

EFFECT OF SINTERING PROTOCOL ON MARGINAL ADAPTATION AND FRACTURE RESISTANCE OF ZIRCONIUM OXIDE BASED THREE UNIT FIXED PARTIAL DENTURES

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ABSTRACT

Aim of the study Multilayer monolithic zirconia, which includes polychromatic layers that reflect natural tooth color gradients, gives an enhanced properties and advanced fabrication efficiency. Controversy, still present regarding the fracture resistance and marginal adaptation of multilayer monolithic zirconia restorations after application of variable sintering protocols. Thus, the aim of this study was to evaluate and compare the effect of sintering protocol on marginal adaptation and fracture resistance of zirconium oxide based three units FPDs.

Materials and methods A total of 24 three-unit monolithic zirconia FPDs, with one premolar pontic and two retainers, were fabricated. The FPDs were divided into two groups (n=12) according to the material used for construction of FPDs Group I: White peak Copra supreme hyperion monolithic zirconia, Group II: White peak Copra supreme Symphony monolithic zirconia. Then each group was further divided into two equal subgroups according to the sintering protocol. Sub group 1: Regular (Normal) sintering protocol, sub group 2: Speed sintering protocol. The master cast was scanned with a laboratory scanner, the scanned data was used to design monolithic FPDs based on manufacturer instructions. The received CAD data were milled for each group of material. FPDs were sintered according to the manufacturers' instructions. Vertical margin gap distance was measured using a stereomicroscope before cementation as control. The FPDs were cemented using self-adhesive resin cement (Rely X Unicem), and then samples were subjected to thermocycling and cyclic fatigue loading. The vertical marginal gap distance for all samples was subsequently re-measured. Fracture resistance test was done using universal testing machine. The fracture load was recorded in Newton. Data was collected, tabulated and statistically analyzed. Repeated measures ANOVA test was used to study the effect of variables and their interactions on mean marginal gap distance. Two-way ANOVA test was used to study the effect of variables and their interactions on mean fracture resistance. The significance level was set at $P \leq 0.05$. **Results** In all tested groups and subgroups, regular sintering showed statistically significantly lower mean gap distance than speed sintering, while there was a statistically significant increase in mean gap distance after cementation, ceramic material regardless the sintering protocol had no statistically significant effect on mean fracture resistance (P -value = 0.821). Sintering regardless of ceramic type had a statistically significant effect on mean fracture resistance (P -value = 0.002). **Conclusions** The sintering protocol is shown to affect both the marginal adaptation and fracture resistance of multi-layered zirconia FPDs.

Key words: Speed sintering, Regular sintering, Monolithic zirconia, Marginal gap, CAD/CAM, Fracture resistance.

INTRODUCTION

Zirconia (ZrO_2) is a strong, versatile and durable ceramic material that has gained extensive acceptance in different dental treatments [1-4] due to its unique properties, including high flexural strength, fracture

toughness, as well as dimensional stability [5]. These properties are of greatest importance for the application of ZrO_2 -based materials in dentistry.

Since the nineties, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) has

been extensively used in dentistry, due to its strength and toughness properties, rendering it an ideal substitute for metal in metal-ceramic restorations [1, 2].

Zirconia is a metastable polycrystalline ceramic structure composed of monoclinic, tetragonal, and cubic phases, with the phase transitions occurring at different temperatures. One of these phases, the tetragonal phase, which shows the highest mechanical properties such as strength, is held at room temperature with the addition of oxide stabilizers, especially 3Y [1-3].

New multilayered translucent zirconia materials have recently appeared in the dental field and progressed for full-contoured (monolithic) restorations [6-9]. The name “multilayer” has been applied for many zirconia products, which are relatively different in terms of color additives and chemical composition. Manufacturers improved multilayered zirconia materials with a gradient shade technology, as the multilayered zirconia disc has different color additives in each layer to direct and control the translucency of the constituent layers, imitating the natural tooth appearance [8, 9]. These multilayered zirconia materials can be used for both posterior and anterior restorations [10].

Multilayered materials have the same chemical configuration, in terms of yttria contents, for constituent layers with almost equivalent mechanical properties by layer [10, 11]. Although, balancing mechanical and optical properties using a gradient shade technology in those multilayered zirconia materials might be considered a difficult and demanding procedure. Selecting monolithic fully contoured fabrication approach gives the advantage of reducing the possibility of chipping accompanied with the bilayered zirconia design [12]. Furthermore, the conservative preparation guidelines associated

with monolithic zirconia have been presented to preserve hard tooth structures as well as pulpal integrity. Moreover, monolithic zirconia has been recommended to permit for the reuse of vertical margin design during preparation for these restorations [13].

One of the benefits of using Y-TZP as the material of choice for dental applications is well-matching with computer-aided design (CAD) and computer-aided manufacturing (CAM) technology and becomes the material of choice for the fabrication of high-quality all-ceramic dental restorations [14–16]. The integration between CAD/CAM technologies and digital era have simplified and accelerated the advancement of fabrication of dental prostheses, making them more accurate and reproducible. [17]. Zirconia offers many advantages such as the ability to mimic tooth shades although of its opacity, superior mechanical properties, radiopacity, reduced susceptibility to corrosion, dimensional stability, and an elastic modulus comparable to that of metal restorations [18].

During the fabrication of different dental prosthesis from zirconia-based materials, they are sintered in a dental furnace at high temperatures. Sintering process can significantly affect the micro-structure and properties of zirconia. In the process of sintering, heat is transferred from the surface to the core to obtain a matured sintered zirconia restoration [19]. Sintering temperature is an important aspect influencing the size and density of zirconia particles, which directly control on the degree of porosities and the rate of particles growth. The sintering temperature can also affect the strength and resistance of zirconia. In the process of sintering, zirconia goes through considerable shrinkage. By adjusting the sintering protocol through placing the restorations into the furnace at room temperature and regulating respectively the

heating rate, holding temperature, holding time, as well as the cooling rate based on the manufactures instructions. Therefore, variations in sintering parameters of zirconia can directly affect its properties [20, 21].

Poor marginal adaptation of FDPs can enhance plaque retention and change the distribution of the microflora, which might lead to the occurrence of microleakage, secondary caries, root canal infection, and can encourage the onset of periodontal diseases [22]. For most researchers, a marginal fit of below 120 μm in in vivo studies is clinically acceptable [22, 23]. However, there is no clear agreement regarding the appropriate marginal adaptation of FDPs on advanced dentistry. So, clinicians should attempt for the best FDPs marginal fit as possible to decrease threatening potential complications.

The mechanical properties of brittle ceramic materials play a crucial role in preventing crack propagation and ensuring the durability of restorations. These ceramics are more prone to cracking under tensile stress [11]. The mechanical properties that have an important role and characterize the long term function in all ceramic restorations are flexural strength and fracture toughness [24]. Preventing chipping is one of the factors toward using monolithic complete contour zirconia restorations. Monolithic zirconia materials with higher translucency have reduced mechanical properties [25].

Marginal gap distance and fracture resistance are important characteristics that can contribute to clinical performance of all ceramic restorations. Thus this study seeks to evaluate and compare the effect of sintering protocol and thermocycling on marginal adaptation and fracture resistance for zirconium oxide based three units' fixed partial dentures. The null hypothesis is stated that the sintering protocol has no influence on the vertical marginal gap or the fracture

resistance of zirconia based fixed partial dentures.

MATERIALS AND METHODS

Sample size calculation:

This power analysis used marginal gap distance as the primary outcome. Based upon the results of a pilot study conducted using three specimens per subgroup, the effect size for repeated measures ANOVA test (Partial Eta squared) was 0.053 and the correlation among repeated measures was 0.7. Using alpha (α) level of (5%) and Power = 80%, the minimum estimated sample size was a total of 24 specimens (Six samples per subgroup). Sample size calculation was performed using G*Power Version 3.1.9.2.

Samples preparation

A total of 24 three-unit monolithic zirconia FDPs, with one premolars pontic and two retainers, were fabricated. The FDPs were divided into two groups ($n=12$; Fig.2) according to the material used for construction of FPDs Group I: White peak Copra supreme hyperion monolithic zirconia (Whitepeaks Dental Solutions GmbH, Germany), Group II: White peak Copra supreme Symphony monolithic zirconia (Whitepeaks Dental Solutions GmbH, Germany).

Two abutment teeth, the canine and the second premolar, were prepared in a plastic model of the upper jaw (Kavo YZ, Kavo Dental, Biberach, Germany) to receive a three unit monolithic FDP with one premolar pontic. The abutment tooth preparations were designed to allow for an all-ceramic restoration with a 120° chamfer finish line and 15° convergence angle. A sectional-arch impression (President, Coltene, Altstätten, Switzerland) was taken, boxed and poured using a die stone (Vel-Mix, Kerr, Orange, CA, USA) and a master cast was created. The master cast was then scanned with a laboratory

scanner (D900L, 3Shape, Copenhagen, Denmark). The scanning data was uploaded to a computer that had CAD software installed (Exocad, Darmstadt, Germany). The software was used to design monolithic FDPs with the following design parameters based on the instructions of the manufacturer: the connector dimensions were set to 3×3 mm, the minimum wall thicknesses were set to 1 mm, and the cement gap was set at 45 µm. In all groups, the gingival and occlusal radii of the embrasures in the connector areas were set to default CAD software settings (Fig.1). twenty four sectional impression (President, Coltene, Altstätten, Switzerland) were taken for the prepared abutments, boxed and poured into epoxy resin (Polypoxy700, polymer, chemical industries for construction Co., CIC, Egypt) as it simulates the modulus of elasticity of dentin, facilitate marginal gap distance measurements before and after cementation as well as fracture resistance testing procedures.

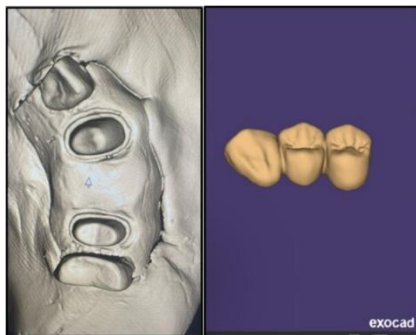


Figure 1. Scanning and designing of the 3 units FDPs.

Sample grouping

The FDPs were divided into two equal groups (n=12; Fig.2) according to the material used for construction of fixed partial dentures. Group I: White peak Copra supreme hyperion monolithic zirconia (WPH), Group II: White peak Copra supreme Symphony monolithic zirconia (WPS). Then each group was further divided into two equal subgroups according to the sintering protocol. Sub group 1: Regular (Normal) sintering protocol, sub group 2: Speed sintering protocol.

FDPs fabrication

The milling unit (InLab MC X5, Dentsply Sirona, Charlotte, NC, USA) received the CAD data to produce 12 monolithic FDPs made of multilayered zirconia material (White peak Copra supreme hyperion monolithic zirconia (WPH)) and 12 monolithic FDPs made of multilayered zirconia material (White peak Copra supreme symphony monolithic zirconia (WPS)). The multilayered disc for white peak material composed of five layers, with a total thickness of 16 mm (Enamel 1, Enamel 2, Dentin 1, Dentin2 and Margin). For group 1(WPH): The first enamel layer represented 16.66% of the total thickness of the disc, the second enamel layer represented 16.66%, and the first dentin layer represented 16.66%, the second dentin layer represents 25% and the margin layer represents 25%. For group 2 (WPS): each of the five layers represents 20% of the total thickness of the disc.

Sintering protocols

Fixed partial dentures were sintered according to the manufacturers' instructions (Table 1) in calibrated sintering furnace (Programat S1 1600, Ivoclar Vivadent-Technical). Each group will be subdivided into two equal subgroups according to sintering protocol. Sub group 1: Regular (Normal) sintering protocol, sub group 2: Speed sintering protocol.

For subgroup 1; the samples was sintered at a heating rate of 10 °C/min up to 950 °C and subsequent heating at 6°C/min up to 1500 °C, followed by a dwell temperature and sintering time of 1500 °C for 90 min, and a cooling rate of 10 °C/min from 1500 °C to 900 °C and 8.3 °C/min from 900 °C to room temperature. For subgroup 2; the samples was sintered at a heating rate of 50 °C/min up to 1100 °C and

subsequent heating at 20°C/min up to 1500 °C, followed by a dwell temperature and sintering time of 1500 °C for 30 min., and a cooling rate of 10 °C/min from 1500 °C to 900 °C and 8.3 °C/min from 900 °C to room temperature.

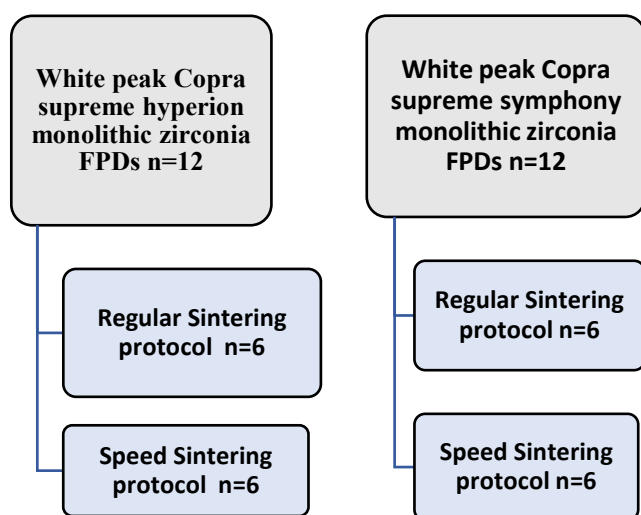


Figure 2. Diagram representing samples grouping.

Table 1. The sintering protocols used in the study.

	Regular(Normal) Program	Speed Program
Heating rate	10° per minute to 950°C	50°C per minute to 1100°C
Holding time	None	None
Heating rate	6°C per minute to 1500°C	20°C per minute to 1500°C
Holding time	at final temperature 90 minutes	at final temperature 30 minutes

Final temperature	1500°C	1500°C
Cooling	unregulated in the closed furnace	unregulated in the closed furnace

Testing procedures

Vertical marginal gap distance measurements

Vertical margin gap distance for all retainers was measured using an image analysis system. This method uses image analysis software (Image J-1b, USA) in combination with a stereomicroscope (Olympus, SZ-PT: Japan) [26]. For all samples, four points were marked with an indelible marking pen in the middle of the buccal, lingual, mesial and distal surfaces of each retainer (canine & premolar). For each retainer, the area of interest was captured by CCD digital camera (DP-10 Olympus, Japan) mounted on the stereomicroscope. The vertical margin gap distance between the cervical margin of the crowns and the finish line was assessed at the previously marked points for each retainer on their corresponding dies before cementation as control. These readings were calculated using the image analysis software. Then FPDs were cemented using self-adhesive Rely X Unicem (3M, ESPE) resin cement following the manufacturer's instructions. A static load of 3Kg (finger pressure) was applied on the occlusal surface of the cemented crowns using a specially designed loading device. The excess cement was removed, and light curing was done for 20 seconds for each surface using light curing unit (Mini LED, 1250 mw/cm², Satelec, Acteon). The load was applied for 10 minutes.

Cyclic fatigue loading

Mechanical aging was performed using programmable equipment; the newly developed four stations multimodal ROBOTA

chewing simulator integrated with thermocyclic protocol (Model ACH-09075DC-T, GERMANY). The specimens were embedded in Teflon housing in the lower sample holder. A weight of 10 kg, which is comparable to 100 N of chewing force, was exerted. The test was repeated 60000 times to clinically simulate 6 months chewing condition, according to previous study [27]. Then, vertical marginal gap distance for all samples were subsequently re-measured after cyclic fatigue loading testing using the same method described previously.

Fracture resistance testing

All samples were individually mounted on a computer-controlled material testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA) with 5 kN loadcell and data were documented using computer software (Instron® Bluehill Lite Software). Samples were secured to the lower fixed compartment of testing machine by tightening screws (Fig. 3). Fracture test was done by compressive mode of load applied occlusally using a metallic rod with spherical tip (5.8 mm diameter) attached to the upper movable compartment of testing machine traveling at cross-head speed of 1mm/min with tin foil sheet in-between to achieve homogenous stress distribution and minimization of the transmission of local force peaks (Fig. 3). The load required to fracture was recorded in Newton (N). All testing procedures sequence were illustrated in (Fig.4).

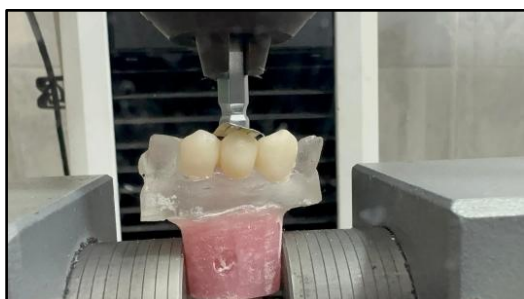


Figure 3. The fracture resistance testing for the samples

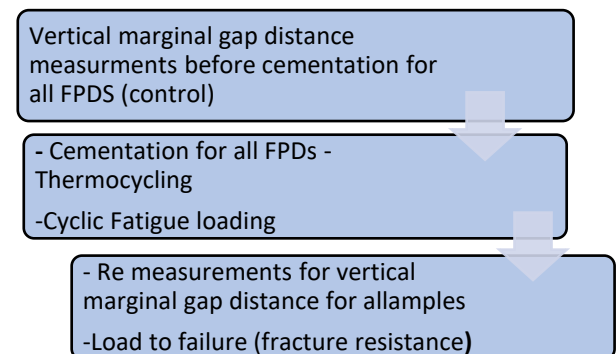


Figure 4. Diagram illustration for the sequence of testing procedures

Data analysis

Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). All data showed normal (parametric) distribution. Data were presented as mean and standard deviation (SD) values. Repeated measures ANOVA test was used to study the effect of ceramic type, sintering, cementation, tooth, surface and their interactions on mean marginal gap distance. Two-way ANOVA test was used to study the effect of ceramic type, sintering and their interactions on mean fracture resistance. Bonferroni's post-hoc test was used for pairwise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY: IBM Corp.

RESULTS

Marginal gap distance (μm)

Repeated measures ANOVA results

The results showed that ceramic type regardless of other variables had no statistically significant effect on mean

marginal gap distance. While sintering regardless of other variables had a statistically significant effect on mean marginal gap distance. The interaction between variables had no statistically significant effect on mean marginal gap distance. Since the interaction between the variables is non-statistically significant, the variables are independent from each other. (Table2).

Comparing both ceramic materials used regarding the marginal gap distance, there was no statistically significant difference for both tested groups (WPH&WPS) regardless the sintering protocol used for both before and after cementations measurements at all surfaces (mesial, distal, buccal &palatal).

Comparison between surfaces regarding the marginal gap distance for the anterior retainer (Canine), for hyperion and regular sintering protocol before cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that distal surface showed the statistically significant highest marginal gap distance. There was no statistically significant difference between buccal and palatal surfaces; both showed statistically significantly lower mean value. Mesial surface showed the statistically significantly lowest mean gap distance.

After cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that distal surface showed the statistically significantly highest gap distance. There was no statistically significant difference between mesial, buccal and palatal surfaces; all showed statistically significant lower mean values, whereas with hyperion and speed sintering protocol whether before or after cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that distal surface showed the statistically significantly highest gap distance. There was

no statistically significant difference between mesial, buccal and palatal surfaces; all showed statistically significantly lower mean values. (Table 3)

With Symphony and regular sintering protocol before cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that distal surface showed the statistically significantly highest gap distance. Buccal surface showed statistically significantly lower mean value. There was no statistically significant difference between mesial and palatal surfaces; both showed statistically significantly lower mean values.

For white peak symphony after speed sintering protocol before cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that distal surface showed the statistically significantly highest gap distance. There was no statistically significant difference between mesial, buccal and palatal surfaces; all showed statistically significantly lower mean values. White peak symphony ceramic material for both sintering protocol after cementation, showed that there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that distal surface showed the statistically significantly highest gap distance. There was no statistically significant difference between buccal and mesial surfaces; both showed statistically significantly lower mean values. Palatal surface showed the statistically significantly lowest mean gap distance. (Table 3).

For the posterior retainer (premolar), Hyperion and regular sintering before cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that mesial surface showed the statistically significantly highest gap distance. There was no statistically

significant difference between distal, buccal and palatal surfaces; all showed statistically significantly lower mean values. After cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that mesial surface showed the statistically significantly highest gap distance. There was no statistically significant difference between distal and buccal surfaces; both showed statistically significantly lower mean values. Palatal surface showed the statistically significantly lowest mean gap distance. (Table 3).

Regarding hyperion ceramic material and speed sintering whether before or after cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that mesial surface showed the statistically significantly highest gap distance. There was no statistically significant difference between distal, buccal and palatal surfaces; all showed statistically significantly lower mean values. For Symphony ceramic material and regular sintering protocol for both before or after cementation, there was no statistically significant difference between surfaces.

With Symphony and speed sintering before cementation, there was no statistically significant difference between surfaces. After cementation, there was a statistically significant difference between surfaces. Pair-wise comparisons revealed that mesial surface showed the statistically significantly highest gap distance. There was no statistically significant difference between distal, buccal and palatal surfaces; all showed statistically

significantly lower mean values. (Table 4).

In all tested groups and subgroups, regular sintering showed statistically significantly lower mean gap distance than speed sintering, while there was a statistically significant increase in mean gap distance after cementation for all tested groups and subgroups. Canine retainers showed statistically significantly lower mean gap distance than premolar retainers except at the distal surface of hyperion before cementation where there was no statistically significant difference between canine and premolar retainers for marginal gap distance measurements.(Table 4).

Fracture resistance (Newton)

Two-way ANOVA results showed that ceramic material regardless the sintering protocol had no statistically significant effect on mean fracture resistance (P -value = 0.821). Sintering regardless of ceramic type had a statistically significant effect on mean fracture resistance (P -value = 0.002). (Table 5) The interaction between variables had no statistically significant effect on mean fracture resistance. Since the interaction between the variables is non-statistically significant, the variables are independent from each other. With both sintering protocols, there was no statistically significant difference between both tested ceramic materials (WPH&WPS). Whereas with hyperion or symphony ceramic material, regular sintering showed a statistically significant lower mean fracture resistance than speed sintering. (Table 5).

Table 2.Repeated measures ANOVA results for the effect of different variables on mean marginal gap distance (μm)

Source of variation	Type III Sum of Squares	df	Mean Square	F-value	P-value	Effect size (<i>Partial eta squared</i>)
Ceramic type	129.5	1	129.5	0.894	0.356	0.043
Sintering	2526.6	1	2526.6	17.449	<0.001*	0.466

Cementation	18550.9	1	18550.9	418.841	<0.001*	0.954
Tooth (Canine or Premolar)	37703.2	1	37703.2	188.091	<0.001*	0.904
Surface	2546.2	3	848.7	11.015	<0.001*	0.355
Ceramic type x Sintering x Cementation x Tooth x Surface interaction	12.3	3	4.1	0.284	0.837	0.014

*df: degrees of freedom = (n-1), *: Significant at $P \leq 0.05$*

Table3. The mean, standard deviation (SD) values and results of repeated measures ANOVA test for comparison between marginal gap distance (μm) of the two ceramic types and comparison between surfaces within each group

Sintering	Cementation	Tooth	Surface	Hyperion		Symphony		P-value
				Mean	SD	Mean	SD	
Regular	Before cementation	Canine	Mesial	39.2 ^C	4.8	40.5 ^C	3.7	0.738
			Distal	54.7 ^A	4.4	52.5 ^A	5.6	0.465
			Buccal	44 ^B	4.1	45.2 ^B	5	0.690
			Palatal	42 ^B	6.8	39.5 ^C	7.1	0.545
			Overall	45	3.8	44.4	4.2	0.815
			P-value	<0.001*		<0.001*		
		Premolar	Mesial	73 ^A	6	68.7	4.2	0.341
			Distal	59.3 ^B	6.8	61.5	5	0.679
			Buccal	59.8 ^B	10	59.5	6.5	0.954
			Palatal	57.8 ^B	7.5	58.8	2.5	0.868
			Overall	62.5	5.8	62.1	3	0.906
			P-value	0.001*		0.068		
	After cementation	Canine	Mesial	51.3 ^B	5.6	55.8 ^B	8	0.302
			Distal	63.2 ^A	5.1	63.8 ^A	6.8	0.845
			Buccal	53.5 ^B	6.3	57.5 ^B	5.7	0.322
			Palatal	51.3 ^B	7.2	47.7 ^C	7	0.408
			Overall	54.8	3.1	56.2	5.3	0.523
			P-value	0.001*		0.002*		
		Premolar	Mesial	84.7 ^A	6.3	84	5.4	0.882
			Distal	72.3 ^B	10.5	73.3	9.6	0.869
			Buccal	73.8 ^B	9.1	76.8	5.8	0.483
			Palatal	68.7 ^C	9	76.3	7.7	0.129
			Overall	74.9	5.5	77.6	4.3	0.095
			P-value	0.002*		0.059		
Speed	Before cementation	Canine	Mesial	49.5 ^B	7.4	54 ^B	9.7	0.266
			Distal	61.2 ^A	5.8	59.8 ^A	4	0.651
			Buccal	51.7 ^B	6	52.5 ^B	4.8	0.776
			Palatal	51.8 ^B	7.8	52.5 ^B	6.3	0.871
			Overall	53.5	5.4	54.7	4.3	0.618
			P-value	<0.001*		0.002*		
		Premolar	Mesial	86.8 ^A	9.7	82.5	9.4	0.341
			Distal	74.7 ^B	14.3	75.7	6.6	0.848
			Buccal	72.7 ^B	12.6	75.8	9.5	0.586
			Palatal	73 ^B	14.3	75.3	12.5	0.698
			Overall	76.8	8.4	77.3	6.2	0.705
			P-value	0.002*		0.147		
	After cementation	Canine	Mesial	64.3 ^B	6.3	66.5 ^B	9	0.616
			Distal	78.7 ^A	6	77.2 ^A	5.3	0.661
			Buccal	64 ^B	8.6	68.5 ^B	6.3	0.267
			Palatal	64 ^B	7.5	59.3 ^C	8.3	0.295
			Overall	67.8	3.1	67.9	5.3	0.915
			P-value	<0.001*		<0.001*		
		Premolar	Mesial	95.7 ^A	10.3	100.2 ^A	7.9	0.323
			Distal	86.2 ^B	14.3	91 ^B	5	0.430
			Buccal	84.7 ^B	7	89.2 ^B	6.7	0.296
			Lingual	88 ^B	9.9	87.7 ^B	6.7	0.946
			Overall	88.6	5.3	92	5.8	0.472
			P-value	0.034*		0.014*		

*: Significant at $P \leq 0.05$, Different superscripts in the same column indicate statistically significant difference between surfaces

Table 4. P-values for comparisons between sintering types, values before and after cementation as well as premolar and molar

*: Significant at $P \leq 0.05$

Comparison	Ceramic	Surface	Canine		Premolar	
			Before cementation	After cementation	Before cementation	After cementation
Regular Vs. Speed sintering	Hyperion	Mesial	0.016*	0.006*	0.005*	0.022*
		Distal	0.037*	<0.001*	0.007*	0.032*
		Buccal	0.015*	0.015*	0.036*	0.018*
		Palatal	0.025*	0.008*	0.019*	0.001*
		Overall	0.005*	0.009*	0.010*	0.012*
	Symphony	Mesial	0.003*	0.021*	0.005*	0.002*
		Distal	0.020*	0.001*	0.012*	0.008*
		Buccal	0.019*	0.011*	0.010*	0.008*
		Palatal	0.004*	0.014*	0.011*	0.030*
		Overall	0.010*	0.002*	0.006*	0.007*
Comparison	Ceramic	Surface	Canine		Premolar	
			Regular sintering	Speed sintering	Regular sintering	Speed sintering
Before Vs. After cementation	Hyperion	Mesial	<0.001*	<0.001*	0.017*	0.048*
		Distal	0.002*	<0.001*	0.027*	0.047*
		Buccal	0.005*	0.001*	0.002*	0.007*
		Palatal	0.001*	<0.001*	0.009*	0.001*
		Overall	<0.001*	<0.001*	0.015*	0.021*
	Symphony	Mesial	<0.001*	<0.001*	0.003*	0.001*
		Distal	<0.001*	<0.001*	0.042*	0.011*
		Buccal	0.001*	<0.001*	<0.001*	0.003*
		Palatal	0.002*	0.009*	<0.001*	0.003*
		Overall	<0.001*	<0.001*	0.001*	0.005*
Comparison	Ceramic	Surface	Regular sintering		Speed sintering	
			Before cementation	After cementation	Before cementation	After cementation
Canine Vs. Premolar	Hyperion	Mesial	<0.001*	<0.001*	<0.001*	0.023*
		Distal	0.273	0.007*	0.004*	<0.001*
		Buccal	0.005*	<0.001*	<0.001*	<0.001*
		Palatal	0.011*	0.003*	0.001*	<0.001*
		Overall	0.035*	<0.001*	<0.001*	<0.001*
	Symphony	Mesial	0.023*	0.042*	0.006*	<0.001*
		Distal	<0.001*	0.010*	<0.001*	0.001*
		Buccal	<0.001*	0.003*	<0.001*	<0.001*
		Palatal	<0.001*	<0.001*	<0.001*	<0.001*
		Overall	<0.001*	0.005*	<0.001*	<0.001*

Table 5 .The mean, standard deviation (SD) values and results of two-way ANOVA test for comparison between fracture resistances (N) with different interactions of variables

	Hyperion		Symphony		P-value
	Mean	SD	Mean	SD	
Regular sintering	1087.6	131.7	1079	78.2	0.902
Speed sintering	1264.7	132.8	1251.1	123.9	0.845
P-value	0.018*		0.021*		

*: Significant at $P \leq 0.05$

DISCUSSION

This study was conducted to investigate the effect of sintering protocol on marginal adaptation and fracture resistance of zirconium oxide based three unit fixed partial dentures. The null hypothesis is rejected, suggesting that the sintering protocol has no influence on the vertical marginal gap distance or the fracture resistance of zirconia based fixed partial dentures for the two types of material used.

Pure zirconia is a ceramic material that occurs in three phases based on the temperature: monoclinic, from room temperature to 1170 °C; tetragonal, from 1170 to 2370°C; and cubic, higher than 2370 °C [28, 29]. Although, monoclinic zirconia has lowered mechanical properties, restricts its clinical application. In order to stabilize tetragonal zirconia at room temperature, Y₂O₃, CaO, MgO, or CeO₂ stabilizers are incorporated in the zirconia-crystal structure during sintering. Amongst these stabilizers, yttria (Y₂O₃) is the utmost popular for dental zirconia. Based upon the Y₂O₃ content, YSZ is categorized as tetragonal zirconia polycrystals (TZP), containing 3-mol% Y₂O₃ and being denoted to as 3Y-TZP, containing 4–6 mol% Y₂O₃ and being referred to as 4–6Y-PSZ. YSZ has superior mechanical properties because of the stress-induced transformation, which raises the fracture toughness of the material.

Conventionally, the manufacturing of zirconia prostheses involves a long sintering process [30]. Whereas, due to the growing request for one-day fabrication of a dental prosthetics, manufacturers introduced speedy sintering schedules, such as speed sintering and, recently, high-speed sintering, which attain higher heating and cooling rates

compared to that obtained with conventional(regular) sintering protocols[30]. Throughout high speed sintering, zirconia undergoes rapid temperature changes as it is rapidly heated to the sintering temperature and then rapidly cooled. The 3~6Y-zirconia, primarily used for dental prosthetics, undergoes phase transformations according to the sintering temperature used [31-33].

CAD/CAM technology used computer-aided design and fabrication of ceramic prostheses and the process is more time consuming than conventional techniques [34]. Sintering is responsible for offering the strengths properties to the zirconia restoration. Several sintering approaches have been improved and they affect the microscopic structure, mechanical properties, as well as esthetics of zirconia [35]. There is no clear definition of speed-sintering of dental zirconia. However, speed-sintering typically consists of a fast heating step, a short dwell time at the sintering temperature, and a fast cooling step, enabling sintering in less than 240 min. Speed-sintering of YSZ within only 10–30 min has even been reported[34-36].

In this study, cementation for all FPD samples, thermocycling as well as cyclic fatigue loading was applied to all samples to test the ceramic material performance under clinical simulating condition.

Marginal gap distance was measured before cementation (as control group) to study the effect of the cement upon the gap distance. Regarding marginal gap distance testing method, many alternatives in the testing approaches, and measuring tools is possible, however, the direct view method is more accessible, easy, rapid, and non-destructive and the crown is retrievable, contrasting sectioning method which causes destruction of

the crown [37]. All measurements were made on the corresponding epoxy die. Each surface was measured three times and the mean value was obtained for more accurate findings. All the measurements were made by one operator to lower the statistical errors as much as possible. This was in accordance with Sulaiman et al, [38]. Image analysis software was used to identify the amounts of space from the values obtained by drawing approximately 6000 perpendicular lines per cross section to obtain more reliable data [37]. And this software has been used in several other studies and has proven its reliability in evaluation of the fit [39, 40].

The results of the present study revealed that in all tested groups and subgroups, regular sintering showed statistically significantly lower mean gap distance than speed sintering, while there was a statistically significant increase in mean gap distance after cementation for all tested groups and subgroups. Canine retainers showed statistically significantly lower mean gap distance than premolar retainers except at the distal surface of hyperion before cementation where there was no statistically significant difference between canine and premolar retainers for marginal gap distance measurements.

This was in accordance with Kunii et al. [41] evaluated the fit of the pontic and non-pontic sides of three and four-unit zirconia FDPs. They found that the non-pontic side developed a better marginal fit. The cause for this is that the volume of zirconia at the pontic area, which is higher than that of the retainer, affected the amount of shrinkage occurring during secondary firing. The difference in the amount of shrinkage between the pontic area and the retainer side caused a dimensional change in the FDPs. The same as our results which showed that the highest marginal gap distance was recorded in the distal surface for

the canine retainer and the mesial surface for the premolar retainer regardless the type of multi-layered zirconia used. In the three-unit zirconia FDPs, it should be considered that the danger of causing marginal discrepancy is higher on the pontic side than on the non-pontic side.

It is important to mention that the results of the vertical marginal gap distance for all samples in this study are within the clinically acceptable ranges of 120µm even after cyclic fatigue loading for all measurements before and after cementation [37-40].

The transformation to the monoclinic phase can disturb the mechanical properties and stability of Y-TZP materials [40]. The mechanical properties of Y-TZP rely on the aforesaid tetragonal to monoclinic transformation [25, 42]. Whereas, the tetragonal phase on the Y-TZP surface can change into the monoclinic phase, which is supplemented by an increase in volume (4–5%) that cause compressive stresses [43, 44]. This transformation can produce severe clinical implications, such as an increased risk and tendency of fracture during function. The multilayered zirconia materials are commonly accompanied by high-speed sintering protocols recommended by the manufacturers. As these high-speed sintering schedules turn out to be more commonly available. [45]. Research on the effects of the sintering speed on the optical and mechanical properties of zirconia is being actively conducted. Many studies studied and compared different (slow and fast) sintering protocols of zirconia [46,47]. Juntavee and Attashu evaluated the role of sintering duration and temperature on the mechanical properties of zirconia and concluded that a long sintering time with high sintering temperature causes a higher flexural strength of zirconia [48]. The speed sintering had increased biaxial flexural strengths which might be due to changes in the material

structure from the degradation of the metal salts [38].

This study assessed the effect of sintering protocol on the fracture resistance of WPH & WPS zirconia blocks. Two-way ANOVA results showed that ceramic material regardless the sintering protocol had no statistically significant effect on mean fracture resistance (P -value = 0.821). Sintering regardless of ceramic type had a statistically significant effect on mean fracture resistance (P -value = 0.002).

This was in accordance with Armaghateh et al, who found that increasing the sintering temperature increased the flexural strength of zirconia. The maximum flexural strength was noted in specimens sintered at 1500°C [49].

The highest fracture resistance was recorded for speed sintering protocol with both ceramic material, this was contrary to the results reported by Ersoy et al, evaluated 120 In-Coris ZI zirconia specimens sintered in three groups: at 1510°C for 120 min, 1540°C for 25 min, and 1580°C for 10 min. They measured the flexural strength of specimens, and reported maximum flexural strength in specimens sintered at 1580°C for 10 min. Also, the flexural strength of specimens

sintered at 1540°C was significantly higher than that of specimens sintered at 1510°C [50].

This study investigated the influence of marginal adaptation as well as fracture resistance of FDPs in vitro in addition to trials to simulate the clinical condition by performing thermocycling and cyclic fatigue loading to all samples. Hence, there was no clear consensus on whether the differences in in each group as shown in the results of this study had a clinical impact. Therefore, it is thought that further clinical research (in-vivo) is necessary to see how each factor compared in this study affects clinically.

CONCLUSIONS

Based on the results of this study, the following conclusions can be drawn:

(1) The sintering protocol of the three-unit zirconia FDPs affected the marginal gap distance. (2) The sintering protocol of the three-unit zirconia FDPs affected the fracture resistance. (3) The regular sintering protocol showed a lower significant statistical results for both marginal gap distance and fracture resistance for all tested samples.

Conflict of interest

The author reports no conflicts of interest to declare with respect to this study.

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REFERENCES

1. Alghazzawi, T.F. Advancements in CAD/CAM technology: Options for practical implementation. *J. Prosthodont. Res.* 2016; 60: 72–84.
2. Camposilvan, E.; Leone, R.; Gremillard, L.; Sorrentino, R.; Zarone, F.; Ferrari, M.; Chevalier, J. Aging resistance, mechanical properties and translucency of different yttria-stabilized zirconia ceramics for monolithic dental crown applications. *Dent. Mater.* 2018; 34:879–890.
3. Kolakarnprasert, N.; Kaizer, M.R.; Kim, D.K.; Zhang, Y. New multi-layered zirconias: Composition, microstructure and translucency. *Dent. Mater.* 2019; 35: 797–806.

4. Huang, B.; Chen, M.; Wang, J.; Zhang, X. Advances in zirconia-based dental materials: Properties, classification, applications, and future prospects. *J. Dent.* 2024; 147: 105111.
5. Nakamura, K.; Harada, A.; Inagaki, R.; Kanno, T.; Niwano, Y.; Milleding, P.; Örtengren, U. Fracture resistance of monolithic zirconia molar crowns with reduced thickness. *Acta Odontol. Scand.* 2015;73: 602–608.
6. Li, X.; Wang, Q.; Qiu, X.; Zhao, B. Effect of different CAD/CAM cutting depths on the post-fatigue load-bearing capacity of novel multilayer zirconia restorations. *J. Dent.* 2021; 111: 103709.
7. Rosentritt, M.; Preis, V.; Schmid, A.; Strasser, T. Multilayer zirconia: Influence of positioning within blank and sintering conditions on the in vitro performance of 3-unit fixed partial dentures. *J. Prosthet. Dent.* 2022; 127: 141–145.
8. Kaizer, M.R.; Kolakarnprasert, N.; Rodrigues, C.; Chai, H.; Zhang, Y. Probing the interfacial strength of novel multi-layer zirconias. *Dent. Mater.* 2020; 36: 60–67.
9. Ziyad, T.A.; Abu-Naba'a, L.A.; Almohammed, S.N. Optical properties of CAD-CAM monolithic systems compared: Three multi-layered zirconia and one lithium disilicate system. *Heliyon.* 2021; 7: 8151.
10. Zhang, Y. Making Yttria-Stabilized Tetragonal Zirconia Translucent. *Dent. Mater.* 2014; 30: 1195–1203.
11. Reyes, A.R.; Dennison, J.B.; Powers, J.M.; Sierraalta, M.; Yaman, P. Translucency and Flexural Strength of Translucent Zirconia Ceramics. *J. Prosthet. Dent.* 2023; 129: 644–649.
12. Kontonasaki, E., Giasimakopoulos, P. and Rigos, A. E. Strength and aging resistance of monolithic zirconia: An update to current knowledge. *Japanese Dental Science Review.* 2020; 56: 1–23.
13. Poggio, C. E., Dosoli, R., & Ercoli, C. A retrospective analysis of 102 Zirconia single crowns with knife-edge margins. *The Journal of Prosthetic Dentistry.* 2012; 107: 316–321.
14. . Jakovac, M.; Klaser, T.; Bafti, A.; Skoko, Ž.; Pavić, L.; Žic, M. The Effect of Y3+ Addition on Morphology, Structure, and Electrical Properties of Yttria-Stabilized Tetragonal Zirconia Dental Materials. *Materials.* 2022; 15: 1800.
15. Machry, R.V.; Borges, A.L.S.; Pereira, G.K.R.; Kleverlaan, C.J.; Venturini, A.B.; Valandro, L.F. Influence of the Foundation Substrate on the Fatigue Behavior of Bonded Glass, Zirconia Polycrystals, and Polymer Infiltrated Ceramic Simplified CAD-CAM Restorations. *J. Mech. Behav. Biomed. Mater.* 2021; 117: 104391.
16. Jakovac, M.; Klaser, T.; Radatović, B.; Skoko, Ž.; Pavić, L.; Žic, M. Surface Characterization and Conductivity of Two Types of Lithium-Based Glass Ceramics after Accelerating Ageing. *Materials.* 2020; 13: 5632.
17. Amornvit, P., Rokaya, D., & Sanohkan, S. Comparison of accuracy of current ten intraoral scanners. *BioMed Research International.* 2021; 1.
18. Denry, I., & Kelly, J. R. Emerging ceramic-based materials for Dentistry. *Journal of Dental Research.* 2014; 93: 1235–1242.
19. Taruta S, Yamaguchi I, Yamakami T, Yamaguchi T. Sintering behavior and mechanical properties of machinable zirconia/mica composites. *J Asian Ceram Soc.* 2019; 7: 342-9.
20. Janney MA, Calhoun CL, Kimrey HD. Microwave sintering of solid oxide fuel cell materials: I, zirconia-8 mol% yttria. *J Am Ceram Soc.* 1992; 75: 341-6.
21. Zhang HB, Kim BN, Morita K, Yoshida H, Lim JH, Hiraga K. Optimization of high-pressure sintering of transparent zirconia with nano-sized grains. *J Alloys Compd.* 2010; 508: 196-9.

22. Reich S, Wichmann M, Nkenke E, Proeschel P, Clinical fit of all-ceramic three-unit fixed partial dentures generated with three different CAD/CAM systems. *Eur J Oral Sci.* 2005; 113:174-9.
23. Abduo J, Judge RB, Implications of implant framework misfit: a systematic review of biomechanical sequelae. *Int J Oral Maxillofac Implants.* 2014; 29:608-21.
24. Vichi, A.; Zhao, Z.; Paolone, G.; Scotti, N.; Mutahar, M.; Goracci, C.; Louca, C. Factory Crystallized Silicates for Monolithic Metal-Free Restorations: A Flexural Strength and Translucency Comparison Test. *Materials* .2022; 15: 7834.
25. I,seri, U.; Özkurt, Z.; Yalniz, A.; Kazazoglu, E. Comparison of Different Grinding Procedures on the Flexural Strength of Zirconia. *J. Prosthet. Dent.* 2012, 107: 309–315.
26. Hamza T, Al-Baili M, Abdel-Aziz M. Effect of artificially accelerated aging on the marginal fit and color stability of laminate veneers. *Stomatological Dis Sci.* 2018, 2: 1-7.
27. Nawafleh N, Hatamleh M, Elshiyab S and Mack F, Lithium Disilicate Restorations Fatigue Testing Parameters: A Systematic Review *Journal of Prosthodontics.* 2016, 25: 116–126.
28. Zhang Y, Lawn BR. Novel zirconia materials in dentistry. *J Dent Res.* 2018; 97: 140–7.
29. Ozdogan A, Ozdemir H. The effects of repetitive firing processes on the optical, thermal, and phase formation changes of zirconia. *J Adv Prosthodont.* 2020; 12: 9–14.
30. Al-Haj Husain, N.; Özcan, M.; Dydyk, N.; Joda, T. Conventional, Speed Sintering and High-Speed Sintering of Zirconia: A Systematic Review of the Current Status of Applications in Dentistry with a Focus on Precision, Mechanical and Optical Parameters. *J. Clin. Med.* 2022; 11: 4892.
31. Yamashita, I.; Tsukuma, K. Phase Separation and Hydrothermal Degradation of 3 mol% Y2O3 -ZrO2 Ceramics. *J. Ceram. Soc. Japan.* 2005; 113:530–533.
32. Belli, R.; Hurle, K.; Schürlein, J.; Petschelt, A.; Werbach, K.; Peterlik, H.; Rabe, T.; Mieller, B.; Lohbauer, U. A revised relationship between fracture toughness and Y2O3 content in modern dental zirconias. *ChemRxiv* 2021.
33. Scott, H.G. Phase relationships in the zirconia-yttria system. *J. Mater. Sci.* 1975; 10:1527–1535.
34. Padrós R, Giner L, Herrero-Climent M, Falcao-Costa C, Ríos-Santos JV, Gil FJ. Influence of the CAD-CAM systems on the marginal accuracy and mechanical properties of dental restorations. *International Journal of Environmental Research and Public Health.* 2020; 17:4276.
35. Kilinc H, Sanal FA. Effect of sintering and aging processes on the mechanical and optical properties of translucent zirconia. *Journal of Prosthetic Dentistry.* 2021, 126(1):129.e121-129.
36. Amat NF, Muchtar A, Amril MS, Ghazali MJ, Yahaya N. 2019. Effect of sintering temperature on the aging resistance and mechanical properties of monolithic zirconia. *Journal of Materials Research and Technology* 8(1):1092–1101.
37. Kale E, Seker E, Yilmaz B, Ozcelik TB. Effect of cement space on the marginal fit of CAD-CAM-fabricated monolithic zirconia crowns. *J Prosthet Dent* 2016;16:890–5.
38. Sulaiman TA, Abdulmajeed AA, Shahramian K, Lassila L. Effect of different treatments on the flexural strength of fully versus partially stabilized monolithic zirconia. *Journal of Prosthetic Dentistry.* 2017; 2:216–220.
39. Almeida e, Silva JS, Erdelt K, Edelhoff D, Araujo E, Stimmelmayer M, Vieira LC, et al. Marginal and internal fit of four-unit zirconia fixed dental prostheses based on digital and conventional impression techniques. *Clin Oral Investig* 2014;18:515–23.

40. Keul C, Stawarczyk B, Erdelt KJ, Beuer F, Edelhoff D, Guth JF. Fit of 4-unit FDPs made of zirconia and CoCr-alloy after chairside and labside digitalization—a laboratory study. *Dent Mater* 2014;30:400–7.
41. Kunii J, Hotta Y, Tamaki Y, Ozawa A, Kobayashi Y, Fujishima A, et al. Effect of sintering on the marginal and internal fit of CAD/CAM-fabricated zirconia frameworks. *Dent Mater J* 2007;26:820–6.
42. I,seri, U.; Ozkurt, Z.; Kazazoğlu, E.; Küçükoğlu, D. Influence of Grinding Procedures on the Flexural Strength of Zirconia Ceramics. *Braz. Dent. J.* 2010;21: 528–532.
43. Jakovac, M.; Klasner, T.; Radatović, B.; Baftić, A.; Skoko, Ž.; Pavić, L.; Žić, M. Impact of Sandblasting on Morphology, Structure and Conductivity of Zirconia Dental Ceramics Material. *Materials*. 2021, 14: 2834.
44. Yi, Y.A.; Ahn, J.S.; Park, Y.J.; Jun, S.H.; Lee, I.B.; Cho, B.H.; Son, H.H.; Seo, D.G. The Effect of Sandblasting and Different Primers on Shear Bond Strength between Yttria-Tetragonal Zirconia Polycrystal Ceramic and a Self-Adhesive Resin Cement. *Oper. Dent.* 2015, 40;63–71.
45. Liu, H.; Inokoshi, M.; Nozaki, K.; Shimizubata, M.; Nakai, H.; Too, T.D.C.; Minakuchi, S. Influence of high-speed sintering protocols on translucency, mechanical properties, microstructure, crystallography, and low-temperature degradation of highly translucent zirconia. *Dent. Mater.* 2022; 3:451–468.
46. Ban, S.; Okuda, Y.; Noda, M.; Tsuruki, J.; Kawai, T.; Kono, H. Contamination of Dental Zirconia before Final Firing: Effects on Mechanical Properties. *Dent. Mater. J.* 2013;32: 1011–1019.
47. Ordoñez Balladares A, Abad-Coronel C, Ramos JC, Martín Biedma BJ. Fracture resistance of sintered monolithic zirconia dioxide in different thermal units. *Materials*. 2022; 15:2478.
48. Juntavee N, Attashu S. Effect of different sintering process on flexural strength of translucency monolithic zirconia. *Journal of Clinical and Experimental Dentistry*. 2018; 108:821-830.
49. Armaghan S, Fariborz V, Amirarslan H, Erfan N, Masood N, Maryam F. Effects of sintering temperature on flexural strengths of two types of zirconia. *Frontiers in dent.* 2022;19:1-7.
50. Ersoy NM, Aydoğdu HM, Değirmenci BÜ, Çökük N, Sevimay M. The effects of sintering temperature and duration on the flexural strength and grain size of zirconia. *Acta Biomater Odontol Scand.* 2015;1:43-50.