Conventional sintering	0.734	0.006	0.826	0.008	<0.001*	0.961
Speed sintering	0.785	0.004	0.879	0.005	<0.001*	0.963
<i>P</i> -value (Effect of sintering)	<0.()01*	<0.001*			
Effect size (Partial Eta Squared)	0.	91	0.917			

*: Significant level $P \le 0.05$.

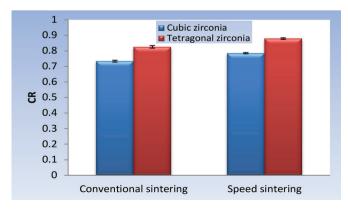


Figure (2) — Bar chart representing mean and standard deviation values for CR with different interactions of variables.

4. DISCUSSION

The impact of conventional sintering protocol and speed sintering protocol on the translucency of the second generation (tetragonal) and third generation (cubic) zirconia was the focus of this study. Quantifying and measuring the translucency can be done using spectrophotometer by three methods namely: light transmittance, contrast ratio (CR), and translucency parameter (TP).^(26,27) In this study, the translucency was evaluated by contrast ratio and translucency parameter, as they are commonly used for measuring ceramic's translucency.^(28, 29)

The stated null hypothesis was rejected as the translucency of cubic and tetragonal zirconia seemed to be well affected by the various sintering protocols as results revealed that conventional sintering showed a statistically significant higher mean TP and lower mean CR than speed sintering.

These results were in agreement with a study performed by Ebeid et al,⁽²³⁾ (2014) as they concluded that there was a decrease in CR as sintering time increases. The results of the current study were also in agreement with Juntavee et al,⁽³⁰⁾ (2018) where CR showed the highest values, and TP showed the least value when sintering time decreased. This study was also in agreement with Lawson et al,⁽³¹⁾ (2020) where the translucency of two cubic zirconia brands decreased when sintering holding time was 30 minutes compared to 2 hours holding time.

This could be explained by the capability of prolonged sintering time to increase the material's density by reduction of porosity as it is the main source of light scattering. When sintering time increases, zirconia particles can be able to join together, causing reduction of porosities between the grain boundaries upon solid-state diffusion, which enhances densification, and consequently, the translucency.^(30, 32)

Indeed, the results of this study indicated that various sintering protocols seem to play a role in determining the translucency of both types of zirconia. Although shorter sintering times are attractive as decreases time of treatment, however, conventional sintering is preferred for dental zirconia ceramics, as translucency mismatch may be evident with speed sintering. The present study has limitations. Only one brand of zirconia was tested which limits the results to this brand. Furthermore, data derived from specimens need to be verified in anatomical reconstructions in order to mimic the clinical condition.

5. CONCLUSION

Different sintering protocols showed a significant effect on the translucency of cubic and tetragonal zirconia.

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