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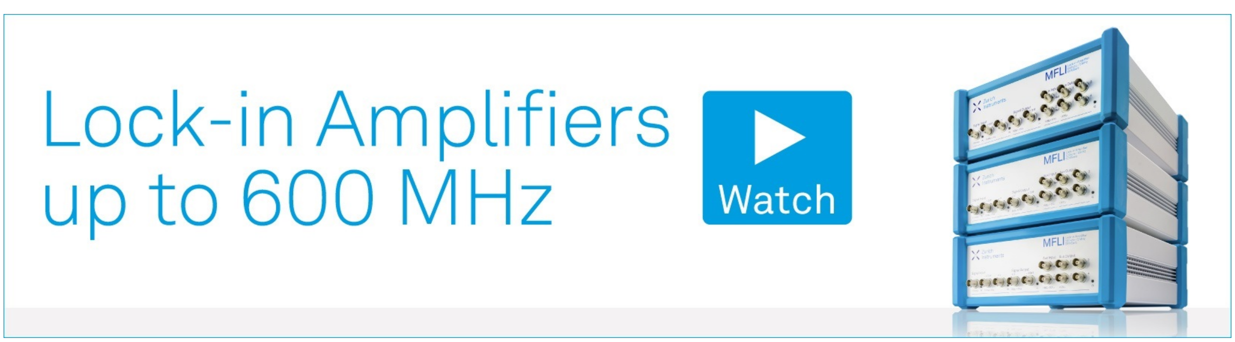

Manufacturing Process and Characterization of Porous Ceramics with AAS, XRD and SEM-EDX

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Abstract. Research has been conducted on the manufacturing process and characterization of porous ceramics with raw materials like alumina, kaolin, fly ash and activated carbon. The composition comparison of alumina raw materials is kaolin (1:4) with fly ash and activated carbon (50:35:15) percent from weight. Alumina, kaolin, fly ash and activated carbon materials have a diameter of 100 mesh each, then added with water, mixed and pressurized homogeneously with a pressure of 5 tons, conditioned for 48 hours and burned at 1100°C for 2 hours. The characteristics of porous ceramics on the composition of 15% activated carbon have the highest porosity of 23.54%, with phase Corundum (Al_2O_3) through the standard JCPDS No. 46-1212 lattice parameters, a = 4.758 Å, c = 12.99 Å, the crystal structure was trigonal and Mullite phase ($3Al_2O_3.2SiO_2$) through standard JCPDS No. 15-776, lattice parameters a = 7.5430 Å, b = 7.6872 Å and c = 2.8842 Å with Orthorhombic crystal structure.

INTRODUCTION

The development of ceramic materials is increasing due to its application in all fields, especially those that prioritize durability at high temperatures, resistance to chemicals, good mechanical strength and low pollutant effects [1,2]. Rapid technological advancement have led to the continuous manufacturing of porous ceramic materials in order to obtain a lower production cost. One type of porous material is an exhaust gas filter. The exhaust gas filter is widely used in the car industry. The use of porous materials in the car industry can be distinguished according to the size of porosity from macro to micro sizes [3-5].

The most common preparation method of manufacturing porous ceramics is the slip casting method, dry pressing and extrusion forming. Most industries apply the extrusion method in manufacturing their products, while the slip casting and dry pressing methods are mostly carried out on a laboratory scale [6]. In the manufacturing process of porous ceramics by the dry pressing method, all ingredients are prepared in powder form with known particle size. All ingredients are mixed to obtain a homogeneous mixture. Afterwards, the material is put into the mold with a certain pressure and the ceramic green body is inserted into the furnace for sintering. There are several variables that can affect the yield of ceramics using the dry pressing method, namely the particle size of material particles, homogeneity of the mixture of materials, compaction pressure, sintering temperature and holding time [7]. The exhaust gas filter that will be produced in this study utilizes volcanic ash from Mount Sinabung, alumina, kaolin and activated carbon as additives in the manufacture of porous ceramics. Porous ceramic in mold with dry pressing method and heated to a temperature of 1100°C for 2 hours [8].

Activated carbon is carbon that has undergone changes in physical and chemical properties due to chemical activators or high temperatures, hence the pores are open which enable greater carbon absorption compared to ordinary carbon absorption. Activated carbon is widely used as an absorbent material [9]. This research aims to produce porous ceramics by utilizing volcanic ash from Mount Sinabung, alumina, kaolin and activated carbon as porous ceramic raw materials [10, 11].

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RESEARCH METHOD

Raw materials such as alumina, kaolin and volcanic ash were sourced from the crater of Mount Sinabung, 4 km from Berastepu village. The activated carbon was sifted at 100 mesh each, with a comparison of alumina material: kaolin is 1:4 [6]. Each ingredient was weighed and the composition of alumina, kaolin, volcanic ash and activated carbon was made as shown in Table 1.

TABLE 1.Comparison of Ceramic Composition

No.	Alumina : Kaolin 1 : 4 (% weight)	Volcanic Ash (% weight)	Activated Carbon (% weight)
1	50	50	0
2	50	45	5
3	50	40	10
4	50	35	15
5	50	30	20

Alumina powder, kaolin, volcanic ash and activated carbon are mixed in several plastic containers evenly. After the powder mixture is evenly distributed, removed and scattered over the plastic container, it is then evenly sprayed with water to soften the powder. Afterwards, it is inserted for dry pressing into the first mold of iron cylindrical hollow shape with an outer diameter of 3.9 cm, inside diameter of 1.6 cm and height 6 cm, to be pressed with 2 tons weights for 5 minutes. The pellet-shaped mold with a diameter of 3 cm with a height of 4 cm, thickness of 2.3 cm, is pressed with 5 tons Hydraulic Press for 5 minutes. Eventually, the sample was removed and conditioned for 6 days [7]. After 6 days, the sample was burned in High Temperature Furnace at 1100^{o}C for 2 hours. Then, the crystal structure and phase were tested with XRD and elemental analysis with SEM-EDX [8]. The analysis of the composition of volcanic ash elements was carried out with AAS [7,8]. Photographs of hollow cylindrical shape samples and pellet samples are shown in Fig. 1.

(a) (b)

FIGURE 1. Porous ceramic sample (a) hollow cylinder, (b) pellet cylinder

RESULT AND DISCUSSION

Analysis of Volcanic Ash Compounds

Volcanic ash from Mount Sinabung erupted on March 14, 2018, the ash were taken at Berastepu village, Karo Regency, 2 km from the crater of Mount Sinabung, and the composition of the compounds with AAS was analyzed. The composition of volcanic dust is consisted of 87.19% SiO_2; 3.08% Fe_2O_3; 6.67% Al_2O_3; 0.13% MgO; 2.83% CaO; 0.02% Na_2O. From the chemical compounds obtained, it can be further classified into types of ceramics in order to be used as ceramic raw material [5].

Analysis of Heavy Metal in Volcanic Ash

Heavy metal analysis is carried out with Atomic Absorption Spectrometry (AAS), the results of the analysis are shown in Table 2.

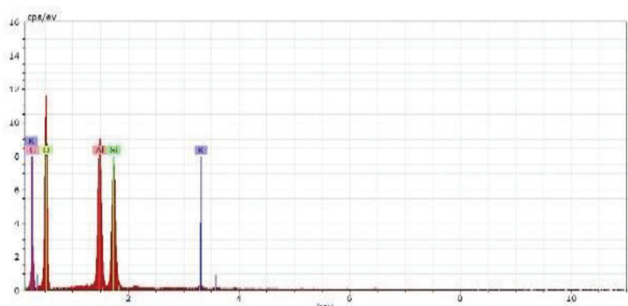
TABLE 2. Composition of Heavy Metal in Volcanic Ash

No	Heavy Metal	Unit	Amount
1	Copper (Cu)	ppm	46,35
2	Lead (Pb)	ppm	< LoD
3	Cadmium (Cd)	ppm	< LoD
4	Arsenic (As)	ppm	< LoD
5	Iron (Fe)	%	4,37
6	Zinc (Zn)	%	0,02
7	Mercury (Hg)	ppm	< 0,001
8	100 mesh smoothness	%	85.20

LoD Lead (Pb) < 50 ppm, LoD Mercury (Hg) < 1 ppm, LoD Cadmium (Cd) < 10 ppm, LoD Copper (Cu) < 5000 ppm, LoD Arsenic (As) < 10 ppm, Iron (Fe) < 5000 ppm. The heavy metal composition of Mount Sinabung volcanic ash (DVGS) shown in table 4.1 above is analyzed. The result showed that DVGS contains heavy metals, such as: Copper (Cu) = 46.35 ppm; Lead (Pb) < LoD; Cadmium (Cd) < LoD; Arsenic (As) < LoD; Iron (Fe) = 4.37%; Zinc (Zn) = 0.02% and Mercury (Hg) = <0.001 ppm. Lead (Pb), Cadmium (Cd) and Arsenic (As) possess smaller composition than the detection limit (LoD). The volcanic ash of Mount Sinabung contains heavy metals, such as Fe, Cu, Hg and Zn, with very low compositions compared to the LoD of each element and the composition of heavy metals Pb, Cd and As are much smaller than LoD.

Kaolin

The chemical composition and pore diameter size of kaolin material were analyzed by Bruker's SEM-EDX, where by the chemical composition of kaolin (% wt) is as follow: Carbon 25.39, Oxygen 45.16, Aluminum 11.42, Silicon 9.98 and Potassium 0.34. EDX Spectrum analysis is displayed as shown in Fig. 2 below.



Spectrum: Acquisition

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Carbon	K-series	21.69	25.39	34.06	4.00
Oxygen	K-series	45.16	52.88	53.26	6.36
Aluminium	K-series	9.75	11.42	6.82	0.51
Silicon	K-series	8.52	9.98	5.72	0.41
Potassium	K-series	0.29	0.34	0.14	0.05
	Total:	85.41	100.00	100.00	

FIGURE 2. Analysis of Kaolin Sample Elements

Microstructure of kaolin samples with magnification of 2.5 KX is shown Fig. 3 below. The pore size of the kaolin sample is carried out at five points as in Fig. 3.

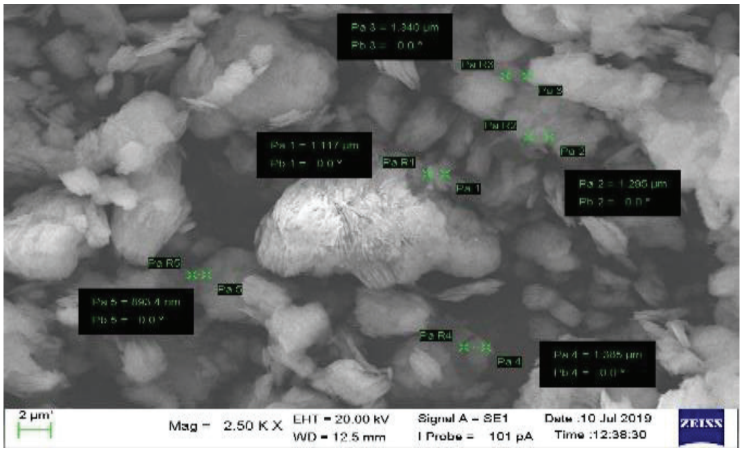


FIGURE 3. Kaolin Microstructure

The five data points obtained in Fig. 3 above are the average pore diameter size of the kaolin sample, which is 1.206 µm. The morphology of the kaolin samples is the same and uniform.

Porosity

Measurement data on wet mass, dry mass and sample volume are shown in the Table 3.

TABLE 3. Results of Porous Ceramic Porosity Measurement

No.	Activated Carbon (%)	Mass (g)		Volume (cm^3)	Porosity (%)
		Dry	Wet		
1	0	43.38	45.92	20.96	12.19
2	5	39.58	42.18	19.59	13.18
3	10	32.49	35.39	17.78	15.86
4	15	24.93	28.57	16.04	23.54
5	20	14.34	17.33	9.88	23.24

From the table above, the relationship between the porosity value of the composition of activated carbon can be plotted in a graph as shown in Fig. 4 below.

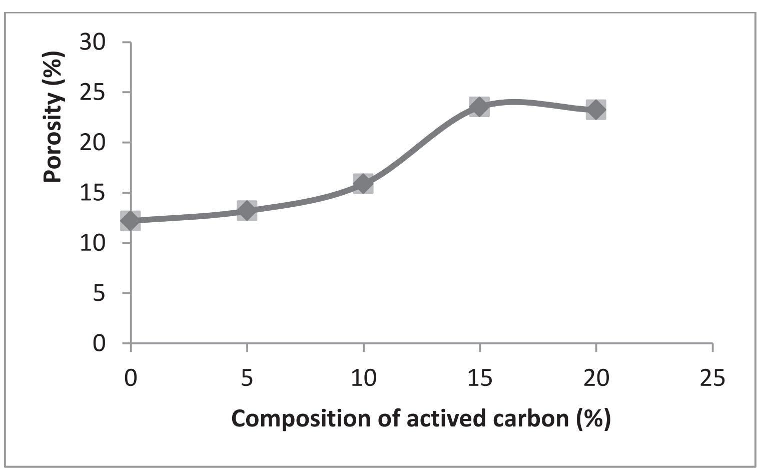


FIGURE 4. Relation between Porosity and Activated Carbon

From Fig. 4, it can be seen that the addition of activated carbon ceramic will result in increased porosity of the ceramic. The ceramic porosity increases because the surface area as active carbon also increases. The porosity value of the ceramic is extremely high, it is estimated that the composition of the addition of activated carbon is 15%.

Structure and Ceramics Phase Testing

Analysis of ceramic structure and phase was carried out by XRD, the XRD diffraction pattern of porous ceramics is shown in Fig. 5 below.

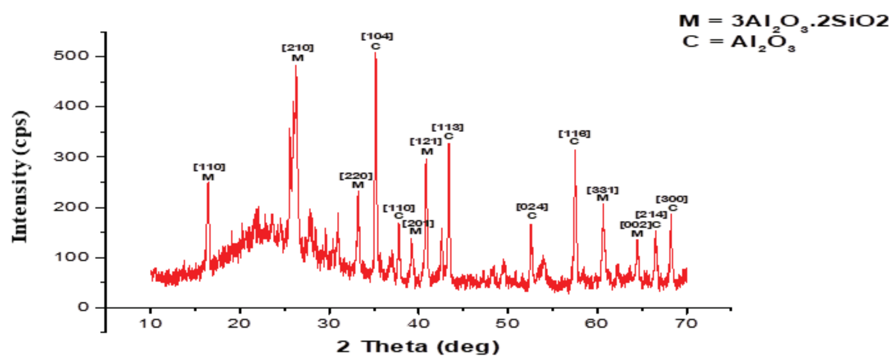


FIGURE 5. Porous ceramic diffraction pattern on 15% activated carbon composition

The ceramic structures and phases with the composition of 15% activated carbon which were burned at a combustion temperature of 1100°C for 2 hours were analyzed using MATCH version2 software. The analysis result showed porous ceramics with Corundum phase (Al_2O_3) through the JCPDS No. standard. 46-1212 with lattice parameters, a = 4.758 Å, c = 12.99 Å with Trigonal crystal structure and porous ceramics with Mullite phase ($3Al_2O_3.2SiO_2$) through JCPDS No. standard. 15-776 with lattice parameters, a = 7.5430 Å, b = 7.6872 Å and c = 2.8842 Å with Orthorhombic crystal structure.

Microstructure and Analysis of Ceramic Elements

Ceramic microstructure with 15% activated carbon and a combustion temperature of 1100°C for 2 hours is shown in Fig. 6.

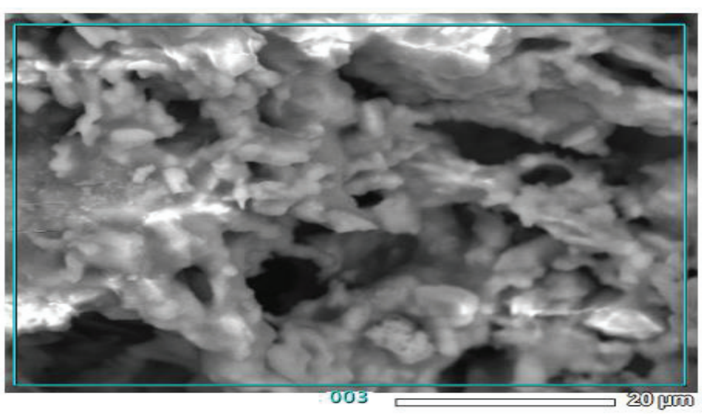
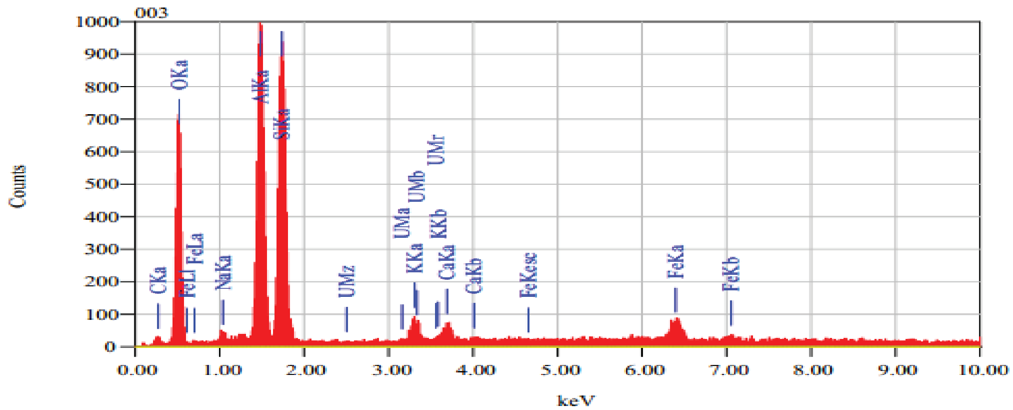


FIGURE 6. Porous microstructure of activated carbon with 15% composition

Ceramic microstructure has pores spread evenly and uniformly on the surface of the ceramic, so that the uniformity of the pores will produce absorption of motorized vehicle exhaust emissions more optimally.



ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.2490

Element	(keV)	Mass%	Error%	Atom%	Compound	Mass%	Cation	K
C K	0.277	5.95	0.23	9.97				0.9101
O K	0.525	45.37	0.19	57.06				50.8233
Na K	1.041	0.96	0.15	0.84				0.8639
Al K	1.486	18.76	0.10	13.99				18.1109
Si K	1.739	20.34	0.13	14.57				18.3999
K K	3.312	1.70	0.14	0.88				2.2280
Ca K	3.690	1.46	0.16	0.74				2.0351
Fe K	6.398	5.46	0.34	1.97				6.6286
U M*								
Total		100.00		100.00				

FIGURE 7. Analysis of porous ceramic elements on 15% activated carbon composition

Elements contained in porous ceramics with a comparison of alumina as raw materials are consisted of: kaolin (1: 4) with fly ash and the composition of 15% activated carbon is consisted of oxygen (O) 45.37 %wt, silica (Si) 20.34 %wt, aluminum (Al) 18.76 %wt, Carbon (C) 5.95 %wt, Iron (Fe) 5.46 %wt, Calsiun (Ca) 1.46 %wt, Sodium (Na) 0.96 %wt and potassium (K) 1.70 %wt.

CONCLUSION

The results of the porous ceramic characterization test can be summarized as follows: Mount Sinabung volcanic ash contains silica (SiO_2) compounds of 87.19%wt. Therefore, it can be used as a raw material to replace quartz materials in ceramic manufacturing. The ceramic porosity on the addition of 15% activated carbon is 23.54% with a density of 1.76 g/cm^3, also with the same pore shape and uniformity. The ceramic structures and phases with the composition of 15% activated carbon possess Corundum phase (Al_2O_3) through the JCPDS No. standard. 46-1212 with lattice parameters, a = 4.758 Å, c = 12.99 Å with Trigonal crystal structure and porous ceramics with Mullite phase ($3Al_2O_3.2SiO_2$) through JCPDS No. standard. 15 -776 with lattice parameters, a = 7.5430 Å, b = 7.6872 Å and c = 2.8842 Å with Orthorhombic crystal structure.

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