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# Characterization of Activated Carbon from Melinjo (*Gnetum gnemon*) Shells with Chemical-Physical Activation

1st Apriliana Dwijayanti
*Chemical Engineering Department*
*University of Serang Raya*
Serang, Indonesia
apriliana.d@gmail.com

2nd Safril Kartika
*Chemical Engineering Department*
*University of Serang Raya*
Serang, Indonesia

3rd Slamet
*Chemical Engineering Department*
*University of Serang Raya*
Serang, Indonesia

4th Yuliusman
*Chemical Engineering Department*
*University of Serang Raya*
Serang, Indonesia

***Abstract*—The synthesis of activated carbon derived from biomass melinjo shells has been carried out This research makes activated carbon from the shell of melinjo with active chemicals and chemical-physics so that it can distinguish the activated carbon produced. The carbonization process was carried out at 400ºC, for 15 minutes. The process continues by activating the chemical with a 65% KOH activator by returning the activator mass and activated carbon 4: 1. The activated process is carried out by immersion 24 hours then stirred at ± 110 ºC for 5 hours. Furthermore, physics was carried out at variations in temperature of 500 ºC, 550 ºC, 600 ºC and 650 ºC for 1 hour. Based on the results of research on activated carbon with active physics at a temperature of 550ºC, the best results were obtained by producing an iodine number of 695.53 mg / g, water content of 4.38%, ash content of 5.955% and area of 412.33 $m^2$ / g.**

***Keywords: melinjo, activation, coal, pore***

## I. INTRODUCTION

At present many material researchers are interested in synthesizing activated carbon specifically those derived from biomass due to the high content of lignin, cellulose and hemicellulose. Many studies use biomass as an activated carbon material. Some researchers have used various carbon sources such as rice husks, durian skins, corncobs, corn stalks, coconut fibers, coconut shells, palm shells, peanut shells, and others [1]. Other sources include used tires [2], lignin [3], cocoa shells [4], siriguela seeds, and grape seeds [5]. Coffee waste is also used as activated carbon for adsorption of $SO_2$, $H_2S$, methane, α-lactalbumin, serum albumin bovin, and organic dyes.

Melinjo shell structure is similar to peanut shells which have been widely used as activated carbon consists of cellulose, hemicellulose, and lignin. This indicates that the melinjo shell has the potential as an active carbon material. Data from the Indonesia Statistics Agency (BPS) in Indonesia melinjo production in 2016 reached 203,625 tons, the highest production in Banten 34,874 tons followed by West Java 33,360 tons [6]. The weight of melinjo shells is around 15% of the weight of the whole melinjo, so the quantity of melinjo shell waste in Indonesia reaches 30,500 tons per year.

Active carbon production involves two steps: carbonization and activation. Carbonization consists of thermal decomposition of carbon materials, eliminating non-carbon species and producing fixed carbon masses with imperfect pore structures. Activation is the process of removing impurities in carbon pores thereby increasing carbon porosity [7]. Activations that can be used to make activated carbon from biomass basic materials are physical activation and chemical activation. Physical activation is carried out by heating at high temperatures while chemical activation is generally carried out by conditioning the base material with a strong dehydrating agent [8]. [9] showed that porosity developed from vegetables using physical or chemical activation was more heterogeneous than coal.

## II. METHODS

The materials used in this research were melinjo shells, sodium thiosulfate ($Na_2S_2O_3$), potassium iodide (KI), iodine ($I_2$), starch indicator, KOH and aquadest. The equipment used in this study is the furnace, desiccator, Erlenmeyer, burette, oven, stirrer, funnel, measuring flask, beaker glass, filter paper and statif. **Activated carbon preparation:** Preparation of this research can be seen in figure 1.

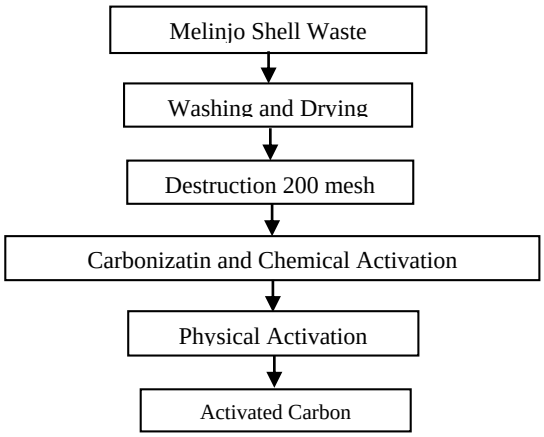



Fig.1. Diagram of activated carbon synthesis from melinjo shells

Melinjo shell waste is washed with distilled water to remove surface impurities and dried. The sample was carbonized with variations in temperature of 450ºC in the furnace for 15 minutes. The sample is then crushed and sifted so that the raw material with small particle size (± 200 mesh) is obtained. Then mixing with a variety of activating agent KOH 65% was carried out. The variation of mixing activating agent with raw material is 4/1 soaked for 24 hours then stirred at 110 rpm at ± 110 ºC for 5 hours. Then dried with an oven at 110ºC for 2 hours to release the water content. The physics activation was carried out at a temperature variation of 500ºC, 550 ºC, 600ºC and 650ºC for 1 hour . The samples is rinsed with distilled water to a pH of around 7. Activated carbon is then dried at 120ºC for one hour to release water content.

TABLE 1. VARIABLE OF ACTIVATED CARBON

| Sample | Treatment |
|---|---|
| KOH activation | Chemical activation with 65%KOH |
| +Physic 500ºC | Chemical activation 65%KOH and Physical activation at 500 ºC |
| +Physic 550ºC | Chemical activation 65%KOH and Physical activation at 550 ºC |
| +Physic 600ºC | Chemical activation 65%KOH and Physical activation at 600 ºC |
| +Physic 650ºC | Chemical activation 65%KOH and Physical activation at 650 ºC |

**Characteristics of activated carbon:**

The water content of activated carbon can be determined by oven drying. The procedure for calculating activated carbon content uses the SNI standard No. 06-37301995.

$$\%water = \frac{s-p}{p} \times 100\% \quad (1)$$

with the sample initial weight (s) and the final weight of the dried sample (p). Calculation of total ash content of activated carbon using SNI standard No. 06-37301995.

$$\%ash = \frac{ash\ weight}{sample\ weight} \times 100\% \quad (2)$$

The iodine test was carried out to determine the active surface area of activated charcoal based on the absorption of iodine in solution**.**

$$I_{absorb} = \frac{\left(H - \frac{b \times a}{Ni}\right) \times 126.9 \times N}{W} \quad (3)$$

with volume of filtrate (H), volume of titrant (b), normality of $Na_2S_2O_3$ (a), normality of $I_2$ (Ni), normality of $Na_2S_2O_3$ (0.1 mgrek / mL) (N).

Calculation of surface area were analyzed using the Brunnauer-Emmett-Teller (BET) method, and SEM. The instrument that used in BET method is the Autosorb Quantachrome Instruments Nova 3200e surface area analyzer with nitrogen as a gas analyzer. The main features of the SEM are an electron source which provides the electrons that nteract with the material to be examined, an arrangement of metal apertures, magnetic lenses and scanning coils or deflectors plates that confines, focuses and turns the beam of electrons into a thin and focused monochromatic beam which is accelerated towards the sample and which irradiates the specimen in a raster fashion [10]

## III. RESULTS AND DISCUSSION

### *A. Analysis of water content*

The analysis of water content is carried out to determine the water content in activated carbon after activation. Table 2 shows the effect of the chemical-physics activation compared to chemical activation. Activated carbon with chemical activation have a highest moisture content 5.19%. The lowest water content 3.55% occurs at 650ºC of chemical-physics activation. The high active water content of carbon means the larger bound water content caused by the structure of activated carbon which is composed of 6 C atoms at each hexagonal angle allowing the water grains to be trapped in it and at high temperatures the grains of loose water so that the water content becomes low [11].

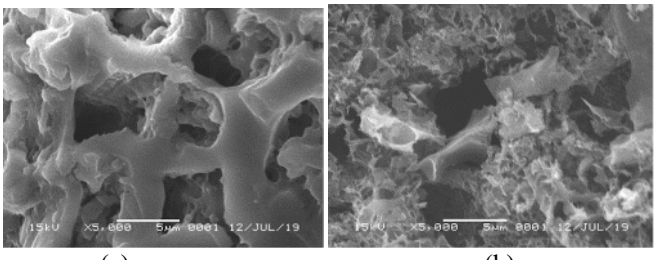
TABLE 2. CHARACTERISTIC OF ACTIVATED CARBON

| Samples | Water content (%) | Ash content (%) | Iodine Absorption (mg/g) | Surface Area ($m^2/g$) |
|---|---|---|---|---|
| KOH activation | 5.19 | 3.78 | 583.88 | 201.06 |
| +Physic 500ºC | 4.53 | 5.72 | 590.21 | 238.93 |
| +Physic 550ºC | 4.38 | 5.95 | 695.53 | 412.33 |
| +Physic 600ºC | 3.86 | 6.57 | 663.87 | 389.72 |
| +Physic 650ºC | 3.55 | 6.89 | 594.63 | 215.12 |

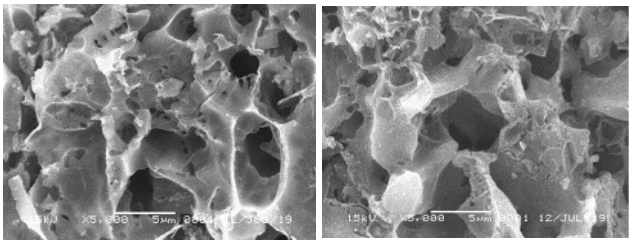
### *B. SEM analysis*

SEM images of activated carbon samples are shown in Figure 2. It can be seen from the micrographs that the external surface of the activated carbons has cracks and crevices in various sizes. The porous structure was formed in all samples because the organic volatiles were evolved.

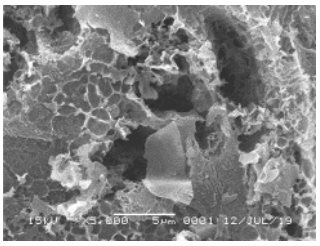
In chemical activation (figure 2a) the oxidized carbon surface forms an imperfect pore. Physical activation (figure 2b-e) aims to widen the pore gap that has been formed by previous chemical activations. In the 500ºC chemical-physical activation the removal of volatile organic substances is not optimal so that the formation of pores is less than optimal. Chemical-physical activation of 550ºC and 600ºC produces a larger pore while the temperature of 650ºC shows that the pore formed is damaged due to high temperature.



(a) (b)



(c) (d)



(e)

Fig.2. SEM Analysis (a) KOH activation, (b) +Physic 500ºC, (c) +Physic 550ºC, (d) +Physic 600ºC, and (e) +Physic 650ºC

### *C. Analysis of ash content*

The principle of determining ash content in this study follows the Indonesian Industry Standard, which is heating at a temperature of 650ºC for 2 hours. It can be concluded from Table 2 that ash content increases with increasing temperature. High temperature made the decomposition of minerals in the form that settles as solids in activated carbon occurs [12]. The amount of ash formed will reduce the yield and can cover the pores formed.

### *D. Analysis of iodine number*

One of the parameters that used to find out the quality of activated carbon is absorption capacity $I_2$ (iodine number). As the iodine number increase, the ability of activated carbon adsorption will be better because the use of activated carbon is generally as an absorbent material. The analysis of iodine number can be seen from Table 2. The smallest iodine number found in chemical activation is 583.88 mg/g. While the largest iodine number is found in the chemical-physical activation of 550ºC which is 695.53 mg/g. Physical activation widen pore slits that have been formed by previous chemical activations. Iodine number decreased at 600ºC and 650ºC due to heat which raises oxidation reactions and leaves more residues. This high residue will reduce the ability to absorb activated carbon against iodine. Judging from the results of SEM also shows pore damage at 650ºC can cause a decrease in the iodine number.

### *E. Analysis of surface area*

The area of pore surface is a very important parameter in determining the quality of an activated carbon as an adsorbent. This is because the pore surface area is one of the factors that influence the adsorption power of an adsorbent [11]**.** The surface area factor affects the adsorption capability of activated carbon. The larger surface area, adsorption capability will higher. Table 2 shows that the surface area of activated carbon produced through chemical-physical activation results in a higher surface area than chemical activation. The largest surface area is 412.33 $m^2/g$ in chemical-physic activation sample 550ºC. At higher temperatures, the surface area has decreased due to an increase in ash content. The presence of this ash can clog the pores in the activated carbon structure thereby reducing its surface area.

## IV. SUMMARY

Melinjo shells which has been a waste, can be used as activated carbon. Metode aktifasi influences the quality of activated carbon from melinjo shells. The optimal conditions for making the best quality activated charcoal from melinjo shells are at chemical-physic activation of 550ºC. Under these conditions iodine number 695.53 mg /g, water content of 4.38%, ash content of 5.955%, and surface area 412.33 $m^2/g$.

## ACKNOWLEDGMENT

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