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Dental zirconia microwave-sintering followed by rapid cooling protocol

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ABSTRACT

Objectives: This study aimed to evaluate the effect of microwave sintering temperature and cooling rate (MS) on 3Y-TZP ceramics and its influence on the ceramic microstructure and mechanical properties. Specifically, to optimize the sintering process, reducing the total sintering time compared to conventional sintering.
Materials and methods: Eighty-four pre-sintered Y-TZP discs (Vipi block Zirconn, VIPI) (ISO 6872) were divided into seven groups (n = 12) according to the sintering conditions: conventional sintering (CS) at 1530 °C for 120 min and microwave sintering at 1400 °C (MS1400) and 1450 °C (MS1450) for 15 min followed by different cooling conditions: rapid cooling (RC), cooling at 400 °C (C400) and 25 °C (C25). The specimens were submitted to apparent density measurements, X-ray diffraction analysis (XRD), scanning electron microscopy, and biaxial flexural strength test. Data was statistically analyzed through two-way ANOVA, Tukey, Sidak, Dunnett and Weibull ($\alpha = 0.05$).
Results: All MS1400 groups presented lower density values than the CS and MS1450 groups. Two-way ANOVA revealed that the MS temperature and cooling rate affected the biaxial flexural strength of the Y-TZP ($p < 0.01$). Group MS1400RC presented lower biaxial flexural strength values (681.9 MPa) than MS1450RC (824.7 MPa). The cooling rate did not statistically decrease the biaxial strength among the groups submitted to microwave sintering at 1450 °C. XRD analysis showed that the sintering and cooling temperature did not induce tetragonal to monoclinic phase transformation.
Conclusions: Microwave sintering at 1450 °C for 15 min followed by rapid cooling can be a viable fast alternative protocol for Y-TZP sintering, compared with the conventional sintering, reducing the total sintering time by 75% and reducing the energy used for the sintering process without affecting the Y-TZP biaxial flexural strength and relative density compared to the conventional sintering. Moreover, the microwave technique promoted smaller grains and did not induce monoclinic phase formation.

1. Introduction

The increase in aesthetic demand by the patients has contributed to the development of metal-free restorations that mimic the natural tooth to achieve the tooth's colour (value and saturation) and translucency (Pecho et al., 2012). The 3 mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) is frequently used as a dental restorative material. It is indicated for infrastructure, framework, implants and monolithic

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crowns since it presents aesthetics associated with high mechanical strength due to its phase transformation toughening mechanism that induces compressive stress at a crack tip, difficulting the crack propagation. (Chevalier et al., 2007; Denry and Kelly, 2008; Piconi and Maccauro, 1999; Kongkiatkamon et al., 2023; Zhang et al., 2013, 2016).

The sintering process can directly influence the mechanical strength of the Y-TZP since it affects its density, degree of porosity, grain size and all final properties of the ceramic material (Kim et al., 2013; Luo et al., 1998; Trunec, 2008). The conventional sintering process of Y-TZP ceramics is conducted in a resistive furnace designed to process this material under pre-established temperature and time conditions. However, in conventional firing, the radiant heating received on the ceramic reaches the core by thermal conduction, producing high-temperature gradients and stresses (Upadhyaya et al., 2001). In this process, the Y-TZP is submitted to a high temperature (1350 °C–1550 °C) for 60–120 min, according to the manufacturer, followed by a slow cooling rate to prevent cracks. The total sintering time can vary around 10 h due to the slow cooling step, which is expensive and time-consuming.

Among the sintering techniques, microwave energy sintering allows a fast and uniform distribution of heat in the ceramic core due to the excitation of each constituent unit of the crystal lattice (Almazdi et al., 2012; Menezes et al., 2007), reducing the thermal stresses that can generate cracks and damage to the ceramic (Marinis et al., 2013). The benefits of microwave energy sintering, compared to the conventional method, are the rapid and volumetric heating of the specimen, lower heating temperature, enhancement in densification and controlled grain growth (Upadhyaya et al., 2001). These advantages reduce processing time, promote energy savings, and uniform temperature absorption (Lazar et al., 2002). Promising results have shown microwave sintering as a viable alternative to conventional sintering concerning laboratory production. The microwave sintering (1.4 KW at 2.45 GHz, 1450 °C for 15 min, 1 h and 45 min total, including the heating process) showed no statistically significant difference in volumetric contraction and surface roughness compared to specimens submitted to conventional sintering (1530 °C for 2 h, 10 h total including the heating and cooling process) (Barchetta et al., 2017). Microwave sintering can reduce the total sintering time from 5 h 46 min (conventional sintering) to 1 h and 45 min (excluding the cooling process) with a decrease of 70% in sintering time (Barchetta et al., 2017). Therefore, this technique is attractive to the restorative dentistry market as it will increase laboratory production and lower energy consumption, reducing laboratory prosthetic costs. A few studies recommend different sintering times and temperatures concerning the sintering method of zirconia through microwave energy (Borrell et al., 2012; Lazar et al., 2008; Wang et al., 2023). However, optimization of this sintering and cooling rate regarding the mechanical and microstructural properties of the Y-TZP is still required. Therefore, this study aimed to optimize the Y-TZP sintering and cooling rate and establish a microwave sintering protocol to obtain microwave-sintered Y-TZP ceramic with comparable or better characteristics than the conventional sintered Y-TZP.

2. Materials and methods

2.1. Specimen preparation

Blocks of pre-sintered 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) (lot: A67407_10/13, VIPI) were machined and sectioned to rounded samples (15 mm Ø x 2 mm height) using a CAD-CAM system (milling machine K4 4 axis, VIPI) according to ISO 6872:2008 (*ISO 6872:2008, Dentistry - Ceramic materials*, 2008). Before sintering, the discs were polished using sandpaper #1200 grit (Norton Saint-Gobain) (Arata et al., 2014).

2.2. Sintering and cooling protocol

Eighty-four discs were divided into seven groups according to the sintering and cooling temperature rate in a conventional resistive furnace (Conventional sintering - CS) or microwave furnace (Microwave sintering - MS) (Fig. 1). The temperature and sintering time were selected as recommended by the Y-TZP manufacturer (1530 °C/120 min) for conventional sintering. The MS groups sintering protocol was performed in a customized microwave furnace for zirconia sintering (Model FE-1700, from 1.4 KW to 2.45 GHz, SiC susceptor, INTI). The Y-TZP microwave sintering temperature defined in this present study was selected according to Kim et al. (2013) and Borrell et al. (2012), which suggested a microwave sintering time of 20 min at 1450 °C and 10 min at 1400 °C, respectively. The heating rate for sintering is presented in Table 1. The samples were cooled down by removing the discs from the microwave under three different conditions: 1) remotion at 400 °C – specimens removed after sintering time when the furnace cools to 400 °C; 2) remotion at room temperature (25 °C)- complete cooling of the sample, inside the oven, according to the manufacturer; 3) rapid cooling - specimens immediately removed in the as-sintered condition after the sintering time at temperatures around 1450 °C and 1400 °C.

2.3. Microstructural characterization

One cross-sectioned specimen per group was polished with 9, 6, 3 and 1 μm diamond suspension and submitted to thermal etching at 50 °C below the sintering temperature for 10 min to reveal the post-sintering microstructure morphology. The polished surface was analyzed under scanning electron microscopy (SEM, XL30, Phillips) in three areas per specimen. The grain size of each group was measured through the Ferret area (Allen, 1990) using the Image J software (National Institute of Health).

The as-sintered ceramic densities and porosity were determined for all groups by an immersion method based on Archimedes' principle (ASTM C 20-00) (ASTM C20-00, 2015). To verify the possibility of tetragonal to monoclinic (t-m) phase transformation induced by the cooling process, one specimen per group was submitted to X-ray diffraction analysis (XDR) (X'pert Powder, PANalytical), Cu-Kα (λ = 1, 54060 Å), 45 kV, 40 mA. Scans were performed from 20° to 80°, with a step size of 0.02°; 25s per step. Quantitative phase analyses were performed using the Rietveld refinement method (General Structure Analysis System - GSAS). The crystal structure data (atom coordinates, thermal parameters and unit cell parameters) were obtained from the Inorganic Crystal Structure Database – ICSD. The computation adjustments performed were the scale factors, unit cell parameter, pattern background polynomial parameter, 2θ-scale offset and peak profile functions (pseudo-Voigt with asymmetry) (Young, 1993).

2.4. Biaxial flexural strength test

Each group (n = 12) was submitted to biaxial flexural strength test underwater (1 mm/min, 100 kgf load) in a universal mechanical testing machine (Emic DL 3000, Emic) until a fracture occurred. The biaxial flexural strength (MPa) was calculated using equations (1)–(3), according to ISO 6872 (*ISO 6872:2008, Dentistry - Ceramic materials*, 2008).

$$S = -0.2397\frac{P(X-Y)}{d^2} \quad (1)$$

Where:

S = Maximum tensile stress (MPa)

P = Total load causing fracture (N)

d = Specimen thickness at the origin of the fracture (mm)

The X and Y values were determined according to equations (2) and (3).

$$X = (1+\nu)ln\left(\frac{r_2}{r_3}\right)^2 + \left[\left(\frac{1-\nu}{2}\right)\left(\frac{r_2}{r_3}\right)^2\right] \quad (2)$$

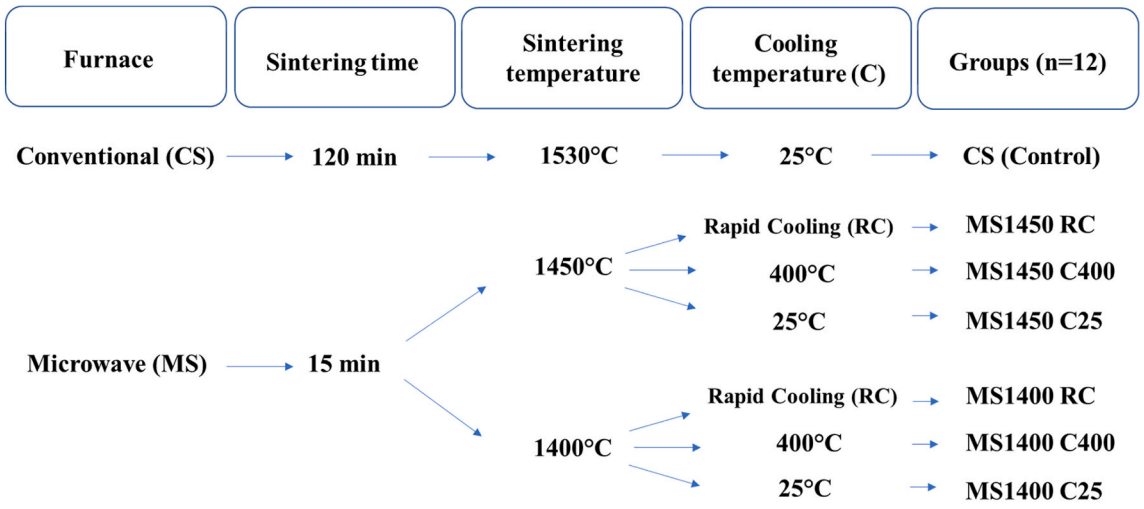

Fig. 1. Experimental design for Y-TZP conventional resistive and microwave sintering.

Table 1
Heating and cooling rate for the microwave and conventional sintering.

Microwave			Conventional		
Heating rate (degree/min)	Temperature range (°C)	Time (min)	Heating rate (degree/min)	Temperature range (°C)	Time (min)
25	0 to 1000	40	10	0 to 800	80
15	1000 to 1300	20	5	800 to 1530	146
5	1300 to1450	30	0	1530	120
0	1450	15			
Cooling	400	150		25	240
Cooling	25	180			
Total		435 min			586 min

$$Y = (1+\nu)\left[1 + ln\left(\frac{r_1}{r_3}\right)^2\right] + (1-\nu)\left(\frac{r_1}{r_3}\right)^2 \quad (3)$$

Where:
ν = Poisson's ratio (=0.25)
$^{r}1$ = Radius of the support circle (mm)
$^{r}2$ = Radius of the loaded area (mm)
$^{r}3$ = Radius of the specimen (mm)

2.5. Statistical analysis

The grain size was submitted to descriptive statistical analysis. The Dunnett test (α = 0.05) was used to compare the density data between all the groups and the biaxial flexural strength (MPa) between the CS and MS groups. The biaxial flexural strength values (MPs) among the MS groups were analyzed through the 2-way ANOVA, Tukey's test (α = 0.05) and Sidak test (α = 0.05).

The reliability of strength was assessed through the Weibull distribution (Quinn and Quinn, 2010). The Weibull parameter m and the characteristic strength σ_c were determined in a diagram (DIN ENV 843-5, 2007):

$$\ln\ \ln\frac{1}{1-F(\sigma_c)} = m\ln\sigma_c - m\ln\sigma_\theta \quad (4)$$

The Weibull modulus (m) determines the distribution function slope and characterizes the spread of the failure data concerning the σ (fracture stress). The σ_θ is the characteristic strength where the stress level led to 63.21% failure of the specimens (Egilmez et al., 2014). The statistical analyses were undertaken using the MINITAB software (Minitab 17.1.0).

$$ln\ \sigma_c - ln\left[ln\left(\frac{1}{1-F(\sigma_c)}\right)\right] \quad (5)$$

3. Results

3.1. Microstructural characterization

SEM image of the Y-TZP group sintered by the conventional resistive furnace (CS) (Fig. 2) showed a defined grain boundary. The same characteristic was observed in the ceramics submitted to microwave sintering (MS) (Fig. 3A–F). However, the MS group sintered at 1400 °C showed higher porosity in its microstructure (Fig. 3A and E).

3.2. Grain size analysis

The grain size analysis of the CS group showed uniform larger grains (600 nm) compared to the groups submitted to microwave sintering (200–300 nm). The grain sizes of the microwave-sintered groups (MS) were similar (Table 2).

3.3. Relative density analysis

The conventional sintering group (CS) presented around 97% relative density in comparison to the Y-TZP theoretical density value (ρ = 6.1 g/cm^3), which was statistically similar to the results observed for all

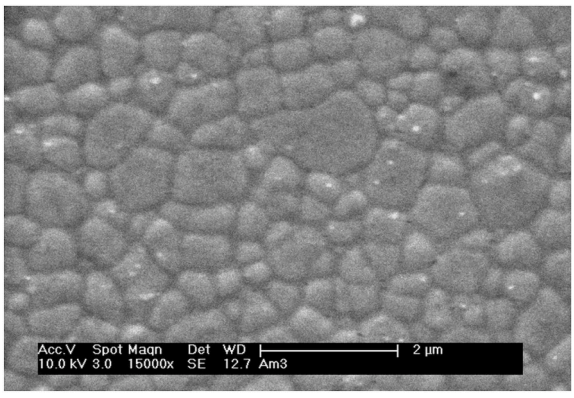

Fig. 2. SEM image of the conventional sintered Y-TZP (CS).

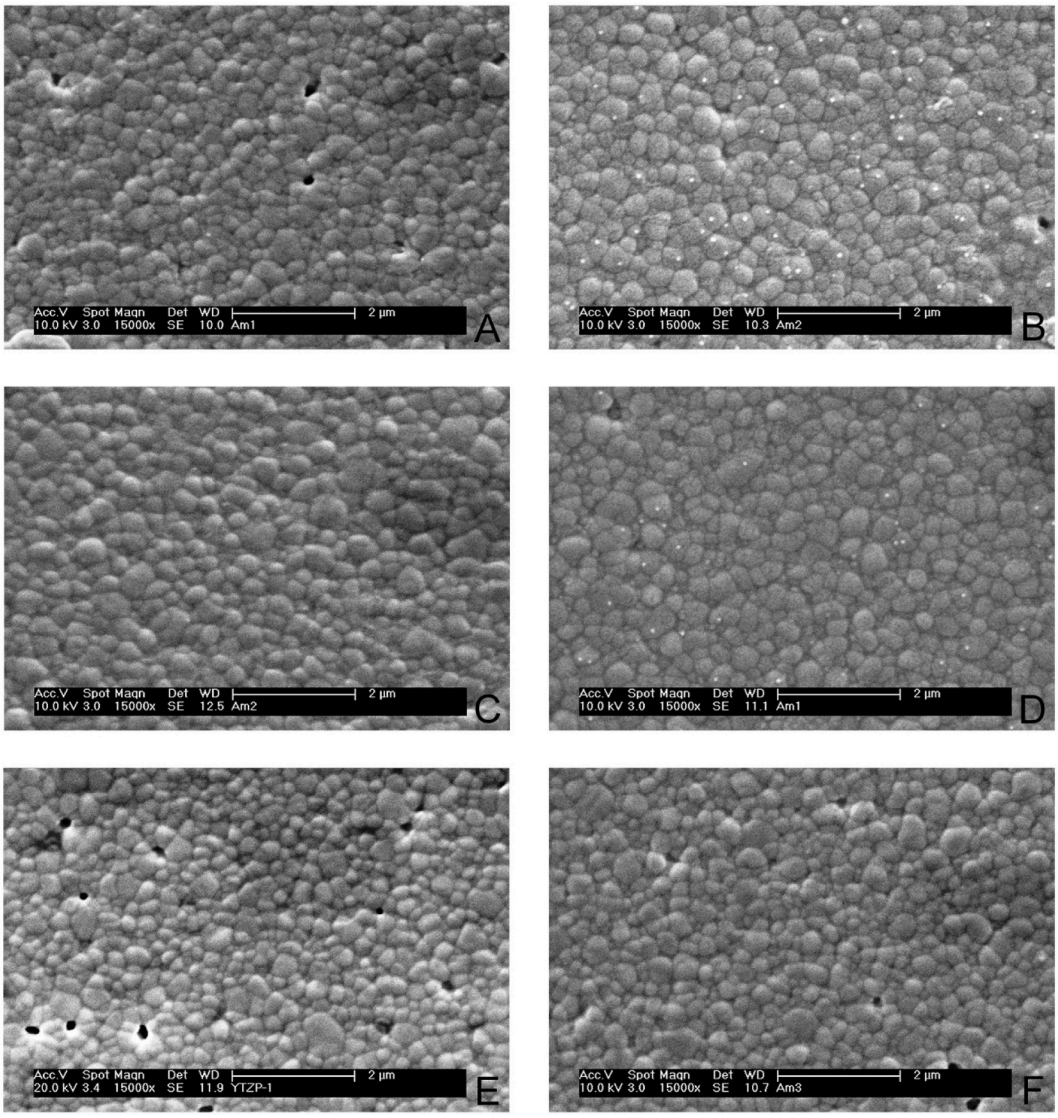

Fig. 3. SEM images of the Y-TZP groups submitted to microwave sintering (MS) at 1400 °C and 1450 °C and cooling temperature at 25 °C, 400 °C and rapid cooling (RC): 3A) MS1400RC; 3B) MS1450RC; 3C) MS1400C400; 3D) MS1450C400; 3E) MS1400C25; 3F) MS1450C25.

Table 2
Average grain size of the conventional- (CS) and microwave-sintered groups (MS).

Groups	Grain Size (μm)
CS	0.66 ± 0.27
MS1400RC	0.32 ± 0.10
MS1400C400	0.34 ± 0.11
MS1400C25	0.29 ± 0.14
MS1450RC	0.35 ± 0.12
MS1450C400	0.36 ± 0.12
MS1450C25	0.37 ± 0.11

CS- conventional sintering MS - sintering temperature C- cooling temperature.
RC- rapid cooling temperature.

groups submitted to microwave sintering at 1450 °C (MS1450) (Table 3). However, the MS groups sintered at 1400 °C presented a statistically significant lower density than the CS and MS1450 groups.

Table 3
Apparent density average and standard deviation (SD) of sintered groups as a function of the sintering protocol.

Groups	Density ± SD (%)
CS	97.74 ± 0.95[a]
MS1400RC	94.07 ± 0.33[b]
MS1400C400	94.96 ± 0.21[b]
MS1400C25	95.80 ± 0.41[b]
MS1450RC	98.08 ± 0.34[a]
MS1450C400	96.72 ± 0.63[a]
MS1450C25	97.04 ± 0.83[a]

CS- conventional sintering MS - sintering temperature C- cooling temperature.
RC- rapid cooling temperature.
Different superscript letters mean a significant statistical difference among groups according to the Dunnett test (α = 0.05).

3.4. X-ray diffraction analysis

X-ray diffraction analysis showed similar patterns between the CS and MS groups sintered at 1400 °C and 1450 °C (Fig. 4a and b,

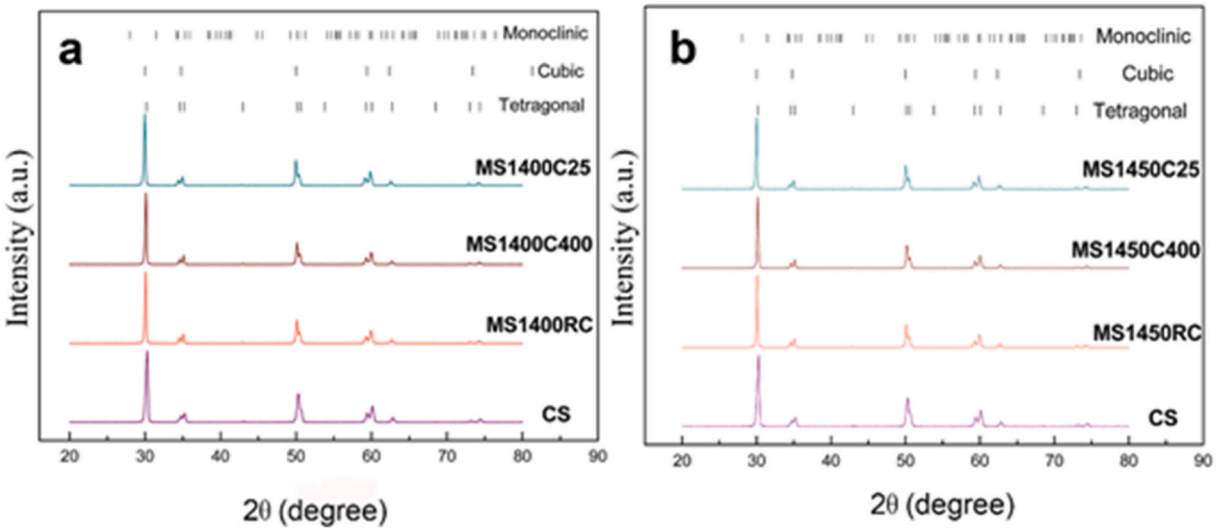

Fig. 4. XRD diffraction patterns show the tetragonal and cubic phases, comparing the conventional sintering group (CS) with a) MS groups sintered at 1400 °C; b) MS groups sintered at 1450 °C.

respectively). The results of the Rietveld refinement showed ~83% of tetragonal phase and ~17% of cubic phase in all groups independent of the sintering procedure (CS or MS), sintering temperature and cooling rate (Table 4).

3.5. Biaxial flexural strength

The results of biaxial flexural strength and its respective standard error are presented in Tables 5 and 6. The average biaxial flexural strength for the conventional sintering group (CS) was ~806 MPa (Table 5). The Dunnet analysis comparing the MS groups with the CS showed that the MS1400C400 (~685.6 MPa) and MS1400RC (~681.9 MPa) presented a statistical decrease in biaxial flexural strength compared with the CS group (Fig. 5).

Two-way ANOVA showed that the sintering temperature statistically influenced the biaxial flexural strength of the MS groups ($p < 0.01$). The Tukey test (5%) revealed that the cooling temperature among the group microwave sintered at 1400 °C did not present a statistical difference, and the same results were observed for the microwaved groups sintered at 1450 °C. However, the Sidak test (5%) (used for comparison between the groups MS1400 °C and MS1450 °C with the same cooling rate) showed a statistical difference in the biaxial flexural strength for the group MS1400RC (681 MPa) when compared to the group MS1450RC (824.7 MPa) (Table 6).

The Weibull statistical analysis (Fig. 6) did not show a statistical difference in the Weibull's moduli (m) (Chi-Square = 0.486; df = 7; $p = 0.998 > 0.05$). The biaxial flexural strength test data were evaluated according to the Weibull distribution. The results of biaxial flexural strength obeyed the Weibull distribution due to the strong correlation (close to 1). The modules (m) were between 7 and 9.

Table 4
Percentage of tetragonal, monoclinic and cubic phases for the CS and MS groups, calculated using the Rietveld method.

	Tetragonal phase (%)	Monoclinic phase (%)	Cubic phase (%)
CS	76.7	0	23.3
MS1400C25	82.8	0	17.2
MS1400C400	83.0	0	17.0
MS1400RC	82.7	0	17.3
MS1450C25	81.9	0	18.1
MS1450C400	83.2	0	16.8
MS1450RC	84.0	0	16.0

Table 5
–Biaxial flexural strength mean (MPa) and standard deviation of the conventional sintered (CS) group.

Group	Mean ± SD	Coefficient of variation (%)
CS	806.1 ± 105.8	13.12

Table 6
Biaxial flexural strength mean (MPa) and standard deviation of the microwave-sintered (MS) groups.

Groups	MS1400 °C	MS1450 °C
C25 °C	704.1 ± 86.95 ^{Aa}	749.4 ± 100.83 ^{Aa}
C400 °C	685.6 ± 94.65 ^{Aa}	728.4 ± 106.94 ^{Aa}
RC	681.9 ± 91.17 ^{Aa}	824.7 ± 99.12^{Ab}

Same uppercase letters indicate no significant difference in the respective column, Tukey test (5%).
Same lowercase letters indicate no significant difference in the respective line, Sidak test (5%).

4. Discussion

Sintering time and temperature have an important role in the Y-TZP microstructure since they can modify its grain size, density, degree of porosity, and crystalline phase, directly influencing the ceramic's mechanical properties. Through the increase of sintering temperature, pores on grain boundaries are reduced by solid-state diffusion, increasing the sintered density (Magnago et al., 2013). Different methods, such as spark plasma sintering, hot pressing, two-step sintering and fast firing, are used to hinder grain growth while maintaining a high densification (Gómez et al., 2016). While spark plasma and hot pressing are available techniques to produce nanometric microstructure, their high cost and complexity make the techniques inviable for dental laboratory applications (Gómez et al., 2016). Two-step sintering and fast firing are also techniques that have been proposed to control the ceramic morphology using different temperatures and times to exploit the grain growth and densification using a conventional furnace, being an alternative for the Y-TZP sintering. However, to suppress the grain growth, the two-step sintering technique "freezes" the microstructure to slow the kinetic to reach full density; therefore, the sintering procedure is carried out at a lower temperature for an extended length of time (20 h) (Hotza et al., 2015).

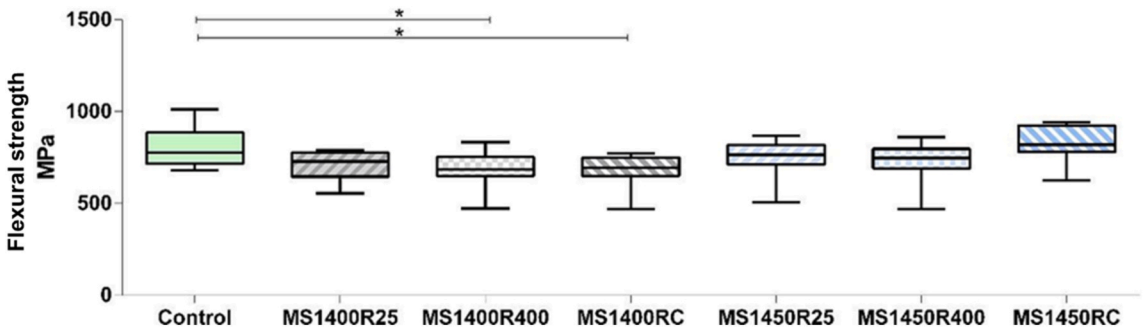

Fig. 5. Graphic showing the mean (MPa) and standard deviation of the biaxial flexural strength values of the CS and MS groups. The (*) indicates a significant statistical difference.

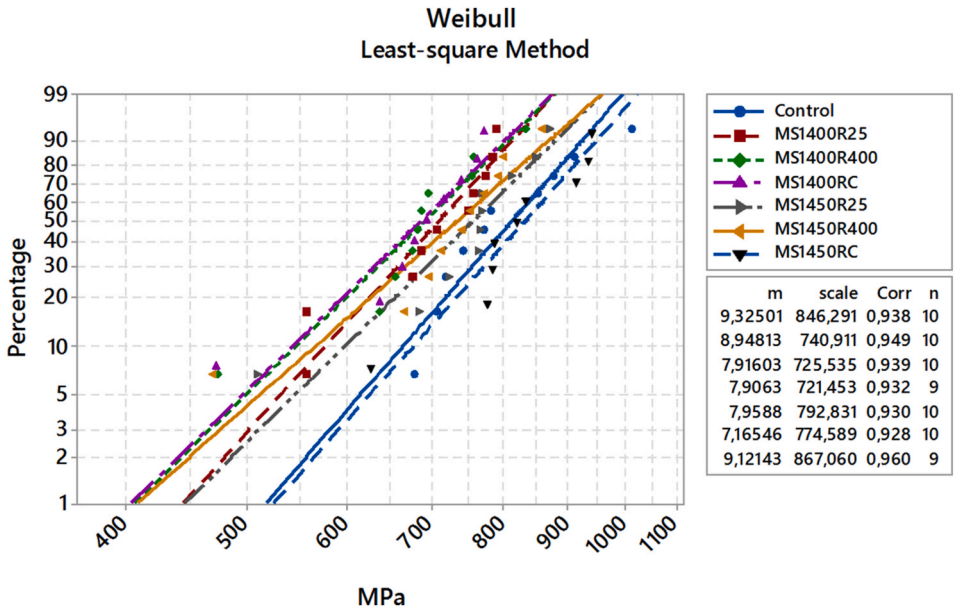

Fig. 6. Weibull diagram according to the Y-TZP sintering protocol.

In contrast, the fast-firing technique increases the densification rate, promoting a microstructure with a small grain size to the detriment of the coarsening rate by rapidly approaching a high temperature. However, the thermal gradient inside the sample can cause cracks. Moreover, large samples can present different microstructures between the inner and outer regions. Therefore, samples with low thermal expansion and higher heat conductivity are more suitable for this technique (Hotza et al., 2015).

Another Y-TZP sintering technique explored in the literature is microwave sintering. The advantage of this technique is that it allows rapid and volumetric heating, where the heat flow occurs from the grain core to the grain boundary, which may slow the mass diffusion among the particles at the grain boundary, decreasing the coarsening rate compared with conventional sintering (Almazdi et al., 2012; Jiang et al., 2011; Upadhyaya et al., 2001).

The sintering time and temperature can alter the Y-TZP grain size and final density (Trunec, 2008). In the present study, the grain size analysis showed that the conventional sintering at 1530 °C for 2 h presented larger grains (600 nm) compared with the microwave sintering at 1400 °C or 1450 °C for 15 min (200–300 nm). Similar results were observed by Kim et al. (2013), where specimens submitted to microwave sintering ranging from 1450 °C to 1500 °C for 20 min presented an average grain size of around 0.3 μm compared to 0.6 μm when the Y-TZP was submitted to conventional sintering at the same temperatures for 2 h. However, the present study did not find difference in grain size among the groups submitted to microwave sintering. The temperature range used for microwave-sintered groups (1400 °C–1450 °C) was probably not significant enough to alter the grain size. Also, between the microwave-sintered groups, the sintering time did not affect the grain size (Table 2).

The microwave sintering technique showed the advantage of reducing the temperature and time (1450 °C and 1400 °C for 15 or 30 min) compared with the conventional sintering process (1530 °C for 2 h), producing a smaller grain size since it inhibited the grain coarsening process for all the microwave groups. Overall, comparing the results with other sintering techniques in the literature (Gómez et al., 2016), the microwave technique also suppressed the grain growth during the zirconia sintering. Moreover, there was a reduction in the Y-TZP overall sintering time from 10 h (conventional method) to 1 h 45 min using microwave technology.

The conventional and microwave sintering groups presented specimens with a defined grain boundary (Figs. 2 and 3). However, the groups submitted to microwave sintering at 1400 °C followed by rapid cooling or cooled at room temperature presented higher porosity in their microstructure (Fig. 3A and E) and Table 3. The microwave heating source of 2.45 GHz allowed a high densification of the groups sintered at 1450 °C (ranging from 96.7% to 98% of the theoretical value), promoting uniform granulated microstructure, presenting statistically similar results of relative density (around 97%) compared with the conventional sintering group, indicating that microwave sintering can

produce high-density ceramics at low temperature (Wang et al., 2023). However, the MS groups sintered at 1400 °C presented a statistically significantly lower density than the CS and MS1450 groups, in agreement with the porosity observed by SEM (Fig. 3A and E). These results are also in accordance with Ribeiro et al. (2019), who observed a higher densification with the increase of the microwave sintering temperature in laboratory-scale synthesized Y-TZP specimens and commercial dental Y-TZP submitted to microwave sintering at 1450 °C for 15 or 30 min and a lower densification of the specimens sintered at 1350 °C for 15 or 30 min. This behaviour is associated with the activation energy for the sintering process. The higher porosity observed at the MS1400 °C groups is probably a result of low activation energy to form the grains and insufficient time to produce a dense formation. For the group MS1400RC, the rapid cooling process could have stopped the grain formation and densification during the sintering process, showing a higher presence of pores. However, there is a limit in sintering temperature and grain size that could interfere with the grain size and final densification. Trunec (2008) observed that a Y-TZP conventionally sintered at 1650 °C for 20 h produced a critical grain size (between 1.8 μm and 2.15 μm) that allowed spontaneous tetragonal to monoclinic phase transformation decreasing the overall density and allowing crack propagation.

The present study's sintering technique, temperature, and time did not induce tetragonal to monoclinic phase transformation. XRD Rietveld results showed the same amount of tetragonal (~83%) and cubic phase (~17%) for all the groups (Table 4). The cooling procedure did not induce the Y-TZP tetragonal to monoclinic phase transformation (Table 4). However, the microwaved group sintered at 1400 °C when cooled at 400 °C (~685.6 MPa) or submitted to rapid cooling (~681.9 MPa) presented a statistical decrease in biaxial flexural strength compared with the CS group (~806 MPa) (Fig. 6). The presence of pores due to the lower sintering temperature and the cooling rate probably influenced the decrease in mechanical strength. Two-way ANOVA showed that the sintering temperature statistically influenced the biaxial flexural strength of the MS groups ($p < 0.01$). However, the cooling temperature rate did not affect the biaxial flexural strength of the Y-TZP when compared to the results within the same microwave sintering temperatures (Table 6).

A statistical difference was observed in the groups submitted to microwave sintering at 1400 °C and 1450 °C, followed by rapid cooling, with 681 MPa and 824.7 MPa, respectively. The group sintered at 1400 °C associated with rapid cooling presented a decreased mechanical strength of ~17% (Table 6). Magnago et al. (2013) observed similar values of density (6.05 g/cm^3) and mechanical strength (~950 MPa) for a dental Y-TZP (VIPI Y-TZP) submitted to conventional resistive furnace sintering (1530 °C/2h).

The mechanical strength of a Y-TZP ceramic is directly related to its microstructure, where differences in the synthesis (Lazar et al., 2008), processing steps and sintering conditions (Kelly and Denry, 2008) promote different results in mechanical strength reported in the literature (Goldstein et al., 1999). The Y-TZP ceramics that were submitted to microwave sintering at 1450 °C, independent of the cooling rate, did not present a statistical difference in mechanical strength compared with the control group (CS- conventional sintering) (Fig. 4) despite the difference in grain size (Table 2). Several authors studied the comparison of mechanical strength between the microwaved and conventional sintered Y-TZP, and no statistically significant difference in mechanical strength among both sintering techniques was observed (Almazdi et al., 2012; Marinis et al., 2013; Upadhyaya et al., 200; Wang et al., 2023). Presenda et al. (2015) observed that a lower sintering temperature could result in a smaller grain size for both conventional and microwave sintering.

Weibull statistical analysis did not show a difference in Weibull's moduli, which were between 7 and 9, within the value reported for most ceramics with modules between 5 and 15 (Johnson, 1983). Meanwhile, the MS1450RC group showed a slightly higher Weibull modulus than the other studied groups, indicating a slightly higher reliability. Also, the values of biaxial flexural strength showed a strong correlation to the Weibull distribution (close to 1).

Therefore, the densification and biaxial flexural strength for the Y-TZP submitted to microwave sintering at 1450 °C for 15 min, and rapid cooling was equivalent to the conventional sintering (1530 °C for 2 h), showing that the microwave energy sintering can be a good alternative for Y-TZP with the advantage of a drastic decrease in the total sintering time. This result concerns the specific dental Y-TZP materials tested in this study and may not be valid for other 3Y-TZP materials processed with different parameters and additives since the mechanical strength depends on the ceramic microstructure. More studies should be done to explore the effects of microwave sintering, grain size and hydrothermal aging of dental Y-TZP.

5. Conclusion

The finding of this study indicates:

- The 3Y-TZP conventional at 1530 °C for 2 h and microwave sintering at 1450 °C for 15 min showed similar density results.
- The microwave sintering method revealed a smaller grain size than CS sintering at 1530 °C for 120 min.
- Microwave sintering at 1450 °C, followed by rapid cooling, did not decrease the 3Y-TZP mechanical strength.
- The microwave sintering method has been confirmed as an efficient sintering method, reducing the total sintering time (105 min) compared to the conventional sintering (481 min), which will lead to the reduction of energy consumption and overall cost of prosthetic laboratory procedures, without decrease the Y-TZP density and biaxial mechanical strength.
- The microwave energy sintering at 1450 °C for 15 min, followed by rapid cooling, presented similar results compared with the conventional sintering, and it is suggested as a suitable protocol for the Y-TZP dental ceramics sintering.

CRediT authorship contribution statement

Nayara Fernanda Barchetta: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Formal analysis, Data curation, Conceptualization. **Anelyse Arata Found:** Writing – review & editing, Writing – original draft, Data curation, Conceptualization. **Walter Kenji Yoshito:** Formal analysis, Data curation, Conceptualization. **Valter Ussui:** Supervision, Resources, Project administration, Conceptualization. **Dolores Ribeiro Ricci Lazar:** Writing – review & editing, Writing – original draft, Visualization. **Ivan Balducci:** Validation, Software. **Sheila Butler:** Writing – review & editing. **Guilherme de Siqueira Ferreira Anzaloni Saavedra:** Supervision, Resources, Conceptualization.

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.

Data availability

Data will be made available on request.

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