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UTILIZATION OF SLIP CASTING PROCESS FOR RECYCLING CAD/CAM DENTAL ZIRCONIA WASTES

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Abstract: This study aimed to find the ideal parameters for shaping waste zirconia powders from dental laboratories using the slip-casting process. Additionally, the qualities of ceramic products created in this manner were evaluated using microstructural characterization and physical-mechanical tests. Various dental laboratories provided the waste CAD/CAM zirconia powder used in the investigation. Wastes in powder form were first calcined. Afterward, an attritor mill was used to grind the grain size until it was usable, following the completion of the grain size distribution analysis. Waste and commercial zirconia powders were combined using various dispersants to create slip-casting slurries. The rheological characteristics of these slurries were then ascertained. By evaluating the rheological properties of slip-casting slurries prepared in this way, the most suitable casting parameters were determined, and ceramic products were formed by slip-casting technique from the slurries to be prepared in accordance with these parameters. The shaped samples were dried and sintered at two different temperatures, 1400-1450°C, and samples were designed for physical, mechanical, and microstructural characterization. The pore percentages, bulk densities, and water absorption of the sintered samples, according to Archimedes' principle, as well as their strengths, were determined by the three-point bending strength test. Phase analysis was performed with XRD (X-ray diffractometer) microstructure studies with SEM (Scanning Electron Microscopy). It has been concluded that waste zirconia can be used in dental applications.

Keywords: Dental zirconia, Waste zirconia, CAD/CAM, Slip casting, Recycling

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1. Introduction

Zirconia (ZrO_2) is one of the most attractive dental materials, with the most research today. Yttria-stabilized tetragonal zirconia (Y-TSZ) was first used in biomedical applications in orthopedics in hip joints thanks to its excellent mechanical properties and biocompatibility (Christel et al., 1989; Denry and Kelly, 2008; Kelly and Benetti, 2011; Zhang and Lawn, 2018).

In dentistry, zirconia is preferred in orthodontic applications, post and support systems, and implants. Among these, its use of ceramic supports in crown and bridge prostheses constitutes the most considerable portion. High mechanical strength (900-1200 MPa) and fracture toughness (9-10 MPa.m$^{1/2}$) make using zirconia in crown and bridge applications on front and back teeth is possible. Zirconia support is obtained by processing semi-sintered and fully sintered blocks in automatic or manual devices with computer-aided design/computer-aided manufacturing (CAD/CAM) systems. While some blocks used after shaping become products, most become waste. Waste zirconia in powder and block form can be reused for different purposes by going through various processes. Considering the economic and environmental factors, it is crucial to evaluate these material wastes, which are widely used today (Shenoy and Shenoy, 2010; Madfa et al., 2014; Mundhe et al., 2015; Duraccio et al., 2015; Gautam et al., 2016; Zhang et al., 2016).

However, the use of different production methods in evaluating dental zirconia waste has been examined in a limited way in the literature, and a basic understanding has not been provided yet. Another production method that is alternative to this production method is the slip casting method. Slip casting is one of the methods used for many years in shaping ceramics. This method is carried out by pouring slurry onto the refractory mold. Thanks to the porous structure of the refractory mold, water is removed from the mud by the capillary effect. This piece is then thickened by firing at high temperatures. Ceramics shaped by slip casting have less porosity and higher density than traditional porcelains (Pröbster and Diehl, 1992; Denry, 1996; Denry and Holloway, 2010; Shen, 2013).

Gouveia et al. (2017) in their study, various physical and mechanical properties of the samples obtained by sintering the powder consisting of 100% CAD/CAM zirconia waste and the mixtures they prepared by adding 5-10-15% waste by volume to commercial zirconia powder and sintering at 1500 °C. According to their results, they concluded that the residue powders are highly suitable for prosthodontic zirconia sandblasting, as well as raw materials in refractory and pigment industries. In another study, Guazzato et al. (2004), in their research on zirconia-based ceramics, showed that the flexural strength (630±58 MPa) they obtained for In-Ceram Zirconia (IZ) processed by slip casting was higher than that of machine-processed ones (476±50 MPa). On the other hand, new applications are being tried in slip-casting studies. Roulet et al. (2021), in a survey conducted by Y-TZP, ceramics produced using a patent-pending slip casting method (Slurry, Decema GmbH) were compared with the hot isostatic pressing (HIP) method. As a result, the biaxial bending strength and characteristic strength of ceramics produced using the Slurry method were significantly higher than those of ceramics produced using HIP. In addition, dispersants are used for good shaping in ceramics. Dispersant selection is significant in obtaining a well-dispersed powder and a homogeneous ceramic suspension. The interaction of dispersant and ceramic powder is one of the most critical parameters. Therefore, it should be well characterized when choosing a dispersant. To determine dispersion properties, sedimentation and viscosity behaviors of suspensions are generally examined at the macroscopic scale (Schultz and Burckhardt, 1993; Singh et al., 2004).

Studies show that evaluating both waste zirconia and considering alternative methods is essential. Since dental zirconia is expensive and imported into our country, the country's economy needs to be recycled. Therefore, in this study, optimum parameters were determined for shaping CAD/CAM tetragonal zirconia waste powders from dental laboratories using the slip casting method, and the usability of these materials in the production of high-density ceramic materials such as dental ceramics was evaluated with various physical and mechanical tests and microstructure characterization.

2. Materials and Methods

2.1. Starting Materials

Commercial yttria-stabilized zirconia powder from Saint-Gobain Ceramics (SGZ) with high purity and waste zirconia (ZrO_2) powders (WZ) gathered from several dental prosthesis laboratories of the machining CAD/CAM were employed in this investigation. Table 1 presents the suppliers' information, including the chemical composition, grain size distribution, and specific surface area. On the other hand, Darvan 821-A and Darvan C-N (Vanderbilt Minerals, LLC) dispersants, which are used in shaping ceramics by slip casting, were used in the experiments. The first of the dispersants used in this study is Darvan 821 A, an ammonium polyacrylate with a molecular weight of 3500g/mol and pH between 7 and 8 at room temperature. The second one is Darvan C-N ammonium polymethacrylate with a molecular weight of 15000g/mol and pH between 7.5 and 9 at room temperature. In addition, polyethylene glycol was used as a binder.

Table 1. Chemical yttria-stabilized zirconia powder from saint-gobain ceramics

Chemical composition	wt%	ppm
LOI	0.61	
Y_2O_3	5.56	
Na_2O		<10
Fe_2O_3		10
SiO_2		135
TiO_2		<10
CaO		<15
MgO		25
Al_2O_3		<20
$SiO_4{}^{-2}$		<100
Cl^-		<50
Physical properties	µm	m²/g
Grain size distribution		
D10	0.15	
D50	0.57	
D90	1.44	
Specific surface area		6.4

2.2. Preparing Recycled Powder, Slip-Casting Slurry and Testing Samples

Materials such as wax processed on the same device can also be mixed into the waste powders generated from zirconia blocks processed with CAD/CAM in dental laboratories. For this reason, the waste powders were calcined at 850 °C for 2 hours to remove wax before use. The powder residues from the machining process presented a more extensive particle size distribution than the commercial powders. The commercial powders gave D50;0.57; the residues ranged from 0.1 to 1000 µm. The waste powder was ground with an attritor milling for 2 hours in isopropyl alcohol to match the commercial powder's mean size. Then, the slip-casting slurry was prepared to be 65% solid and 35% water. 6% by weight of selected dispersant and 2% by weight of binder were added and mixed in a magnetic stirrer for 2 hours. The prepared mixture was shaped by the slip-casting method. After the shaped samples were removed from the mold, they were kept covered with nylon for 4–5 days in order to dry slowly and in a humid environment, and then they were dried in an oven at a speed of 5 °C/hour to 100 °C and dried at this temperature for 1 night. The dried samples were pre-sintered at 1000 °C, and then their surfaces were smoothed and their dimensions adjusted using grinding and polishing machines. The oven was allowed to cool naturally after the sintering process, which was conducted at temperatures of 1400 and 1450 °C with a heating rate of 5 °C/min for two hours.

2.3. Characterization

In the first phase of this study, Zeta potential and sedimentation tests were carried out to determine the optimum parameters for shaping two groups of samples consisting of zirconia and waste zirconia powders. Zeta potential measurements were made between pH 1-10 to determine both zirconia powders' isoelectrostatic points (IEP). Measurements were made on the Malvern Nano-Z Zeta potential measuring device. Sedimentation tests were conducted on zirconia powders in the pH range of 1 to 10. Subsequently, two distinct dispersants were added at pH 7 with different additive ratios for both zirconia powders. Tests were performed using 4 wt% zirconia powder in 25 mL tubes. First, the pH of the required amount of deionized water for each pH was fixed using HCl or NaOH. Then, the same amount of powder was weighed for each tube, added into the pH-adjusted water, and mixed in a magnetic stirrer for 4 hours. At the end of 4 hours, pH measurement was made, the suspension was transferred into the tube, its mouth was closed, and the height of the suspension was measured (h0). After being kept vertically for 24 hours, the height of the precipitate formed was measured and recorded (h). Finally, h/h0 values were calculated for the tubes prepared according to each pH value. These processes were repeated for both zirconia and dispersants for increasing amounts of dispersant (0-0.2-0.4-0.6-0.8-1 wt%) at pH 7. The total porosity, mass density, and water absorption percentages of the sintered samples were calculated based on Archimedes' method. The equation 1 calculated the relative density:

$$D = {}^{\rho}/_{\rho_0}\, x\ 100 \qquad (1)$$

where ρ_0 is the theoretical density of yttrium-stabilized tetragonal zirconia polycrystals (6.10 g/cm^3 (Jiang et al., 2011). A cold modulus of rupture (3-point bending strength) test was applied to the samples to measure the strength of the materials. The tests were conducted on a Shimadzu AG-IS model 100kN capacity mechanical testing device. Three-point bending strength tests followed ISO 6872 'Dental Ceramic' Standard. Furthermore, SEM microstructure investigations and XRD phase analysis were performed on the samples.

3. Results and Discussion

3.1. Waste Zirconia Characterization

First, it was necessary first to analyze the crystal phases and particle sizes to comprehend the existing state of waste zirconia. Figure 1a presents the particle size distribution, and Figure 1b X-ray diffraction pattern of sample WZ. The waste powders are currently undergoing examination for their potential application in dental ceramics. Their particle size distribution is quite complex, ranging from 0.1-1000 µm, and featuring multimodal modes. Upon conducting X-ray diffraction analysis, it was discovered that the powders contain monoclinic and tetragonal zirconia crystalline phases, which are highly desirable for dental ceramics. No other phases indicating contamination or impurities were detected during the analysis. Based on these findings, it can be confidently concluded that the waste powders are suitable for dental ceramics.

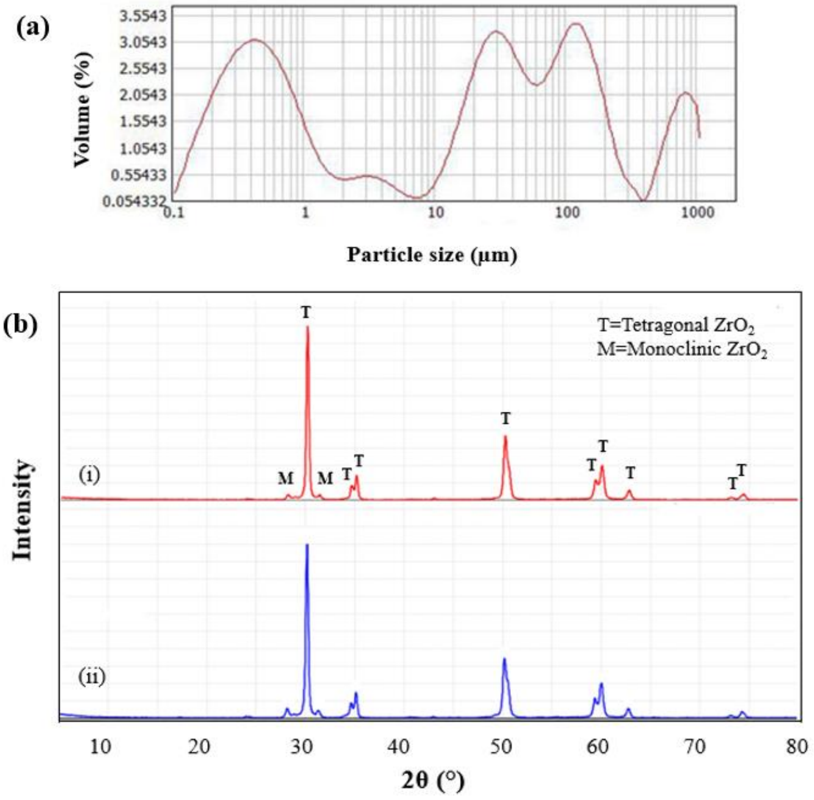

Figure 1. (a) Particle size distribution of waste zirconia (b)Waste zirconia, i-block, ii-powder XRD patterns.

3.2. Zeta Potential - Sedimentation Analysis and Preparing Testing Samples

The rheological properties of ceramic slurry depend on its pH, which determines its zeta potential. Zeta potential was measured to find the isoelectric points of SGZ and WZ samples. As a result of zeta potential measurements, it was observed that the IEP points of SGZ zirconia and WZ powders before the addition of dispersant were between pH = 6-7. Zeta potential plots are given in Figures 2a and 2b, respectively. It is seen that both zirconia have negative zeta potential values at pH values above IEP and positive zeta potential values at pH values below IEP. The obtained values agree with the reported values (Schultz and Burckhardt, 1993; Greenwood and Kendall, 1999; Agrafiotis et al., 2000).

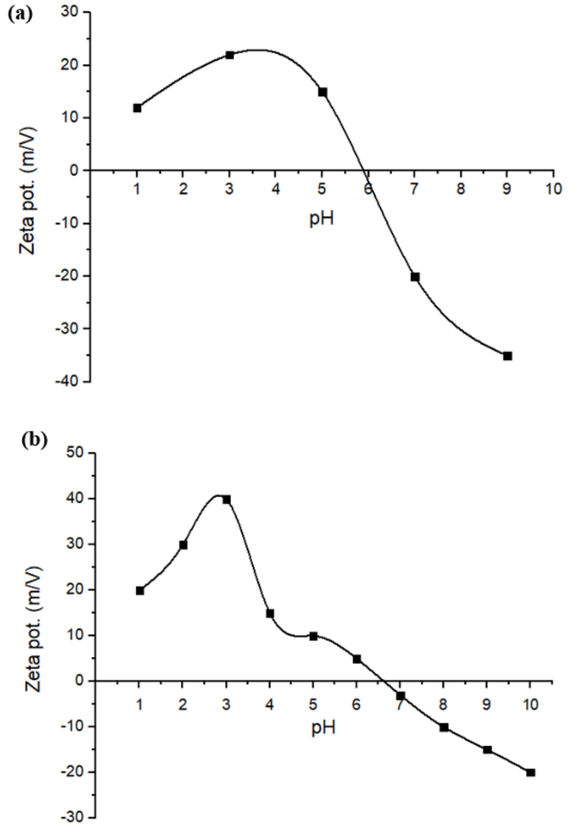

Figure 2. (a) SGZ and (b) WZ zeta potential graphs of zirconia at different pHs.

In the ceramic industry, knowing which dispersant provides the best stability in suspension is important. Determining how much dispersant should be added for economic reasons is vital (Greenwood and Kendall, 1999). For this purpose, Darvan 821-A and Darvan C-N dispersants were added to SGZ and WZ samples at ratios 0-0.2-0.4-0.6-0.8-1 wt% at pH 7, and sedimentation experiments were carried out. Both Darvans belong to the class of ammonium polymeric monomers that can disperse in water. This deflocculant can be used in ceramic dispersions, giving low viscosity slip and low foam production, being proper for extended times of mixture or grinding. Figures 3(a) and (b) show the sedimentation rates varying according to the amount of Darvan 821-A and Darvan C-N dispersants added to SGZ and WZ, respectively. In both graphs, it is observed that the sedimentation rate decreases as the amount of dispersant increases. Both waste zirconia and commercial zirconia behaved more stable in Darvan C-N dispersant. Since there was no change in sedimentation rate after 0.6%, this rate was chosen for slurry containing 65% solid and 35% water. The same dispersant was used in previous studies on dental zirconia production using the slip casting method (Kim and Lee, 2020).

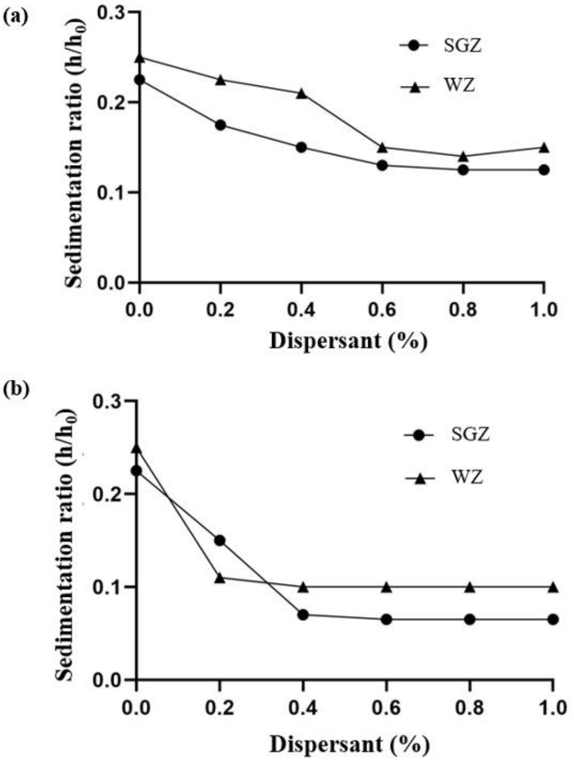

Figure 3. Changing sedimentation rates of waste zirconia and S.G. zirconia at pH7 with increasing rates of (a) Darvan 821A (b) Darvan C-N additive.

After determining the dispersion conditions, the slip-casting masses were prepared and transferred to the plaster mold, as can be seen in Figure 4a. Pre-heat treatment was applied at 1000 °C to ensure condensation after shaping and to smooth the surface. The sample obtained after pre-heat treatment is seen in Figure 4b.

3.3. Characterization of the Sintered Samples

XRD patterns of waste zirconia and commercial zirconia samples sintered at 1450 °C are given in Figure 5. When the XRD patterns are examined, it is seen that there is no change in the crystallographic structure after sintering; waste zirconia is mainly composed of tetragonal zirconia and contains a deficient proportion of monoclinic zirconia phase. In commercial zirconia, slightly higher monoclinic zirconia peaks were observed after sintering than in waste zirconia. Garvie et al. (1990) stated that zirconia can increase both the strength and toughness of ceramics due to the transformation of metastable tetragonal zirconia grains to monoclinic form by the effect of the stress field in front of a crack (Stevens 1986).

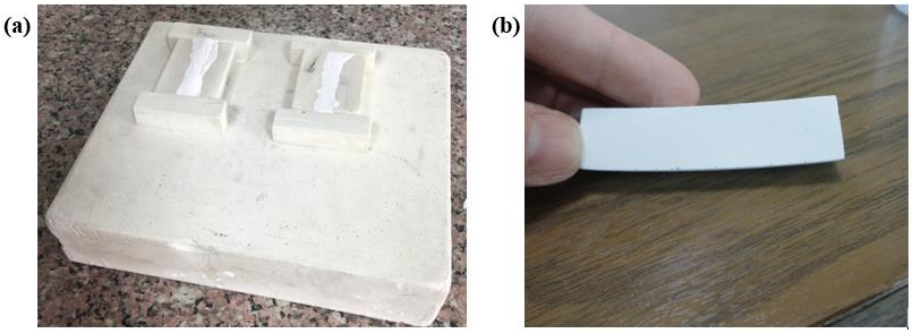

Figure 4. Image of (a) slip casting masses transferred to plaster mold (b) the sample obtained after pre-heat treatment.

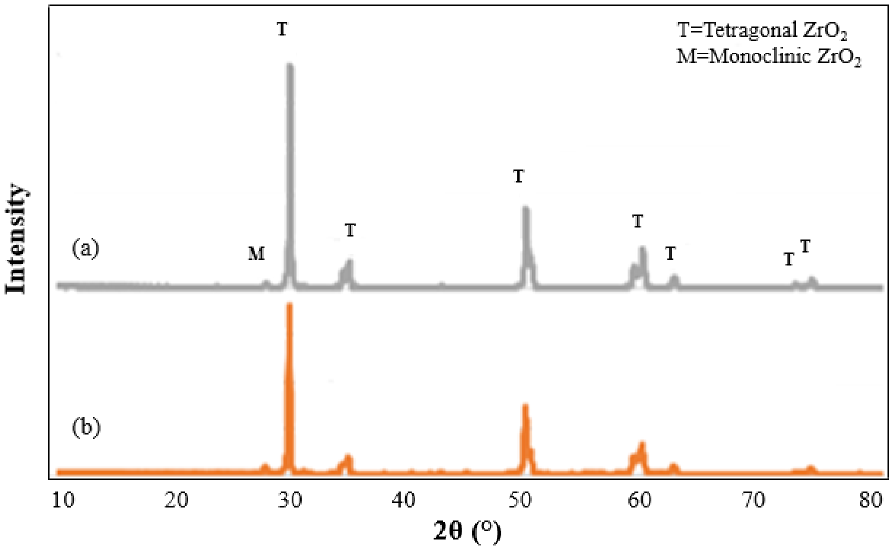

Figure 5. XRD patterns of samples containing 100% waste zirconia (a) and 100% commercial zirconia (b) sintered at 1450 °C.

The tetragonal-monoclinic phase transformation, similar to the martensitic transformation seen in steels, results in a 3 to 5% volume increase in the structure. Thus, in response to the tensile stress caused by the crack, a compressive stress field is formed in the matrix, especially at the crack's tip. This situation prevents crack propagation and increases the toughness of the ceramic (Christel et al., 1989; Piconi and Maccauro, 1999; Grigoriadou, 2006). For this reason, the waste zirconia must have the desired tetragonal structure so that it can be reused, especially in dental applications that require high fracture toughness.

Microstructure and grain size were analyzed by scanning electron microscopy. This analysis mainly aimed to compare waste and commercial zirconia. Figure 6 shows SEM images of waste zirconia sintered at 1400 °C and 1450 °C at high and low magnifications. It is clear from the microstructures that homogeneous and dense structures were obtained. As expected, relatively less porosity and a denser structure were observed in the samples sintered at 1450 °C. As the temperature increased, the crystal structure of zirconia became denser, reducing porosity, defects, and flaws. Figure 7 shows SEM images of commercial zirconia sintered at 1400 °C and 1450 °C at different magnifications. When the images of both samples are compared, it is seen that the microstructure obtained with waste zirconia is very similar to the microstructure of the samples obtained using commercial zirconia.

Figures 8a and 8b show the relative density and porosity values of sintered ceramics of waste and commercial zirconia as a function of temperature, respectively. The density increases, and porosity decreases with increasing temperature. According to Figure 8a, the relative density increased from 0.96 to 0.98 when the temperature was increased from 1400°C to 1450 °C, respectively. In addition, porosity decreased with increasing temperature. Density increases and porosity should decrease with increasing temperature. In Figure 8b, it is also seen that the relative density increases, and porosity decreases with increasing temperature. However, the density increased to 0.98% in waste zirconia and remained around 0.94% in commercial zirconia. The porosity value at 1450 °C was 1.95% in waste zirconia and 5.63% in commercial zirconia. The nearly 3-fold difference in porosity values increases the importance of using waste zirconia.

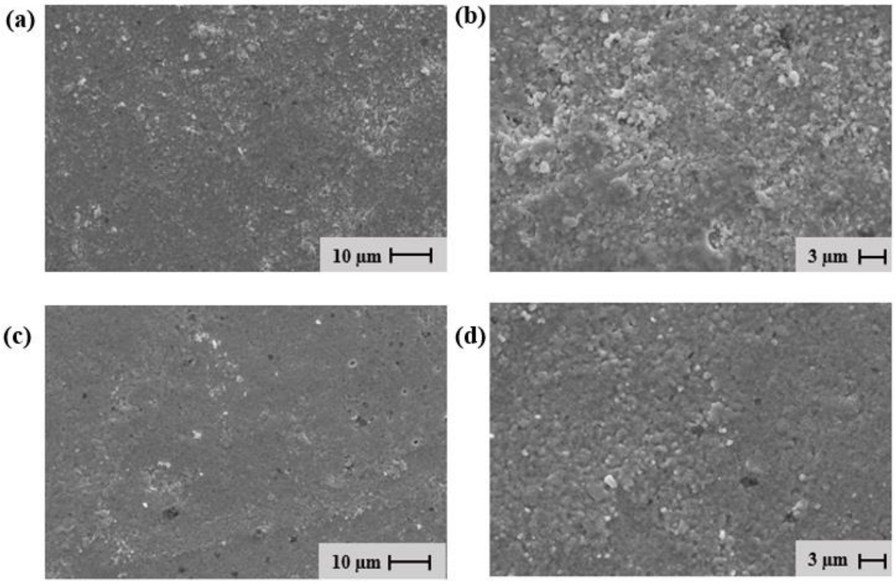

Figure 6. SEM images of waste zirconia samples sintered at different temperatures (a) - (b); 1400 °C, (c) - (d); 1450 °C.

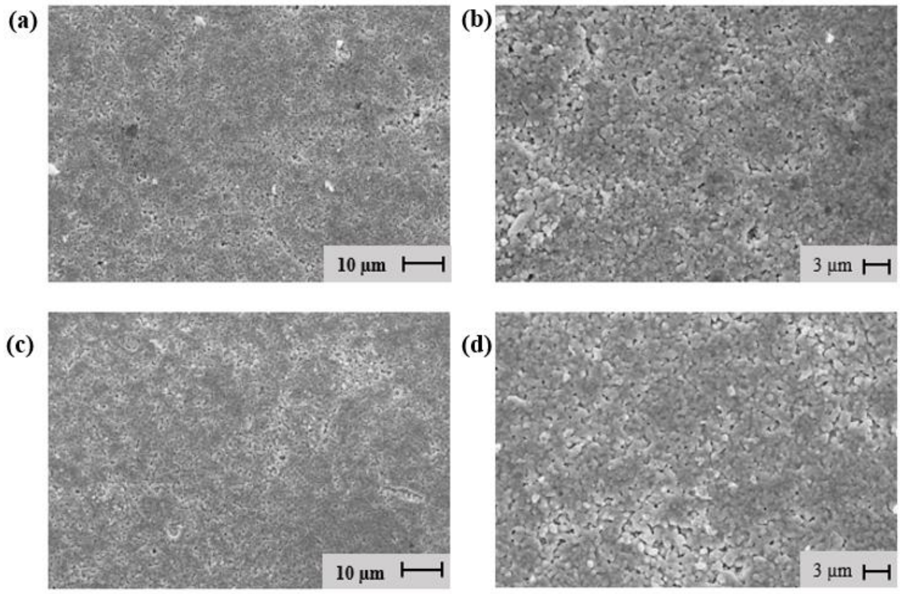

Figure 7. SEM images of commercial zirconia samples sintered at different temperatures (a) - (b); 1400 °C, (c) - (d); 1450 °C.

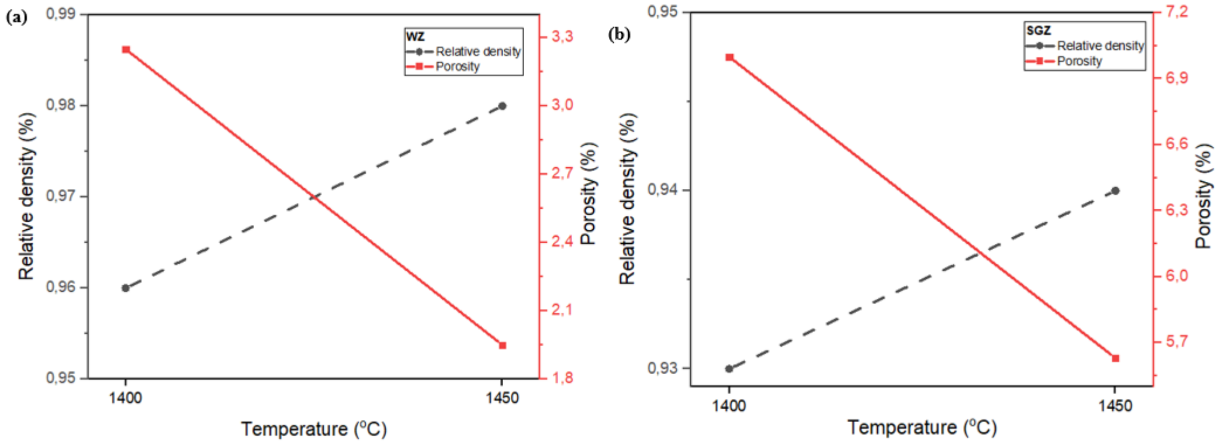

Figure 8. Evolution of relative density and porosity of (a) waste and (b) commercial zirconia as a function of temperature.

Figure 9 shows the 3-point flexural strength test results of waste and commercial zirconia sintered at 1400 and 1450 °C. According to this, it is seen that the bending strength of the samples obtained from waste at both temperatures is higher than the commercially purchased zirconia samples. The surface flaws of the tensile-stressed surface highly influence the 3-point bending strength of dental ceramics. This is because fractures typically initiate from surface defects. Additionally, the flexural strength of dental ceramics can be impacted by the shape and size of the specimens, as well as the test setup conditions (Jin et al., 2004).

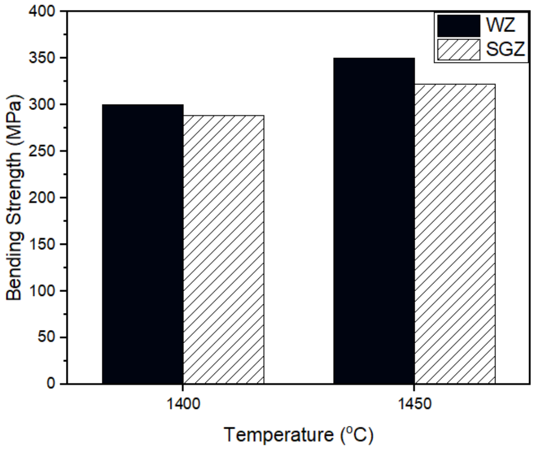

Figure 9. Bending strength of waste and commercial zirconia as a function of temperature.

4. Conclusion

As per the study's findings, the waste zirconia sintered at 1450 °C exhibited superior relative density (0.98%) and bending strength (350 MPa) compared to the commercially available Saint Gobain zirconia. Additionally, the porosity of the former was observed to be lower. These results imply that waste zirconia can be a feasible alternative to Saint Gobain zirconia in specific industrial applications. Based on the study's results, the powdered residues exhibit exceptional properties that make them highly suitable for dental ceramic zirconia. Therefore, it is possible to promote the utilization of waste powder generated from the zirconia CAD/CAM milling process. At the same time, this study has shown that dental ceramics can also be shaped by different methods, thus enabling less material loss. In the continuation of this study, zirconia wastes produced by other companies can be evaluated. The waste method is extremely important for the country's economy. With this study, it has once again shown that waste management is critical in every field.

Author Contributions

The percentage of the author(s) contributions is presented below. All authors reviewed and approved the final version of the manuscript.

	C.B.E.A.	H.Ş.Ç.T.	E.A.
C	100		
D	100		
S	100		
DCP	60		40
DAI	80		20
L	40	30	30
W	50	50	
CR	40	30	30
SR	40	30	30
PM	100		
FA	100		

C=Concept, D= design, S= supervision, DCP= data collection and/or processing, DAI= data analysis and/or interpretation, L= literature search, W= writing, CR= critical review, SR= submission and revision, PM= project management, FA= funding acquisition.

Conflict of Interest

The authors declared that there is no conflict of interest.

Ethical Consideration

Ethics committee approval was not required for this study because of there was no study on animals or humans.

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