

The Effect of Rice Husk Charcoal and Sintering Temperature on Porosity of Sintered Mixture of Clay and Zeolite

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Abstract

Objectives: Sintered porous clay for possible as a water filter was fabricated from clay and zeolite mixtures (50:50 ratio weight by weight) with adding of rice husk charcoal as porous template. **Methods/Statistical Analysis:** The effect of particle size (<88 μm , 88-250 μm , and >250 μm) and amount (25, 35, and 50 wt%) of rice husk charcoal and sintering temperature (800, 900, and 1000°C) for linear shrinkage, density, water absorption, compressive strength, and functional groups on sintered mixture clays were investigated. The FTIR and SEM were also used for characterization of sintered porous clays. **Findings:** The results showed that the water absorption and the open porosity increased with increasing amount and size of rice husk charcoal, while the density and compressive strength decreased. However, these properties are inverse as increasing of sintering temperature from 800 to 1000°C. The results of FTIR and SEM confirmed occurring of glassy form, high open porosity and macropore in sintered mixture clays. The study results could be concluded that the mixtures of rice husk charcoal and clays with ratios 25 wt% rice husk charcoal for <88 - >250 μm size : 37.5 wt% Thapo clay : 37.5 wt% zeolite at 800-1000 °C sintering temperature are suitable for water filter fabrication with values of 30.44-42.80 %porosity and 3349.94-4629.66 kN/m² of compressive strength. **Application/Improvements:** Rice husk charcoal was used as template for water filter fabrication at 800-1000°C sintering temperature which values of porosity and compressive strength are according to traditional water filter manufacture standards..

Keywords: Porous Ceramic, Rice Husk Charcoal, Thapo Clay, Water Filter, Zeolite

1. Introduction

Porous ceramics endowed with properties of high temperature resistance, high thermal shock resistance, high specific strength, low thermal mass, low thermal conductivity etc.¹ are interesting materials for a wide range of industrial applications². They are widely used for differ-

ent purposes such as thermal insulation, catalyst support, filtration, heat exchangers, high efficiency combustion burners, bio-medical implants etc¹. They are usually prepared from the mixture of clay and pore-forming agents. During the heating process, the mixture loses its water and other volatile compounds, which leads to the cre-

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ation of pores³. For instance, porous ceramics with three different porosities have been fabricated by the sintering of redart clay and sawdust as the pore-forming agent in porous ceramic⁴. They are also produced from preceramic polymers, which offer advantages in terms of simple processing methodology, low processing cost, and ease of control over porosity and other properties arising from the composition of the resultant ceramics⁵. Among these, mesoporous systems of zeolites have been developed with the carbon particles template⁶, sacrificial template⁷, and an organic foam template⁸. For this research, the zeolite and clay are used for porous ceramics fabrication with rice husk as the pore-forming agent. Zeolites are defined as a diverse group of inorganic, alkaline, and hydrated crystalline aluminum-silicates, where tetrahedral units are connected to neighboring corners by sharing their oxygen atoms, thus forming a regular spatial arrangement of uniform cages, cavities or channels of molecular dimensions with unique hydrophilic properties, high ion exchange capacity, molecular sieving, large porous surface area, flexible shape selectivity, intrinsic charge-balancing cations, and electrochemical behavior promoting strong adsorption affinity within their encapsulated network⁹. These materials are widely used as sorbents, catalysts, and ion-exchange materials⁶. The pore-forming agent, rice husk, is an agricultural waste, which is abundantly available in rice producing countries such as China, India, Bangladesh, Brazil, US, Cambodia, Vietnam, Myanmar, and South East Asia. Its major components are carbon and silica. Roughly 150 million tons/year of rice husk is generated in the world from rice production¹⁰. The use of rice husk in the ceramic industry is favorable due to the economical, energy, and environmental advantages¹¹. The main advantages of rice husk, in comparison with other agricultural residues, are its high silica content, high porosity, lightweight, and very high external surface area, which make it a valuable material for industrial applications¹². Rice husk has been used for the production of materials with enhanced mechanical resistance¹³. It was

also utilized in the production of fired bricks making them into an insulating material thereby saving energy. Furthermore, its use can reduce energy consumption in the firing process and even generate a large amount of energy by its combustion. Thus it also results in considerable fuel savings¹⁴.

In this research, the porous ceramics were produced from a mixture of clay, and rice husk charcoal additive used as a pore forming agent. The purpose of these materials is their use for water filtering. The effects of particle size, amount of rice charcoal, and sintering temperature on the properties of the sintered clay mixtures were investigated. The physical and mechanical properties such as linear shrinkage, density, water absorption, porosity, and compressive strength, as well as the presence of functional groups and morphologies on sintered clay mixtures were investigated for evaluation of suitable conditions for water filter fabrication.

2. Materials and Methods

The rice husk charcoal, which was prepared by carbonization of rice husk at 500°C, was mashed and sieved (Laboratory test sieve, Retsch, Germany) to <88 µm, 88-250 µm, and >250 µm. Local clay (obtained from Tambol Thapo, Muang District, Phitsanulok Province, Thailand) and zeolite (commercial grad) were also mashed and sieved to >250 µm. All starting materials were mixed in weight ratios of rice husk charcoal: local clay: zeolite of 25:37.5:37.5, 35:32.5:32.5, and 50:25:25. Separate mixtures were prepared for the charcoals with different particle sizes. All mixtures were wetted with water (20% v/v) and then pressed into a PVC pipe ($\phi = 12.7$ mm, length = 50 mm). The specimens were oven dried (SL shellab, 1350 FX, USA) at 110°C for 3 h. After that, the specimens were sintered at 800°C, 900°C, or 1000°C in an electric furnace (Fisher Scientific Isotemp ® Muffle furnace) under an oxidizing atmosphere. The physical and chemical properties of sintered products such as linear shrinkage, density, % water absorption (following the procedure of ³), open

porosity (following the procedure of ¹⁵), and compressive strength (by Compression Machine, Shimadzu Autograph, model AG-25TB) were investigated. The FTIR (Spectrum GX, Perkin Elmer) and SEM (PHILIPS LEO 1455 VP) instruments were also used for the characterization of functional groups and surface morphology of the sintered products, respectively.

2.1 Sample Characterization

2.1.1 Measurement of the Percent of Linear Shrinkage

Linear shrinkage was determined by measuring the length of the specimens over the whole process. It is expressed as a percentage and calculated according to the following formula³.

$$\text{Linear shrinkage (\%)} = [(L_{\text{initial}} - L_{\text{fired}}) / L_{\text{dried}}] \times 100 \quad (1)$$

where, L_{initial} is the length of the wetted specimens (mm) and L_{fired} is the length of the sintered specimens (mm).

2.1.2 Measurement of Bulk Density

Bulk density of each specimen was measured by calculating the ratio of the dry mass to volume of the sintered specimen³.

2.1.3 Water Absorption Measurement

When pore volume of the pores and capillaries increases, relative wall thickness of the pores and capillaries decreases. Reserve pore volume represents part of the volume that initially is not filled with water. It is filled gradually, while samples are soaked in water. This reserve is determined by the existence or absence of the closed defective areas in the ceramic body, as well as by the dimensions of the pores and capillaries¹⁶. The amount of water absorption is depend on the arrangement of the pores and the way they are linked together, this can penetrate the material more or less easily, with a preferential pathway¹¹. In this measurement, water absorption

of sintered clay mixtures was determined by the procedure of³. The specimens were dried at 110 °C for 24 h and weighed to constant mass. They were then cooled for 24 h and totally immersed in water. After soaking for 24 h, the specimens were weighed, dried and reweighed to constant mass. The water absorption (A) expresses the relationship of the weight of water absorbed to the weight of the dry specimen.

$$A(\%) = [(W - D) / D] \times 100 \quad (2)$$

where, D is the dry weight and W is the saturated weight with water.

2.1.4 Measurement of the Percent of Open Porosity

Porosity is the measure of interconnected voids in the ceramic material¹⁷. The percent of open porosity of sintered clay mixtures was measured following the procedure of ¹⁸. The sintered clay mixtures were weighted in air and then immersed in boiled distilled water. All weightings were conducted on an analytical balance (Sartorius, Germany). The densities of the specimens and immersed specimens were calculated. Porosity was calculated using the following equation:

$$\text{Open porosity (\%)} = [1 - (\rho_s / \rho_o)] \times 100 \quad (3)$$

where, ρ_s is the measured specimen density and ρ_o is the theoretical density of a specimen having no porosity.

2.1.5 Compressive Strength Measurements

The compressive strength of the sintered clay mixtures was determined by a compression machine (Shimadzu Autograph, model AG-25TB). The compressive strengths of the sintered clay mixtures were obtained by compressive loading (1 kN) of cylindrical rod with height = 50 mm and diameter = 12.7 mm. The tensile tester works by gradually increasing the pressure on the sintered clay mixtures. The press is simulated by the string principle. String has one end created by the tip, which is pressing on

the sintered clay mixtures. The pressure is increased until the sintered clay mixtures become crashed.

3. Results and Discussion

3.1 Effect of Sintering Temperature on Clay Mixtures without Rice Husk Charcoal

For clay mixtures without rice husk charcoal, the values of linear shrinkage (7.69-14.00%), density (1.40-2.09 g/cm³), and compressive strength (5199.53-12998.83 kN/m²) of sintered clay mixtures increase with increasing sintering temperature from 800 to 1000°C, except for values of open porosity (16.07-14.41%) and water absorption (10.48-9.38%), which show an inverse trend (Table 1). This is because particles of clay mixtures fuse together leading to enhanced linear shrinkage during sintering at high temperatures³. Furthermore, a reduction of rate of linear shrinkage of sintered products was observed between 900°C to 1000°C in comparison to the rate from 800°C to 900°C. This result is attributed to the development of crystalline phases with an excess of glassy phase and an increase of closed porosity at 900°C and higher sintering temperature, which cause a lower increase of linear shrinkage for between temperatures 900°C and 1000°C¹⁹. The density increase is a direct result of the increase of sintering temperature as this enhances the densification mechanisms²⁰. Porosity is reduced due to shrinking of the products during the sintering process. Furthermore, this is also the result of liquid phase or vitrification process, which tends to fill the pores²⁰, or fusion of clay mixture particles³. These phenomena result in the decrease of the water absorption and open porosity of the sintered clay mixtures. On the other hand, these processes also affect the compressive strength, which increased with increasing of sintering temperature. This result could be explained by densification and denser packing of clay mixture particles, which occurs after sintering and helps to increase the effective load bearing area hence increasing the compressive strength²¹ of the sintered products. It was also observed that the sintered products are of reddish color (Figure 1 A₁-A₃). This is explained

by the oxidation of Fe²⁺ present in raw clays to Fe³⁺²⁰ in the sintered products during the sintering process. It can be seen that the sintered clay mixtures without rice husk charcoal do not possess the properties required for water filter fabrication, except for compressive strength. For general a traditional water filter manufacture in different countries, the range of porosity is between 35% and 44%¹⁷, and a compressive strength should not be below 700±10 kN/m²²².

3.2 Effects of Size and Amount of Rice Husk Charcoal with Sintering Temperature

The addition of rice husk charcoal at different loading ratios and with different particle sizes has a significant effect on the properties of the fabricated materials. The values of percent water absorption and porosity are higher for all sintered clay mixtures containing rice husk charcoal in comparison to pure clay mixtures. On the other hand, density and compressive strengths show an inverse trend (Table 1). This is because the addition of rice husk charcoal into the clay mixtures creates open porosity in the system during the drying and sintering processes by dehydroxylation and combustion. The removal of rice husk charcoal portions leaves holes inside the materials and these holes are the sources of voids that move from the center to the outer surface during sintering process²¹. This formation of voids leads to the observed decrease in the densities, which likely affects the compressive strength of the sintered products through weakening of the structure³. It was seen that the sintered products containing rice husk charcoal have reddish color as well as a grey tone (Figure 1 BI₁-DIII₃). The grey color content increased with increasing content of rice husk charcoal. This shows that some Fe₃O₄, which is related to magnetite²⁰, is also formed by reduction during combustion of carbon in rice husk charcoal.

The effects of amount and size of rice husk charcoal in sintered clay mixtures formed between 800 and 1000°C showed increased water absorption and open porosity with increasing amount and size of rice husk

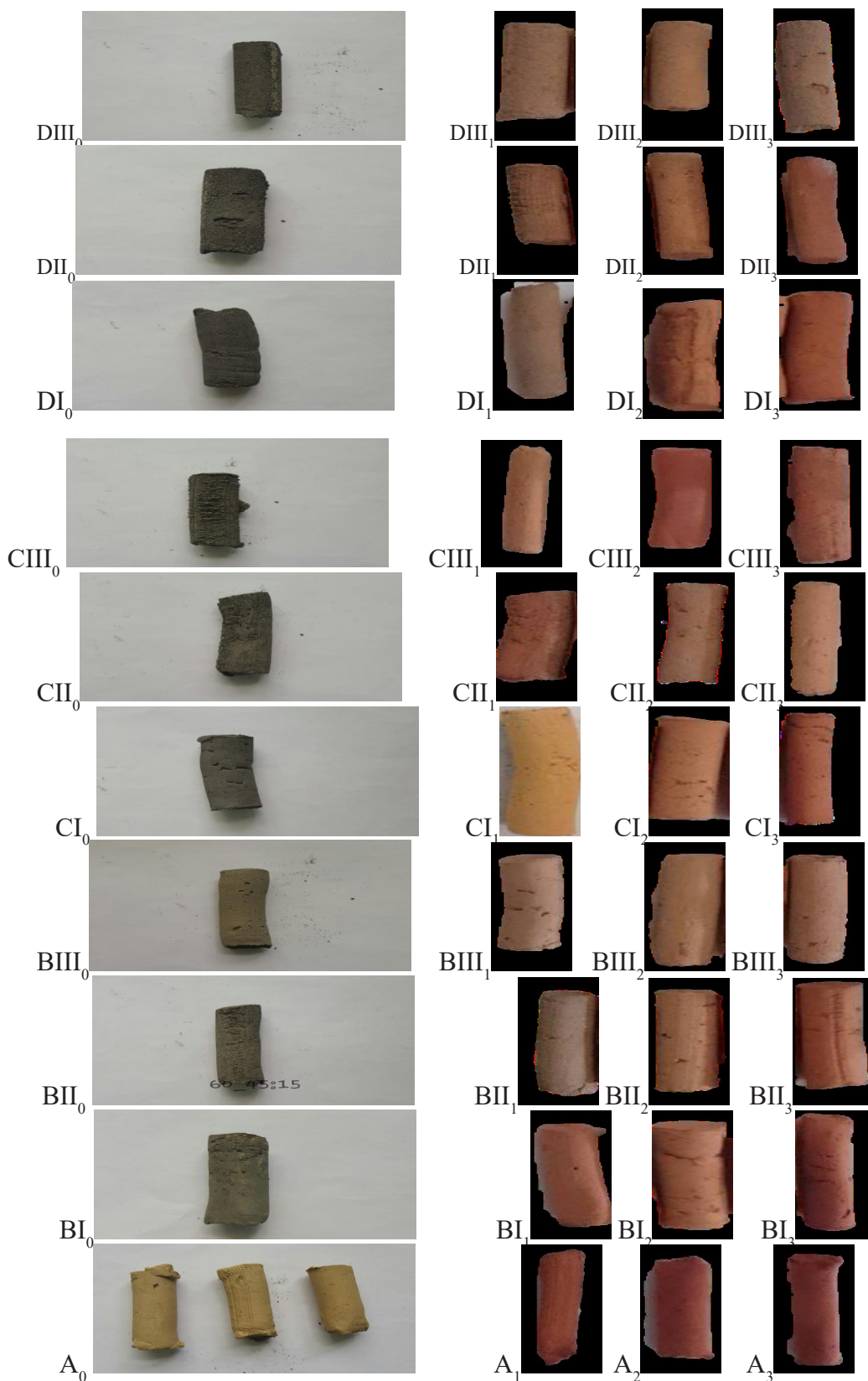


Figure 2. Specimens of rice husk charcoal: Thapo clay:zeolite with ratios of; A= 0:50:50, B=25:37.5:37.5, C=35:32.5:32.5, D=50:25:25; particle size of rice husk charcoal: I= <88 μm , II= 88-250 μm , III= >250 μm ; and sintering temperature; subscript number: 0= no firing, 1= 800 °C, 2= 900 °C, 3 = 1000 °C.

charcoal, while the density decreases. In terms of shrinkage, which occurs as the rice husk charcoal is burned out, leaving behind only the clay particles. These were initially packed loosely but then approached each other and densified at high temperatures²¹. Thus, the compressive strength is also reduced with increasing amount of rice husk charcoal. This result could be explained by the fact that the increase of porosity, achieved with rice husk charcoal addition, is considered as a source of mechanical strength defects though impeding the mechanisms of densification and pore-filling processes²⁰. The compressive strength increases with decreasing particle size of the rice husk charcoal for the same sintering temperature. This observation could be rationalized based on the findings of ²¹. Essentially, small particles lead to a higher amount of solid content in the pores of the clay mixture. Furthermore, after sintering the rice husk charcoal, containing significant portion of silica, is burned out and the clay particles are fused with the silica fused to obtain crystalline phases¹⁹, which leads to the shrinkage of the specimens. In addition, as the particle size of the rice husk charcoal increases, for fixed ratios of rice husk charcoal, the open porosity of the specimens' increases. This is due to two reasons. Firstly, specimens prepared from rice husk charcoal with bigger particle size have a lower density when compared with those prepared from rice husk charcoal with smaller particle size. Secondly, large pores created during burning out of the larger rice husk charcoal particles undergo less shrinkage in comparison to materials prepared from charcoal with smaller particle sizes. These effect lead to overall increase in the porosity of the sintered products¹.

The effects of the adding 25% rice husk charcoal to the clay mixtures can be glimpsed from Table 1 where the values of linear shrinkage, density, water absorption, porosity, and strength are reported in combination with the effects of particle size and sintering temperature. As far as the particle size effects at same sintering temperature are concerned, the linear shrinkage (9.40% to 8.08%), density (1.10 g/cm³ to 0.97 g/cm³), and strength (4512.66 kN/m² to 3349.94 kN/m²) decreased with increasing particle size (<88 μ m to >250 μ m) for sintering at 800°C. On the other hand, percent water absorption (39.72% to 42.80%) and

porosity (39.90% to 42.80%) increased. Similar trends for the effects of particle size are observed at both 900 and 1000°C. Inversely, for fixed particle size of rice husk charcoal, the linear shrinkage (9.40% to 14.22%, density (1.10 g/cm³ to 1.43 g/cm³), and strength (4512.66 kN/m² to 4629.66 kN/m²) increased with increasing sintering temperature (800°C to 1000°C) for charcoal with <88 μ m particle size. The percent water absorption (39.72% to 27.27%) and porosity (39.90% to 30.44%) decreased in the same series. The results for charcoals with particle sizes 88-250 μ m and >250 μ m showed similar trends in their properties. The same trends for these parameters have been observed for starting mixtures containing 35 wt% and 50 wt% rice husk charcoal. Furthermore, it was seen that the compressive strength of sintered specimens obtained from mixtures containing 50 wt% of rice husk charcoal added to clay mixtures is sharply lower in comparison to those made with 25 wt% and 35 wt% rice husk charcoal addition. This may be attributed to the bubble formation during sintering process² of mixtures with excessive content of rice husk charcoal. The compressive strength normally decreased rapidly above a critical pore content (50 vol%)¹. Finally, the optimum mixture of rice husk charcoal and clays for water filter production purpose is made from 25 wt% rice husk charcoal (<88 - >250 μ m particle size) : 37.5 wt% Thapo clay : 37.5 wt% zeolite and sintered at 800-1000°C. This is because of the values of porosity and compressive strength for sintered specimens prepared in this manner are complying with traditional water filter manufacture standards.

3.3 FTIR Analysis

FTIR spectra of Thapo clay (Figure 2a) and zeolite (Figure 2b) are very similar. They exhibit hydroxyl stretching of AlO-H groups (very weak peaks at 3693-3696 cm⁻¹)²³, as well as the bending bands of Al-OH at 910 cm⁻¹ and 690-700 cm⁻¹ ²⁴ Furthermore, signals for bridging acidic hydroxyls Si-O(H)-Al (small weak peaks at 3610-3625 cm⁻¹)²³ are observed in these samples as well. The broad band at about 3400 cm⁻¹ is due to extra bridging by hydroxyl ion or due to moisture incorporated in the porous structure of Thapo clay and zeolite. This feature is also related to a weak O-H deformation peak at 1634 cm⁻¹

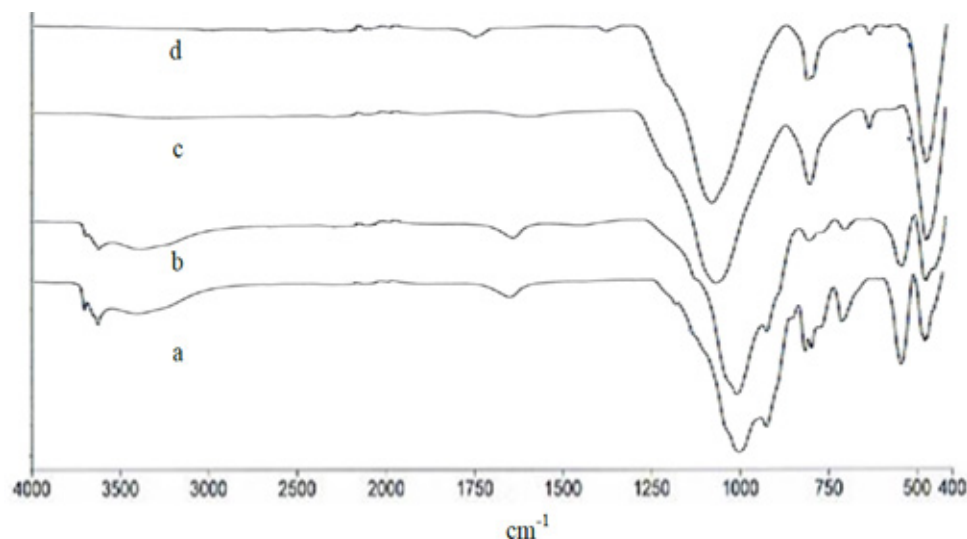


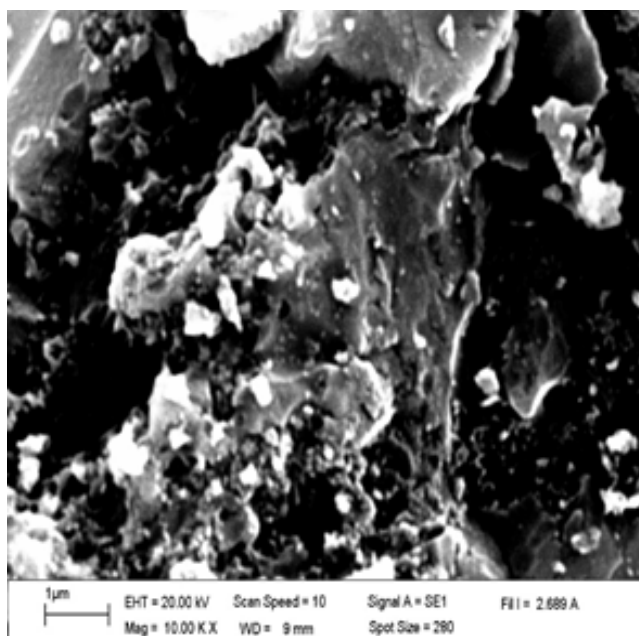
Figure 2. FTIR transmission of Thapo clay (a), zeolite (b), rice husk charcoal (c) and sintered product made from a 32.5:32.5:35 mixture of clay:zeolite : >250 μm rice husk charcoal at 1000 $^{\circ}\text{C}$ (d).

²⁵ these bands are attributed to vibrations stemming from OH in the di-octahedral layer²⁶. The strong peak found in Thapo clay and zeolite at about 1000 cm^{-1} is assigned to bending of Al-O²⁷ and Si-O-Si stretching vibrations²⁰. The weak peak at about 780 cm^{-1} is assigned to Si-O stretching²⁷. The two weak peaks at about 525 cm^{-1} and 460 cm^{-1} are assigned to bending of Si-O-Al²⁴ and Si-O-Si deformation²⁵, respectively. These functional groups are found in mineral silicate of clay and zeolite.

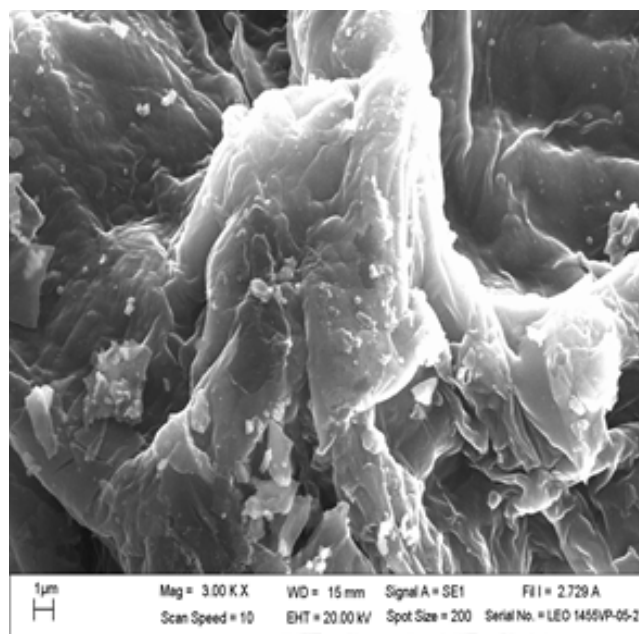
The FTIR spectrum of rice husk charcoal (Figure 2c) contains a dominant broad band with maximum at 1062.73 cm^{-1} , which could be assigned to the stretching vibrations of the C-O bonds of esters, phenols, alcohols or ethers²⁸ after carbonization. This peak is also attributed to the inter-tetrahedral vibration modes of SiO_4 tetrahedra, which are associated with the intra-tetrahedral vibration modes of the SiO_4 tetrahedra at 619.17 cm^{-1} ²⁹. The peaks at 761.09 cm^{-1} and 462.50 cm^{-1} could be assigned to stretching and bending of Si-O-Si in amorphous silicates³⁰, respectively.

The FTIR spectrum of a representative sintered clay mixture (Figure 2d) exhibits showed very weak peaks at 1739.5 and 1365 cm^{-1} , a strong peak at 1067.34 cm^{-1} , a

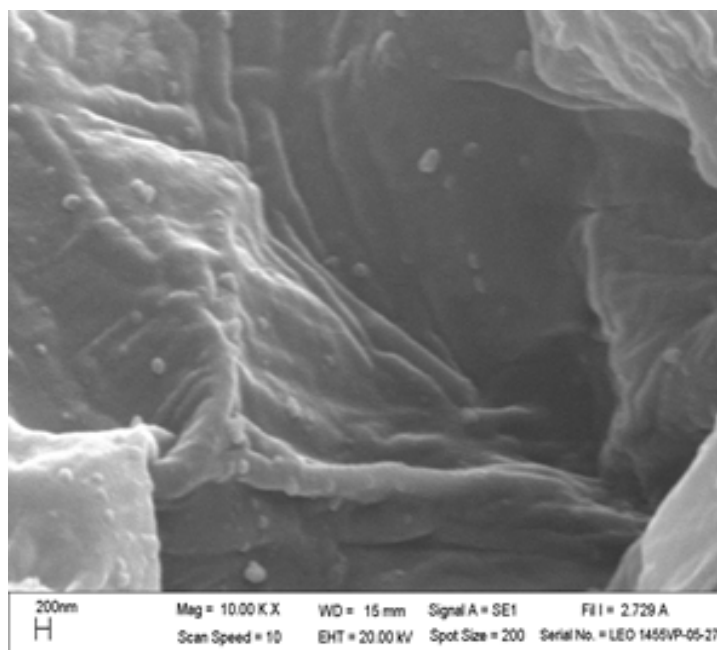
weak peak at 795.18 cm^{-1} , a very weak peak at 619.32 cm^{-1} , and strong peak 459.24 cm^{-1} . The peaks of -OH group, which is bound to Al and Si atoms, at 3693-3696 cm^{-1} , 910 cm^{-1} and 690-700, and 3610-3625 cm^{-1} observed in samples of local clay and zeolite have disappeared after sintering of the clay mixtures. This shows that the OH group in the tetrahedral sheet of starting clay mixtures is thermally degraded²⁶ after sintering. The peaks of the inter-tetrahedral and intra-tetrahedral vibrational modes of the SiO_4 tetrahedra, bending of Si-O-Si, bending of Al-O, stretching of Si-O, bending of Si-O-Al and Si-O-Si deformation found in the starting materials are still present in the sintered clay mixture. This is because these functional groups are thermally stable. However, the strong peak at about 1000 cm^{-1} found in the starting clays is shifted to 1067.34 cm^{-1} after sintering. This could be attributed to the formation of a silica network during sintering³¹. Two new peaks with very weak intensity are found in the sample of sintered clay mixture at 1739.05 and 1365.06 cm^{-1} and are attributed to harmonics of Si-O and Si-O-Si bridge vibrations³². These new peaks originate from silicates of the starting clay mixtures, which melt to a glassy form³³ after sintering at 1000 $^{\circ}\text{C}$. The sili-



(a)



(b)



(c)

Figure 3. SEM images of 25 wt% rice husk charcoal : 37.5 wt% Thapo clay : 37.5 wt% zeolite sintered specimens after sintering at temperatures of 800 °C (a), 900 °C (b), and 1000 °C (c).

cate glass provides the hardness of sintered clay mixtures. Finally, the small peak at 540 cm^{-1} of sintered clay mixtures reveals the presence of iron oxide responsible of the reddish color of the sintered clay mixtures²⁰.

3.4 SEM Analysis

Figure 3 shows SEM images of sintered specimens with 25 wt% rice husk charcoal added to the clay mixtures. It can be seen that the morphologies of the specimens

Table 1. Effects of particle size, amount of rice husk charcoal, and sintering temperature on linear shrinkage, bulk density, water absorption, open porosity, and compressive strength of sintered clay mixtures.

Clay:Zeolite: Husk charcoal	Size (μm)	Temperature (°C)	% Linear shrinkage	Density g/cm ³	% Water absorption	% porosity	Strength (kN/m ²)
25:25:50	>250	800	5.67	0.69	67.30	68.52	566.17
		900	6.95	0.83	60.45	63.46	753.77
		1000	8.91	0.99	54.71	56.51	866.17
	88-250	800	5.78	0.72	61.85	65.23	753.77
		900	7.28	0.93	56.60	58.05	868.89
		1000	8.78	0.95	53.56	56.44	907.55
	<88	800	6.32	0.73	57.20	62.26	1397.02
		900	7.65	0.95	55.22	56.57	1489.42
		1000	9.43	0.97	50.52	52.66	1599.64
32.5:32.5:35	>250	800	7.28	0.83	48.35	49.69	2832.34
		900	7.78	0.97	45.76	46.94	2907.56
		1000	10.40	1.05	40.12	41.51	3015.11
	88-250	800	7.52	0.94	47.79	48.23	3248.68
		900	8.40	1.02	44.09	45.16	3260.89
		1000	11.00	1.09	39.30	40.03	3449.70
	<88	800	7.76	0.95	45.07	47.56	3286.68
		900	8.87	1.08	39.44	43.64	3264.68
		1000	12.56	1.15	37.61	39.64	3492.56

Table 1 Continued

37.5:37.5:25	>250	800	8.08	0.97	42.70	42.80	3349.94
		900	9.06	1.10	38.19	38.14	3649.94
		1000	12.68	1.22	35.72	36.51	4249.47
	88-250	800	9.02	0.99	40.61	40.65	4199.65
		900	9.54	1.16	35.98	36.48	4299.76
		1000	13.03	1.28	33.08	34.91	4349.59
	<88	800	9.40	1.10	39.72	39.90	4512.66
		900	10.00	1.23	35.60	35.27	4522.59
		1000	14.22	1.43	27.27	30.44	4629.66
		800	9.69	1.40	10.48	16.07	5199.53
50:50:0		900	12.52	1.53	9.39	15.99	7799.29
		1000	16.00	2.09	9.38	14.41	12998.83

change in response to changes of the sintering temperature. For sintering at 800°C (Figure 3a), the specimen shows the sheet-like morphology with high open porosity and macropores on the surface. High open porosity and macropores are both advantageous for the improvement of permeability and the filtration velocity of aqueous solution. For the specimen prepared by sintering at 900°C (Figure 3b), it can be seen that the surface is deflated. Furthermore, the surface of the specimen obtained after sintering at 1000°C is quite smooth in comparison to specimens prepared with sintering at 800°C and 900°C. These results confirm that the clay mixtures with rice husk charcoal additive melt to crystalline phases at high sintering temperatures, especially at 1000°C, which con-

sequently influences the mechanical properties of the sintered specimens as mentioned above. The observations from the SEM images are consistent with the results of mechanical properties (Table 1) and FTIR transmission (Figure 2). All of these methods point to a decrease in open porosity and water absorption, and an increase in linear shrinkage, bulk density and compressive strength in response to increasing the sintering temperature from 800°C to 1000°C for a fixed ratio of rice husk charcoal.

4. Conclusion

The effect of particle size, amount of rice husk charcoal, and sintering temperature on the properties of sintered clay mixtures show highly significant effect of these vari-

ables on the properties of the sintered products. The results show that the levels of percent water absorption and porosities are higher for all sintered clay mixtures containing the rice husk charcoal additive in comparison to the pure clay mixtures, while the density and compressive strengths show a reverse trend. Furthermore, the results indicate increased water absorption and open porosity being achieved with increasing amount and size of rice husk charcoal, while the density and compressive strength decrease. In terms of water filter production, the mixtures of rice husk charcoal and clays with ratios of 25 wt% rice husk charcoal for all sizes: 37.5 wt% Thapo clay : 37.5 wt% zeolite sintered at 800-1000°C result in materials with suitable properties. The values of porosity and compressive strength are according to traditional water filter manufacture standards. The FTIR and SEM results support the results of evaluations of physical and mechanical properties of the sintered specimens.

5. Acknowledgement

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