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Effect Sintering Temperature On Silica Production Using Rice Husk from Pasir Tumboh, Kelantan

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Abstract: This paper presents were to study the physical properties of the samples ceramic water filter in different sintering temperature that produce high silica content which in crystalline to reduce water pollution and agricultural waste. Rice husk ash from Pasir Tumboh, Kelantan divided by composition which 45wt%, 50wt% and 55wt%, and each of the ash were mixture with Polyethylene Glycol (PEG) by 1 wt%. The mixture material was compacted by hydraulic press machine and going through with thermal treatment to produce the filter samples. The filter samples were sintered at temperatures of 900°C, 950°C, 1000°C, 1050°C and 100°C to observe the effect of sintering temperature on the ceramic filter samples. Density and porosity test plus water absorption test was performed to evaluate the physical properties of the samples while Scanning Electron Microscopy (SEM) were used to analyze the microstructure of the filter samples. The results showed that the filter composition 45wt% has lowest percentage of water absorption and apparent porosity plus highest bulk density.

Keywords: Rice husk ash, Density, Water absorption, Physical properties, Compaction, Crystalline silica.

1. Introduction

Rice husk is an agro waste material that is abundant in all rice producing countries and it has high and convenient production found all over the world. Current world rice production was estimated at around 700 million tons and 100 million tons per year which accounts for 20% of total world rice production [1]. Most of the rice husk was usually disposed and burned in open areas which this will cause soil degradation and air pollution [2].

Rice husk contains about 70-90% organic matter such as 50% cellulose and 25-30% lignin and 15-20% silica [12 full report]. According to other sources, the rice husk ash burned at moderate temperatures consists of 80%-97% amorphous silica [4]. The silica content of rice husk ash can also be

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commercialised for use in water filters because it contains an active carbon source, cheap, contains no dangerous materials and has been tested as an adsorbent material [5].

Silica forms quartz, crystals, silica gels, and aerogels. In addition, silica has a microstructure with high porosity, low density, and stable heat resistance [6]. Silica phase includes amorphous, crystalline, and zeolite silica. When rice husk was burned to ash, the silica phase becomes amorphous silica. For the crystal phase, rice husk ash must be burned at above 800°C, while the zeolite phase required above 1500°C [4]. Crystalline silica has hard properties, high melting point and chemically inert. The characterization of crystalline silica can be studied using X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM), Fourier-Transform Infrared Spectroscopy (FTIR) and X-Ray Fluorescent (XRF) [7]. The crystalline silica percentage was increased by subjecting the rice husk ash to a higher temperature with more period time or by maintaining the temperature and increasing the time.

The objectives of this study are to determine the effect of sintering rice husk ash and the physical properties of the ceramic filter samples at different sintering temperatures that produce high silica content. The presence of crystalline silica in RHA ceramics filter samples sintered at temperature range 900-11100°C were tested by density, porosity and water absorption test. The microstructure of this crystalline silica is also observed.

2. Materials and Methods

2.1 Raw Materials

Rice husk ash from Pasir Tumboh, Kelantan was used to make samples. It was divided into three filter compositions of rice husk ash burned at 45wt%, 50wt% and 55wt%.

2.2 Methods

There are several processes to produce samples from burned rice husk ash. The process begins with milling, sieving, compaction, and sintering process. Lastly, physical tests and microstructural analysis were performed on the samples to determine their physical properties and microstructure.

2.2.1 Milling process

Rice husk ash was milled to a micron-size in a Planetary Mono Mill Pulverisette 6. The milling process must start with dry ash. The High Carrot Oven dried ash at 90°C to 114°C before starting the mill. After the ash was dried, it was milled to get in powdery state for 30 minutes at 200 rpm. After the milling process is finished, the container would be cleaned using silica sand and dishwashing liquid if there were still impurities.

2.2.2 Sieving process

Analysette 3 creates uniform powdery ash. The powdery ash was filtered 250, 200, 180, and 160 microns. The powdery ash went into the machine's sieve and the container was closed. The conveyor strap helps to keep the sieve container in place during sieving. The sieving machine was set up in 15 minutes and the concussion rate of the machine is 0.7 amplitude. The conveyor belt was opened, and the sieve container was separated to get 160-micron ash.

2.2.3 Compaction process

Compaction process was done by using hydraulic press machine (Carver model 38510). The shape and size of the sample refer to the percentage of set composition silica and mixed with 1 wt% PEG. The mold set was pre-washed with lubricating oil and mixed powder were added into mold set. The mold set compacted with a load of 3 tons in 2 minutes.

2.2.4 Sintering process

The sintering process was performed by heating a furnace (Protherm Furnaces model PLF 140/5) with different temperatures, holding time and heating rate. The samples were sintered between 900°C and 1100°C. The samples are placed in the furnace and the panel instrument would be set to the sintering temperature. After sintering, samples are removed and cooled to room temperature.

2.2.5 Microstructural analysis.

Rice husk ash samples were characterized using Scanning Electron Microscope (SEM) brand *JSM-6370 LA model* from Germany. It was used for identifying the microstructure of the water filter sample produced from the burn rice husk ash after going through the sintering process with different composition of silica and temperatures.

2.2.6 Density, porosity and water absorption test.

Standard ASTM C20-00 measured sample density and porosity water absorption. The sample's dry weight (D) was measured to the closest 0.1 g. The sample was boiled in water for 2 hours and kept the samples submerged away from the beaker's heated bottom. The samples were submerged in distilled water for 12 hours. Submerged samples will be weighed to determine suspended weight (S). The samples were taken from distilled water and absorbed by filter paper. The samples were reweighed to determine saturated weight (W) in grams. The data were calculated by using the below equations to get the Exterior volume (V), Apparent porosity (P), Bulk density (B) and Water absorption (A) data.

$$\text{Exterior Volume, } V = W - S \quad \text{Eq. 1}$$

$$\text{Apparent Porosity, } P = [(W - D)/V] \times 100 \quad \text{Eq. 2}$$

$$\text{Bulk density, } B = D/V \quad \text{Eq. 3}$$

$$\text{Water absorption, } A = [(W - D)/D] \times 100 \quad \text{Eq. 4}$$

3. Results and Discussion

3.1 Discoloration of the sample after sintering process

In this experiment, samples were sintered by 45wt% composition of silica. Sample A-E represent 45wt% silica sintered at 900°C, 950°C, 1000°C, 1050°C, 1100°C. Figure 1 shows sintering samples by temperature.

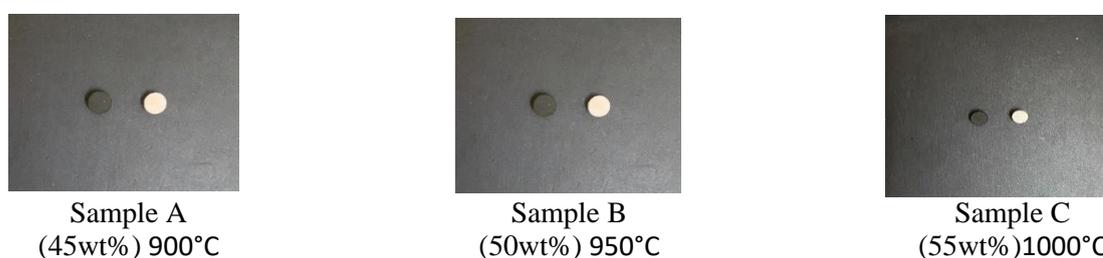


Figure 1: Sintering samples by temperature

Figure 1 shows that sample A (45%) changed from black to brownish white after sintering at 900°C, while sample B (45wt%) changed from black to brownish white but was brighter. Sample C (45wt%) sintered at 1000°C changed from black to white and less greyish. Next, samples sintered at 1050°C, sample D (45wt%) changed from black to white but were brighter than those sintered at 1000°C. Lastly, 1100°C sintered sample E (45wt%). At 1000°C, 1050°C, and 1100°C, the samples were completely sintered and contained a lower percentage of carbon, while at 900°C and 950°C, they were not completely sintered and contained a higher percentage of carbon. A high-quality sample required a high sintering temperature and long sintering times. The study found that as the temperature increases, the black samples turned white. This evidence shows that as the samples sintered at higher temperatures, their colour brightened because the carbon content decreased.

3.2 Density, porosity and water absorption test

Rice husk ash silica composition ranged from 45wt%, and sintering temperatures were 900°C until 1100°C were test. Table 1 shows the bulk density, apparent porosity and water absorption of each sample, followed by 45wt% silica composition and different temperature. The example for calculation result of the average value for bulk density, percentage of apparent porosity and water absorption show in Appendix A.

Table 1: Calculation result of the average value for bulk density, apparent porosity and water absorption

Composition of silica (%)	Sample	Sintering temperature (°C)	Bulk density, B (g/cm ³)	Apparent porosity, P (%)	Water absorption, A (%)
45	A	900	0.75116	66.96102	89.1437
	B	950	0.7625	64.9364	85.1641
	C	1000	0.8275	61.8506	74.7488

Based on the data on Table 3.1, samples composition of silica 45wt% was selected in this research due to its smooth data compared to the other sample’s silica composition. Figure 2 shows the graph for the bulk density and apparent porosity against the sintering temperature for 45% composition of silica.

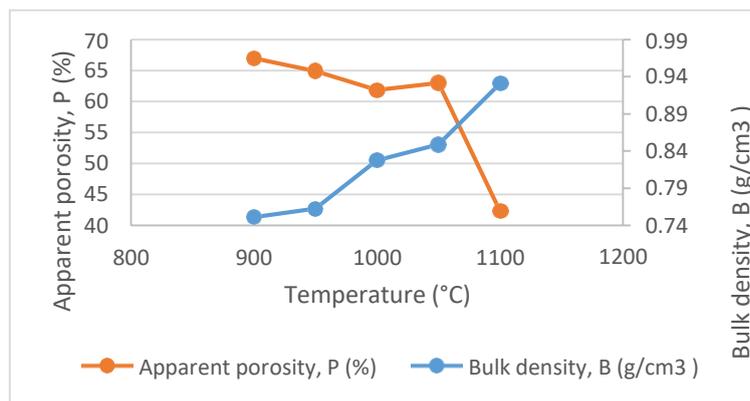


Figure 2: Graph for the bulk density and apparent porosity against the sintering temperature by 45%

Table 1 and Figure 2 show that the average bulk density of samples by 45wt% silica increases with sintering temperature. Sample A bulk density was 0.7512 g/cm³, while sample E was 0.9309 g/cm³. The graph also shows bulk density increase with the rising sintering temperature. Sample A has

the largest apparent porosity 66.96% while sample E has the lowest 42.36%. Figure 3 shows the graph for water absorption and bulk density results against the sintering temperature. Meanwhile, Figure 4 displays the graph for water absorption and apparent porosity results against the sintering temperature for 45% composition of silica.

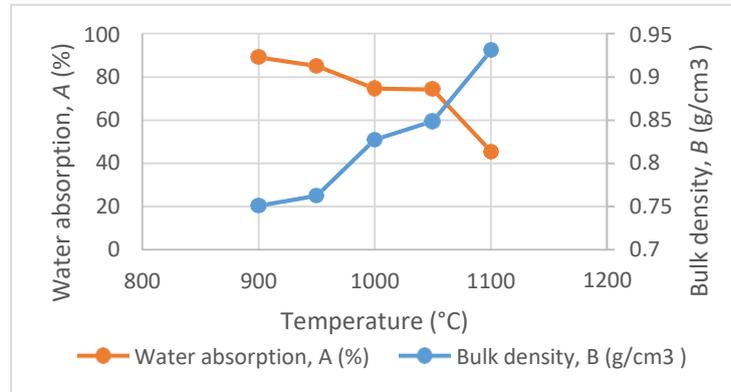


Figure 3: Graph for water absorption and bulk density results against the sintering temperature by 45%

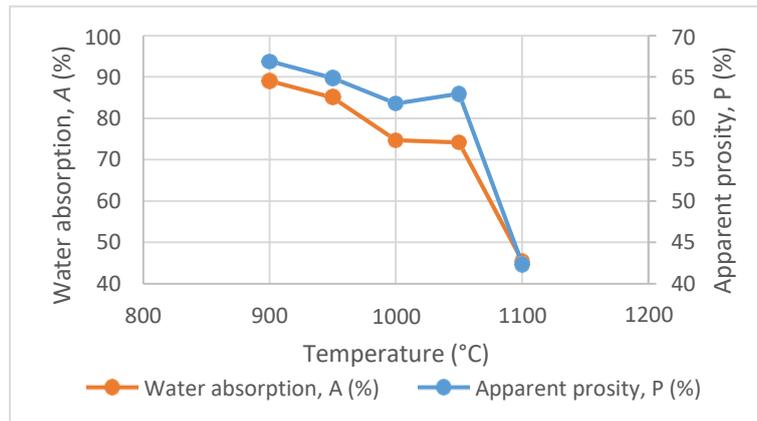


Figure 4: Graph for water absorption and apparent porosity results against the sintering temperature by 45%

Based on Figure 3, the graph shows percentage water absorption for each sample will decrease when the sintering temperature increases. The highest value water absorption percent was sample A which 89.14% and the lowest value water absorption percent were sample E which 45.51% and it can be concluded that the water absorption decreased, and the bulk density will be increase when the sintering temperature of the sample increase. Besides, refer to Figure 4, it can be measured from the graph that the percentage of water absorption will decrease as apparent porosity decreased when the sintering temperature of the samples increase.

Based on the results, the apparent porosity will decrease, and the bulk density will increase as the sintering temperature rises. The sample will shrink with increased sintering temperature which affecting its density. Due to sample shrinking, the density increases and the volume decreases.

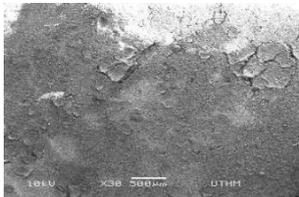
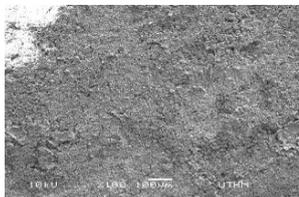
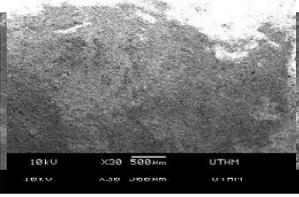
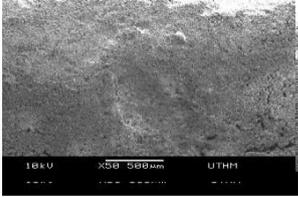
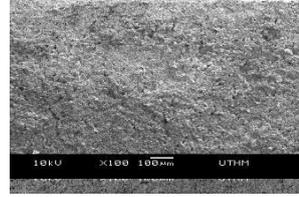
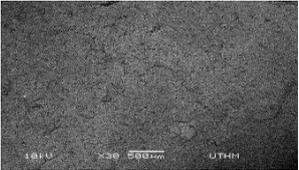
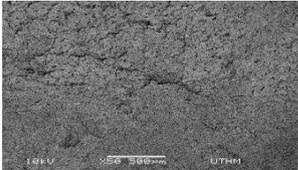
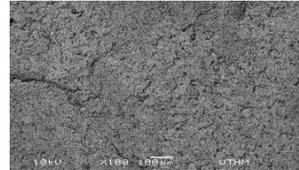
Other than that, based on Figure 4, water absorption for each sample decreases steadily. This finding was also found by J.P. Nayak & J. Bera (2009), who employed rice husk ash as their silica source [8]. Based on their findings, water absorption decreases, and bulk density increases as sintering temperature increases. This result shows sample E was the best sample since it has the low water absorption percentage and high bulk density. High bulk density values and low percentage of porosity provide mechanical strength on water sample [9] and the percentage of water absorption depended on the sample pores, and because the pores were small, only a small amount of water could be absorbed.

This is quite good for ceramic water filter because its pores are used to keep pollutants and foreign stuff out [10].

3.3 Microstructural Analysis

Scanning Electron Microscopy (SEM) machine used is the brand JSM-6370 LA model from Germany. Microstructural Analysis was used to determine the morphology and distribution of sintered samples at 900°C, 950°C, 1000°C, 1050°C, and 1100°C by 45wt% silica composition. In this research, 45wt% of samples were chosen for optimum experiment data. The microstructure that has been measured is the size of the porosity and the surface area of the samples. Table 2 illustrates micrographs of sintered samples by 45wt% silica composition at 30x, 50x, and 100x magnifications.

Table 2: Micrograph for each sample by 45wt% composition of silica in different temperature.

Sample	Magnification of micrograph 45		
	30x	50x	100x
A			
B			
C			

Samples A and B in Table 2 had large pores and particle sizes and it shows particle production, but not in high density. Sample B has smaller pores and larger particles than sample A. Other than that, the particle of sample C occurs and has a higher density than sample A and B. Other than that, sample C, sintered at 1000°C, has bigger particles and higher density than samples A and B.

Microstructure analysis shows sintering temperature affects sample microstructure. When the sintering temperature increases, the sample's pores narrow, and its particle size increases due to particle bonding. Other than that, the density value also increases when sintering temperature increases. Table 3.2 shows that sample A has the greatest pores and smallest particles, while sample E has the smallest pores and largest particles in the 45wt% silica samples. Microstructure analysis study involved density, porosity, and water absorption tests. Sample E that has been sintered at 1100°C has high value density and low percentage of porosity. It shows that sample E has the smallest pores, due to the low porosity percentage and high-density value because the particles bond together to form a big particle. This narrow pore affects the percentage of water absorption because it only allows a little

amount of water to be absorbed into the sample. The relation of microstructure analysis between density and porosity test has been explored by J.P. Nayak & J. Bera (2010) [9].

Conclusion

In the nutshell, this experiment shows sintering temperature influences water filter sample qualities. When the sintering temperature rises, the water filter sample will turn bright. As sintering temperature increases, apparent porosity will increase and bulk density increase. Increasing sintering temperature causes shrinkage, which affects sample density. Sample shrinkage increases density and decreases volume. Other than that microstructural research shows that when sintering temperature rises, pores will shrink, and particles increase. Besides, the sample E porosity of 42.36 % meets the requirement for a ceramic water filter. Previous research found 30%–44% porosity in ceramic filters. Sample E has the lowest porosity in that range. Less porous ceramic filters can eliminate bacteria more efficiently.

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Appendix A

Example of Calculating Bulk Density, Percentage of Apparent Porosity and Water Absorption.

Sample E was selected as an example with the stated weight;

$$\begin{aligned} D &= 0.6219 \\ S &= 0.2369 \\ W &= 0.9049 \end{aligned}$$

$$\begin{aligned} \text{Exterior Volume, } V &= W - S \\ &= 0.9049 - 0.2369 \\ &= 0.6681 \end{aligned}$$

$$\begin{aligned} \text{Bulk Density, } B &= D / V \\ &= 0.6219 / 0.6681 \\ &= 0.9309 \end{aligned}$$

$$\begin{aligned} \text{Apparent Porosity, } P &= [(W - D) / V] \times 100 \\ &= [(0.9049 - 0.6219) / 0.6681] \times 100 \\ &= 42.36\% \end{aligned}$$

$$\begin{aligned} \text{Water Absorption, } A &= [(W - D) / D] \times 100 \\ &= [(0.9049 - 0.6219) / 0.6219] \times 100 \\ &= 45.51\% \end{aligned}$$

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