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Original Article

Effects of sintering protocols, yttria content, and zirconia thickness on the optical properties of monolithic zirconia

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Abstract

Purpose: This laboratory-based study evaluated the effects of sintering protocols, yttria content, and zirconia thickness on the optical properties of monolithic zirconia.
Methods: Three partially stabilized zirconia (PSZ) materials, one monolayer (HT) and two multilayer (GE, FX) samples, with thicknesses of 0.5-1.5 mm were tested under conventional and speed sintering protocols. Translucency parameter (TP), color difference (ΔE_{00}), and spectral reflectance were measured using a spectrophotometer against standardized black and white backgrounds. Statistical analyses were performed using a one-way analysis of variance, Tukey's *post-hoc* tests, and correlation analysis.
Results: The TP values decreased with increasing zirconia thickness; significant differences were observed between the sintering protocols. Speed sintering resulted in lower transparency in certain materials, particularly GE. Conversely, HT exhibited consistent optical properties across the sintering protocols. The ΔE_{00} values for multilayer zirconia exceeded clinically acceptable thresholds, with greater variations observed for thicker specimens. Spectral reflectance curves were minimally influenced by the sintering protocol and thickness for monolayer zirconia, whereas they showed significant variations for multilayer zirconia.
Conclusion: The results indicate that sintering protocols and material composition significantly influence the optical properties of zirconia, underscoring the necessity of optimizing processing conditions to enhance both esthetic and functional performance in clinical applications.

Keywords: color difference, computer-aided design-computer-aided manufacturing, esthetic dentistry, spectral reflectance, translucency parameter

Introduction

Zirconia is a fundamental material used in prosthetic dentistry owing to its superior mechanical properties, biocompatibility, and esthetics [1]. When zirconia was first introduced in dentistry approximately 20 years ago, tetragonal zirconia polycrystals (TZP) containing 3 mol% yttria (3Y-TZP) were primarily used as frameworks. However, zirconia frameworks are typically veneered with porcelain because of their white color and limited esthetic appeal [2]. The transformation toughening mechanism of the material provides excellent crack resistance and durability, making it suitable for high-load posterior regions [3]. Since 2011, high-translucency zirconia with reduced alumina content and improved optical properties has been developed [4]. This advancement has improved the clinical success rate and enabled its use as a monolithic material for molar restorations. However, its esthetic limitations make it less suitable for anterior applications. To this end, ultra-high-translucency zirconia has been introduced, offering significantly enhanced transparency that allows for monolithic restorations in the anterior regions [4].

Ultra-high-translucency zirconia is a partially stabilized zirconia (PSZ) with an increased yttria content, ranging from approximately 4 mol% (4Y) to 6 mol% (6Y). Its microstructure comprises a combination of tetragonal and cubic crystal phases, which contribute to its high transparency and improved esthetics. However, these esthetic improvements come at the cost of reduced flexural strength, which is approximately half that of the conventional 3Y-TZP [4]. To further enhance esthetics, multilayered zirconia materials have been developed [5]. These materials incorporate layers of various shades to mimic the appearance of natural teeth. Monocomposition discs, which have a uniform homogeneity, are available as shade-gradation discs in 3Y-TZP, 4Y-PSZ, or 5Y-PSZ compositions. Alternatively, mixed-composition discs are designed with high-strength 3Y-TZP or 4Y-PSZ used for the cervical layer and highly translucent 4Y-PSZ or 5Y-PSZ used for the incisal layer, striking a balance between strength and esthetics.

Increasing the yttria content in zirconia enhances its translucency, which is a critical parameter for esthetic applications [3,6]. These findings have driven the development and widespread adoption of high-translucency zirconia in clinical practice [7,8]. However, sintering remains a time-intensive step in zirconia production, often requiring several hours to achieve optimal mechanical and optical properties. Recent advancements in speed sintering protocols have shown the potential to significantly reduce processing time while maintaining material performance [9].

With the integration of intraoral scanning, computer-aided design-computer-aided manufacturing (CAD-CAM) technology, and high-speed sintering furnaces, it is now feasible to perform single-visit treatments with highly translucent zirconia employed to make monolithic zirconia (MZ) crowns [10]. Shorter sintering times allow for shorter treatment times, fewer treatment appointments, and lower costs [11]. However, rapid sintering can produce zirconia materials with large grains and increased porosity [12]. Additionally, shade-gradated zirconia materials with mixed compositions (3Y-TZP and 5Y-PSZ) exhibit color differences depending on the sintering method employed (conventional vs. speed sintering) [13].

The final color of dental ceramics is influenced by multiple factors, including the material transparency [14], thickness [6,15], and spectral reflectance of the surface [15]. Hence, a careful selection of zirconia discs and the use of stains to adjust the shades are essential for achieving color consistency with the adjacent teeth. Despite these advancements, limited information is available on the effects of sintering protocols on the transparency and color of zirconia with varying yttria content. Although rapid sintering has evident time-saving benefits, its impact on the esthetic and optical properties of the material remains insufficiently explored.

This study aimed to evaluate the influence of different sintering protocols, yttria content, and zirconia thickness on translucency parameter (TP), color difference (ΔE_{00}), and spectral reflectance of sintered zirconia. The null hypothesis of this laboratory-based study was that the sintering protocol, yttria content, and zirconia thickness have no significant effect on the optical properties of the material.

Materials and Methods

Three types of PSZ materials with varying yttria contents were selected (Table 1). The materials used included one type of monolayer zirconia (HT:

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Table 1 Materials used

	Code	Materials	Manufacturer	Type of materials	Shade	Batch code
Monolayer	HT	Ceramill zolid HT+ Preshade	Amann Girrbach	4Y-PSZ	A2	1808000
Multilayer	GE	Ceramill zolid Gen-x	Amann Girrbach	4Y-PSZ, 5Y-PSZ	A2	2209000
	FX	Ceramill zolid FX Multilayer	Amann Girrbach	5Y-PSZ	A2/A3	2209004

Table 2 Sintering parameters for the conventional and speed sintering

	Code	Heating rate (°C/min)	Temperature (°C)	Holding time (min)	Sintering time (min)
Conventional		8	1,450	120	480
Speed	HT	60	1,450	65	120
	GE	250	1,580	5	21
	FX	60	1,450	65	120

Ceramill zolid HT+ Preshade, Amann Girrbach, Koblach, Austria) and two types of multilayer zirconia (GE: Ceramill zolid Gen-x, and FX: Ceramill zolid FX Multilayer, Amann Girrbach). Specimens were fabricated using a dental CAD-CAM system (Ceramill DNA Generation, Amann Girrbach). Each specimen was cut from a 20-mm-thick zirconia disk, and three plate specimens were prepared for each material with the final dimensions after sintering being 15 mm × 15 mm and thicknesses being 0.5, 1.0, and 1.5 mm ($n = 8$ per thickness). Sintering was performed in a sintering furnace (Ceramill Therm 3, Ceramill Therm DRS, Amann Girrbach) following the manufacturer's instructions under both conventional and speed sintering protocols (Table 2). The conventional sintering process involved gradual heating to a peak temperature of 1,450°C, with a total cycle time of approximately 8 h. Depending on the zirconia material, the speed of the sintering process was varied, while following manufacturer-specific protocols. After the sintering, all the specimens were wet-ground using #500 grit silicon carbide (SiC) abrasive paper (Silicon Carbide Grinding Paper, Struers, Copenhagen, Denmark), followed by wet-grinding with #1,200 grit SiC abrasive paper to achieve a uniform surface finish.

A spectrophotometer (CM-700d, Konica Minolta, Tokyo, Japan) was used to estimate the TP, ΔE_{00}, and spectral reflectance values of each specimen, in accordance with the CIE 1976 L*a*b* color scale relative to the CIE standard illuminant D65 (as defined by the International Commission on Illumination).

Measurement of TP values

Each specimen was positioned on standardized black ($L^* = 4.7$, $a^* = -0.1$, $b^* = 0.0$) and white ($L^* = 98.1$, $a^* = -0.5$, $b^* = 2.8$) backgrounds, and TP was determined as the difference in the reflectance between the two backgrounds. Measurements were conducted at two locations: on the cervical and incisal layer sides. The measurement point was set at the center of each layer, 5 mm from the edge of the specimen. To ensure accuracy, the measurements were repeated thrice per specimen, and the average TP value was recorded.

The TP values were calculated from the L*, a*, and b* values using the following formula:

$$TP = [(L_W^*-L_B^*)^2 + (a_W^*-a_B^*)^2 + (b_W^*-b_B^*)^2]^{1/2}$$

where W and B denote the measurements obtained for the specimens placed on the white and black backgrounds, respectively.

Measurement of ΔE_{00}, and spectral reflectance

Color measurements were conducted to evaluate the color differences resulting from the different sintering protocols applied to the zirconia specimens. The measurements were performed against a standardized white background, with three measurements taken per specimen. The average of the measurements was recorded as the final value. For the single-layer zirconia specimens, measurements were performed at the center of the specimen. For multi-layer zirconia specimens, measurements were taken at two distinct locations, one on the cervical side and the other on the incisal side, following the same protocol used for TP measurements.

The color differences (ΔE_{00}) between specimens subjected to conventional sintering and speed sintering were calculated using the CIEDE2000 formula:

$$\Delta E_{00} = \{[(L_i\text{-}L_j/K_LS_L)]^2 + [(C_i\text{-}C_j/K_CS_C)]^2 + [(H_i\text{-}H_j/K_HS_H)]^2 + R_T[(C_i\text{-}C_j/K_CS_C)] \times [(H_i\text{-}H_j/K_HS_H)] \}^{1/2},$$

where L represents lightness, C represents chroma, H represents hue, and subscripts i and j denote values obtained under different sintering conditions. Moreover, S_L, S_C, and S_H are the weighting functions; K_L, K_C, and K_H are the parametric factors (set to 1 in this study); and R_T is the rotation term [16]. The ΔE_{00} values quantify perceptible color differences, with higher values indicating greater deviations. The reflectance values for all the specimens were measured at intervals of 10 nm in the wavelength range of 400-700 nm using a standardized black background plate ($L^* = 4.7$, $a^* = -0.1$, $b^* = 0.0$) [17]. Each measurement was repeated thrice, and the average value was recorded as the representative result.

Statistical analysis

The total sample size was determined using statistical software (G*Power 3.19.7, Heinrich Heine University Düsseldorf, Düsseldorf, Germany) with consideration of α error (0.05), power (0.05), mean of difference (0.81), and standard deviation (SD) of difference (0.9), which were based on the results of previous studies [16].

The Shapiro-Wilk W test was primarily used in this study to determine if the distribution was normal. For the TP analysis, Welch's *t*-test was used to compare the sintering protocols at the same measurement points for the same material. For ΔE_{00}, Bartlett's test was employed to assess color differences due to variations in sintering protocols at the same measurement points for the same material across different thicknesses. When the measured values demonstrated homoscedasticity, the Tukey-Kramer honestly significant difference (HSD) test was conducted following a one-way analysis of variance (ANOVA). In cases where homoscedasticity was not observed, the Steel-Dwass test was used. A significance level of $P < 0.05$ was considered statistically significant. All the statistical analyses were performed using the JMP Pro software (version 17.0.0; SAS Institute, Cary, NC, USA).

Results

Figure 1 presents the TP values for all specimens, demonstrating a decrease with increasing specimen thickness. A comparison of TP values between sintering protocols revealed a tendency toward lower transparency in the speed-sintered GE samples. The statistical analyses revealed significant differences in the TP values between sintering protocols under specific conditions: 0.5 mm thickness for the HT sample ($P = 0.0096$), all the conditions for the GE sample ($P < 0.05$), 0.5 mm thickness for the FX_I sample ($P = 0.0039$), and 3.0 mm thickness for the FX_C sample ($P = 0.0033$). For the GE and FX specimens, the TP values generally did not exhibit significant differences between the measurement points under the same conditions.

The ΔE_{00} values for the GE were greater than 3 across all the conditions, and its ΔE_{00} values increased with specimen thickness. In contrast, the ΔE_{00} values for HT and FX ranged from 1.0 to 1.7, and no consistent trend in ΔE_{00} was observed with changes in the thickness (Fig. 2). For HT, a significant difference in ΔE_{00} was observed between the 3.0 mm thickness and both the 0.5 mm and 1.5 mm thicknesses ($P < 0.01$). In both the GE_C and GE_I samples, significant differences in ΔE_{00} were noted for each thickness ($P < 0.01$) (Fig. 3). However, thickness variations did not produce any significant differences in the ΔE_{00} values of the FX_C and FX_I samples ($P > 0.05$).

Figures 4-6 show the effects of the specimen thickness and sintering protocols on the spectral reflectance curves. The standard deviation-to-mean ratios for each data point on the curves ranged from 3% to 8%. Across all the specimens, the spectral reflectance curves showed an increasing trend in the wavelength range of 400-460 nm, a decrease at approximately 520 nm (green) and 650 nm (red), followed by an increase and transition to a flat line. For the HT and FX specimens, the spectral reflectance curves remained unaffected by the material thickness or sintering protocol. In contrast, the GE specimens subjected to speed sintering with thicknesses of 1.5 and 3.0 mm on both the cervical and incisal sides exhibited spectral reflectance curves approximately 20% higher than those under the other conditions.

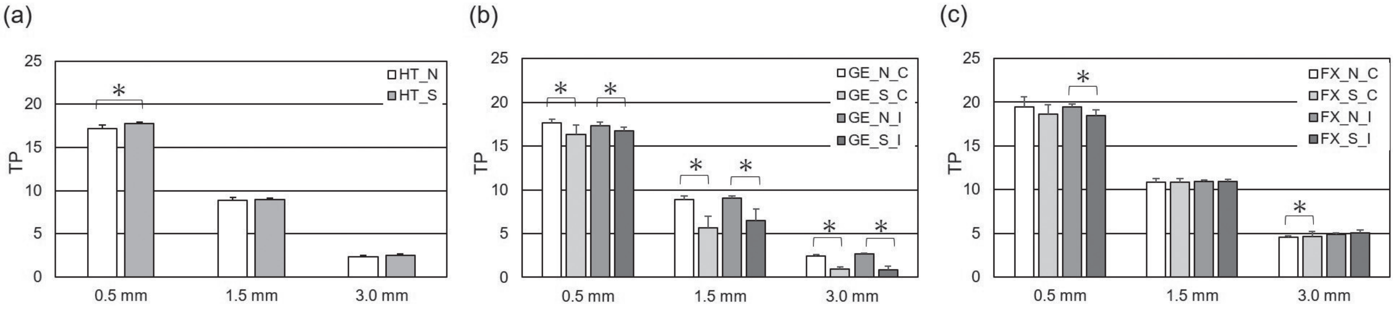

Fig. 1 TP values of zirconia with different thicknesses
*Significant difference between conventional sintering and speed sintering within the same thickness. (a) HT, (b) GE, (c)FX. N: normal (conventional), S: speed, C: cervical side, I: incisal side

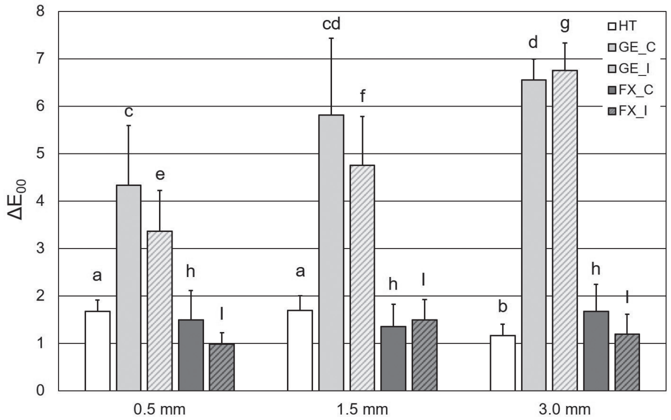

Fig. 2 Color difference (ΔE_{00}) between conventional sintering and speed sintering for the same material and thickness
Different letters indicate significant differences between the same material and same sintering program ($P < 0.05$). C: cervical side, I: incisal side

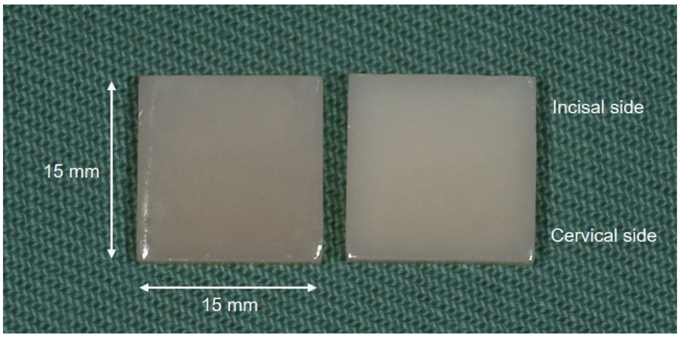

Fig. 3 GE sample with 3.0 mm thickness
left: conventional sintering, right: speed sintering

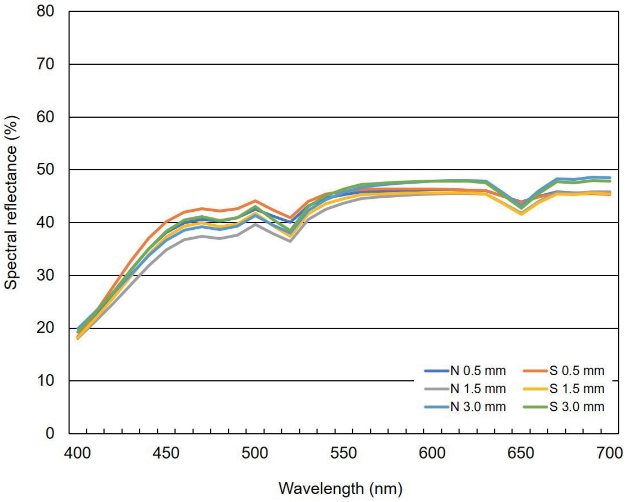

Fig. 4 Spectral reflectance of HT

Discussion

A laboratory-based study was conducted on monolithic zirconia samples to determine the influence of various parameters on their optical properties. The statistical analysis of the experimental results led to the partial rejection of the null hypothesis. For the HT and FX specimens, the transparency, ΔE_{00}, and spectral reflectance were generally within acceptable ranges, whereas the GE specimens were rejected under all conditions. The transparency, ΔE_{00}, and spectral reflectance curves were evaluated using a spectrophotometer across two zirconia materials, three material thicknesses, and two sintering protocols. A black background was employed to simulate the darkness of the oral cavity because all the specimens were translucent at clinically relevant thicknesses. The use of a spectrophotometer to assess the color by measuring the CIE Lab* color coordinates of specimens is a well-established method in dental research [18]. This technique provides unbiased and objective numerical data, allowing for the precise evaluation of colors. The obtained coordinates can be used to calculate the color differences between specimens or objects, enabling the assessment of their perceptual similarity and clinical acceptability [19]. In color science, color-difference formulae have evolved from the simple CIEDE76 to the more complex and accurate CIEDE2000, which assigns appropriate weights to color coordinates and attributes. In this study, the CIEDE2000 formula was applied with parameters $K_L = 1$, $K_C = 1$, and $K_H = 1$ [16]. Previous studies have shown that an excellent color match can be achieved when $\Delta E_{00} \leq 0.8$, while a clinically acceptable match is obtained when $0.8 < \Delta E_{00} \leq 1.8$ [20].

The sintering process significantly affects the properties of zirconia ceramics by altering their crystalline phases and microstructure [21]. Consequently, sintering conditions, including the temperature, holding time, and total sintering duration, can affect the optical properties of zirconia restorations [22]. In speed sintering, a rapid temperature increase and a shorter holding time may result in distinct sintering behaviors between layers with different yttria contents [22]. This can suppress or incompletely facilitate the phase transformation, particularly in layers with a predominantly tetragonal crystal structure. In the 4 mol% yttria layer, where the tetragonal phase is dominant, the rapid temperature changes associated with speed sintering may significantly affect the phase transformation. In contrast, the 5 mol% yttria layer, which predominantly contained a stable cubic phase, was less affected by phase transformations. For the GE specimens, the variations in the crystal grain growth and sintering density across the layers may have resulted in differences in the transmittance and color between the 4 mol% and 5 mol% layers. This disparity may contribute to the observed increase in the overall ΔE_{00}. Speed sintering tends to exacerbate nonuniform grain growth and the density between layers compared

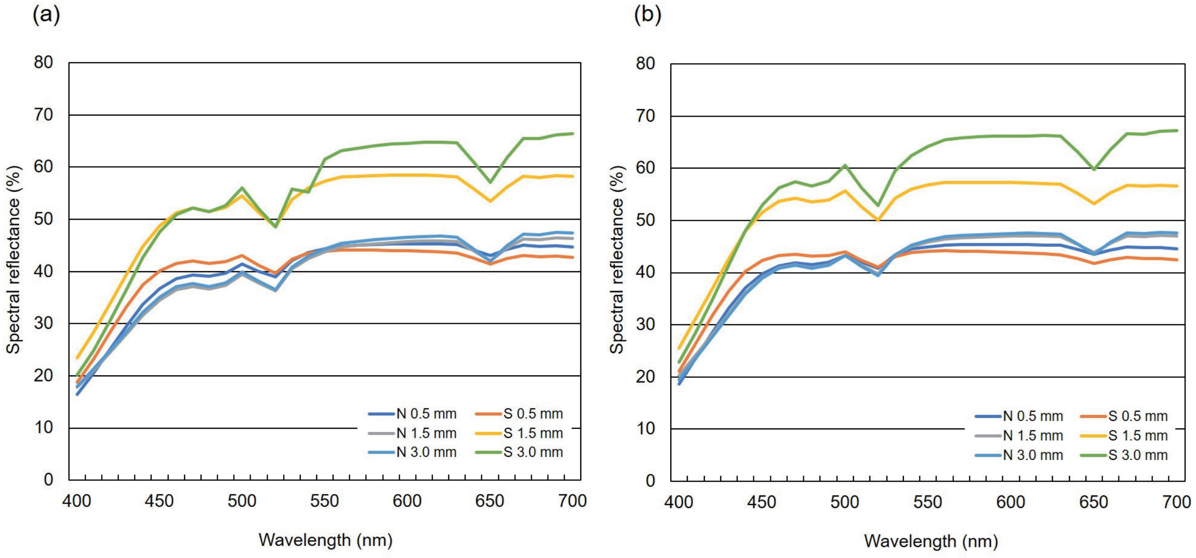

Fig. 5 Spectral reflectance of GE for cervical side (a), and incisal side (b)

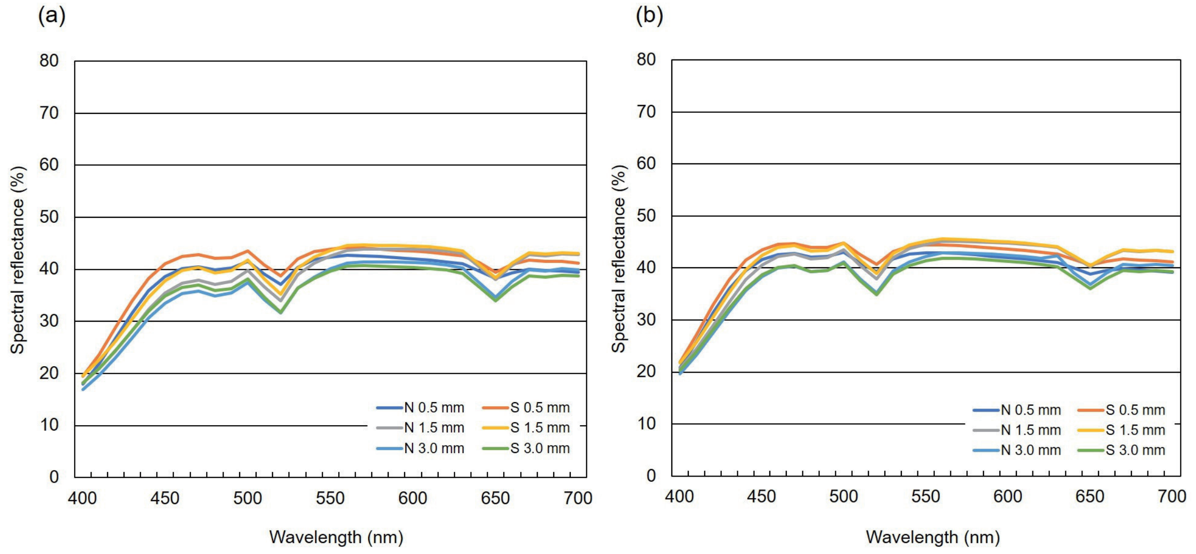

Fig. 6 Spectral reflectance of FX for cervical side (a), and incisal side (b)

with conventional sintering, leading to increased light refraction and scattering, which further affects the optical properties of the material [7]. As a result, the overall ΔE_{00} values may be higher. Additionally, the zirconia used in this study contained oxide-based colorants designed to replicate the color of natural teeth. During speed sintering, uneven oxidation reactions and volatilization of these colorants across different layers, caused by significant temperature fluctuations, may lead to color variations. Consistent with previous research, which compared the color differences between conventionally sintered and speed-sintered mixed-composition layered zirconia (3Y-TZP, and 5Y-PSZ), higher ΔE_{00} values were reported than those observed for mono-composition layered zirconia [13]. These findings align with the results of the present study. These differences were perceptible to the human eye. Although the material compositions differed between the studies, it is likely that the presence of distinct layers contributed to the observed color differences.

HT is a mono-composition type zirconia with an yttria content of 4 mol% and is characterized by a high proportion of tetragonal phases. A gradual increase in the temperature during conventional sintering is crucial for maintaining the stability of the tetragonal phases and achieving optimal strength and transparency. In contrast, during speed sintering, an insufficient temperature control may result in an incomplete sintering of the tetragonal phase. On the other hand, FX is also a mono-composition type zirconia with an yttria content of 5 mol%, making it less prone to the structural inconsistencies between layers during sintering. Regardless of the type of sintering method employed (conventional or speed sintering), the sintering process for FX tended to be uniform across the layers. Even under high-speed sintering conditions, the mono-composition nature of FX reduced the likelihood of an inhomogeneous sintering behavior, and the ΔE_{00} values after sintering were relatively low. Therefore, FX appears to be a suitable choice for clinical applications, where esthetics and consistency in the sintering process are essential.

The TP is closely influenced by the particle size, yttria content, and proportion of chemical impurities in zirconia [23]. When light interacts with the zirconia surface, a part of it is reflected, most of it is scattered at the grain boundaries and internal defects, and the remaining light passes through the pores [7]. The transparency of zirconia increases with higher yttria content and larger particle sizes [24,25]. In this study, the HT and FX samples showed a trend toward higher TP values, particularly the FX sample, which was classified as 5-PSZ. FX exhibits TP values ranging from 10.8 to 10.9. Although these values differ between products, they are slightly lower than the reported TP values (12-13) for 5Y-PSZ at a thickness of 1.5 mm [25,26]. These findings suggest that the TP values may vary depending on the zirconia product. For comparison, the TP values of human enamel and dentin with a thickness of 1.0 mm are 18.1 and 16.4, respectively [27]. The zirconia used in this study, with a thickness of 0.5 mm, exhibited a transparency comparable to those of human enamel and dentin.

The shapes of the spectral reflectance curves for the HT and FX samples were minimally influenced by the specimen thickness. The results obtained in this study are consistent with the spectral reflectance behavior reported for ceramics in previous research [14,17]. Studies, including ours, have demonstrated that the reflectance is lower at shorter wavelengths and increases at longer wavelengths [14,17]. Changes in the magnitude of the spectral reflectance values are known to influence the L* coordinates, whereas changes in the shape of the spectral reflectance curve affect the a* and b* coordinates [28]. In the case of the GE sample, the color difference between conventional and speed sintering was more pronounced with increasing specimen thickness. This suggests that the change in the spectral reflectance between fast-sintered specimens with thicknesses of 1.5 and 3.0 mm is likely greater. Therefore, the fact that GE samples with thicknesses of 1.5 and 3.0 mm exhibited larger color differences could have been strongly influenced by the change in L*.

A limitation of this laboratory-based study is that the three zirconia products investigated were from a single manufacturer. The obtained results may have differed if zirconia products from other brands were included in the study. Furthermore, no changes in mechanical properties or crystalline phases associated with phase transitions were detected in the tested materials during speed sintering, emphasizing the necessity for further investigation in this area. In conclusion, this study demonstrated that the TP, color difference, and spectral reflectance of zirconia with varying yttria content are influenced by the sintering protocol and specimen thickness. For mixed-composition zirconia, significant differences in transparency between conventional and high-speed sintering were observed compared with mono-composition zirconia. The color differences were also sufficient to be perceptible, and significant differences were identified based on the specimen thickness.

Abbreviations

ANOVA: analysis of variance; CAD-CAM: computer-aided design and computer-aided manufacturing; HSD: honestly significant difference; MZ: monolithic zirconia; PSZ: partially stabilized zirconia; SD: standard deviation; TP: translucency parameter; TZP: tetragonal zirconia polycrystal; ΔE_{00}: color difference; 3Y: 3 mol% yttria; 4Y: 4 mol% yttria; 5Y: 5 mol% yttria; 6Y: 6 mol% yttria

Ethical Statements

Not applicable

Conflicts of Interest

The authors declare that they have no conflicts of interests.

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Author Contributions

SM: conceptualization, investigation, data curation, formal analysis, funding acquisition, writing, and editing; ST: methodology, investigation, formal analysis, writing, and review; TF: investigation, formal analysis, and review; MF: formal analysis, review, and supervision; PV: conceptualization, review, and supervision; LL: methodology, review, and supervision. All authors read and approved the final version of the manuscript.

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Data Availability Statements

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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