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EFFECT OF SINTERING TECHNIQUE ON SHADE AND SHEAR BOND STRENGTH OF CAD/CAM MONOLITHIC TRANSLUCENT ZIRCONIA

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ABSTRACT

Introduction: The fabrication of zirconia dental restoration is a time-consuming process due to traditional slow sintering schemes. A speed sintering protocol has been developed to meet the demand for time and cost-effective chair-side CAD/CAM-produced restorations, as well as to control ceramic microstructures for better translucency. Fast heating and cooling rates, as well as shorter sintering dwell times are known to affect the microstructure and properties of zirconia. **Aim of study:** This study aimed to investigate and compare between three types of monolithic translucent zirconia in terms of the shade and the shear bond strength of the zirconia samples to adhesive resin cement after conventional and speed sintering. **Material and Methods:** Three types of zirconia discs were cut into 20 square shaped samples, 10 large square shaped and 10 small square shaped and bonded to each other. Bonded samples were divided according to type of zirconia into 3 groups: Group 1: Dental direct ZX^2, Group 2: Dental Direct ZW iso, Group 3: Zolid HT+. Each group was subdivided according to sintering technique into 2 subgroups. Shade and shear bond test were measured. **Results:** The speed sintering of the three types of zirconia showed a lower ΔE values than that showed with the conventional sintering, and among the speed and conventional sintering subgroups Dental Direkt Bio ZX^2 showed the lowest ΔE value and highest shear bond strength value followed by Dental Direct Bio ZW iso and Zolid Ht+. **Conclusion:** The speed sintering technique of the monolithic translucent zirconia gave better color shade reproduction and higher shear bond strength to resin cement than that of the conventional sintering technique. Dental Direkt ZX^2 produced better color shade reproduction and higher shear bond strength to resin cement than that of the Dental Direkt ZW iso and Zolid Ht+ in speed and conventional sintering technique.

INTRODUCTION

The rapid evolution of dental zirconia, 3 yttrium stabilized tetragonal zirconia polycrystalline (3Y-TZP) ceramics have gained tremendous popularity as restorative materials as a result of their excellent mechanical properties and good biocompatibility, but quite opaque owing to the large grain size and the presence of porosity which is evident at the microstructural level of these materials [1,2].

Newer translucent varieties of zirconia have been developed recently, to improve their transmittance, so that they can be used in esthetically demanding clinical situations. Studies [3,4] done on these newer materials have shown that they are more translucent than conventional zirconia.

The use of translucent zirconia has the potential to eliminate delamination of the veneering ceramic, which has been known to be a common clinical problem and also reduce the amount of tooth preparation required.

But the fabrication of zirconia dental restoration is a time-consuming process due to traditional slow sintering schemes. A speed sintering protocol has been developed to meet the demand for time and cost-effective chair-side CAD/CAM-produced restorations, as well as to control ceramic microstructures for better translucency. Although the speed sintering protocols have already been used to densify the dental Y-TZP, the wear properties of these restorations remain elusive. Fast heating and cooling rates, as well as shorter sintering dwell times are known to affect the microstructure and properties of zirconia [(4)].

Still the consequence of these alterations on mechanical and physical properties of zirconia remains questionable. So, the objective of this study was to explore the effect of changing sintering protocols long and speed cycles on optical and mechanical properties of monolithic translucent zirconia.

MATERIAL AND METHODS

This study was approved by the research ethical committee of Faculty of Dentistry- Suez Canal University (n.2022/118). Low speed cutting saw device was employed to cut three different materials of translucent zirconia discs: Dental Direkt Bio ZX² [1], Dental Direkt Bio ZW iso color [2] and Zolid Ht+[3].

Each disc was cut into 20 square shaped samples, 10 large square shaped (9.6x9.6x2.4mm) and 10 small square shaped (4.8x4.8x2.4mm).

1 Dental Direkt GmbH Industriezentrum, Germany

2 Dental Direkt GmbH Industriezentrum, Germany

3 Amann Girrbach AG, Herrschaftswiesen, Austria

These dimensions were larger than the selected dimensions by 19% according to the manufacture instruction in order to compensate the sintering shrinkage of zirconia, so that small square shaped samples were (4.8x4.8x2.4mm) before sintering and (4x4x2 mm) after sintering while the large square shaped samples were (9.6x9.6x2.4mm) before sintering and (8x8x2) after sintering.

Tabeo-1/M/Zirkon-100 [4] was used for the conventional and speed sintering of all the samples.

Each type of the translucent zirconia was sintered at 1450°C and heating rate started from 20°C, but conventional and speed sintering technique have different heating rate, holding time and cooling time in each material according to manufacture instructions (Table 1).

Polishing of the surface of the small square shaped samples was done using orange disc polisher [5], epoxy resin [6] bases were made for mounting large square shaped samples.

The bonded surface of the large and small square shaped samples was treated by sandblasting [7], using 110 µm aluminum oxide particles (Al_2O_3) under 2.5 bar pressure at an angle 45°, distance 10 mm for 15 seconds, then placed in an ultrasonic water bath cleaner [8] for 10 minutes to remove the alumina dust produced from sandblasting, then dried with Oil free air blast.

4 Tabeo-1/M/Zirkon-100, MIHM-VOGT Gmbh & Co.KG. Friedrich-List-StraBe 876297 Stutensee-Blankenloch Germany.

5 Coltene/Whaledent AG. Feldwiesenstrasse 20-9450 Altstatten, Switzerland

6 Chemapoxy. CMB, 69 Zone G.Khofo Gate Hadaek Al Ahram. Giza, Egypt.

7 Vaniman Manufacturing Co.25799 Jefferson Ave Murrieta, CA 92562

8 Merck SA, an affiliate of Merck KGaA, Darmstadt, Germany

Table (1) *Summarizes the grouping of the bonded square shaped samples after sintering.*

Material	DD Bio ZX[2]		DD Bio ZW iso		Zolid Ht+	
Group no.	Group 1		Group 2		Group 3	
Sub group.	C1	S1	C2	S2	C3	S3
Sintering time	9 hs	2 hs	9 hs	2 hs	8 hs	2 hs
Sintering temperature	1450°C	1450°C	1450°C	1450°C	1450°C	1450°C
No of bonded samples.	5	5	5	5	5	5
No of XRD samples.	1	1	1	1	1	1

C: Conventional sintering *S: Speed sintering*

Each small square shaped samples size (4x4x2mm) was bonded to the large square shaped samples size (8x8x2mm) of the same material and the type of sintering using Panavia sa resin cement[1], following the manufacturer's instructions mixing of the cement was done.

To bond the samples under the same conditions, a specially designed device was used; the device was used to maintain a static load of 3 Kg weight was placed on the top of the small square shaped sample for bonding of all samples. Excess cement was then removed using a probe after 3 seconds (Tacky curing) and adhesive cement was then light-cured [2] for 20 seconds on each surface of the square shaped sample.

Shade measurement Test

A digital spectrophotometer[3] was used to measure the color shade differences (ΔE) value between the selected color shade (A3 classical shade) and the produced color shade of the samples in all subgroups. The middle part of the un-bonded surface of the small square shaped sample in each bonded sample was selected for the tip of the probe of the easy shade device with white background. The ΔE reading values was calculated and considered as the final reading for the investigated restoration. The results were recorded, tabulated and statistically analyzed. The ΔE values were lesser than 2 take us a reference in this study which was considered clinically acceptable based on previous study [(5)].

Shear Bond Strength Test

The shear bond strength was performed using a computerized universal testing machine [4]. The bonded square shaped sample was firmly attached to the lower fixed jaw of the machine with horizontal orientation using a simple vice. Meanwhile, the chisel edge blade was firmly attached to the underside of the upper movable jaw of the device, So that the chisel edge was parallel and adjacent to the bonded interface. The bonded square shaped sample was loaded till failure (de-bond) at a crosshead speed of 0.5 mm/min. The initial failure load was

1 Kuraray dental, Inc, Japan

2 3M™ Elipar™ DeepCure-L wavelength 430–480 nm

3 Vita easy shade , VITA Zahnfabrik H. Rauter GmbH & Co. KG Postfach 1338 D-79704 Bad Säckingen, Germany

4 TIRA test 2805, Tira GmbH, Eisfelder Strabe23/25 D-9528, Schalkau,Germany.

defined when any two signs of either a sharp fall in the loading curve appeared, or an audible cracking sound was heard. All data was recorded, tabulated, and subjected to statistical analysis. Additionally, the bonded samples separation was recorded for all samples. The shear bond strength value greater than 25 MPa was taken as a reference in this study which was considered clinically acceptable based on previous study [6].

X-ray diffraction (XRD)

In order to investigate the effect of sintering on the crystalline structure of the investigated zirconia in this study, two square shaped samples of each of the three types of zirconia (6 more samples) were fabricated. 3 samples were subjected to conventional sintering while the other 3 samples were subjected to speed sintering. The square shaped samples were scanned using XRD to determine the degree of tetragonal to cubic conversion, Y_2O_3 % and Al_2O_3 % content.

RESULTS

1. Shade measurements results:

The speed sintering of the three types of zirconia discs employed in this study showed a lower ΔE values than that showed with the conventional sintering, as summarized in **(Table 2)** and presented in **(Figure 1)**. Statistical analysis using one-way ANOVA and the post-hoc test showed statistically significant difference between studied subgroups ($F=32.58$ & $P<0.001$).

Among the speed sintering subgroups Dental Direkt Bio ZX^2 showed the lowest ΔE value followed by Dental Direct Bio ZW iso and Zolid Ht+: (1.20± 0.15, 1.70±0.15 and 2.22±0.13) respectively, as summarized in **(Table 2)** and presented in **(Figure 1)**.

Among the conventional sintering subgroups Dental Direkt Bio ZX^2 showed the lowest ΔE value followed by Dental Direct Bio ZW iso and Zolid Ht+: (1.78± 0.35, 2.46±0.33 and 2.82±0.08) respectively, as summarized in **(Table 2)** and presented in **(Figure 1)**.

Table (2) *Showing ΔE results of the three groups (conventional and speed sintering)*

	DD Bio ZX^2 (Group 1)		DD Bio ZW iso (Group 2)		Zolid Ht^+ (Group 3)	
	C 1	S 1	C 2	S 2	C 3	S 3
Mean ±SD	1.78 ± 0.35^a	1.20 ± 0.15	2.46 ± 0.33^b	1.70 ± 0.15^a	2.82 ± 0.08	2.22 ± 0.13^b
Test of significance	F=32.58 P<0.001*					

F: ONE Way ANOVA test **statistically significant (if p<0.05).*

The similar superscripted letters denote significant differences between groups within the same row by the post-hoc Tukey test.

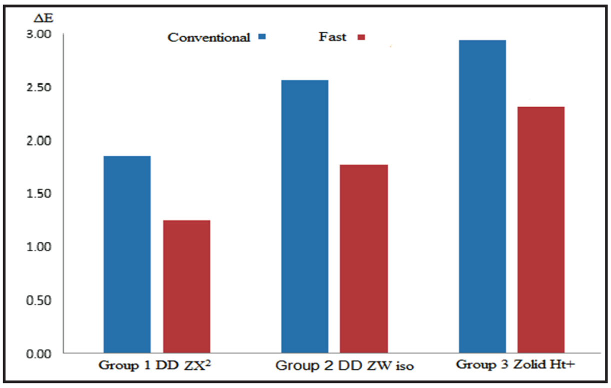

Fig. (1) Bar chart showing ΔE results of the three groups (conventional and speed sintering).

2. Shear bond strength test:

The speed sintering of the three types of zirconia discs employed in this study showed higher shear bond strength to the adhesive resin cement than that showed with the conventional sintering, as summarized in (**Table 3**) and presented in (**Figure 2**). Statistical analysis using one-way ANOVA and the post-hoc test showed statistically significant difference between studied subgroups (F=5.57 & P=0.002).

Among the speed sintering subgroups Dental Direkt Bio ZX^2 showed the highest shear bond strength value followed by Dental Direct Bio ZW iso and Zolid Ht+: (26.76± 0.96, 24.22±1.64 and 22.87±1.59) respectively, as summarized in **(Table 3)** and presented in **(Figure 2)**

Among the conventional sintering subgroups Dental Direkt Bio ZX^2 showed the highest shear bond strength value followed by Dental Direct Bio ZW iso and Zolid Ht+ (22.32±1.22, 18.86±0.95 and 17.22±0.59), as summarized in (**Table 3)** and presented in **(Figure 2).**

Table (3) *Showing shear bond strength results of the three groups (conventional and speed sintering).*

	DD Bio ZX^2 (Group 1)		DD Bio ZW iso (Group 2)		Zolid Ht+ (group 3)	
	C 1	**S 1**	**C 2**	**S 2**	**C 3**	**S 3**
Mean ±SD	22.31 ± 1.22 ^{bdh}	26.76 ± 0.96 ^{ei}	18.86 ± 0.95 ^{abc}	24.22 ± 1.64 ^{adefg}	17.22 ± 0.59 ^{cfh}	22.87± 1.59 ^{gi}
Test of significance			F=5.57 P=0.002*			

*F: One Way ANOVA test * statistically significant if p<0.05.*
The similar superscripted letters in the same row denote significant differences between groups by the Post-hoc Tukey test.

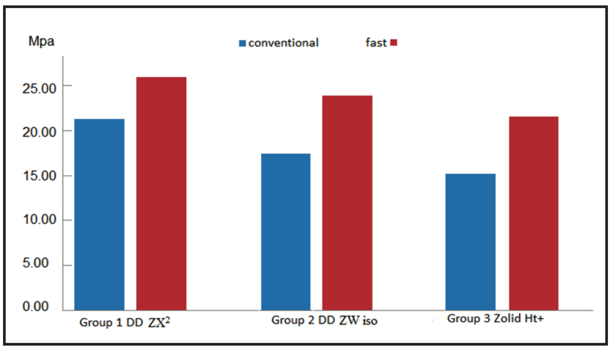

Fig. (2) Bar chart showing shear bond strength results of the three groups (conventional and Speed sintering).

3. X-ray diffraction (XRD) results:

The speed sintering of the three types of zirconia discs employed in this study showed a higher cubic phase % than that showed with the conventional sintering. The speed sintering subgroups Dental Direkt Bio ZX^2 showed the highest Cubic phase % followed by Dental Direct Bio ZW iso and Zolid Ht+ (42.1%, 38.8% and 29.7%) respectively.

The conventional sintering subgroups Dental direkt Bio ZX^2 showed the highest Cubic phase %

followed by Dental direct Bio ZW iso and Zolid Ht+ (35.3%, 31.3% and 24.3%) respectively. Y_2O_3 % in the speed sintering subgroups S1 (6.1%), S2 (5.8%) and S3 (5.2%) was higher than conventional sintering subgroups C1 (5.7%), C2 (5.5%), and C3 (5.0%). Al_2O_3% in the speed sintering subgroups S1 (0.11%), S2 (0.26%), and S3 (0.34%) was lower than conventional sintering subgroups C1 (0.15%), C2 (0.48%) and C3 (0.50%).

DISCUSSION

In this study, the results revealed that the ΔE values with the speed sintering subgroups: S1, S2 and S3 were lower than that of the conventional sintering subgroups C1, C2 and C3. The low values of ΔE with speed sintering could be due to the change in the crystal phase percentage. This explanation was detected from X-Ray Diffraction (XRD) which showed that the cubic phase percentage with the speed sintering subgroups was higher than that of the conventional sintering subgroups. The speed sintering technique increased the cubic phase percentage and therefore increased the number of the small grains size than that of the tetragonal crystals, which could be responsible for high light transmittance and translucency. This is in agreement with **Stawarczyk et al.** (7) who emphasized on the role of speed sintering in decreasing the grain size and **Zhang** (8) who emphasized on the role of smaller grain size in increasing the translucency of zirconia.

Also, X-Ray Diffraction (XRD) showed that the yttrium oxide % with the speed sintering subgroups was higher than that of the conventional sintering subgroups. In addition, Al_2O_3% with the speed sintering subgroups was lower than that of the conventional sintering subgroups. The speed sintering technique increased the yttrium and decreased the Al_2O_3% and therefore could be responsible for increasing the translucency, this was in agreement with **Kim et al.** (9) who reported that the percentage of cubic phase increases with increasing percentage of Y_2O_3. Also, **Carrabba et al.** (10) who reported that the increases of Y_2O_3 and decrease of Al_2O_3 content strongly improved the optical properties of zirconia. This could allow less absorption and reflection of the light passing through, while most of the light transmitted to the underlying translucent cement and subsequently to the underneath sample.

The translucency of the zirconia is determined by multiple combined parameters such as grain size, yttrium content, aluminum oxide content and the degree of sintering which supposed to be responsible for the transformation of the zirconia from the tetragonal to the cubic phase that is supposed to turn zirconia to be more translucent This explanation could be in agreement with **Heffernan et al** (11) who reported that the amount of absorbed, reflected and transmitted light would depend on the percentage and size of crystalline structure.

Furthermore, **Kaizer et al.** (4) reported that the speed sintering improved the optical properties of monolithic translucent zirconia. This could explain why all the samples in the speed sintering groups gave ΔE values lower than that of the conventional sintering groups.

The results seem to be in agreement with **Kim et al.** (12) who concluded that in order to obtain more translucent dental zirconia restorations, shorter sintering times should be considered. Also, the results revealed that the ΔE values in Group 1 (ZX^2) were lower than that in Group 2 (ZW) and Group 3 (HT+). According to X-Ray Diffraction (XRD) the yttrium oxide % in Group 1 was higher than that in Group 2 and Group 3. In addition, Al_2O_3% in the Group 1 was lower than that in the Group 2 and Group 3. The higher content of yttrium oxide % and lower content of Al_2O_3% could be highly responsible for increasing the translucency.

In this study, the results revealed that the shear bond strength values with the speed sintering subgroups S1, S2 and S3 were higher than that of the conventional sintering subgroups C1, C2 and C3. The high values of shear bond strength with the speed sintering could be due to the change in the cubic phase percentage. This explanation was detected from X-Ray Diffraction (XRD) scan results revealed that the cubic phase percentage with the speed sintering subgroups was higher than that of the conventional sintering subgroups. These scientific findings could be held together to be responsible for increasing the number of small grains size due to cubic crystals which are smaller than tetragonal and therefore increased the number of densities of grain-boundaries, which might increase the surface area for bonding of the cement and subsequently increased the degree of bonding between the sample and the adhesive molecules in PANAVIA SA resin cement. This could be in agreement with **Jeon et al** [13] who proved that the speed sintering technique reduce the grain size and enhance the degree of bonding between resin cement and a dental zirconia due to the increase of interfacial free energy.

Also, X-Ray Diffraction (XRD) showed that the yttrium oxide % with the speed sintering subgroups was higher than that of the conventional sintering subgroups. In addition, Al_2O_3% with the speed sintering subgroups was lower than that of the conventional sintering subgroups. Inspired of the fact that the Al_2O_3 content was decreased however the yttrium oxide content was increased with speed sintering. The higher yttrium oxide content could be held responsible for forming a stable chemical bond with the phosphate ester group of the adhesive monomer (MDP) present in PANAVIA. This scientific assumption could be in agreement with **Cavalcanti et al.** [14] who emphasized on the role of oxides content in zirconia in forming a durable bond with MDP of PANAVIA.

Also, the results revealed that the shear bond strength values in the Group 1 (ZX^2) were higher than the Group 2 (ZW) and Group 3 (HT+). According to X-Ray Diffraction (XRD) the yttrium in the Group 1 was higher than in Group 2 and Group 3. The higher content of yttrium oxide content could increase the shear bond strength.

The current study showed some limitations, among which was the absence of factors simulating the oral environmental conditions, consequently further investigations might be recommended to assess the effect of the sintering technique after aging. Another limitation was the absence of a mode of failure test which is important to know the type of failure. Also, recommend that more studies would be carried out testing the speed sintering technique on shear bond strength to resin cement of monolithic translucent zirconia.

CONCLUSION

Under the conditions of this in-vitro study the following could be concluded that the speed sintering technique of the monolithic translucent zirconia gave better color shade reproduction and higher shear bond strength to resin cement than that of the conventional sintering technique. Dental Direkt ZX^2 produced better color shade reproduction than that of the Dental Direkt ZW iso and Zolid Ht+ in speed and conventional sintering technique. Dental Direkt ZX^2 produced higher shear bond strength to resin cement than that of the Dental Direkt ZW iso and Zolid Ht+ in speed and conventional sintering technique.

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