Characteristics of activated carbon resulted from pyrolysis of the oil palm fronds powder

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Abstract. Activated carbon is the product of a charcoal impregnation process that has a higher absorption capacity and has more benefits than regular char. Therefore, this study aims to cultivate the powder of oil palm fronds into activated carbon that meets the requirements of Standard National Indonesia 06-3730-1995. To do so, the carbonization process of the powder of oil palm fronds was carried out using a pyrolysis reactor for 30 minutes at a temperature of 150 °C, 200 °C, and 250 °C in order to produce activated char. Then, the char was impregnated using Phosphoric Acid activator (H3PO4) for 24 hours. Characteristics of activated carbon indicate that the treatment of char by chemical activation of oil palm fronds powder has an effect on the properties of activated carbon. The activated carbons that has the highest absorption properties to Iodine (822.91 mg/g) were obtained from the impregnation process with 15% concentration of Phosphoric Acid (H3PO4) at pyrolysis temperature of 200 °C. Furthermore, the activation process resulted in activated carbon with water content of 8%, ash content of 4%, volatile matter 39%, and fixed carbon 75%, Iodine number 822.91 mg/g.

1. Introduction
Oil palm is a type of plant that produces various kinds of byproducts, such as empty fruit bunches, shells or oil palm fronds [1]. Unfortunately, these products have not been utilized into products that have high economic value. As a result, these products only become useless garden waste [2]. One product with huge potential is oil palm fronds, as it is the largest biomass resources are available in oil palm plantations. The average weight of palm fronds that had entered the cutting period is equal to 4 kg per dry frond, with total production of palm fronds around 5500 kg per hectare per year [3].

Recognizing the enormous potential of palm oil stem, this study aims to utilize palm oil fronds into a more valuable products, such as activated carbon. Furthermore, this study also tries to produce activated carbon in accordance with Indonesian National Standard SNI (06-3730 - 1995), where there is a minimum value of quality that must be possessed in order to meet the criteria of high quality activated carbon.

2. Theory
Palm oil fronds is one of the solid waste resulted from oil palm plantations that can be obtained throughout the year during harvest time. It consists of three main elements: cellulose (31.5%), hemicellulose (19.2%) and lignin (14%) [4]. The potential of palm oil fronds in Indonesia is very promising, with the total production in Indonesia reaching 10.14 million tons in 2016 [5].

There are two stages of the manufacture of activated carbon, which is the stage of carbonization and activation [6-7]. Carbonization is defined as a process of destruction of organic matter by heating to charcoal in the absence of oxygen [8]. Meanwhile, the activation process is required to change the results of carbonization into the adsorbent which has a high porosity and a large surface area. The quality of activated carbon depends on the type of raw materials and processing technology used.

The quality standards for activated carbon has been set in the Indonesian National Standard, SNI 06-3730-1995 [9] as described in Table 1.
Table 1. Quality standards of activated carbon according to SNI (06 - 3730 - 1995)

| No | Description     | Unit | Requirements |
|----|-----------------|------|--------------|
| 1  | Water content   | %    | Max. 15      |
| 2  | Ash content     | %    | Max. 10      |
| 3  | Volatile matter | %    | Max. 25      |
| 4  | Fixed carbon    | %    | Min. 60      |
| 5  | Iodine absorption | mg/g | Min. 750    |

3. Method
The production of activated carbon from palm oil fronds powder was carried out through four stages: (1) preparation of palm oil fronds; (2) charcoal production (carbonization) through pyrolysis process; (3) immersion (impregnation); and (4) analysis of products (activated carbon). At the preparatory stage, the palm oil fronds was cut into small pieces and dried in an oven at a temperature of 105°C. The resulting product was then mashed using a ball mill and sieved using a 50 mesh filter.

Thereafter, the chemical activation was performed using a Phosphate Acid activator (H₃PO₄) with concentrations of 10%, 15% and 20% in the oven at a temperature of 80°C for 2 hours. Following this, the impregnation process was performed for 24 hours at a room temperature. Then, the activated carbon was filtered and washed using distilled water until it reached a pH of 7, to be dried. Finally, two types of analyses were conducted on the activated carbon: (1) proximate analysis in the form of water content, ash content, volatile matter, and fixed carbon; and (2) qualitative analysis in the form of FTIR and Scanning Electron Microscope (SEM) analysis.

4. Result
The results of the study based on the parameters in SNI (06 - 3730 - 1995) can be seen on Table 2.

Table 2. Results of the research on the activated carbon from oil palm fronds

| Time (Minutes) | Temperature (°C) | Concentration of Activator (%) | Water Content (%) | Ash Content (%) | Volatile Matter (%) | Fixed Carbon (%) | Iodine Number (mg/g) |
|----------------|------------------|--------------------------------|-------------------|-----------------|--------------------|------------------|----------------------|
| 30             | 150              | 10                             | 1.48              | 8.03            | 13.59              | 76.90            | 597.52               |
|                |                  | 15                             | 1.72              | 8.90            | 16.00              | 73.38            | 566.02               |
|                |                  | 20                             | 3.36              | 7.55            | 16.04              | 73.04            | 603.99               |
| 200            |                  | 10                             | 5.00              | 6.00            | 12.00              | 77.00            | 685.70               |
|                |                  | 15                             | 8.00              | 4.00            | 30.39              | 75.00            | 822.91               |
|                |                  | 20                             | 7.00              | 6.00            | 13.46              | 73.54            | 741.47               |
| 250            |                  | 10                             | 5.34              | 8.73            | 29.41              | 56.52            | 429.02               |
|                |                  | 15                             | 6.70              | 7.13            | 17.00              | 55.78            | 402.46               |
|                |                  | 20                             | 3.27              | 7.90            | 39.00              | 49.83            | 404.67               |

4.1 Water content
Water content increases with higher activator concentration. This is due to the dissolving process of residual pyrolysis and organic minerals on the surface of activated carbon which will get better as the activator concentration increases. Increased concentration of activator will increase the amount and volume of pores and surface area, which cause an increase in the performance of activated carbon to absorb water from air [10].

The increase in pyrolysis temperature will cause the C and H bonds in charcoal to be released completely. Thus, a shift of the crystallite carbon plate forms a new pore and develops a pore that has
been formed [11]. These results indicate that activated carbon has met the requirements of the National Industrial Standard 06-3730-1995.

4.2 Ash content
Ash content has an influential role in affecting the quality of activated carbon produced. The presence of excessive ash can lead to clogging of the pores on the activated carbon so that the surface area of activated carbon becomes reduced [12]. Ash content increases with increasing concentration of activator so that organic compound of activated carbon decreases, however the content of inorganic compound is relatively fixed.

The interaction in the reactor during the pyrolysis process causes the inorganic compound to trigger the formation of the metal oxide. The increasing number of metal oxide causes the ash content in the activated carbon to be increased, especially with the rise in temperature. There is an increase in ash content at 250°C, indicating that there is an increase of metal oxide formation, which resulted in the increase of ash content as well. The concentration of activators that are too high can also cause damage to the structure of activated carbon, which resulted in the increase of ash content [7]. The increase in ash content can also be caused by mineral salt formation during the pyrolysis process. In addition, the ash content is influenced by the silica content of the raw material. This would mean that the higher the silica content, ash content is also higher [7]. Thus, in general the ash content of activated carbon in all variables has met the SNI standards of 06-3730-1995.

4.3 Volatile Matter
The levels of volatile substances decrease as activator concentration increases. This is because the addition of activators causes changes in the structure and properties of activated carbon absorption. H₃PO₄ activators cause degradation of organic material that weakens the surface structure of activated carbon. In addition, this activator also releases volatile substances and develops an active carbon micro pore structure [13].

At higher temperatures, volatile materials will be released into more and cause less carbon to be formed [13]. This is due to higher pyrolysis temperatures, so that the decomposition of components contained in raw materials such as water, tar, and volatile materials is also higher [15].

The impregnation process using the activator is capable of reducing the non-carbon compound attached to the surface of the activated carbon and entering the bottom surface of the charcoal through the pores of the charcoal. Thus, the presence of the activator will cleanse and enlarge the pore surface. Activators will free non-carbon elements, especially hydrogen, oxygen and nitrogen in liquid form known as tar and gases [16].

4.4 Fixed Carbon
The value of the resulting fixed carbon content fluctuates as the pyrolysis temperature increases. Fixed carbon content is also affected by cellulose and lignin content that can be converted to carbon atoms. At a temperature of 250 °C there is a decrease in fixed carbon caused by the breakdown of activated carbon structures with high temperatures or burning of carbon [16]. Decrease in carbon content can also be caused by the reaction between the carbon and the activator at high concentrations, which can damage the micropore on the carbon surface [16].

The findings show that fixed carbon which meets SNI 06-3730-1995 is at operating condition 150°C and 200°C for all activator concentration.
4.5 Iodine Number

The highest iodine number of the activated carbon is at a temperature of 200°C and drops to 250°C for all concentrations of activator. This shows that there is damage to the structure of the activated carbon in the form of the collapse of the pore wall so that the iodine number decreases at a temperature of 250°C. Besides pore structure damage, there is also a burning carbon, as well as a micro pore expansion to the macro pore, all of which cause a decrease in the iodine number [18]. All this is due to high activator concentration and/or too high carbonization temperature.

The findings show that the iodine number fulfilling SNI 06-3730-1995 is under operating conditions 200°C for all activator concentrations.

4.6 Scanning Electron Microscope (SEM) Analysis

SEM analysis is used to visualize the surface morphology of the product which has been carbonized and activated. Figure 1 shows the surface characteristics of the activated carbon of the study that has a pore and cavity distribution on its surface.

![SEM images of charcoal and activated carbon](image)

(b) Charcoal  (a) Activated Carbon

**Figure 1.** Scanning Electron Microscope (200x) of Activated Carbon

The structure of the pores formed can result from evaporation and breakdown of the non-carbon compounds contained in the feedstock. Activated carbon also has a rougher surface and a wider irregularity. The presence of the activator can enlarge the pores of activated carbon and expand the surface.
4.7 Fourier Transform Infra-Red (FTIR) Analysis

FTIR analysis is used to characterize the functional group on the surface of activated carbon. Figure 2 shows the characteristics between Palm Oil (a) and Active Carbon (b).

![Figure 2. FTIR spectrum of palm oil fronds and activated carbon](image)

As can be seen in Table 3, the wave numbers between palm fronds and activated carbon has a peak with no significant difference. The presence of OH bonds and C-O indicated that activated carbon tends to be more polar. Therefore, the FTIR results show that the activated carbon produced can be said as activated carbon.

Table 3. Comparison between infrared fraction of oil palm fronds and activated carbon

| No | Functional Group | Oil Palm Frond | Activated Carbon |
|----|------------------|----------------|------------------|
| 1. | O-H              | 3394.72        | 3437.15          |
| 2. | C-H              | 2924.09        | 2962.66          |
| 3. | C-O              | 1323.17        | 1238.30          |
| 4. | C-X              | 447.49         | 559.36           |

5. Conclusion

This study obtained several highest test value for indicators mentioned in the National Industrial Standards (SNI) 06-3730-1995. In particular, the following test value were discovered: water content of 8.00%, ash content of 8.90%, volatile matter of 39.00%, fixed carbon of 77.00%, and iodine number of 822.91 mg/g. Hence, it can be concluded that the activated carbon produced from palm oil fronds has met the requirements of the National Industrial Standard 06-3730-1995.

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