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THE EFFECT OF TWO SINTERING CYCLES AND AGING ON BIAXIAL FLEXURAL STRENGTH OF STRENGTH GRADIENT MULTI-LAYERED ZIRCONIA (IN VITRO STUDY)

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ABSTRACT

Objective: Impacts of thermocycling aging and variation in sintering parameters using conventional and speed sintering cycles on biaxial flexural strength (BFS) of IPS e.max ZirCAD Prime and Katana Ultra-Translucent zirconia to be used as a veneer material and comparing them to IPS e.max CAD as a control group.

Materials and Methods: Strength gradient multilayered zirconia (IPS e.max ZirCAD Prime) and color gradient multilayered zirconia (Katana UTML) were investigated to be compared to lithium disilicate (IPS e.max CAD). Zirconia Specimens were sintered following speed and conventional sintering protocols. 0.5 mm circular shaped sample of IPS e.max CAD was obtained using a precision sawing machine. Dimensions of the zirconia specimens after sintering were 0.5 mm. it was designed and milled with 20%-25% oversize in order to compensate for the shrinkage that occurs after sintering. Thermocycling was applied for 5000 cycles to simulate 6 months in the oral environment, A universal testing machine was used to measure biaxial flexural strength.

Results: In normal cycle, BFS had the highest value in IPS e.max ZirCAD Prime, followed by IPS e.max CAD, while the lowest value was found in Katana UTML Zirconia. In speed cycle, IPS e.max ZirCAD Prime had the highest value, followed by Katana UTML Zirconia, while IPS e.max CAD was the lowest. In both zirconia groups, speed cycle had a higher value than normal cycle, however, the difference wasn't significant.

Conclusion: Biaxial flexural strength wasn't affected by both sintering cycles after thermocycling aging for both Zirconia groups.

KEYWORDS: Sintering cycles, biaxial flexural strength, thermocycling aging, Gradient Zirconia.

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INTRODUCTION

The usage of dental ceramics and zirconia is widely increasing with superior results for different restorations. Zirconia, which is a polymorphic Ceramic, could be found in 3 different crystalline forms, which are; monoclinic, tetragonal and cubic, and that depends on the temperature. The monoclinic phase is stable at room temperature; however, its mechanical and optical properties are not suitable for clinical use. On another hand, stabilizing oxides are used to stabilize tetragonal and cubic phases at room temperature [(1)].

Yttrium oxide is known to be the most used stabilizer[(2,3)]; Where yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) has been reported to have the most pronounced phase transformation toughening. The yttria content within zirconia has a direct effect on mechanical and optical properties. Generally, increasing the amount of yttria content within zirconia enhances its optical properties while decreases its mechanical properties[(4,5)]. Zirconia stabilized with 3 mol% is the first introduced yttria-stabilized tetragonal zirconia polycrystalline[(6,7)] that has very high mechanical properties[(7)] and extraordinary biocompatibility[(8)].

The high opacity and white color is the main drawbacks of 3 mol%. That was the reason for which it has only been used as a core and to be veneered with highly translucent ceramics. Chipping of the veneering ceramic is still a standing problem[(9,10)]. Meanwhile, due to its resistance to chipping and tooth conservation during reduction[(3,11)] monolithic zirconia has gained a great attention.

Throughout the past years, several modifications have been added, to enable zirconia to be used as a monolithic restoration with adequate esthetics[(12)]. The modifications included the following; increasing the yttria content from 3 mol% to 4 mol% and 5 mol% to gain higher translucency, in addition to introducing the poly-chromatic (multi-layered) zirconia.

The multi-layered technology has shown an improvement in color of zirconia to mimic the natural teeth without affecting its mechanical properties, by only Using pigmentations to provide shade gradient as natural teeth, while having the same generation in each blank[(13,14)]. A New multi-layered technology has been introduced in recent days; Where it merges 2 different zirconia generations in one blank, in order to get the advantages of both generations. For better translucency, this merge occurs between 3Y-TZP in the dentin/body area for better mechanical properties, and 5Y-TZP in the incisal area for superior esthetics.

Monolithic 3Y-TZP showed in several studies, a wear behavior similar or slightly higher than natural teeth[(15,16)]. With regard to the mechanical properties, 5Y-TZP has showed better mechanical properties than that of lithium disilicate ceramics, despite the fact that it has shown the least flexural strength among zirconia generations [(17,18)].

With the evolution of ceramics and the increased usage of single visit chairside restorations, a major drawback of conventional zirconia has showed up, which is the long-time sintering process. Using Conventional Sintering Process needs at least 2 appointments for delivering the restoration, this happens due to the fact that the process of sintering Y-TZP is time consuming[(19,20)]. High-speed sintering was able to make the overall time for sintering less than 30 minutes; this has been used to overcome this problem by using special furnaces, which allowed zirconia to be a suitable material for single visit restorations[(21)].

The changes of the properties of zirconia after speed sintering have to be investigated intensively, as it has been claimed that changing the sintering parameters have several effects on the properties of the zirconia such as flexural strength[(22,23)]

The newest generation of zirconia combines both 3Y-TZP and 5Y-TZP in one blank, it is labeled as a universal zirconia that is used in different clinical situations from veneer to anterior single crowns to full-arch restorations. This research is done to study the usage of gradient zirconia as a veneer and comparing its properties to lithium disilicate.

MATERIALS AND METHODS

Sample size calculation: A power analysis was designed to have adequate power to apply a statistical test of the null hypothesis that there is no difference between tested groups regarding biaxial flexural strength. By adopting an alpha (α) level of 0.05 (5%), a beta (β) level of (0.05) (i.e. power=95%) and an effect size (f) of (0.943) calculated based on the results of a previous study[24]; the predicted total sample size (n) was found to be (30) samples. Sample size was increased by (15%) to compensate for possible procedural errors during testing to be (35) samples. Sample size calculation was done using G*Power version 3.1.9.7[25].

In this in vitro study, A total of 35 samples were constructed as follow; Group A: 7 discs of IPS e.max CAD (Control group), Group B: 14 discs of Katana Ultra translucent zirconia. Group C: 14 discs of IPS e.max ZirCAD Prime. Group B and C were subdivided according to the sintering cycles (standard cycle and speed cycle). Group (B1),(C1): Normal cycle , Group (B2), (C2): Speed cycle

Study Design: in vitro comparative study

Procedure methodology

A. IPS e.max CAD samples preparation:

A 3D cylinder shaped IPS e.max CAD of 12x10 mm dimensions was designed using windows 3D builder software to produce STL file which was transferred to CAM software (inLab CAM SW 18.5). By using inLab MCXL milling machine, the block was milled into circular cylinder with 12 mm diameter and 10 mm length. After fabrication of the cylinder, it was attached to its holder to be held during sawing.

IPS e.max CAD blocks were placed in a precision sawing machine. Then the blocks were sawed to obtain circular shaped samples of 0.5 mm. The samples were cut while using a coolant delivery system. The thickness of the blade was 0.7 mm, with a constant rate of cutting of 16.7 mm/min. The blade was of 2500 rpm in 50 rpm increments. The thickness of the samples was checked after sawing. After that, crystallization of IPS e.max CAD samples were done following the manufacturer's instructions using Programat P310 furnace. The samples were placed into the furnace then the program was started. The holding time was 10 minutes at 840 ◦C. All samples were adjusted to the definitive dimension, a thickness of 0.5mm. All samples were finished and polished with Diasynt plus - diapro eve system, following the manufacturer`s instructions. A perfect high gloss was achieved. The samples thicknesses were re-checked by a digital caliper, where the samples with improper thickness were discarded. Finally, Glazing of IPS e.max CAD samples were done following the manufacturer's instructions. IPS e.max CAD Crystall./Glaze Paste/FLUO was mixed and applied on the surface of the sample using small brush by the same technician for standardization. After that glazing was conducted in a compatible ceramic furnace. Ivoclar Vivadent, Programat® P310 furnace.

B. Katana ultra-translucent zirconia and IPS e.max ZirCAD Prime samples preparation

Both zirconia groups were following the same steps:

Zirconia blocks of 15 mm diameter was accurately designed using a digital 3D builder software as follow: A cylinder dimension of 15 mm diameter was designed. The designed cylinder was then saved as STL file, then exported to MillBox DGSHAPE software for fine editing using a free form tool. The cylinder shape was accurately confirmed, saved and exported to the CAM software system.

The blank was inserted into Roland DWX-51D milling machine, then it was milled according to the designed cylinder dimensions. A cylinder was milled from the blank with an increase in size of 20%-25%. Each blank has a barcode with an enlargement factor used to exactly calculate the oversize needed to compensate for the shrinkage that occurs after sintering of the samples.

Zirconia discs were machined from their respective cylinder by using IsoMetTM 4000 saw as being used with IPS e.max CAD, with an oversize in thickness of 20%-25% (0.62 mm) to compensate for the shrinkage that occurs after sintering of zirconia samples to reach a uniform standard thickness of (0.5mm). Thickness of the samples was verified using a digital caliper

Conventional Sintering of Katana UTML discs: 7 discs were put into a sintering boat made of pure alumina and a 2mm single layer of sintering beads was placed below the discs. After that the discs were put into TABEO-1/M/ZIRKON-100 mihmvogt sintering furnace and sintered following the recommended sintering temperatures and times. The recommended sintering parameters for Katana UTML is 1550 ºC and holding time is for 2 hours, rate of temperature increases 10ºC/min. After sintering, thicknesses were verified using a digital caliper.

Conventional sintering cycle of IPS e.max ZirCAD Prime: 7 discs were sintered according to the recommended sintering parameters of IPS e.max ZirCAD Prime is 1500ºC for 2 hours, rate of temperature increases 10 C/min, holding time: 2.5 hours, and cooling rate: -10 C/min. After sintering, thicknesses were re measured using a digital caliper.

Speed sintering cycle of Katana UTML: 7 discs were sintered following the recommended speed sintering parameters; 1560ºC, holding time 30 mins, and rate of temperature increase is 35ºC/min, using TABEO-1/M/ZIRKON-100 mihmvogt furnace.

Speed sintering cycle of IPS e.max ZirCAD Prime: 7 discs were sintered following the recommended speed sintering parameters for 2h and 26 min with 1530ºC for 1-hour, heating rate 60ºC/min and cooling rate -6 C/min.

All the samples were finished and polished minimally using water coolant, polishing was done using Eve Rotary Grinding & Polishing instruments (Diasynt plus & Diacera zirkonoxid zirconia Eve). All samples were cleaned ultrasonically for 3 minutes in a solution of 99% isopropanol then dried with the Robocam drying unit to get rid of any contamination.

Glazing of Zirconia samples were done following the manufacturer's instructions. Ceramotion paste glaze was mixed and applied on one surface of the samples using small brush. After that glazing was conducted in a compatible ceramic furnace. Ivoclar Vivadent, Programat® P310 furnace.

Thermocycling

The samples were stored for 5 days at 37ºC. They were applied into two chambers one with cold water bath, immersed for 30 seconds at temperature of 5ºC and the other with hot water bath, immersed for 30 seconds at temperature 55ºC. Thermocycling was applied for 5000 cycles for dwell time of 10 seconds by automatic thermocycling machine. THE-1100 thermocycler* stimulates artificial aging by means of cyclical temperature changes. All parameters can be easily adjusted such as temperatures, exposure time and number of cycles.

Biaxial flexural strength testing

The biaxial flexural strength (BFS) of all samples was measured using the following samples: 7 discs of each group (n=7) were tested for flexural strength after thermocycling aging. Measurements was done withthe piston-on-three ball technique using an Instron testing machine** model 3345

* THE-1100, SD MECHATRONIK GMBH, feldkirchen-westerham. Germany

** Instron testing machine model 3345, England, United Kingdom

England according to the ISO 6872 specifications for testing ceramic materials and recording data was done using computer software BlueHill universal Instron England version 3.3. A 3mm diameter steel balls that were equidistant were rested above a 12mm diameter metallic platform. samples were placed on the steel balls and load was applied on it by a piston of 1.5mm diameter and 0.5mm/min crosshead speed.

Statistical analysis

Numerical data were explored for normality by checking the data distribution using Shapiro-Wilk tests. Data showed parametric distribution so; they were represented by mean and standard deviation (SD) values. Comparisons of independent variables were done using one-way ANOVA followed by Tukey's post hoc test. P-values were adjusted for multiple comparisons utilizing Bonferroni correction. The significance level was set at $p \leq 0.05$ within all tests. Statistical analysis was performed with R statistical analysis software version 4.1.3 for Windows[26].

RESULTS

Biaxial flexural strength (MPa)

A) Effect of material with each sintering cycle:

In normal cycle; a significant difference was found between different groups (p=0.011). IPS e.max ZirCAD Prime (1060.10±455.57) had the highest value, followed by IPS e.max CAD (551.55±101.71), while the lowest value was found in Katana UTML Zirconia (459.67±130.40). Post hoc pairwise comparisons showed IPS e.max ZirCAD Prime to have a significantly higher value than other groups (p<0.001). while in speed cycle a significant difference was found between different groups (p<0.001). The highest value was found in IPS e.max ZirCAD Prime (1144.42±143.56), followed by Katana UTML Zirconia (573.58±234.01), while the lowest value was found in IPS e.max CAD (551.55±101.71). Post hoc pairwise comparisons showed IPS e.max ZirCAD Prime to have a significantly higher value than other groups (p<0.001).

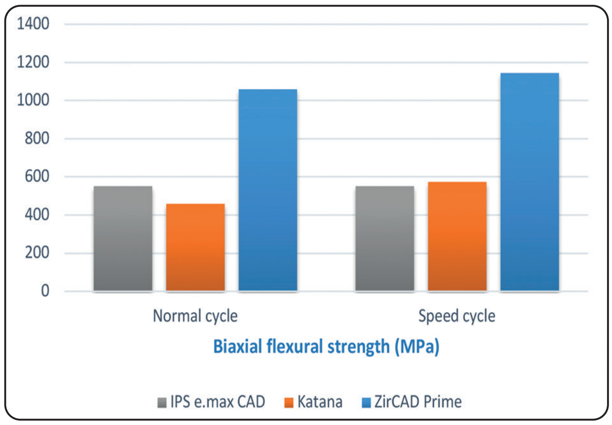

Fig. (1): Bar chart showing average biaxial flexural strength (MPa) for different materials within each sintering cycle

B-Effect of sintering cycle within each material:

In Katana UTML Zirconia: Speed cycle (573.58±234.01) had a higher value than normal cycle (459.67±130.40) but there was no statistically significant difference (p=0.370). while in IPS e.max ZirCAD Prime: Speed cycle (1144.42±143.56) had a higher value than normal cycle (1060.10±455.57) but the difference was not statistically significant (p=0.703).

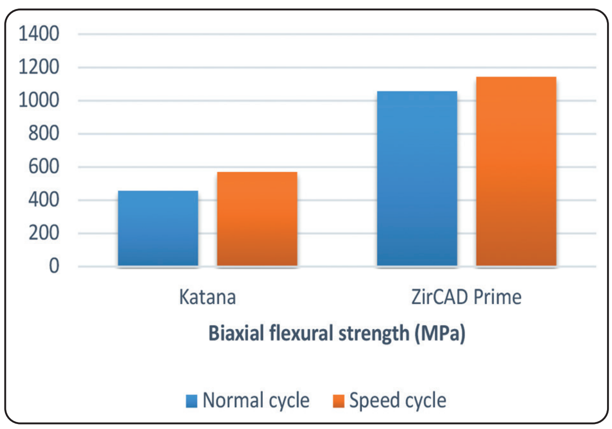

Fig. (2): Bar chart showing average biaxial flexural strength (MPa) for different sintering cycles within each material

DISCUSSION

Nowadays, all-ceramic restorations have provided superior esthetics and biocompatibility for dental restorations. Ceramic materials are recently introduced with improved properties that made it used widely in esthetic dentistry as lithium disilicate and zirconia.

Zirconia restorations are now used in long-span restorations and this is as a result of their superior mechanical properties. Zirconia restorations main challenges was their opacity and the chipping of the porcelain that masks the opacity of the zirconia core. In order to solve this problem, monolithic and cubic zirconia are introduced to construct full-contour zirconia restorations without the need of using any veneering porcelain [27,28].

The latest zirconia generation is made of tetragonal and cubic zirconia which has superior optical properties in comparison to conventional and monolithic zirconia. As cubic zirconia translucency is comparable to lithium disilicate. yet, its mechanical properties are not to be compared to the past zirconia generations[29].

Two different Zirconia materials were used in this study; strength gradient multilayered zirconia (IPS e.max ZirCAD Prime) and color gradient multilayered zirconia (Katana UTML) with 0.5 mm thickness as a suggestion to be used as a veneer material and it was compared to IPS e.max CAD as a control group, as lithium disilicate is the gold standard regarding veneers restorations.

A drawback of conventional zirconia is the long time of sintering cycles, Recently, high-speed sintering is used to overcome this problem by using special furnaces, which allowed zirconia to be a suitable material for single visit restorations

The true comparative values of biaxial flexural strength of the available cubic zirconia brands remain in question and more clinical data are still needed for the long-term survival of new-formulation, zirconia-based restorations.

That's why our main concern was to study the effect of thermocycling aging and 2 different sintering cycles on biaxial flexural strength of two types of zirconia and compare them to lithium disilicate.

In this in vitro study, in order to compare the different properties among available zirconia products, two of commercially available zirconia were selected Katana UTML and IPS e.max ZirCAD Prime with the shade A2 in order to compare their different properties with IPS e.max CAD as a control group.

E.max cylinders of 12x10 mm dimensions was designed using digital 3D builder software system, Then the block was milled into a circular cylinder with 12 mm diameter and 10 mm length by using inLab MCXL milling machine, IPS e.max CAD blocks were placed in a precision sawing machine, Then the blocks were sawed to obtain circular shaped samples of 0.5 mm. After that, the IPS e.max CAD samples were crystallized & glazed using a Programat P310 furnace [30].

Zirconia cylinders of 15 mm diameter was designed using digital 3D builder software system. The blank was milled into a cylinder with 20%-25% increase in size to compensate for the shrinkage that occurs after sintering to reach two uniform standard thicknesses of (0.5mm) which were selected in our study. 0.5 mm thickness was used to simulate the average thickness of the veneer. Thicknesses before and after sintering were verified using a digital caliper.

Samples were divided into normal and speed sintering cycles to study the effect of two sintering cycles on the optical and mechanical properties of zirconia.

Finishing and polishing were done minimally in order to not affect the microstructure of the samples. Rough surfaces was avoided as roughness facilitates water sorption[31]. A dental surveyor was used to standardize minimal finishing and polishing for all samples[32].

Ultrasonic cleaning using 99% isopropanol for 3 mins followed by dryness of samples was also done for cleaning purposes, and removal of any contamination to avoid any negative effect on our results[33].

Thermal aging is a naturally occurring phenomenon. As aging occurs to all the ceramic materials that we use for dental restorations, and this aging affects their optical and mechanical performances, it was done to simulate the oral environment, Aging was done by thermocycling technique using a thermocycling device. A total of 5000 thermal cycles were done, which simulates 6 months of in vivo function. Samples were immersed for 30 seconds in a temperature between 50ºC and 55ºC with 10 seconds dwell time and 5 seconds transfer time[34].

Biaxial flexural strength results showed that in speed cycles; the difference between the groups were significant, the highest value BFS was found in IPS e.max ZirCAD Prime, after that comes Katana, while the least values were found in e.max, While in normal cycle the highest value was also found in IPS e.max ZirCAD prime followed by IPS e.max and Katana UTML with insignificant difference between the 2 groups.

IPS e.max ZirCAD Prime flexural strength values may be explained due to that it contains both 3Y-TZP and 5Y-TZP, where 3Y-TZP core has high content of tetragonal crystals in comparison to 5Y-TZP in Katana UTML, this leads to the increase in its flexural strength [35].

The grain size of zirconia strongly affects its mechanical behavior and its ability to undergo transformation toughening, in Katana UTML zirconia, it has high yttria percentage in order to stabilize cubic phase at room temperature; that's why, cubic zirconia is not undergoing transformation toughening. this means that its more liable to mechanical damage, while in case of lithium disilicate, its microstructure consists of small randomly oriented interlocking crystals. This greatly increases its strength as the needle-like crystals cause cracks deflection, and arrest the crack propagation through the material. giving an increase in its flexural strength. This may be the explanation to the in significant difference between IPS e.max CAD and Katana Ultra-translucent zirconia in normal cycle.

Our results were in accordance with a study performed by Eduardo M. Murioz et al. (2017) [36] where AMZ (anterior cubic monolithic zirconia) had lower biaxial flexural strength values in comparison with PMZ (posterior non-cubic monolithic zirconia) which was affected by mechanical and hydrothermal aging. Our results were also similar to Jihad G. Hamed et al.[37] who stated that Katana UTML group had significantly higher biaxial flexural strength values than those in IPS e.max Press Multi group.

As for the effect of sintering cycles, for both katana and ZirCAD prime, Speed cycle had higher values than normal cycle yet the difference wasn't statistically significant. In accordance with Jenni Hjerppe et al. (2009)[38] he revealed that after using different holding and rising time in sintering process of zirconia, the reduced time of sintering had no effect on the biaxial flexural strength. Ebied et al. (2014), [24] also concluded that according to the applied sintering parameters, it didn't affect the biaxial flexural strength of the material used.

CONCLUSION

Both sintering cycles did not affect the biaxial flexural strength for both UTML and ZirCAD Prime

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