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The effect of staining and vacuum sintering on optical and mechanical properties of partially and fully stabilized monolithic zirconia

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The effect of staining and vacuum sintering on optical properties and the bi-axial flexural strength of partially and fully stabilized monolithic zirconia (PSZ, FSZ) were evaluated. Disc-shaped specimens divided into three subgroups (n=15): non-stained, stained and non-stained with vacuum sintering. After staining and sintering, optical properties were evaluated using a reflection spectrophotometer and bi-axial flexural strength was tested using the piston-on-three balls technique. Statistical analysis was performed using multivariate analysis of variance (MANOVA) followed by *post-hoc* Tukey's tests ($p<0.05$). Staining decreased translucency parameter (TP) values of FSZ ($p<0.05$). Sintering under vacuum enhanced TP values for PSZ ($p<0.05$). Staining enhanced surface gloss for both types of zirconia ($p<0.05$). Staining increased bi-axial flexural strength of FSZ ($p<0.05$), while it decreased the strength of PSZ ($p<0.05$). Sintering under vacuum provided minimal benefits with either type of zirconia.

Keywords: Partially stabilized zirconia, Fully stabilized zirconia, Staining, Bi-axial flexural strength, Optical properties

INTRODUCTION

Recently, zirconium oxide has attracted significant attention in the dental field. Indeed it has proven to possess adequate biological properties and the transformation toughening phenomenon is a significant advantage contributing to clinical success. However with layered zirconia, issues primarily related to chipping of the veneering porcelain have been reported. In four published reports of different zirconia systems, 8–50% of prostheses developed crazing or cracking with minor loss of material after only 1–2 years of observation[1-4]. However, fracture of the zirconia core seems to be relatively uncommon. With the goal to eliminating the risk of veneer chipping or fracture, full anatomical monolithic zirconia restorations were introduced. However, little clinical data on the performance of monolithic zirconia restorations is available. The use of monolithic full-contour zirconia in dental applications such as single and multi-unit restorations raises a set of unique challenges stemming from shade production, tribological behavior, and long-term chemical stability[5]. Many properties of monolithic zirconia have yet to be explored. However, it is well-established that every step of the fabrication process of zirconia ceramics has to be carefully controlled to achieve expected mechanical and chemical properties. This includes blank fabrication, green machining, sintering process, coloring, and surface treatments, whether chemical, thermal, or mechanical[5].

Unalloyed zirconia can exist in three crystallographic forms; monoclinic (at room temperature to 1,170°C), tetragonal (1,170°C to 2,370°C) and cubic (2,370°C to melting point (2,716°C)[6,7]. Upon cooling, tetragonal phase zirconia transforms to monoclinic phase accompanied by a 4.5% volume increase, which can fracture the zirconia. Adding oxides such as CaO, MgO and Y_2O_3 maintains the tetragonal structure at room temperatures preventing the phase transformation and leading to high toughness[8-10]. The stability of zirconia is influenced by grain size, nature, and amount of stabilizer[11]. Y_2O_3 (yttria) is the most widely used stabilizer in high-tech application of zirconia. Partially stabilized zirconia (PSZ) (also known as yttria tetragonal zirconia polycrystals), where the yttria content can occur at concentrations 2–5 mol% depending on grain size, is considered the standard for manufacturing zirconia blocks used in dentistry, most widely researched and microstructurally complex ceramic[12]. The term 'partially' stabilized zirconia refers to the yttria addition in concentrations lower than that required for full cubic zirconia stabilization. Increasing yttria concentrations to no less than 8 mol% is needed to achieve full stabilization[7,13,14], hence the term 'fully' stabilized zirconia (FSZ). Recently, some dental zirconia manufacturers have produced zirconia in its full cubic stabilized form in efforts to improve zirconias translucency (due to increased cubic phase).

Accurate tooth color reproduction presents one of the most significant clinical challenges associated with

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monolithic zirconia. Two main approaches for coloring zirconia are available. One method is to incorporate metal oxides into the zirconia powders prior to pressing. Another is using solutions containing metal-salts, which are applied to the surface when the zirconia is in its pre-sintered stage through infiltration. The latter method can have some drawbacks such as presence of porosity gradients that can result in a non-uniform color[15], and issues related to limited diffusion depth of the coloring solutions which leads to lighter areas after grinding adjustments[16]. These coloring methods can have an effect through crystallographic and microstructural changes that can in turn affect the mechanical properties of zirconia. Little data is available in the literature on the effect of zirconia coloring using metal-salts on esthetic and mechanical properties.

The sintering process can also affect the final properties of zirconia. Sintering temperatures and duration determine the grain size, amount of cubic phase, and yttria segregation that in turn dictate metastability, mechanical properties, and resistance to low temperature degradation[5]. Sintering consists of at least two heat-activated processes, densification and grain growth. The most common method for sintering zirconia would be the use of conventional furnaces at temperatures between 1,350–1,600°C and holding times ranging from 2–4 h. Multiple alternative methods for sintering zirconia have been introduced such as microwave sintering, spark plasma sintering, and use of vacuum furnaces, all aiming to improve the mechanical and esthetic properties of the zirconia and to some extent save on sintering time and energy. In a vacuum furnace, the absence of air or other gases prevents heat transfer with the product through convection and removes a source of contamination. Some manufacturers claim that use of vacuum furnaces can enhance mechanical properties and increase the translucency of the zirconia.

The aim of this study was to evaluate the effect of a commercial acid-free aqueous metal-salt coloring solution on the optical properties of zirconia in terms of translucency parameter (TP), contrast ratio (CR), and surface gloss. In addition the bi-axial flexural strength of partially stabilized versus fully stabilized zirconia. Another aim was to determine the effect of sintering in a vacuum furnace on the translucency and bi-axial flexural strength of the fully and partially stabilized zirconia.

MATERIALS AND METHODS

Sample preparation

Two types of monolithic zirconia were used in this study; PSZ (Prettau®, Zirkonzahn, Taufers, Italy) and FSZ (Prettau®, Anterior, Zirkonzahn) (Table 1). Zirconia cylinders (20 mm diameter) where milled (Milling unit M5, Zirkonzahn) and then cut using a cutting device (Struers Secotom-50, Copenhagen, Denmark) to a 1.8 mm thickness. In the green stage, each specimen was sequentially ground to a 1.5 mm thickness using silicon carbide grinding paper (FEPA #1200, 2400, and 4000) (Struers LaboPol 21, Struers A/S, Rodovre, Denmark) under finger pressure. The final thickness (±0.01mm) was measured using a digital caliper (Mitutoyo, Kanagawa, Japan). The specimens were divided into three groups for each type (non-stained, stained, non-stained vacuum sintered, *n*=15/subgroup). Prettau® Anterior specimens were stained using shade A2 Color Liquid Prettau® Anterior Aquarell (Zirkonzahn), and Prettau® specimens were stained using shade A2 Color Liquid Prettau® Aquarell (Zirkonzahn). A touchup brush was used to apply two coats of the mentioned liquid stain. The specimens were placed for 20 min under an infrared drying lamp, Zirkonlamp 250 (Zirkonzahn), following the manufacturers' instructions.

The stained and non-stained groups were sintered in a non-vacuum sintering furnace (Zirkonofen 600/V2, Zirkonzahn). Another non-stained group of both zirconia types was sintered using a vacuum furnace (Zirkonofen 700 Ultra-Vakuum, Zirkonzahn). The sintering conditions for Prettau® specimens started at room temperature with an elevating heat rate at 6°C/min until 1,600°C where it holds for 2 h and then cools at 6°C/min rate to room temperature. Prettau® Anterior sintering conditions started at room temperature and heat elevates at 5°C/min rate till 1,450°C where it holds for 2 h and then cools down at a 5°C/min rate until room temperature. Final specimen size after sintering was (16 mm diameter ×1.2 mm thickness). The same operator polished each specimen using a straight lab handpiece (K5plus, Kavo, Germany) connected to an electrical control unit (K-control 4960, Kavo) using diamond polishing burs (Zircpol Plus and Zircoshine Plus, Diatech, Switzerland) followed by a polishing paste (Zircon-Brite, Dental Ventures of America, Corona, CA) at a constant speed of 10,000 rpm, under constant pressure and standard time in a single directed motion, following manufacturers' instructions. The specimens

Table 1 Materials used in this study

Brand name	Stability	Manufacturer	Composition
Prettau® Zirconia	Partially stabilized	Zirkonzahn, Taufers, Italy	4–6% Y_2O_3, <1% Al_2O_3, max. 0.02% SiO_2, max. 0.01% Fe_2O_3, max. 0.04% Na_2O
Prettau® Anterior	Fully stabilized	Zirkonzahn, Taufers, Italy	<12% Y_2O_3, <1% Al_2O_3, max. 0.02% SiO_2, max. 0.01% Fe_2O_3, max. 0.04% Na_2O

were cleaned ultrasonically in distilled water for 10 min (Quantrex 90, L&R Ultrasonics Manufacturing, Kearny, NJ, USA). Each specimen was air-dried for 20 s before testing.

Optical measurements

To estimate the TP, CR, and surface gloss values of each specimen, a reflection spectrophotometer (CM-700d, Konica Minolta Sensing, Tokyo, Japan) was used according to the CIE 1976 L*a*b* color scale relative to the CIE standard illuminant D65 (as defined by the International Commission on Illumination) which corresponds to “average” daylight (including ultraviolet wavelength region with a correlated color temperature of 6504K). The Specular Component Excluded (SCE) and Specular Component Included (SCI) geometries were determined according to the CIE L*a*b* color scale using standard illuminant D65 over white (CIE L*=98.1, a*=−0.5 and b*=2.8) and black (CIE L*=4.7, a*=−0.1 and b*=0.0) background.

Differences in surface gloss (ΔE*SCE−SCI) values were calculated by the following equation[17]:

$$\Delta E^*_{SCE-SCI}=[(\Delta L^*_{SCE-SCI})^2+(\Delta a^*_{SCE-SCI})^2+(\Delta b^*_{SCE-SCI})^2]^{1/2}$$

Calibration of the spectrophotometer was executed before measurement of each specimen. Then the TP of each specimen was obtained by calculating the color difference between the specimen against the white background and against the black background using the following equation[18]:

$$TP=[(Lb^*-Lw^*)^2+(ab^*-aw^*)^2+(bb^*-bw^*)^2]^{1/2}$$

L* refers to the lightness, a* to redness to greenness, and b* to yellowness to blueness.

The CR values were calculated from the spectral reflectance of light of the specimen (Y) over a black background (Yb) and over a white background (Yw), using the following equation[19]:

$$CR=Yb/Yw$$

Bi-axial flexural strength test

Bi-axial flexural strength values were determined in accordance with the ISO Standard 6872[20]. The thickness and diameter of the sintered disks were confirmed with a digital caliper before the fracture test. The specimens were tested dry at room temperature with a universal testing machine (Model LRX, Lloyd Instruments, Fareham, UK) where they rested on three symmetrically based steel balls and the load was applied to the center of the top surface by the piston (diameter 1.60 mm) until fracture occurred (Fig. 1). The cross-head speed of the piston was 1.0 mm/min. Results were recorded with Nexygen software (Lloyd Instruments). The bi-axial flexural strength was calculated as[20]:

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

S: bi-axial flexural strength (MPa); P: fracture load (N); d: specimen disk thickness at fracture origin (mm).

$$X=(1+v)\ln(B/C)^2+[(1-v)/2](B/C)^2$$

$$Y=(1+v)\ln(A/C)^2+[(1-v)/2](A/C)^2$$

v: Poisson’s coefficient (ceramic=0.25, ISO 6872); A: radius of support circle (mm); B: radius of loaded area (mm); C: radius of specimen disk (mm).

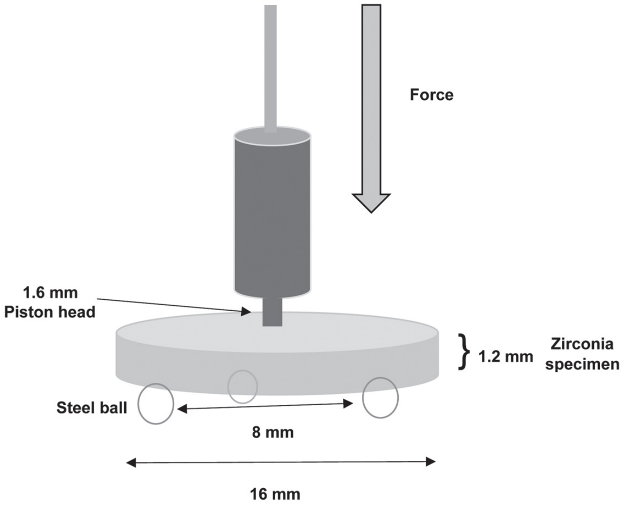

Fig. 1 llustration of the bi-axial flexural strength testing. The specimen rested on three symmetrically based balls and the load (F) was applied to the center of the top surface by the piston (diameter 1.6 mm) until fracture occurred.

Scanning electron microscopy (SEM)

Specimens from the regular and under vacuum sintering of both zirconia types were sputter-coated with gold (Polaron SC 502, Fisons Instruments, UK) and secondary electron SEM images were taken (Leo Gemini 1530, Zeiss, Oberkochen, Germany). Visual examination was used to inspect the grain structure of both groups after sintering and no quantitative data regarding grain size was extracted.

Statistical analysis

Statistical analysis was performed with Statistical Package for the Social Sciences (Version 22.0, SPSS, Chicago, IL, USA). The type of zirconia, sintering furnace and staining are the independent variables. The surface gloss, TP, CR and bi-axial flexural strength are the dependent variables. Thus, the data were analyzed using multivariate analysis of variance (MANOVA) followed by Tukey’s post-hoc test. Differences were considered significant at 95% confidence level.

RESULTS

The TP values are presented in Fig. 2. The TP values for FSZ were significantly higher than PSZ ($p<0.05$) regardless of the staining or the type of sintering used. For PSZ, staining had no significant effect on TP values ($p>0.05$), while vacuum sintering improved the values ($p<0.05$). However, in case of FSZ, staining decreased the TP values ($p<0.05$), but sintering in a vacuum furnace had no effect ($p>0.05$).

The ranking of all groups according to their CR

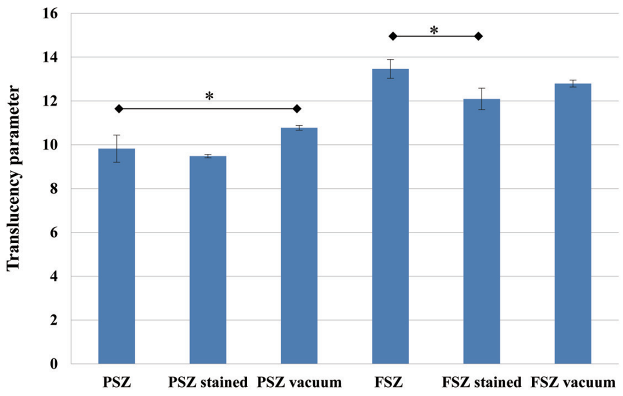

Fig. 2 Mean values and standard deviations of translucency parameter for partially stabilized zirconia (PSZ) and fully stabilized zirconia (FSZ).
FSZ was statistically higher than PSZ regardless of the staining or sintering method ($p<0.05$). Vacuum sintering of PSZ improved translucency parameter (*$p<0.05$). Staining FSZ decreased translucency parameter (*$p<0.05$).

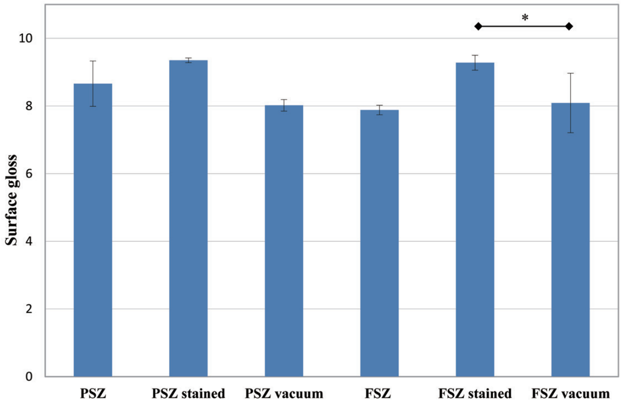

Fig. 3 Mean values and standard deviations of surface gloss for partially stabilized zirconia (PSZ) and fully stabilized zirconia (FSZ).
Staining increased surface gloss of FSZ (*$p<0.05$).

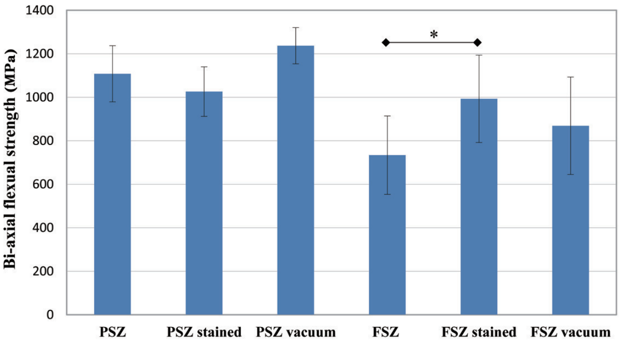

Fig. 4 Mean values and standard deviations of bi-axial flexural strength for partially stabilized zirconia (PSZ) and fully stabilized zirconia (FSZ).
Bi-axial flexural strength of PSZ zirconia was higher than FSZ ($p<0.05$). Staining increased the bi-axial flexural strength of FSZ (*$p<0.05$).

values, from least to most translucent is: Prettau® stained<Prettau®<Prettau®vacuum sintered<Prettau® Anterior stained<Prettau® Anterior vacuum sintered= Prettau® Anterior ($p<0.05$).

The surface gloss values are presented in Fig. 3. Staining increased the surface gloss of FSZ ($p<0.05$), but had no significant effect on PSZ. Type of sintering had no effect on either of the zirconia types.

The bi-axial flexural strength of PSZ zirconia was higher than FSZ ($p<0.05$). Staining increased the bi-axial flexural strength of FSZ ($p<0.05$), but the type of sintering had no effect. Neither staining nor the type of sintering had an effect of the bi-axial flexural strength of the PSZ zirconia (Fig. 4).

By visual inspection, after regular oven and vacuum sintering the FSZ zirconia grains showed minor crack-like surface changes of ca. 200 nm in length. The cracks were fewer on the PSZ zirconia surface and almost absent after vacuum sintering (Fig. 5).

DISCUSSION

This study compared the optical and mechanical properties of partially (Prettau®) and fully (Prettau® Anterior) stabilized zirconia materials before and after staining. Another goal was to discover whether sintering under vacuum conditions has an effect on these properties. FSZ has yttria content around 12 mol%, with more cubic form zirconia grains than in PSZ in which yttria content is 4–6 mol% and the zirconia grains exists in tetragonal form. In its cubic form zirconia has enhanced optical properties, while mechanical properties are inferior to that of tetragonal form of zirconia. The results of this study supports the manufacturers' data that FSZ has indeed lower bi-axial flexural strength and more translucency compared to PSZ.

Coloring liquids can either be acidic or neutral. The main purpose for using an acid-based liquid is that its acidity (preferably pH 1–3) will allow the color to penetrate deep within the restoration. In addition, certain metal-salts can only be dissolved in acid. Different metal-salts have been discovered to be able to dissolve in water, creating similar results in terms of color to acid-based liquids. Additionally, aqueous coloring liquids used in our study are promoted by the manufacturers as being more efficient and friendlier to the technician and the environment as less acidic fumes are released during the liquid application and sintering of the restoration and also with more enhanced drying time (Hellweger M., Zirkonzahn, personal communication).

Our finding that staining of PSZ had no significant

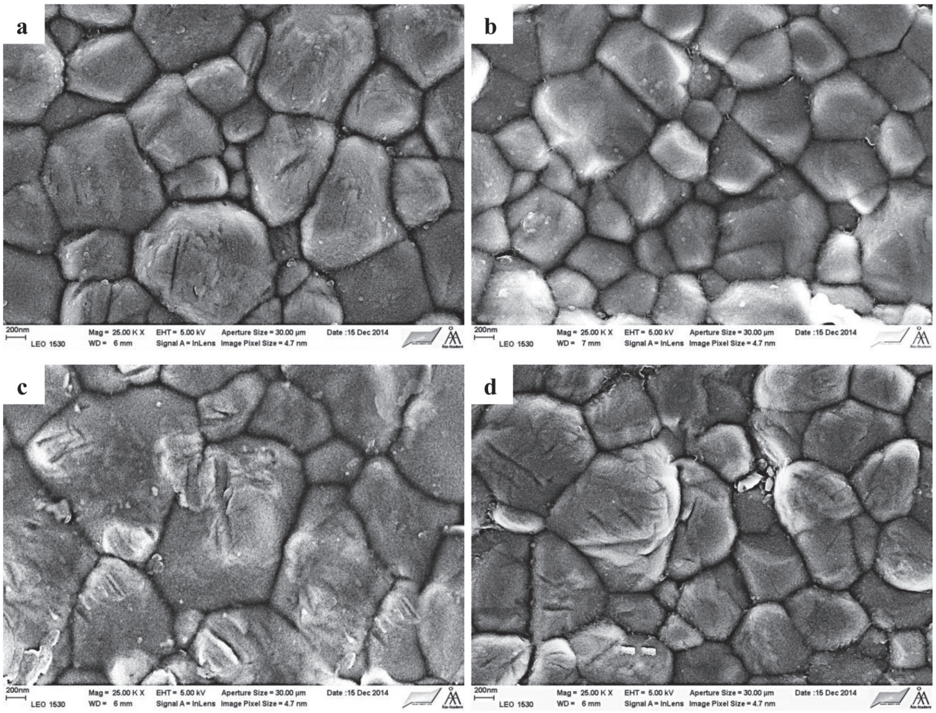

Fig. 5 Scanning electron micrographs of Prettau® (partially stabilized zirconia, PSZ) and Prettau® Anterior (fully stabilized zirconia, FSZ) sintered in regular and vacuum furnaces.
(a) PSZ regular sintering, (b) PSZ vacuum sintering, (c) FSZ regular sintering, and (d) FSZ vacuum sintering.

effect on its translucency values, while staining of FSZ decreased its translucency significantly is interesting. This can be possibly interpreted by the different microstructure and grain boundary dimensions of the FSZ which may lead to more stain absorption than the PSZ. In addition, the chemical nature of the coloring liquid can lead to more absorption of light energy and thus decreasing the amount of light transmission. Although the decrease was statistically significant, the difference in translucency is most likely not perceivable to the eye. According to Liu *et al.*[21)], the human eye can perceive a 0.06 contrast ratio difference in translucency. Based on the data of the present study, the mean CR for non-stained PSZ and FSZ was 0.92 and 0.87 respectively. After staining with an aqueous, acid free Aquarell A2 coloring liquid, the mean CR raised to 0.94 and 0.90 respectively. Regarding the measurements of translucency using contrast ratio values, 0 indicates most translucent and 1 most opaque outcome. In our study the difference in CR values before and after staining was 0.04 and 0.03 respectively. Therefore, having a CR difference less than 0.06, will render it difficult to detect such translucency change with the naked eye.

In regards to the effect of staining on the flexural strength, staining enhanced significantly the flexural strength of FSZ, while it decreased the flexural strength of PSZ. Interpretation of such behavior for FSZ is rather scarce. A possible explanation might be a result to the different microstructure, nature of grain form, and its relation to the high yttria content, which all can have an influence on the mechanical properties of the stained FSZ. Also, coloring liquids contain metal oxides which have the potential to lead to crystallographic and microstructural changes that could in turn affect the mechanical properties of zirconia[15)]. Further studies are needed to investigate the mechanism of such behavior in detail. A recommendation for future studies would be to compare whether the use of an acidic versus neutralstaining liquid causes any differences in the mechanical properties of PSZ or FSZ.

The finding that staining enhanced surface gloss of fully stabilized zirconia can be explained by the fact that it inhibited light transmission through the zirconia. This in turn increases the amount of reflected light rendering the zirconia more luminous, thus increasing surface gloss.

Coloring liquids are applied to the surface of the pre-sintered zirconia in an attempt to create a restoration that can mimic the esthetic appearance of a natural tooth. Techniques such as dipping, brushing or spraying have been advocated to incorporate the color[22)]. There is some disagreement regarding which technique provides a more predictable and reliable result. Although, dipping the restoration in the coloring liquid is a straightforward procedure, it can lead to an increase in porosity, oversaturation and affect translucency[15)]. The brushing technique, on the other hand, allows application of multiple colors in a more controlled manner with fewer ramifications on the material's mechanical and optical properties.

Concentrations of the coloring liquids can have a considerable effect on the mechanical properties of zirconia materials. Certain types of metal-salts at elevated concentrations have been proven to decrease the flexural strength of zirconia[15,22]. It has also been claimed that elevated concentrations can render a different coloring effect of the same coloring liquid at lower concentrations. In general, higher concentrations of coloring liquids have a lower value (darker) and a more yellow-red hue than the same coloring liquids at lower concentrations[23].

A second aim of our study was to evaluate the effect of vacuum sintering on the optical and mechanical properties of the zirconia materials. It has been claimed that sintering zirconia in a vacuum furnace can increase its flexural strength and enhance translucency (Manufactures' data), since the absence of oxygen or other gases will provide an environment with little or no surface oxidation. This preserves the yttria content within the zirconia and renders it more strong and stable. Moreover, it is claimed to prevent heat transfer within the product through convection and to remove a source of contamination.

Indeed, our results indicated that sintering in a vacuum furnace seem to play a role in enhancing the bi-axial flexural strength for both types of zirconia, although the difference was statistically insignificant under current study set up. Whether this has any clinical significance is still to be researched.

In regards to the claim on enhancing the translucency of zirconia, our results indicate that this is true for PSZ, while it has no significant effect on FSZ. However, the drop in CR value was minimal and it cannot be perceived by human eye[21] and may not have any clinical relevance.

In conclusion, *in vitro* laboratory research is crucial to provide information on the behavior of restorative materials. It has always been a challenge to link *in vitro* testing to clinical conditions. *In vitro* studies can be a definite blueprint for designing a proper clinical study. Closely monitored clinical studies of full contoured monolithic zirconia restorations with critical failure analysis should be performed prior to making definitive clinical recommendations.

CONCLUSION

The recently introduced FSZ behaves differently than the traditional PSZ. Staining had different effects on FSZ than it had on PSZ. After staining, the flexural strength increased with FSZ while the strength decreased with PSZ. Sintering under vacuum provided minimal benefits with either type of zirconia.

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