Synthesis and Characterization of Activated Carbon from Oil Palm Empty Bunches

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Abstract. In this study, activated carbon was prepared from empty oil palm bunches obtained from Padang Bulan, Medan, North Sumatra. The empty oil palm bunches are cleaned, then mashed to obtain a particle size of 200 mesh which is then referred to as the oil palm empty bunches bio-sorbent. This bio-sorbent is then washed with water to remove impurities that may come from fertilizers or insecticides during processing in oil palm plantations such as K, Ca, Mg, Si and P, which are visible from the EDX analysis results of bio-sorbent before washed. From the EDX data after washing, the was observed if there are no peaks of these impurity elements. From the XRD pattern it can also be seen that there are some peaks missing after washing if compared to XRD bio-sorbents. Activated carbon then prepared by heating the bio-sorbent at 500 °C for 1 minute, and the XRD results show an amorphous structure as a characteristic of the formation of activated carbon. SEM images can also be observed that the difference in the surface of the activated carbon is smoother than the surface of the bio-sorbent, besides the activated carbon looks more homogeneous.

1. Introduction
Heavy metals such as Pb$^{2+}$, Cd$^{2+}$ and Hg$^{2+}$ caused environmental pollution which have negative impact to the community. Several modern methods such as deposition, electrochemistry, ion exchange, and microorganisms were developed to eliminate heavy metal in liquid waste [1-5]. These method give the best result but the operational cost is very expensive so it is less effective. The adsorption method is an alternative choice, because it is cheaper and more effective in its application [4-8]. Environmental pollution by heavy metal ions causes severe socio-environmental problems. Lead is a common heavy metal, depleted and accumulates in large quantities into the environment as a result of various industrial activities such as tanning, dyeing, paints and pigment manufacturing, wood preservation, electroplating etc., which in turn have had detrimental impacts on health, society and ecosystem in general [9,10].

Lead (Pb) is a heavy metal that has the highest affinity for sulfur and attacks its bonds in the enzyme. As a heavy metal, Pb is classified as a dangerous pollutant. It is in the form of Pb(OH)$_2$ in the water [11]. It is widely used in industry and pipeline work. Lead gasoline is the main source in the atmosphere and earth. Oil palm empty fruit bunches (OPEFB) are a collection of fibers left after separating the fruit from the sterilized fresh fruit bunches (by evaporating at 294 kPa for 1 hour). It is
cheap, decomposable, non-toxic, and is a natural fiber that is widely used. It is a natural material that contains thick and coarse filaments. This makes it more effective than non-renewable industrial materials, hazardous to health and the environment, and expensive for small-scale production [12-14].

Adsorption became a new popular method to reduce heavy metal content in water due to their simple preparation and low cost of usage. Adsorption has several advantages over other methods such as coagulation, flocculation and electrochemical separation. This is because the adsorption method is effective and efficient in terms of process, absorption capacity, cost, and also the adsorbent can be regenerated. Activated carbon is one type of adsorbent which used and widely developed for heavy metal adsorption because it has a large adsorption capacity and can be regenerated. Activated carbon most produced from natural product because contain high carbon compounds.

The palm oil industry also produces a number of by-products in the form of solid and liquid waste. Around 25 million tons of oil palm empty fruit bunches (OPEFB) were produced in Indonesia in 2013. Thus, it can be estimated that there will be an increase in solid waste production - oil palm empty fruit bunches. If it is not used and handled properly, it will be a big problem in the future. The technique of exploiting and processing it is currently more focused on managing “waste” and providing added value to the palm oil industry. Palm oil mill liquid waste is brownish in color, consisting of dissolved and suspended solids in the form of colloids and oil residues with high COD and BOD content of 68,000 ppm and 27,000 ppm, acidic (pH 3.5 - 4), consisting of 95% water, 4 -5% dissolved and suspended ingredients (cellulose, protein, fat) and 0.5 –1% oil residue mostly in the form of an emulsion. The TSS content of palm oil mill effluent is high around 1,330 - 50,700 mg / L, copper (Cu) 0.89 ppm, iron (Fe) 46.5 ppm and zinc (Zn) 2.3 ppm and ammonia 35 ppm [15].

2. Experimental

2.1. Oil Palm Empty Bunch Preparation
Oil palm empty fruit bunches were collected from Padang Bulan, Medan, North Sumatera. A total of 100 grams of empty oil palm bunches was collected and cleaned. Put in the oven at a temperature of ± 105 °C for 24 hours. mashed in a blender and sieved with a 200 mesh sieve. Then the bio-sorbent was purified by washing it three times using distilled water.

2.2. Bio-sorbent Characterization
The characterization of oil palm empty bunches bio-sorbent was carried out using XRD (X-ray Diffractometer) to determine the structure of oil palm empty bunches, FTIR (Fourier-Transform Infra Red) for functional group analysis, determination of surface area with BET (Brunauer-Emmett-Teller), and Morphological analysis using SEM-EDX (Scanning Electron Microscope and Energy Dispersive X-ray).

2.3. Bio-sorbent Carbonization
The prepared palm empty bunches are put into the furnace at 500°C for 2 minutes. Cooled to a room temperature around 30°C. After that, continue to actication process.

2.4. Activated Carbon Activation
The prepared carbon palm empty bunches were immersed in H₃PO₄ for 24 hours. Filtered with Whatman filter paper and washed with distilled water until a neutral pH. Then dried in an oven at 105oC for 1 hour.

3. Results and Discussion

3.1. EDX Characterization of Oil Palm Empty Bunches Content
EDX analysis results on oil palm empty fruit bunch bio-sorbent show that the amount of impurities that may come from fertilizers or insecticides during the processing process in oil palm plantations or
due to the storage process in open air. EDX analysis shows several impurity metals are observed, such as K, Ca, Mg, Si and P (Figure 1). To obtain pure bio-sorbent, it is purified by washing it three times with distilled water. The filtrate from the third washing was subjected to a qualitative test of Phosphate anion. With HNO₃ and Ammonium Molybdate reagent. The qualitative test conducted and it was found that in the first wash there was a large amount of yellow sediment, which means that in 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 showed no phosphate content. From the EDX data after washing (Figure 2), no peaks of these impurity elements were observed.

![Figure 1. Initial EDX Bio-sorbent Analysis of Oil Palm Empty Bunches](image1)

![Figure 2. EDX Analysis of Biosorbent of Oil Palm Empty Bunches After Washing](image2)
3.2. X-Ray Diffraction (XRD) Characterization
To observe the structural changes of the prepared empty oil palm bunches into bio-sorbent and to active carbon, XRD analysis was performed for all three samples (Figure 3). The XRD patterns of the initial bio-sorbent before washing shows the number of sharp peaks indicating the presence of impurity metals which may come from the processing of empty oil palm bunches in oil palm plantations or during the transportation process. In oil palm empty fruit bunch bio-sorbent before preparation it has a character in the form of X-ray diffraction which has an intensity with a wide peak value at $2\theta = 15.26^\circ$; $2\theta = 21.72^\circ$; $2\theta = 24.38^\circ$; $2\theta = 64.34^\circ$ with the highest peak $2\theta = 21.72^\circ$. Bio-sorbent after the purification preparation has a character in the form of X-ray diffraction which has an intensity with a peak value that widens at $2\theta = 15.3^\circ$; $2\theta = 21.9^\circ$; $2\theta = 24.54^\circ$; $2\theta = 64.29^\circ$ with the highest peak $2\theta = 21.9^\circ$.

| No | Before Preparation | After Preparation |
|----|--------------------|-------------------|
| 1  | 15.26              | 15.30             |
| 2  | 21.2               | 21.90             |
| 3  | 24.38              | 24.54             |
| 4  | 64.34              | 64.29             |

Table 1. Diffractogram Analysis of Oil Palm Empty Bunches

After washing, supporting the EDX analysis results, the XRD patterns also indicate the impurities in the missing bio-sorbent which was indicated by several missing peaks in the $2\theta$ region around 20 to 35. The three XRD adsorbents showed an amorphous structure, where the amorphous structure was a more reactive and suitable structure to be applied as an adsorbent. Amorphous also has high purity, small particle size and large surface area. The change from bio-sorbent to activated carbon is also shown by a peak in the $2\theta$ region around 22.

![Figure 3. XRD pattern of oil palm empty bunches](image-url)
The amorphous activated carbon is formed which is more homogeneous when compared to bio-sorbent before washing or after washing. It suggest of the degradation process of several organic and organic compounds present in the bio-sorbent of oil palm empty banches has been lost and activated carbon is formed from empty oil palm banches. Several intensities at the highest peak also show the uniformity of the amorphous structure of the activated carbon and also show the degradation of organic and inorganic compounds as previously described and form the skeletal structure of the activated carbon. The formation of this activated carbon that will be observed in the Pb (II) metal adsorption process, how the availability of space or pore frames on activated carbon can bring more Pb (II) as a source of contaminants in liquid waste.

3.3. SEM Characterization
To support the opinion of the homogeneity of activated carbon particles compared to the bio-sorbent of oil palm empty bunches, SEM image was measured. SEM images observe the difference in the surface by the activated carbon is smoother than that of the bio-sorbent, and besides that the activated carbon looks more homogeneous. The particle size of activated carbon also looks smaller than that of the bio-sorbent which allows a larger surface area of activated carbon, thereby increasing its adsorption ability.

![Biosorbent](image1.png) ![Activated Carbon](image2.png)

**Figure 4.** SEM image of Bio-sorbent and Activated Carbon of Oil Palm Empty Bunches

4. Conclusion
The XRD results show an amorphous structure as a characteristic of the formation of activated carbon. SEM images can also be observed that the difference in the surface of the activated carbon is smoother than the surface of the bio-sorbent, besides the activated carbon looks more homogeneous.

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