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Characterization of 3Y-TZP/TiO_2 hybrid experimental dental ceramics

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ABSTRACT

The addition of titania to zirconia dental implants has been considered a promising choice to improve its bioactivity. This study aimed to evaluate the effect of different sintering conditions on the microstructure, density, optical properties and flexural strength of a 3Y-TZP/TiO_2 dental ceramic based on zirconia with two different titania contents (7.5 mol% and 12.5 mol%). 3Y-TZP/TiO_2 ceramic powders were synthesized by coprecipitation, uniaxially pressed and sintered at six different sintering conditions. Microstructural analysis of the sintered samples was performed by scanning electron microscopy and X-ray diffraction. Optical properties were measured using a spectrophotometer. The density was determined by Archimedes principle. Flexural strength was estimated by the biaxial flexure device. The microstructure and flexural strength of the 3Y-TZP/TiO_2 dental ceramic with 7.5% and 12.5 mol% were affected by the sintering conditions. Sintering the specimens at 1460 °C for 2 h increased the grain size and significantly decreased the flexural strength of 3Y-TZP/TiO_2 dental ceramic. The interaction (titania content x sintering conditions) affected the relative density and optical properties. A relative density greater than 98% was achieved for the T7.5 groups (sintered at 1260 °C/1 h, 1300 °C/1 h and 1300 °C/2 h) and for the T12.5 groups (sintered at 1260 °C/1 h, 1260 °C/4 h, 1300 °C/1 h and 1300 °C/2 h). The highest values of L*, a* and b* were respectively 87.2 (T7.5 group sintered at 1460 °C/2hs), 4.3 (T12.5 group sintered at 1300 °C/2hs) and 15.8 (T12.5 group sintered at 1300 °C/1 h). The material developed with 12.5 mol% of titania and sintered at 1300 °C/2 h showed high densification, flexural strength of 670 MPa and has good potential to be used in dentistry.

1. Introduction

Implant supported dental prostheses increased the quality of life of patients, often recovering their self-esteem and social (re)integration [1, 2]. The gold standard material to replace a tooth root is titanium alloy which has excellent mechanical behavior and very good osseointegration properties [3]. It is currently expected that biomaterials used in oral rehabilitation, in addition to showing high clinical longevity concerning functional and biological aspects, should also mimic the natural dentition as much as possible [4]. Titanium has a grayish color which often leads to visible shadowing of tissues surrounding implants. This might compromise the aesthetic result, especially in clinical cases involving anterior teeth of patients with thin gingival biotype [5]. In clinical situations in which the aesthetic condition is required, the use of yttria-stabilized tetragonal zirconia ceramic implants (3Y-TZP) is an alternative. By having a whitish color, such materials allows an oral rehabilitation with better aesthetic results and patient satisfaction [6,7].

Along with good esthetics, 3Y-TZP offers excellent biocompatibility and high fracture toughness [8–10]. The great ability of zirconia to resist crack propagation is related to its high crystalline phase content and mostly to a toughening mechanism known as martensitic transformation [8,11,12]. This phenomenon occurs when stresses originating from mastication are concentrated close to pre-existing defects. This triggers a transformation from tetragonal zirconia grains to monoclinic grains. Simultaneously, a volumetric expansion occurs due the phase transformation, resulting in compressive stresses at the crack tip. This

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behavior hinders crack propagation and growth of defects, therefore improving the mechanical behavior of 3Y-TZP [8,11,13,14].

On the other hand, the interaction of 3Y-TZP with living tissues is limited, being considered as biologically inert materials. Such feature negatively affects the osseointegration of dental implants, and can lead to implant loss and therefore decrease the longevity of oral rehabilitations [15,16]. Several studies aimed to improve the mechanical behavior and bioactivity of 3Y-TZP implants by means of developing ceramic dental implants with faster and more reliable osseointegration [17,18]. It is reported the addition of different ceramics in the zirconia matrix, such as titania, hydroxyapatite, bioactive glasses and alumina/ceria showing promising results [19–22].

Titania is a bioactive material and can be successfully used together with zirconia to form 3Y-TZP/TiO_2 [23–25]. Such dental ceramics has the ability to form hydroxyapatite when immersed in simulated body fluid (SBF) and also shows suitable adhesion, proliferation and differentiation of cells related to osseointegration [19,23,25]. In addition to this osteogenic effect, the 3Y-TZP/TiO_2 has been studied in relation to shape memory behavior [26]. New dental ceramics with ductile behavior can better withstand mechanical damage and, consequently, would allow clinicians and patients to perform oral rehabilitation with greater longevity [18,22].

The development of dental ceramics is usually carried out by performing some experiments in order to characterize densification, microstructure, mechanical and optical properties of the sintered materials [18,19,27,28]. Previous studies have performed an characterization of the 3Y-TZP/TiO_2 with the addition until 60 mol% of titania [23–25].However the mechanical properties of this material are not high enough for manufacturing ceramic dental implants that are more aesthetic, biocompatible and structurally reliable [19,23,25]. Thus, the objective of this study was to evaluate the effect of different sintering conditions on the microstructure, density, optical properties and flexural strength of a 3Y-TZP/TiO_2 dental ceramic containing two small titania contents (7.5% or 12.5 mol%). The null hypotheses were as follows: (I) the titania content would not affect the properties studied; (II) the sintering conditions would not affect the properties studied; and (III) the interaction (titania content x sintering conditions) would not affect the properties studied.

2. Experimental procedures

3Y-TZP/TiO_2 powders with two different titania contents (7.5 and 12.5 mol%, Table 1) were synthesized by coprecipitation method from zirconium oxychloride, titanium chloride and yttrium chloride [25,29]. The suspensions obtained after coprecipitation with ammonium hydroxide were filtered and washed using water, ethanol and n-butanol. The ceramic powders were dried at 60 °C, calcined at 800 °C/1 h, (Fornitec, Brazil) and milled in a high energy mill for 4hs using ethanol and Y-TZP beads. After milling, the suspension was again dried at 60 °C.

The ceramic powder was uniaxially pressed at 50 MPa (n = 5 for each group and N total = 60) into disks and sintered (Lindberg, SPX Thermal Product Solutions, White Deer, USA) at six different sintering conditions: 1260 °C/1 h, 1260 °C/2 h, 1260 °C/4 h, 1300 °C/1 h, 1300 °C/2 h and 1460 °C/2 h. These sintering conditions were based on preliminary dilatometry data and previous studies [24,25,30]. After grounded in a semi-automatic polisher (Ecomet II, Buehler, USA) with a diamond disc (Dia-Grid Diamond, 120 Grit Resin Bond, 12, Allied, USA), the samples had a diameter of approximately 12 mm and a thickness of 1.1 mm.

Table 1
Composition of 3Y-TZP/TiO_2 ceramic powder.

Groups	Composition (mol%)	
	3Y-TZP	TiO_2
T7.5	92.5	7.5
T12.5	87.5	12.5

Microstructural analysis was performed by scanning electron microscopy (SEM, Quanta 650 FEG, FEI, USA) and X-ray diffraction (Rigaku, DMAX 3000, Japan) with $Cuk_{\alpha}1$ radiation with an angular step of 0.02° and acquisition time of 5 s. To visualize the intergranular boundaries, the specimens were polished and thermal etched at 50 °C below the sintering temperature for 30 min. The grain size was estimated by the linear intercept method based on SEM micrographs. Optical properties were measured using a CM 3700d spectrophotometer (Konica Minolta, Japan) in the visible light range (360–740 nm) and with the following standardized parameters: observer function at 2° and D65 illuminant (daylight). The color difference (ΔE_{00}) was calculated between the two titania contents using the CIEDE2000 equation [31]. The density of the specimens was determined by the Archimedes principle and estimated as a percentage in relation to the theoretical density, that was calculated by Rietveld refinement. For the biaxial flexural strength evaluation, the specimens were fractured in a piston on three balls device in a Universal testing machine (EMIC DL 2000, Brazil) with a loading rate of 0.5 mm/min and immersed in water at 37 °C according ISO 6872/2008 [32].

The data analysis involved descriptive statistics and normality test. Data from relative density, flexural strength and optical parameters (L*, a* and b*) were statistical evaluated by analysis of variance two way, followed by the Tukey's test. Data from color difference were statistical evaluated by analysis of variance one way, followed by the Tukey's test. The level of significance was set at 5% at Jamovi Computer Software (Version 2.0, Australia).

3. Results

3.1. Microstructure

Fig. 1 shows scanning electron micrographs of the specimens after polishing and thermal etching. It is possible to observe multiple crystalline grains for both groups, according to titania content (7.5 and 12.5 mol%). For the sintering conditions of 1260 °C/1 h (Fig. 2a and b), 1260 °C/2 h (Fig. 2c and d), 1260 °C/4 h (Fig. 2e and f), 1300 °C/1 h (Fig. 2g and h) and 1300 °C/2 h (Fig. 2i and j) the zirconia grain sizes varied from 0.1 to 0.15 μm. For the sintering condition of 1460 °C/2 h (Fig. 2k and l) the grains were larger: 0.6 μm for T7.5 (Figs.. 2k) and 0.9 μm for T12.5 (Fig. 2l).

Fig. 2 shows the X-ray diffraction patterns of sintered specimens as a function of sintering conditions for samples with 7.5 (Figs.. 2a) and 12.5 mol% of titania (Fig. 2b). Three experimental groups (T7.5 1260/2 h; T7.5 1260/4 h and T12.5 1260/2 h) showed only peaks corresponding to the tetragonal zirconia crystalline phase. For the other nine experimental groups, in addition to the tetragonal phase, peaks corresponding to the monoclinic and cubic phases were identified.

3.2. Relative density, flexural strength and optical properties

According to the 2-way ANOVA (Table 2), titania content affected the flexural strength ($p = .027$), L ($p < .001$), a ($p < .001$), and b ($p < .001$). Sintering conditions affected all properties ($p < .001$) and interaction (Titania content x sintering conditions) affected relative density ($p = .014$), L ($p < .001$), a ($p < .001$), and b ($p < .001$).

Fig. 3 shows the mean values, standard deviations and coefficients of variation for relative density and flexural strength of the sintered samples. The highest mean relative density value was 98.9% for group T7.5 sintered at 1300 °C/1 h. This value was statistically similar to those obtained for groups T7.5 1260 °C/1 h, T7.5 1300 °C/2 h, T12.5 1260 °C/4 h, T12.5 1300 °C/1 h, T12.5 1300 °C/2 h and T12.5 1260 °C/1 h. The lowest relative density values were obtained for specimens sintered at 1460 °C/2 h (85.5% for group T7.5 and 86.3% for group T12.5). The highest mean flexural strength value was 670 MPa for group T12.5 sintered at 1300 °C/2 h and the lowest value was 364 MPa for group

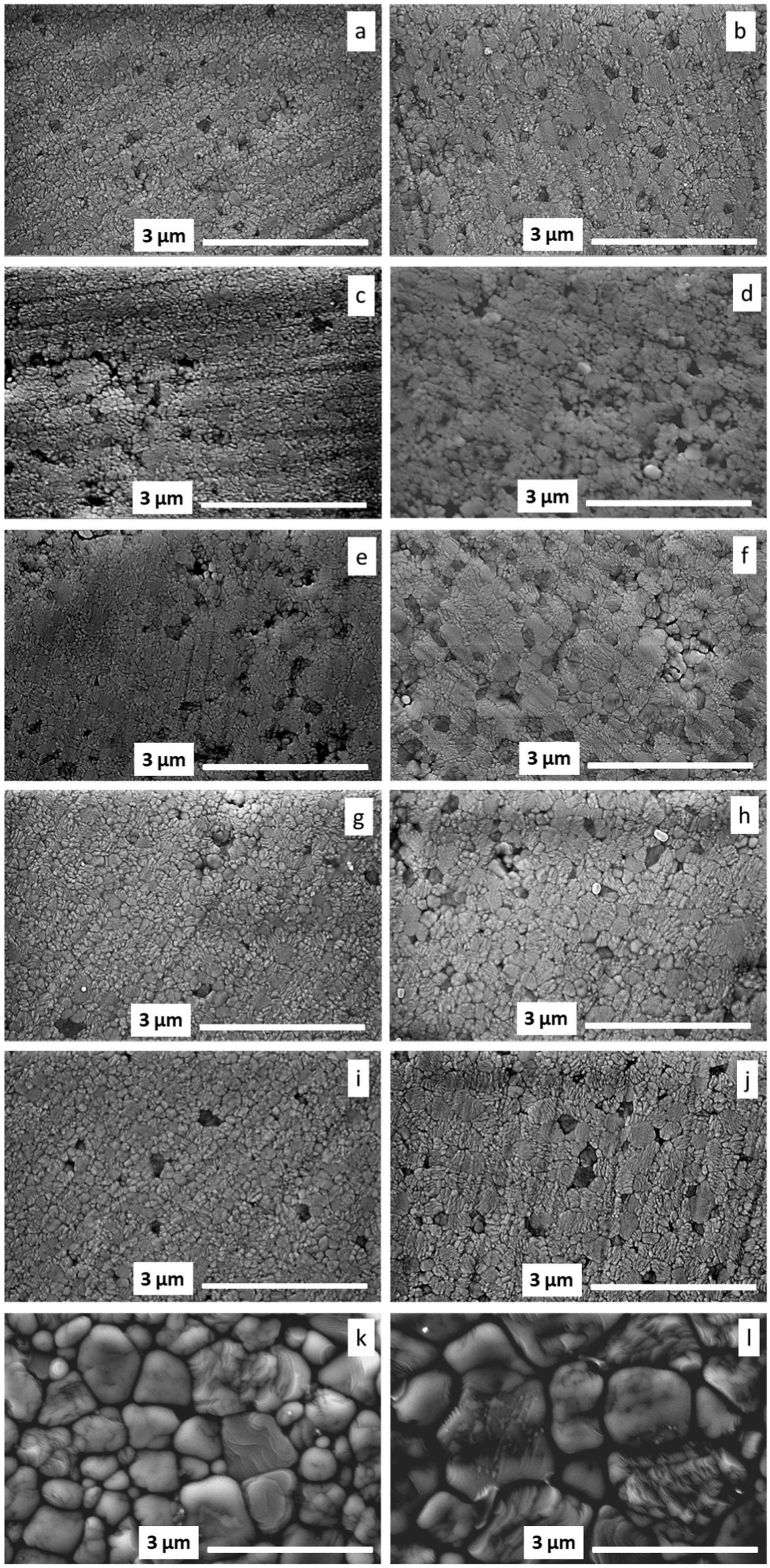

Fig. 1. Scanning electron micrographs of T7.5 (left) and T12.5 (right) groups, after polishing and thermal etching, sintered at: (a) and (b) 1260 °C/1 h; (c) and (d) 1260°/2 h; (e) and (f) 1260°/4 h; (g) and (h) 1300°/1 h; (i) and (j) 1300°/2 h; (k) and (l) 1460°/2 h.

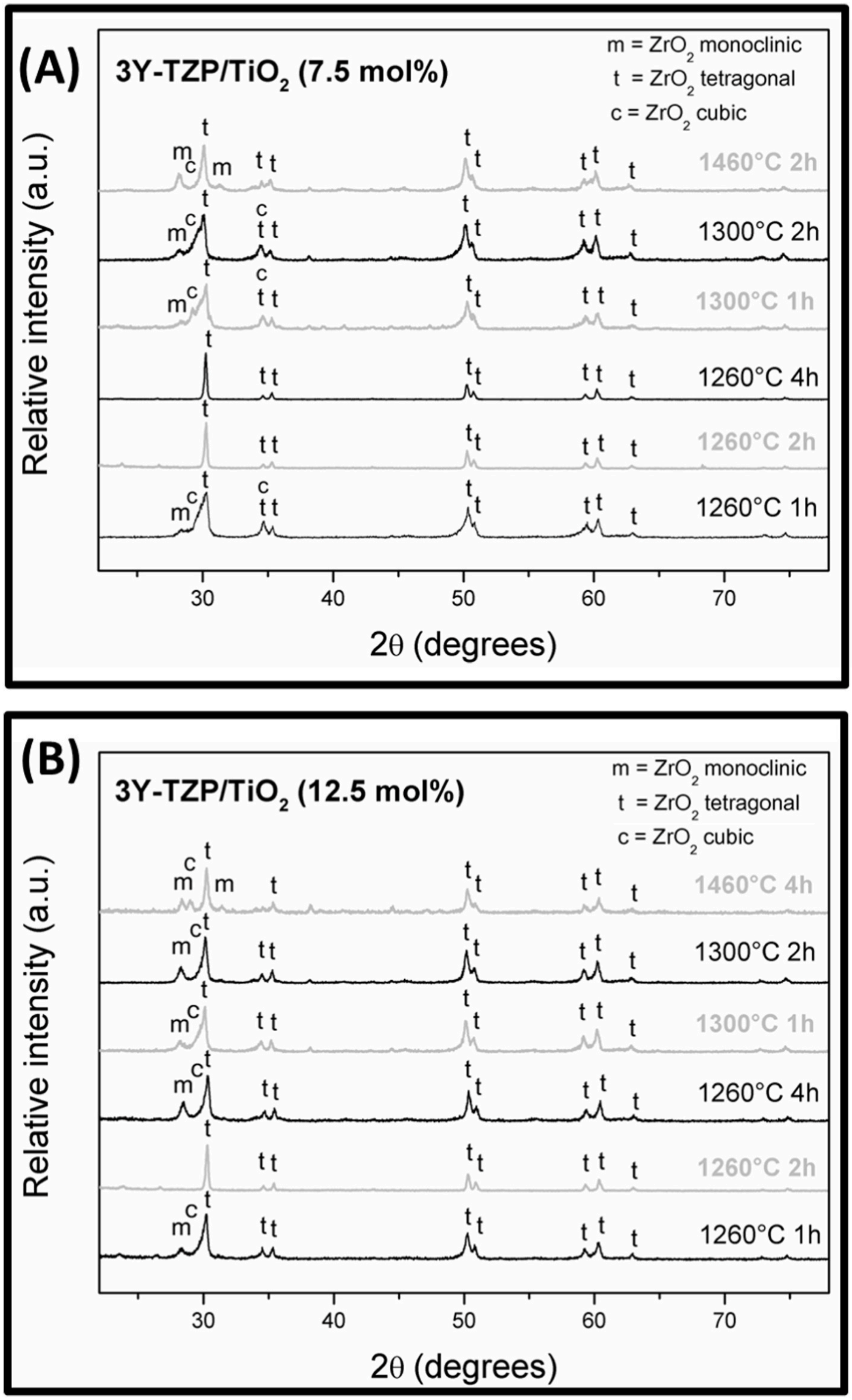

Fig. 2. X-ray diffraction patterns of T7.5 (A) and T12.5 (B) pellets after different sintering conditions.

T12.5 group sintered at 1460 °C/2 h.

There were significant differences among the values of L*, a* and b* (Table 3) and color difference (Table 4) obtained for the different experimental groups. The highest values of L*, a* and b* were respectively 87.2 (T7.5 group sintered at 1460 °C/2hs), 4.3 (T12.5 group sintered at 1300 °C/2hs) and 15.8 (T12.5 group sintered at 1300 °C/1 h). The lowest values of L*, a* and b* were respectively 66.2 (T12.5 group sintered at 1260 °C/4hs), −0.4 (T7.5 group sintered at 1460 °C/2hs) and 10.8 (T7.5 group sintered at 1260 °C/4hs). The lowest color difference (7.2) was obtained between groups T7.5 and T12.5 (1460 °C/2hs). The color difference for this sintering condition was statistically lower than those obtained for all of the other five conditions, which showed color differences varying from 10.7 (1300 °C/2hs) to 11.6 (1300 °C/1 h), which were statistically similar to each other.

4. Discussion

The results of the current investigation can be considered promising

Table 2
Summary of ANOVA of flexural strength, relative density, L, a and b.

Test	Factor	df	F	P
Flexural strength	*Titania content*	*1*	*5.21*	*.0273*
	Sintering conditions	*5*	*11.06*	*<.001*
	Interaction: Titania content x Sintering conditions	*5*	*.873*	*.507*
Relative density	*Titania content*	*1*	*.939*	*.3374*
	Sintering conditions	*5*	*535.97*	*<.001*
	Interaction: Titania content x Sintering conditions	*5*	*3.201*	*.014*
L*	*Titania content*	*1*	*8516.64*	*<.001*
	Sintering conditions	*5*	*399.64*	*<.001*
	Interaction: Titania content x Sintering conditions	*5*	*31.85*	*<.001*
a*	*Titania content*	*1*	*2696.15*	*<.001*
	Sintering conditions	*5*	*359.93*	*<.001*
	Interaction: Titania content x Sintering conditions	*5*	*13.59*	*<.001*
b*	*Titania content*	*1*	*545.89*	*<.001*
	Sintering conditions	*5*	*20.01*	*<.001*
	Interaction: Titania content x Sintering conditions	*5*	*17.31*	*<.001*

as group T12.5 sintered at 1300 °C/2 h reached a relative density close to 99% and a flexural strength of 700 MPa. These values are the highest found for the 3Y-TZP/TiO_2 dental ceramic and show an evolution in relation to previous studies in which the flexural strength ranged from 420 to 455 MPa (17, 22). The most significant contribution of this research with respect to the 3Y-TZP/TiO_2 dental ceramic was to set different sintering conditions (based on temperature and holding times) in order to find the right combination that would result in high densification.

The titania content affected flexural strength and optical properties of the 3Y-TZP/TiO_2 dental ceramic; therefore, the first null hypothesis was partially rejected. The second null hypothesis was totally rejected, since sintering conditions affected all properties studied. About the third null hypothesis, the interaction (titania content x sintering conditions) only not affected flexural strength, then this hypothesis was partially rejected. The literature reports that adding titania to a zirconia matrix promotes an increase in grain size, since the mobility of the grain boundaries is increased with the addition of tetravalent cations such as Ti^{4+} [33]. The microstructure is also affected by the sintering temperature, since high temperatures and long holding times increase the grain size [25,26,34]. In accordance to that, group T12.5 sintered at 1460 °C/2 h showed the largest mean grain size in the present study, with size of approximately 0.9 μm (Fig. 1l). Such grain size is related to both the addition of titania and the high sintering temperature used during processing of these specimens.

Regarding structural behavior, it is known that the mechanical properties of titania are inferior to those usually measured for zirconia. Previous studies have shown that the flexural strength, hardness and fracture toughness of 3Y-TZP/TiO_2 is lower than those of 3Y-TZP, especially when titania is added in large amounts [25,34–36]. Thus, the present study used smaller titania contents and different sintering

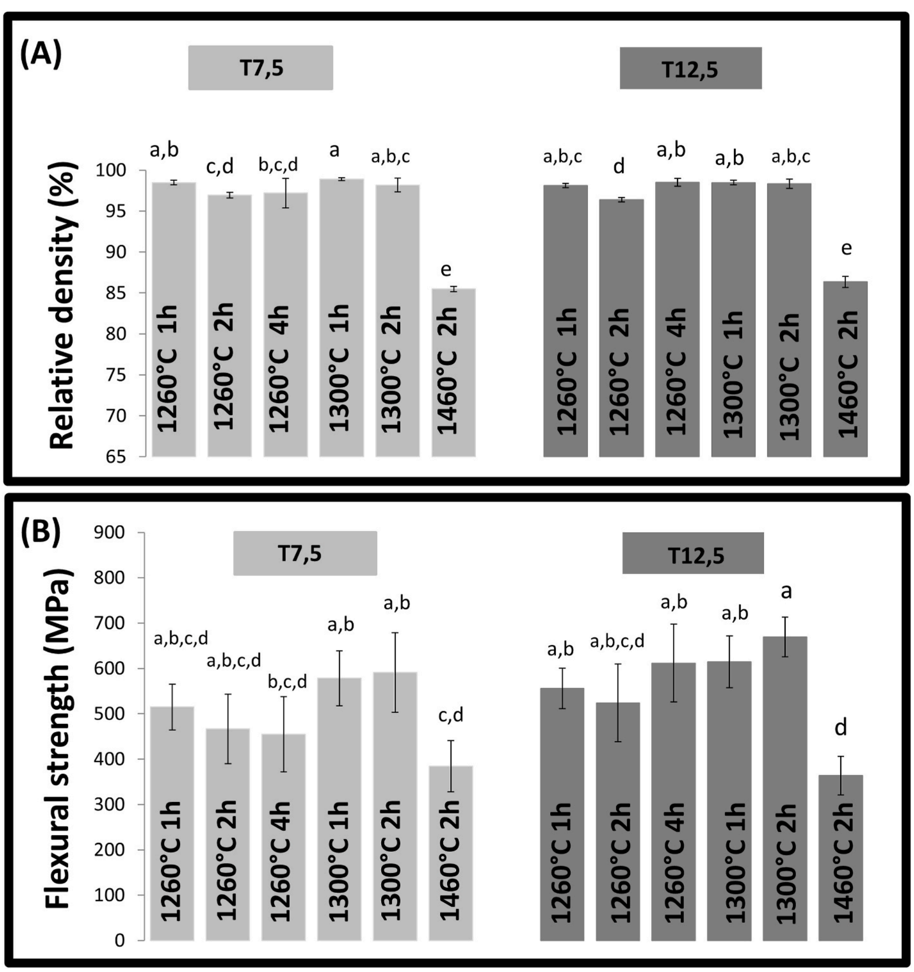

Fig. 3. Relative density (A) and flexural strength (B) of T7.5 and T12.5 groups sintered at different sintering conditions: Bars topped by different letters are statistically different (p ≤ .05).

Table 3
Optical properties of 3Y-TZP/TiO_2 ceramics. Mean ± standard deviation (coefficient of variation) of L, a and b of T7.5 and T12.5 groups sintered at different sintering conditions. Values followed by the same letter are statistically similar ($p > .05$). For each variable, the letters representing statistical differences represent statistical grouping for all groups shown in the Table.

Sintering conditions	L*		a*		b*	
	Titania content (mol %)		Titania content (mol %)		Titania content (mol %)	
	7.5	12.5	7.5	12.5	7.5	12.5
1260 °C 1 h	84.0 ± 0.5^{b} (0.6%)	69.1 ± 0.6^{f} (0.9%)	1.5 ± 0.1^{f} (6.5%)	3.8 ± 0.1^{b} (2.1%)	15.1 ± $0.2^{a,b}$ (1.1%)	12.8 ± $0.4^{g,h,i,j}$ (3.0%)
1260 °C 2 h	81.0 ± $0.4^{c,d}$ (0.5%)	67.1 ± 0.7^{g} (1.1%)	1.9 ± $0.1^{d,e}$ (6.2%)	3.9 ± 0.1^{b} (3.7%)	15.1 ± $0.3^{a,b,c}$ (1.7%)	12.2 ± $0.4^{j,k}$ (3.4%)
1260 °C 4 h	79.8 ± 0.5^{d} (0.6%)	65.2 ± 0.4^{h} (0.6%)	2.2 ± 0.2^{d} (9.3%)	4.1 ± $0.1^{a,b}$ (2.5%)	14.4 ± $0.3^{b,c,d}$ (2.1%)	10.7 ± 0.4^{l} (3.9%)
1300 °C 1 h	81.5 ± 0.3^{c} (0.37%)	66.9 ± 0.3^{g} (0.4%)	1.8 ± $0.1^{e,f}$ (4.1%)	4.1 ± $0.1^{a,b}$ (1.7%)	15.8 ± 0.1^{a} (0.5%)	12.2 ± $0.4^{j,k}$ (3.4%)
1300 °C 2 h	80.8 ± $0.2^{c,d}$ (0.2%)	67.1 ± 0.7^{g} (1.1%)	2.8 ± 0.1^{c} (3.1%)	4.3 ± 0.1^{a} (2.0%)	15.1 ± $0.2^{a,b,c}$ (1.4%)	12.2 ± $0.4^{j,k}$ (3.4%)
1460 °C 2 h	87.2 ± 0.2^{a} (0.2%)	77.7 ± 0.4^{e} (0.5%)	−0.4 ± 0.1^{h} (23.7%)	2.1 ± $0.2^{d,e}$ (8.7%)	14.7 ± $0.2^{b,c,d}$ (1.1%)	14.0 ± 0.5^{d} (3.6%)

Table 4
Mean ± standard deviation (coefficient of variation) of color difference (ΔE_{00}) between the two dental ceramics sintered at different sintering conditions. Values followed by the same letter are statistically similar ($p > .05$).

Sintering conditions					
1260 °C 1 h	1260 °C 2 h	1260 °C 4 h	1300 °C 1 h	1300 °C 2 h	1460 °C 2 h
11.2 ± 0.9^{b} (8.0%)	10.9 ± 0.8^{b} (7.3%)	11.5 ± 0.4^{b} (7.8%)	11.6 ± 0.3^{b} (2.6%)	10.7 ± 0.4^{b} (3.7%)	7.2 ± 0.3^{a} (4.1%)

conditions so that the dental ceramic would have the lowest probability of failure in clinical scenarios. It is well-known that the mechanical performance of ceramics is directly related to high densification of specimens. 3Y-TZP is usually sintered between 1400 °C and 1500 °C and achieves high relative density and very good mechanical properties [10,37,38]. The coprecipitated powder synthesized from 3Y-TZP/TiO_2 dental ceramic when sintered at temperatures close to that of 3Y-TZP showed a relative density of 86.3% (group T12.5 sintered at 1460 °C/2 h) and 85.5% (group T7.5 sintered at 1460 °C/2 h). The literature shows that high porosity impairs the structural reliability of materials, since it leads to significant stress concentrations around pores [39,40]. The results of this study showed that the 3Y-TZP/TiO_2 dental ceramic, when sintered at lower temperatures than those recommended for 3Y-TZP, showed density greater than 98% and flexural strength exceeding 600 MPa. This can be considered a promising result, as this material can be applied for three-unit fixed dental prostheses according to the biaxial flexural strength recommendation in ISO 6872/2008 [32]. In addition, the processing method used here allows greater productivity due to decreased electricity consumption and faster sintering.

Considering the parameters established by the International Commission on Illumination, in general the groups with the addition of 7.5 mol% of titania showed greater brightness than the groups with 12.5 mol% of titania, that is, their L* values were higher. Regarding the color coordinates, the additions of the two different titania contents made the dental ceramic more yellowish and reddish. However, there were differences between these two titania contents, with the T12.5 group being more reddish (higher a* coordinate) than the T7.5 group and the T7.5 group being more yellowish (higher b* coordinate) than the T12.5 group. Regarding the color difference between the group with the addition of 7.5 mol% of titania and the group with 12.5 mol% of titania, the results showed that for all six different sintering conditions this value was greater than 2.25. This means that untrained observers can identify the color difference between the two dental ceramic with different titania contents [41].

When searching the literature for CIELAB values reported in the literature for teeth and 3Y-TZP, it can be considered that the material developed (group T12.5 sintered at 1260 °C/1 h, L* = 69.1; a* = 3.8 and b* = 12.8) has values closer to central incisors (L* = 75.1; a* = 3.4 and b* = 14.8) than 3Y-TZP (L* = 93.2; a* = −1.6 and b* = 0.8) [42,43]. Caution must be exercised when making this comparison, since tooth color is extremely complex and influenced by many factors, such as age, gender, ethnic group and enamel and dentin thickness [44]. On the other hand, the color of restorative materials is influenced by their composition, microstructure and thickness [45]. At a time when dental esthetics is increasingly valued by society and monolithic zirconia is gaining popularity as a material for oral rehabilitation [14,46,47], our proposed 3Y-TZP/TiO_2 dental ceramic shows great potential as full-contour crowns that mimic more naturalized tooth structure.

Although the 3Y-TZP/TiO_2 dental ceramics exhibits high bioactivity, promoting the adhesion and proliferation of cells related to osseointegration, as previously studied, it is essential that future studies investigate other mechanical properties. The dynamic loading test for endosseous dental implants, Weibull analysis and step-stress accelerated life testing are fundamental characterizations to assess the structural reliability and failure probability of this material and should be carried out in future studies [48]. The possibility of controlling the sintering conditions and the stabilization of the tetragonal crystalline phase would help in this challenge, since the tetragonal phase has the highest mechanical properties among the allotropic forms of zirconia [49,50].

5. Conclusion

The interaction (titania content x sintering conditions) affected relative density and optical properties. A relative density greater than 98% was achieved for the T7.5 groups (sintered at 1260 °C/1 h, 1300 °C/1 h and 1300 °C/2 h) and for the T12.5 groups (sintered at 1260 °C/1 h, 1260 °C/4 h, 1300 °C/1 h and 1300 °C/2 h). The groups with 7.5 mol% of titania showed greater brightness (L*), more yellowish (b*) and less reddish (a*) coloration than the groups with 12.5 mol% of titania. The microstructure and flexural strength of the 3Y-TZP/TiO_2 dental ceramic with 7.5 mol% and 12.5 mol% were affected by the sintering conditions used in this study. The sintering conditions of 1460 °C/2 h increased the grain size and significantly decreased the flexural strength of 3Y-TZP/TiO_2 dental ceramics. The material developed with 12.5 mol% of titania and sintered at 1300 °C/2 h showed high densification, flexural strength of 670 MPa and has good potential to be used in dentistry.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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