



## Preparation and Characterization of Activated Carbon Made from Robusta Coffee Skin (*Coffea Canephora*)

Raudhatul Raihan<sup>1</sup>, Adi Setiawan<sup>2\*</sup>, Lukman Hakim<sup>3</sup>, Muhammad<sup>3</sup>, Muhammad Arif<sup>2</sup>, Hadi Hosseiniamoli<sup>4</sup>

<sup>1</sup> Post-graduate Program in Renewable Energy Engineering, Faculty of Engineering, Universitas Malikussaleh.

<sup>2</sup> Mechanical Engineering Department, Faculty of Engineering, Universitas Malikussaleh

<sup>3</sup> Chemical Engineering Department, Faculty of Engineering, Universitas Malikussaleh

<sup>4</sup> Western Sydney University, Centre for Infrastructure Engineering, Sydney, 2747, Australia

\*E-mail: [adis@unimal.ac.id](mailto:adis@unimal.ac.id)

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### Abstract

Indonesia is one of the coffee-producing countries where production tends to increase from year to year. Currently, residuals from coffee-fruit processing such as coffee-skin and husks are thrown away without any use and this biomass residual contains several toxic chemicals such as alkaloids, tannins, and polyphenols. One of potential uses of coffee-industry by-product is to make activated carbon (ACs), which is made through a carbonation process and followed by an activation process. In this study, chemical activation was carried out using chemical activators ZnCl<sub>2</sub> and NaOH. The purposes of this study were to prepare and investigate the characteristics of chemically activated coffee skin bio-char, especially on the surface area topography and iodine adsorption capacity. Prior to carbon activation, a purpose built pilot-scale reactor was fabricated and tested at temperatures of 400 °C and 500 °C. The difference in carbonation temperature and variations in activator concentration altered the absorption properties. All activated carbon samples were analyzed to find-out the composition, morphology, topography under SEM-EDS analyses as well as absorption test by iodine absorption method. The results showed that the coffee-skin pyrolyzed at 500 °C and activated by 2% NaOH solution which exhibit the highest absorption value of 720.2 mg/g. Lower absorption values were observed in any ZnCl<sub>2</sub> activator samples. SEM-EDS analysis result suggested significant changes in composition of the ACs before and after the activation. Most of impurities were gone during activation and washing. The value of C atoms increases and the pores structure of the activated carbon expanded showing its suitable properties for commercial adsorbent.

Keywords: Chemical activation, Biomass, Coffee skin, Iodine absorption, SEM-EDS analysis

### 1. Introduction

Coffee is a commodity ranked second after petroleum in terms of currencies traded throughout the world. It is, quite relevant to the economies of the main producing countries such as Brazil, Vietnam, Indonesia, Colombia, Ethiopia, India and Mexico. Brazil is the largest coffee producer and exporter in the world, also the second largest consumer. Coffee processing produces significant amounts of agricultural waste, depending on the type of processing ranging from 30% to 50% of the total weight of coffee produced, (Oliveira & Franca, 2014). Coffee production in Indonesia in 2017 reached 666,992 tons, resulting more than 66 thousand tons of agricultural waste. Utilization of coffee waste to date has not been maximized (Badan Pusat Statistik, 2018). The current expansion of coffee plantations in Indonesia will result in an increase in the amount of coffee waste produced. Therefore, a new breakthrough is needed to utilize the coffee waste so that it

can be beneficial. Coffee waste contains several toxic chemicals such as alkaloids, tannins, and polyphenols.

As summarized in Table 1, there are many potential biomass wastes which can be further processed into activated carbon with low cost. Activated carbon is an amorphous compound produced from materials containing carbon that is specially treated to get high adsorption power. Activated carbon can adsorb certain gases and chemical compounds or selective adsorption properties, depending on the size or volume of pores and surface area (Arsad & Hamdi, 2010).

Fabrication of activated carbon is done through a carbonization followed by an activation process. To make activated carbon from organic ingredients there are two methods that can be used: the physical and the chemical activation. The choice of activator type affects the quality of the

resulting activated carbon. Each type of activator has different effects or effects on the surface area and volume of the pores of the resulting activated carbon (Rohmah & Redjeki, 2014).

**Table 1.** List of precursor materials from various agricultural sources for low cost activated carbon production

No	Raw material	Reference
1.	Cassava peel	(Moreno-Piraján & Giraldo, 2010)
2.	Jatropha curcas fruit shell	(Tongpoothorn et al., 2011)
3.	Spent coffee ground	(Castro et al., 2009)
4.	Olive seeds	(Michailof et al., 2008)
5.	Rice husk	(Rahman et al., 2005)
6.	Corn cob	(El-Hendawy et al., 2001)
7.	Coconut shell	(Tsamba et al., 2006)
8.	Pecan shell	(Cheng et al., 2010)
9.	Rice straw	(Oh & Park, 2002)
10.	Macadamia nutshell	(Oh & Park, 2002)
11.	Fruit stones & nutshell (hazelnut shells, peanut hulls)	(Aygün et al., 2003)
12.	Apricot stones	(Youssef et al., 2005)
13.	Almond shell	(Marcilla et al., 2000)
14.	Grain of sorghum	(Diao et al., 2002)
15.	Cedar wood	(López de Letona Sánchez et al., 2006)
16.	Date stone	(M. Danish et al., 2010)
17.	Acacia mangium wood	(Mohammed Danish et al., 2014)
18.	Pomegranate seeds	(Uçar et al., 2009)
19.	Neem leaves	(Babu & Gupta, 2008)
20.	Palm oil shell waste	(Foo & Hameed, 2009)
21.	Wheat straw	(Dang et al., 2009)
22.	Rice husk ash	(Naiya et al., 2009)
23.	Coconut coir dust	(Macedo et al., 2006)
24.	Natural condensed tannin	(Zhan & Zhao, 2003)
25.	Vetiver roots	(Altenor et al., 2009)

Activated carbon has several important uses including refining (such as in cleaning cane, beets and corn sugar solutions), eliminating flavors and odors from domestic and industrial water supplies, vegetable and

animal fats and oils, alcoholic beverages, chemicals and pharmaceuticals and in water treatment waste (Kumar et al., 2009). Chemical activation methods are widely used to produce activated carbon from carbon-containing materials. Chemicals that can be used as activators are acids, bases and salts including  $H_3PO_4$ , NaOH,  $ZnCl_2$  (Emmy Sahara, 2017). Chemical agents help in develop activated carbon porosity by means of dehydration and degradation (Yorgun & Yildiz, 2015). It is well-known that activated-carbon quality is influenced by some factors such as the nature of raw material, the method of activation, the activating agent and the conditions of the carbonization process.

An earlier research (Nae-Wen & Wu, 2003) reported about processing of coffee husk waste using a rotary reactor with a temperature of 800°C within 1 hour 30 minutes to 4 hours 30 minutes and the activator used with concentrations between 10 to 40%. Later, an investigation (Kurniawan et al., 2006), showed the performance of absorbents from various agricultural wastes in adsorbing heavy metals in the form of (Cd (II), Cr (III), Cr (VI), Cu (II), Ni (II) and Zn (II)) from water metal contaminated waste. Agricultural waste used is candlenut skin, orange peel, soybean skin and jackfruit. Agricultural waste shows extraordinary ability to remove heavy metals (Cr (VI): 170 mg/g activated carbon nutmeg bark; Ni (II): 158 mg/g orange peel; Cu (II): 154.9 mg/g soybean peels treated with NaOH and citric acid; Cd (II): 52.08 mg/g jackfruit), compared to activated carbon (Cd (II): 146 mg/g; Cr (VI): 145 mg/g, Cr (III) ): 30 mg/g; Zn (II): 20 mg/g).

Recently, development of activated carbon from coffee husk was carried-out through hydrothermal carbonization under low temperature conditions (180 °C) and followed by chemical activation with KOH (Tran et al., 2020). The absorption capacity was analyzed with methylene blue solution. This study showed that activated hydrocarbon (AH) had a larger specific surface area ( $862.2 \text{ m}^2\text{g}^{-1}$ ) than its carbon precursor ( $33.7 \text{ m}^2\text{g}^{-1}$ ). The maximum methylene blue absorption capacity is 415.8 mg/g. This finding indicates that coffee agro-industry residues are potential raw materials for activated carbon. More investigation is needed to explore the potency of others kind of biomass from coffee plantation.

This study mainly focused on the evaluation of carbonization process of Robusta Coffee

Skin (*Coffea canephora*) and activation procedures in relationship with the resulting textures, adsorption capacity and chemical composition. The raw material that has been used in this investigation was the exocarp and mesocarp parts of the Robusta coffee fruit. Based on literatures that have been reviewed, the use of coffee peels as raw material for activated carbon is still very limited. Previous studies used a relatively high carbonization process and a high activator concentration. In this study, activated carbon production involved a pilot plant pyrolysis reactor at medium temperatures, i.e. 400-500 °C. The use of chemicals as activators was kept low in order to develop a low-cost synthesis procedure.

## 2. Methodology

### 2.1. Materials

Robusta Coffee skin used in this study was obtained from coffee Plantation in Bener Meriah, Aceh. Experimental procedure carried-out in this investigation mainly consists of two stages. First stage was bio-char preparation via pyrolysis process in a pilot-scale reactor. The second was chemical activation of bio-char using two activator agents. Resulting activated carbon samples were then tested under iodine adsorption method and SEM-EDS analysis.

### 2.2. Active Carbon Preparation

At this stage, the dry Robusta coffee skin was processed with a slow pyrolysis reactor. This process was carried out using a set-up described in Fig. 1 at maximum temperatures

of 400 °C and 500°C. About 3 kg of dried coffee-skin was placed in this reactor and heated for three hours. When the reactor was cooled down to room temperature, the resulting bio-char was collected and grounded using a mortar and pestle. Bio-char sample was then sieved to 50 mesh size.

The next step was bio-char chemical activation using 1%, 1.5%, 2% NaOH solutions and 10%, 15% and 20% ZnCl<sub>2</sub> solutions. 10 g of each bio-char carbonized at 400 °C was placed into three glass beakers. Then, 100 ml of NaOH (Sodium Hydroxide) at a concentration of 1%, 1.5% and 2% was added into each glass beaker. The mixture was then left for 24 hours then filtered and rinsed with distilled water until it reached a neutral pH. The sample was then heated in an oven at 105 °C for 30 minutes to dry. The activated carbon was then cooled to room temperature and weighed. Similar activation process was also carried out for bio-char which was carbonized at 500 °C. Another set of activated carbon was prepared using chemical activator ZnCl<sub>2</sub> concentration of 10%, 15% and 20%. Table 2 lists sample ID and brief information of each sample.

$$(m/v) (\%) = \left(\frac{m}{v}\right) \times 100\% \quad (1)$$

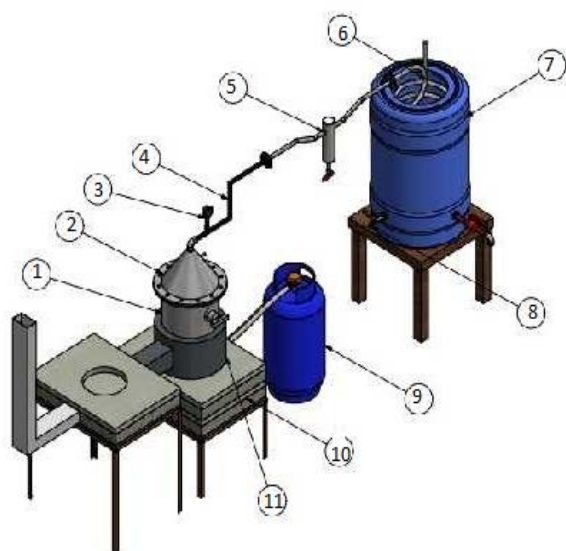
Keterangan:

(m/v) (%) : persen massa per volume

m : massa zat terlarut

v : volume zat terlarut

For example, calculation of concentration of 10% ZnCl<sub>2</sub>, 10 grams of mass of ZnCl<sub>2</sub> is divided by 100 ml of solution, then multiplied by 100%.



Remarks:

1. Pyrolysis Tube;
2. Cover the pyrolysis tube;
3. Pressure gauge;
4. Distribution pipe;
5. Tar container;
6. Condenser spiral pipe;
7. Drum;
8. Liquid smoke valve;
9. Gas cylinders;
10. Fireplace;
11. Pyrolysis tube cover

**Figure 1.** Set Up the Robusta Coffee Slow Pyrolysis Skin reactor

**Table 2.** Robusta coffee skin sample ID

No	Sample ID	Information
1.	CP Raw	Raw coffee skin that has been dried in the sun
2.	CPBC400	Coffee skin with slow pyrolysis method preparation with a temperature of 400°C
3.	CPBC500	Coffee skin with slow pyrolysis method preparation with a temperature of 500°C
4.	CP400A2	Coffee husk with slow pyrolysis method preparation with a temperature of 400°C and chemical activation using NaOH concentration of 2%(m/v).
5.	CP400B10	Coffee husk with slow pyrolysis method preparation with a temperature of 400°C and chemical activation using ZnCl <sub>2</sub> concentration of 10%(m/v).
6.	CP500A2	Coffee husk with slow pyrolysis method preparation with a temperature of 500°C and chemical activation using a concentration of NaOH 2%(m/v).
7.	CP500B10	Coffee husk with slow pyrolysis method preparation with a temperature of 500°C and chemical activation using ZnCl <sub>2</sub> concentration of 10%(m/v).
8.	CP500B15	Coffee husk with slow pyrolysis method preparation with a temperature of 500°C and chemical activation using ZnCl <sub>2</sub> concentration of 15%(m/v).
9.	CP500B20	Coffee husk with slow pyrolysis method preparation with a temperature of 500°C and chemical activation using ZnCl <sub>2</sub> concentration of 20%(m/v).

### 2.3. Characterization of Active Carbon

Topography and pore structures of activated carbon were analyzed by backscattered electron detector (BSE) of JEOL JSM-6510 Series Scanning Electron Microscope. Surface chemical composition was analyzed by JED-2300 X-ray dispersive energy spectroscopy.

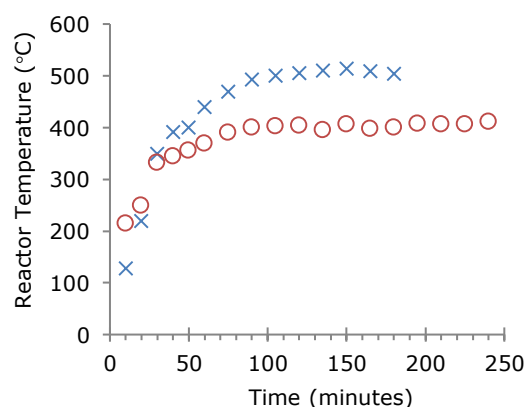
### 2.4. Adsorption Experiment

Adsorption properties of activated carbon was performed by adsorbing iodine solution. One gram of activated carbon was initially heated in oven at 110 °C for 3 hours, and then cooled in a desiccator. Subsequently, 50 ml of 0.1 N iodine solution was added and stirred with a magnetic stirrer for 15 minutes. After mixture filtration, 10 mL of filtrate was taken and titrated with 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution until the yellow color is reduced. A few drops of 1% amilum (C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>) were added and titrated again until the solution was colorless. Titration was also carried out for blank solutions, such as titration of iodine solutions without the addition of activated carbon (Idrus et al., 2013).

## 3. Result and Discussion

### 3.1. Coffee Skin Pyrolysis and Activation

Carbonization of 3 kg of coffee-skin was performed at maximum temperatures of 400 °C and 500°C. Fig. 2 displays temperature profiles recorded during this process. The temperature was recorded after 10 minutes heating-up. The process was stopped when no pressure built-up in the reactor as well as no more pyrolytic oil dropped down after condenser.



**Figure 2.** Reactor temperature profiles recorded during slow pyrolysis process at temperatures of ○ = 400 °C and × = 500 °C

Temperature profile of 400 °C pyrolysis process as displayed in Fig. 2 suggested a heating ramp of 2.3 °C/ min. A maximum temperature of 400 °C was reached after 90 minutes of heating using LPG burner. Carbonization temperature was then kept at about 400 °C by adjusting LPG burner regulator. The process was stopped after 240 minutes at a final temperature of 412°C.

Fig. 2 shows a slightly higher temperature ramp during carbonization process at 500 °C. Targeted temperature of reaction was reached after 105 minutes. This revealed that the heating rate applied during the process was 3.9 °C/ min. Indeed, a shorter processing time was experienced during pyrolysis of coffee-skin at 500 °C where 3 kg of coffee-skin was pyrolyzed within 180 minutes reaching a final temperature of 504°C. It is noteworthy that under our pilot-plant set-up condition, carbonization of coffee-skin at higher temperature reduced processing time significantly.

Carbonization of coffee-pulp via pyrolysis process produced three products namely biochar, bio-oil, and un-condensable gas. Table 3 provides mass of products of Robusta Coffee-skin pyrolysis at the temperatures of 400 °C and 500°C. This data indicates that the carbonization temperature affects slightly on the production of biochar.

**Table 3.** Robusta coffee skin pyrolysis product

Products	Pyrolysis Temperature	
	400 °C	500 °C
Biochar (g)	1286	1228
Bio-oil (g)	784	673
Un-condensable gas (g)	balanced	balanced

For activation process, two chemical activators *i.e.* NaOH and ZnCl<sub>2</sub> were used at different concentrations. Initially, NaOH activators were prepared at concentrations of 1 %, 1.5 %, and 2 %. ZnCl<sub>2</sub> activators were then prepared at concentrations of 10%, 15% and 20%. Differentt activators were prepared in this experiment in order to find the best procedure for activating the biochar. The activation process, is an important aspect in preparing activated carbon. The activation process, significantly increased the absorption capacity. This happened due to impurities covering the carbon pores were removed (via evaporation) as the activation temperature increased. The higher the activator concentration the larger the pores of activated carbon (Heo & Park, 2015).

### 3.2. Adsorption Test of Active Carbon

After chemical activation process, the resulting activated carbon was then tested for its absorption capacity by using iodine adsorption method. Table 4 displays the data of iodine adsorption test for all activated carbon prepared for this investigation. In

general, all samples exhibit significant absorption capability.

**Table 4.** Iodine absorption

No	Sample ID	Volume Titration (ml)	Iod absorption (mg/g)
1.	CP400A2	3,4	704,9
2.	CP400B10	4,9	609,8
3.	CP500A2	3,2	720,2
4.	CP500B10	5	605,3
5.	CP500B15	8,5	383,9
6.	CP500B20	10,9	231,6

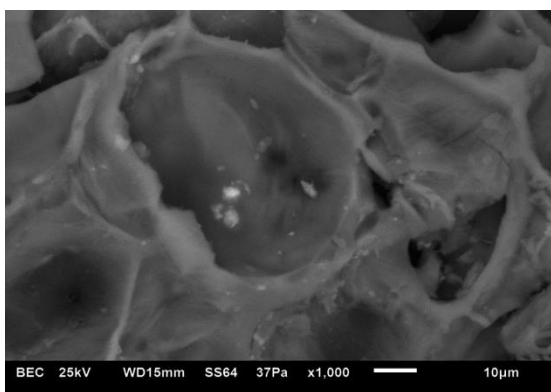
Basically, the adsorption power of activated carbon to iodine correlates well with the surface area of activated carbon. The greater the iodine number, the greater its ability to adsorb or solutes (Maulinda et al., 2017). Common method used in the analysis of activated carbon adsorption power of iodine solution is the iodometric titration method. The reactivity of activated carbon can be seen from its ability to adsorb substrates. The adsorption power can be shown by the amount of iodine which is a number that shows how much the adsorbent can adsorb iodine. The greater the value of the iod number, the greater the adsorption power of the adsorbent. The addition of iodine solution functions as an adsorbate which will be absorbed by activated carbon as the adsorbent. The absorption of iodine solution is indicated by the reduction in the concentration of iodine solution. Measurement of residual iodine concentration can be done by titrating the iodine solution with sodium triosulfate 0.1 N and the indicator used is starch.

In Table 4, iod adsorption per gram of sample is shown. The difference in the amount of iod adsorbed was due to the provision of different concentrations in each activator used. The increase in values occurred as a result of more impurities being released from the surface of activated carbon. As the temperature and activator concentration increased, impurities which were initially found in the pore and cover the pore, came apart or evaporated so that they expanded the surface of activated carbon. The greater the surface area of activated carbon, the greater the ability of activated carbon adsorption. Iodine numbers obtained in this experiment meets the Indonesian National standards (SNI No. 06-3730-1995).

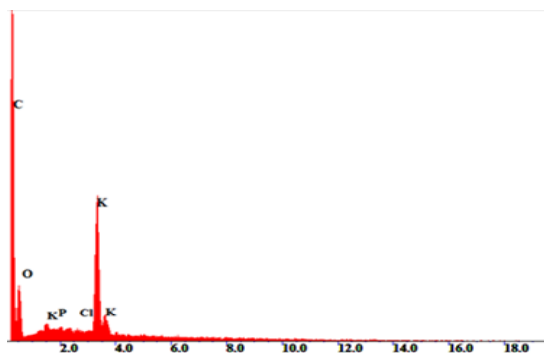
### 3.3. Morphology and Elemental Compound of Activated-Carbon

Observation on the topography and morphology of activated-carbon samples were performed under SEM analysis. The surface characteristics, the texture and pore size were captured simultaneously. EDS analysis was then continued in order to find-out the surface composition of each samples.

At beginning of SEM and EDS analysis, the surface texture and topography of biochar samples namely CPBC 400 and CPBC 500 were examined. Later, samples of activated carbon which show higher absorption value were analyzed in order to compare the effectiveness of both NaOH and ZnCl<sub>2</sub> activators.



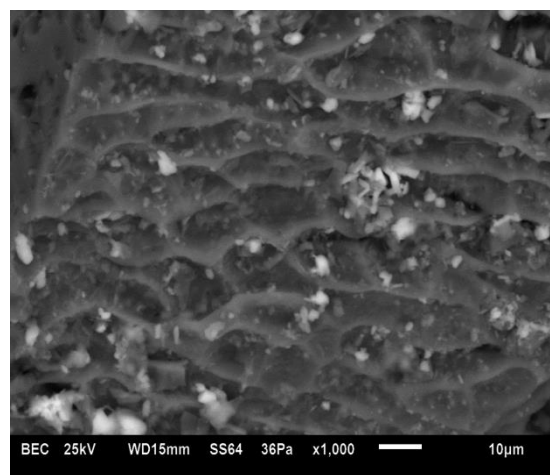
**Figure 3.** CPBC400 sample with a magnification of 1000 times



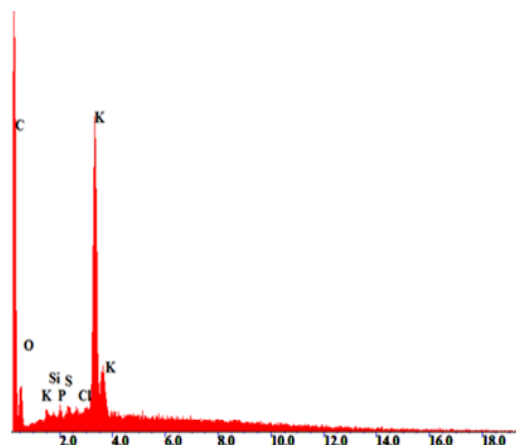
**Figure 4.** CPBC400 sample spektrum

Fig. 3 shows SEM image of CPBC400 sample with a magnification of 1000 times. It can be seen that the pores of the activated carbon were still dense causing the char unable to absorb optimally. EDS spectra of biochar carbonized at 400 °C are shown in Fig. 4. This spectrum indicated that the value of the carbon element was still mixed with many other impure elements such as the potassium.

The surface topography captured from CPBC400 sample were not much different compared to the surface of CP500 sample as shown in Fig. 5. However, EDS spectra of CP500 sample as shown in Fig. 6 had a greater potassium content, which was most likely related to the higher pyrolysis temperature. Increasing pyrolysis temperature leads to an increase in potassium content of activated carbon. The rise in temperature can speed up the reaction and cause the molecules to react and move faster. Increasing temperature also expands the surface of the particles of substances which facilitates the reaction between one substance and another substance (Maulinda et al., 2017).



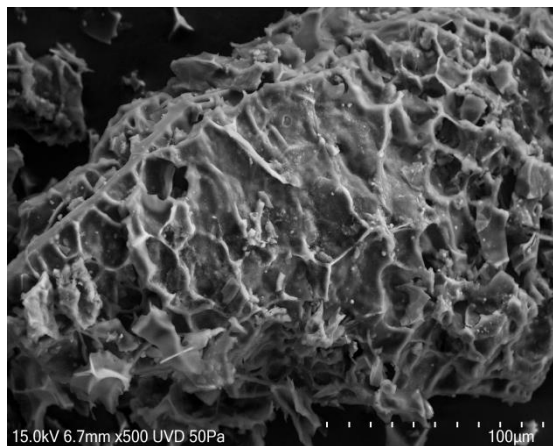
**Figure 5.** CP500 sample with magnification 1000 times



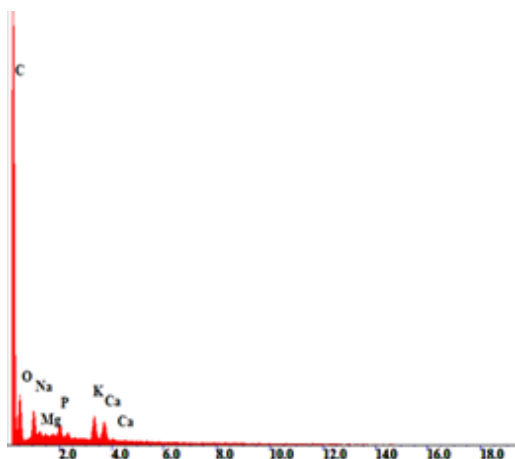
**Figure 6.** CP500 sample spektrum

In Fig. 7, SEM image of CP500A2 samples with 500 times magnification are shown. It can be seen clearly that the pores of activated carbon have been opened. This indicates that biochar has been successfully activated and this sample demonstrated a high absorption

of 720.2 mg/g as shown in Table 3. Interestingly, EDS spectra in Fig. 8 shows that the carbon element was very large and other impurities were very small. This suggests that activation procedure applied for this sample could manage to reduce the levels of potassium in CPBC500 sample as shown in Fig. 8. A higher concentration of chemical activator, and pyrolysis temperature will increase the pores and absorption of activated carbon (Md Arshad et al., 2016).

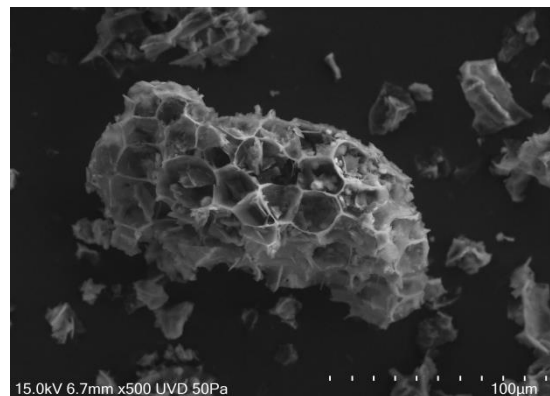


**Figure 7.** CP500A2 samples with 500 times magnification.

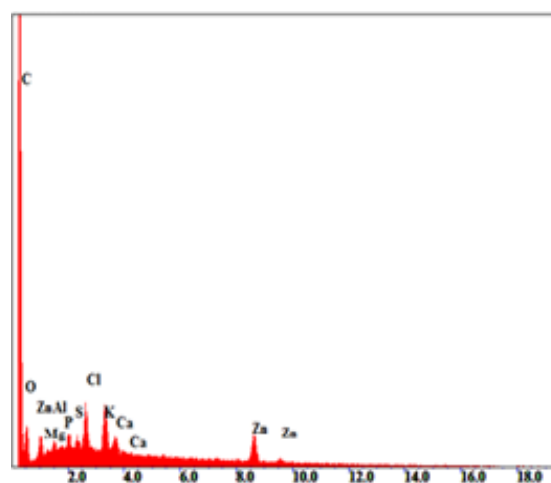


**Figure 8.** CP500A2 sample spectrum

Fig. 9 displays SEM image of CP500B10 sample captured at a magnification of 500 times. It can be seen that the CP500B10 sample had large structure and pores indicating a successful activation. This sample has been shown to have a high absorption value as indicated in Table 3. EDS spectra in Fig. 10 also indicates a high level of carbon element, but other impurities are very small. It also demonstrates that there were still elements of Zn and Cl which have not been completely decomposed from the activated carbon.



**Figure 9.** CP500B10 sample with a magnification of 500 times



**Figure 10.** CP500B10 sample spectrum

#### 4. Conclusion

Evaluation of carbonization process of Robusta Coffee Skin (*Coffea canephora*) and activation procedures have been carried-out by preparing the samples under two carbonization protocols and six chemical activation procedures. The highest iodine absorption value was observed from coffee-skin pyrolyzed at 500 °C followed by chemical activation using 2% of NaOH. SEM images confirmed that the shape and the structure of the pores enlarged after chemical activation. EDX spectrum indicates that C element increased and the other binding components decreased. Overall, this investigation has shown a potential utilization of Robusta Coffee Skin as a raw material for preparation of activated carbon which can be used further as adsorbent material with high absorption capacity.

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