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# Preparation of Activated Carbon from Palm Shells Using KOH and $ZnCl_2$ 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 $ZnCl_2$. Then the physical activation is done by flowing $N_2$ gas for 1 hour at 850°C and followed by gas flow through the $CO_2$ 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 $ZnCl_2$. 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 $m^2$/g. While the use of $ZnCl_2$ 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 $m^2$/g.

## 1. Introduction

One of the purification method of the byproducts of petroleum refinery yields off gases is by adsorbing the impurities using activated carbon as a biosorbent [1]. Activated carbon is a biosorbent with high adsorption capabilities, carbonization-processed and chemically or physically activated. According to the structure pattern, activated carbon is an amorphous carbon consisted mostly of free carbons and has inner surfaces; resulting the high adsorption capabilities compared to other adsorbents [2]. Organic as well as inorganic compounds containing lignin, hemicelluloses, and cellulose can be utilized as the raw material of producing activated carbon because of the high effectivity for adsorption process. One of the highly potential and currently developed raw material for making activated carbon are palm shell.

Palm shell contains 26.6% cellulose and 27.7% hemicelluloses, which are good to be utilized as activated carbon. Palm shell is one of the largest waste produced from palm oil refinery, achieving 12% weight of palm fruit. The average production of palm fruit per year is 5.6 million tones, meaning there could be around 672,000 tonnes of shells produced. With the availability of the waste, an advanced process is needed to convert the palm shell waste into a high economical valued product [3].

The quality of activated carbon that can be converted into biosorbent on pollutant adsorption is strongly affected by the production condition. Parameters affecting activated carbon production process are dehydration, carbonization, and activation. On the other hand, adsorption process affectivity is strongly affected by several parameters including adsorption properties, temperature, contact time, and surface area. Previously, there has been a lot of method producing activated carbon from organic materials but the carbon produced has yet achieved the required standard. Hence, this research aims on producing an activated carbon that has a good characteristic as a biosorbent.

Activation using activating agent on activated carbon production has been done by adding several chemicals like NaOH, $H_3PO_4$, dan $ZnCl_2$. The usage of $H_3PO_4$ produced activated carbon with the surface area of 438.9 $m^2/g$ [4]. The usage of KOH as an activating agent can produce activated carbon with the surface area of 3000 $m^2/g$ [5]. On the other hand, usage of $ZnCl_2$ as an activating agent can produce activated carbon with the surface area of 1100 $m^2/g$ [6]. Activating agents KOH and $ZnCl_2$ are activating agents that can potentially produce activated carbon with a high surface area. Therefore, this research utilized activating agents KOH and $ZnCl_2$ in the production of activated carbon from palm shells.

## 2. Experimental

### *2.1. Carbonization*

In the research, palm shells are used as the raw material of activated carbon. The research starts by doing a carbonization on the temperature of 400°C for 2 hours. The outcome of carbonization is palm shell charcoal that has been deprived of water content and volatile matters. Palm shell charcoal then shattered using mortar. The process continues to sieving with mesh to yield smaller sized particles (1-2 mm). Smaller particle size increases the charcoal's surface area, expected to increase the number of activated pores.

### *2.2. Chemical and physical activation*

Carbons with the size of 1-2 mm is then chemically activated with activating agents KOH (75% KOH solution) and (25% $ZnCl_2$ solution). The mixing composition of activating agent and carbon is 1:4 (carbon : activating agent). Activation process is done with the stirring speed of 100 rpm, temperature ± 85°C for 2 hours. Activated carbon from the chemical activation then physically activated by using $N_2$ gas on 850 °C for 1 hour. Activation is done by staged heating in order to reach the desired activation temperature. $N_2$ gas is channeled with the flow rate of 100 cc/min. After 1 hour, physical activation is continued with channeling $CO_2$ gas for 1 hour on the temperature of 850°C [8]. After the activation process is finished, the activated carbon is cooled with channeling $CO_2$ gas into the reactor. The activated carbon yielded is then washed with 0,1 N HCN solution until it reaches neutral pH. The activated carbon is then washed with distilled water to get rid of the chloride. The size of the activated carbon is then reduced into 212-150 µm, 150-106 µm, 106-50 µm, 50-37 µm.

Dried activated carbon is characterized based on Indonesian Industrial Standard. Characterization includes activated carbon yield gain, iod number test, water content test, ash content test and losses from heating. To change the iodine number into surface area a linear regression equation is used that refers to ASTM D-4607-94 [7]. The equation is as follows

$$\text{Iod Number} = 0.6366 \times \text{surface area} + 174.34 \tag{1}$$

## 3. Results and Discussion

### *3.1. Carbonization of palm shell*

Carbonization process occur on 400°C for 2 hours. The results of palm shell carbonization is displayed as yield percentage data on Table 1. From the table we can conclude that the process produced charcoal with the average yield percentage of 38%. Charcoal yield shows that volatile matters contained on the palm shell is quite high, causing large mass loss through the carbonization process. Charcoal yield produced is also an indication of the number of fixed carbon contained on palm shell.

**Table 1.** Palm shell carbonization yield

| No | Initial Mass (g) | Final Mass (g) | Yield (%) |
|---|---|---|---|
| **1** | 587 | 218 | 37.2 |
| **2** | 625 | 253 | 40.5 |
| **3** | 502 | 181 | 36.2 |
| | **Average** | | 38.0 |

*3.2. Chemical activation with activating agent KOH and physical activation*

Choice of KOH as activating agent is based on the ability to open the pores of the carbon. Pores formation on the chemical activation is then completed on the physical activation through heating. The more pores formed, the more surface area produced on the activated carbon. Physical activation is a heating process on high temperature without the presence of free oxygen, by channeling $N_2$ gas into the reactor throughout the activation process, continued by channeling $CO_2$ gas functioning as cooler gas. Yield percentage gained is presented on Table 2. The average yield percentage is 94.3%, showing the number of activated carbon formed from physical activation process. The high activated carbon content is produced because of a carbonization process prior activation; removing volatile matters and isolating the material from air, thus producing high yield percentage [9].

**Table 2.** Physical activation process activated carbon yield (KOH)

| No | Initial Mass (g) | Final Mass (g) | Yield (%) |
|---|---|---|---|
| **1** | 100 | 94.3 | 94.0 |
| **2** | 101 | 95 | 94.2 |
| **3** | 101 | 96.1 | 94.5 |
| **4** | 101 | 95.7 | 94.7 |
| | **Average** | | 94.4 |

*3.2.1. Water content and ash aontent*

Water content percentage of the activated carbon is 13.6%. According to Indonesian Industrial Standard, the minimum water content of activated is 15%. Therefore, the activated carbon produced from palm shell in the research has met the Indonesian Industrial Standard's criteria of water content. Ash content percentage of activated carbon on the research is 9.4%, which has met the Indonesian Industrial Standard's criteria of ash content of 10%.

*3.2.2. Surface area using Iod number method*

Activated carbon quality can be observed from the adsorption ability towards iodine. Activated carbon's adsorption ability towards iodine solution indicates its ability to adsorb components with low molecular weight. Activated carbon with high iodine adsorption ability has larger surface area and has larger micro- and mesoporous structure. There is a correlation between iod number and specific surface area of activated carbon, which can be determined using linear regression equation referencing on ASTM-4607-94. Figure1shows specific surface area based on activation treatment.

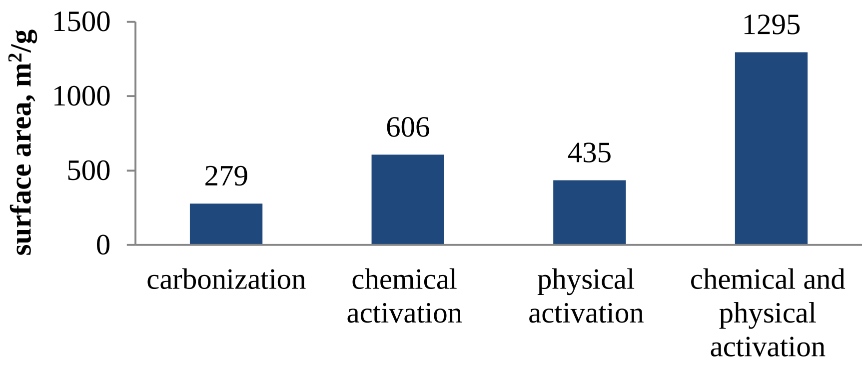


**Figure 1.** Effect of treatment on specific surface area (activating agent KOH)

From Figure 1, it can be seen that activation treatment increases specific surface area. Charcoals without activation has the smallest specific surface area. The principle of carbonization is to form pores, but the number of pores formed is still small compared to activation process. Small number of pores causes charcoal to be unable of adsorbing iodine solution effectively. The number of iod adsorbed is 352 mg/g and the surface area of the activated carbon is also small, which is 279 $m^2/g$. Activation process greatly affects the formation of surface area of activated carbon, the more complex the activation process will form more pores so that the activated carbon can absorb more iodine and the surface area formed will be greater. On the other hand, the effect of activated carbon particle size on surface area can be seen at Figure 2.

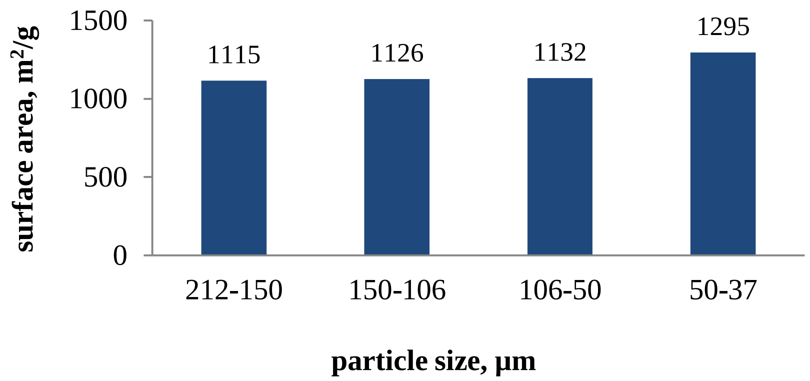


**Figure 2.** Effect of activated carbon particle size on specific surface area (activating agent KOH)

The difference in activation treatment on activated carbon sample affects the specific surface area significantly. But on the variation of the particle size, the specific surface area formed is not so different. This is caused by the formation of pores from the physical and chemical activation process, thus the variation of the pore quantity is uniform. There is a little difference from the specific surface area formed, because the smaller the particle size thus the number of particle will be greater and the contact with iodine solution will be more intense. The more particles contacted with iodine solution will cause an increase on iod number and surface area. But on the fine particles, the contact between activated carbon and iodine solution is not going smoothly because the carbon is on the surface of iodine solution.

### *3.3. Chemical activation with activating agent $ZnCl_2$ and physical activation*

After size reduction, the palm shell will next be activated chemically by adding activating agent solution, $ZnCl_2$. Choice of $ZnCl_2$ is because it is acidic thus has the ability of opening pores on carbon by removing impurities on carbon. The process after wards is carbon activation by heating on 850°C with the absence of free oxygen by channeling $CO_2$ and $N_2$ gas into the reactor through out the activation process. Yield percentage gained is presented on Table 3. Activation process cause mass loss. The quantity of mass loss percentage is a representation of water content and impurities contained on the solid mixture between activating material/raw material and volatile matters that are still present, thus can be removed throughout the heating process.

**Table 3.** Physical activation process activated carbon yield ($ZnCl_2$)

| No | Initial Mass (g) | Final Mass (g) | 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** | | 77.1 |

*3.3.1. Water content and ash content*

Water content percentage of the activated carbon is 14.5%. According to Indonesian Industrial Standard, the minimum water content of activated is 15%. Therefore, the activated carbon produced from palm shell in the research has met the Indonesian Industrial Standard's criteria of water content. Ash content percentage of activated carbon on the research is 9.0%, which has met the Indonesian Industrial Standard's criteria of ash content of 10%.

*3.3.2. Surface area using Iod number method*

Activated carbon quality can be observed from the adsorption ability towards iodine. Activated carbon's adsorption ability towards iodine solution indicates its ability to adsorb components with low molecular weight. Activated carbon with high iodine adsorption ability has larger surface area and has larger micro- and mesoporous structure. Figure 3 shows the effect of treatment on specific surface area. Highest activated carbon surface area is gained when chemical and physical activation is done, which is 743 $m^2/g$. Meanwhile, the lowest surface area is gained on carbon without activation, which is 275 $m^2/g$.

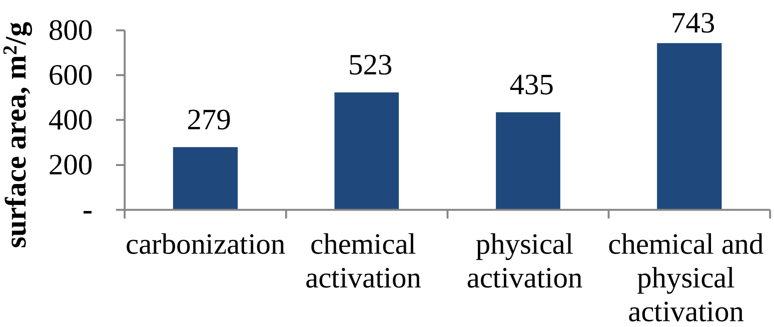


**Figure 3**. Effect of treatment on specific surface area (activating agent $ZnCl_2$)

Figure 4 shows the effect of particle size on activated carbon's surface area. Highest surface area is yielded on activated carbon with the particle size of 50-37 µm, which is 743 $m^2/g$. Particle size affects the activated carbon's surface area by a little. The smaller the particle size, the larger the surface area. Therefore, from the results gained we can get that the most optimal particle size of activated carbon in adsorbing iodine is 50-37 µm.

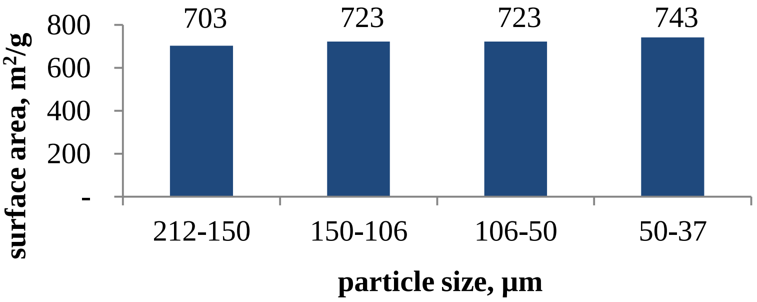


**Figure 4**. Effect of activated carbon particle size on specific surface area (activating agent $ZnCl_2$)

Research results show that activated carbon using KOH and $ZnCl_2$ can increase iodine adsorption ability (showing surface area). There is a high iodine adsorption ability increase between carbon without and activated carbon using KOH and $ZnCl_2$. This shows that in the activation process, the activating agent forms new pores so that the adsorption ability increases. Amorphous carbons prevent pores from reacting through early oxidation stage, resulting the opening of closed pores and forming new pores. Research results show that KOH is more effective on increasing the surface area than $ZnCl_2$ [10], shown by the surface area produced on activated carbon.

## 4. Conclusion

From the research results, data processing, and analysis; we can conclude that an activated carbon produced from the research has met the Indonesian Industrial Standard with carbon based on palm shell has a carbon yield of 38%. Chemical activation with activating agent KOH and physical activation on 850°C yields activated carbon with the specific surface area of 1295.20 $m^2/g$, water content of 13.6%, ash content of 9.4%. Chemical activation with activating agent $ZnCl_2$ and physical activation on 850°C yields activated carbon with the specific surface area of 743 $m^2/g$, water content of 14.5%, ash content of 9.0%.

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