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TRANSLUCENCY AND BIAXIAL FLEXURAL STRENGTH OF MONOLITHIC ZIRCONIA AS AFFECTED BY SINTERING SPEEDS

Hanaa I. Sallam* *and* Elzahraa Eldwakhly*

ABSTRACT

Statement of the problem: Approaches towards reducing fixed prosthetic restoration fabrication times include; (CAD/CAM) chair side systems in addition to rapid sintering cycles, which can be carried out in minutes rather than hours in order to deliver zirconia-based restorations in one visit .However, limited information is available regarding the effect of rapid sintering on properties of monolithic translucent zirconia.

Aim of the study: The aim of this study was to evaluate the effect of changing the sintering speed (classic, speed and super speed cycles) on translucency and biaxial flexural strength of monolithic translucent zirconia before and after thermodynamic aging.

Materials & Methods: 36 monolithic translucent zirconia discs (10mm diameter×0.8mm thickness) were CAD/CAM fabricated from translucent zirconia blocks(In Coris TZI) and randomly divided into three main groups (n=12 each) according to the sintering speed; **group I**: samples sintered by classic sintering cycle (sintering at 1510°C for 120 min holding time & 8 hours total cycle time), **group II**: speed sintering (sintering at 1540°C for 25 min holding time & 2 hours total cycle time) and **group III**: super speed sintering (sintering at 1580°C for 10 min holding time representing the total firing cycle). In each group, half of the samples were evaluated in the non-aged condition (**subgroup1**, n=6) while the other half were evaluated after thermodynamic aging (**subgroup2**, n=6) in a chewing simulator. Translucency parameter (TP) and contrast ratio (CR) were measured using a digital reflective spectrophotometer. Biaxial flexural strength (MPa) was tested using piston on three ball technique in a universal testing machine. Representative sample from each subgroup was analyzed by Scanning electron microscope. Statistical analysis was performed using Two-way ANOVA and Tukey's post-hoc tests ($P \leq 0.05$).

Results: Two-way ANOVA revealed that sintering speed and thermodynamic aging had a statistically significant effect on mean (TP), (CR).Either before or after aging, classic cycle showed the statistically significantly highest mean (TP) & lowest (CR) values. On the other hand, biaxial flexural strength was statistically significantly affected by the sintering speed only and not by thermodynamic aging; the super speed cycle registered the lowest mean biaxial flexural strength before and after thermodynamic aging.

* Associate Professor of Fixed Prosthodontic , Faculty of Oral and Dental Medicine, Cairo University

Conclusions: Translucency of monolithic zirconia was reduced by rapid sintering and thermodynamic aging, however, biaxial flexure strength decreased only by rapid sintering. Although reducing the translucency and biaxial flexure strength, speed and super 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.

KEY WORDS: Monolithic zirconia, Translucent zirconia, Translucency, Biaxial flexural strength, Sintering speed, Thermodynamic aging.

INTRODUCTION

Increase of esthetic demands has led to the development of advanced dental ceramics with the rising application of partially stabilized zirconia in restorative dentistry.[1] Superior mechanical properties as well as outstanding biocompatibility attributed to Zirconia-based restorations being highly attractive for clinicians. [2] However, low translucency and hydrothermal instability are amongst the challenges being faced when using dental zirconias.[3] Owing to their high opacity resulting from the reduced amount of light transmission and much scattering through the restoration, zirconia cores are usually veneered with porcelain overlay to mimic the appearance of natural adjacent teeth.[4,5] Chipping and delamination of the veneering ceramic have been cited to be the most frequent failures observed during clinical service. [3,6]

Recently, the anatomic or monolithic polycrystalline translucent zirconia has attracted increasing attention due to the distinctive combination of mechanical and optical properties, making it the material of choice for fabricating fixed veneer-less Zirconia restorations.[7,8]

Reduction in the amount of required tooth preparation and restorative material thickness in comparison to veneered zirconia restoration, accounts for the clinical advantage of these full contour zirconia restorations. [6,9] Absences of enamel contact wear after exposure to polished monolithic zirconia in a chewing simulator accounts for one more claimed clinical advantage of monolithic zirconia restorations. [10,11] Whereas some of the potential issues encountered with monolithic zirconia restorations include; limitation of esthetic characterization and the higher susceptibility of aging or Low Thermal Degradation (LTD) of zirconia in the oral cavity. [10]

CAD/CAM technologies enable milling of zirconia into restorations with different geometries. Two types of zirconia milling processes are currently available: soft-milling (partially sintered state) and hard-milling (full sintered). Soft-milled frameworks are subsequently sintered to full density.

Different sintering parameters may show a strong influence on the properties of the zirconia restorations.[12]

Alterations in the sintering parameters such as final temperature, holding time and total sintering duration may affect the grain size, translucency and biaxial flexural strength of translucent zirconia. [12,13] Increasing either the sintering temperatures and/or durations leads to larger tetragonal zirconia grain sizes which are claimed to enhance translucency of zirconia. [13]

A Study examining the effect of sintering temperature on the biaxial flexural strength, contrast ratio and grain size of dental zirconia demonstrated that the highest flexural strength was attained at temperatures between 1.400°C and 1.550°C whereas, a decrease in flexural strength was manifested with temperatures above 1.550°C. Their results also showed that both the contrast ratio and the grain size increased with increasing the sintering temperature.[12] Another study carried out by Ersoy et al. [6] investigated the influence of different sintering temperatures and times on the flexural strength of zirconia, and concluded that high sintering temperature and short sintering time combination increased the flexural strength of zirconia. Contradictory results were reported by researches evaluating the effect of different sintering parameters on translucency and biaxial flexural strength of monolithic zirconia, where it was postulated that longer sintering cycles and higher temperatures resulted in reduction of contrast ratio,[7] while biaxial flexural strength was not affected by changes in the sintering parameters.[7,14]

The adverse intra oral environment with fluctuating temperatures, humidity, moisture and mechanical masticatory stresses host the ideal circumstances for the development and exacerbation of low temperature degradation (LTD) of monolithic zirconia restorations.[15,16] These restorations are more sensitive to (LTD, hydrothermal aging) due to two main factors. Firstly, the direct contact of the veneer-less zirconia with the oral fluids as a result of the absence of the porcelain veneer layer that acts as a barrier against water penetration.[17,18] Secondly, the desired increase in grain size to enhance the translucency of the monolithic zirconia, rendered it less resistant to LTD.[19]

Many researches [20,21]evaluated the effect of aging on translucency and biaxial flexure strength of zirconia and found that they were deteriorated after aging, while others reported that they were not influenced by aging.[22]

Several authors had investigated the influence of changing sintering parameters (time and/or temperature) and aging on the translucency and biaxial flexural strength of translucent zirconia. However, scarce studies had been carried out to study the effects of changes in sintering speeds (combination between changing sintering temperature, time and heating conditions) and thermodynamic aging on the translucency and biaxial flexural strength of monolithic translucent zirconia in terms of affecting its long-term performance. Therefore, the aim of this study was to investigate the effect of changing the sintering speed (classic, speed and super speed cycles) on translucency and biaxial flexural strength of monolithic translucent zirconia with and without thermodynamic aging.

MATERIALS AND METHODS

Study Design

In this in-vitro study, 36 monolithic translucent zirconia discs (10mm diameter×0.8mm thickness) were CAD/CAM fabricated and randomly divided into three main groups (n=12 each) according to the sintering speed; **group I:** samples were sintered by classic sintering cycle (sintering at 1510°C for 120 min holding time with 8 hours approximate total cycle time), **group II:** speed sintering (sintering at 1540°C for 25 min holding time with 2 hours total cycle time) and **group III:** super speed sintering (sintering at 1580°C for 10 min holding time representing the total time of the cycle). In each group, half of the samples were evaluated in the non-aged condition (subgroup1, n=6) while the other half were evaluated after thermodynamic aging (subgroup 2, n=6). Translucency measurements [translucency parameter (TP) and contrast ratio (CR)], biaxial flexural strength testing, roughness measurements and scanning electron microscopic analysis were carried out.

Fabrication of Zirconia Discs

a. Designing of the discs

Design of zirconia discs (10mm diameter ×0.8mm thickness) was performed by the use of an open source 3D computer graphics software (Blender 2.78, Amsterdam, Netherlands). The disc shape was designed in the form of 2D model that is 20% larger than the desired final size (12mm diameter ×0.96mm thickness) to compensate for sintering shrinkage that occurred during the sintering stage, so that the final dimensions of the discs would be 10mm diameter ×0.8mm thickness after sintering.

b. Milling of the discs

The 2D model of the disc was exported to the CAM system (computer aided machining system, Sirona Dental Systems GmbH, Bensheim, Germany). The in Lab 15 CAM software was used for the milling procedure of the translucent zirconia blocks (In Coris TZI F0, Mono L, block size : 20/19, Sirona Dental Systems) which was carried out in the in Lab MC x5 milling machine (Sirona Dental Systems), (Figure1) following the dry milling protocol. The discs were then ultrasonically cleaned for 10 minutes in distilled water and air dried with oil free compressed air and left for 24 hours for complete dryness.

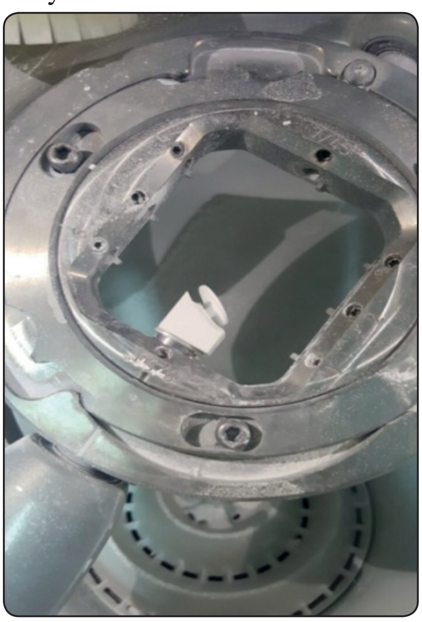

Fig. (1) Milling of the In Coris TZI block in the in Lab MC x 5 milling machine

c. Sintering of the discs

After completion of the milling procedure, the discs were sintered in a high temperature sintering furnace (in Fire HTC speed, Sirona Dental Systems) according to the following sintering speeds as recommended by the manufacturer:

Classic cycle: Samples of group I were placed on the sintering beads in the sintering tray which was loaded into the furnace at room temperature, then the temperature was gradually raised till reaching the sintering temperature (1510^{o} C) which was held for 120 minutes, after which the samples were cooled down to room temperature. The total time of the cycle was approximately 8 hours.

Speed cycle: Samples of group II were sintered by the speed cycle which was similar to the classic one but with (1540^{o} C) sintering temperature that was held for 25 minutes. The total time of the cycle was 2 hours.

Super Speed cycle: This cycle was carried out by the aid of special tools supplied by the manufacturer. The empty furnace was first preheated till (1580^{o} C) then the furnace door opened and by the aid of the super speed fork, the super speed crucible with the samples on its top was loaded to the hot furnace and kept for 10 minutes sintering time. The total time of the cycle was 10minutes. Immediately after that, the furnace door opened and the crucible was lift from the furnace door using the fork and placed on the fire-proof resting tray for two minutes, then the samples were placed on a metal base at room temperature.

After sintering of the discs with the different sintering cycles, the diameter and thickness of each disc were checked with a digital caliper. The final dimensions were 10mm diameter×0.8mm thickness (± 0.02mm).

d. Polishing of the discs

Polishing was carried out by low speed hand piece and an electric motor with a rate of 7000-1200 rpm using zirconia polishing kit (EVE DIACERA Set HP 321, EVE Ernst Vetter GmbH, Germany).Polishing procedure was started by the green medium grit wheel (H8DCmf)followed by the orange fine grit wheel (H8DC) to get the high gloss mirror finish. Polishing was carried out for one surface of the discs and the other surface left as sintered without polishing. Finally, the discs were ultrasonically cleaned for 10 min in distilled water and air dried with oil free compressed air.

Thermodynamic Aging

Half of the samples in each group were subjected to thermodynamic aging which was carried out using the four stations multi-modal ROBOTA chewing simulator integrated with thermo-cyclic protocol operated on servo-motor (Model ACH-09075DC-T, AD-TECH Technology CO., LTD., Germany). Mechanical (dynamic) aging was performed by vertically loading samples with 50 N by a sliding stainless steel sphere-shaped piston for 75000 cycles at 3 Hz to clinically simulate 6 months of chewing condition.[23] The piston vertical movement was 2 mm (with rising speed of 90 mm/s and descending speed of 40 mm/s) and its horizontal movement was 2 mm (with forward speed of 90 mm/s and backward speed of 40 mm/s) at a torque of 2.4 N.m. Simultaneous thermal aging was carried out by thermo cycling in distilled water at temperatures of 5°C and 55°C with one cycle lasting 60s.[24]

Assessment of Surface Roughness (Ra)

A 3D-surface analyzer system was used for contactless quantitative analysis for surface roughness of the tested samples before and after thermodynamic aging.[25] In-Coris zirconia samples were photographed using USB Digital microscope with a built-in camera (Scope Capture Digital Microscope, Guangdong, China) connected with an IBM compatible computer using fixed magnification of 120X. The images were recorded with resolution of1280 X 1024 pixels per image and then cropped to 350 X 400 pixels using Microsoft office picture manager to specify and standardize area of roughness measurement. Cropped images were analyzed using WSxM software (Version 5 develop 4.1, Nanotec, Electronica, SL).[26] Three separate areas were measured on each disc and WSxM software was used to calculate average surface roughness (Ra) expressed in mm.[27]

Translucency Measurements

a. Translucency Parameter Measurements (TP)

Translucencies of all samples were measured before and after thermodynamic aging using a digital reflective spectrophotometer (Model RM200QC, X-Rite, Neu-Isenburg, Germany). The aperture size was set to 4 mm and the samples were aligned with the device. Three measurements were taken for each sample on white (CIE L*= 88.81, a*= -4.98, b*= 6.09) and black (CIE L*= 7.61, a*= 0.45, b*= 2.42) backgrounds and the average of each parameter (L*, a*and b*) was recorded relative to the CIE standard illuminant D65.

These measurements were used to calculate the translucency parameter (TP) according to the following formula:[28,29]

$TP= [(Lb^* - Lw^*)^2 + (ab^* - aw^*)^2 + (bb^* - bw^*)^2]^{1/2}$, where letters “b” and “w” refer to color coordinates over the black and white backgrounds, respectively. ΔL*, Δa* and Δb* were considered as the differences of the L*, a*, b* values of the specimens over the black and white backgrounds. In all calculations, 0 was considered as totally opaque and 100 as totally transparent. The greater the TP value, the higher the translucency of the material.[30]

b. Contrast Ratio Measurements (CR)

The contrast ratio was measured before and after thermodynamic aging using the same Reflective spectrophotometer at daylight under the light source of CIE illuminant D65 brightness. The measurement was made 3 times in flashing mode with an interval of 3 s, in steps of 0.1 s. Mean values were then calculated and contrast ratios were measured from the luminous reflectance (Y) of the specimens with a black (YB) and a white backing (YW) to obtain CR=YB/YW. In all calculations, 0 was considered as totally transparent and 1 as totally opaque.

Biaxial Flexure Strength Testing

Samples were tested for biaxial flexural strength using piston on three ball technique [31] in a universal testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA). Tested samples were supported centrally on three hardened steel balls with a diameter of 3.2 mm, positioned equidistant to each other at 120° angles. [32] Samples were loaded centrally by means of a piston with a flat punch of 1.50 mm diameter at a crosshead speed of 1mm/min and a 5KN load cell in compressive mode of loading. Load was applied on the unpolished surface of the sample as the polished surface was the tension side. [33]The fracture load (N) for each specimen was recorded using computer software (Instron® Bluehill Lite Software) and the biaxial flexural strength was calculated using the following equation: [32, 34-36] according to the ISO 6872 standard (ISO 6872 1995).[31, 37-39]

$$S = -0.2387\ P(X-Y)/d^2$$

where **S:** the biaxial flexure strength (MPa), **P:** the measured load at fracture (N), **d:** is the sample disc thickness at fracture origin (mm), **X** and **Y** were determined as follows;

$$X = (1+\gamma)\ \ln\ (r_2/r_3)^{2} + [(1-\gamma)/2]\ (r_2/r_3)$$

$$Y = (1+\gamma)\ [1+\ln\ (r_2/r_3)^{2}] + (1-\gamma)\ (r_1/r_3)2$$

Where γ: is Poisson's ratio (0.25), **r1:** is the radius of support circle (mm), **r2:** is the radius of loaded area (mm), **r3:** is the radius of sample (mm).

Scanning Electron Microscopic (SEM) Analysis

After sintering and before thermodynamic aging, representative samples of each subgroup were ultrasonically cleaned for 10 min in distilled water and air dried with oil free compressed air. Scanning Electron Microscope (Quanta FEG 250 Environmental Scanning Electron Microscope, Netherlands), was used for analysis of the unpolished surface of the discs (as sintered). Gold sputtering of the samples were done in a Sputter Coater (SMITECH K550 X Sputter coater, England). The sputtered samples were examined with accelerating voltage 20 K.V. The results were recorded photographically at magnifications 200× , 4000× , 25000× & 50000× .Similarly, after thermodynamic aging and biaxial flexural strength testing, representative samples for fractured pieces of the discs of all subgroups were ultrasonically cleaned, dried and prepared for SEM examination of the fractured surfaces.

Statistical Analysis

Two-way ANOVA test was used to study the effect of sintering speed and thermodynamic aging on mean (Ra), (TP), (CR) and biaxial flexural strength. Tukey's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. Pearson's correlation coefficient was used to study the correlation between (TP) and (CR).

The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM (IBM Corporation, NY, USA) SPSS (SPSS, Inc., an IBM Company) Statistics Version 20 for Windows.

RESULTS

The data collected was checked for normal distribution by checking data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests. All presented data showed parametric (normal) distribution.

1. Surface Roughness (Ra) before and after aging

Two- way ANOVA results showed that sintering speed, thermodynamic aging as well as the interaction between the two variables had non-statistically significant effect on mean (Ra). The interaction between the two variables was non-statistically significant indicating that the two variables are independent from each other. Results of the different interactions of variables are presented in (Table1), (Figure 2).

Effect of sintering speed: Either before or after aging, there was no statistically significant difference between classic, speed and super speed cycles in (Ra) values.

Effect of aging: Either with classic, speed as well as super speed cycles, there was a statistically non-significant increase in mean (Ra) after aging than before aging.

2. Translucency Results

a. Translucency parameter (TP) measurements before and after aging

Two-way ANOVA revealed that sintering speed and thermodynamic aging had a statistically significant effect on mean (TP). The interaction between the two variables had non-statistically significant effect on mean (TP) indicating that the two variables are independent from each other. Results of the different interactions of variables are presented in (Table2), (Figure3).

Effect of sintering speed: Either before or after aging, classic cycle showed the statistically significantly highest mean (TP). There was no statistically significant difference between speed and super speed cycles; both showed the statistically significantly lowest mean (TP) values.

Effect of aging: Either with classic, speed as well as super speed cycles, there was a statistically significant decrease in mean (TP) after aging.

b. Contrast Ratio (CR) measurements before and after aging

Two-way ANOVA results showed that sintering speed and thermodynamic aging had a statistically significant effect on mean (CR). The interaction between these two variables had non-statistically significant effect on mean (CR) indicating that the two variables are independent from each other. Results of the different interactions of variables are presented in (Table3), (Figure 4).

TABLE (1) Descriptive statistics and results of two-way ANOVA test for (Ra) values of the different interactions

Aging	Classic cycle		Speed cycle		Super speed cycle		*P*-value (Effect of sintering speed)
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**	
Before aging	0.2557	0.0011	0.2558	0.0012	0.2559	0.0008	0.981
After aging	0.2582	0.0027	0.2580	0.0028	0.2588	0.0021	0.769
***P*-value (Effect of aging)**	0.157		0.116		0.059		

: Significant at $P \leq 0.05$

TABLE (2) Descriptive statistics and results of two-way ANOVA test for (TP) values of the different interactions

Aging	Classic cycle		Speed cycle		Super speed cycle		*P*-value (Effect of sintering speed)
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**	
Before aging	13.41 ^{A}	0.50	11.78 ^{B}	0.46	11.41 ^{B}	0.39	<0.001*
After aging	12.65 ^{A}	0.47	11.24 ^{B}	0.32	11.08 ^{B}	0.28	<0.001*
***P*-value (Effect of aging)**	0.003*		0.029*		0.045*		

: Significant at $P \leq 0.05$, Different superscripts in the same row are statistically significantly different

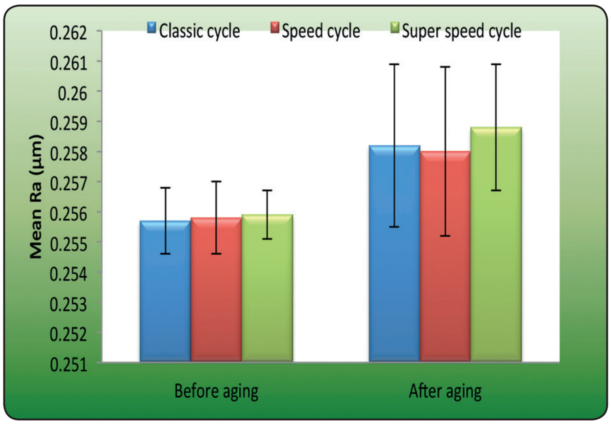

Fig. (2): Mean and standard deviation values of (Ra) in the different groups.

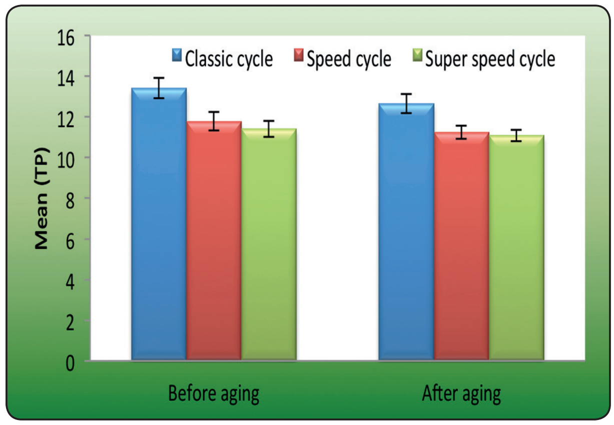

Fig. (3): Mean and standard deviation values of (TP) in the different groups

TABLE (3) Descriptive statistics and results of two-way ANOVA test for (CR) values of the different interactions

Aging	Classic cycle		Speed cycle		Super speed cycle		*P*-value (Effect of sintering speed)
	Mean	SD	Mean	SD	Mean	SD	
Before aging	0.651 [B]	0.022	0.703 [A]	0.007	0.711 [A]	0.009	<0.001*
After aging	0.681 [B]	0.009	0.722 [A]	0.015	0.732 [A]	0.019	<0.001*
***P*-value (Effect of aging)**	0.001*		0.033*		0.019*		

: Significant at $P \leq 0.05$, Different superscripts in the same row are statistically significantly different

Effect of sintering speed: Either before or after aging, there was no statistically significant difference between speed and super speed cycles; both showed the statistically significantly highest mean (CR) values. Classic cycle showed the statistically significantly lowest mean (CR).

Effect of aging: Either with classic, speed as well as super speed cycles, there was a statistically significant increase in mean (CR) after aging**.**

Pearson correlation showed that there was a statistically significant inverse (negative) correlation between (TP) and (CR) with correlation coefficient (r = - 0.874, P-value <0.001).

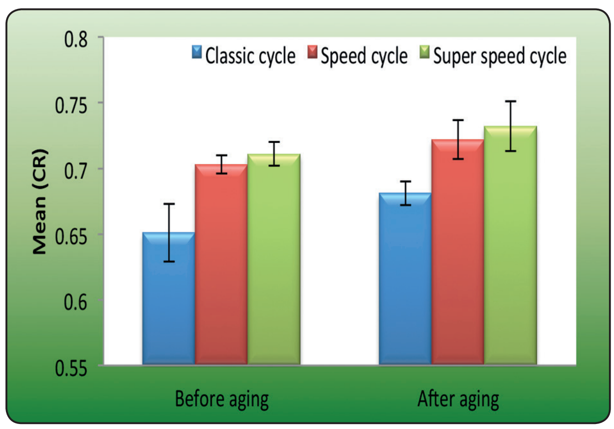

Fig. (4): Mean and standard deviation values of (CR) in the different groups

3. Biaxial flexural strength before and after aging

None of the samples fractured during thermodynamic aging.

The mean biaxial flexural strength of the tested groups ranged from 766 MPa to 1169 MPa. Two-way ANOVA results showed that both the sintering speeds and thermodynamic aging had a statistically significant effect on mean biaxial flexural strength regardless of the other variable (P-value = 0.045). However, the interaction between the two variables had non-statistically significant effect on mean biaxial flexural strength indicating that the two variables are independent from each other. Results of the different interactions of variables are presented in (Table4), (Figure 5).

Effect of sintering speed: Regarding the results for both before or after aging, there was no statistically significant difference between classic and speed cycles; both of which showed the statistically significantly highest mean biaxial flexural strength values. Whereas, the Super speed cycle showed the statistically significantly lowest mean biaxial flexural strength.

Effect of aging: After aging, all tested groups; classic, speed as well as super speed cycles, showed lower mean biaxial flexural strength than before aging. However, the difference between before and after aging mean values was statistically insignificant.

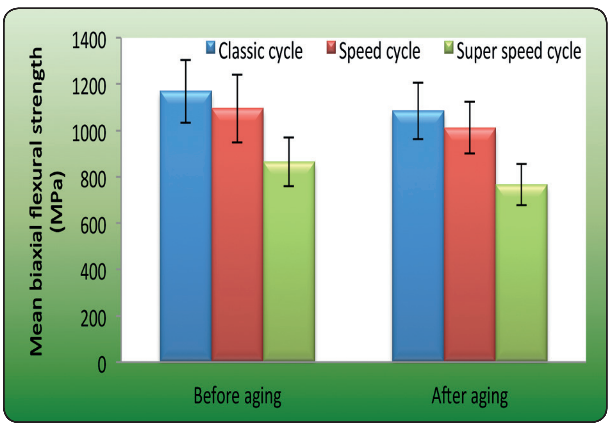

Fig. (5): Mean and standard deviation values of biaxial flexural strength in the different groups

4. Scanning Electron Microscopic (SEM) Results

(SEM) images of representative samples of each sintering speed (without thermodynamic aging) are illustrated in (Figure 6) to show the microstructure of zirconia (grain size and orientation). All samples sintered at the different sintering speeds were completely sintered to the tetragonal phase.[6,12] Qualitative visual analysis of images (6 a, b& c) showed apparent difference in the zirconia grain size as a result of the different sintering speeds. Samples sintered by the classic long cycle showed large well organized compact grins with little porosity and increased density (Figure 6a), While samples sintered by the speed cycle showed compact smaller grains in comparison to those of the classic cycle (Figure 6b). However, samples sintered by the super speed cycle showed less organized bizarre arrangement of different sized grains with increased number of pores and decreased density (Figure 6c).

Due the comparable microstructural pattern of the SEM samples investigated after fracture (with and without thermodynamic aging), a representative sample of the classic sintering speed cycle was selected for presentation in this article to shed light on the microstructural behavior of the samples after fracture. Distinct subsurface transformation zone was observed after aging in the SEM images (Figure 7 c, d &e) that could hypothetically resulted from the tetragonal to the monoclinic transformation[18,21], it was measured at × 200 and found to be 62.05μ which represented 7.5% of the total thickness of the sample, however, the transformation zone was not clearly detected in the fractured sample without aging (Figure7,a &b) .

TABLE (4): Descriptive statistics and results of two-way ANOVA test for biaxial flexural strength values of the different interactions

Aging	Classic cycle		Speed cycle		Super speed cycle		*P*-value (Effect of sintering speed)
	Mean	SD	Mean	SD	Mean	SD	
Before aging	1169.4 ^{A}	136.1	1094.5 ^{A}	147.1	863.8 ^{B}	104.9	<0.001*
After aging	1084.2 ^{A}	121.5	1011.9 ^{A}	110.8	766.4 ^{B}	88.8	<0.001*
***P*-value (Effect of aging)**	0.228		0.242		0.169		

****: Significant at $P \leq 0.05$, Different superscripts in the same row are statistically significantly different***

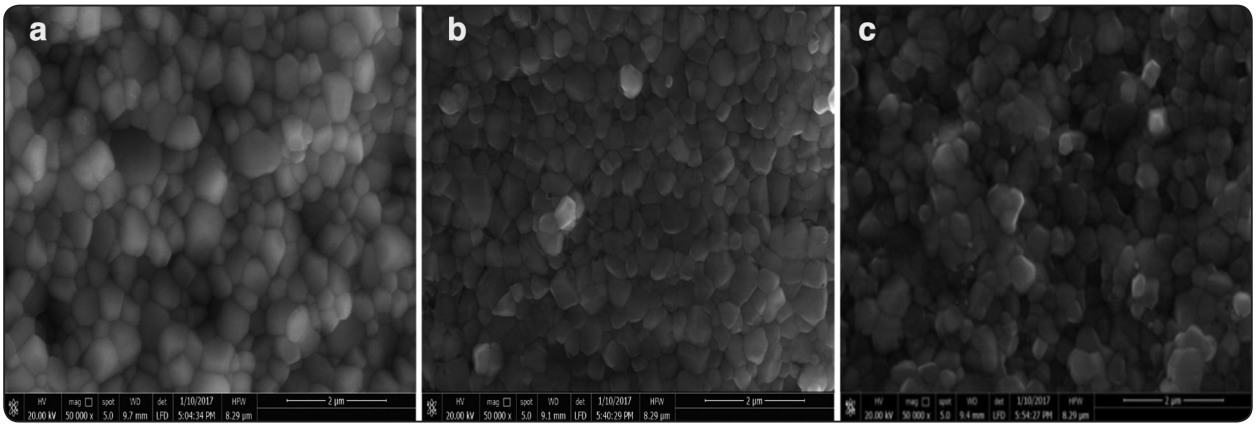

Fig. (6) : SEM images of zirconia samples sintered by A: classic, B: speed and C: super speed cycles without thermodynamic aging and before fracture showing the different grain patterns at 50000× (Top views)

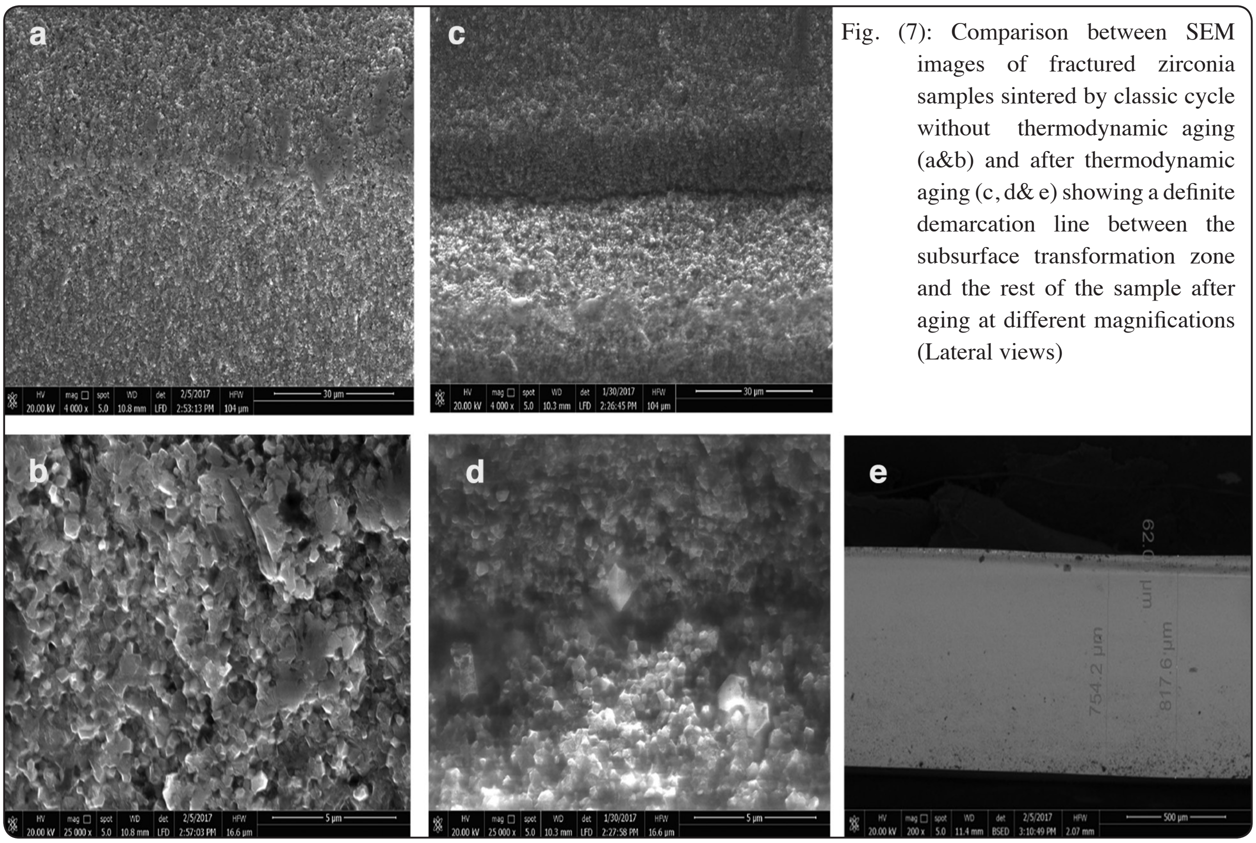

Fig. (7): Comparison between SEM images of fractured zirconia samples sintered by classic cycle without thermodynamic aging (a&b) and after thermodynamic aging (c, d& e) showing a definite demarcation line between the subsurface transformation zone and the rest of the sample after aging at different magnifications (Lateral views)

DISCUSSION

The advent of monolithic zirconia with its proposed enhanced translucency coupled with superior strength properties, endorsed its use as an esthetic restoration that fulfills the desires of both patients and doctors. [28] This study was performed following recommendations of previous studies to determine how changing the sintering parameters affect the clinical serviceability of monolithic zirconia as a fixed dental restoration.[6,12] Clinically, this is of paramount importance due to the recent interest of shortening the sintering cycle's time in order to deliver the fixed restoration to the patient in a single setting after its chair-side CAD/CAM milling .

Translucency is a vital parameter that controls esthetics during material selection in terms of mimicking the appearance of natural teeth. Translucency is the phenomenon of light transmission through a material. [40] It is primarily affected by multiple scattering of light within the ceramic grains due to the different refractive indices and the inhomogeneous nature of the ceramic phases.[41] Grain boundaries, pores, and light scattering from rough surfaces, are factors known to have a negative effect on translucency.[42]

Several methods have been proposed to evaluate translucency of esthetic restorative materials, such as: absolute translucency; measured by direct transmittance of light, and relative translucency; measured either by the contrast ratio (CR) or the translucency parameter (TP). Translucency parameter (TP) is the color difference of a given thickness of a material over white and black backgrounds. (TP) is equal to zero when a material is absolutely opaque. The higher the value of (TP), the more translucent the restoration is. When (TP) equals 100, this means that the material is absolutely transparent.[40,8] The contrast ratio (CR) is a measure of the opacity of the restoration. It is the measured reflectance of a material over black and white backgrounds. The (CR) value of a perfectly transparent material is 0,while the value of a completely opaque material is one.[7,8] In this study, there was an agreement with other studies [43] that there was a correlation between (TP) and (CR) results ($r = -0.874$, P-value <0.001).

Flexural strength is an important indicator of a material's mechanical properties. Brittle materials are much weaker in tension than in compression. The piston on three ball method was used for biaxial flexural strength testing in this study as it was approved by the "American Society of Test and Materials" and by the International Standard Organization to be a standard test method (ASTM F 394-78, 1995) for thin ceramic substrates.[37] Samples were tested in the polished state following the ISO standard 13356:2008(E) for 3Y-TZPceramics.[38,44]

Rough surfaces are considered to have a detrimental effect on translucency and biaxial flexural strength of esthetic restorative materials.[42] Results of this study showed that sintering speeds and thermodynamic aging did not affect the mean roughness (Ra) values of the samples (Table 1). Since there were no significant differences between the subgroups regarding the surface roughness, it can be assumed that the changes in the TR, CR and biaxial flexural strength are mainly due to changes in the microstructure rather than changes in surface morphology. Microstructure analysis by scanning electron microcopy imaging of representative tested samples was carried out according to the hypothesis that changes in the sintering conditions of zirconia affects its grain and micro-structural patterns.[45]

In the present study, regarding the effect of sintering speeds on translucency, acceleration of the sintering cycle resulted in a decrease in translucency and an increase in opacity as evident from the TP and CR results (Tables 2&3). The classic cycle (before and after aging) recorded the highest translucency among the tested subgroups. This could be attributed to the fact that sintering parameters have an effect on the grain structure and the crystalline phases. [46] It has been shown that the increase in the holding time during sintering (as with the classic cycle,120 min. holding time) caused grain growth which increased the grain size and allowed the zirconia grain structure to become more compact, decreasing the porosity and consequently, improving the translucency. [47] SEM image of the classic cycle sample (Figure 6a) supported these findings. These results were in accordance with other studies which reported that increasing the holding time during sintering improves translucency of monolithic zirconia.[7,12,48]

The speed sintering cycle results in smaller grains as could be demarcated from the SEM analysis (Figure 6b). This smaller grain size with abundant grain boundaries may be attributed to the short holding time during sintering (25 min.) which decreased the grain growth when compared to the classic cycle samples.[47] This abundant microstructure with a non-uniform light/grain-boundary interaction resulted in lower translucency and impaired optical characteristics.

The lowest TP and highest CR were recorded with the super speed cycle. This could be referred to the less organized bizarre arrangement of the different sized grains with an increased number of pores as shown in the SEM image (Figure 6c). These SEM findings could be attributed to; firstly, the excessively short holding time of the cycle (10 min.). Secondly to the sudden exposure of the zirconia samples to the high sintering temperature (1580°C) without any gradual heating, causing an added detrimental effect on the zirconia grain orientation and leading to more light diffraction.[49] Our findings could further be clarified by stating that the tetragonal zirconia crystal is birefringent, meaning that the index of refraction is anisotropic in different crystallographic directions. [50,51] In polycrystalline Y-TZPs, birefringence results in the discontinuity of the refractive index at the grain boundaries when the adjacent grains do not have the same crystallographic orientation (as clearly seen in SEM image ,Figure 6c). This causes both reflection and refraction at grain boundaries, leading to diversions in the incident beam and thus reductions in light transmittance. [17] Additionally, the increased number of pores present in samples sintered by the super speed cycle (SEM figure 6 c) leads to the "pore scattering" phenomenon which probably caused more light scattering, less light transmission and reduced translucency.

The increase in the sintering temperature with the decrease in the sintering time in this study did not improve the grain size (as seen in SEM images 6 b &c) or translucency results (Tables 2&3) of zirconia. This could be due to the little range of temperature increase between the three tested cycles (1510,1540 &1580) in addition to the extremely short holding time which did not allow the increased sintering temperature to affect the grain size or translucency.[12] Our findings were against those of others [7,12] who claimed an enhanced translucency as a result of higher sintering temperatures leading to; reduction in the pores between the grains; increase in the final density of zirconia; decrease in the light scattering; and increase in the light transmission.[30] This opposition in results may be attributed to the difference in the methodologies employed in each research, as they tested different sintering temperature ranges at fixed longer holding times , so they changed only one sintering parameter not a combination between sintering time and temperature as in the present study.

Biaxial flexural strength was found to be significantly higher for both the classic and speed longer sintering cycles in comparison to the super speed ultra short sintering cycle before and after thermodynamic aging (Table 4). This was in accordance with previous studies who reported comparable strength results with the change in sintering parameters.[12] This could be attributed to faster sintering cycles causing un-homogenous grain pattern compared to the speed and classic longer sintering cycles that leads to a more densely packed grain pattern with reduced number of pores (Figure 6 a,b&c). [52,53]

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.[7, 14,54] Our results support the proposed linear relationships between grain sizes, density, and biaxial flexural strength. [52]

On the other hand, other investigators showed that changes in the sintering parameters had no effect on the biaxial flexural strength of zirconia.[7, 14] These differences in results may be due to the difference in zirconia type and sintering parameters tested in the different studies.

Artificial accelerated aging or thermodynamic fatigue allows for the simulation of the adverse intra oral clinical conditions to which restorations are exposed, causing alternations in their physical and mechanical properties, and the quantifications of such changes over the course of time. [20,55,56] Thermodynamic aging of monolithic zirconia performed in this study could be considered as a good simulation of clinical situations.[23,24] It has been proposed that hydrothermal aging has a detrimental effect on the mechanical properties of zirconia, causing a degradation of up to 30% in the elasticity and hardness.[57] Accordingly, thermodynamic aging was performed in this study to ascertain the effect of aging on the long-term stability of monolithic zirconia.

Concerning the effect of thermodynamic aging on translucency, exposing the tested samples to aging reduced the translucency of monolithic zirconia at the three tested sintering speeds as evident from the (TP) and (CR) results (Tables 2&3). This may be attributed to the hypothesized transformation from tetragonal to monoclinic grain structure (t–m) at the subsurface layer which was confirmed by the SEM image e (Figure 7 c, d &e). [18,21,58] This initial transformation results in further spontaneous transformations in adjacent grains as it is accompanied by considerable volume expansion stressing the neighboring grain and leading to the formation of microcracks which alter the light reflection and transmission, thus, negatively affecting translucency. The results of this study were in agreement with those of Alghazzawi[59] who reported that the optical properties of zirconia were affected by aging as it increased CR and lowered TP of the tested samples.

The results of our study complemented other studies in that the biaxial flexural strength was reduced for all tested sintering speed groups after exposure to thermodynamic aging, though this decrease was statistically non significant (Table 4). [14,60-64] This may be explained by the fact that the total thickness of the samples was 8 mm (±0.02 mm), and the thickness of the transformed (t–m) layer was 62.05μ (Figure 7e).Thus, it is highly unlikely that thermodynamic aging would affect the biaxial flexural strength values given the remained thickness of the untransformed zone (tetragonal_phase layer). Indeed, the insignificant results of the biaxial flexural strength may lead to the misconception that mechanical properties of Y-TZP materials are not affected by aging, but the most probable hypothesis is that the flexural strength test is not sensitive enough to detect the influence of surface phase transformation on mechanical properties of aged Y-TZP. [63]

On the other hand, our results were inconsistent with those of other researchers who found a statistically significant negative effect for the aging procedure of zirconia on its flexural strength.[21,58,65] These differences in results may be referred to the difference in zirconia types used and the testing methods as they used 4 point bending test for a bar shaped specimens. Additionally, there was a difference in the aging protocols examined.

Low thermal degradation (LTD)as a result of aging of zirconia is characterized by phase transformation manifested in a shift from the tetragonal crystalline form to the monoclinic form. This phase transformation occurs over time when the material is in contact with water which is able to penetrate the crystalline structure.[15] This Intracrystalline water diffusion resulting from LTD leads to; enhancement of microcrack propagation, promotion of release of small zirconia grains pull- outs causing roughening of the surface, and degradation of both mechanical as well as aesthetic properties.[20, 66]

Three different theories have been postulated about the initiating criteria for the hydrothermal aging mechanism in zirconia. The first proposed rational states that ceramic corrosion occurs as a result of the reaction between water (H_2O) and yttrium (Y_2O_3) to form yttrium hydroxide ($Y(OH)_3$), gradually depleting the stabilizer and inducing the conversion of the tetragonal to the monoclinic phase.[67] The second mechanism suggested that the bond between Zr and O, is broken by water causing localized stress growth as a result of –OH ingression inside the crystal structure resulting in lattice fault that acts as nucleating agents for crystalline phase transformation from the tetragonal to the monoclinic forms. The third theory states that oxygen from water breakdown fills oxygen vacancies.[66, 67]

The instrumental (CR) measurements presented in this study need to be verified for their clinical relevance. This was done by calculating the mean difference in CR (ΔCR) between any two subgroups, and then compareing it with the Translucency Perception Threshold (TPT) following Liu et al [68] who stated that the translucency perception threshold for people was 0.07 , above which it can be considered as clinically perceivable and below which it can be considered clinically undetectable to people. In the current study, regarding (ΔCR) induced by the different sintering speeds in the non aged subgroups, it was found that (ΔCR) between classic cycle (0.651) and speed (0.703)as well as super speed(0.711) cycles was (0.052) and (0.06), respectively, while it was (0.008) between the speed and the super speed cycles. Similarly, in the aged subgroups, (ΔCR) between classic cycle (0.681) and speed (0.722) as well as super speed(0.732) cycles was (0.041) and (0.051),respectively, while it was (0.01) between the speed and the super speed cycles. Concerning the (ΔCR) induced by thermodynamic aging, it was found that (ΔCR) between non aged (0.651) and aged (0.681) subgroups in the classic cycle was(0.03).While it was (0.019) between non aged (0.703) and aged (0.722) subgroups in the speed cycle. Finally, the (ΔCR) between non aged (0.711) and aged (0.732) subgroups in the super speed cycle was (0.021). [All ΔCR values were calculated from (Table 3)]

All (ΔCR) values obtained in this study were less than the translucency perception threshold (TPT=0.07) [68] therefore ,would not have a clinically significant impact on translucency of monolithic zirconia as they would not be perceived by lay people.

The results achieved from monolithic zirconia tested in this study at different sintering speeds, with or without thermodynamic aging, met the requirements of ADA specifications that recommends a minimum biaxial flexural strength value of 100MPa for this type of ceramic material.[60] Also, they fulfilled the ISO standard 13356:2008(E)[44], which stated that the ceramic material strength after simulated ageing should not be less than 80% of the initial strength measured before ageing.[44] This was verified in our study as the biaxial flexural strength values of the thermodynamic aged samples recorded the following percentages of the non aged samples: 92.7% for the classic group, 92.4% for speed group, and 88.7% for the super speed group (calculated from Table 4).

Some of the limitation of this study include; first, tests were carried out using flat discs of a standardized thickness which does not resemble the fixed restoration geometry and this may affect the results. Second, only one brand of zirconia material and one specimen thickness were tested. The zirconia samples were tested without being dipped in the coloring liquid as this procedure could affect the translucency and biaxial flexural strength as proved previously in the literature.[69,70] Future investigation should be performed directly on an anatomical restoration for greater clinical relevance.

CONCLUSIONS

Within the limitations of this study, the followings could be concluded:

1. Translucency of monolithic zirconia was reduced by rapid sintering and thermodynamic aging, however, biaxial flexure strength decreased only by rapid sintering.

2. Although reducing the translucency and biaxial flexure strength, speed and super 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.

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