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Effect of changes in sintering parameters on monolithic translucent zirconia

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ABSTRACT

Objectives. Aim of this study was to evaluate the effect of different sintering parameters on color reproduction, translucency and biaxial flexural strength of monolithic zirconia.
Methods. Translucent zirconia discs having 15 mm diameter, 1 mm thickness, and shade A3 were milled and divided according to the sintering temperatures (1460 °C, 1530 °C, and 1600 °C) into three groups ($n = 30$). Each group was later divided into three subgroups ($n = 10$) according to the sintering holding time (1, 2, and 4 h). Easyshade spectrophotometer (Vita, Bad Säckingen, Germany) was used to obtain the ΔE between the specimens and the shade A3. Mean ΔE values below 3.0 were considered "clinically imperceptible", ΔE values between 3.0 and 5.0 were considered "clinically acceptable" and ΔE values above 5.0 were considered "clinically unacceptable". Contrast ratio (CR) was obtained after comparing the reflectance of light through the specimens over black and white background. Biaxial flexural strength was tested using the piston-on-three balls technique in a universal testing machine.
Results. Mean ΔE results ranged from 4.4 to 2.2. Statistically significant decrease in the Delta E was observed as the sintering time and temperature increased. CR decreased from 0.75 to 0.68 as the sintering time and temperature increased. No significant change in the biaxial flexural strength was observed.
Significance. Sintering zirconia using long cycles and high temperatures will result in reduction of ΔE and CR. Biaxial flexural strength is not affected by changes in the evaluated sintering parameters.

1. Introduction

All ceramic restorations have proved to be a promising alternative to metal-ceramic restorations mainly due to their excellent esthetics, chemical stability, and biocompatibility [1]. Zirconia restorations became very popular due to their unique mechanical properties which made it possible to use them in long span restorations [2,3]. Zirconia is polymorphic in nature and exists in three forms: cubic, tetragonal, and monoclinic. At room temperature zirconia is present in its monoclinic form and is stable up to 1170 °C. Above this

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temperature a transformation occurs to the tetragonal phase that is stable up to 2370 °C. Beyond this temperature, zirconia assumes its cubic form [4].

Zirconia restorations are fabricated either in a partially sintered state by soft machining followed by a final sintering cycle, or they are fabricated in a fully sintered state by hard machining [5,6]. Hard machining may induce tetragonal-monoclinic transformation, introduce cracks, and wears the milling hardware at a higher rate. Soft machining however is much easier, but may produce less accurate frameworks due to the sintering shrinkage accompanied with the final sintering process [7].

The phenomenon of light scattering largely affects the translucency of dental ceramics. If the majority of light passing through a ceramic is scattered, the material will appear opaque. However, if most of the light passing is transmitted through the ceramic it will appear translucent [8]. The amount of light that is absorbed, transmitted, and reflected mainly depends on the microstructure of the ceramic itself [9,10].

Differences in perceived color (ΔE) can be determined using the CIELAB coordinates. The CIELAB system has provided a quantitative representation of color and it has been extensively applied in dentistry to study esthetic materials, shade guides, and color reproductions [11–13]. The perceptibility and acceptability thresholds of the ΔE vary widely in literature mainly due to the diversity of observers, objectives, and methodologies among the studies [14,15]. Clinically the tooth, restorations available, surrounding, and blending effect tend to expand the clinically acceptable range previously reported [16,17]. The mean ΔE values as "clinically imperceptible" ($\Delta E < 3$), "clinically acceptable" (ΔE between 3 and 5) and "clinically unacceptable" ($\Delta E > 5$) seem to be consistent with the clinical practice considering a non-color expert, which usually is the patient's condition [11,18,19].

In order to overcome the main disadvantage of zirconia which is its opacity, the zirconia core is veneered with veneering porcelain to enhance its esthetics. However, the most common mode of failure that faced clinicians was the chipping of this veneering porcelain while the zirconia core remained unaffected [20,21].

The differences in sintering parameters of zirconia can directly affect its microstructure and properties [22]. The extent of this effect have become of interest in the field of dental research especially after the introduction of short sintering cycles by manufacturers. Several authors have studied the effect of the changes in sintering time and temperature on the translucency, grain size, and biaxial flexural strength of zirconia core ceramics; however the effect of these changes on the properties of monolithic nanozirconia remains in question [10,23–25].

The aim of this study was to evaluate the effect of using different sintering times and temperatures on the color reproduction, translucency, surface roughness, biaxial flexural strength, and the surface hardness of monolithic zirconia ceramic.

2. Materials and methods

Ninety translucent shaded zirconia ceramic discs (Bruxzir, Glidewell, Frankfurt, Germany) with a diameter of 15 mm, a thickness of 1 mm, and shade A3 were milled and divided into three groups ($n = 30$) according to the sintering holding time (1, 2, and 4 h). Each group was later divided into three subgroups ($n = 10$) according to the sintering temperature (1460 °C, 1530 °C, and 1600 °C). All specimens were sintered as milled in the manufacturers sintering furnace (Bruxzir Fast-Fire, Glidewell, Frankfurt, Germany) at a heating and cooling rate of 10 °C per minute. The temperature was controlled using the furnace's internal thermometer.

2.1. Color evaluation

Specimens were placed over a neutral gray background (CIE $L^* = 62.1$, $a^* = 1.3$, $b^* = -0.02$) and the CIELAB coordinates were measured for each specimen using a spectrophotometer (Easyshade compact, Vita Zahnfabrik, Bad Säckingen, Germany). The Easyshade was set to the restoration mode and the shade A3 was selected. In this mode the color difference is determined by comparing the selected shade and the measured shade. For each specimen three measurements were taken at the center and their average was recorded. After each specimen was measured the Easyshade was recalibrated. Mean ΔE values below 3.0 were considered "clinically imperceptible", ΔE values between 3.0 and 5.0 were considered "clinically acceptable" and ΔE values above 5.0 were considered "clinically unacceptable".

2.2. Translucency evaluation

A quantitative measurement of translucency was obtained by measuring the CIELAB coordinates of the specimens after backing with a white (CIE $L^* = 96.7$, $a^* = 0.1$, $b^* = 0.2$) and black (CIE $L^* = 10.4$, $a^* = 0.4$, $b^* = 0.6$) background using the spectrophotometer. For each specimen three measurements were taken and their average was recorded. The contrast ratio (CR) for each specimens was calculated according to the following equation: $CR = Y_b/Y_w$ where $Y = [(L + 16)/116]^3 \times 100$ and Y_b is the reflectance over a black background and Y_w is the reflectance over a white background [8,11]. In all calculations "0" is considered the most transparent and "1" is considered the most opaque.

2.3. Surface roughness evaluation

All specimens were cleaned ultrasonically in 99% isopropanol solution for 3 min and then dried with air. The average surface roughness (Ra) for the specimens was measured using a 3D laser scanning microscope (Keyence VK-X100, Keyence GmbH, Neu-Isenbuerg, Germany). The wavelength of the laser was 658 nm. Three separate areas were measured on each specimen, the measured area was 500 μm × 750 μm and the distance between the separate scans was over 3 μm. The mean Ra for each specimen was later recorded.

2.4. Microstructure analysis

Three specimens were selected randomly from each subgroup for X-ray diffraction (XRD) surface analysis to detect the amount of tetragonal and monoclinic phases available. The specimens were placed in the holder of a diffractometer