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Empty Bunches

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## Characterization of Modified Fe-Cu Nanoparticle Activated Carbon Derived of Oil Palm Empty Bunches

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**Abstract.** We prepared activated carbon from empty oil palm bunches by modifying the metal (Fe and Cu) in the structure of the activated carbon. Empty oil palm bunches obtained in Padang Bulan, Medan, North Sumatera and after washed, it mashed then heated at 500 °C for 1 minutes. For modification, activated carbon was dissolved in a 0.1 M solution of Fe(NO<sub>3</sub>)<sub>2</sub> and Cu(NO<sub>3</sub>)<sub>2</sub> which is heated at 60 °C for 3 hours. Activated carbon without and with modification was then characterized using XRD and SEM / EDX. The XRD results showed slightly change after modification with new peaks appearing and missing peaks. SEM images also show that the activated carbon before modification is irregular in shape, while the activated carbon after modification is more regular and homogeneous. The presence of Fe and Cu on the modified activated carbon is also evidenced by the EDX image, which shows the presence of Fe and Cu peaks when compared to activated carbon before modification which does not show the presence of Fe and Cu metals. It successful to modified Fe and Cu inside the activated carbon of empty oil palm bunches which consider to improve their adsorption properties.

### 1. Introduction

The use of activated carbon synthesized from natural materials is one of the potential alternative solutions in dealing with the problem of clean water availability [1,2]. Activated carbon is generally used as an adsorbent or adsorbent. In the water purification process, activated carbon will adsorb metals such as iron, copper, nickel, lead and can remove odors, colors and tastes in water so that activated carbon can increase the overall purity of water. The use of activated carbon can be maximized by making modifications to activated carbon to increase the pore volume and surface area. One of the modifications to activated carbon is to use metal oxides [2-6].

Metal modification on the surface of activated carbon will increase the reduction and adsorption ability of activated carbon. In the process, metal ions are absorbed on the surface of the activated carbon, then the reducing properties of activated carbon will reduce metal ions to ions in the form of simple or low-valence substances. Due to the strong ability of metal ions to absorb substances, the



adsorption capacity of activated carbon in absorbing increases from before modification [7-10]. Modifications that occur on the surface of activated carbon cause structural changes such as the addition of atoms, the creation of new functional groups and changes in texture. Surface modification greatly affects the adsorption capacity. The adsorption capacity can be determined by many factors, such as, surface (functional groups), porous structure, adsorbate properties and adsorbate / adsorbent interactions. Modifications to the surface of activated carbon affect the chemical properties of the surface, through the reactions that occur in the structure, especially in surface functional groups. Changes in surface properties are based on the results of surface modifications so that they have an impact on the absorption capacity of substances [10,11].

One of the potential ingredients for obtaining activated carbon is oil palm empty bunches. Oil palm empty bunches are the main waste that has not been utilized optimally. According to statistical data, North Sumatra is in the third position of the largest palm oil producer in Indonesia, amounting to 12.02% with a production of 1,758,936 tonnes (Central Statistics Agency and Directorate General of Horticulture, 2017). Based on these data, it indicates that the raw material in the form of oil palm empty bunches is very abundant. Solid waste originating from the oil palm processing process consists of oil palm empty bunches (PEB), shells or shells, fibers or fibers, mud, and cake. Oil palm empty bunches solid waste is the main waste, which is 23% of the oil palm processing process. For each processing of 1 ton of fresh fruit bunches, 22-23% or 220-230 kg of empty oil palm bunches will be produced. Oil palm empty bunches contain 22.2 - 65% cellulose, 19.5 - 38.8% hemicellulose and 10 - 34.37% lignin from dry weight [12-14].

## 2. Experimental

Palm Empty Bunches was taken from Padang Bulan, Medan, North Sumatera. The chemicals used are  $H_3PO_4$ , distilled water,  $Cu(NO_3)_2$ ,  $Fe(NO_3)_2$ ,  $K_2Cr_2O_7$ ,  $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ ,  $H_2SO_4$ , sulfuric acid - silver sulfate reagent, indicator ferroin, Nessler's reagent, HCl, n-hexane,  $HgSO_4$  powder,  $NH_4OH$ , and anhydrous  $Na_2SO_4$ . For biosorbent characterization were used Scanning Electron Microscope (SEM), Energy Dispersive X-ray (EDX), Fourier Transform Infra red (FTIR).

### 2.1. Oil Palm Empty Bunch Preparation.

Total of 100 grams of Palm Empty bunches were collected and then cleaned with distilled water. Then put it in the oven at  $\pm 105^\circ C$  for 24 hours to remove the moisture content. Then grind it with a blender to get a 200 mesh particle size.

### 2.2. PEB Adsorbent Characterization

To determine the structure change of adsorbent after modification was observed by XRD analysis and to analyze the organic content in PEB adsorbent was carried out using Fourier-Transform Infra Red (FTIR). SEM-EDX (Scanning Electron Microscope and Energy Dispersive X-ray) were analyzed to investigate the morphology change of modification adsorbent.

### 2.3. Biosorbent Carbonization

PEB adsorbent prepared sample then insert to the furnace and setup the temperature at  $500^\circ C$  for 2 minutes. After finished the carbonization the sample was cooled to a room temperature.

### 2.4. Activated Carbon Activation

PEB Activated carbon were activated with  $H_3PO_4$  for 24 hours. After immersed for 24 hours, the sample was filtered and neutralized with distilled water until a neutral pH. After neutral, insert to oven at  $105^\circ C$  for 1 hour.

### 2.5. Modification of Activated Carbon with Fe-Cu Metal

15 grams activated carbon was put into 150 mL (1: 1)  $Fe(NO_3)_2$  and  $Cu(NO_3)_2$  0.1 M. After that, it is heated to  $60^\circ C$  for 3 hours. Cooled, filtered, and oven at  $105^\circ C$  for 24 hours.

### 2.6. Characterization of Activated Carbon Fe-Cu

The final characterization of activated carbon of palm empty bunches modified with Fe-Cu was carried out using XRD (X-ray Diffractometer) to determine the structure of palm empty bunches after modification, determine the surface area with BET (Brunauer-Emmett-Teller), and morphological analysis by SEM- EDX (Scanning Electron Microscope and Energy Dispersive X-ray).

### 3. Results and Discussion

EDX analysis results on palm empty fruit bunch bio-sorbent shows the amount of impurities that may come from fertilizers or insecticides during the cultivation process in oil palm plantations or due to the storage process in open air. EDX analysis indicate the several impurity metals were observed, such as K, Ca, Mg, Si and P. To obtain pure bio-sorbents, purification was carried out with three repetitions of washing with distilled water. The filtrate from the third washing was observed with a qualitative test of Phosphate anion by using  $\text{HNO}_3$  and Ammonium Molybdate reagent [15]. The qualitative test found in the first wash there was a large amount of yellow sediment, indicate the first wash there was phosphate, in the second wash there was sediment but in a small amount, and in the third filtrate there was no sediment, which confirm no phosphate content. It was supported from EDX analysis (Figure 1) which no peaks of these impurity elements were observed.

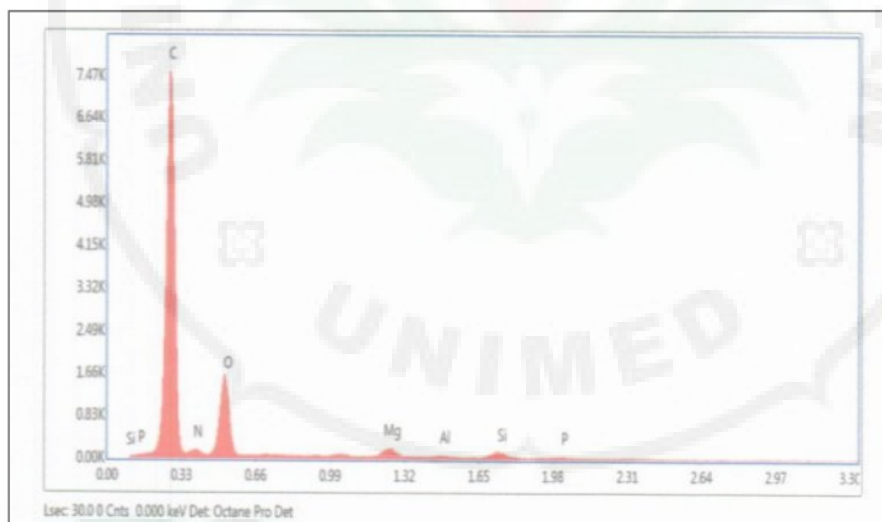
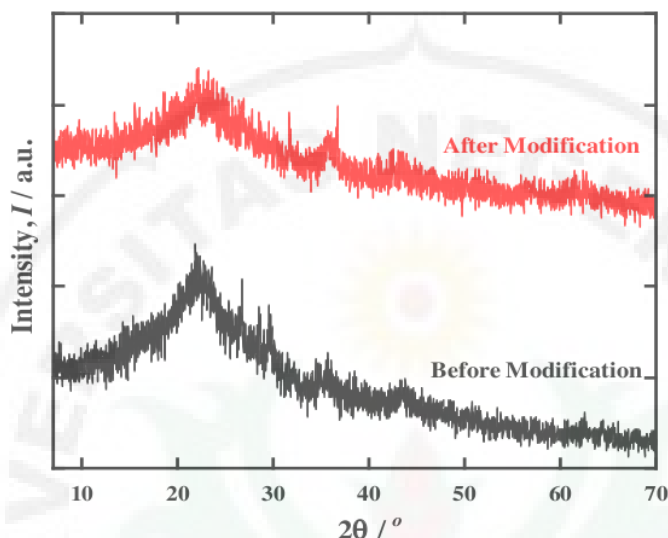


Figure 1. EDX Analysis of Biosorbent Palm Empty Bunches After Washing

To observe the the structure changes of activated carbon after modification and before modification, it were analyzed using XRD. Both of the XRD patterns changed but were not significant, it was observed from the presence of missing peaks in the area  $2\theta = 20^\circ - 30^\circ$  and the appear of several new peaks in the  $2\theta = 20^\circ - 30^\circ$  area which indicate Fe-Cu metal entered the activated carbon framework. The amorphous region at  $2\theta = 10^\circ - 30^\circ$  also shows amorphous properties the activated carbon after modification is slightly reduce if compare with before modification. It indicate the presence of Fe and Cu was effect to increase the crystallinity of activated carbon which have a potential to increase a stability of activated carbon by reducing the thermal energy of adsorption [9].





**Figure 2.** XRD pattern of Activated Carbon of Palm Empty Bunches before and after modification

To observe a more detailed difference properties due to the effect of metal modification on PEB activated carbon, a crystal structure analysis approach was carried out using the 2014 EXPO application (Table 1) from the XRD data obtained above. Although the crystal structure of both of them does not change, they are both triclinic, but the modification of Fe-Cu on activated carbon shows a change in the length of the bond on each side of the crystal, where the bonds between atoms on the activated carbon after modification are longer than the previous modification. The bond angle of activated carbon modified with Fe-Cu also changes significantly when compared to before modification.

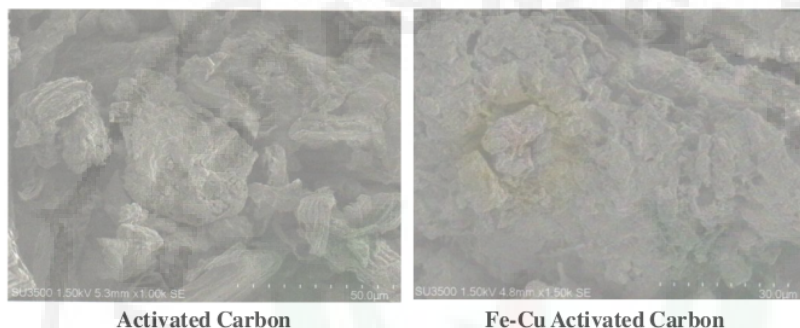
**Table 1.** Analysis of EXPO 2014 Activated Carbon EFB before and after modification

Activated Carbon Type	Crystal Parameter						d Spacing (Å)	Cell Volume (Å) <sup>3</sup>	Crystal System
	a(Å)	b(Å)	c(Å)	α(°)	β(°)	γ(°)			
PEB AC	8.39	9.49	8.38	110.3	103.5	74.8	1.85	596.5	Triclinic
Fe-Cu PEB AC	12.37	15.50	11.30	92.34	105.96	91.60	3.47	2084	Triclinic

Interestingly, this modification of Fe-Cu on activated carbon increases the distance between the lattices from 1.85 Å to 3.47 Å, which indicates a pore space is formed which is almost 2 times larger in the activated carbon modified with Fe-Cu. This is also supported by data on the volume of activated carbon cells which also increased three times from 596.5 Å<sup>3</sup> to 2084 Å<sup>3</sup>. It is suggest that the increasing of the pore framework will increase the absorption capacity of activated carbon against several adsorbates, especially useful for removing impurities in the water purification process.

To support this opinion of the homogeneity of activated carbon particles after modification compared to pre-modified activated carbon palm empty bunches, observations were made through SEM images observation. SEM image shows the difference performance in the surface of activated carbon after modification with Fe-Cu which smaller size than the activated carbon before modification which allows a larger surface area to increase its adsorption ability. In addition, activated carbon after modification with Fe-Cu looks more homogeneous compared to before modification. Besides being able to expand the surface of activated carbon, the inclusion of Fe and Cu metals between the carbon

shells makes the carbon structure more regular and the distance between the carbons more homogeneous. This SEM image (Figure 3) supports the results of XRD analysis which shows a significant change in the distance between the lattices in the activated carbon structure after being modified with Fe and Cu metals. This modified carbon is expected to be able to increase the absorption of impurities in the water when it is tested later for the process of purifying the absorption water.



**Figure 3.** SEM images of palm empty bunches activated carbon before and after modification.

In this study we prepared metal modification of Palm Empty Bunches with successful changes the properties if PEB activated carbon. The presence of Fe and Cu on the modified activated carbon is also evidenced by the EDX image, which shows the presence of Fe and Cu peaks when compared to activated carbon before modification which does not show the presence of Fe and Cu metals. It successful to modified Fe and Cu inside the activated carbon of empty oil palm bunches which consider to improve their adsorption properties.

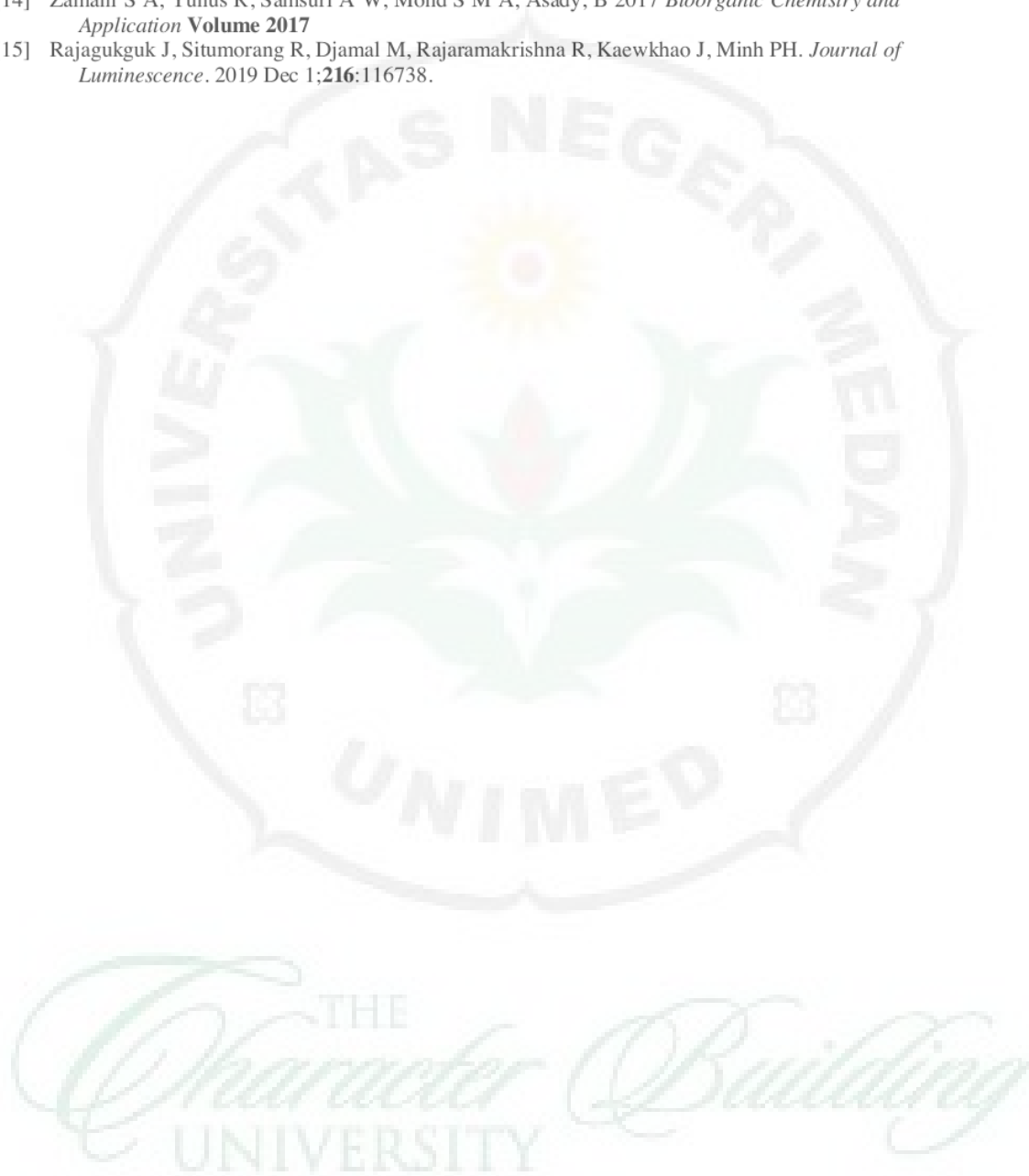
#### 4. Conclusion

The research successful to modified Fe and Cu inside the activated carbon of empty oil palm bunches which consider to improve their adsorption properties.

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