Characterization of Activated Charcoal Oil Palm (*Elaeis Guineensiss Jacq*) Shell Waste using SEM and FTIR: Effect of Activation Temperature

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Abstract
This study aims to determine the structure of morphology and functional groups from activated charcoal for palm oil waste with variations in activation temperature. The process of two stages: the carbonation and activation stage. Carbonation process using a pyrolysis reactor at a carbonation temperature of 200ºC -400ºC for 6 hours. Then, the sample activated at a temperature of 700 °C, 750ºC, 800ºC, and 850 °C. The samples were characterized using SEM and FTIR. The SEM analysis results show that the largest pore size at a temperature of 850 ºC with a diameter of 48.3 nm, and the lowest temperature was 700 °C with a pore size 35.9 nm. Activated charcoal from oil palm shell waste at mesopore size. The FTIR analysis results show wave numbers 2165,18 cm^{-1}, 1554 cm^{-1}, 1030,76 cm^{-1} dan 424,11 cm^{-1}. The removal of some absorption bands and the formation of new absorption bands, caused by the activation temperature.

Keywords: activation temperature, SEM, FTIR, charcoal oil palm

I. Introduction

One of the most effective adsorbents is activated charcoal, it is a multifunction material, and could be used to vanish the pollutant of gas and contaminated fluid flow as it has a large capacity of the absorption, a high surface area, suitable to develop porous structure, rapid kinetic absorption and good mechanical [1]. The charcoal surface is still covered by hydrocarbon precipitation that inhibits its activity. The while the surface of activated charcoal is relatively free of deposit and able to absorb as the surface area and has open pores [2].

The use of activated charcoal to overcome intoxication has been recognized since 1830 by a French chemist, Bertrand. Activated charcoal commonly produced through the pyrolysis process of material containing carbon and activated by oxidation with high-temperature vapor [3]. Activated carbon produced from different precursors, including coal from a different structure, and lignocellulose materials, with physical or chemical activation process [4]. Most of the material used to create activated carbon is organic materials that contain a lot of carbon [5]. Therefore, the development of a method to reuse organic waste materials like activated carbon is hugely recommended to solve an environmental problem. Agricultural waste and waste of factory production such as corn cobs, palm bunches, and palm oil shells, sawdust, cashew shells unusual to be used for activated carbon materials as it belongs to lignocellulose materials that have a high content of carbon [6].

Palm oil with the Latin name *Elaeis Guneensis Jacq* is one of the natural resources which is very rich in Indonesia. Global production of palm oil has increased more than nine times since 1980 to 45.1 million tons in 2009 where Indonesia and Malaysia lead as the producer contributes for about 85% of all palm oil production in the world [7]. For a palm oil factory production to produce raw palm oils (CPO), it provides a considerable amount of solid waste and

wastewater, which has a high impact on the environment in 2015, The produce solid waste is about 17,7 million tons [8]. One of the waste is palm oil shell. Palm oil shell is the final product of the palm oil manufacture process [9]. Palm oil is the recommended raw material candidate to produce activated carbon with developed priority and surface area under several reasons: high carbon content and price, which is relatively inexpensive. It shown that palm oil shell has 55,7% carbon content compared to palm oil fiber (49,6%), coffee bark (50,3%), and bagasse (53,1%)[10].

The research aims to discover the effect of activated temperature variation toward surface morphology and functional group of activated carbon from palm oil shell. Based on the description and data above, researchers are interested in researching the title of characterization of activated charcoal oil palm (*Elaeis Guineensiss Jacq*) shell waste using SEM and FTIR

II. Theory

The Process of Making Active Charcoal

Activated charcoal made of several materials containing carbon, both derived from animals, plants, and minerals. These materials include wood, wood sawdust, coal, coconut shells, grain shells, rice husks, animal bones [11]. Activated charcoal making process consists of three stages:.

a. Drying Phase: This stage is the stage of the water removal process. The material dried in the sun or heated material to ± 170 ℃
b. Carbonancy phase: This stage is the process of breaking down organic materials into carbon. This process is a process to convert organic material into charcoal by heating without the presence of oxygen so that the complex compounds that make up organic material decompose into charcoal with high carbon content. Temperatures above 170 ℃ will produce CO, CO_2, and acetic acid. At a temperature of 275 ℃ decompositions produces tar, methanol, and charcoal formation at a temperature of 400-600 ℃ [12].
c. Activation phase: This stage gave a treatment of charcoal which aims to enlarge the pore, namely by breaking the hydrocarbon bonds or oxidizing surface molecules so that they experience physical changes, both chemical properties and physical properties with a larger surface area and effect on absorption [13]. Activation method commonly used in the manufacture of activated charcoal there are two types of physical activation, and chemical activation described as follows: (1) This activation is the process of breaking carbon chains from organic compounds using chemicals. Activator used is chemicals such as KOH, NaCl, H_2SO_4, HCl, and H_3PO_4. (2) This activation is the process of cutting carbon chains from organic compounds with the help of heat, water vapor, CO_2, or N_2.

The quality of the surface of activated charcoal produced is very dependent on the raw material, activating material, temperature, and method of activation. The main effect of activating charcoal with hot steam is to create and expand the charcoal pore. It is clear that hot steam activation not only removes unmanaged material but is also quite useful in forming and widening micropores with rising temperatures. Increasing the temperature from 750 ^{0}C to 800 ^{0}C increases the volume of activated charcoal micropore. At a specific limit temperature increases, it will decrease the volume micropore [14]. Activated carbon mass is affected by the activation temperature. The higher the activation temperature, the lower the mass of carbon activated carbon. In addition, the higher the activation temperature of activated carbon, the more water content evaporates, which affects the quality of activated carbon [15]. It explained that temperature greatly affects the quality of activated charcoal.

Scanning Electron Microscope (SEM)

Scanning Electron Microscopy (SEM) is a type of electron microscope that uses electron beams to describe the surface shape of the material analyzed. Electrons interact with atoms that make samples produce samples that provide information about the surface topography of the sample, composition, and other properties such as electrical conductivity. SEM can produce high-resolution images from a sample surface, capture in full with a size of about 1-5 nm, and produce the desired image SEM has a considerable focus width usually 25-250000 magnification [16].

The working principle of SEM consists of electron optics and electron coulomb console. SEM sample is placed in a specimen chamber in the electron optical column with a high vacuum level of about two x 105 Torr. Electron beam generated by an electron gun will supply such as to specimen/sample. This electron beam will pass through an optic column that serves to focus the electron beam up to the sample [17].

Fourier Transform-Infrared Spectroscopy (FTIR

Fourier Transform-Infrared Spectroscopy (FTIR) is an analytical technique used to identify organic matter. This procedure measures the absorption of infrared radiation by sample material versus wavelength. Infrared absorption groups identify molecular components and structures. When a material irradiated with infrared radiation, absorbed radiation usually excites molecules into a higher vibration state. The wavelength of light absorbed by specific molecules is a function of the energy difference between the state of vibration at rest and excited wavelength absorbed by the sample is a characteristic of its molecular structure.

FTIR spectrometers use interferometers to modulate wavelengths from broadband infrared sources. The detector measures the intensity of light emitted or reflected as a function of wavelength. The signal obtained from the detector is an interferogram, which must be analyzed by computer using the Fourier transform to obtain the infrared spectrum of a single ray. FTIR spectra usually presented as a plot of intensity versus wave number (in cm^{-1}). Intensity plotted as a percentage of light transmission or absorbance at each wavenumber [18].

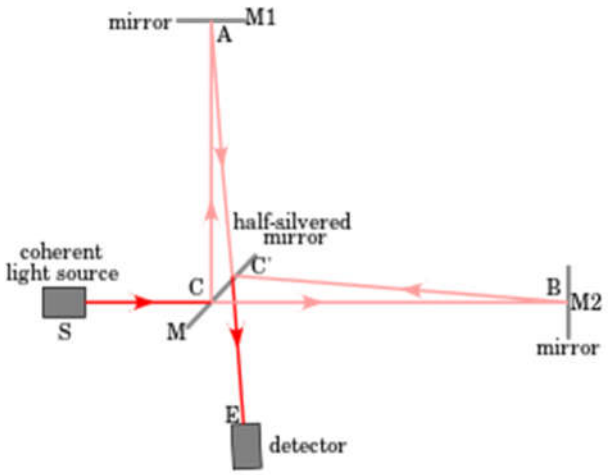


Figure 1. Michelson interferometer design[19]

FTIR usually based on The Michelson Interferometer Experiment Setup, an example shown in Figure 1. The interferometer consists of a board splitter, a framed mirror, and the mirror that translates back and forth, very precise. The beam splitter made of special material that transmits half of the radiation struck and reflected the other half. Radiation from the source hits the beam breaker and separates into two beams. One beam transmitted through a beam divider to a fixed mirror and the second reflected from the beam divider to the moving mirror. Fixed and moving mirrors reflect radiation beams to the breaker. Again, half of the repeated radiation transmitted and half reflected at the beam splitter, producing a passing beam and a second detector to go back to the source [20].

III. Methodology

Raw materials

This study uses materials that are oil palm shells taken from Plasma Jaya Village, Polinggona District, Kolaka Regency. The tools used in this study include glass tools, oven, 100 mesh sieve, carbonation tube, infrared thermometer, electric furnace, and mortar. Characterization sample using (Scanning Electron Microscope and Fourier Transform Infrared Spectroscopy).

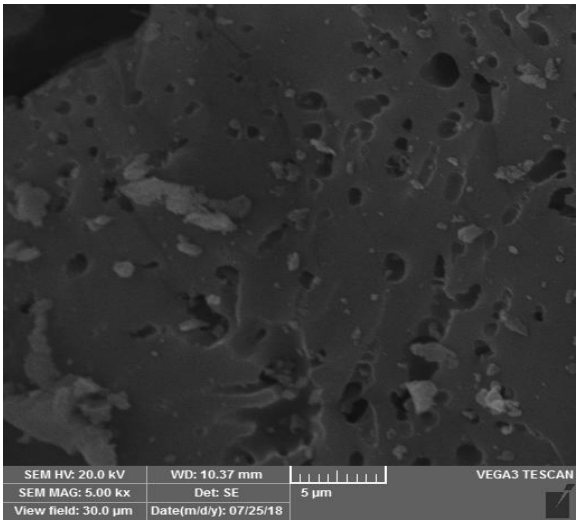
Preparation and Characterization of Charcoal Oil Palm

The oil palm shell that will sample cleaned and washed with water until it is clean and then dried in the sun for 24 hours. Next, the sample is carbonated using a pyrolysis reactor for 5-6 hours with a temperature of 400 °C – 500 °C. Then the sample is crushed using a mortar and sieved using a 100 Mess sieve. Furthermore, charcoal activated by steam of carbon dioxide (CO_2) or oxygen (O_2) using an electric furnace at temperature variations of 700 °C, 750 °C, 800 °C and 850 °C for 60 minutes and the last sample characterized by using Scanning Electron Microscope (SEM) and Fourier Transform Infra Red (FTIR).

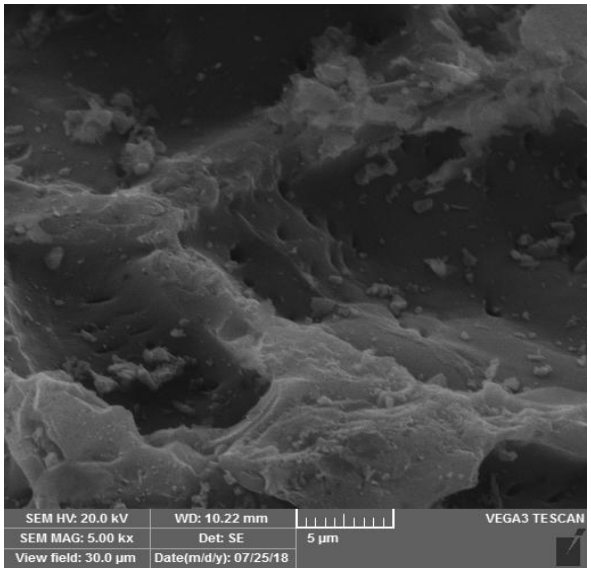
IV. Results and Discussion

The Effect of Activation Temperature Variations on Activated Charcoal Surface Morphology

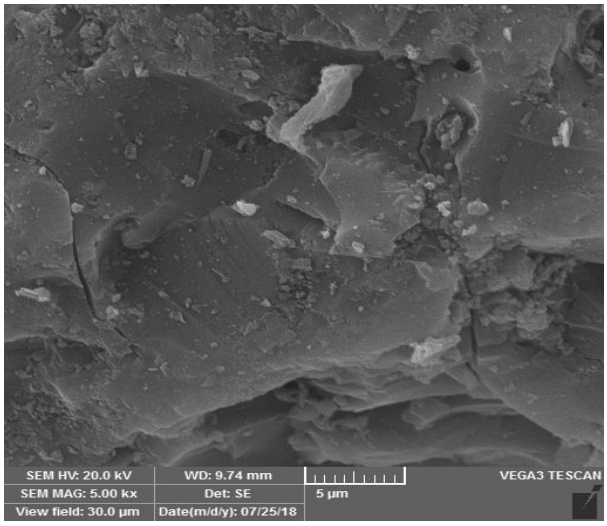
The active charcoal of oil palm shell that activated with varying temperatures of 700ºC, 750ºC, 800ºC, and 850ºC analyzed using Scanning Electron Microscope (SEM) with 5000x enlargement as shown in Figure 2.



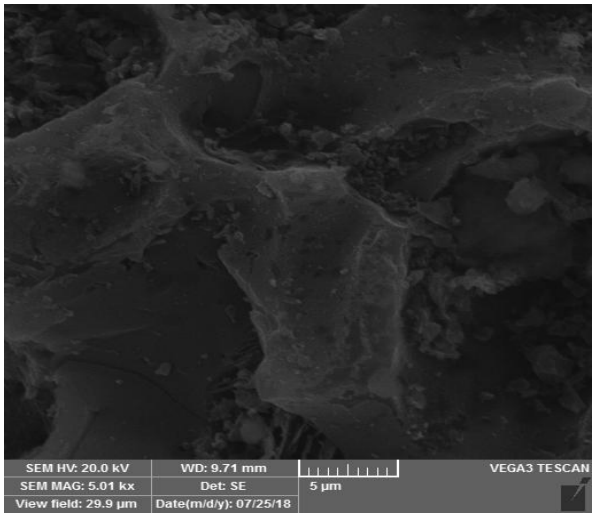
(a)



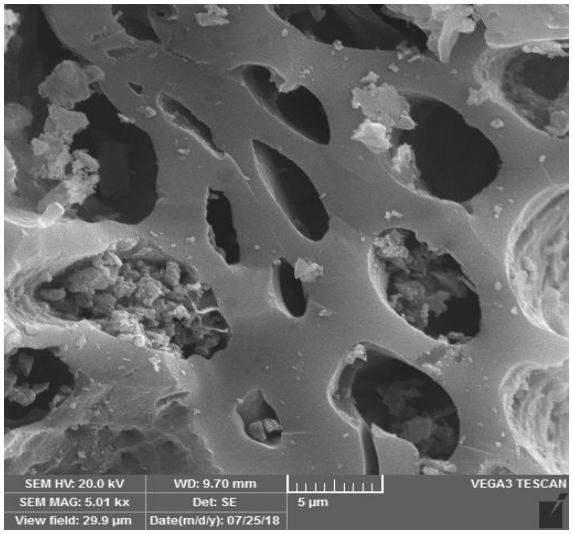
(b)



(c)



(d)



(e)

Figure 2. Active charcoal of oil palm that is the characterization results by SEM (a) without activation; (b) activation of 700 ºC; (c) activation of 750 ºC; (d) activation 800 ºC; (e) activation of 850 ºC.

Based on Figure 2, the results of the SEM characterization show that the surface structure of activated charcoal morphology shows differences in pore size images at each activation temperature. In this confirmed from the results of ImageJ analysis that shows differences in pore size distribution at each temperature activation (Table 1). Jawad said that the pore activation process has a different surface structure with features of rough and irregular surfaces with heterogeneous cavities that are distributed randomly throughout the pore surface [21]. In Figure 2e with an activation temperature of

850 ºC showed an apparent pore size of 48.3 nm pore size than the activation temperature of the others. Because of the effect of activation temperature, which resulted in the evaporation process of volatile matter from the raw material of the activation process [22][23]. The activation temperature has caused the elemental composition reduced and produces some gas products such as CO, CO_2, and hydrogen and methane [24]. For more details on the differences in the pore size of any activation, temperature difference presented in Table 1 and Figure 3.

Table 1. The average pore size of active charcoal determined on Image-J analysis

Activation temperature	Pore size (nm)
No activation	35.3
700 ºC	35.9
750 ºC	38.4
800 ºC	39.0
850 ºC	48.3

Table 1 provides information that affects the activation temperature effect on the pore size distribution at each temperature activation. Table 1 interpret that as temperature increases resulting activation of the pore size the higher (Figure 3). In this line with the expression Nurdiansyah and Diah that the higher the activation temperature of the activated carbon produced diminishing of the number of impurities that cover the surface of the pores so that the resulting pore sizes getting bigger[25]. Next, Quach et al. explained that there was an increase in pore surface size on carbon xerogels at activation temperatures 700 ºC-1000 ºC [26].

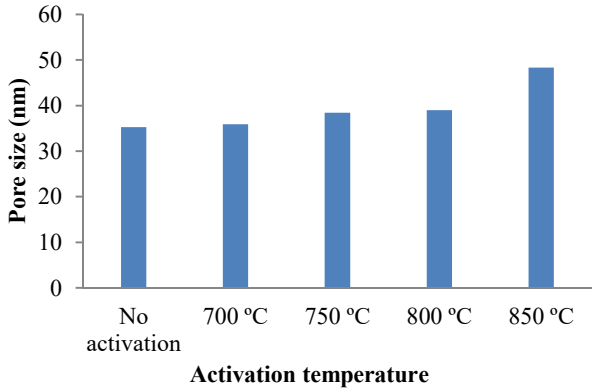


Figure 3. The effect of the activation temperature on the activated charcoal oil palm pore

The results of characterization by SEM analyzed using Image-J showed that the best pore surface area was at temperatures of 850ºC (Figure 2e) with a pore surface area of 48,262 nm. In this because the activation process can enlarge the pores by breaking hydrocarbon bonds or surface oxidizing surfaces that change the properties of activated charcoal, both physically and chemically and can increase the size of the surface area [27]. From the results of the analysis of the active pore size of oil palm shells in the mesoporous category (Table 1), in this based on the mesopore size scale, which ranges from 2-50 nm [28]. Margaret et al. and Herawan et al. suggest that the average pore size for palm oil active charcoal samples is in the micropore and mesopore categories[28][29].

The Influences of Activation Temperature on the Function Cluster Activated Charcoal

FTIR analysis aims to find out the number of particles in a material, as shown in Figure 4. FTIR shows the pattern of IR absorption in the graphical form of the connection between wave numbers and the quantity of IR transmissions. With the intake on the particular wavenumber domain, it shows that there is a vibration of function cluster.

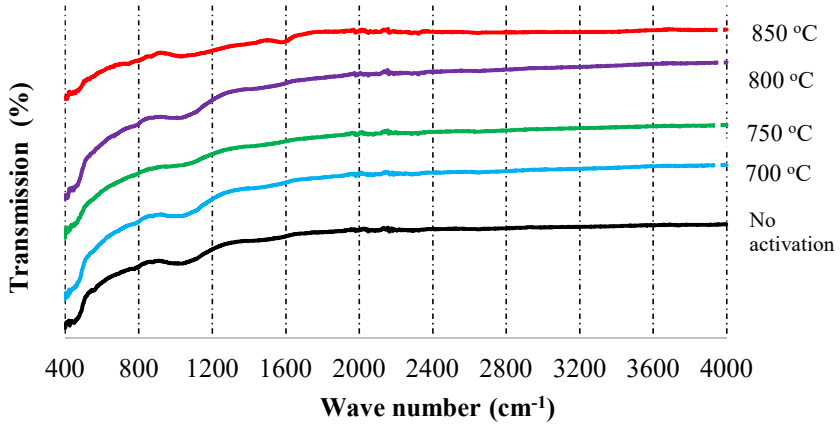


Figure 4. IR spectrum of activated charcoal from palm shells with activation temperature variations

FTIR absorption pattern Figure 4 shows that the charcoal of oil palm tree (non-activation) there is absorption in the wavenumber domain of 2165,18 cm^{-1}, which suspected as cluster vibration of alkyne. The absorption in wavenumber domain of 1554 cm^{-1} which is allegedly caused by the vibration of C=C (aromatic), absorption in wavelength domain of 1030,76 cm^{-1} which is reportedly caused by C-O (secondary alcohol) and absorption in wavenumber domain of 424,11 cm^{-1} which suspected as cluster vibration of C-H. The identification results of FTIR show that the charcoal of oil palm tree (non-activation) contains function cluster of Alkyne, C=C, C–O, and C-H. While, The activated charcoal of oil palm tree for variation of activation temperature suspected of containing function cluster of Acetylene, C–H (Alkyne), C=O, C=C, C-O, and C-H. In this expected due to heteroatoms (in this case, the oxygen atoms of carbon dioxide-enriched atmosphere during the activation process) on the sheet or in a cluster of aromatic carbon [30]. The charcoal function cluster of oil palm shell with a variety of activation temperatures seen in the following Table 2.

Table 2 Charcoal functional cluster and activated charcoal in oil palm shell

Activation Temperature	**Wave number (cm^{-1})**						
No activation		2165,18			1554	1030,7	424,11
700 °C		2188,34			1515	1037	427,18
750 °C		2286,07			1521	1038	411,21
800 °C	2324,04		2051,26	1981,59	1511	1026	444,75
850 °C	2323,83		2050,16	1980,05	1576	1027,0	428
Cluster Functions	C_2H_2 Acetylene	$C \equiv C$ Alkuna	$C \equiv C - H$ Alkuna	C=O Carbonyl	C=C Aromatic	C-O Secondary Alcohol	C-H

Changes in functional groups did not occur at 700 °C and 750 °C activation temperatures. However, there was a decrease in absorption intensity in secondary alcohol groups. It is because the higher the activation temperature will weaken the bond or vaporize the group, which causes the vibration frequency to decrease. Inactivation temperature of 800 °C and 850 °C, there is a friction of absorption pattern toward the wavelength domain of 2323 – 2424 cm^{-1} which suspected as C_2H_2 (Acetylene) cluster. Furthermore, the appearance of the new absorption band in the wavelength domain of 2050-2051 cm^{-1} which alleged as the vibration of C-H (alkyne) and in the wavenumber domain of 1980 cm^{-1} which assumed as C=O (carbonyl) cluster. This possibility caused by the interaction between activated charcoal with free radicals compounds from outside (H_2O) when the process of temperature activation is in progress [31]. While Swaidan (2013) argues that the rise due to the carbonyl group of the cellulose in plants itself, the statement was proven by the writings of Maulina and Iriansyah that the high cellulose content in oil palm charcoal was 31.5% compared to lignin content (14%) and hemicellulose (19.2%) [32].

The FTIR analysis results pada Tabel 2 show that the heat treatment or activation temperature variation can permute the function cluster. Yashim et al. reported that changes in the functional groups of the surface treated with the activation and carbonized at high temperatures[33]. It is seen in the friction of absorption pattern, the loss of some absorption pattern, the formation of new absorption pattern, and the reduction of absorption intensity. Carbonization and activation process has also formed a bond of C=C aromatic in approximately 1511-1576 cm^{-1}. It shows that carbonization and activation to be activated charcoal will increase the aromatic compound [34]**.** This compound is the composer of charcoal hexagonal structure and activated charcoal. The higher activation temperature it will reinforce the aromatic cluster

V. Conclusion

Activation temperature has a significant effect on activated charcoal morphology and activated charcoal functional groups of oil palm shells. The higher of activation temperature, the more active charcoal pore area of the oil palm shell formed, the included in the mesopore category. Palm shell charcoal has alkuna functional groups, C=C (aromatic), C-O and C-H, while charcoal activated in temperatures of 700℃, 750℃, 800℃, and 850℃ undergoes functional group changes followed by atomic realignment carbon that viewed in shifting absorption bands. In addition, the higher activation temperature will strengthen the aromatic compounds; the compound is a constituent of charcoal and activated hexagonal structures

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