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To cite this article: Yuliusman *et al* 2017 *IOP Conf. Ser.: Mater. Sci. Eng.* **180** 012282

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Preparation of Activated Carbon from Palm Shells using KOH and ZnCl₂ as the Activating Agent

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Abstract. Palm shell is a potential source of raw materials for the produce of activated carbon as biosorbent for quite large numbers. The purpose of this study is to produce activated carbon qualified Indonesian Industrial Standard (SNI), which will be used as biosorbent to purify the impurities in the off gas petroleum refinery products. Stages of manufacture of activated carbon include carbonization, activation of chemistry and physics. Carbonization of activated carbon is done at a temperature of 400°C followed by chemical activation with active agent KOH and ZnCl₂. Then the physical activation is done by flowing N₂ gas for 1 hour at 850 °C and followed by gas flow through the CO₂ for 1 hour at 850 °C. Research results indicate that activation of the active agent KOH produce activated carbon is better than using the active agent ZnCl₂. The use of KOH as an active agent to produce activated carbon with a water content of 13.6%, ash content of 9.4%, iodine number of 884 mg/g and a surface area of 1115 m²/g. While the use of ZnCl₂ as the active agent to produce activated carbon with a water content of 14.5%, total ash content of 9.0%, iodine number 648 mg/g and a surface area of 743 m²/gram.

1. Introduction

Off-gas is one of the by-product produced by crude oil refining process. Off-gas contains light hydrocarbon such as CH₄, C₂H₆ and etc. It also known for containing large amount of impurities such as CO, SO₂, NO_x, and H₂S. Usually, off-gas will be combusted and emitted to the air. Whereas, off-gas still has an economical value and very potential to be exploited as a fuel or as a petrochemical feedstock. Basically, utilization of an emitted off-gas can boost oil company's profit, by optimizing surface condition rather than exploring new wells. Therefore, utilizing off-gas is an alternative way of increasing company's profit. Before an off-gas can be employed to a more useful function, it should be cleaned up from its impurities. One of the method available to clean an off-gas is by adsorption of impurities by an activated carbon as biosorbent. Recently, activated carbon is employed to absorb pollutant gas [5].

Activated carbon is a biosorbent with a high absorption capacity, which processed by carbonization process and activated by physical and chemical drive. Based on its structure, activated carbon is an amorphous carbon which consists of free carbon atoms and a deep-surface which creates high absorptivity compared to another adsorbent [3]. Organic matters which contain lignin, hemicelluloses, and cellulose, can be used as an activated carbon feedstock, due to its effectiveness in adsorption process. One of the potential source as an activated carbon feedstock is palm shell.



Palm shell contain 26.6% of cellulose and 27.7% of hemicelluloses, which contributed to high absorptivity characteristic in an activated carbon. Palm shell is one of the most abundant waste in palm oil processing, with a share of 12% from total weight of palm fruit. Palm oil production averaging at 5.6 million tons annually, which resulted 672,000 tons of palm shell produced. The amount of palm shell will continue to rise as palm oil production projected to grow in recent future. Hence, with palm shell waste is in abundance, advanced process to convert palm shell waste to a product with higher economical value is needed [6].

Activated carbon quality as a biosorbent in pollutant adsorption, is very much affected by production process. Several parameters are responsible in activated carbon synthesis process, such as, dehydration, carbonization, cathodization, and activation. Besides that, effectivity of an absorption process is determined by several parameters such as, absorptivity, temperature, contact time, and surface area. Until now, synthesis of an activated carbon from organic feedstock still produced a sub-standard product which didn't meet certain criteria. Therefore, this research aims to produce an activated carbon with good characteristics as a biosorbent.

Activation treatment which utilize activating agent in activated carbon synthesis has been used by adding several chemicals such as NaOH, H₃PO₄, and ZnCl₂. H₃PO₄ treatment has been used to produce activated carbon with surface area of 438.9 m²/g [7]. KOH usage as an activating agent produced larger surface area of 3000 m²/g [9]. Furthermore, ZnCl₂ application will produce activated carbon with a surface area of 1100 m²/g [8]. KOH dan ZnCl₂ is a proven activating agent in activated carbon synthesis to produce a product with large surface area. Therefore, this research used KOH dan ZnCl₂ as an activating agent in activated carbon synthesis from palm shell.

2. Material and Method

2.1. Carbonization

Palm shell is used as feedstock in activated carbon synthesis. Early stage of this research is conducting carbonization at 400°C for two hours. Carbonization process resulted a palm shell with no water content and volatile matter. Palm shell ash then crushed with a mortar and sieved by a mesh equipment to produced small particles, which sized 1 – 2 mm. Smaller particle will create larger surface area and more activated pores.

2.2. Physical Activation and Chemical Activation

Carbon which sized at 1 – 2 mm then activated by using KOH solution which consist of 75% KOH solution and 25% ZnCl₂ solution, as an activating agent. Composition ratio between carbon and activating agent is 1:4 respectively. Activation process done in mixing speed at 100 rpm, and 85°C temperature for two hours. Resulting a slurry which will be dried at an oven with temperature at 100°C for 24 hours. Activated carbon produced from chemical activation will be activated by physical activation using Nitrogen (N₂) gas at 850°C for an hour. Activation is done by gradual heating in order to reach desired temperature. N₂ gas flow at 100 cc/minute rate. After an hour, physical activation resumed by flowing CO₂ gas in an hour at 850°C temperature. Subsequently, activated carbon cooled by flowing CO₂ into the reactor, to remove free oxygen in the reactor. Removing free oxygen is done to prevent damage to product pores which can result losses in the end activated carbon product. Later, activated carbon will be washed by 0.1 M HCl solution until it reached neutral pH. Once, neutral pH reached, activated carbon will be washed by aquadest to remove the remaining of chlorides. Activated carbon then resized to a smaller size such as 212-150 µm, 150-106 µm, 106-50 µm, 50-37 µm and <37 µm.

Activated carbon will be characterized based on Indonesia's industrial standard. Characterization covers amount of activated carbon yield, iod numbers, water content, ash content, and volatile matter

which was removed in heating process. Converting iodine number into surface area can be done by a regression equation is used that refers to ASTM D-4607-94. The equation is as follows;

$$\text{Iod Numbers} = (0,6366 \times \text{specific area}) + 174,34$$

3. Results and Discussion

3.1. Carbonization of Palm Shell

Carbonization process took place at 400°C temperature in two hours. From the process, activated carbon yield is shown in Table 1. From the table, carbonization process results an ash yield averaging at 38%.

Table 1. Palm Shell Carbonization Yield

No	Initial Mass (gram)	Final Mass (gram)	Yield (%)
1	587	218	37,2%
2	625	253	40,5%
3	502	181	36,2%
Average			38,0%

Carbonization yield shows that there were a lot of volatile matter consisted in palm shell. Therefore, there were a lot of mass reduced in carbonization process. Ash yield can also be indicated as fixed carbon content in palm shell.

3.2. Chemical with KOH as an Activating Agent and Physical Activation

KOH selection as activating agent is based on its capability on opening pores in carbon surface. Pore formation in chemical activation process will be maintained in physical activation by heating. Activated carbon's surface area will increased as a result in an increase of pore formation. Physical activation is a heating process in a high temperature without a presence of free oxygen, which can be done by flowing Nitrogen gas to the reactor during the activation process and in the cooling process by flowing of Carbon Dioxide gas.

Yield of activated carbon is shown in Table 2. Average yield of activated carbon is 94.3%. Yield percentage shows the amount of activated carbon formed from physical activation process.

Table 2 Activated carbon yield from physical activation process

No.	Initial Mass (gram)	Final Mass (gram)	Yield (%)
1	100	94,3	94,0%
2	100	95,0	94,2%
3	101	96,1	94,5%
	101	95,7	94,7%
Average			94,4%

High activated carbon yield is caused by its feed which has been carbonized, and subsequently, has removed most of its volatile matters.

3.2.1. Water and Ash Content

Water content percentage in the activated carbon is 13.6%. According to Indonesia's Industrial Standard, maximum water content in an activated carbon is 15%. Therefore, activated carbon from palm shell

meets the criteria from Indonesia's Industrial Standard (SII). In another parameter, ash content in activated carbon produced from palm shell is 9.4% and meet the regulatory standard from SII, which stood at 10%.

3.2.2. Surface Area Calculation with Iodine Numbers Method

Activated carbon quality can be determined from its absorptivity of iodine. Absorptivity of an activated carbon related to iodine solution indicating activated carbon's capability to adsorb component with low molecular weight. Activated carbon with a high capability of iodine absorption indicates more surface area with larger mesoporous and micro-structure. Correlation between iodine number and specific surface area can be determined by linear regression in ASTM-4607-94. Figure 1 shows specific surface area based on activation treatments.

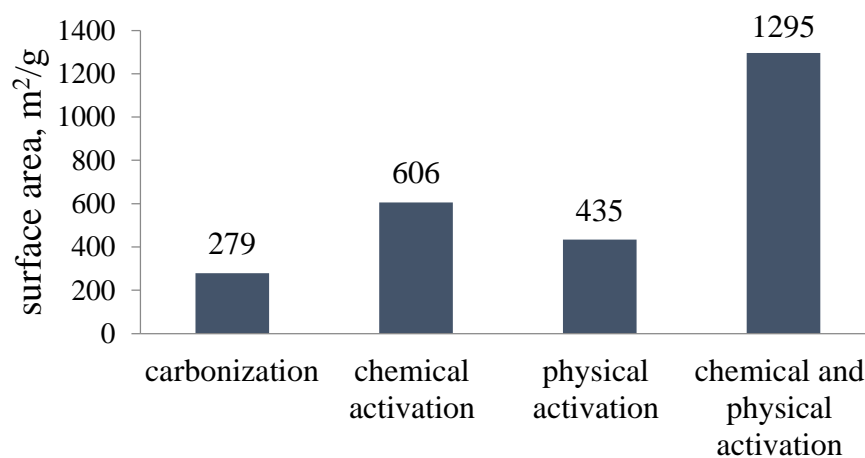


Figure 1. Effects of treatment to Specific Surface Area with KOH as activating agent

From Figure 1, activation treatments increase specific surface area. Ash with no activation process will have the least specific surface area. Basically, carbonization process creates pores in the process, but, the number of pores formed is relatively low if compared to ash with activation process. Less pore will result reduced capability in absorbing iodine properly, iodine absorbed by the ash is 354 mg/g with smaller specific area, 279 m²/g. Activation process affect specific surface area formation, more complex of activation process can result more pores formed, therefore activated carbon can absorb more iodine and have larger specific surface area. While, the effect of activated carbon particle size on surface area can be observed from Figure 2.

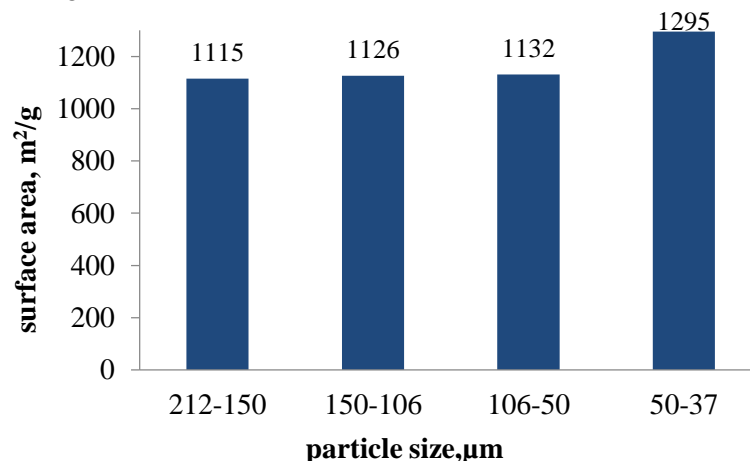


Figure 2. Effect of activated carbon particle on surface area with KOH as activating agent

Different activation treatment in activated carbon sample has significant effect on specific surface area. But, variation in activated carbon size proved not to be a significant factor on specific surface area. This is due to pores has been formed at physical and chemical activation process, thus resulting a similar pore size. Differences in specific surface area is due to smaller size of particle. Smaller size of particle will create more particles, thus, increasing the contact of carbon with the iodine. More contact between iodine and carbon will result increasing iodine number and surface area. But, contact between carbon and iodine won't be done in very fine particle size, because activated carbon will spread across iodine solution's surface. In a very small size, activated carbon will saturated rapidly and won't be able to absorb matters in iodine solution, or have passed its saturated limit, resulting to-be-absorbed matter to diffused out of particle and reducing iodine number.

3.3. Chemical with $ZnCl_2$ as an Activating Agent and Physical Activation

After reducing particle size, palm shell will be activated chemically by adding activating agent, such as $ZnCl_2$. $ZnCl_2$ is chosen due to its acid characteristic, thus, able to open pore in carbon surface by scraping impurities.

Ratio of activating agent and carbon feedstock is 1:4 respectively, and the ratio of aquadest to carbon feedstock is 4:1, respectively. Mixture then stirred and heated by using magnetic stirrer at 85°C for two hours. Later, drying process is done in the oven, in order to evaporates remaining water content available.

Then, activation of activated carbon can be done by heating at 850°C without free-oxygen presence. This condition can be reached by flowing carbon dioxide and nitrogen gas to the reactor during the activation process. Presence of free oxygen can make a damage to the carbon pore, and produce losses subsequently. Table 3. will show percentage of ash yield:

Table 3.Yield from Activation Process

No.	Initial Mass (gram)	Final Mass (gram)	Yield (%)
1	100	78,4	78,4
2	100	76,7	76,7
3	100	77,9	77,9
4	100	78,1	78,1
Average			77,8

Activation process results reduced mass, results are due to water content and impurities that has been eliminated by heating process.

3.3.1. Water and Ash Content

Water content on the activated carbon was measured at 14.5% and meet SII standard which stands at 15%. Whereas, ash content on activated carbon measured at 9.0% and also meet regulatory SII standard which stands at 25.0%.

3.3.2. Surface Area Calculation with Iodine Numbers Method

Activated carbon quality can be determined from its iodine absorptivity. Absorptivity of an activated carbon related to iodine solution indicating activated carbon's capability to adsorb component with low molecular weight. Activated carbon with a high capability of iodine absorption indicates more surface area with larger mesoporous and micro-structure. Figure 3. shows an influence of activation treatment to specific surface area. Highest surface area is obtained from physical and chemical treatment with 743 m²/gram. In contrary, the least surface area is obtained from carbon without any activation with 275 m²/gram.

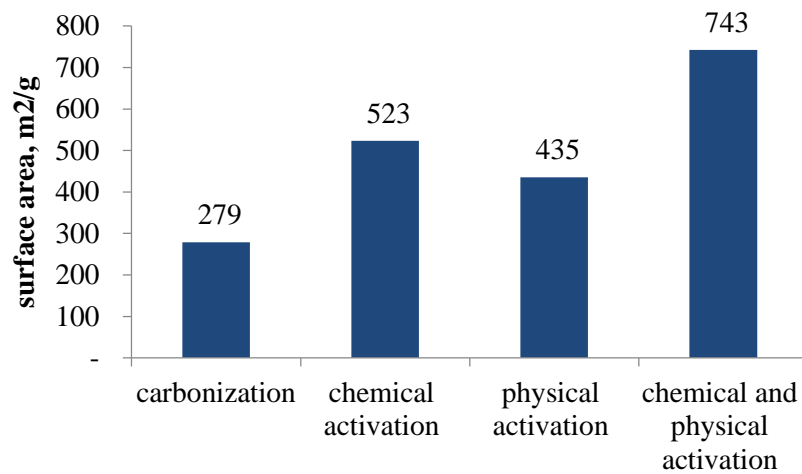


Figure 3. Effect of activation treatment to specific surface area

Figure 4 shows the influence of particle size to activated carbon surface area. Highest surface area is obtained from 50-37 μm particle with 743 m^2/gram . In general, particle size has a little effect on activated carbon surface area. Smaller the particle, the surface area will be larger. Thus, the most optimum particle size is 50-37 μm .

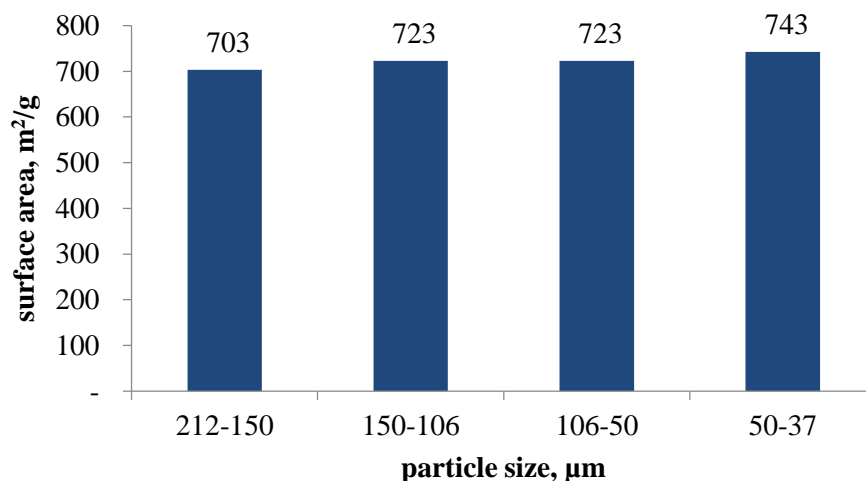


Figure 4. Effect of particle size to surface area

Results shown that activated carbon resulted from activation by KOH dan ZnCl_2 can increase iodine absorptivity. Significant increase is observed in iodine absorptivity between activated carbon without activation and with KOH dan ZnCl_2 activation. Increase of absorptivity is due to new pores formation across carbon surface. During activation, activation agent will form new pores by creating holes in the surface. Amorphous carbon blocked pore reaction in early oxidation step, so closed pores will open and form new pores. Results show that KOH activation agent is more effective in increasing surface area rather than ZnCl_2 .

4. Conclusion

Conclusion obtained from research results, data processing, and analysis can be shown as below:

- Activated carbon produced from the research meet the standard from Standar Industri Indonesia (SII)
- Activated carbon with palm shell as a feedstock has carbon yield averaging at 38%
- Activation by using activating agent KOH with physical activation at 850°C, results activated carbon with specific surface area of 1295.20 m²/g, 13.6% of water content, and 9.4% of ash content.
- Activation by using activating agent ZnCl₂ with physical activation at 850°C, results activated carbon with specific surface area of 743 m²/g, 14.5% of water content, and 9.0% of ash content.

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