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Influence of Sodium Silicate, Sodium Aluminate, and *Filicium Decipiens* Charcoal on The Conductivity of Artificial Zeolite

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ABSTRACT

Fly ash-bottom ash of coal is a solid waste in many industries and electric power plants. One method to reduce that solid waste is by using it to produce artificial zeolite. The Filicium decipiens charcoal was added into artificial zeolite to increase its performance to adsorb water pollutants. The pumice stone is also used in this work as a source of silica. The manufacture of artificial zeolite was carried out by the hydrothermal method by optimizing the composition of sodium silicate from Lampung pumice silica, sodium aluminate from aluminum scrap, and charcoal of Filicium decipiens. From the characterization of the LCR meter, the optimum artificial zeolite obtains the conductivity value from 9.14×10^{-6} (S/cm) to 6.09×10^{-6} (S/cm), confirming that artificial zeolite is a semiconductor material. The highest conductivity of artificial zeolite will increase its performance as a catalyst and absorbent. The composition of the artificial zeolite after XRF characterization was aluminum, silica, phosphorus, potassium, calcium, titanium, vanadium, chromium, Mangan, and ferrite. From the XRD characterization result obtained, the dominant phase is Zeolite-A. SEM-EDX characterization results obtained a spherical and cylindrical microstructure of artificial.

Keywords: *Zeolite, Lampung Pumice, Aluminum Can Waste, Conductivity, Charcoal*

1. INTRODUCTION

Zeolite is one of the active materials that is widely used in chemical processes and water treatment. Many types of zeolites were synthesized and used as adsorbents. Recently, zeolite has been manufactured using cheap natural-based materials such as rice husk [1], kaolin [2], fly ash [3], and volcanic ash [4]. The basic framework of the zeolite structure consists of units tetrahedral (AlO_4) and (SiO_4) [5]. Artificial zeolites are compounds that have physical and chemical properties like natural zeolites. This zeolite is made from other materials by a synthetic process. Zeolite is grayish-white, greenish-white, or yellowish-white with a zeolite density of 2.0-2.3 $g\ cm^{-3}$ and the shape is smooth and soft [6]. The types of zeolites can be distinguished into two types, namely natural zeolite, and synthetic/artificial zeolite. However,

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the purity of natural zeolite is usually lower than synthetic/artificial zeolite [7]. One artificial zeolite type is zeolite-A which has the mineral composition of $Na_{12}[(AlO_2)_{12}(SiO2)_{12}].27H_2O$, which has a low ratio Si/Al of close to 1 [9-11]. This type of zeolite is good performance for wastewater treatment, hard water treatment, and ion exchangers [10]. Saraswati (2015) has successfully produced Zeolite-A from Glass waste as a silica source [11]. This artificial zeolite was synthesized at the ratio of sodium silicate, and sodium aluminate (Si/Al) was 1.05: 1.

Artificial zeolite is commonly synthesized using synthetic sodium silicate and sodium aluminate [8]. However, some of them utilized natural resources to replace sodium silicate with natural resources like pumice and synthesized with sodium aluminate [5]. Many waste resources can be utilised to produce sodium aluminate. An aluminum can waste contains a large amount of aluminum that can be extracted and used as raw material. Beverage cans contain around 92 % -99 % aluminum [12]. By utilizing aluminum can waste, the pollution to the environment can be reduced.

In this work, charcoal was used for the absorbance performance of artificial zeolite. The pores in activated charcoal are generally larger than the zeolite pores, which means that activated charcoal can adsorb a wider range of molecules. This makes activated charcoal more effective at eliminating odors caused by a variety of sources [13]. Adsorbents from biomass resources produced by charcoal commonly possess excellent performance in the adsorption process due to their large surface area. Indonesia has an abundance of forest potential that can be turned into a beneficial product. *Filicium decipiens* originate from tropical Asia and Africa but are now common in various parts of Indonesia. The biomass waste from the trees can be transformed into charcoal. Charcoal is a porous solid material that contains large carbon elements. It is commonly produced by the carbonization process and forms activated carbon (charcoal). Activated carbon is carbon with space very large pores of a certain size that can trap the absorbed particles [10].

This study aims to produce artificial zeolite, which is supported by utilizing cheap material abundantly available in Indonesia. The main raw materials, such as sodium silicate and sodium aluminate produced from Lampung pumice and aluminum can waste, respectively. In addition, charcoal made of *Filicium decipiens* leaves was introduced in the artificial zeolite to improve the adsorption capacity.

In this work, the concentration of sodium silicate, sodium aluminate, and *Filicium decipiens* charcoal was varied and optimized for a high conductivity value. Understanding the conductivity value of artificial zeolite is very important, as zeolite with a high conductivity value will have a good performance as an absorbent and catalyst. The physical characteristic such as metal composition, conductivity, and morphology of the synthesis of artificial zeolite was also examined.

2. METHODOLOGY

2.1 Material and Chemicals

The materials used in this study were Lampung pumice (LP), aluminum can waste (AC) and *Filicium decipiens* leaves (FD), caustic soda flake (NaOH) 98 % from Merck ASC, and distilled water.

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2.2 Preparation of Raw Material

The artificial zeolite (AZ) was prepared by mixing three main chemical formulations, i.e., sodium silicate (Na_2SiO_3), sodium aluminate ($NaAlO_2$), and charcoal.

Firstly, charcoal was prepared using biomass sources (Filicium decipiens leaves) which are abundant in Indonesia. The leaves were exposed to the sunlight until fully dried and then carbonized into charcoal. After that, the charcoal was ground to ±100 mesh particle size and kept for further use.

The second material was the production of sodium silicate (Na_2SiO_3) from volcano rock (Lampung pumice). The Lampung pumice (LP) was sun-dried and milled until a fine particle was produced. A 10 g of LP was mixed with 40 g of NaOH (flake) in a beaker. Then the mixture was heated at 350 °C for 4 hours. Subsequently, the solid was diluted in an aqueous solution and left for 12 hours. The chemical reaction that occurred is expressed by Equation 1 [5]. The sample was sent for characterization by XRF to determine the silica composition.

$$SiO_{2(s)} + 2NaOH_{(l)} \rightarrow Na_2SiO_{3(s)} + H_2O_{(l)} \tag{1}$$

Lastly, sodium aluminate ($NaAlO_2$) was prepared using an aluminum can waste from local residency. The Aluminium can (AC) was cleaned, dried, and cut into small pieces. In a beaker, 55 g of NaOH (flake) was mixed with 600 ml aqueous or 2.3 M NaOH. Then, 70 g of AC was slowly added into the beaker filled with NaOH solution and stirred until the color changed to brownish and yellowish. The chemical reaction that occurs during the reaction is expressed by Equation 2 [5]. Afterwards, the solution was filtered, and then the solid was dried for 4 hours in the oven. Finally, the $NaAlO_2$ sample was ground into small particles size of 60 mesh and analyzed using XRF to determine the composition of aluminum.

$$2Al + 2NaOH_{(s)} + H_2O_{(l)} \rightarrow 2NaAlO_{2(s)} + H_{2(g)} \tag{2}$$

2.3 Preparation of Artificial Zeolite

The AZ was prepared by varying the weight concentration of Na_2SiO_3, $NaAlO_2$, and charcoal (FDC) in the formulation. All compounds were mixed with different weights, as shown in Table 1.

Table 1: Formulation of AZ samples with the different masses of Na_2SiO_3, $NaAlO_2$, and charcoal (FDC)

AZ Sample	Na_2SiO_3 (g)	$NaAlO_2$ (g)	FDC (g)
1	10	15	10
2	12.5	15	10
3	15	15	10
4	17.5	15	10
5	20	15	10
6	15	15	6
7	15	15	8
8	15	15	10
9	15	15	12
10	15	15	14

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The mixed compounds of Na_2SiO_3, $NaAlO_2$, and charcoal (FDC) were stirred for 24 hours at room temperature and filtered. Subsequently, the mixed sample was heated at 100 °C for 5 hours. Then the solid was washed with distilled water until its pH reached 7. Afterwards, the solid sample was filtered, and oven dried at 100 °C for 2 hours. Finally, the mineral composition in the sample was characterized using XRF (X-Ray Fluorescence) from Malvern Panalytical (PANalytical Epsilon 3XLE- Holland). The conductivity of the sample was analyzed using LCR (inductance (L), capacitance (C), and resistance (R)) meter. LCR meter is a type of electronic test equipment that measures the inductance (L), capacitance (C), and resistance (R) of an electronic component. The powder of artificial zeolite was filled with the Hollow Cubical Acrylic (1 cm x 1 cm x 1 cm), which has a copper electrode at both sides, as shown in Figure 1. The copper electrodes were connected to an LCR meter (LCR-6002, Good Will Instrument Co., Ltd., Taiwan). The powder of artificial zeolite was filled with hollow cubical acrylic.

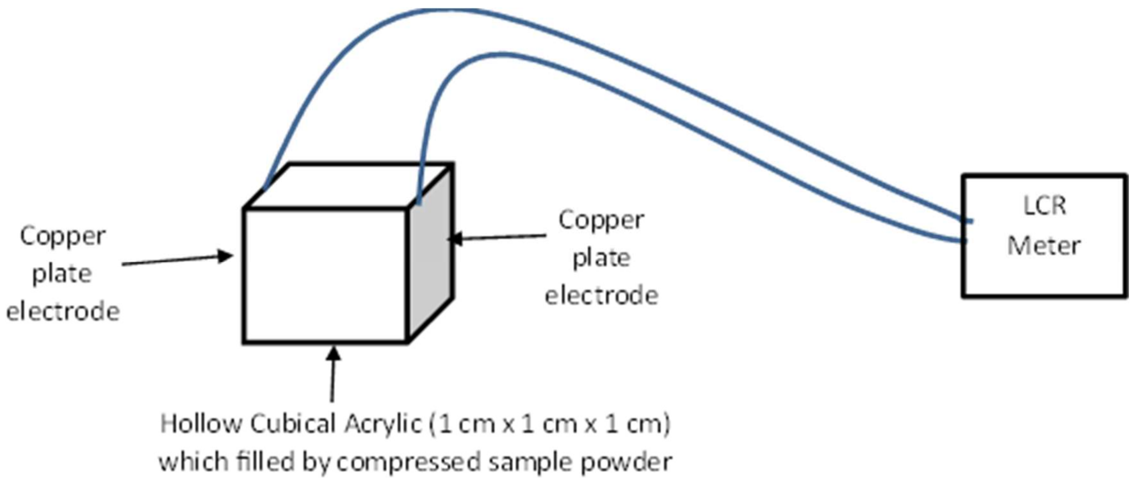

Figure 1. Schematic diagram of LCR meter to measure the conductivity of zeolite

The optimum sample was determined based on the highest conductivity. The conductivity was calculated according to Equation 3 [14].

$$\sigma = G\left(\frac{l}{A}\right) \tag{3}$$

where σ is conductivity (S/cm), G is conductance (Siemen, S), A is cross-section area (cm^2), and l is length (m). The morphology of artificial zeolite with optimum conductivity was analyzed using Field Emissions - Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray Spectroscopy (EDX) from Thermo Fisher Scientific Inc. (Thermo Scientific Quattro S)- United States. The crystal structure analysis was conducted using X-Ray Diffraction (XRD) from Malvern Panalytical (PAnalytical X'Pert3 Powder)- Holland.

3. RESULT AND DISCUSSION

3.1.1 Characterization of sodium silicate (Na_2SiO_3) and sodium aluminate ($NaAlO_2$)

In this study, sodium silicate (Na_2SiO_3) and sodium aluminate ($NaAlO_2$) were obtained from Lampung pumice, and aluminum can waste, respectively, after being melted with an alkaline

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solution (NaOH). Then, the metal composition of Na_2SiO_3 and $NaAlO_2$ was determined by XRF characterization. Table 2 shows the result obtained from XRF.

Table 2: Metal composition of Na_2SiO_3 and sodium aluminate $NaAlO_2$ using XRF

No	Metal	Concentration Unit (%)	
		Na_2SiO_3	$NaAlO_2$
1	Na	29.6	15.8
2	Al	8.0	75.0
3	Si	26.5	1.9
4	P	1.2	2.6
5	K	1.5	0.3
6	Ca	8.8	1.3
7	Ti	1.6	0.2
8	V	111.0 ppm	363.6 ppm
9	Cr	291.5 ppm	-
10	Mn	0.6	0.2
11	Fe	-	2.1

The XRF result shows that Na_2SiO_3 from LP contains a high amount of Silica (Si) which is 26.5 %. This indicates the suitability of LP to be used as raw material for Na_2SiO_3 production. A similar observation can be seen from $NaAlO_2$. The high percentage of aluminum (Al) contained in $NaAlO_2$ (75 wt%) denotes the significance of aluminum can from waste to be used in $NaAlO_2$ production.

3.1.2 Effect of AZ material loading on Conductivity

Electric conductivity value (ECV) is an important parameter for AZ material identification. It indicates the physical properties of a material to identify the type of material, either conductor (> 10^3 S/cm), insulator (<10^{-9} S/cm), or semiconductor (10^{-8} – 10^3 S/cm) [14]. The conductivity of AZ was tested using an LCR meter, and ECV on various AZ material loading is presented in Figure 2. The variation of AZ material loading indicated by samples 1-10 can be referred to in Table 1.

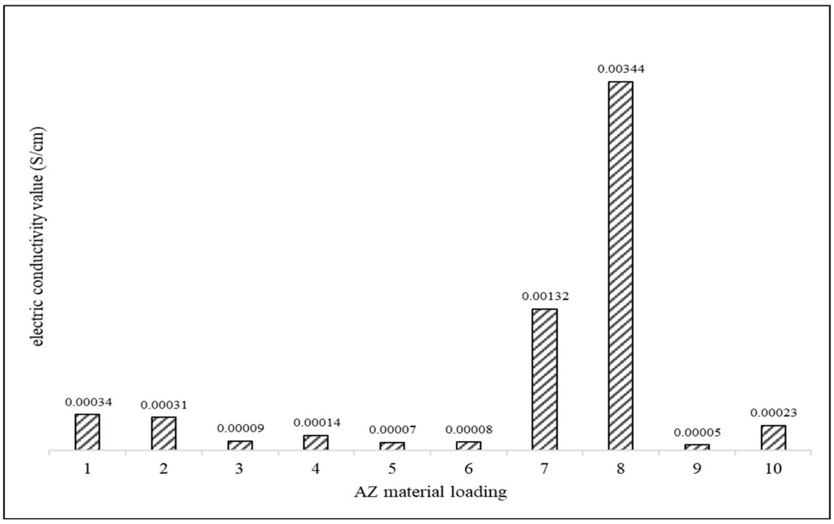

Figure 2: electric conductivity value on the effect of AZ material loading

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Figure 2 shows the ECV for samples 1-10. All samples indicate the ECV sets the range of 10^{-5} to 10^{-3}; thus, AZ is classified as a semi-conductor. In general conductivity of the material is influenced by crystallinity, microstructure, and specific area of material cause the conductivity depends on the material loading [15]. According to Mahaddila and Putra [16], zeolite with a high ECV possesses a higher ionic capacity, thus increasing the ability to accept cations and being suitable to act as an adsorbent.

Higher ECV denotes better AZ material than lower ECV [14]. It can be seen AZ sample no. 8 (AZ-8), which contains 15:15:10 g of Na_2SiO_3, $NaAlO_2$, and FDC, respectively, indicates the highest ECV. Hence, AZ-8 is the optimum formulation suitable to be applied in hard water or wastewater treatment [5, 15, 16].

3.1.3 Relationship between AZ conductivity with sodium silicate (Na_2SiO_3), sodium aluminate ($NaAlO_2$), and Filicium decipiens charcoal (FDC)

As can be seen from Figure 2, the material loading has a significant influence on electric conductivity value (ECV). The influence of sodium silicate (Na_2SiO_3), sodium aluminate ($NaAlO_2$), and Filicium decipiens charcoal (FDC) will be discussed further in the following section.

3.1.3.1 Influence of sodium silicate (Na_2SiO_3) loading

Figure 3 shows the influence of different Na_2SiO_3 loading on ECV. It can be observed, Artificial zeolite no. 1 (AZ-1) to no. 5 (AZ-5) obtain values of different conductivity. Where AZ-1 has the highest conductivity value with a value of 5.93×10^{-5} S/cm and AZ-5 has the lowest conductivity value with a value of 1.29×10^{-5} S/cm. Electric conductivity values (ECV) of AZ-3 decrease with a ratio of sodium silicate and sodium aluminate 15:15 g. Comparison between silica (Si) in Na_2SiO_3 and aluminum (Al) in $NaAlO_2$ atoms that vary will be produced by different types of zeolite species [17] which pose different physical and chemical properties. Therefore, the different Si and Al ratio attribute to different characteristics and influence the conductivity of AZ. Therefore, the increase in Al concentration will increase the value of conductivity.

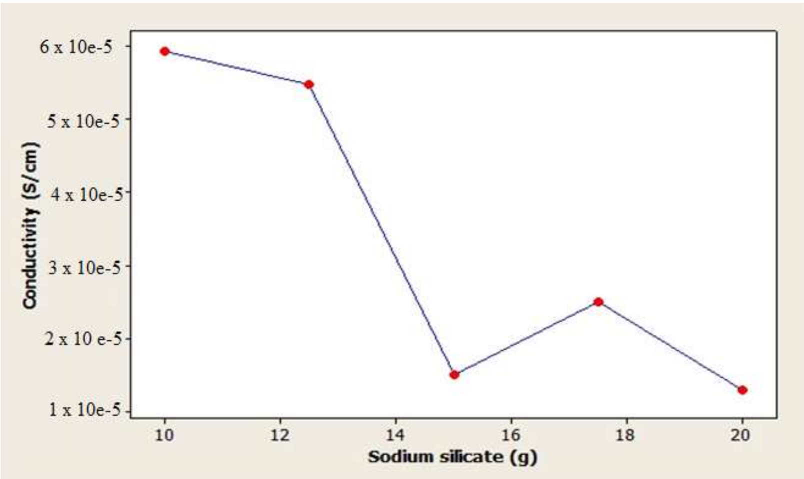

Figure 3: Electric conductivity value on different mass loading of Na_2SiO_3

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3.1.3.2 Influence of Filicium decipiens charcoal loading

A comparison between variations of *Filicium decipiens* charcoal (FCD) towards ECV is presented in Figure 4. The sample of FCD loading represents samples 6 (AZ-6) to 10 (AZ-10) from Table 1.

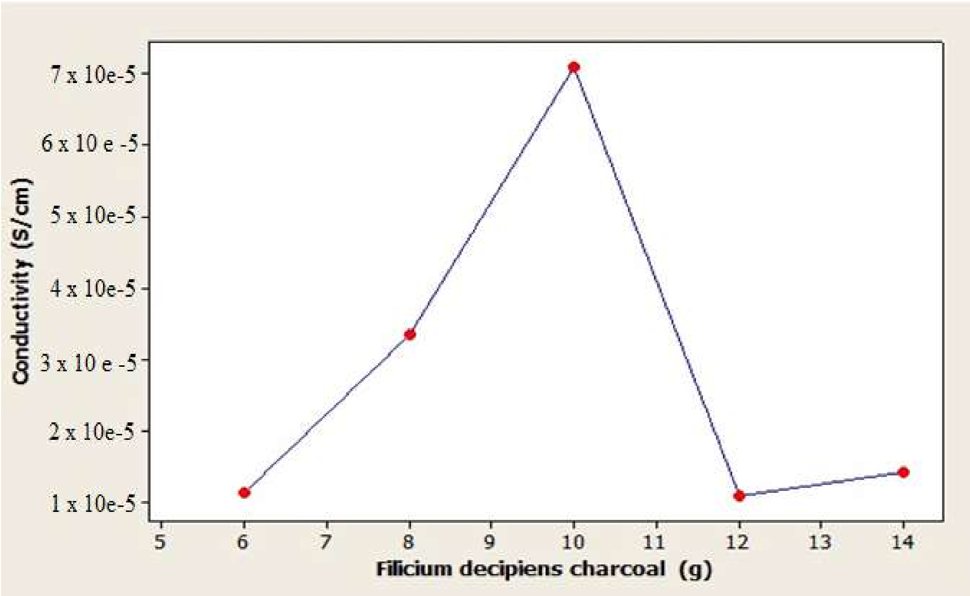

Figure 4: Electric conductivity value on different mass loading of FDC

Based on Figure 4, the AZ ECV has increased from AZ-6 to AZ-8 and decreased afterwards. Meanwhile, AZ-8 shows the highest ECV among other samples, with the mass ratio of *Filicium decipiens* charcoal and sodium aluminate is 10:15 (g). In the study by Maaddila and Putra [5], the observation shows that the ratio of sodium silicate and sodium aluminate is 10:15 (g). However, when adding charcoal from *Ficilium decipiens*, the result indicates differently. The difference in the best sample results can be influenced by the addition of *Filicium decipiens* charcoal in the zeolite manufacturing process.

3.1.3.3 Comparison of AZ conductivity with sodium silicate (Na_2SiO_3), sodium aluminate ($NaAlO_2$), and Filicium decipiens charcoal (FDC).

The overall conductivity of AZ samples (AZ-1 to AZ-10) towards the mass loading of each material can be observed in Figure 5.

The result shows the mass comparison of sodium silicate over total sodium aluminate and charcoal is obtained between the range of 0.51 to 0.75. Where sample 10 (AZ-10) has the smallest ratio of 0.51 with a conductivity value of 4.08×10^{-5} S/cm and sample 1 (AZ-1) has the largest value of 0.75 with a conductivity value of 5.93×10^{-5} S/cm. The mass loading of sodium silicate, sodium aluminate, and *Filicium decipiens* charcoal of AZ-1 is 10:15:10 (g), respectively. Based on Figure 5, the highest ratio among the material does not show the highest conductivity, as sample 8 (AZ-8) shows the highest conductivity with a ratio value of 0.6.

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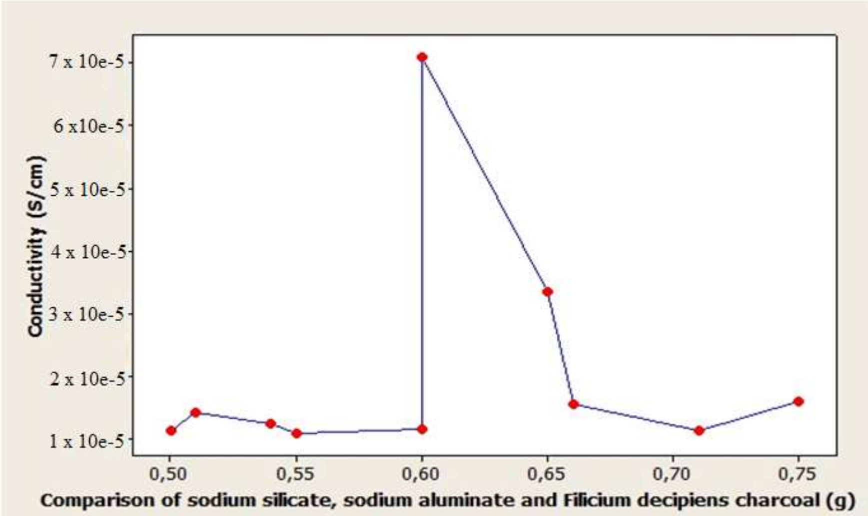

Figure 5: Comparison of AZ conductivity on mass loading of sodium silicate on the total mass of sodium aluminate and charcoal

3.1.3 Artificial Zeolite (AZ) physical properties- XRD and SEM-EDX Characterization

From the results of the LCR meter characterization, the optimum sample is AZ-8. The sample was characterized by XRD and SEM-EDX, which obtained the results shown in Figure 6 and Figure 7, respectively. From Figure 6, three components are obtained, namely Zeolite-A, Albite, and Nacrite. Where the peak is dominated by Zeolite-A, which has the name Sodium Aluminum Silicate. According to Hossein et al. [18], the use of zeolite is based on its ability to perform adsorption and catalytic cation exchange. Zeolite-A usually possesses catalyst properties. AZ-8 is found to dominate by Zeolite-A type, which this finding agrees with the work obtained by Mahadilla and Putra [5]. Thus, it can be concluded that the manufacture of zeolite using sodium silicate, sodium aluminate, and *Filicium decipiens* charcoal does not affect the type of artificial zeolite.

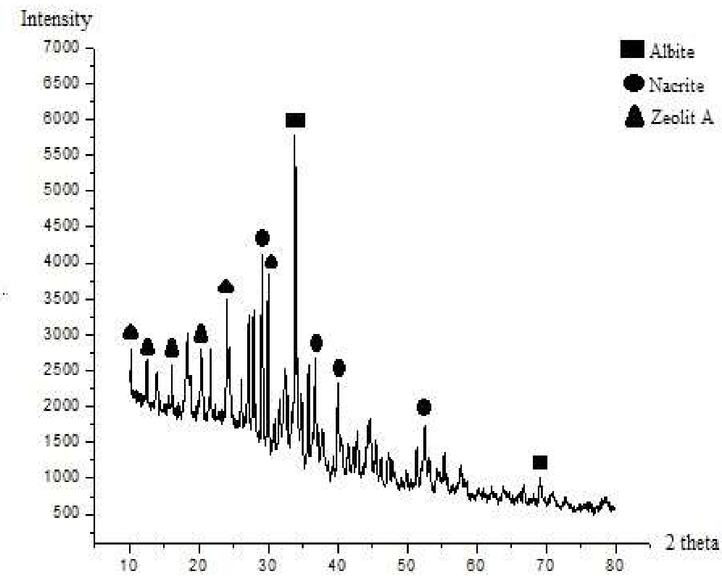

Figure 6: XRD characterization on AZ-8

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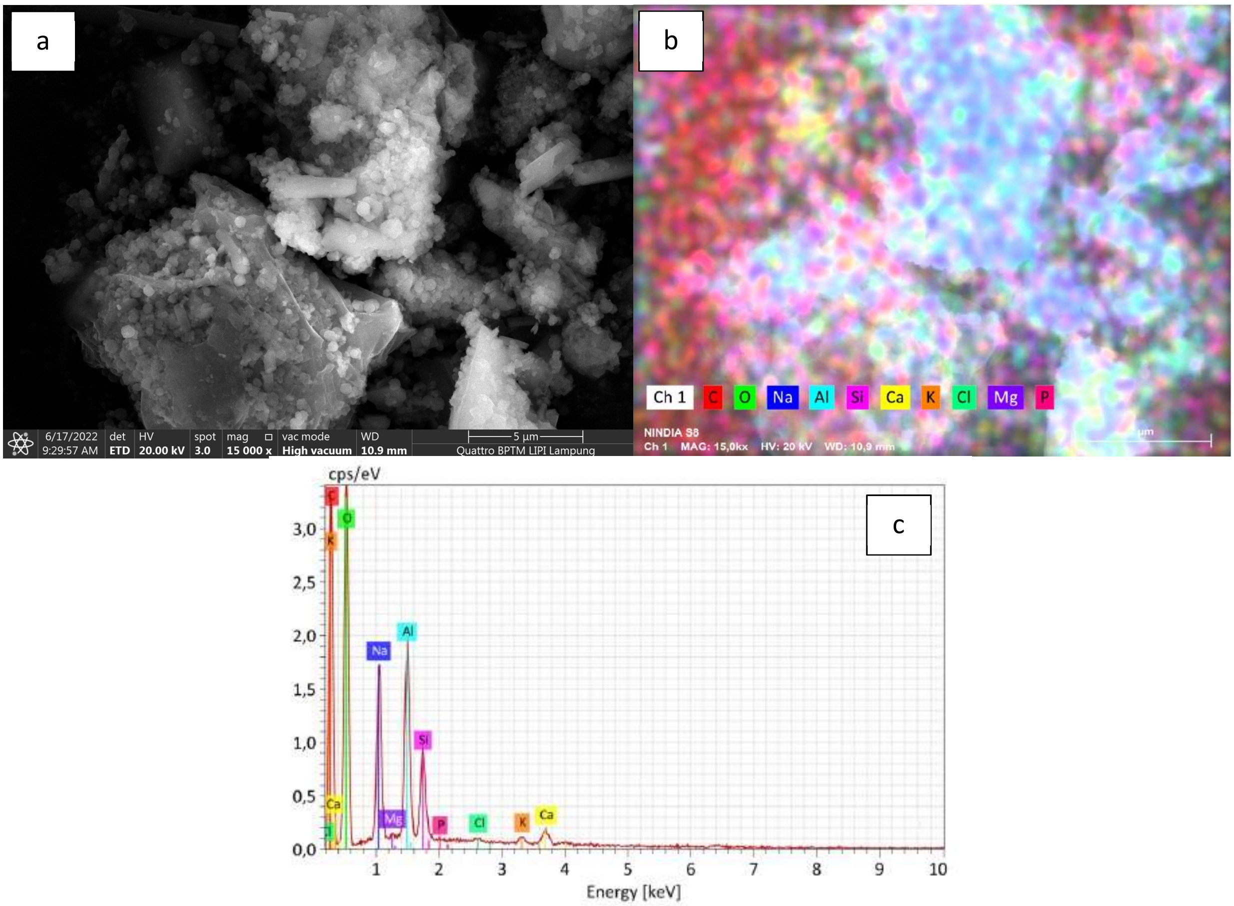

Figure 7: Physical characterization of AZ-8 (A) SEM image with 15000X magnification (B) metal distribution mapping (C) metal composition by EDX

Figure 7 (a-c) shows the microstructure obtained from the AZ-8 sample. The grains formed composed of several spherical and cylindrical particles can be observed at 15000x magnification, as shown in Figure 7(a). Meanwhile, the same figure shows different colors, which are bright white and pale white, indicating the distribution of aluminum and silica element, respectively. Figure 7(b) illustrates the metal element distribution on the surface of AZ-8. It shows that aluminum (Al) and carbon (C) are the dominant element in the artificial zeolite. This matter is due to the presence of a mixture of sodium aluminate and *Filicium decipiens* charcoal in the manufacture of artificial zeolite. Furthermore, the composition of Al, C, Silica (Si), and sodium (Na) had significantly contained in AZ-8, as shown in Figure 7(c).

4. CONCLUSION

Based on the research conducted, it is concluded that artificial zeolite obtains a large conductivity value within the range of 9.14×10^{-6} S/cm to 6.09×10^{-4} S/cm. From the characterization of the LCR meter, the optimum sample, which is AZ-8, is obtained with a conductivity value of 6.09×10^{-4} S/cm. Meanwhile, the metal composition of the artificial zeolite is aluminum, silica, phosphorus, potassium, calcium, titanium, vanadium, chromium, manganese, and ferrite. The AZ-8 shows a structured crystal dominant to the Zeolite-A type as it appears at an angle of 30.888° at 2θ and is identified as round and cylindrical. The zeolite resulting from the characterization of the LCR meter is a zeolite with catalytic properties.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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