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Porosity and pore morphology characteristics of zirconia-alumina bioceramics

Rusul Ahmed Shakir* and Róbert Géber

Faculty of Materials and Chemical Engineering, Institute of Energy – Ceramic and Polymer Technology, University of Miskolc, Miskolc-Egyetemváros, Hungary

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ABSTRACT

Manufacturing ceramic green structures using starch consolidation casting is an established process that is simple, non-hazard, and low-cost. In this study, starch consolidation casting is used to prepare ceramics based on submicron monoclinic zirconia with additions of alumina and magnesia. Scanning electron microscopy results indicate that the size of pores decreased and the morphological irregularity increased when the tapioca starch content increased. The sample with 30 wt.% tapioca starch in a 55 wt.% slurry concentration had the highest estimated apparent porosity (around 56%), whereas the sample with 10 wt.% in a 68 wt.% suspension concentration had the lowest (about 35%).

KEYWORDS

biomaterials, porous ceramics, starch consolidation casting, tapioca starch, pore morphology

1. INTRODUCTION

The major purpose of porous ceramic materials is that they combine the beneficial features of the controlled microstructure of ceramic and varying grades of porosity. Porous ceramics that have closed pores used in insulation applications [1, 2], while porous ceramics with open pores may serve as filtration systems [3, 4]; membranes [5, 6]; support for catalyst [7, 8]; biomaterials [9–12]; ceramic burner [13, 14]; gas sensors [15, 16]; therefore, different microstructures and, hence, different preparation methods are required for porous ceramic bodies. Ceramic implants with certain porosity are preferred in bio-ceramics because they facilitate tissue integration [17]. Several methods, including direct foaming, sacrificial template, and gel-casting, have been devised to create porous ceramics [18–23]. Lyckfeldt and Ferreira [24] created and initially published this method. The principle behind starch consolidation casting is the subsequent capacity of starch for absorbing water coming from the ceramic slurries due to its swelling in water at increased temperature. Heat up to 80 °C in the starch/ceramic mixture causes the intermolecular connections inside the granules to weaken, resulting in fast and irreversible swelling by water absorption. The swelling and gelatinizing of starch granules, forcing the ceramic particles to compressed and packed into the spaces between the starch grains. This transforms the water-based ceramic slurries into a green body that can then be dried and burned [25]. Sintering of ceramics at high temperatures pyrolyzes and burns the starch, while the sintering of the ceramic matrix continues. During this stage of processing at high temperatures, nothing but pores remain from the starch. The size of the formed pores is depending on the swelling of starch and shrinkage of ceramic matrix, while the porosity or volume fraction of the formed pores is closely associated is strongly correlated with the starch weight percent content [26–29].

Zirconia has been used in many studies as a support for alumina in porous structured ceramic composites, known as zirconia toughened alumina. However, the impact of alumina on zirconia, referred to as alumina toughened zirconia, has been less well studied. In the present investigation, porous-structured ceramic composites of ZrO_2–Al_2O_3–MgO were

*Corresponding author.
E-mail: rusulahmed1990@gmail.com

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formed utilizing tapioca starch as the pore and body forming agent. The current work employed Scanning Electron Microscopy (SEM) to investigate the impact of ceramic oxides and tapioca starch on the porosity and pore morphology of the composites that were prepared. Moreover, determine how adding of varying quantities of the starches in a variety of suspension concentrations influences the apparent density and apparent porosity, and also on the volume shrinkage of the prepared porous structures.

2. EXPERIMENTAL WORK

2.1. Materials

Ceramic oxides like monoclinic zirconia, alumina, and magnesia were chosen in this research as biomedical substances because of their biological compatibility, excellent mechanical characteristics, and environmental inertness. As a pore-forming and body-forming agent, tapioca starch was chosen to give the solidified body strength and make it easier to de-mold before drying. Dolapix CE64 deflocculant and distilled water were both used as basic requirements in the production of slurries. Table 1 detailed the purity, density, typical particle size and raw-material supplier.

2.2. Preparing of the slip slurries

As the initial stage the milling machine was used to blend various concentrations of ceramic powders at a speed of 60 rpm for about 5 h. Figure 1 shows the slurries preparation procedure.

Slurries were prepared using 1.5 wt.% of the deflocculant in the distilled water and a magnetic stirring device with 600 rpm stirring speed for 2 h at ambient temperatures. The various mixed compositions that were produced for this study are detailed in Table 2.

2.3. Preparation of molds

The 3D printing technique was used to produce an impermeable plastic mold for use in the casting process. High impact polystyrene filament was utilized as a printing material for preparation of these molds.

2.4. Starch consolidation casting technique

With the starch consolidation casting (SCC) technique, plastic molds were filled with slurries of varying concentrations. Plastic wrap was then used to completely cover the filled plastic molds to prevent the water from evaporating and to

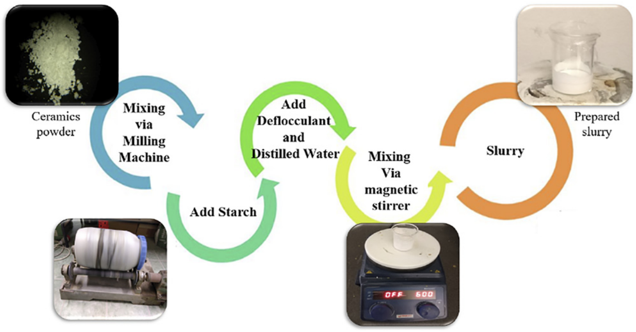

Fig. 1. Slurries preparation procedure

allow the starch granules to absorb that water from the suspension and subsequently to swell. The mold holding the slurry was placed in an 80 °C laboratory oven for a period of two hours to allow the starch to swell. The ceramic bodies were demolded the next day after setting overnight at room temperature. Then, the dried samples were pre-sintered at 1,150 °C and sintered at 1,600 °C for 2 h with a heating and cooling rate of 2 °C/min in the laboratory furnace. Figure 2 shows the starch consolidation casting procedures. Pre-sintering is a process that is used to decrease shrinkage during the final sintering step and to minimize thermal stress. It does this by enabling the ceramic material to undergo partial densification in the pre-sintering stage. Thermal stress and cracking may occur as a result of rapid and uneven heating during the final sintering process. Pre-sintering mitigates these concerns by dividing the densification process into two parts.

2.5. Characterization techniques

The pycnometer was used to measure the density of zirconia, alumina, and magnesia ceramic powders. CILAS 715 apparatus was used to describe the size distribution and the average grain size of the ceramic powder particles. Experiments were performed at laboratory temperature with adding of two drops of a dilute solution of sodium tripolyphosphate. The purpose of adding the dilute solution of sodium tripolyphosphate is to improve the dispersion of ceramic particles in the distilled water and avoid their accumulation. The ultrasonic treatment, which lasted for 60 s, aided in the dispersion process. Using laboratory sieving analytical equipment, the size distribution of the tapioca starch particles was evaluated. In the sieve analysis, it is assumed that all particles are completely round and thus pass through the square opening when their diameter is smaller than the size of the square aperture in the screen.

Table 1. Characteristics and source of the base materials

Material	Purity	Density (g cm^{-3})	Average particle size (μm)	Source
m-ZrO_2	99.99	5.64	0.10	Interkeram Kft. (distributor)
Al_2O_3	99.99	3.13	0.29	Interkeram Kft. (distributor)
MgO	99.5	3.56	0.25	Sigmaaldrich.com, Germany
Tapioca starch	–	–	45	Balance food Kft. (distributor)

Table 2. Mix compositions

m-ZrO_2	MgO	Al_2O_3	Deflocculant	Tapioca starch	Suspension Conc.
72.5	2.5	25	1.5	10	62
72.5	2.5	25	1.5	10	65
72.5	2.5	25	1.5	10	68
72.5	2.5	25	1.5	20	62
72.5	2.5	25	1.5	20	65
72.5	2.5	25	1.5	20	68
72.5	2.5	25	1.5	30	62
72.5	2.5	25	1.5	30	65
72.5	2.5	25	1.5	30	68

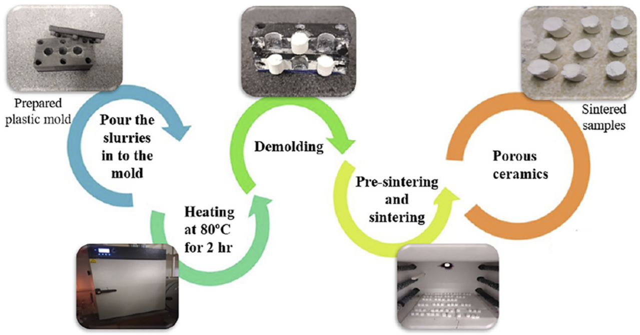

Fig. 2. The starch consolidation casting procedures

The present study used Scanning Electron Microscopy (SEM) (Carl Zeiss EVO MA10) to examine the influence of ceramic oxides and tapioca starch on the porosity and pore morphology of the produced composites. Through the use of Archimedes' principle, apparent densities of the prepared porous samples were calculated. Measurements of apparent porosity in the prepared porous samples were made using the water absorption technique. For estimating the Apparent Porosity (AP), the weight of porous samples was measured after sintering ($w_{sint.}$), Following that, after a 24-h soaking in lap-temperature water ($w_{imm.}$), in addition, after being saturated in distilled water ($w_{sat.}$). The following equation was used to calculate the AP:

$$AP = \frac{W_{imm} - W_{sint}}{W_{imm} - W_{sat}} \cdot 100\%. \tag{1}$$

The determination of volume shrinkage (V_s) in the porous-structured samples was conducted by measuring the initial volume of the samples (V_0) before to sintering at a temperature of 1,600 °C, as well as the final volume of the samples (V_1) following the sintering process. The determination of sample volume is achieved by computing the mean value of three measurements obtained via the use of a digital Vernier caliper. The following equation used for volume shrinkage calculation:

$$V_s = \frac{V_0 - V_1}{V_0} \cdot 100(\%). \tag{2}$$

The apparent density, apparent porosity, and volume shrinkage tests were conducted three times for the same composition, averaging the results each time to obtain the most accurate representation of the measured value.

3. RESULTS AND DISCUSSION

3.1. Characterization of tapioca starch

The shape, size, and quantity of pore-forming particles have a significant impact on the porous structure that has been generated. In the biomedical application, the shape and size of generated pores must be suitable to facilitate the cell migration, proliferation and vascularization. The increasing surface area of porous materials, on the other hand, leads to improved bonding with host tissues. As it is shown in Fig. 3, the scanning electron microscopy was used to evaluate the tapioca starch particle shape. The SEM image shows that the tapioca starch particles present approximately round (spherical) shapes with different particle size distribution.

The sieve analysis results of tapioca starch powder are shown in Fig. 4. The particle diameters of tapioca starch are around 78, 48 and 28 μm for 90, 50 and 10% cumulative over size, respectively.

3.2. Apparent density of porous-structured ceramics

Packing behavior of the ceramic particles in the slurries and the gelatinization operation that occurs throughout the casting are the primary causes of densification. Figure 5 shows how the starch content in the slurries correlates with the apparent densities of the porous samples.

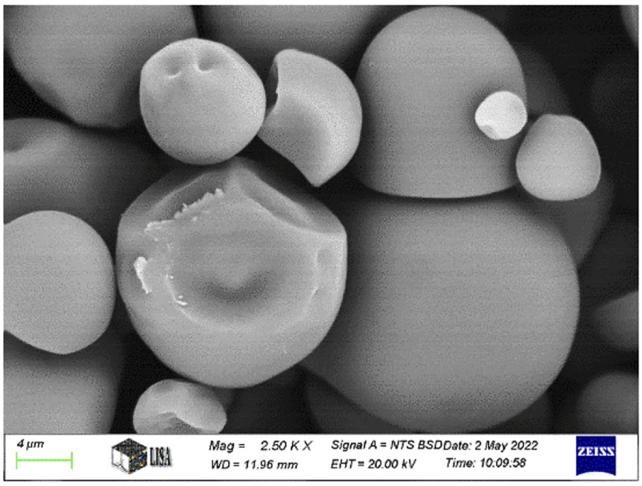

Fig. 3. SEM image for potato starch particles

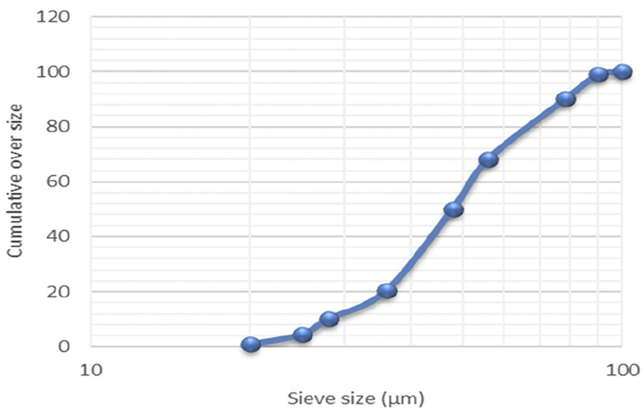

Fig. 4. Particle size distribution of tapioca starch

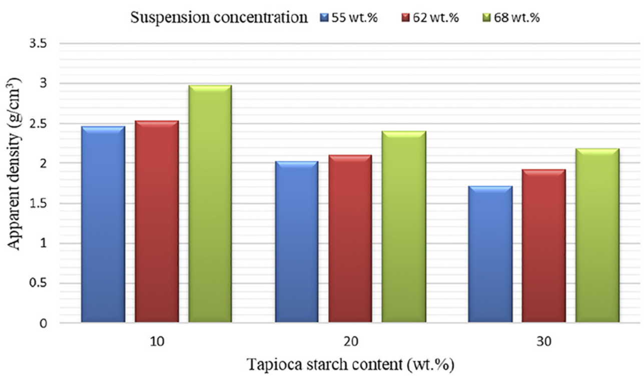

Fig. 5. Effect of tapioca starch content on apparent density

The results show that the apparent densities produced by the sintered specimens are correlated with the quantities of starch present. Increasing tapioca starch results in less apparent density in the sintered samples. As more starch was added, fewer ceramic granules could be packed into the same green volume, resulting in lower density. The results also demonstrate that the apparent densities of the porous specimens effected by the concentration of the suspensions. As the concentration of the suspensions increase, the apparent densities of the porous specimens increase.

3.3. Apparent porosity of porous-structured ceramics

Figure 6 compares the apparent porosity of many types of porous-structured ceramics produced by utilizing varying concentration of tapioca starch.

This figure indicates that the amount of the nominal starch in the slurries is an important factor in determining the final porosity of the ceramics (after burnout). Due to the raised starch concentration present in the slurry, a considerable quantity of moisture from the surrounding environment may be drawn towards it. Consequently, the slurry will undergo gelatinization and experience swell throughout the drying process. During the sintering process, the combustion of starch will occur, leading to the formation of pores inside the structure. These pores will result in the structure having a lower density and a higher porosity. Additionally, for the majority of samples, the results indicate that an increase in slurries concentrations (which implies a decrease in the amount of water inside the slurry) induces a decrease in the apparent porosity. The sample consisting of 30 wt.% of tapioca starch in a 55 wt.% slurry concentration had the greatest estimated apparent porosity (near 56%), whereas the sample consisting of 10 wt.% of tapioca starch in a 68 wt.% suspension concentration had the lowest (around 35%).

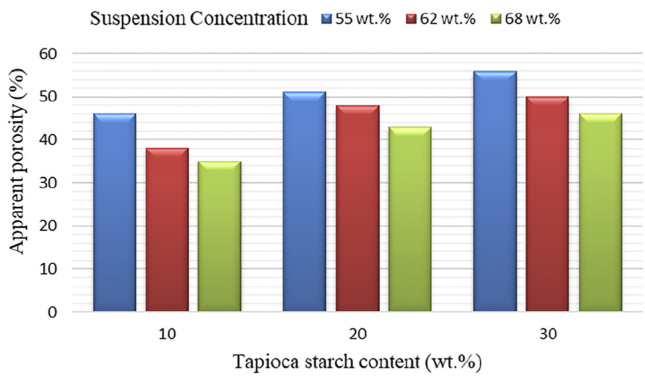

Fig. 6. Effect of tapioca starch content on apparent porosity

3.4. Volume shrinkage of porous-structured ceramics

Figure 7 provides a compared study for the volume shrinkage values subsequent to burnout, taking into account the suspension concentration and the quantity of starch inside the suspension. Figure 7 shows that volume shrinkage is reduced with increasing ceramic powder concentration and decreasing water content in the solution. Furthermore, the findings indicate that the magnitude of volume shrinkage is positively correlated with the quantity of starch content. The distinct swelling kinetics of tapioca starch and the consequent variable relative densities of the ceramics matrix inside the green body are the primary factors responsible for the reliance of shrinkage on the starch type. Higher shrinkage may occur at high starch concentrations and low suspension concentrations due to the free water content in suspension, which is required for full swelling of the starch particles, decreasing the ceramic particle packing density between starch particles.

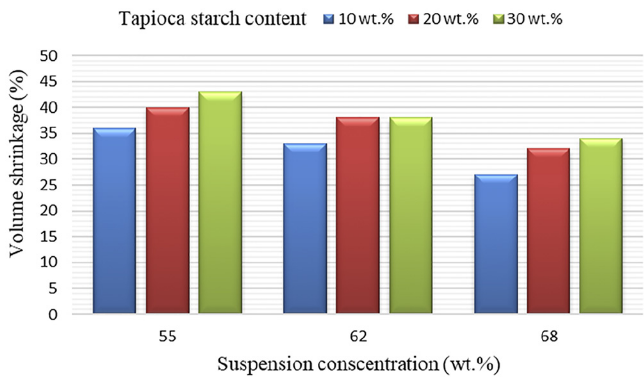

Fig. 7. Volume shrinkage via suspension concentration using potato starch

3.5. Scanning electron microscopy of porous-structured ceramics

Figure 8 depicts a sample with a suspension concentration equal to 68 wt.% with a concentration equal to 10 wt.% of tapioca starch. The present sample exhibits a significantly reduced pore fraction (nearly 35%) compared to others prepared using tapioca starch. The zirconia particles are brighter than the alumina particles. The microstructure in this sample is quite uniform, and the produced phase does not seem to have a significant agglomeration. Therefore, it is deemed that the utilization of both the milling stage, lasting for duration of 5 h, and the magnetic stirrer, used for a period of 2 h, yields composites characterized by a highly refined and uniform microstructure.

The scanning electron micrograph in Fig. 9a depicts a 10 wt.% tapioca starch with 68 wt.% suspension sample, exhibiting apparent porosity of roughly 35%. There is a relatively small amount of starch, indicating that the pores exhibit full closure and possess a circular morphology. In Fig. 9b, the suspension concentration is 55 wt.% with 30 wt.% tapioca starch presents. As compared to other samples, this one seems to have a higher porosity that is around 56%. The micrograph suggests that there are a many of pores in this sample, but closer inspection reveals that they are irregularly shaped. It can be deduced from the results that as the concentration of tapioca starch increased, the pore size reduced and the morphological irregularity increased. These findings are consistent with those of Garrido et al. [30]. In a general sense, it is evident that porous ceramic samples produced from suspensions containing a lower concentration of starch 10 wt.% exhibit much bigger holes compared to those derived from slurries with a greater amount of starch 30 wt.%. This conclusion confirms a significant variety of results that have been proved to be equivalent in earlier studies [24, 27, 31, 32], and is consistent with idea of the starch consolidation casting.

Figure 10 is a scanning electron micrograph illustrating the presence of ceramic shells within the pores. The creation of these shells may be attributed in large part to the connection between the ceramic particles and the starch

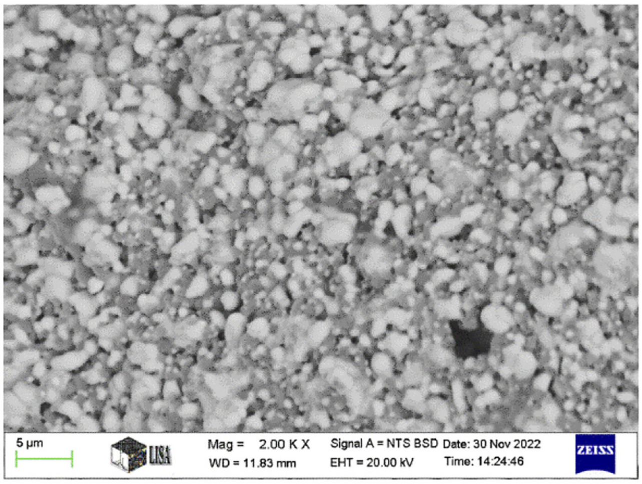

Fig. 8. SEM photograph of a 10 wt.% starch, 68 wt.% suspension sample

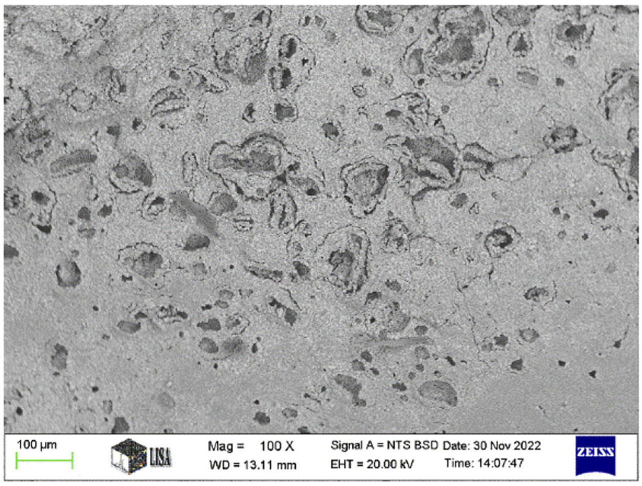

Fig. 10. Ceramic shells inside the pores

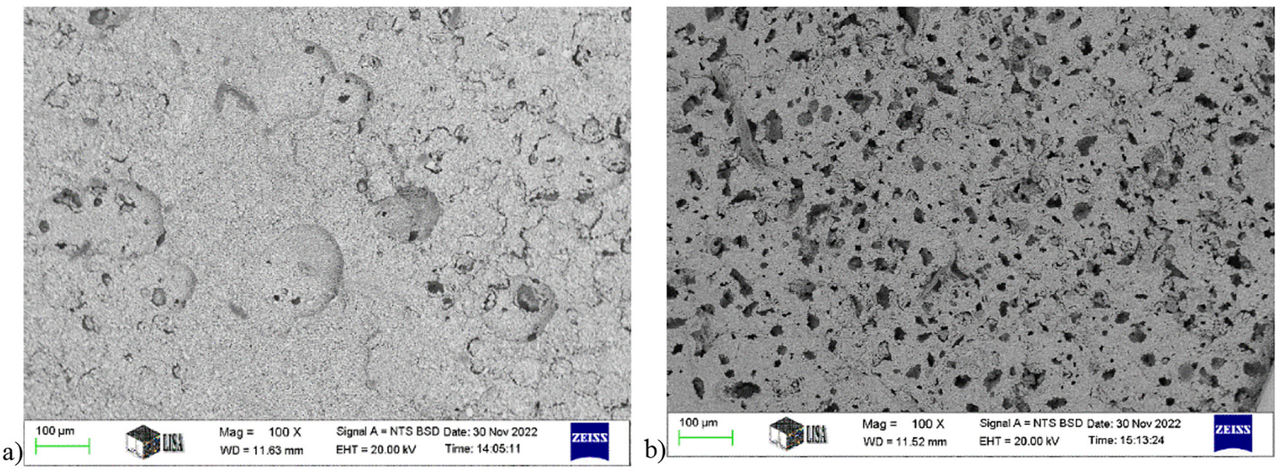

Fig. 9. SEM micrographs with a) 10 wt.% and b) 30 wt.% of tapioca starch

granules. When heated, a layer of ceramic powder adhering to the starch granules surface detaches from the remainder of the enclosing matrix and migrates in a congruent trajectory with the starch particles.

The diameters of the originating spherical pores with 10 wt.% tapioca starch ranged from 156.0 to 259.3 μm, but the diameters of the pores that were generated with 30 wt.% tapioca starch were less than 35 μm, as it is shown in Fig. 11a and b. The starch consolidation casting process involves the absorption of water by starch, causing it to expand, when the temperature exceeds 50 °C. The starch granules are unable to expand to their maximum potential when the starch concentration increases because there is not enough water in the solution. To accomplish total swelling, a significant quantity of water is necessary in order to increase the starch content. When the starch content is about 30 wt.%, the pore size is smaller than the size of starch granules due to the volume shrinkage that occurs to the ceramic material during sintering. As the volume shrinkage measurement results showed, the volume shrinkage increased with an increase in the starch content.

4. THE PEARSON CORRELATION MATRIX OF THE EXPERIMENTAL DATA

The Pearson correlation coefficient values for the experimental data were calculated by using data analysis tool Pak in Microsoft Excel to represent the strength of correlation between the experimental data. Table 3 shows all the experimental data in the Excel sheet. Table 4 shows the correlation matrix of the experimental data. The abbreviations T.S., Susp., A.D., A.P., and V.S., stand for the tapioca starch content, suspension concentration, apparent density, apparent porosity, and volume shrinkage, respectively.

The correlation coefficient values range from +1 (which means that there is a perfectly positive linear correlation) to −1 (which means that there is a perfectly negative linear correlation), while the zero value mean that there is no linear correlation between the two variables. The results of

Table 3. The experimental data Excel sheet

Tapioca starch wt.%	Suspension wt.%	Apparent density (g cm^{-3})	Apparent porosity (%)	Volume shrinkage (%)
10	55	2.46	46	40
20	55	2.03	51	44
30	55	1.72	56	47
10	62	2.53	38	37
20	62	2.10	48	42
30	62	1.92	50	42
10	68	2.98	35	31
20	68	2.40	43	36
30	68	2.19	46	38

Table 4. The correlation matrix

	T.S. (wt.%)	Susp. (wt.%)	A.D. (g cm^{-3})	A.P. (%)	V.S. (%)
T.S. (wt.%)	1				
Susp. (wt.%)	0	1			
A.D. (g cm^{-3})	−0.8177	0.5125	1		
A.P. (%)	0.7318	−0.6453	−0.9523	1	
V.S. (%)	0.5749	−0.7813	−0.9306	0.9529	1

Pearson correlation matrix indicate that there is a strong negative correlation between the starch content and the apparent density, which mean that with increasing of starch content the apparent density decrease. The results also show that there is a strong positive correlation between the starch content and the apparent porosity. The suspension concentration has a strong negative correlation with the volume shrinkage, which mean that with decreasing of water content the volume shrinkage decreases.

5. CONCLUSION

This study focuses on the synthesis and characterization of Starch Consolidation Cast (SCC) porous oxide ceramics

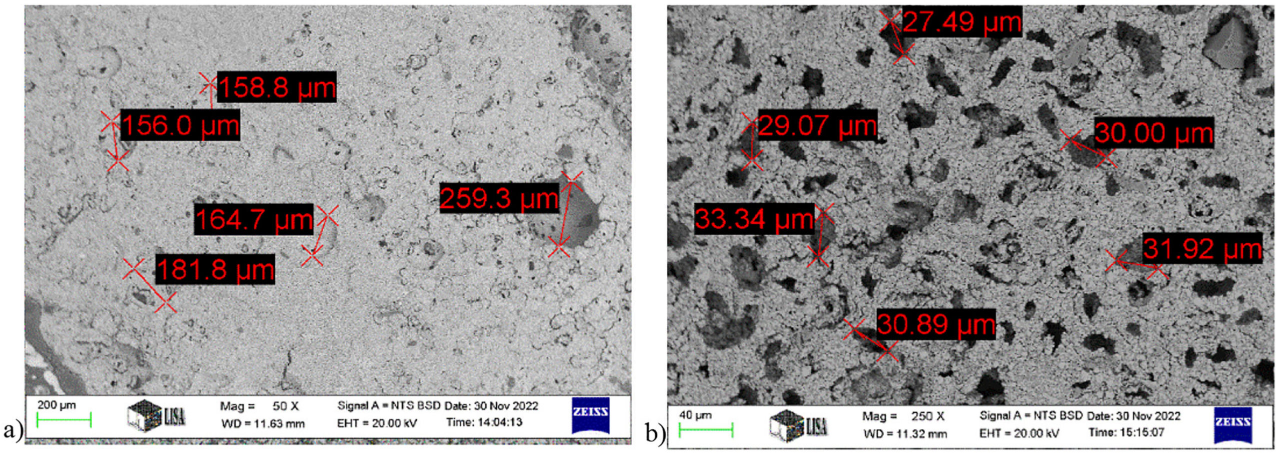

Fig. 11. Pore size for samples with a) 10 wt.% and b) 30 wt.% of tapioca starch

(made from ZrO_2, Al_2O_3, and MgO system) fabricated using starch of tapioca as the agent for pore and ceramic body forming. One of the most promising shaping techniques for the manufacturing of the porous structures is the starch consolidation casting procedure, which benefits from advantageous properties of starch like water dispersibility, gelling potential, flame retardancy, and economical value. If the slurry has a high starch content, it will absorb a lot of water from the environment and then gelatinize and swell as it dries. During sintering, the starch will be burnt, creating pores in the structure. The results of the density measurements indicate a negative correlation between the densities of the sintered specimens and the quantity of starch present. In summary, the observed porosity measurement indicates that the overall porosity of ceramics is influenced by the prescribed starch concentration in the solution. A decrease in the values of apparent porosity was also seen as a function of increasing suspension concentration. The volume shrinkage measurements indicate that the higher shrinkage may occur at high starch concentrations and low suspension concentrations due to insufficient free water content in suspension, which is required for full swelling of starch granules. As the starch granules enlarge, the ceramic particles between them become less tightly packed. The SEM findings suggest that when starch content increases, the pore size decreases and the form irregularity increases.

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