Membranes adsorber from oil palm empty fruit branches (OPEFB): preparation and fabrication

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Abstract. Oil Palm empty fruit branches (OPEFB) are solid waste that are numerous produced from palm oil mills. OPEFB is economically and potentially used as membrane adsorber material due to has good thermal stability, chemical resistance and biodegradability. The objectives of this work is to preparte and fabricate the OPEFB membrane adsorber which is activated by physical activation. The OPEFB has been cleaned and dried, subsequently heated at 500 °C for 30 min via pyrolysis. The activated OPEFB was sieved using 200-400 mesh and followed by the addition of 2-propanol, NH\(_4\)Cl, polyvinyl alcohol (PVA) and polyethylene glycol (PEG) to become a mixture. The activated OPEFB ratio were varied in the mixtures to obtain the best composition in order to produce a good membrane adsorber texture for casting. FTIR shows on wavenumber at 1082 cm\(^{-1}\) indicates that there is O-H stretching functional groups and bands at 943 cm\(^{-1}\) correspons to C=O functional group. It is concluded that the membrane mixtures can be employed as membrane adsorber due to carbon content which creates strong matrix applied for gas separation.

1. Introduction
Indonesia is the largest palm oil producer in the world, especially in South Kalimantan-Indonesia \([1, 2]\). Production of fresh palm fruit oil branches is increased as much as the demand every year. Nevertheless, the increase in fresh palm fruit oil branches also brings out increasing solid and wastewater production \([3]\). Oil palm empty fruit branches (OPEFB) is one of the solid waste produced by palm oil mills is about 23% of the total fresh palm fruit oil \([4]\). Whereas, OPEFB still containing high hemicellulose (37.76%), cellulose (66.07%), fiber (72.67%), and lignin \([5]\). Cellulose is a carbon compound that consists of much glucose which is bound by beta 1.4 glycoside bonds and easily decomposes into sample C by cellulosic organisms compounds. Meanwhile, lignin is a component of OPEFB which is relatively difficult to deteriorate. This compound is a structural polymer associated with cellulose and hemicellulose. When observed from its structure, OPEFB has economic value and

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potential to be used for gas adsorption. It due to the OPEFB causes a polar character in the adsorber [6].

Cellulose derived OPEFB is a renewable natural polymer that is superfluous available and has unique characteristics such as good thermal stability, chemical resistance, and biodegradability [2, 7]. Furthermore, the fibrous structure on the nanometer-scale of cellulose also has several advantages i.e. high surface area, controllable porous structure, and outstanding mechanical properties [8]. Cellulose has the desired structure which is a challenge due to its strong hydrogen bonds. This leads to very poor solubility in several solvents [9]. This material is appropriate and favorable to tailored the membrane adsorber [10].

In generally, OPEFB was used to preparation of activated carbon due to its porous material containing carbon. Moreover, activated carbon also offering a high surface area [11-13]. Activated carbon can adsorb particularly gases and chemical compounds depend on its adsorption properties by the size, pore-volume, and surface area [14]. The adsorption capacity of activated carbon ranges from 25 to 100%. Activated carbon can be made through a physical or chemical activation process [15]. Adsorption is a physical phenomenon that occurs when gas or liquid molecules are in contact with a solid surface and some of the molecules condense on the solid surface. One of the adsorption technologies for biogas purification can use adsorbents such as activated carbon for CO2 adsorption [16]. Some purification technologies were developed with various types including water scrubbing, chemical adsorption, and membrane purification technology [17].

Membrane technology has come a long way in recent decades [18-21]. Membrane technology has several advantages both technically and economically [22-24]. Thus, it is often used in the environmental, health, and industrial fields [25]. The membrane functions as a thin, selective barrier between the two phases by only passing certain components and holding the other components [26-29]. Recently, adsorbent membranes have emerged as a popular technology [30]. When compared to conventional separation methods such as chemical precipitation, ion exchange, and adsorption column, the adsorbent membrane technology has unique advantages. These advantages are high removal efficiency, normal productivity, low-pressure drop, and good stability [31]. Therefore, this study was focused to designing the membrane adsorbent structure properties derived from OPFEB witch suitable to apply for gas purification.

2. Method

2.1. Chemicals and Materials

Preparation of OPEFB membrane adsorber by employing of the chemicals and materials such as oil palm empty fruit branches (OPEFB) was taken from PT. Kharisma Alam Persada South Kalimantan Indonesia, isopropyl alcohol (2-propanol, Merck), ammonium chloride (NH4Cl, Merck), polyethylene glycol 400 (PEG 400), polyvinyl alcohol (PVA), filter paper, deionized water (DI water Merck).

2.2. Pre-treatment of OPEFB

Oil palm empty fruit branches (OPEFB) must be firstly mashed until it becomes fibrous. Then dry the OPEFB under the sun for ±7 days and stored in a closed container before use.

2.3. Preparation of OPEFB Membrane Adsorber

After Oil palm empty fruit branches (OPEFB) have been dried under the sun for ±7 days. Afterwards, the pyrolysis process was carried out with a temperature of 500°C for 30 minutes, then the membrane was sieved to get a size of 200 mesh to 400 mesh. After OPEFB has been carbonized, it is ready to be used in the fabrication membrane processed.

2.4. Fabrication of OPEFB Membrane Adsorber

The preparation of the adsorber membrane dope solution in this research was carried out by trial and error method to obtain the best adsorber membrane. Weigh the OPEFB activated carbon to a predetermined size, then mix with 35 ml 2-propanol, then centrifuged the media for 10 minutes at a speed of 600 rpm. Filter the media that has been centrifuged, then add 3.5 gr of NH4Cl that has been dissolved with 300 ml of DI water and stirred for 1 h to form pores on the membrane. Once in the
stirrer, the media is filtered. Then, add 3.4 gr of PVA and 5 ml of PEG so that the media can be produced. Schematic set up fabrication of membrane adsorber OPEFB can be seen in Figure 1, as follow.

![Figure 1](image)

Figure 1. Schematic set up fabrication of membrane adsorber OPEFB

3. Results and Discussion

3.1. Effect of Composition and Method Fabrication Membrane Adsorber OPEFB

A comparison of the composition fabrication of membrane adsorber OPEFB can be seen in Table 1, as follow. Table 1 shows that the polymerization process in the fabrication of membrane adsorber OPEFB is influenced by the polymer concentration and the treatment of the fabrication membrane. In experiment 7th to 10th, the OPEFB membrane adsorber were produced good physic texture as shows in Figure 2.

### Table 1. Comparison of the composition fabrication of membrane adsorber OPEFB

| Trial | Composition | Method | Texture result |
|-------|-------------|--------|----------------|
| 1     | OPEFB: 10 g | PEG: 5 mL | Dried under the sun until the membrane is dry and heated by oven at 70 °C for an hour | Moist |
| 2     | OPEFB: 10 g | PEG: 5 mL | Heated by oven at 80 °C for 24 h | Moist |
| 3     | OPEFB: 10 g | PEG: 5 mL | Dried under the sun for 4 h and heated by a furnace at 105 °C for 30 min | Too sticky |
| 4     | OPEFB: 10 g | PEG: 5 mL | Dried under the sun for 5 to 6 days and heated by a furnace at 105 °C for 30 min | Too thick |
| 5     | OPEFB: 10 g | PEG: 5 mL | Dried under the sun for 5 to 6 days and heated by a furnace at 105 °C for 30 min | Moist |
| 6     | OPEFB: 5 g  | PEG: 3-3.2 mL | Dried under the sun for 2 to 3 days | Crushed |
| 7     | OPEFB: 15 g | PEG: 0.5 mL | Dried under the sun for ± 2 days and heated by a furnace at 105°C for 1 h | Good but too thick |
| 8     | OPEFB: 12.5 g | PEG: 5 mL | Dried under the sun for ± 2 days and heated by a furnace at 105°C for 1 h | Good but easily crack |
| 9     | OPEFB: 5 g  | PEG: 5 mL | Dried under the sun for ± 2 days and heated by a furnace | Good but the edge is fragile |
| Trial | Composition | Method | Texture result |
|-------|-------------|--------|----------------|
|       | OPEFB (g)   | PEG (mL) | at 105°C for 1 h |
| 10    | 17.5        | 5       | Dried under the sun for ± 2 days and heated by a furnace at 105°C for 1 h |
|       |             |         | Good membrane casting |

After the membrane was made according to the membrane fabrication procedure. Then the membrane was fabricated and dried in the sun until the membrane dried. After the membrane was heated by oven at 70°C for 1 h and result in the membrane become fragile. After the membrane fabrication process, is carried out according to the procedure. The fabrication process is carryout out in the oven at 80°C for ± 24 h. The process is too long for heated the membrane causes the upper of the membrane to dry faster so that the reversing process is required. This causes the center of the membrane to be imperfect and still moist.

![Figure 2. The OPEFB membrane adsorber obtained by added different weight of biomass at 6 to 10 trials](image)

In the next Experiment, the OPEFB activated carbon was firstly soaked in DI water for ± 1 h, then filtered. After the membrane was fabricated, the membrane was heated by an oven at 40°C for 10 h and followed by drying under the sun for ± 4 h. After the membrane dry, then the membrane was heated by a furnace at 105°C for 30 minutes. This process makes the membrane adsorber OPEFB is less than perfect because the bottom of the membrane is sticky to cast. Then, the procedure for fabrication membrane adsorber OPEFB was the same as in the previous work. However, the membrane is not in the oven, however, the membrane is dried under the sun for 5 to 6 days then it dried by a furnace at 105°C for 30 minutes. As a result, the membrane in this experiment was too thick, so the weight composition of OPEFB must be reduced.

In the next experiment, 10 g of OPEFB were used. After all the ingredients are mixed on the media, the result is still not as desired. As a result, the membrane is still too wet, this is presumably because of the PEG was added too much, in this research used 5 ml PEG. 6th experiment, using only 5 gr of OPEFB and divided into 2 spaces, each weight is 2.5 g. The membrane fabrication procedure is still the same as the previous procedure. PEG was measured as much as 3 ml and 3.5 ml respectively and then put into each activated carbon OPEFB weight 2.5 g. Without the addition of NH₄Cl and DI water, the membrane was immediately fabricated and press with a pressure of 80 bar. Then the membrane is dried under the sun for ± 2 to 3 days. After the membrane is dried and then the membrane is removed from the casting. The result, the membrane was still too fragile, this is presumably because the PEG added was still lacking.

In the 7th experiment, activated carbon OPEFB was washed with DI water and filtered. The procedure is still the same as the previous adsorber membrane preparation. However, only 0.5 ml of PEG were added in this step. The media was stirred using a mixer, then added the remaining solution of NH₄Cl and 5 ml of DI water. Then the membrane is fabricated and pressed with a pressure of 100 bar. The membrane was dried under the sun for ± 2 days. After drying, the membrane dried by a
furnace at 105°C for 1 h. In this research, its procedures a good membrane, and the membrane surface look flat. Therefore, the 8th experiment to 10th experiment was carried out by varying the weight of activated carbon OPEFB, namely 12.5 g, 15 g, and 17.5 g with the same procedure as the 7th experiment to fabricated a good membrane.

3.2. Characterization of Membrane Adsorber OPEFB

The FTIR spectra of membrane adsorber OPEFB were given in Figure 3, based on these figures, activated carbon OPEFB showed at 1082 cm\(^{-1}\) which attributed to O-H stretching functional group. The bands at 943 cm\(^{-1}\) corresponded to C=O functional group. Rani, et al. [32], conducted a study compare FTIR spectra result of the raw material OPEFB, activated carbon OPEFB and commercial activated carbon. Based on the research, showed the raw material OPEFB has a complicated and clear spectrum than activated carbon OPEFB and commercial activated carbon.

![FTIR spectra of membrane adsorber OPEFB](image1)

**Figure 3.** FTIR spectra of membrane adsorber OPEFB

Based on research Yacob [33], the functional groups from raw material were released as volatile materials when the heat was supplied to the sample during the carbonization and activation process. This proved that the activation process has taken place successfully. It could be seen from Figure 3 that the curves of membrane adsorber OPEFB. The membrane adsorber OPEFB that has been fabricated can be seen from Figure 4, as follow. The color of the membrane is black due to the physical activation process. Membrane adsorber OPEFB has a diameter of 7.2 cm and the thickness of the membrane is 0.6 cm.
**Figure 4.** (A) Front of image membrane adsorber OPEFB, (B) Back of image membrane adsorber OPEFB and (C) Thickness of image membrane adsorber OPEFB

Morphology of membrane adsorber OPEFB observe used mini microscope with 1000x magnification to observe the surface and cross-section of the membrane. **Figure 5,** there exist very small pores available on the surface of membrane adsorber OPEFB. This is maybe due to the temperature given during the physical activation of activated carbon OPEFB. According to Rani, et al. [32], physical activation with a temperature of 756°C and activation time 77 minutes, can be making larger pores on the membrane.

**Figure 5.** (A) The surface section and (B) cross section of the microscope image of membrane adsorber OPEFB with 1000x magnification

Oil palm empty fruit branches (OPEFB) have fibers belonging to the type of lignocellulose so that the OPEFB has a complex structure [1]. On figure 3 and figure 4 can be seen that the OPEFB fibers are rod-shaped. The OPEFB structure can be divided into two types, namely fibers with a rough surface and porous surfaces containing silica. It is according to the content of OPEFB. The OPEFB is covered by lignin, hemicellulose, and another component that binds cellulose [1].

4. **Conclusions**

Membrane adsorber from palm empty fruit branches have been successfully fabricated by varied composition and activated via physical activation. The best composition of OPEFB membrane adsorber is OPEFB:2-propanol:PEG:NH₄Cl: PVA of 17.5 g: 35 ml: 5 ml: 3.5 g: 3.4 g. The membrane has diameters of 7.2 cm and a thickness of 0.6 cm. Morphology of the membrane seen using a mini microscope showed that the membrane has very small pores and the fibers are rod-shaped. The FTIR spectra of membrane absorbent OPEFB showed that at 1082 cm⁻¹ which attributed to O-H stretching functional group. The bands at 943 cm⁻¹ corresponded to C=O functional group. In conclusion, the membrane can be used as membrane adsorber due to containing rich by carbon which affordable for gas separation.

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