Effect of different immersion times and sintering temperatures on translucency of monolithic nanocrystalline zirconia

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ABSTRACT

Purpose: The objective of the study was to investigate the effect of different immersion times, in coloring liquid, and sintering temperatures on translucency of monolithic nanocrystalline zirconia.

Materials and methods: Forty five specimens of nanocrystalline zirconia were obtained by cutting InCoris TZI blocks into slices using a stainless steel disc mounted on a custom made milling machine. The slices were divided into three groups (n = 15) according to immersion times (3, 5 and 7 min) then each group was further subdivided into 3 subgroups (n = 5) according to the sintering temperatures (1400 °C, 1500 °C and 1600 °C). CIE-Lab coordinates were measured for each slice against black and white backgrounds using Vita easy shade spectrophotometer and translucency parameter (TP) was calculated. One way analysis of variance combined with a Tukey-post hoc test were used to analyze the data obtained (P = 0.05).

Results: Results of the present study showed that at temperature 1400 °C there was statistically significant decrease in TP between 7 min immersion time and the other two groups, while there was no statistically significant difference in TP using different immersion times at temperatures 1500 °C and 1600 °C.

Conclusion: Our results showed that the best translucency was obtained by the combination between lower dipping times and higher sintering holding temperatures.

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1. Introduction

In recent years, esthetic dentistry has become more prevalent because of increasing demands from patients and the development of new techniques that help to reproduce natural tooth translucency and color.

Zirconia-based restorations have become very popular due to their superior mechanical properties and excellent biocompatibility. Due to their high opacity, zirconia core is usually veneered with veneering porcelain. However, in clinical service, the most frequent failure is the chipping of the veneer, while the high-strength zirconia substructure is mostly not affected [1] and [2]. In specific clinical situations, such as when the occlusal or palatal space is limited or in cases where a patient’s parafunctional activity (e.g., bruxism) may contraindicate this veneering application, the use of unveneered zirconia ceramic seems to be an option for all-ceramic restorations [3]. Recently nano-crystalline zirconia, which showed satisfactory optical and mechanical properties, was introduced allowing the use of monolithic zirconia restorations without the need for any veneering ceramic [4].

Zirconia restorations are usually milled in a partially sintered state then these machined restorations are infiltrated with special coloring liquids either by immersion, spraying, or brushing to produce work pieces of various shades [5] and [6]. This is followed by a sintering process to allow the restoration to reach its maximum density [7] and [8]. Several manufacturers replaced their long sintering cycles with shorter ones by changing the heating and cooling rates and by modifying the sintering holding temperatures. The effect of these changes on the final properties of the zirconia restoration remains in question.

Natural teeth tend to have varying shades and translucencies
throughout their structures. Therefore translucency is as important as color in the shade matching procedure to achieve optimal esthetic outcome of these dental restorations, where translucency is the relative amount of light transmitted through the material. A material’s translucency could be measured in terms of contrast ratio or translucency parameter. As for the translucency parameter (TP) [9] and [10], it was first described by Johnston et al in 1995 [10] and used to describe translucency of dental materials. It is defined as the color difference of a material of a given thickness over white and black backgrounds, and corresponds directly to common visual assessments.

The study was designed to investigate the effect of different immersion times, in coloring liquid, and sintering temperatures on translucency of monolithic nano-crystalline zirconia. The null hypothesis was that both the change in dipping times and sintering temperatures would affect the translucency.

2. Materials and methods

Forty five slices of dimensions (15.5 mm × 19 mm x 1.25 mm) were milled from nano-crystalline Yttria-stabilized tetragonal zirconia blocks (inCoris TZI). The slices were divided into three groups of fifteen slices each according to the immersion time in their coloring liquid. Group A was immersed for three minutes; Group B was immersed for five minutes and Group C for seven minutes. Each group was further sub-divided into three subgroups of five slices each according to the final sintering holding temperature. Subgroup 1 was sintered at 1400 °C, subgroup 2 was sintered at 1500 °C, and Subgroup 3 was sintered at 1600 °C (Table 1).

2.1. Slicing of the zirconia samples

Slices of 1.25 mm thickness each were cut out of the pre-sintered inCoris TZI blocks using water cooled diamond blade with a low speed cutting saw.1 Thickness was adjusted to allow for 20–25% shrinkage during sintering so that the final slice thickness would be 1 mm.

2.2. Finishing and thickness adjustment of the milled specimens

Each slice was finished wet using water-proof silicon carbide sandpaper2 of different grit size ranging from 320 to 1200 in order to adjust the thickness and provide a smooth surface. Each specimen thickness was checked using a micrometric caliper.3

The slices were washed under running water and ultrasonically cleaned in distilled water for five minutes to wash out any debris that might affect the surface and hence affect the coloring procedure and the measurement results.

2.3. Coloring of the zirconia slices

Specimens coloring was done by immersion technique in TZI sirona coloring liquid of shade A3. Each sample was immersed solely at a time in a dipping container and time was calibrated using a stopwatch according to the previously mentioned sample grouping.

The samples were removed from the coloring liquid with a pair of plastic tweezers and hung for 30 s in a vertical position to allow dropping of excess liquid traces before being placed on a non-absorbable surface. Samples were then left to bench dry for 24 h.

2.4. Sintering of the colored samples

The samples were sintered using Sirona infire HTC speed sintering furnace4 according to the below mentioned sintering cycle (Table 2). They were placed on specially designed sintering boats filled with sintering alumina beads.

Samples of each group were arranged spaced at least 1 cm from each other to allow even shrinkage. Groups were sintered simultaneously, each in a single sintering cycle.

2.5. Checking and verification

After sintering, samples were left to cool down to room temperature before final dimension of (12 mm × 15 mm x 1 mm) was checked using a micrometric caliper.

2.6. Color and translucency measurement

Each specimen was tested for translucency parameter (TP) using a portable digital spectrophotometer (Vita EasyShade Compact5).

The Vita Easy shade was used in the “Tooth Single” mode to determine the values of the CIE-Lab coordinates. The probe tip of the device was centrally and perpendicularly placed in full contact with each specimen’s surface till a confirmation beep was heard. Each specimen was measured against a white and a black background. Each specimen was measured three consecutive times and mean values for L*, a*, and b* co-ordinates were recorded. The device was calibrated in the calibration slot before each measurement for maximum standardization. Translucency parameter was measured according to the following formula;

\[ TP = \left[ (L^* - L^*_w)^2 + (a^*_b - a^*_w)^2 + (b^*_b - b^*_w)^2 \right]^{1/2} \]

Where the subscript (b) refers to the color co-ordinate against black background while the subscript (w) refers to the color co-ordinate against white background.

2.7. Environmental scanning electron microscopy

One random representative sintered sample from each sintering group was scanned under Environmental Scanning Electron Microscope6 (Quanta-FEG-ESEM250) for assessment of grain growth and micro-structure.

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1 ISOMET 4000, Buehler, Lakebluff, U.S.A.
2 Matador SoftFlex, Germany.
3 Mitutoyo, Tokyo, Japan.
4 Sirona Dental Systems GmbH, Bensheim, Germany.
5 VITA, Zahnfabrik H. Rauter GmbH & Co. KG.
6 FEI company, Netherlands.
4.2. Effect of sintering temperature

Scanning electron micrographs of inCoris TZI slices at the 3 used different temperatures showed that Zirconia specimens sintered at 1400 °C had greater porosity (Fig. 3). The grain size ranged from 250 to 700 nm at 1400 °C. At temperature of 1500 °C the proportion of larger grains increased (Fig. 4). However, at 1600 °C the grains grew more even and varied between 500 nm and 1.2 μm (Fig. 5).

5. Discussion

The null hypothesis for that study was partially accepted where translucency parameter revealed a general rise with increasing the final sintering temperature from 1400 °C to 1600 °C. The effect was more pronounced with prolonged dipping time (7 min), where Group C1 showed TP mean value of 12.75, while Groups C2 and C3 showed TP mean values of 13.68 and 13.78 respectively. This means that the effect of temperature change was more prominent with the maximum dipping time used.

This can be attributed to the color change when coloring agents are baked at higher temperatures. Since these coloring agents must be added in the form of an oxide, obtained by dissolving in hydrochloric acid, the color may be damaged when the coloring agents are subjected to high temperatures. This can occur in a temperature range from 1350 to 1600 °C according to the liquid’s composition where for the inCoris TZI coloring liquid-used in the current study-this disintegration temperature happened to be 1600 °C [11]. Furthermore, sintering was performed in air without any argon atmosphere. Therefore, a degree of oxidation might have happened at such a high temperature which would also negatively affect the color saturation and hence increase the translucency [4].

The above results were supported by Tuncel et al in 2013 [5] in a study that correlated the use of coloring liquid to the translucency of zirconia. The control group of zirconia specimens, which did not undergo a coloring procedure, showed significantly more translucency than other groups which were colored by dipping in different shades of the coloring liquid. This suggested that incorporation of coloring liquid within the specimens decreased the translucency of zirconia frameworks when compared to non-colored frameworks.

Our results were partly similar to a study performed by Jiang et al [12], where sintering temperatures 1350, 1400, 1450, and 1500 °C were used and their effect on the translucency of zirconia discs was measured. They concluded that as the sintering temperature increased from 1400 °C to 1500 °C, the translucency of the discs increased. However, further increase showed no significant improvement of the translucency.

Likewise, the previously mentioned results came out agreeing with Stawarczyk et al [13] who studied the effect of sintering temperature on contrast ratio of zirconia discs. Zirconia specimens (Ceramill ZI, Amann Girbach) were sintered at temperatures ranging from 1300 °C to 1700 °C. Specimens were sintered at a heating rate of 8 °C/min and a holding time of 2 h. Authors concluded that as the sintering temperature increased, the contrast ratio of the zirconia specimens decreased and better translucency was achieved.

The results also showed acceptance with Ebeid et al in 2014 [14] who evaluated the effect of different sintering parameters on color

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Table 1

<table>
<thead>
<tr>
<th>Change in temperature</th>
<th>Rate °C/Min</th>
<th>Temperature °C</th>
<th>Holding Time in Minutes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heating</td>
<td>25</td>
<td>800</td>
<td>7</td>
</tr>
<tr>
<td>Heating</td>
<td>15</td>
<td>1400/1500/1600</td>
<td>120</td>
</tr>
<tr>
<td>Cooling</td>
<td>30</td>
<td>1000</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 2

The sintering cycle details.

<table>
<thead>
<tr>
<th>Change in temperature</th>
<th>Rate °C/Min</th>
<th>Temperature °C</th>
<th>Holding Time in Minutes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heating</td>
<td>25</td>
<td>800</td>
<td>7</td>
</tr>
<tr>
<td>Heating</td>
<td>15</td>
<td>1400/1500/1600</td>
<td>120</td>
</tr>
<tr>
<td>Cooling</td>
<td>30</td>
<td>1000</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 3

Mean, standard deviation (SD) values and results of one-way ANOVA and Tukey’s tests for comparison between translucency values after different immersion times.

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>Time</th>
<th>Group A</th>
<th>Group B</th>
<th>Group C</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1400</td>
<td>3 min</td>
<td>13.94</td>
<td>13.88</td>
<td>13.22</td>
<td>0.003</td>
</tr>
<tr>
<td>1500</td>
<td>3 min</td>
<td>14.28</td>
<td>13.77</td>
<td>13.78</td>
<td>0.287</td>
</tr>
<tr>
<td>1600</td>
<td>3 min</td>
<td>14.65</td>
<td>13.78</td>
<td>13.78</td>
<td>0.103</td>
</tr>
</tbody>
</table>

*: Significant at P ≤ 0.05. Different superscripts in the same row are statistically significantly different.

3. Statistical analysis

Data were presented as mean and standard deviation (SD) values. Data were explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests. All data showed parametric (normal) distribution.

One-way Analysis of Variance (ANOVA) was used to compare between the three immersion times as well as to compare between the three sintering temperatures. Tukey’s post-hoc test was used for pair-wise comparisons when ANOVA test is significant.

The significance level was set at P ≤ 0.05. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

4. Results

4.1. Effect of time

At 1400 °C, sintering temperature there was no statistically significant difference for between 3 min and 5 min immersion times where both showed the statistically highest mean translucency. Yet, 7 min immersion time showed the statistically significantly lowest mean translucency. On the other hand, at both 1500 °C and 1600 °C sintering temperatures there was no statistically significant difference between the three immersion times (Table 3) and (Fig. 1).

4.2. Effect of sintering temperature

After both 3 and 5 min immersion times, there was no statistically significant difference between the three sintering temperatures. For the 7 min immersion time, there was no statistically significant difference between 1500 and 1600 °C sintering temperatures where both showed the statistically significantly highest mean translucency. However, 1400 °C sintering temperature showed the statistically significantly lowest mean translucency (Table 4) and (Fig. 2).

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7 IBM Corporation, NY, USA.
8 SPSS, Inc., an IBM Company.
reproduction and translucency of monolithic zirconia. Translucent zirconia discs were sintered at three temperatures (1460 °C, 1530 °C, and 1600 °C) with three sintering holding times. Easy-shade spectrophotometer was used to obtain the ΔE between the specimens and the shade A3. Contrast ratio decreased as the sintering time and temperature increased. It was thus concluded that sintering zirconia using long cycles and high temperatures would result in better translucency.

On the contrary, Zhang et al [4] who worked on Cubic SPS zirconia proved that as the sintering temperature is raised, the absorption and scattering coefficients increased for both the as-sintered and annealed specimens which accounts for the decrease of transparency above 1100 °C.

The increase in translucency by raising the final sintering holding temperature is mainly due to the effect of sintering parameters on the microstructure and the crystalline phases [15]. Sintering can eliminate the inter-particle pores in a granular material by atomic diffusion driven by capillary forces. As the temperature rises, the particles are sintered together and pores on grain boundaries are reduced by solid-state diffusion and consequently the sintered density increases [16]. This reduction in the pores may be the main factor responsible for the increase in TP.

This was supported by the scanning electron micrographs of inCoris TZI slices at the three used different temperatures where microstructure revealed the very fine grains. Zirconia specimens sintered at 1400 °C had greater porosity and larger pores of irregular shapes were often located at the inter-boundary regions. With the increasing temperature, the zirconia crystal structure became more compact whereas porosity, defects, and flaws decreased. The

Table 4
Mean, standard deviation (SD) values and results of one-way ANOVA and Tukey’s tests for comparison between translucency values of different sintering temperatures.

<table>
<thead>
<tr>
<th>Time</th>
<th>Temperature</th>
<th>P-value</th>
<th>Group 1</th>
<th>Group 2</th>
<th>Group 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Mean</td>
<td>SD</td>
<td>Mean</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1460 °C</td>
<td>1530 °C</td>
<td>1600 °C</td>
</tr>
<tr>
<td>Subgroup A</td>
<td>3 min</td>
<td>13.94</td>
<td>0.17</td>
<td>14.28</td>
<td>0.73</td>
</tr>
<tr>
<td>Subgroup B</td>
<td>5 min</td>
<td>13.88</td>
<td>0.32</td>
<td>13.77</td>
<td>0.76</td>
</tr>
<tr>
<td>Subgroup C</td>
<td>7 min</td>
<td>12.75</td>
<td>0.75</td>
<td>13.68</td>
<td>0.22</td>
</tr>
</tbody>
</table>

*: Significant at P ≤ 0.05. Different superscripts in the same row are statistically significantly different.

Fig. 1. Bar chart representing comparison between mean translucency values after different immersion times.

Fig. 2. Bar chart representing comparison between translucency values of different sintering temperatures.
Grain size ranged from 250 to 700 nm at 1400 °C. At temperature of 1500 °C, the proportion of larger grains increased. However, at 1600 °C the grains grew more even and varied between 500 nm and 1.2 μm. It was also found that distinct grain boundaries were formed. This supported the fact that nano crystalline zirconia inCoris TZI can be sintered with fewer pores thus giving it the better translucency than regular zirconia core materials. Also, the increase in the sintered density of the zirconia may lead to a more uniform crystalline arrangement thus promoting better light transmission and penetration.

This agreed with the study done by Jiang et al [12] who showed that increasing the sintering temperature of zirconia from 1400 °C to 1500 °C led to more compaction, higher density, and less porosity. Likewise, Gómez S et al [17] stated that porosity decreased and density increased with increasing temperature where the relative density increased from 50% in the starting powders to 99% and null porosity at sintering temperature 1400 °C.

Results obtained from scanning electron micrographs were also in acceptance with Stawarczyk et al [13] who stated that the grain size of zirconia increased with higher sintering temperatures above 1300 °C, with the highest results perceived at 1700 °C. However, specimens with a final sintering temperature above 1600 °C were accompanied by hollow opening in the zirconia microstructure crystal. In this study, the grain size of zirconia increased with increasing sintering temperature.

Hao Chin Chuin et al [18] in their study showed that sintering temperature significantly influenced the density, light transmission, and microstructure of YSZ, where high sintering temperatures produced YSZ with a compact, homogeneous microstructure and a high density. This porosity-free microstructure exhibited light transmission as high as 37% in YSZ sintered at 1650 °C which was probably due to the effective densification of grains and elimination of pores at high temperatures, and consequently eliminating the light scattering effect of the pores.

6. Conclusions

Our study showed that the best optical properties were obtained by the combination between lower dipping times and higher sintering holding temperatures. The shade was affected by the dipping time and sintering temperature while the translucency parameter was more affected by the final sintering holding temperature. This was supported by the data obtained from the scanning electron micrographs.

7. Limitations of study

Our 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 dipping time and sintering temperature after aging. Authors also recommend that more studies would be carried out testing the effect of different sintering temperatures on the mechanical properties of nano-crystalline Y-TZP.

References


