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EFFECT OF DIFFERENT SINTERING TECHNIQUES ON THE BIAXIAL FLEXURAL STRENGTH AND CRYSTAL STRUCTURE OF MONOLITHIC ZIRCONIA (IN VITRO STUDY)

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ABSTRACT

INTRODUCTION: For monolithic zirconia restorations to be a viable chair-side treatment option, recent studies are aiming to reduce the sintering time required without altering its properties.

OBJECTIVES: This study compared the influence of high-speed sintering, speed sintering and conventional sintering on monolithic zirconia regarding bi-axial flexural strength, phase transformation and dimensional changes after sintering.

METHODS: Thirty six monolithic zirconia disc specimens were prepared with the dimensions 12mm x 1.5mm and categorized into 3 groups (n=12) based on the sintering technique: High-speed sintering (HS: 1580 °C, total time is approximately 10 minutes) Speed sintering (SS: 1515 °C, total time is approximately 90 minutes), and Conventional sintering (CS, 1500 °C, total time is approximately 10 h). The ball on ring design was used to determine biaxial flexural strength (BFS). Dimensional changes after sintering was evaluated by digital micrometer (with an accuracy of 0.001 mm). The specimen's crystallography was investigated by x-ray diffraction technique (XRD).

RESULTS: All groups exhibited a uniform sintering shrinkage of about 20% in all dimensions. Crystallographic analysis revealed only tetragonal and cubic characteristic peaks. BFS test results showed statistically insignificant difference between conventional (1105.5 ± 55.85MPa), speed (1078.3 ± 56.22 MPa), and high speed (1050.6±53.42 MPa) sintering cycles ($p<0.05$).

CONCLUSION: Speed and high speed cycles can be recommended for sintering of monolithic zirconia in order to reduce fixed prosthetic restoration fabrication times as the changes they induced were within the clinically acceptable ranges.

KEYWORDS: Monolithic zirconia, speed sintering, biaxial flexural strength.

RUNNING TITLE: Sintering's effect on BFS and monolithic zirconia's crystallography.

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INTRODUCTION

Cosmetic demands in dentistry influenced the growth of modern dental zirconia in the era of restorative dentistry. Tetragonal zirconia polycrystalline (Y-TZP) was introduced in dentistry due to its great mechanical properties, improved esthetics, good biocompatibility, superior structural and chemical stability, high flexural strength (700–1200 MPa) and fracture toughness (7–10 MPa m1/2) (1,2).

Fully contoured monolithic zirconia restorations are developed to overcome the delamination issues of the veneering layers, with the benefits of less budget and processing time, as well as acceptable properties within the minimally - invasive preparation and simplicity of application (3-5).

Conventional sintering of zirconia usually takes 8-10 hours. The sintering furnace's heat is transferred to the surface of the material and then through thermal conduction to the core, resulting in mature sintered zirconia (6). Despite the fact that chair side technology (CAD/CAM) have considerably shortened procedure times and allowed for the delivery of the majority of prosthesis in a single visit; however, the sintering process still takes long time, hindering the production of restorations at the same visit.

In order to meet the desire for chairside restorations that are both time and cost effective, rapid sintering furnaces with various sintering parameters have recently been featured in the dental field. However,

Alteration in the zirconia sintering process can have a direct impact on its properties and strength (7-9).
Flexural strength is usually regarded as a valid and trustworthy approach for determining the longevity of ceramics. Superior strength means that restorations are less likely to fracture (10). Changing the sintering conditions of zirconia can have a direct impact on its characteristics (11).
The magnitude of this influence has gained the attention of dental researchers, particularly when manufacturers began using short sintering cycles. Several researchers have investigated the influence of sintering duration and temperature changes on biaxial flexural strength and phase transformation of zirconia. Yet, the consequences of these modifications on the features of monolithic zirconia is still unknown (12-15).
Therefore, this in vitro study was conducted to investigate the properties of monolithic zirconia blocks sintered by speed and high-speed protocols as compared with those sintered using the conventional technique.
The null hypothesis is that there is no difference between the study groups after the different sintering techniques regarding the biaxial flexural strength, phase transformation, and dimensional change.

MATERIALS AND METHODS

2.1 Study Design

This study was parallel, controlled experimental study, to determine the required sample size in each group, a power analysis was performed using G*Power statistical software (Version 3.1.9.2; Dusseldorf, Germany). With an effect size of 0.4, power of 90% and significance level of 95% (accepted alpha error of 0.05) the calculations revealed that 12 specimens per group would be needed. Thus, a total of 36 discs of 3Y-PSZ monolithic zirconia (12mm diameter×1.5mm thickness) were CAD/CAM fabricated and randomly classified into three main groups (n=12 each) based on the sintering parameter.

2.2 Specimens' preparation

Design of discs (12mm diameter and 1.5 mm thickness) was carried out by the use of dental CAD software (ExocadGmbH; Fraunhofer IGD, Darmstadt, Germany). Multilayered monolithic zirconia block (Katana Zirconia ML, Kuraray Noritake Dental, Inc.,Tokyo, Japan) (Table: 1) was milled using 5-axismilling machine (DWX-50, Roland DG Corp.,Shinmiyakoda,Shizuokaken, Japan) to obtain 36 disc specimens (Figure 1). All specimens were divided at random into three groups (n=12) based on the following manufacturer's specified sintering conditions:

Group I: Conventional sintering (CS): The samples were placed in a high-temperature sintering furnace for Zirconia (KaVo EverestTherm,Germany) at 1500 °C, starts at room temperature. Total time is approximately 10 hours.

Group II: Speed sintering (SS): specimens were placed in(inFire® HTC, Sirona Dental Systems GmbH, Bensheim,Germany) at 1515 °C, starts at room temperature. Total time is approximately 90 minutes.

Group III: High-speed sintering (HS): specimens were sintered in(CEREC Speed Fire, Sirona Dental Systems GmbH, Bensheim, Germany) begins at 1580 °C and has a dwell time of 10 minutes. Specimens were removed from the furnace immediately after sintering. Total time is 10 minutes.

Pre and Post-sintering measurements: Specimens of each group (n=12) were tested to determine their linear shrinkage. Pre and post sintering dimensions were measured by electronic digital micrometer (within the accuracy of 0.001mm)with respect to their diameter and thickness in millimeters(Figure 2) to estimate linear sintering shrinkage ΔL (%) from the equation (16):

$$\Delta L = \frac{(L_0 - L)}{L_0} \text{X } 100$$

Where:
ΔL: Linear sintering shrinkage (%),
L_0: The specimen's diameter prior sintering, and
L:The specimen's diameter post sintering.

X-ray diffraction analysis (XRD)

Three Specimens were selected randomly from each group for detection of microstructural crystalline phase content present by X-ray diffractometer (X- Ray 7000 Shimadzu-Japan), operating with Cu Kα radiation (λ=0.154060nm) generated at voltage and current of 30kv and 30mA respectively. Diffraction profiles were recorded at 2°step size at min-1for 2θ degree of 20°- 80°.
Using the **Schererr** relationship (17), the crystalline size (D) in nm was determined from the reflection of t-ZrO2 peaks at 112 peak.

$$D = \frac{k\boldsymbol{\lambda}}{\beta \cos\theta}$$

Where **k** denotes the crystallite structure constant (0.9), **λ** is the wavelength of radiation (A˚), **β** is the breadth (radians), and **θ** is referred to Bragg angle.

Table 1: Showing material used in the study, its manufacturer, LOT and chemical components

Table 1: Materials used in the study, its manufacturer, LOT and chemical components.

Monolithic Zirconia	Manufacturer	Lot.	Chemical composition in weight percentage
ML KATANA Zirconia blank.	Kuraray Noritake Dental Inc,Tokyo, Japan	DIGTH	$ZrO_2 + HfO_2 + Y_2O_3 > 99$; $2.5 < Y_2O_3 \leq 3$; $HfO_2 \leq 5$; Al_2O_3 + other oxides ≤ 1

Bi-axial flexural strength test

Biaxial flexural testing was chosen since it is considered as a dependable technique and method of preference (ISO 6872) (18). Using a grinding/polishing turbine (Struers TegraPol-25, Denmark), one side of each specimen was wet ground with grit 600, 800, and 1200 for 15 seconds before being cleansed in a sonicator bath for 10 minutes (Codysonultrrasonic cleaning 4820, Shandong, China). For biaxial flexural strength test, ball on ring design was selected. The Universal Testing Machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) was used. Twelve specimens from each group were positioned on a 10-mm size knife-edge and properly loaded by a 5-mm radius sphere indenter at a cross - head speed of 1-mm/min up till the fracture occurred (Figure 3).While testing, all samples were positioned with the grounded side oriented towards the direction of the applied load. A small sheet of rubber was inserted under the disc to ensure even distribution of the load (19). The bi-axial flexural strength was calculated using the formula below (20):

$$\sigma \max = P/h^2 \{(1+v)\ [0.485 \times \ln (a/h) + 0.53] + 0.48\}$$

σ max denotes the highest tensile stress, **P** represents the recorded load of fracture, The knife-edge support's radios is given by **a**, **v** is the Poisson's ratio for the material (a value of 0.3 was substituted for zirconia) (21), **h** denotes the thickness of the specimen determined with a digital calliper and **ln** is the natural logarithm.

Statistical analysis

All tests' outcomes were uploaded into the computer and evaluated with the IBM SPSS software program version 20.0. IBM Corporation, Armonk, New York. The Shapiro-Wilk test was done to ensure that the distribution was normal. Range (minimum and maximum), mean, standard deviation, median, and interquartile range were used to illustrate quantitative data (IQR). The significance of the acquired results was determined at the 5% level.

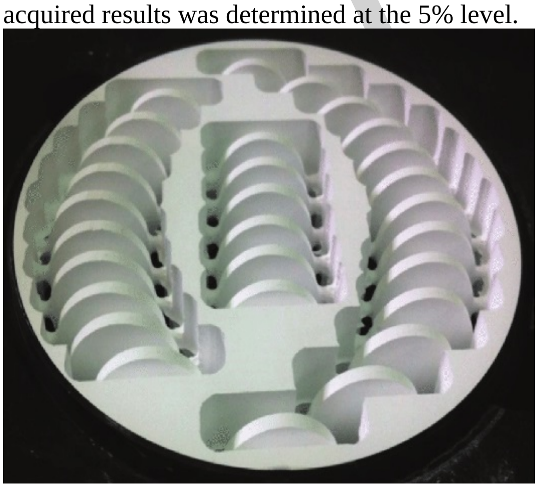

Figure 1: Milled zirconia disc specimens.

Figure 2: Digital caliper measuring (a&b) diameter and (c&d) thickness of specimens before and after sintering respectively.

Figure 3: Biaxial flexural strength test using universal testing machine.

RESULTS

Linear sintering shrinkage (ΔL)

Pre and post-sintering measurements in respect of thickness and diameter in mm of each sample were determined to calculate their Linear sintering shrinkage. In this study, all samples exhibited a uniform linear sintering shrinkage (ΔL) of ≈ 20% in all dimensions. The mean of linear shrinkage of the three groups (CS, SS and HS) was compared using one-way ANOVA. The results showed no statistically significant difference at ($p<0.05$) level between three groups (Table 2)

X-ray diffraction analysis (XRD)

Representative XRD patterns of tested specimens are shown in (Figure 4), where no monoclinic peaks were observed. Moving between diffraction angle positions at about 30°, 34°, 50° and 60° 2theta, **CS group** showed 1 cubic phase c-ZrO_2 (111)peak, and 3 tetragonalt-ZrO_2 (002,112 and 211) peaks respectively. While **SS group** demonstrated simultaneous peak combination of 2c-ZrO_2 (111 and 200), and the 2 t-ZrO_2 (112 and 211) peaks respectively. In contrast, **HS group** showed 1 t-ZrO_2 peak (112) in between 3 distinct c-ZrO_2 peaks (111,002 and 113) respectively.

The crystallite size (D) of pre-sintered and post sintered samples in (nm) are listed in (Table 2). Statistical analysis of crystallite size of the three groups (CS, SS and HS) showed **insignificant difference** at ($p<0.05$) level between three groups, (Table 2)

Table 2: Statistical analysis of mean and SDs of applied tests' values of the three studied groups

		Conventional Sintering (n = 12)	Speed Sintering (n = 12)	High Speed Sintering (n = 12)	Test P value)
Linear shrink age (%)	Min. – Max.	20.0 – 20.13	20.03 – 20.12	20.04–20.18	0.084
	Mean ± SD.	20.07± 0.05	20.09 ± 0.03	20.11± 0.04	
	Median (IQR)	20.10 (20.0 – 20.1)	20.09 (20.1 – 20.1)	20.11 (20.1 – 20.2)	
Crystallite size (D) nm	Min. – Max.	56.55 – 59.42	56.92 – 60.31	59.85 – 61.67	0.194
	Mean ± SD.	58.18 ± 1.47	59.03 ± 1.84	60.64 ± 0.93	
Biaxial flexural strength (MPa)	Min. – Max.	999.8 – 1221.4	953.8 – 1145.8	937.7– 1140.9	0.066
	Mean ± SD.	1105.5 ± 55.85	1078.3 ± 56.22	1050.6±53.42	
	Median (IQR)	1111.0 (1072.5 – 1135.5)	1065.5 (1052.5 – 1129.8)	1064.5 (1036.2 – 1077.2)	

Biaxial Flexural Strength Test (BFS)

Biaxial Flexural Strength test of monolithic zirconia discs sintered in 3 distinct sintering conditions are shown in (Figure 5) demonstrating that the BFS measurements of conventionally sintered discs varied from 999.8 MPa to 1221.4 MPa, while speed sintered group recorded 953.8 MPa to 1145.8 MPa, and a range values of 937.7MPa to 1140.9MPa for high speed sintered samples. However, the statistical test revealed insignificant difference (P=0.066) in flexural strength values among the three groups (Table2).

DISCUSSION

Within the limitation of this study, the null hypothesis was accepted since statistical analysis (Table 2) revealed statistically insignificant difference among conventional, speed and high speed sintered groups (P= 0.066). It was found that all 3Y-PSZ monolithic zirconia discs exhibited a uniform firing shrinkage of approximately 20 % in all dimensions in the three groups. This result is in agreement with Qin (22) who mentioned that because around 20% shrinkage will take place during the sintering, zirconia samples should be cut oversize. Oh (16) observed that the linear sintering shrinkage rate is proportional to the density of the pre-sintered blank as well as the stability of the sintering process. The shrinkage reduces as the density of the pre-sintered blank increases. It is crucial that the block exhibits minimal sintering shrinkage, since this will produce in superior coping precision and fit.

Characteristic XRD patterns for each group presented in (Figures 4) showed similar peak positions of the three groups. The results of the present study showed the presence of tetragonal and cubic phases. XRD patterns revealed that the samples had nearly identical crystalline structure. As a result, it is obvious that there was a negative influence of altering the sintering programs on crystalline composition. According to Stawarczyk et al. (14), with rising sintering heat rates, zirconia crystal size increases. However, a significantly negative association was observed between sintering heat rate and flexural strength, and recommended that the zirconia sintering temperature is better to be maintained around 1550 ºC. The current study findings contradict their conclusions, because the grain size values slightly decreased with high speed 1580ºC sintering.

Also, in line with the findings of recent investigations, larger grain sizes were qualitatively observed for the longer classic speed cycles that allow coalescence and growth in between the grains than for the shorter speed and super speed sintering cycles (4, 23).

This variability can be attributed to various factors, including the various types of zirconia used in studies, as well as limited thermal range and different durations. Measurements of BFS in MPa revealed that highest value (1221.375± 55.85 MPa) was observed with **CS group**, whereas the least value (932.646 ±54.19MPa) was recorded with **HS group**. For **CS group**; A value range of 999.751MPa to 1221.375 MPa was recorded, with an average value of 1105.45MPa, with a SD of 55.85 For **SS group**; A value range of 953.767 MPa to 1145.833 MPa was calculated, having an average value of 1078.337 MPa and a standard deviation of 56.218. For **HS group**; values ranging from 933.646 MPa to 1140.863 MPa, was reported, with an average value of 1027.721 MPa, and standard deviation of 54.19.

These results were equivalent to some BFS reported for monolithic zirconia in the literature. Schatz et al. (24) investigated the BFS of zirconia specimens (ZENOSTAR®ZR translucent; Wieland Dental GmbH, Pforzheim, Germany), and their records ranged from (1139 to 1202 MPa). Other brands of monolithic zirconia BFS values with the same test settings gave similar results to the current study (Ceramill Zolid, BFS=1090 MPa to 1152MPa; DD Bio zx2, BFS=1346 MPa to 1472MPa). This variation in flexural strength scores could be caused by not only using various commercial brands of zirconia, but also by altering the sintering cycles' heating and cooling rates, holding temperature and holding time.

Concerning our findings, modification of sintering temperature and duration of sintering time had no effect on the study's findings, as Li et al (25) who stated that dental zirconia showed similar bending strength, hardness and fracture toughness when sintered for 20 min as with conventional sintering time. In addition Jansen et al (26) also asserted that the fast-sintering protocols, at different maximum temperatures, the biaxial flexural strength of3Y-TZP or 4Y-TZP were unaffected. Furthermore, Cokic et al (27) studied the impact of short sintering cycles on two types of zirconia, 5Y-PSZ and 3Y-TZP that were sintered at 1560ºC in a 30 min cycle and 1578ºC in a 15 min cycle respectively, and reported that all samples exhibited comparable to the conventional sintered control group, same density, composition, hardness, fracture toughness, and mean biaxial flexural strength, as well as hydrothermal ageing resistance.

In contrast to that, Sallam and Eldwakhly (28) tends to refuse these findings by stating that the biaxial flexural strength reported higher values for both the classic and the speed sintering protocols in comparison to the super-speed ultra-short sintering protocol, which resulted in the least mean biaxial flexural strength.

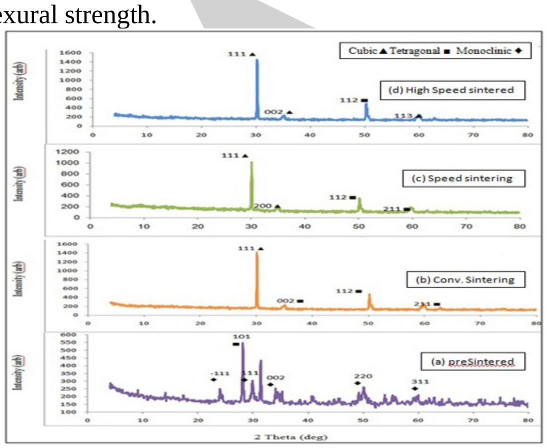

Figure 4: X-ray diffraction pattern of (a) pre-sintered, (b) conventionally sintered, (c) speed sintered, and (d) high speed sintered zirconia discs.

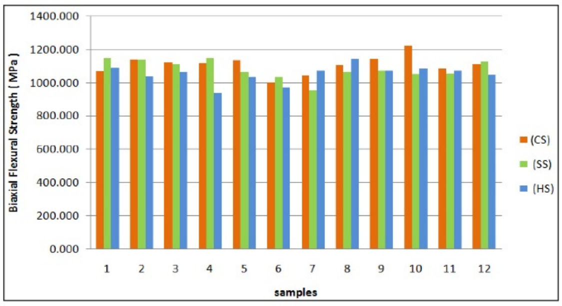

Figure 5: Biaxial flexural strength test results of conventional (CS), speed (SS), and high speed (HS) sintered zirconia discs.

CONCLUSION

- Alternation in sintering protocol within certain ranges, did not significantly affect specimens' dimensions, which in turn will not affect the restoration fit.
- Irrespective of sintering protocol, all groups have nearly the same bi-axial strength values, which satisfy the optimum requirements for clinical use.
- Speed and high-speed cycles can be recommend for sintering of monolithic zirconia in order to reduce fixed prosthetic restoration fabrication times as the changes they induced were within the clinically acceptable ranges.

CONFLICT OF INTEREST

The authors deny any conflict of interests related to the current study.

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