Measurements and analysis of crystal structures of activated carbon of empty fruit bunch from oil palm biomass waste

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Abstract. The purpose of this research is to measure and analyze the properties of activated carbon from biomass waste of palm oil empty fruit bunch by using x-ray diffraction (XRD). Biomass waste that has been clean and dried is carbonated at 400°C for 10 minutes to produce charcoal. Next, the charcoal is activated through a chemical process with the activating agent NaOH by soaking it for twelve hours. Activated carbon with 2N activated agent NaOH have the best properties because it has a largest surface area (27.07). The results of this research are useful for technological developments of activated carbon, and provide an economic added value to biomass waste.

1. Introduction

The estimated area of oil palm land in Indonesia in 2019 according to the Directorate General of Estate Indonesia is around 14,677,560 ha, with growth from 2016 to 2017 at 25.42%. The Indonesian Palm Oil Association (GAPKI) notes, 70 percent of 2018 palm production is allocated to meet export needs and the remaining 30 percent for domestic consumption. The value of the contribution of Indonesia's palm oil foreign exchange throughout 2018 reached US $ 20.54 billion or equivalent to Rp 289 trillion. In addition, local residents also depend on palm oil land, where the Directorate General of Estate Indonesia records that 5.8 million ha or 40% of oil palm land in Indonesia is community-owned plantations.

However, the superiority of oil palm in Indonesia has its own dark side. Besides clearing land activities for oil palm planting that arguably contributes to forest destruction and threatens the extinction of Indonesia's endemic animals, the waste produced by oil palm plantations also has the potential to pollute the environment. On average each fresh fruit bunch produces an oil palm empty fruit bunch (OPEFB) of 23% of its total weight, where each hectare (ha) of gardens can produce 4-5 tons of OPEFB per year [2]. OPEFB as biomass waste has a very slow decomposition time of 6-12 months, so it will cause environmental pollution if it is not managed properly. Therefore, this study aims to process the OPEFB biomass waste into a material that is more environmentally friendly and has better economic aspects.

In this research, OPEFB synthesized into activated carbon using NaOH as activated agent. Carbon activation by alkaline hydroxides has three unique characteristics that make them very attractive for new and potential applications. These are; i) their low ash content, even using precursors having high ash contents, ii) their very high adsorption capacity and iii) their controlled narrow porosity distribution (not possible to achieve using other activating agents) [7]. Activated carbon characterized using an XRD machine. OPEFB is a material that has the potential to become activated carbon because it has a high
lignocellulose content with approximately around 44.2% cellulose, 33.5% hemicellulose and 20.4% of the lignin [1]. Lignocellulosic material found in OPEFB has the ability to absorb heavy metals [6]. With activated carbon properties that have large surface area, activated carbon from the OPEFB biomass waste can be very good to be applied as a waste water purifier, such as a heavy metal absorber in the liquid waste.

2. Materials and Methods

2.1. Raw Materials
Oil palm empty fruit bunches biomass waste were subjected to activation. OPEFB is obtained from palm oil field waste in Pasaman Barat Regency, West Sumatra. NaOH 0.5 N, 1N, 2N solutions are prepared as activated agents.

2.2. Carbonization
OPEFB that has been clean, cut into small pieces, and dried for 2 days under the sun, then water content removed the with an oven at 105°C for 2.5 hours. After drying, the sample is carbonated with the furnace at 400°C for 10 minutes to produce charcoal

2.3. Activation
The charcoal that has been produced is activated by soaking it into NaOH solution for 12 hours. NaOH concentrations used vary: 0.5N, 1N, and 2N. After the soaking process is finished, the sample is dried using an oven at 105°C for 3 hours. The dried sample then crushed to a powder, and stored in a desiccator to maintain its moisture.

2.4. Characterization
Activated carbon in powder form was characterized using X-ray Diffraction machine (XRD). The conditions used include: The patterns were run with copper (Cu) radiation (λ = 1.5406 Å), 40kV energy, 30mA current. Measurement starting position at [° 2θ]: 10.0131, end at [° 2θ.]: 99.9731, and recorded every [° 2θ]: 0.0260. Later, you will get a graphic of the relationship between the diffraction angle and the resulting intensity. Other parameters set are, crystal lattice, inter-layer spacing (d) and crystallite height (Lc), based on Bragg and Scherrer's formula [5,4] as follows:

\[ Lc_{002} (\text{nm}) = K \cdot \frac{\lambda}{\beta \cos \theta}; \]

where, \( K = 0.89; \)
\( \lambda [\text{nm}] = \) the X-ray wavelength;
\( \theta = \) Half diffraction angle;
\( \beta = \) FWHM (Full width at half the maximum intensity).

3. Result and Discussion
Activated carbon prepared from OPEFB biomass waste with NaOH as an activator was characterized using XRD. The results were listed in Table 1 and Figure 1.

| NaOH Conc. (N) | 2θ (°) | d [Å] | Int. [%] | FWHM (2θ) | h k l | Crystal | Lc_{002}(nm) |
|---------------|--------|-------|---------|-----------|------|---------|-------------|
| 0.5           | 29.2958| 3.04865| 29.24283| 0.307     | 2 0 2 | CO      |             |
| 29.9324       | 2.98525| 33.12161| 0.307   | 0 0 2     | Na_2CO_3|         |             |
| 34.1796       | 2.62339| 51.89877| 0.307   | 2 1 1     | CO    |         |             |
| 35.1216       | 2.55516| 33.48967| 0.307   | 3 1 0     | Na_2CO_3|         |             |
| 37.6866       | 2.38694| 27.53671| 0.409   | 0 0 2     | C     | 20.28   |             |
Observed from the figure 1, there are three dominant elements or compounds diffracted in activated carbon samples, which is carbon (C), carbon monoxide (CO), and Sodium Carbonate (Na$_2$CO$_3$). Carbon monoxide (CO) is produced by the direct oxidation of carbon in a limited supply of oxygen or air [11]. This can happen because the carbonation of the dried OPEFB into charcoal is carried out in a closed furnace. Na$_2$CO$_3$ is obtained from chemical reactions during the carbon immersion process with the activated agent NaOH with chemical formula C + 2NaOH + H$_2$O = Na$_2$CO$_3$ + 2H$_2$.

The increase in Carbon (002) peak intensity (fig. 1) is directly proportional to the increase in activator concentration, while inversely proportional to its width diffraction pattern. High and low peaks resulting from the characterization of XRD is influenced by the activation process that causes a hexagonal plate shift which was originally a high level of order (crystalline) to irregular (amorphous) [10, 8].
Found at table 1 that Crystallite height (Lc) is increases whereas lattice spacing (d_{002}) decreased with increases concentration of NaOH. It means Activated carbon with activated agent NaOH 2 N has the best properties. That was because a good activated carbon property is activated carbon with high carbon content, the smaller width of the layer (La), and the largest height of layer (Lc). The greater the value of the layer height (Lc), the surface area of the activated carbon is higher [3]. The smaller the width of the layer (La) crystallite has a greater surface area of activated carbon [9].

4. Conclusion
Measurements have been made on the properties of activated carbon derived from OPEFB that is activated by NaOH as an activating agent using XRD machine. The results obtained are three dominant elements or compounds diffracted in activated carbon samples, which is carbon (C), carbon monoxide (CO), and Sodium Carbonate (Na_{2}CO_{3}). Carbon (002) peak are increase and width diffraction pattern decrease during the increases of activated agent concentration. Meanwhile, the NaOH 2N as an activating agent produces the best activated carbon because it has a large surface area.

Acknowledgements
This work was supported by Direktorat Riset dan Pengabdian Masyarakat, Kementrian Riset dan Teknologi/Badan Riset dan Inovasi Nasional, Republik Indonesia, under scheme Penelitian Tesis Magister contract No. 055/SP2H/LT/DRPM/2020.

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