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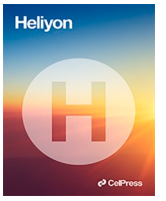

Research article

Effect of speed sintering process on the microstructure, flexural strength and translucency of zirconia

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A R T I C L E I N F O

Keywords:
Speed sintering
Flexural strength
Grain size
Multilayer zirconia
Translucency

A B S T R A C T

Objectives: To determine the impact of the speed sintering program on the microstructure, flexural strength and translucency of zirconia in comparison with those of the conventional sintering program.

Materials and methods: rectangular shape specimens (12.5 × 15.5 × 1.2 mm) were prepared from four commercial pre-sintered zirconia ceramics (KATANA HTML, KATANA STML, InCoris TZI and InCoris ZI) that were sintered with conventional and speed sintering programs according to the manufacturer's instructions. The phase composition of the sintered specimens was determined by X-ray diffraction (XRD). The grain size was evaluated using scanning electron microscopy (SEM), while the three-point flexural strength was assessed based on the ISO 6872: 2015 standard. Translucency was assessed using a spectrophotometer. The data were analyzed using independent t tests ($\alpha = 0.05$) and one-way ANOVA.

Results: The XRD patterns were similar for all the groups, indicating that there was no phase transformation. SEM revealed that the average grain size was lower than 1 μm. The grain size, flexural strength and translucency results showed increasing trends when speed sintering is compared with the conventional one but the differences were not significant ($P > 0.05$).

Conclusions: The results of this research indicate that the speed sintering program had no significant impact on the microstructure, flexural strength and translucency of the examined zirconia, a speed-sintering program can process the ceramic material within a short time with slightly increase their flexural strength and translucency Therefore, a speed-sintering program is appropriate for zirconia (Y-TZP).

1. Introduction

Zirconia has gained considerable attention owing to its exceptional chemical stability, superior mechanical properties, biocompatibility, high aesthetic value, and low thermal conductivity [1,2]. Zirconia has polymorphism characteristics, meaning that it has the same chemical composition and occurs in several atomic configurations. It has three crystalline phases. monoclinic is stable at room temperature to 1170 °C. In contrast, the cubic phase becomes dominant at temperatures greater than 2370 °C, but the tetragonal phase exhibits stability within the temperature range of 1170 °C–2370 °C [3,4].

In response to stress, zirconia undergoes a phase change from the metastable tetragonal stage to the stable monoclinic stage, which is responsible for its excellent mechanical qualities [5]. Consequently, to obtain better mechanical characteristics, stabilizers such as

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CaO, MgO, CeO_2, and Y_2O_3 need to be added to the raw zirconia powder, and the resulting tetragonal phase should be maintained at room temperature. Y_2O_3 is the most common stabilizer in dental zirconia, and its amount has a significant impact on the material's characteristics.

The mole percent of yttria, typically expressed as a number (3, 4, or 5), often defines the mechanical and physical properties of zirconia. The variance of mole percentages, measured in tenths of a mole percent, explains the physical and mechanical characteristics of each category.

In the field of prosthodontics, the most commonly used Y2O3 stabilized zirconia are 3 mol% (3Y zirconia), 4 mol% (4Y zirconia), and 5 mol% (5Y zirconia). The strongest (85 %–90 % tetragonal phase) is opaque zirconia with approximately 3 mol% or 5 wt percent yttria. This composition had the highest flexural strength and fracture toughness, which were 1200–1500 MPa and 3.5–4.5 MPa·m1/2, respectively. Zirconia, which contained about 4 % or 7.1 % yttria, had a flexural strength of 600–900 MPa and a fracture toughness of 2.5–3.5A. Zirconia, containing about 5 mol%, or 8.8 wt%, yttria, produces a more translucent material with around 50 % cubic phase. 5 Y-TZP zirconias have a flexural strength of 700–800 MPa and a fracture toughness of 2.2–4 MPa·m1/2. Yttria increases the zirconia grain size and decreases the thermal expansion coefficient [6].

Translucency is a characteristic of substances that enables the passage of certain light waves while scattering or reflecting others. As light travels through a material, it undergoes a variety of interactions, including reflection, absorption, and transmission, which are all dependent on the material's properties and the wavelength of the light. Grain boundaries, crystallographic flaws, and micro-pores all affect the translucency of polycrystalline material on the inside surfaces of the Y-TZP [7]. Sintering zirconia at higher temperatures for longer periods of time can increase its transparency by increasing grain size. Generally, as the stabilizer concentration increases, the translucency increases; conversely, the mechanical properties decrease [8].

The sintering process of zirconia has been regarded as a crucial step in ceramic manufacturing and has garnered significant attention from many investigators [9,10]. Previous studies have demonstrated that changes in the sintering process have a direct effect on the density. including phase composition and particle size, which have a great impact on zirconia optical properties and mechanical properties [11].

The traditional sintering program for Y-TZP ceramics is often time consuming and costly [12]. So, speed sintering has been suggested as a prospective approach to achieve a more refined microstructure through the inhibition of particle growth using rapid heating while preserving a high material density and reducing processing time [13].

Several research investigations have been conducted to examine the effects of changing sintering conditions on the strength, microstructure, and optical characteristics of zirconia core ceramics. Nuri Murat Ersoy et al. [14] showed that the flexural strength of zirconia increased when a high sintering temperature and short sintering time were combined. Liu et al. [15] showed that changing the sintering program does not have a substantial impact on the flexural strength. However, there is still ongoing debate over the impact of changing sintering conditions on the strength and translucency monolithic zirconia [16,17]. Therefore, this work aimed to study the influence of speed sintering on the microstructure, flexural strength and translucency of zirconia. The null hypothesis that was investigated was that the speed-screening process would not have an impact on the microstructure, flexural strength, or translucency of zirconia.

2. Materials and methods

2.1. Sample grouping

For this in vitro investigation, 168 specimens were constructed from four pre-sintered Y-TZP blocks and disks were utilized and divided into four primary groups. The sample size of this study was determined with using G*Power 3.1.9.7 (a program developed by Franz-Faul at the University of Kiel in Germany).chemical composition and properties of material according to mamufacture was showed in Table (1).

Group A: high translucency multilayer zirconia (HTML) (Katana, Kuraray, Noritake)

Group B: Super translucent multilayer zirconia (STML) (Katana, Kuraray, Noritake)

Group C: InCoris TZI (TZI) zirconia (Sirona, Bensheim, Germany).

Group D: InCoris ZI (ZI) zirconia (Sirona, Bensheim, Germany).

Each group was further subdivided into two subgroups (I & II) according to the sintering programms used (I: Conventional sintering, II: Speed sintering) (Table 2)

Table 1
Chemical composition and the properties of the zirconia using in this study.

Material	manufacture	Flexural streng (MPa)	Fracture toughness (MPa m1/2)	ZrO2 + HfO%	(Y2O3) %
KATANA STML	Kuraray Noritake	600–800	2.2–2.7	88–93	7–10
KATANA HTML	Kuraray Noritake	900–1100	3.5–4.5	90–97	5–5.5
InCoris ZI (ZI)	Sirona, Bensheim, Germany	>900	5.8	≥99.0 %	>4.5 - ≤ 6.0 %
InCoris TZI (TZI)	Sirona, Bensheim, Germany	>900	7.1	≥99.0 %	>4.5 - ≤ 6.0 %

Table 2
Classification of the zirconia samples according to sintering program.

Group	Material	Subgroup	Sintering temperature	Holding time (min)	Total time (h)
A	HTML	AI	1500 °C	120 min	7.0
		AII	1515 °C	30 min	1.5
B	STML	BI	1550 °C	120 min	7.0
		BII	1560 °C	30 min	1.5
C	IncorisTZI	CI	1510 °C	120 min	8.0
		CII	1540 °C	25 min	2.0
D	IncorisZI	DI	1510 °C	120 min	8.0
		DII	1540 °C	25 min	2.0

2.2. Sample preparation

Rectangular shape samples (n = 80) were cut from CAD/CAM blocks and disks. The cutting procedure was performed with a diamond disc bur (manufactured by Detaurum, Germany) with a diameter of 30 mm and a thickness of 1 mm. The bur was affixed to a low-speed handpiece, revolved in a counterclockwise direction at a speed of 20,000 rpm and then ground by using 600 grit abrasive paper. The sample size was 19 × 15.5 × 1.6 mm. The samples were cut oversized by percentages specified by the manufacturers due to shrinkage associated with sintering of zirconia. After the cutting process, the dimensions of each of the samples were examined utilizing a digital caliper (Digital caliper, China) to an accuracy of 0.001 mm. Subsequently, a SpeedFire furnace (Dentsply Sirona, York, USA) was used for sintering the specimens following the sintering program provided by the manufacturer. After sintering the final dimension of samples was 12.5 × 15.5 × 1.2 mm. Eight additional specimens (n = 1 per sub-group) were prepared to be examined under SEM to study the effect of speed sintering versus conventional sintering on the microstructure of the different zirconia materials used in this study.

Also eighty samples (n = 10 per subgroup) were prepared with dimension of (12.5 × 15.5 × 0.5) to be examined under spectrophotometer to study the effect of speed sintering on the translucency of the different zirconia materials tested in this study.

2.3. Crystalline structure analysis (XRD)

X-ray diffraction for surface analysis was performed (n = 3per subgroup) using x-ray diffractometer (ADX2700; Nan-L-11, Angstrom Advanced Inc., America) to identify and determine the crystalline phases present. The samples were positioned within the diffractometer holder at room temperature and irradiated with Cu K α (Cu K-alpha). The scanning range spans from 10 to 90° with a step size of 0.5°, and each scan occurs at an interval of 2 s. The voltage was set at 40 kV, and the current was set at 30 mA. The time for irradiation was 4:30 min. The Rietveld analysis was performed for determining the phase composition and lattice parameters based on the cell unit parameters of the tetragonal ZrO2 phase (a, c), the Y2O3 content in the phase by using the X'Pert Plus software developed by Philips in Almelo, Netherlands, as described by Nonaka K et al. [18] and Gibson et al. [19].

2.4. Microscopic examination (SEM)

For grain size measurements and microstructure analyses, specimens (n = 1 per subgroup) were polished with a diamond paste (Polir Diamant, Celit, Russia), subjected to ultrasonic cleaning with acetone for 10 min and left to dry for 24 h at room temperature. Then, the samples were thermally etched (heating the polished sample at a temperature lower than the sintering temperature usually at 50–200 °C below the sintering temperature for a certain amount of time) at 1200 °C for 20 min to expose the grain boundary network

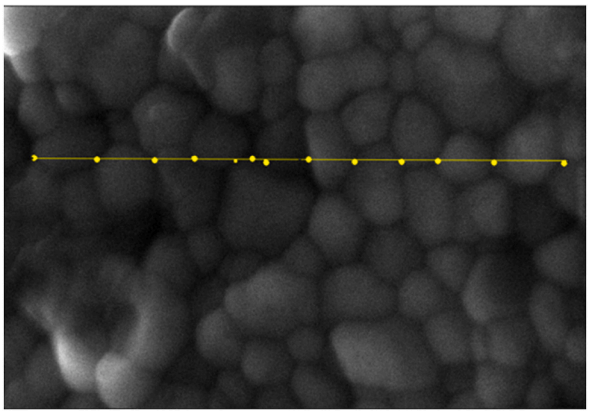

Fig. 1. Linear intercept method.

[18]. A scanning electron microscope (Quattro S- FE SEM, Thermo Fisher Scientific, USA) was used for evaluating the surface topography at 30000 × magnification. ImageJ software (NIH, Bethesda, MD, USA) was used to estimate the average grain size according to the linear intercept method (one or more test lines of known length was fixed in place over a photomicrograph of the polished section, and the number of intercepts between the test line(s) and grain boundaries were counted) [20]. (Fig. 1). The average grain size, denoted as ($\bar{D}$), was determined utilizing the following equation (1):

$$\bar{D} = 1.56\ C/MN. \quad (1)$$

where $\bar{D}$ is the average grain size, N is the number of intercepts, C is the overall length of the test line utilized, and M is the magnification of the photomicrograph.

2.5. Flexural strength test

Eighty samples (n = 10 per sub-group) with dimensions of 1.2.5 mm × 15.5 mm × 1.2 mm were subjected to three-point flexural strength estimation (ISO 6872:2015 standard) [21] in a universal testing machine (Wdw-50; 300 KN; LARYEE, China) under dry conditions at a cross head speed of 1 mm/min until failure. A digital caliper (Digital Vernier Caliper; Herman; Jiangxi, China) was used to measure the sample dimensions before the flexural strength test was performed.

The flexural strength was calculated using equation (2) as below:

$$\sigma = 3Nl/2bd^2 \quad (2)$$

where σ is the flexural strength, N is the fracture load (N), l is the space between the supports (mm), b is the sample width (mm) and d is the sample thickness (mm).

3. Translucency characterization

The UV–visible dual beam spectrophotometer (V360, Jasco, Japan) was used to measure the translucency of the zirconia sample (n = 10 per subgroup, thickness = 0.5 mm) on double-sided mirror-polished plate samples.

The spectrophotometer works on the principle of Beer–Lambert law. The Beer–Lambert law was an amalgamation of two laws each dealing independently with the absorption of light associated to the concentration of the absorber (the substance responsible for absorbing light) and the path length or thickness of the layer (related to absolute amount of the absorber). the ratio of the intensities of transmitted and incident light gives the transmittance T, expressed in equation (3): [22].

$$T = I/Io \quad (3)$$

where Io was the intensity of incident radiation and I was the intensity of transmitted radiation.

3.1. Statistical analysis

Data description, analysis, and presentation were conducted using the SPSS statistical program (SPSS, Version 20, Chicago, IL, USA). One-way ANOVA followed by Bonferroni correction was used for the statistical analysis. Additionally, an independent *t*-test was performed to compare two subgroups, with a significance level of α = 0.05.

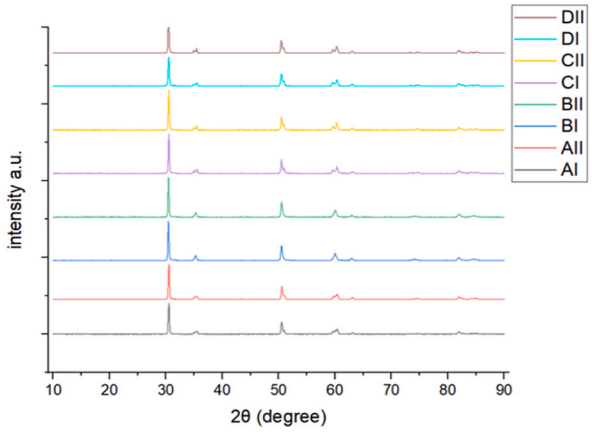

Fig. 2. X-ray diffraction patterns of zirconia sintered at various temperatures: AI, AII BI, BII. CI, CII, DI and DII.

4. Results

4.1. Phase characterization

The XRD patterns depict the zirconia phase composition of each zirconia ceramic, and the results of Rietveld analysis are shown in Fig. 2 and Table 3. All the groups exhibited similar XRD patterns, and no monoclinic phase peaks were observed in the X-ray diffraction (XRD) patterns.

According to the present investigation, the sintering program did not have a noteworthy effect on the observed phase transition on the surface of the zirconia samples.

4.2. Microstructural characterization

SEM images and grain sizes are shown in Fig. 3 and Table 4. The results demonstrated that speed sintering program had no impact on grain size and the speed sintering groups had slightly larger grains, and the results indicated that all four materials tended to produce larger grains as the sintering temperature increased.

4.3. Flexural strength test

The descriptive statistics for the flexural strength test, including the mean and standard deviation, are presented in Table 4. The results of this study show that in comparison to the conventional sintering groups, the speed sintering group has a high flexural strength. Statistical analysis revealed no significant difference in the subgroups' flexural strengths between conventional and speed-sintered zirconia ceramics, regardless of the increase in flexural strength.

4.4. Translucency characterization

The descriptive statistics for the translucency test, including the mean and standard deviation, are presented in Table 4. Fig. 4 presents the translucency of the zirconia ceramics. The results showed that the TP content was slightly greater in the speed sintering group than in the conventional group. The results also showed no statistically significant difference in translucency between subgroups subjected to conventional and speed sintering. However, the group with 5Y-TZP (BI&BII) showed increased translucency compared to the other groups.

5. Discussion

Zirconia restorations with high mechanical properties are the current treatment options for fixed restorations with advantages of high biocompatibility and low pulp irritation The sintering process is responsible for the strength of zirconia restoration. Alteration of sintering parameters alters the microstructural, mechanical, and optical properties of zirconia. This will consequently impact the clinical performance of zirconia prosthese [23]. Speed -sintering could be a viable alternative to conventional sintering procedures because it offers shorter sintering times, which are more cost-effective and time-efficient. Several studies investigated the effect of sintering parameter on zirconia mechanical and optical properties. The effect of these parameter of zirconia strength still uncertian. Therefore this study aimed to investigate and compare the effect of changing sintering parameters on the microstructure, flexural strength and translucency of monolithic zirconia.

The results of this study showed that the phase composition, grain size, flexural strength and translucency of four commercial

Table 3
Rietveld analysis of phase composition and lattice parameters.

Group	Sub-group	Tetragonal					cubic	
		Fraction (%)	c (Å)	a (Å)	Y_2O_3 (mol%)	$c/a\sqrt{2}$	Fraction (%)	c(Å)
A	**AI**	75 ± 2	5.1440 ± 0.002	3.6067 ± 0.006	4.3 ± 0.1	1.0085 ± 0.001	25 ± 2	5.0463 ± 0.006
	AII	75 ± 3	5.0917 ± 0.007	3.574 ±0.003	4.5 ± 0.3	1.0073 ± 0.007	25 ± 3	5.0664 ± 0.004
B	**BI**	46 ± 2	5.0773 ± 0.002	3.6090 ±0.008	6.0 ± 2	0.9948 ± 0.004	54 ± 2	5.0763 ± 0.001
	BII	46 ± 4	5.0667 ± 0.002	3.6093 ±0.007	6.2 ± 3	0.992 ± 0.002	54 ± 4	5.0776 ± 0.003
C	**CI**	81 ± 0.5	5.1427 ± 0.008	3.6153 ±0.004	4.6 ± 0.01	1.0058 ± 0.004	19 ± 0.5	5.0596 ± 0.000
	CII	81 ± 1	5.1397 ± 0.002	3.6153 ±0.001	4.7 ± 0.04	1.0052 ± 0.005	19.0.5	5.0626 ± 0.001
D	**DI**	83 ± 1	5.1427 ± 0.008	3.6053 ±0.001	4.6 ± 0.08	1.0058 ± 0.004	17 ± 1	5.0641 ± 0.005
	DII	83 ± 2	5.1400 ± 0.001	3.5047 ±0.001	4.7 ± 0.03	1.0055 ± 0.004	17 ± 2	5.0629 ± 0.007

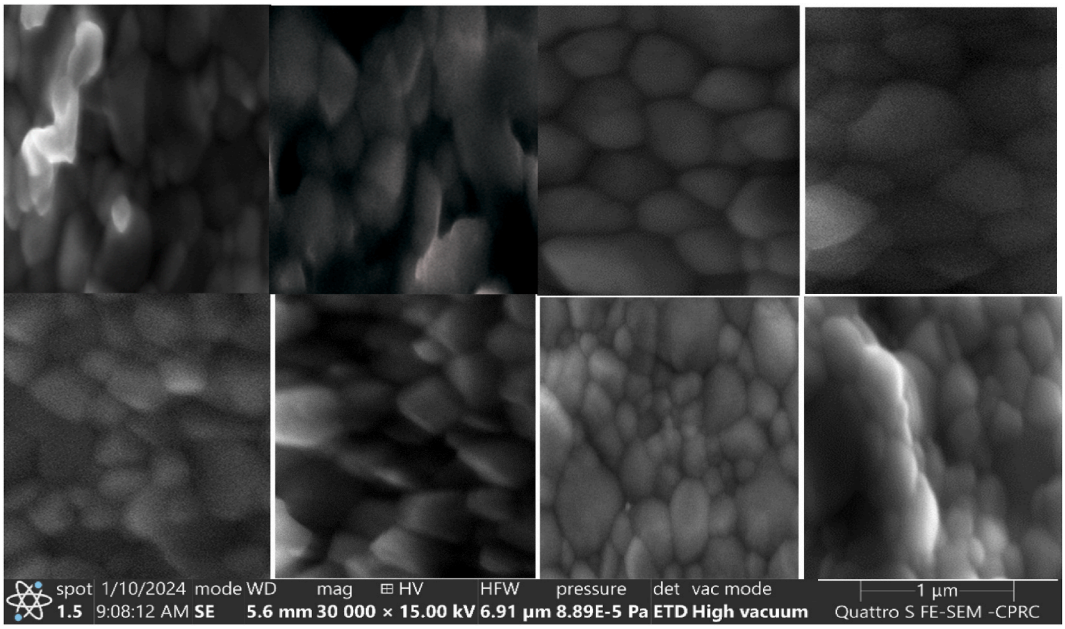

Fig. 3. SEM images (X30000) revealing the grain size distribution of conventionally sintered (AI, BI, CI, DI) and rapidly sintered (AII, BII, CII and DII) samples. The bar indicates 1 µm.

Table 4
Mean standard deviation of the flexural strength, the grain size and translucency of the tested zirconia.

Conventional -sintering				Speed-sintering			
Sub-Group	Flexural strength Mean ± SD	Grain Size (µm)	Translucency Mean ± SD	Sub-Group	Flexural strength Mean ± SD	Grain Size (µm)	Translucency Mean ± SD
AI	830 ± 51	0.46	0.76 ± 0.019	AII	846 ± 43	0.51	0.79 ± 0.024
BI	769 ± 55	0.53	1.61 ± 0.026	BII	787 ± 57	0.57	1.66 ± 0.064
CI	826 ± 46	0.43	0.60 ± 0.14	CII	845 ± 50	0.47	0.63 ± 0.104
DI	840 ± 62	0.40	0.55 ± 0.040	DII	857 ± 60	0.43	0.58 ± 0.087

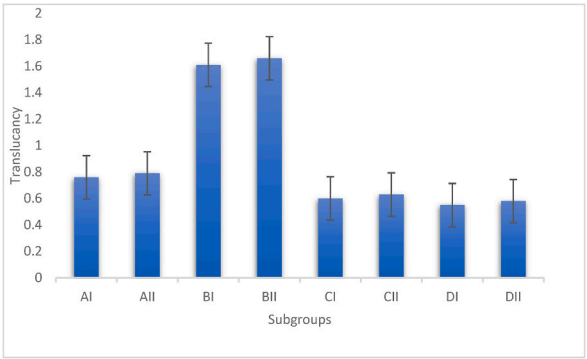

Fig. 4. Comparison of the translucency of speed-sintered and conventional sintered materials (AI, BI.CI. DI) were conventionally sintering, and AII, BII, CII, and DII were speed-sintering.

zirconia materials were not affected by speed sintering in a speeding fire furnace (Dentsply Sirona, York, USA). so the null hypothesis was accepted.

5.1. XRD analysis

X-ray diffraction and Rietveld Refinement were used to identify the phases presenting in each zirconia specimen to estimate the

amount of each phase and the percentage of Y2O3.

According to the result of current study the crystalline analysis indicated showed that all groups display similar XRD pattern; no monoclinic phase peaks have been observed in X-ray diffraction (XRD) patterns. This mean that the samples showed a composition that includes both cubic and tetragonal structures, without undergoing any phase transition. The amount of the cubic phase,Y2O3 contents in the residual tetragonal ZrO2 phase and tetragonality (c/a $\sqrt{2}$) would not effected by speed sintering program.

According to the present investigation, the speed sintering program did not have an imprint effect on the observed phase transition on the surface of the zirconia samples. This was in agreement with the findings of Stevan et al. [24] and Ebeid et al. [25]. While Inokoshi et al. [9] showed that an increase in the holding time and temperature of the sintering program can led to an increase in the cubic phase in the surface structure, which may be ascribed to the use of higher sintering temperatures.

5.2. SEM analysis

Scanning electron microscopy (SEM) was used for microstructure analysis and grain size measurement. SEM micrographs showed the absence of pores, voids or any other defects in the structure of the material, irrespective of the sintering method employed. This observation holds particular significance in the investigation of the speed sintering process.

Zirconia has a critical grain size of around 1 μm. As the grain size of zirconia rises, particularly beyond the critical grain size, its stability decreases and it becomes more prone to spontaneous change from the t-phase to the m-phase. This leads to a drop in the strength of zirconia [26].

The measured zirconia grain size in this study was less than 1.0 μm in all sup groups, suggesting that it was below the crucial size for spontaneous transformation.

The results of this research showed that BII had highest grain size mean (0.57 μm) while the DI had the lowest grain size mean (0.40 μm) , There was a slight increase in the mean of the grain size in the all groups after speed sintering and this indicate that zirconia had a tendency to increase their grain size as the temperature increase.

This result was in agreement with that of Ersoy1 et al. [14], who reported that the variation in grain size due to various sintering processes is typically small and hard to detect. However, Marcela F et al. [27] reported that the grain size increased as the sintering temperature increased, which could be related to the high sintering temperature used.

5.3. Flexural strength test

Zirconia is often regarded as the most durable ceramic substance utilized in the field of dentistry owing to its exceptional mechanical properties This study demonstrated that the flexural strength was greater in the speed-sintering groups than in the conventional groups, although the statistical analysis indicated that there was no significant difference between the conventional and speed sintering. This could be attributed to the formation of mature crystal structures, a reduction in boundary defects, with slight increase in grain size. This was in line with the findings of Ebeid et al. [25] and Hjerppe et al. [16] whose reported that shorter sintering time didn't affect the flexural strength.

The results of this study were not in agreement with those of Stawarczyk et al. [28], who reported that the flexural strength of zirconia decreased with increasing sintering temperature. They found that the maximum flexural strength may be obtained within the temperature range of 1400 °C–1550 °C, whereas the lowest flexural strength was noted at lower temperature.

5.4. Translucency analysis

The translucency of zirconia can be affected by microstructure, its composition and light scatter are significantly affected by grain size. Generally, the amount of visible light that can flow through zirconia materials made of small particles increases as their grain size decreases. This is because, despite the increased scattering caused by an increase in the number of grains, there is less reflection and light absorption. On the other hand, light is reflected more by larger grains, making them opaque [29]. Therefore, zirconia materials with large grains size show less dispersion and less opacity because there are fewer particles per unit volume [30].

Our study showed no significant difference in the translucency but this slight increase in the means of translucency may be contributed to slight increase in grain size; as the grain size increases, the intercept areas at the grain boundaries decrease, and the light scatter decreases and light reflection increase result increase translucency. This in agreement with the findings of CardosoKV et al. [31] and Liu et al. [14], who reported that alterations in sintering parameters did not affect the translucency of zirconia. Different results were obtained by Stawarczyk et al. [28] as the zirconia translucency increased with increasing sintering temperature.

The limitation of current study was that the sintering parameters were determined following manufacturer's recommendation regarding sintering temperature and holding time intervals. Therefore, Investigating the impact of sintering at different temperatures and holding durations on zirconia properties requires additional research. Also, it's important to study how heating rate and sintering environment affect zirconia properties.

6. Conclusion

Speed sintering program did not have a significant impact on the microstructure, flexural strength and translucency of the monolithic zirconia which might be due to the minor increase in sintering temperature used. So that speed -sintering could be a viable alternative to conventional sintering procedures.

Source of funding

This research project received no funding.

Ethics approval statement

The work did not involve the use of humans or animals; hence, ethical approval was not needed.

Data availability statement

Data will be made available on request.

CRediT authorship contribution statement

Mayada Hadi Abed: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Alaa Jawad Kadhim:** Writing – review & editing, Visualization, Validation, Supervision, Methodology, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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