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THE EFFECT OF TWO SINTERING CYCLES AND AGING ON OPTICAL PROPERTIES OF GRADIENT MULTI-LAYERED ZIRCONIA (IN VITRO STUDY)

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ABSTRACT

Objective: This study aims to assess the effects of thermocycling aging and changes in sintering parameters, employing both conventional and speed sintering cycles, on the optical properties of two types of strength gradient multilayered zirconia.

Materials and Methods: The study examined the strength gradient multilayered zirconia; IPS e.max ZirCAD Prime (Ivoclar, Vivadent, USA) and Katana UTML (Kuraray Noritake Dental Inc), comparing them to lithium disilicate IPS e.max CAD (Ivoclar, Vivadent, USA). Thirty five disc shaped samples of 12 mm diameter and 0.5 mm thickness were constructed. Half of the zirconia samples were constructed using conventional sintering cycle, and half were constructed using speed sintering cycle. Thermocycling was performed for 5000 cycles to simulate six months in the oral environment. Color parameters and translucency were measured using a spectrophotometer device before and after thermocycling.

Results: Significant differences were observed among the various groups both before and after aging in both conventional and speed cycles. The highest translucency values were recorded for IPS e.max CAD, followed by Katana, with the lowest values observed in IPS e.max Zircad Prime. The conventional cycle had a higher value than speed cycle both before and after aging. The values measured before aging was significantly higher than the value measured after aging in IPS e.max CAD group while in both zirconia groups, values measured after aging were higher than the values measured before aging in conventional and speed cycles. As for color change, there was a significant difference between different groups. The highest value of color change was found in Katana UTML Zirconia, followed by IPS e.max ZirCAD Prime, while the lowest value was found in IPS e.max CAD in both conventional and speed cycles. Speed sintering cycle had a higher value of color change than conventional sintering cycle.

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Conclusion: In both types of translucent zirconia investigated, the conventional sintering cycle exhibited significantly higher translucency values compared to the speed cycle. Additionally, the speed cycle resulted in a greater color change than the conventional cycle in Katana UTML. Thermocycling aging led to an increase in translucency for Katana Ultra Translucent and IPS e.max ZirCAD Prime, while it decreased the translucency of IPS e.max CAD.

KEYWORDS: Gradient zirconia, optical properties, translucency, color stability

INTRODUCTION

Dental ceramics and zirconia are increasingly used for various dental restorations, offering great results. Zirconia, a versatile ceramic, comes in three forms; monoclinic, tetragonal, and cubic depending on temperature. While the monoclinic phase is stable at room temperature, it lacks suitable mechanical and optical properties for clinical use. However, stabilizing oxides can make the tetragonal and cubic phases stable at room temperature, making them more suitable for dental applications (1).

Yttrium oxide is commonly used as a stabilizer (2,3), particularly in yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP), known for its significant phase transformation toughening. The quantity of yttria in zirconia directly impacts its mechanical and optical characteristics. Typically, increasing yttria content in zirconia improves its optical qualities but reduces its mechanical strength (4,5). Zirconia stabilized with 3 mol% yttria was the first Y-TZP introduced (6,7), boasting high mechanical strength(7) and excellent biocompatibility(8).

The main drawback of 3 mol% zirconia is its high opacity and white color, which limits its use to being a core material veneered with highly translucent ceramics. However, chipping of the veneering ceramic remains a persistent issue (9,10). Conversely, monolithic zirconia has gained significant attention due to its resistance to chipping and preservation of tooth structure during reduction (3,11). Monolithic 3Y-TZP, as indicated by various studies, exhibits wear behavior similar to or slightly higher than natural teeth(12,13). Regarding mechanical properties, 5Y-TZP demonstrates superior characteristics compared to lithium disilicate ceramics, despite having the lowest flexural strength among zirconia generations(14,15).

Over the years, zirconia has undergone various modifications to enable its use as a monolithic restoration with improved esthetics (16). These modifications include increasing the yttria content from 3 mol% to 4 mol% and 5 mol% to enhance translucency, as well as introducing poly-chromatic (multi-layered) zirconia.

The multi-layered technology enhances the color of zirconia to mimic natural teeth without compromising its mechanical properties. By utilizing pigmentations to create shade gradients similar to natural teeth, each blank maintains uniformity(17,18). Recently, a new multilayered technology has emerged, combining two different zirconia generations in one blank to leverage the benefits of both. This merger occurs between 3Y-TZP in the dentin/body area for improved mechanical properties and 5Y-TZP in the incisal area for superior esthetics and translucency. This 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.

With the advancement of ceramics and the growing adoption of single-visit chairside restorations, a significant drawback of conventional zirconia has emerged which is the lengthy sintering process. Conventional sintering processes require at least two appointments for restoration delivery due to the time-consuming nature of sintering Y-TZP (19,20). High-speed sintering has addressed this issue by reducing the overall sintering time to less than

30 minutes, facilitated by special furnaces, making zirconia suitable for single-visit restorations(21).

The effects of speed sintering on zirconia properties, particularly optical properties, need thorough investigation, as altering sintering parameters can impact zirconia properties significantly (22,23). Therefore, this research is done to study the usage of gradient zirconia as a veneer and comparing its properties to lithium disilicate.

MATERIALS AND METHODS

The calculation for sample size was conducted using G*Power version 3.1.9.7 to ensure adequate statistical power for testing the null hypothesis of no difference between groups (24). With an alpha (α) level of 0.05, a beta (β) level of 0.05 (power = 95%), and an effect size (f) of 0.943 derived from a previous study (25), the anticipated total sample size (n) was determined to be 30 samples. To accommodate potential procedural errors during testing, the sample size was increased by 15% to reach 35 samples.

The samples were prepared as follows; Group A: seven IPS e.max CAD disc (control group), Group B: fourteen Katana Ultra translucent zirconia discs, and Group C: fourteen IPS e.max ZirCAD Prime discs. Groups B and C were further divided based on sintering cycles into conventional cycle (B1, C1) and speed cycle (B2, C2).

Study Design: in vitro comparative study

Procedure methodology:

Samples preparation:

IPS e.max CAD samples: A 3D cylinder-shaped IPS e.max CAD measuring 12x10 mm was designed using Windows 3D Builder software to generate an STL file, which was then transferred to CAM software (inLab CAM SW 18.5). The block was subsequently milled into a circular cylinder with a diameter of 12 mm and a length of 10 mm using the inLab MCXL milling machine. After fabrication, the cylinder was affixed to its holder for stability during sawing. IPS e.max CAD blocks were placed in a precision sawing machine IsoMet™ 4000 (Buehler, Dusseldorf, Germany) and sawed to obtain circular samples with a thickness of 0.5 mm. A coolant delivery system was used during cutting, with a blade thickness of 0.7 mm and a cutting rate of 16.7 mm/min. The blade operated at 2500 rpm in 50 rpm increments. The thickness of the samples was verified post-sawing. Subsequently, crystallization of IPS e.max CAD samples was performed according to the manufacturer's instructions using a Programat P310 furnace. The samples were placed in the furnace, and the program was initiated with a holding time of 10 minutes at 840 °C. All samples were adjusted to a final thickness of 0.5 mm. Following manufacturer's instructions, all samples were finished and polished using the Diasynt plus-diapro eve system to achieve a perfect high gloss. Sample thicknesses were re-checked using a digital caliper, and any samples with improper thickness were discarded. Finally, glazing of IPS e.max CAD samples was conducted according to the manufacturer's instructions using IPS e.max CAD Crystall/Glaze Paste/FLUO.

Zirconia samples: Zirconia blocks with a diameter of 15 mm were precisely designed using digital 3D builder software. Initially, a cylinder with a 15 mm diameter was designed and saved as an STL file, then fine editing was performed using MillBox DGSHAPE software with a freeform tool. After confirming the accuracy of the cylinder shape, it was saved and exported to the CAM software system. The blank was then placed into a Roland DWX-51D milling machine and milled according to the dimensions of the designed cylinder, with an increase in size of 20%-25%. Each blank was equipped with a barcode indicating the enlargement factor used to precisely calculate the oversize needed to compensate for shrinkage post-sintering. Zirconia discs were machined from their respective cylinders using an IsoMetTM 4000 saw, with an oversize in thickness of 20%-25% (0.62 mm) to achieve a uniform standard thickness of 0.5 mm after sintering. The thickness of the samples was verified using a digital caliper.

For the conventional sintering of Katana UTML discs, the samples were sintered in a TABEO-1/M/ZIRKON-100 mihmvogt sintering furnace according to the recommended sintering temperatures and times (1550ºC for 2 hours, with a rate of temperature increase of 10ºC/min). After sintering, thicknesses were verified using a digital caliper. Similarly, for the conventional sintering cycle of IPS e.max ZirCAD Prime, the samples were sintered according to the recommended parameters (1500ºC for 2 hours, with a rate of temperature increase of 10ºC/min, holding time of 2.5 hours, and cooling rate of 10ºC/min). After sintering, thicknesses were remeasured using a digital caliper.

For the speed sintering cycle of Katana UTML, the samples were sintered according to the recommended parameters: 1560ºC, holding time of 30 minutes, and a rate of temperature increase of 35ºC/min using the TABEO-1/M/ZIRKON-100 mihmvogt furnace. In the speed sintering cycle of IPS e.max ZirCAD Prime, the samples were sintered according to the recommended parameters: 1530ºC for 1 hour, with a heating rate of 60ºC/min and a cooling rate of 6ºC/min, totaling 2 hours and 26 minutes.

All samples underwent minimal finishing and polishing using water coolant, with polishing performed using Eve Rotary Grinding & Polishing instruments (Diasynt plus & Diacera zirkonoxid zirconia Eve). Subsequently, all samples were cleaned ultrasonically for 3 minutes in a solution of 99% isopropanol and dried with the Robocam drying unit to eliminate any contamination.

Glazing of the zirconia samples was carried out according to the manufacturer's instructions. Ceramotion paste glaze was mixed and applied to one surface of the samples using a small brush, followed by glazing in a compatible ceramic furnace, specifically the Ivoclar Vivadent Programat® P310 furnace.

Thermocycling

The samples were stored for 5 days at 37ºC. They were then placed into two chambers: one with a cold water bath, where they were immersed for 30 seconds at a temperature of 5ºC, and the other with a hot water bath, where they were immersed for 30 seconds at a temperature of 55ºC. Thermocycling was conducted for 5000 cycles, with a dwell time of 10 seconds, using an automatic thermocycling machine, specifically the THE-1100 thermocycler. This machine simulates artificial aging through cyclical temperature changes, with adjustable parameters such as temperatures, exposure time, and number of cycles.

Color stability and translucency testing

The CIE-Lab color parameters and translucency of all samples was measured using spectrophotometer (Agilent Cary 5000 spectrophotometer, Santa Clara, United States) before and after the thermocycling process.

Translucency parameter (TP) values have been identified through the assessment of color variances between readings taken against white and black backgrounds using the equation: $TP = \sqrt{((L1-L2)^2 + (a1-a2)^2 + (b1-b2)^2)}$, where L1, a1, b1 denote the initial color values of each sample, and L2, a2, b2 represent the color values after experimentation. Subsequently, the change in TP before and after thermocycling was determined. Color differences (ΔE) were computed using the formula: $\Delta E = ([L1-L2]^2 + [a1-a2]^2 + [b1-b2]^2)^\wedge 1/2$. Thus, the extent of color alteration (ΔE) is established by comparing the color of baseline samples with that of samples post-thermocycling.

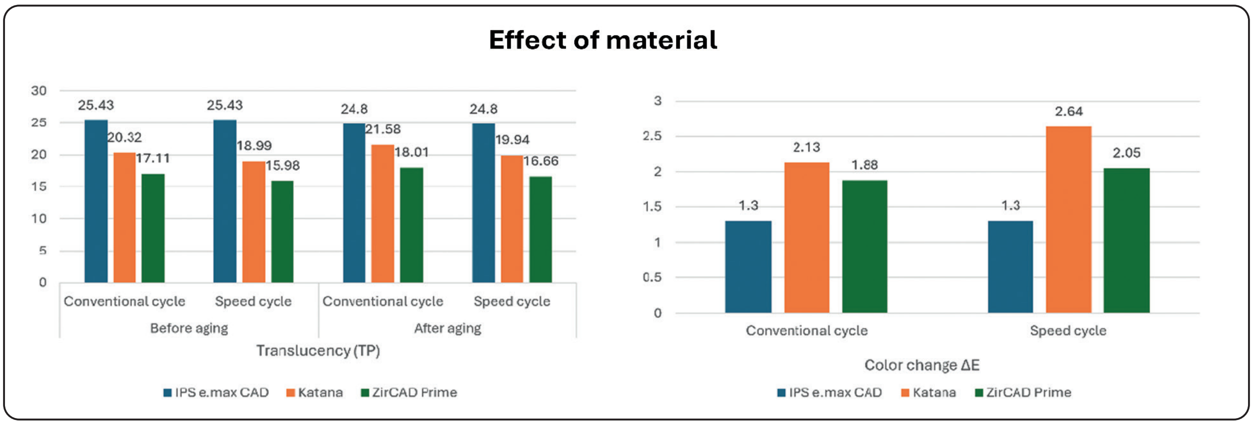
Statistical analysis

The normality of the numerical data was assessed using Shapiro-Wilk tests, indicating a parametric distribution. Mean and standard deviation (SD) values were utilized to represent the data. Independent variable comparisons were conducted via one-way ANOVA, followed by Tukey's post hoc test. P-values were adjusted for multiple comparisons using Bonferroni correction, with a significance level set at $P \leq 0.05$ for all tests. Statistical analysis was carried out using R statistical analysis software version 4.1.3 for Windows.(26)

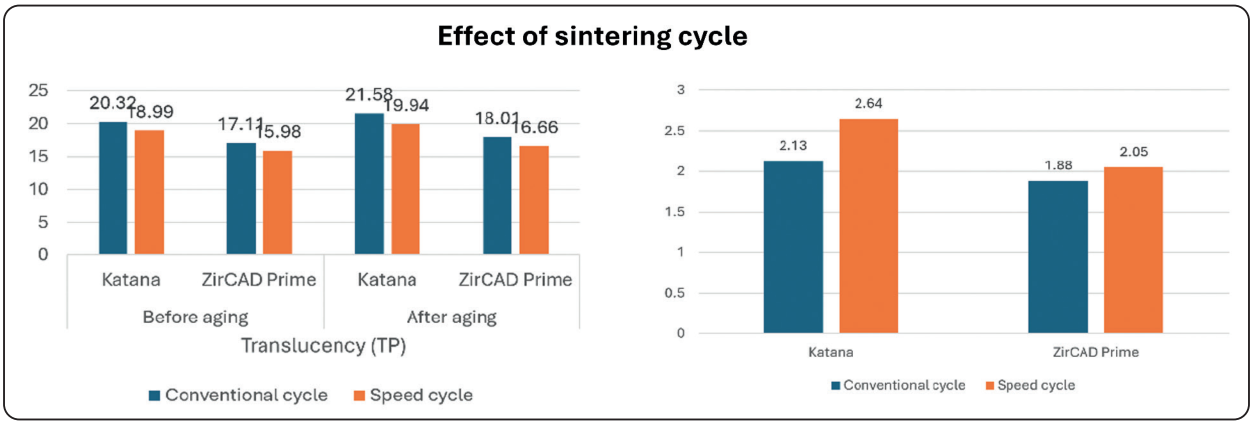
RESULTS

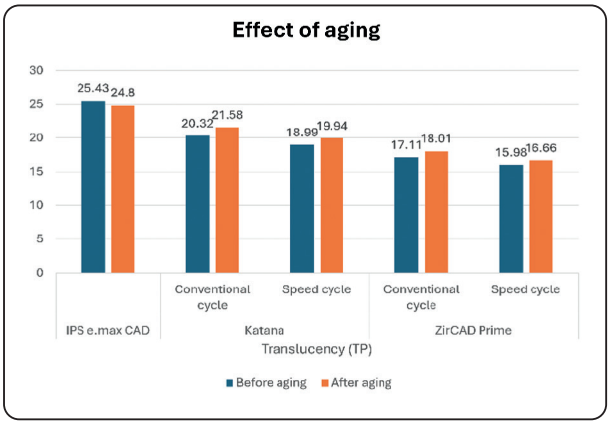
As for the effect of material on the TP; there was a significant difference between the investigated materials ($P < 0.001$) when measured before and after sintering. IPS e.max CAD showed the significantly highest translucency followed by Katana UTML Zirconia then IPS e.max ZirCAD Prime with both conventional and speed sintering cycles.

For both sintering cycles, different materials showed significant change in color. For the conventional cycle, IPS e.max CAD showed the significantly lowest change, while the difference between Katana UTML Zirconia and IPS e.max ZirCAD Prime was not significant. For the speed cycle, IPS e.max CAD showed the significantly lowest change followed by IPS e.max ZirCAD Prime and Katana UTML Zirconia showed the significantly lowest change.


Effect of material

The conventional sintering cycle produced significantly higher TP than speed cycle for both Katana UTML Zirconia ($P < 0.001$) and IPS e.max ZirCAD Prime ($P < 0.001$) when measured before and after aging. Conventional sintering cycle induced a significantly lower change in color for Katana UTML Zirconia ($P = 0.004$) when compared with the speed sintering cycle while the change in color of IPS e.max ZirCAD Prime was not significant between the two sintering cycles ($P = 0.6$) and while aging caused a significant decrease in the TP of IPS e.max CAD ($P = 0.002$), it significantly increased the TP of both Katana UTML Zirconia ($P < 0.001$) and IPS e.max ZirCAD Prime when measured before and after aging ($P < 0.001$).


Effect of sintering cycle



DISCUSSION

In modern dentistry, all-ceramic restorations offer superior esthetics and biocompatibility. Ceramic materials like lithium disilicate and zirconia have gained widespread use due to their improved properties.

Zirconia restorations, known for their exceptional mechanical strength, are now utilized in long-span restorations. However, challenges such as opacity and porcelain chipping have been addressed with the introduction of monolithic and cubic zirconia. These materials allow for the construction of full-contour zirconia restorations without the need for veneering porcelain(27,28). The latest zirconia generation, composed of tetragonal and cubic zirconia, exhibits superior optical properties compared to conventional and monolithic zirconia. Cubic zirconia, in particular, offers translucency comparable to lithium disilicate, although its mechanical properties may not match those of previous zirconia generations(29).

This study utilized two different zirconia materials, strength gradient multilayered zirconia (IPS e.max ZirCAD Prime) and color gradient multilayered zirconia (Katana UTML), with a thickness of 0.5 mm, as potential veneer materials. These were compared to IPS e.max CAD, serving as the control group, as lithium disilicate is considered the gold standard for veneer restorations.

A drawback of conventional zirconia is the lengthy sintering cycles. However, recent advancements in high-speed sintering have addressed this issue, making zirconia suitable for single-visit restorations.

Therefore, our primary focus was to investigate the impact of thermocycling aging and two different sintering cycles on the optical properties of two types of zirconia and compare them to lithium disilicate.

In this in vitro study, to compare the properties of available zirconia products, two commercially available zirconia types were selected: Katana UTML and IPS e.max ZirCAD Prime, both in shade A2, for comparison with IPS e.max CAD as the control group.

Thermal aging is a natural process affecting the optical and mechanical properties of ceramic materials used in dental restorations. To simulate oral conditions, thermocycling was performed using a specialized device. A total of 5000 thermal cycles, equivalent to 6 months of in vivo function, were conducted. Samples were immersed for 30 seconds in a temperature range of 50ºC to 55ºC, with a dwell time of 10 seconds and a transfer time of 5 seconds(30).

A spectrophotometer, a standardized scientific device developed for measuring and matching colors numerically, was utilized in our study to assess color change (ΔE) and translucency (TP) of the samples(31)**.** Spectrophotometers offer increased accuracy by 33% and provide a more objective match in 93.3% of cases compared to human observations or conventional techniques(32). Additionally, spectrophotometers have advantages over colorimeters, including longer working life and resistance to object metamerism(33).

The Agilent Cary 5000 spectrophotometer was used in our study. This device is controlled by the Cary WinUV software, which is a modular Windows-based software. This software facilitates powerful analysis and enables the integration of spheres for spectral and diffuse reflectance measurements.

CIE Lab measurements provide a standardized method for evaluating noticeable color alterations in samples, utilizing a consistent 3-dimensional color classification system. The L* value assesses the brightness or darkness of the sample, where higher L* values indicate lighter samples. The a* value represents chroma along the red-green axis, with positive values indicating red tones and negative values indicating green tones. Similarly, the b* value measures chroma along the yellow-blue axis, with positive values indicating yellowness and negative values indicating blueness. The overall color variation (ΔE) is calculated using the equation $\Delta E = ((\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2)$^1/2. Human perception of color can vary, making it challenging to detect subtle color changes. Clinically acceptable thresholds for color disparities rely on individual evaluations.

According to a clinical study, it was determined that 50% of dentists could discern a color difference of ΔE_{ab} 2.6. Restorations were deemed in need of replacement when the color difference reached 5.5 units due to color mismatch**(35)**.

The research categorized mean ΔE values as follows: those less than 2.6 were termed clinically imperceptible, values ranging from 2.6 to 5.5 were considered "clinically acceptable," and any values exceeding 5.5 were labeled as clinically unacceptable.(35)

Analyzing our findings, particularly concerning translucency, we observed a noteworthy disparity among the various groups. IPS e.max CAD exhibited the highest translucency value, followed by Katana Ultra translucent zirconia, whereas IPS e.max ZirCad Prime demonstrated the lowest value. This trend persisted both before and after aging, across both conventional and speed cycles.

These findings corroborated a study conducted by Aljanob et al. (36), which observed significant discrepancies in translucency between all examined groups before and after thermocycling aging. Notably, IPS e.max CAD exhibited the highest TP value, which was statistically significant. Moreover, all zirconia groups demonstrated a notable increase in TP values following the testing phase, consistent with our findings. Reyes et al. (37) also supported our results, indicating that translucent zirconia Katana and ultra-translucent zirconia were more translucent than high-strength zirconia but less translucent than lithium disilicate material. This difference might be attributed to IPS e.max ZirCAD prime being a multilayered zirconia containing both tetragonal and cubic zirconia. Tetragonal zirconia crystals are birefringent, causing light refraction in various directions, while highly translucent zirconia UTML comprises fully stabilized cubic crystals due to increased yttria content, resulting in isotropic behavior and reduced scattering within the grains. (37).

While increasing cubic crystals does enhance zirconia's translucency, it still exhibits lower TP values compared to glass ceramics, primarily due to the significantly lower light transmission through zirconia as opposed to glass-based ceramics. Regarding the impact of zirconia sintering cycles on translucency, the conventional cycle yielded significantly higher values than the speed cycle for both types of translucent zirconia investigated. This can be attributed to longer sintering times, particularly the dwell time at the final temperature, allowing for a larger area under the sinter curve and thus more time for grain formation. Consequently, this prolonged duration can lead to grain growth, resulting in increased translucency (3, 19).

As for the effect of aging on translucency; our results showed that there was significant increase in translucency of Katana Ultra Translucent and IPS e.max ZirCAD Prime after thermocycling aging, while there was a significant decrease in translucency after aging of IPS e.max CAD The increase in TP after aging in zirconia might be related to the transformation to monoclinic form and to the grain size, which occur in the superficial layer only**(38)**. The aging effect on TP values of Lithium disilicate may be attributed to the slow crack propagation within the material, facilitating water penetration and subsequent dissolution of the silica network. This process leads to a decrease in the material's crystallinity, resulting in a subsequent decrease in TP (39). Our findings align with a study by Kim and Kim (40), which reported a significant increase in the translucency of Katana as monolithic zirconia after autoclaving for up to 10 hours. However, their results regarding lithium disilicate differed, as they observed an increase in translucency after aging. Conversely, our results contrast with those of Fathy et al. (41), who suggested that the formation of pores and differences in refractive indices between monoclinic and tetragonal zirconia crystals induce more light scattering, leading to decreased translucency after hydrothermal aging.

Our study revealed significant color changes among the three tested materials, with ultra-translucent zirconia exhibiting significantly higher changes than IPS ZirCAD Prime, while lithium disilicate showed the least color change. This finding is consistent with the results of Kurt et al. (42), who demonstrated that color changes due to aging are greater in monolithic zirconia than in lithium disilicate. They also found that lithium disilicate ceramic displayed superior esthetics in terms of color stability and translucency. Similarly, Kim et al.(40) reported that the color change in Katana monolithic zirconia was significantly higher than that in IPS e.max CAD following artificial aging in an autoclave. These results may be attributed to the susceptibility of zirconia to low-temperature degradation (LTD), which involves phase transformation from a tetragonal to a monoclinic structure(43). This phase transformation results in a 4% increase in volume (zirconia grains pull out), leading to structural disintegration, surface roughness, and the development of microcracks, which may adversely affect their optical properties(44).

The presence of at least 5.5 mol% of yttria increases the cubic phase content, which is responsible for the improved translucency and the decrease of hydrothermal degradation while undergoing aging**(5)**.

This absence of low-grade thermal degradation, and its consequences of surface roughness and cracks, is speculated to improve the color stability of the highly translucent zirconia as compared to the low-translucency type. It was also proposed in another research that the alumina content is accountable for the resistance of low-temperature degradation. They reported that monolithic zirconia is more susceptible to LTD than core zirconia due to lower alumina content**(41)**.

The presence of at least 5.5 mol% of yttria increases the cubic phase content, contributing to enhanced translucency and reduced hydrothermal degradation during aging (5).

This absence of low-grade thermal degradation, along with its associated effects such as surface roughness and cracks, is speculated to improve the color stability of highly translucent zirconia compared to low-translucency types. Another study proposed that the alumina content plays a role in resisting low-temperature degradation. They found that monolithic zirconia is more susceptible to LTD than core zirconia due to its lower alumina content (41).

Another study noted that 3Y-TZP with 0.05 wt.% Al_2O_3 undergoes tetragonal to monoclinic transformations during hydrothermal aging much more rapidly than 3Y-TZP samples with 0.25 wt.% Al_2O_3. This suggests that a decreased alumina content diminishes the tetragonal hydrothermal stability due to stress-assisted corrosion (45).

According to the manufacturer's microstructure, Katana ultra-translucent zirconia has no alumina while in IPS ZirCAD Prime has a percentage of aluminum oxide, this may explain why IPS ZirCAD Prime has more color stability than Katana ultra-translucent zirconia.

Our study results were in opposite with Tamer A. Hamza et al. (2016), where he stated that translucent zirconia showed color stability after being subjected to artificial accelerated aging(46).

In terms of sintering cycles, we observed that the speed cycle yielded a higher color change value compared to the conventional cycle in both IPS e.max ZirCAD and Katana UTML. However, the difference in IPS e.max ZirCAD Prime was not statistically significant. Our findings align with a study conducted by Ebeid et al. (47), which demonstrated a decrease in color change with increasing sintering time (conventional cycle). This phenomenon could be attributed to the assumption that the sintering process reduces the pores between the grains and enhances the final density of zirconia, thereby decreasing light scattering and increasing light transmission, resulting in improved optical characteristics. This assumption was consistent with our results, as an increase in sintering time (conventional cycle) correlated with enhanced color stability of zirconia.

CONCLUSION

The conventional sintering cycle had a significantly higher translucency value than the speed cycle for both types of investigated translucent zirconia. Speed cycle had a higher color change than conventional cycle in Katana UTML

Thermocycling aging increased translucency of Katana Ultra Translucent and IPS e.max ZirCAD Prime while it decreased translucency of IPS e.max CAD.

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