Effect of carbonisation temperature and activating agents on the characteristics of activated carbon produced from oil palm empty fruit bunch

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Abstract. This study aimed to analyse the effect of carbonisation temperature and type of activating agent which were best to be used in the chemical activation process in the manufacture of activated carbon from oil palm empty fruit bunches (EFB). The process of making activated carbon consisted of three stages, i.e. dehydration, carbonisation, and activation process. The experimental design was a randomised block design arranged as factorial with two factors, i.e. the first factor was variations of carbonisation temperature: 300 °C, 400 °C, and 500 °C; the second factor was the variations of activating agent type: ZnCl₂, CaCl₂, CH₃COOH, and without activation. The results showed that carbonisation temperature and the type of activating agent had a significant effect on the characteristics of the activated carbon. The best results were achieved using CH₃COOH as the activating agent at 500 °C. The characteristics of the best-activated carbon consisted of 5.78% of ash content, 19.84% volatile matter content, 74.39% fixed carbon content, 1007.320 mg/g adsorption of iodine solution. The Brunauer–Emmett–Teller (BET) surface areas were up to 1110.87 m²/g and had a hollow surface structure, and open pores with a weight percentage atoms component of carbon reached 77.132%.

1. Introduction
Santika [1] states that Indonesia is a country that has the most extensive palm oil plantations in the world. The development of the vegetable oil industries strongly relies on the supply of palm oil. This has led to the continued development of oil palm plantations in Indonesia. In the process of processing palm oil into oil, a by-product in the form of palm empty fruit bunches (EFB) is also produced [2-4]. So far, EFB is mainly used as fertiliser through the composting process [5]. EFB has a high cellulose content [6], which consists of 66.07% holo-cellulose and 37.76% α-cellulose with a fibre content of 72.67%. The biomass is abundant with carbon. The presence of the carbon makes EFB has excellent potential as a raw material for the manufacture of activated carbon [7]. The carbon source is beneficial in many bio-applications [8].
Activated carbon is a porous solid material with a carbon content reaching 95%. The world demand for activated carbon for the industry is still relatively high due to the increasingly widespread use of activated carbon in the industrial sector [9]. Activated carbon has many applications [10,11]. In all these applications, activated carbon is most often used as an adsorbent. Adsorption is an essential property of activated carbon. Activated carbon can have adsorption properties because it is rich in pores.

The production process of activated carbon consists of three stages, i.e. dehydration, carbonisation, and activation [12]. The dehydration phase aims to remove the water content. The carbonisation phase is for the decomposition of volatile organic components. The last stage is the activation process which is carried out chemically and physically to generate pores in the carbon so that the surface area and adsorption capacity of the activated carbon can be significantly increased. According to Jiang [13], the absorption capacity of activated carbon can be enhanced with the addition of the activating agent, which is also affected the type of activating agent used. The manufacture of activated carbon by chemical activation has been carried out by Gonzalez [14] and Hendrawan [15] in the manufacture of activated carbon-based on corn pericarp and Sengon woods. In their study, the results showed that the type of activating agent in the chemical activation process significantly affected the physical properties of activated carbon, including water content, ash content, and absorption of iodine solution.

The quality of activated carbon as an adsorbent can be determined in terms of the ash content, the content of the volatile substances, and the level of pure activated carbon [16]. The adsorption capacity of activated carbon can be determined with respect to iodine number. Brunauer, Emmett, and Teller (BET) can be used to explain the physical adsorption of the specific surface area of materials [17]. Morphology and elemental compositions of activated carbon can be investigated using scanning electron microscopy coupled with elemental dispersive X-ray spectroscopy (SEM-EDX). The purpose of this study is to study the effect of chemical activating agents (ZnCl$_2$, CaCl$_2$, and CH$_3$COOH) carbonisation temperature on the characteristics of activated carbon EFB.

2. Materials and Methods

The materials used in this study included: the main ingredient, i.e. Dura type EFB from solid waste of the processed palm oil industry at PT Sawit Arum Madani, Blitar, Indonesia. Chemicals as activating agents include ZnCl$_2$, CaCl$_2$, and CH$_3$COOH. Iodine number test material: 0.1 N iodine solution; 0.1N sodium thiosulfate solution; and 1% starch solution. Equipment used included: Oven MMM MED Center - Ecocell 55 55 L capacity to remove moisture from the material. Heraeus furnace 1200 W capacity of 9 L as a place for carbonisation and ash material. Hitachi Quantax70 SEM-EDX device to determine the morphology and composition of the active carbon.

The experimental parameters include carbonisation temperature (300, 400, and 500 °C) and types of the activating agent (ZnCl$_2$, CaCl$_2$, and CH$_3$COOH). The tests include ash content, volatile matter, fixed carbon content, iodine absorption according to the BET surface area of activated carbon [18, 19]. The best conditions of carbonisation temperature and types of the activating agent were carried out morphological imaging and components of activated carbon using SEM-EDX.

In the preparation of EFB material, the oil palm bunches were first cleaned and then dried. EFB material was cut into particles of 1-3 cm. The next process was the manufacture of activated carbon, including dehydration, carbonisation, and activation. Dehydration stage was a stage that aims to remove the moisture content contained in the EFB sample preparation results. The dehydration process was carried out at 110 °C using an oven for four hours or until the weight of the EFB sample became constant. The carbonisation phase was carried out using a furnace at 300, 400, and 500 °C for 10 minutes, respectively. The carbon was then ground. The result of carbon powder was sieved into 100 mesh. The chemical activation process used a aqueous solution of ZnCl$_2$, 10% (w/v), 10% CaCl$_2$ (w/v), and 60% CH$_3$COOH (v/v). Comparison of the mass ratio of the activating agent solution to the carbon mass of 3:1.

The parameters measured in this study included ash content, volatile matter, fixed carbon, and absorption of iodine solution according to the calculated BET surface area of the activated carbon
The ash content value was determined via equation 1. The volatile matter was calculated through equation 2. The fixed carbon was measured using equation 3. The absorption value of the iodine number was measured by equation 4. Calculation of BET used the following equations [21]:

$$\text{Ash content}(\%) = \frac{b}{a} \times 100\%$$  \hspace{1cm} (1)

$$\text{Volatile matter}(\%) = \frac{e-d}{c} \times 100\%$$  \hspace{1cm} (2)

$$\text{Fix carbon}(\%) = 100\% - (e + f)$$  \hspace{1cm} (3)

$$\text{IAN} = \frac{(10-\left(\frac{8x5}{D}\right)) \times 12.693 \times 2.5}{W}$$  \hspace{1cm} (4)

$$\text{BET} \left(\frac{m^2}{g}\right) = \frac{\text{IAN}}{538.9} \times 594.3$$  \hspace{1cm} (5)

where: $a =$ initial mass of the sample (g); $b =$ final mass of the sample (g); $c =$ initial mass of the sample before heating (g); $d =$ final mass of sample after heating (g); $e =$ volatile matter (%); $f =$ ash content (%); $\text{IAN} =$ iodine number (mg iodine / g activated carbon); $B =$ volume of total sodium thiosulfate used in titration (ml); $C =$ normality of sodium thiosulfate (N); $D =$ iodine normality (N); $W =$ mass of activated carbon (g); $12.693 =$ the amount of iodine corresponding to 1 ml of 0.1 N sodium thiosulfate solution; $538.9 =$ the results of iodine numbers (mg iodine / g activated carbon) [21]; $594.3 =$ BET surface area results (m$^2$/g) [21].

3. Results and Discussion

Figure 1 shows ash content values in the range of 3.73-16.44%. The lowest ash content of 3.73% was obtained at a carbonisation temperature of 300 °C using a CH$_3$COOH (acetic acid) solution as an activation agent. The highest ash content of 16.44% was obtained at a carbonisation temperature of 500 °C without a chemical activation process. The carbonisation temperature factor showed a significant effect on the ash content of activated carbon. The carbonisation temperature factor had a very significant effect on 500, 400, and 300 °C.

![Figure 1](image1.png)

**Figure 1.** Effect of carbonisation temperature and type of activating agent on ash content.

Figure 2 shows the value of volatile matter which was in the range of 17.22–35.52%. The lowest volatile matter was obtained at 500 °C using zinc chloride as an activating agent, and the highest volatile matter was obtained at 300 °C without a chemical activation process. The carbonisation temperature factor gave a significant effect on activated carbon volatile matter. Based on Duncan’s advance test, the carbonisation temperature factor had a very significant effect on 500, 400, and 300 °C.
°C. The activating agent type had a very significant effect on ZnCl₂, CaCl₂, CH₃COOH, and in the control treatment or without activation.

Figure 3 shows the fixed carbon value, which was between 51.97 and 74.39%. The highest fixed carbon was obtained at 500 °C with activation using an acetic acid activator, and the lowest fixed carbon was obtained at the 300 °C without a chemical activation process. The effect of the carbonisation temperature factor gave a very significant effect on fix carbon. Based on Duncan’s advance test, the carbonisation temperature treatment had a very significant effect on 300, 400, and 500 °C. The activating agent type had a very significant effect on the control treatment, ZnCl₂, CaCl₂, and CH₃COOH. The high levels of fixed carbon using acidic activator compounds were due to the lignocellulosic material contained in EFB, which had a high oxygen content and the acidic activating agent reacts with oxygen-containing functional groups [22].

![Figure 2](image1.png)

**Figure 2.** Effect of carbonisation temperature and type of activating agent on volatile matter.

![Figure 3](image2.png)

**Figure 3.** Effect of carbonisation temperature and type of activating agent on fix carbon.

Figure 4 shows the iodine number, which was between 763.803–1007.320 mg/g. The highest iodine number was obtained at a 500 °C carbonisation temperature treatment by activating agents using acetic acid. In contrast, the lowest iodine absorption was obtained at a 300 °C without a chemical activation process. Based on Duncan’s advance test, the carbonisation temperature factor had a very significant effect on 300 °C and 400 °C and had a significant effect at 500 °C. The activating agent type factor results in a very significant effect on the control treatment but had no significant effect on the types of activating agents ZnCl₂, CaCl₂, and CH₃COOH.

Figure 5 shows the BET surface area values, which were between 842.33–1110.87 m²/g. The highest BET surface area was obtained at a 500 °C carbonisation temperature treatment by activation using acetic acid. In contrast, the lowest BET surface area was obtained at a 300 °C without a chemical activation process. Based on Duncan’s advance test, the carbonisation temperature factor had a very significant effect on 300 °C and 400 °C and had a significant effect at 500 °C.
The activating agent type resulted in a significant effect on the BET surface area, the activating agent type factor had a very significant effect on the control treatment, but no significant effect on the types of activating agents ZnCl₂, CaCl₂, and CH₃COOH. Based on the calculation of BET surface area, using acetic acid and carbonisation temperature of 500 °C gave the highest BET surface area of 1110.87 m²/g. The chemical activation process with variations in the type of activating agent can increase the surface area value of activated carbon BET.

![Figure 4](image.jpg)

**Figure 4.** Effect of carbonisation temperature and type of activating agent on iodine number.

![Figure 5](image.jpg)

**Figure 5.** Effect of carbonisation temperature and type of activating agent on the BET surface area

The determination of the best treatment in this study was obtained using the multiple attribute method. The best treatment results were the treatment of a carbonisation temperature of 500 °C with a type of activating agent of CH₃COOH. The results of the treatment had 5.78% ash content, 19.84% volatile matter, 74.39% carbon fix, iodine solution absorption of 1007.320 mg/g, and BET surface area of 1110.87 m²/g.

SEM imaging was carried out to study the surface structure of the adsorbent, especially the pores, formed [23]. Figures 6 (a), (b), and (c) show the structure of activated carbon obtained from carbonisation temperature of 500 °C without using an activating agent. It can be seen that the sample had a hollow structure or pores but was still covered with various impurities. Figures 6 (d), (e), and (f) show the structure of activated carbon obtained from the carbonisation temperature of 500 °C with activation using activating agent CH₃COOH. The sample had large hollow structures and open pores and fewer impurities compared to Figure 6 (a), (b), and (c). The pores or cavities in Figure 6 (d), (e), and (f) had various pore diameters, i.e. mesopore and macro-pore.

Table 1 shows the EDX analysis of the activated carbon obtained at 500 °C with/without activating agent. It was found the activated carbon without chemical activation processes had impurities such as
potassium, silica, and calcium as well as chlorine ions that attach to or cover the cavity and pore surfaces of activated carbon. The sample treated by CH$_3$COOH had few impurities.

**Table 1.** EDX elemental composition analysis of EFB activated carbon.

| Elemental composition | A (%weight) | B (%weight) |
|-----------------------|------------|-------------|
| Carbon                | 71.685     | 77.132      |
| Oxygen                | 19.091     | 21.856      |
| Magnesium             | 0.404      | -           |
| Silica                | 1.918      | 0.720       |
| Chlorine              | 0.814      | -           |
| Potassium             | 5.577      | 0.292       |
| Calcium               | 0.510      | -           |

A) Treated at carbonization temperature of 500 °C without activation  
B) Treated at carbonization temperature of 500 °C with activating agent CH$_3$COOH

**Figure 6.** SEM images of activated carbon temperature control carbonization 500 °C: (a) magnification 1000X; (b) magnification 1500X; (c) magnification 2000X and activated carbon temperature 500 °C with activating agent CH$_3$COOH; (d) magnification 1000X; (e) magnification 1500X; (f) magnification 2000X.

**4. Conclusions**

Based on the results of the analysis of variance, it was found that carbonisation temperature and the type of activating agents had a significant effect on the characteristics of the activated carbon. The optimal activation conditions are carbonisation temperature of 500 °C and activating agent of CH$_3$COOH, giving the best characteristics of the activated carbon, i.e. ash content of 5.78%, fix carbon of 74.39%, absorption of iodine solution 1007.320 mg/g, and the BET surface area of 1110.87 m$^2$/g.

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