Preparation and Characterization of Adsorbent from Oil Palm Empty Fruit Bunches Activated with KOH

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Abstract. Preparation and characterization of adsorbent from oil palm empty fruit bunches (OPEFB) activated with KOH has been done. Adsorbent was made from carbonated at 100 °C of oil palm empty fruit bunches and activated by using potassium hydroxide (KOH). The experimental design used was a randomized block design (RBD) which was arranged factorial with 1 (one) factor. The variations in the carbonization temperature of this study were 100 °C, 350 °C, 500 °C, 650 °C and 850 °C. The physico-chemical characteristics of activated OPEFB were evaluated by using Brunauer-Emmet-Teller (BET) surface area, surface morphology using Scanning Electron Microscope (SEM) and Fourier Transform Infrared (FTIR) spectrometer. The best result was obtained in the carbonation treatment at 650 °C. The adsorbent surface area of activated OPEFB is 0.302 m^2/g, the FTIR showed that most of the peaks of functional groups disappeared. The results of the SEM analysis showed that the OPEFB sample micrograph had an irregular shape.

Keywords: Activated · adsorbent · KOH · oil palm empty fruit bunches

1 Introduction

The adsorbent that is often used is in the form of activated carbon and is commonly used for adsorption, such as coconut shells, peat, tree wood, fly ash and other general resources capable of producing activated carbon [1, 2]. Activated carbon has many advantages so it can be categorized as good adsorbents include activated carbon has a very large absorption, i.e. 25–1000% by weight of activated carbon, large surface activated carbon is about 300 m^2/gr up to 2000 m^2/gr [3, 4]. OPEFB from palm oil waste is one of the materials used for activated carbon. The OPEFB consists of Fe, Mg, Na, and K, with the highest potassium content of 463,50 g/kg in the form of an alkaline K_2CO_3 compound, which can absorb FFA [5].

The development of OPEFB as a bio-adsorbent material is an alternative because it has a large surface area, is easy to obtain, and is relatively inexpensive compared to other adsorbents [6]. The adsorbent of OPEFB can be made through carbonation with chemical activation. Chemical activation has advantages such as the required absorption time is shorter, and the adsorption power is relatively short [7]. Multiple activators chemicals

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that can be used, among others H_3PO_4 [8], $ZnCl_2$ [9], H_2SO_4, HCl [10], and KOH [11]. Chemical activation with KOH is used because it can increase micropores with large surface area [12], greater adsorption capacity [13], yield, and microporosity [14].

The study aims of study to preparation and characterization of adsorbent from oil palm empty fruit bunches activated with KOH. Activation and non-activation of OPEFB were carried out with characteristics such as moisture content, ash content, lignin content, cellulose content, and hemicellulose content, and using Fourier Transform Infra-Red (FT-IR), Scanning Electron Microscopy (SEM), and Brunauer Emmett Teller (BET) instruments.

2 Method

1. Materials

   The research materials were oil palm empty fruit bunches (OPEFB) from PTPN IV Jambi and potassium hydroxide 5%.
2. Sample preparation

   Charcoal was burned in a muffle furnace. The sample was divided into two by activated in potassium hydroxide 5% and non-activation procedures after being sifted through a 100-mesh sieve after being burned. Activated is done by immersing the sample with potassium hydroxide 5% for 24 h, followed by oven drying at 105 °C for 4 hours and furnace drying the activated and non-activated samples with temperature variations of 100 °C, 350 °C, 500 °C, 650 °C, 800 °C (Table 1) for 2 h.
3. Characteristics of OPEFB

   a. Determination of water content

      1 g of prepared OPEFB ash is placed in a porcelain exchange and then baked for 1 h at 100 °C. It was then cooled after 30 min in a desiccator, and the moisture content of the OPEFB was calculated. The analysis was carried out three times.

   b. Determination of ash content

      One gram of OPEFB ash that has been prepared and put into a porcelain exchange oven for 3 h at a temperature of 500 °C. Then it was allowed to cool for 30 min in a desiccator, and the ash content was calculated. The analysis was carried out three times.

   c. The concentration of lignin, cellulose, and hemicellulose

      A total of 1 g of OPEFB was oven-dried at 108 °C and then extracted with an alcohol-benzene ratio of 1:2 as much as 200 mL. The extract was added 15 mL of sulfuric acid 72% while stirring for 2–3 min, then covered with a watch glass and soaked for 2 h while stirring and then rinsed with 360 mL of distilled water until the sulfuric acid concentration became 3%, the sample was heated for 4 h at a temperature of 125 °C using reflux as a reverse cooler, then filtered and washed the precipitate until neutral, the precipitate was heated at 105 °C for 1 h and cooled in a desiccator for 15 min and then weighed. The analysis was carried out 3 times repetition.

   d. Chemical characterization was carried out by attenuated total reflectance (ATR)-FTIR using FTIR Perkin Elmer two spectrum in the wavelength range of 4000–400 cm^{-1} to identify the functional groups at the surface of carbon materials.

Table 1. Research design

Temperature °C	A1 (KOH)
T1 100	T1A1
T2 350	T2A1
T3 500	T3A1
T4 650	T4A1
T5 850	T5A1

Table 2. Lignin, Cellulose and Hemicellulose Levels in OPEFB

Content	Activation (%)	Non-Activation (%)
Lignin	26,8	26,7
Cellulose	33,7	33,8
Hemicellulose	17,4	17,7

3 Result and Discussion

3.1 Characteristics of Oil Palm Empty Fruit Bunches (OPEFB)

a. Content of lignin, cellulose, and hemicellulose

Table 2 shows the results of the OPEFB characteristic test to determine the content of lignin, cellulose, and hemicellulose from the treatment that has been carried out. It can be seen that the lignin content of 26,8% activation and 26,7% non-activation, cellulose is 33,7% and non-activation by 33,8% and hemicellulose by 17,4% activation and 17,7% non-activation. The increase in temperature and treatment time will cause the dissolved lignin, cellulose, and hemicellulose content to be more, and the dissolution process at 5% KOH activation and non-activation with OPEFB are perfect. Still, if the treatment time is long enough and the temperature is higher, it will trigger the degradation of lignin compounds. Thus, the appropriate lignin content in OPEFB will facilitate good enzyme performance to decompose cellulose into glucose.

b. Water content

Based on Table 3 shows the results of the OPEFB characteristic test to determine the water content. From the treatment that has been carried out it can be seen that the water content that is close to the standard is the lowest water content, which is shown in the treatment with a temperature of 650 °C with the indicated water content being treated with 5% KOH activation of 0.01 and non-activation of 0.01. The selection of low water content is based on the smaller the water content in OPEFB, the greater the ability of the adsorbent to adsorb adsorbate because of its less content [15].

Table 3. Water Content of OPEFB

Temperature (°C)	Water Content of OPEFB	
	Activation (%)	Non-Activation (%)
100	0.14	0.17
350	0.12	0.13
500	0.09	0.05
650	0.01	0.01
800	0.07	0.06

Table 4. Ash Content of OPEFB

Temperature (°C)	Ash Content of OPEFB	
	Activation (%)	Non-Activation (%)
100	0.098	0.228
350	0.51	0.66
500	0.81	1,305
650	1,919	1.9
800	2.37	2.08

c. Ash content

Based on Table 4 shows the results of the OPEFB characteristic test to determine the ash content. From the treatment that has been carried out it can be seen that the ash content that is close to the standard is the highest ash content, which is shown in the treatment with a temperature of 650 °C with the indicated ash content being treated with 5% KOH activation. of 1,919 and non-activation of 1.9. The selection of high ash content is based on the smaller the ash content in OPEFB, the greater the ability of the adsorbent to adsorbate because of its less content [15].

3.2 FT-IR, BET, and SEM Analysis

a. FT-IR Analysis

Microscopic characteristics were carried out by FT-IR analysis. FT-IR absorption analysis was carried out to determine the functional groups contained in OPEFB before and after treatment [16]. FTIR is a fast, simple, and non-destructive analytical technique where all chemical properties in the sample can be revealed and displayed on the FTIR spectrum [17]. OPEFB fibre has components, namely lignin, cellulose,

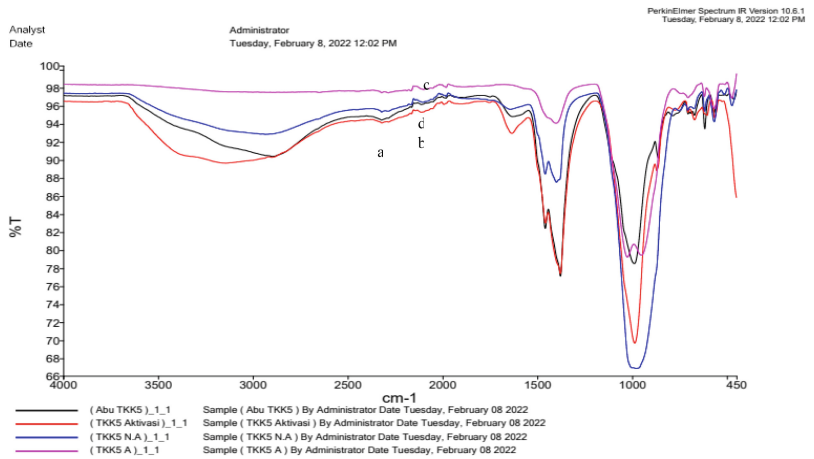

Fig. 1. FTIR Spectrum of OPEFB (a) Activated OPEFB, (b) non-Activated OPEFB, (c) Activated OPEFB 650 °C (d) Non-Activated OPEFB 650 °C

and hemicellulose. The results of the FTIR OPEFB spectrum or graph are shown as follows (Fig. 1).

The FTIR spectra were used to investigate the functional group presence in the raw material OPEFB, activated OPEFB, non-activated OPEFB 650 °C, and activated OPEFB 650 °C. The results of the FTIR spectra analysis of the OPEFB sample showed that there was a wave number of 3172 cm^{-1}, which indicated H_2O molecules (a, b, c) a wave of 2330 cm^{-1}, which included nitrile compounds, a wave of 1648 cm^{-1}, which was identified as carbonate (CO_3) and a wave of 1393 cm^{-1} which included compound -glycosidic bonds and the stretching vibration of C-O at a wave number of 985 cm^{-1}.

The peak at around 1730 cm^{-1} can be seen from the spectra was corresponding to the C=O functional group. The peak at 1635 cm^{-1} corresponded to the bending vibration of the hydroxyl groups of cellulose. The bands at 1730 cm^{-1} was corresponding to the C=O functional group.

b. BET Analysis

BET analysis was carried out to determine the size of the adsorbent surface area and the pore volume contained on the adsorbent surface [18]. The results of the BET test in this study are shown as follows (Figs. 2 and 3).

Based on Figs. 2 and 3, it can be seen that the test results show that the size of the surface area of the activated OPEFB adsorbent is greater than the surface area of the Non-Activated OPEFB adsorbent. It is known that the adsorbent surface area of Activated OPEFB is 0.302 m^2/g, while the surface area of Non-Activated EFB adsorbent is 0,150 m^2/g.

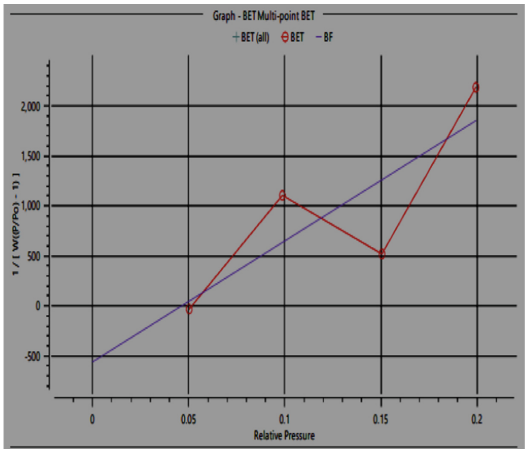

Fig. 2. Activated OPEFB

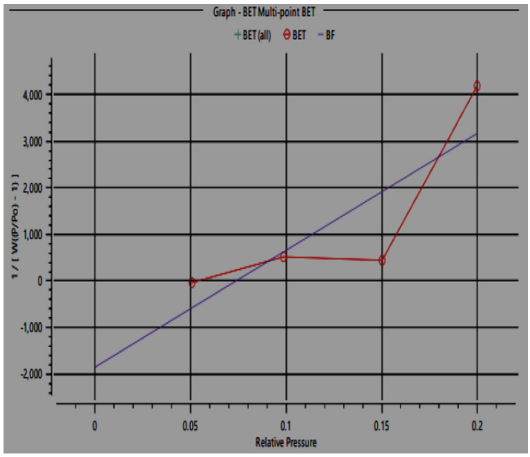

Fig. 3. Non-Activated OPEFB

3.3 SEM Analysis

SEM analysis (SEM-EDX JEOL JSM-6510LA) was conducted to determine the cross-sectional image of the adsorbent surface with magnifications of 500x, 1000x, 1200x, and 1500x. SEM testing was carried out on the treatment of Activated OPEFB, Non-activated OPEFB, 650 °C Activated OPEFB, and 650 °C Non-Activated OPEFB, which were shown as follows:

1) SEM morphological structure of activation OPEFB
2) SEM morphological structure of non-activation OPEFB
3) SEM morphological structure of activation 650 °C OPEFB
4) SEM morphological structure of non-activation 650 °C OPEFB

Based on Figs. 4, 5, 6, and 7, which are the results of the SEM analysis above, the SEM micrograph of the OPEFB sample has an irregular shape. Micrographs were taken for each particle size of 0.859 μm and 2.676 μm with various magnifications. Overall the sample is clearly seen that the surface is uneven, which indicates the presence of quite diverse particle sizes with uneven distribution on the surface and the particles have pores.

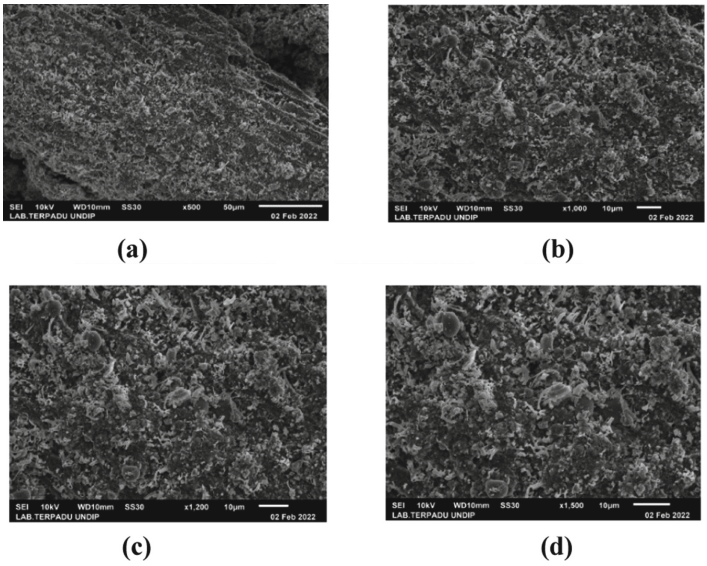

Fig. 4. Results of OPEFB Activation with SEM at magnifications (a) 500x, (b) 1000x, (c) 1200x, and (d) 1500x

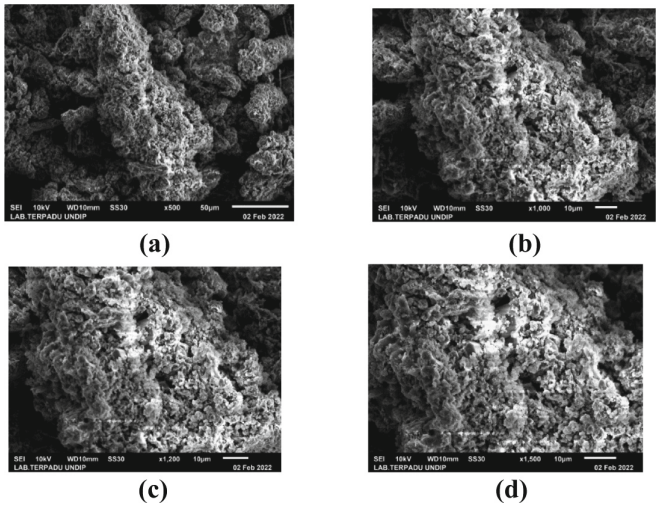

Fig. 5. Results of OPEFB Non-Activation with SEM at magnifications (a) 500x, (b) 1000x, (c) 1200x, and (d) 1500x

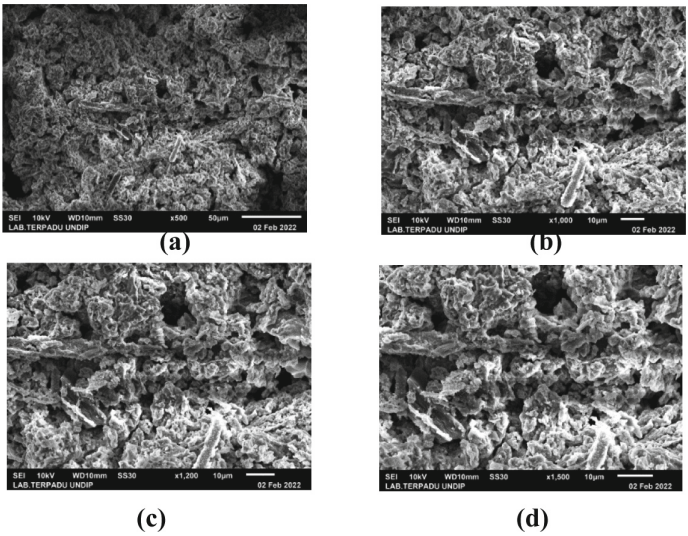

Fig. 6. Results of OPEFB Activation at a temperature of 650 °C with SEM at magnifications (a) 500x, (b) 1000x, (c) 1200x, and (d) 1500x

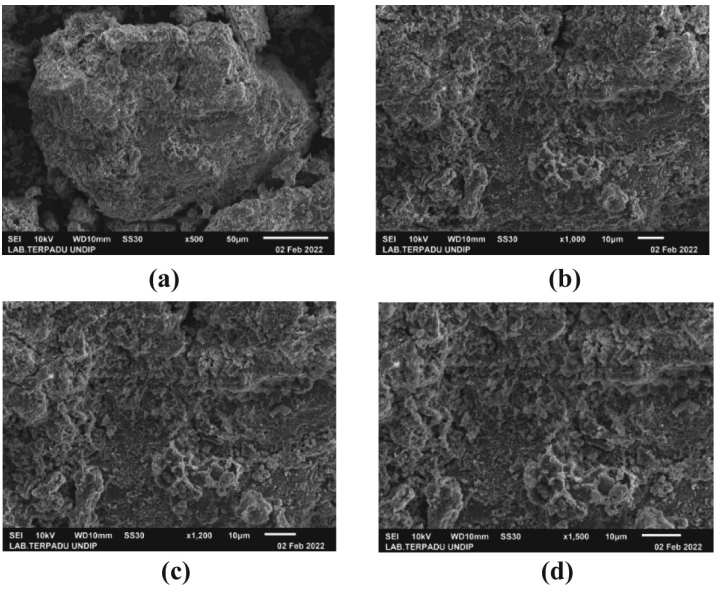

Fig. 7. Results of OPEFB Activation at a temperature of 650 °C with SEM at magnifications (a) 500x, (b) 1000x, (c) 1200x, and (d) 1500x

The activation process aims to enlarge the pores by breaking KOH bonds or oxidizing surface molecules so that KOH changes. Namely, the surface area of OPEFB increases and affects the adsorption power. The structure of the pores that are formed comes from

the evaporation and dissolution of the compounds contained in the raw material caused by the treatment process, which can leave some empty spaces that form pores. While the non-activation process is carried out as a comparison to the activation process where in the non-activation process there is no enlargement of the pores because there is no addition or breakdown of chemical compounds that oxidize molecules, the surface area of OPEFB is denser and does not affect the adsorption power. The structure of the pores is not formed and does not leave some space that forms the pores [19].

4 Conclusion

The results of the FTIR analysis showed that at a wave number of 1648 cm^{-1}, which was identified as carbonate (CO_3), the results of the BET analysis showed that the surface area of the activated OPEFB adsorbent was more significant than the surface area of the Non-Activated OPEFB adsorbent. It is known that the adsorbent surface area of Activated OPEFB is 0.302 m^2/g, while the surface area of Non-Activated OPEFB adsorbent is 0.150 m^2/g. The results of the SEM analysis showed that the OPEFB sample micrograph had an irregular shape. Micrographs were taken for each particle size of 0.859 μm and 2.676 μm with various magnifications.

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