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Temperature carbonization effect on the quality of activated carbon based on rubber seed shell

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Abstract. Successful activated carbon-based rubber seed shell was made using a chemical activation method using H_3PO_4 activator 7% at 400, 500 and 600°C carbonization temperature variations. Based on the results of testing the activated carbon which was burned at 600°C had a better quality compared to activated carbon which was burned at 400 and 500°C. Activated carbon burned at 600°C has a low moisture content of 2.03%, the high ash content of 2.57%, the low volatile matter of 19.64% and high fixed carbon of 76.15%. From the calculation of the density and porosity of the activated carbon has been made known that the higher the carbonization temperature from 400°C to 600°C there is an increase in porosity from 7.689% to 14.809% and there is a decrease in density from 0.01198 gr/cm^3 to 0.01182 gr/cm^3. While from FTIR characterization seen the functional group of P-OH, O-H and C-H aliphatic at active activated carbon.

1. Introduction

Based on Indonesian plantation statistical data since 1980 the Indonesian rubber industry has experienced stable production growth. This is due to the expansion of the rubber plantation area every year. The increase in the area of rubber plantations also causes an increase in rubber production. One of the largest rubber producing regions in Indonesia is South Sumatra, which in 2017 saw rubber production amounting to 970,678 with an area of 845,167 ha. Increasing rubber production every year will also have an impact on the increase in by products of rubber plantations in the form of rubber seeds. The main product of rubber plants is the sap, while the by-products in the form of rubber seeds have not been used optimally. However, along with the development of technology and science several studies have been carried out on rubber plant waste, one of which is the research based on rubber seed shell waste in the manufacture of activated carbon. The selection of basic materials for the process of making activated carbon must meet several criteria, namely low inorganic elements, availability of materials (inexpensive and easily obtainable), good durability, and easy to activate. In addition, the requirement for the selection of basic ingredients for making activated carbon is the content of lignin and cellulose in the raw material. The hard shell construction indicates that the rubber seed shell contains active compounds in the form of lignin, so that the rubber seed shell has the potential to be used as a material for making activated carbon.

Activated carbon is an amorphous carbon compound that can be produced from carbon-containing materials or from charcoal which is treated in a special way to obtain a wider surface [2]. According [3] activated carbon is amorphous carbon from flat plates composed of C atoms that are covalently bound in a hexagonal lattice with one C atom at each angle as seen in Figure 1,

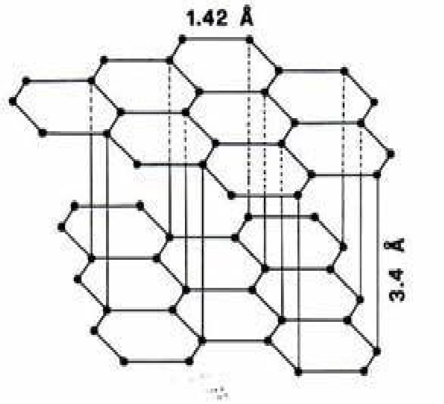


Figure 1. Graphite structure of activated charcoal [3]

There are three stages of making activated carbon.
Dehydrated
The process of removing water in rubber seed shells is heated to a temperature of 170^{o}.
Carbonization
Carbonization is a process of heating organic matter at a certain temperature with a very limited amount of oxygen. The purpose of carbonization is to eliminate volatile substances contained in the base material. This process causes the decomposition of organic compounds that make up the structure of the material to form water, acetic acid vapor, tar, and hydrocarbons. Solid material that lives after carbonization is carbon in the form of charcoal with narrow pores. At the time of carbonization, there are several stages which include removal of water or dehydration, evaporation of cellulose, lignin evaporation, and carbon purification. At heating temperatures up to 400^{o} C there is water removal, cellulose evaporation, and lignin evaporation, while for carbon refining processes occur at a temperature of 500 - 800^{o}C [4].
Activation
Activation is part of the process of making activated carbon which aims to open, add or develop pore volume and enlarge the pore diameter that has been formed in the carbonization process. Through the activation process activated carbon will have increased adsorption power because the activated carbon produced by carbonization usually still contains substances that cover the surface pores of the activated carbon. In the activation process activated carbon will experience changes in properties, both physical and chemical so that it can affect the adsorption power [5]. There are 2 commonly used activation methods, namely physical and chemical activation.

- Physics Activation. The basic ingredients of activated carbon are activated using activating agents from CO_2 or steam at a temperature of 500-800° C Factors that affect the characteristics or properties of the activated carbon produced by physical activation process include the base material, heat flow rate, gas flow rate, before the carbonation process, temperature at the time of the activation process, the activating agent used and the length of the activation process [5].
- Chemical Activation. This activation is the process of breaking carbon chains from organic compounds with the use of chemicals. In this way, the activation process is carried out using chemicals (activators) as activating agents. Activators are substances or chemicals that function as activating reagents on activated carbon adsorbents so that they can cause better absorption. The activator substance is binding to water which causes water that is tightly bound to the carbon pores which do not disappear when carbonization becomes loose. Then the activator substance will enter the pore and open the surface of the activated carbon which is still closed. Activation of activated carbon is done by soaking charcoal into acidic chemical solutions (H_3PO_4 and H_2SO_4)), bases (KOH and NaOH) and are salt (ZnCl and NaCl) [6]. Of the two types of activation processes, according to [7] chemical activation has certain advantages compared to physics activation, such as:

1. In the process of chemical activation, activating chemicals already exist in the preparation stage so that the carbonization process and carbon activation process accumulate in one steps commonly called *one-step activation*.
2. In the process of chemical activation, the temperature used is generally lower.
3. The effects of dehydration agents on chemical activation can improve the development of pores in the carbon structure.
4. Products that are produced from chemical activation are more than the results of physics activation.

Characteristics of activated carbon made either on a conventional scale or laboratory scale must be appropriate to the quality standards set by the Indonesian Industry Standard, SII 0285 - 1988.

Table 1. Active Carbon Quality Requirements (SII. 0258-88)

Types of Test	Requirements for	
	Granules	Powder
Lost Parts at 950^o C	Max. 15%	Max. 25%
Moisture Content	Max. 4.5%	Max. 15%
Ash Content	Max. 2.5%	Max. 10%
Fixed Carbon	Min. 80%	Min. 65%

Few studies have been done in the manufacture of activated carbon from rubber plants including Srinivasakannan and Abu Bakar, 2003, which studied the manufacture of activated carbon from rubberwood powder using H_3PO_4 60% as an activator with a ratio of 1; 1.5 and 2 on the activation temperature of 400^o C and 500^o C. Studied manufacture of activated carbon from coconut rind rubber with an activator H_3PO_4 7%, 100 mesh and impregnation ratio of 1: 4. In this research, the production of activated carbon based on rubber seed shell waste activated using 7% phosphoric acid with carbonization temperature variations of 400^o C, 500^o C, and 600^o C. The activated carbon that has been produced is then tested for its quality and functional characterization using FTIR [9].

2. Research Methodology

The stages of making activated carbon in this study are as follows:

2.1. Rubber Seed Shell Preparation Stage

1. Prepare 300 gr of rubber seed shell that has been separated from the cake.
2. Wash and clean the rubber seed shell from dirt and dust.
3. Dry it in the sun for 12 hours until the shell is dry.

2.2. The Carbonization Stage Dry

1. shells of rubber seeds are burned in a *furnace* for 30 minutes with temperature variations of 400, 500 and 600 ^{o}C.
2. The charcoal is then crushed and sifted to pass the 100 mesh sieve.
3. Three samples of charcoal were obtained with different combustion temperatures.

2.3. Stage of Activation of Charcoal Into Activated Carbon

1. Each charcoal is immersed in H_3PO_4 for 24 hours.
2. Charcoal is then filtered and rinsed with aquadest to neutral pH.
3. Dry activated carbon in the oven at 105^o C.

2.4. Characterization of Activated Carbon

The characterization of activated carbon in this study includes:

2.5. Calculation of the yield of charcoal

shell of rubber seeds is weighed before carbonization, then after carbonization weigh the charcoal mass. Calculate the percentage of charcoal yield using equation 1.

$$\text{Carbon Rendemen} = \frac{m_{carbon}}{m_{material}} \times 100\% \quad (1)$$

2.5.1 Test for Quality Standards for Active Carbon

Moisture Content

Activated carbon is weighed as much as 1 gram and put in a porcelain dish that has constant mass, then put it in the oven at 105° C for 1 hour, then cool the sample in a desiccator and weigh it. Perform the procedure until the sample mass is constant. Calculate water content with equation 2.

$$\text{MC} = \frac{m_a - m_b}{m_{sample}} \times 100\% \quad (2)$$

Ash Content

Activated carbon is weighed as much as 1 gram and is inserted into the porcelain exchange rate which has known the weight. Burn back the charcoal in the furnace at 800° C for 4 hours to become ash. When all carbon has become ash, cool it in a desiccator and then weigh the activated carbon sample. Ash content can be calculated using equation 3.

$$\text{AC} = \frac{m_{ash}}{m_{sample}} \times 100\% \quad (3)$$

Volatile Matter

The activated carbon is weighed as much as 1 gram and heated to a temperature of 950° C in the furnace. After the temperature is reached, leave it for 7 minutes, then the carbon is left to cool in the furnace with no contact with outside air. After chilling enter into the desiccator and weigh. The level of evaporated substance is calculated using equation 4.

$$\text{VM} = \frac{m_a - m_b}{m_a} \times 100\% \quad (4)$$

Fixed Carbon

The carbon content bound to the activated carbon is obtained from the results of 100% activated carbon by summarizing the water content, ash content and the missing part at 950^0 C in accordance with equation 5.

$$\text{FC} = 100\% - (MC + AC + VM) \quad (5)$$

2.5.2 Characterization of Activated Carbon Function Groups.

Characterization of functional groups on activated carbon that has been made is done using FTIR tools.

3. Discussion

Table 2 Data on Shell Carbonization

No.	Description of	carbonization of rubber seed shell at temperatures of		
		400° C	500° C	600° C
1	Mass of rubber seed shell		**55 gr**	
2	Charcoal mass	20,936 gr	17,758 gr	15,233 gr
3	Mass lost	34,064 gr	37,242 gr	39,767 gr
4	% Charcoal yield	38,065%	32,287%	27,696%
5	% The mass lost	61,934%	67,712%	72,303%

Based on Table 2 it can be seen that the carbonization temperature greatly affects the yield of charcoal produced. Along with the increased carbonization temperature, the yield of charcoal produced will decrease. The low yield of charcoal is due to the reaction between carbon and water vapor increases with increasing carbonization temperature, so that carbon reacts to CO_2 and H_2 more but produces less carbon [9].

Table 3 Active Carbon Quality

No.	Type of Test	for Active Carbon Quality Standards for Active Carbon	Samples		
			Carbonization 400° C (%)	Carbonization 500° C (%)	Carbonization 600° C **(%)**
1	Moisture Content	Max. 15%	3.21	2.67	2.03
2	Ash Content	Max. 10%	0.99	2.09	2.57
3	Volatile Matter	Max. 25%	32.89	26.70	19.64
4	Fixed Carbon	Min. 65%	62.51	69.14	76.15

Based on Table 3 it can be seen that the activated carbon made has met the quality standards of activated carbon. Carbonized activated carbon at a temperature of 600°C tends to be closer to the specified quality standard when compared with activated carbon which is carbonized at a temperature of 400° C and 500° C. This is due to lower carbon content, higher ash content, volatile matter low and high fixed carbon.

3.1. Moisture Content

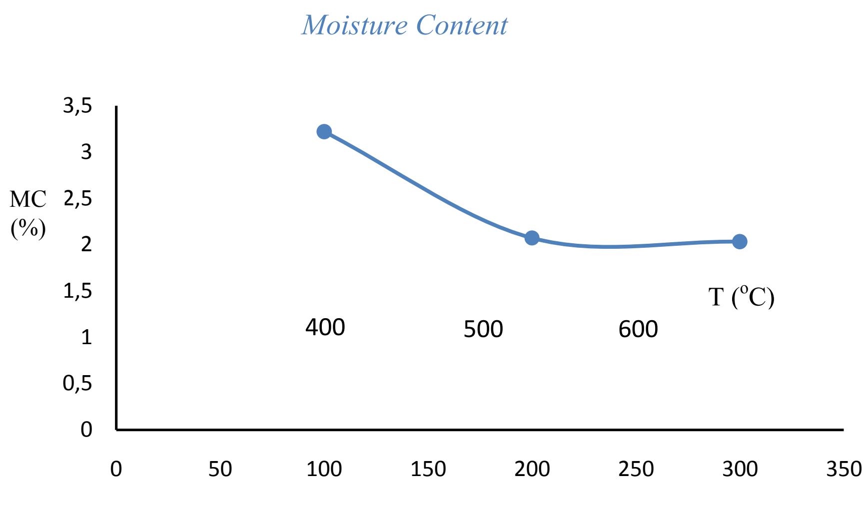


Figure 2. Active Carbon Water Content

From figure 2 it can be seen that the increase of carbonization temperature from 400°C to 600°C causes a decrease of water content from 3.21% to 2.03%. These results are in line with the research conducted by [9][10], where there was a decrease in water content as the carbonization temperature increased. This is because chemically H_2O begins to experience changes in the phase into the gas when it reaches its boiling point, which is at a temperature of 100 ° C. At this point, free H_2O bound to carbon is released and forms a gas phase.

3.2. Ash Content

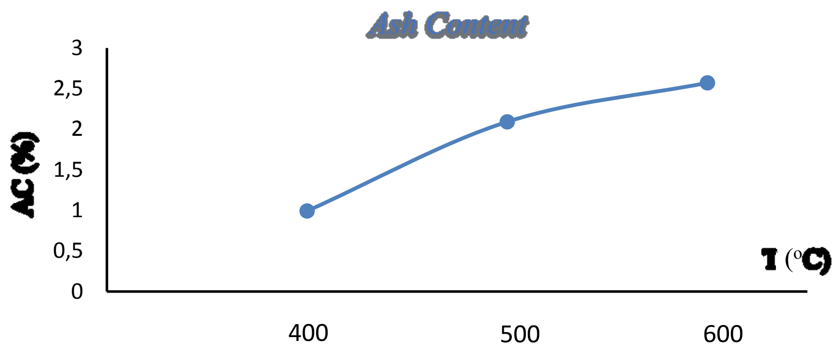


Figure 3. Active Carbon Ash Level

From figure 3 it can be seen that the increase in carbonization temperature from 400°C to 600°C can cause an increase in ash content from 0.99% to 2.57%. This result is in line with the research conducted by [9]. Increased ash content can occur due to the formation of mineral salts during the process of drying which if continued will form fine particles of the mineral salt. This is due to the mineral content contained in the biomass starting material for carbon making. This mineral material will then form into an ash compound when the oxidation process is carried out [11].

3.3. Volatile Matter

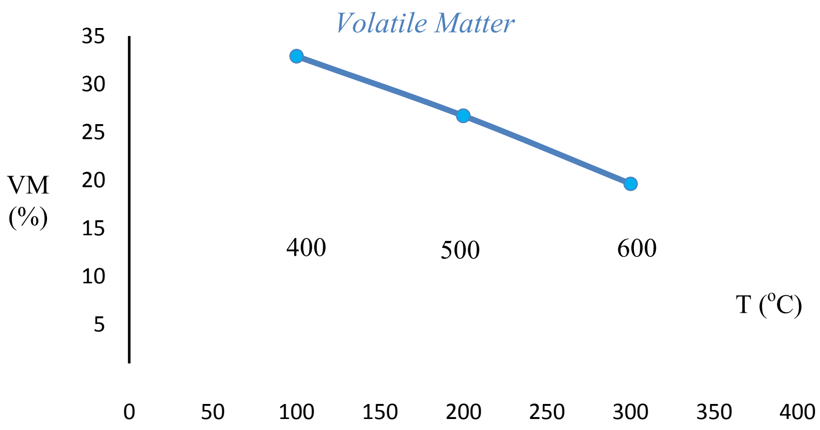


Figure 4. Levels of Active Carbon Evaporating Substances

From figure 4, it can be seen that the increase in carbonization temperature from 400°C to 600°C causes a decrease in evaporating content from 32.89% to 19.64%. According to Fauziah (2011), the amount of the volatile substance is determined by the time and temperature of carbonization. The higher the carbonization temperature, the more wasted substances will be, so that the evaporating content will be lower. The presence of evaporating substances that are still attached to carbon will affect carbon absorption. The higher the temperature, the more substances that cover the carbon evaporate so that the previously closed carbon pore surface will open up and increase the absorption capacity.

3.4. Fixed Carbon

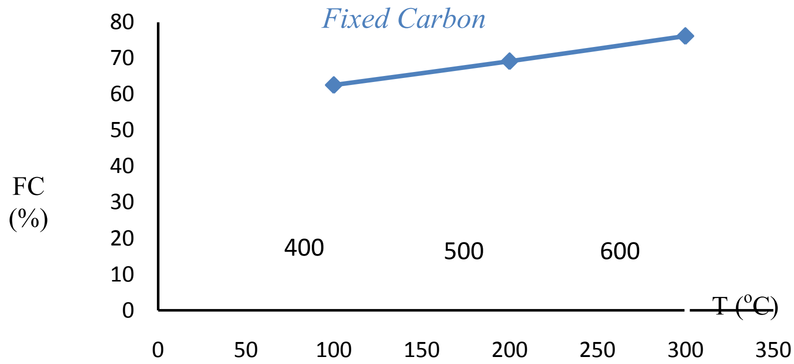
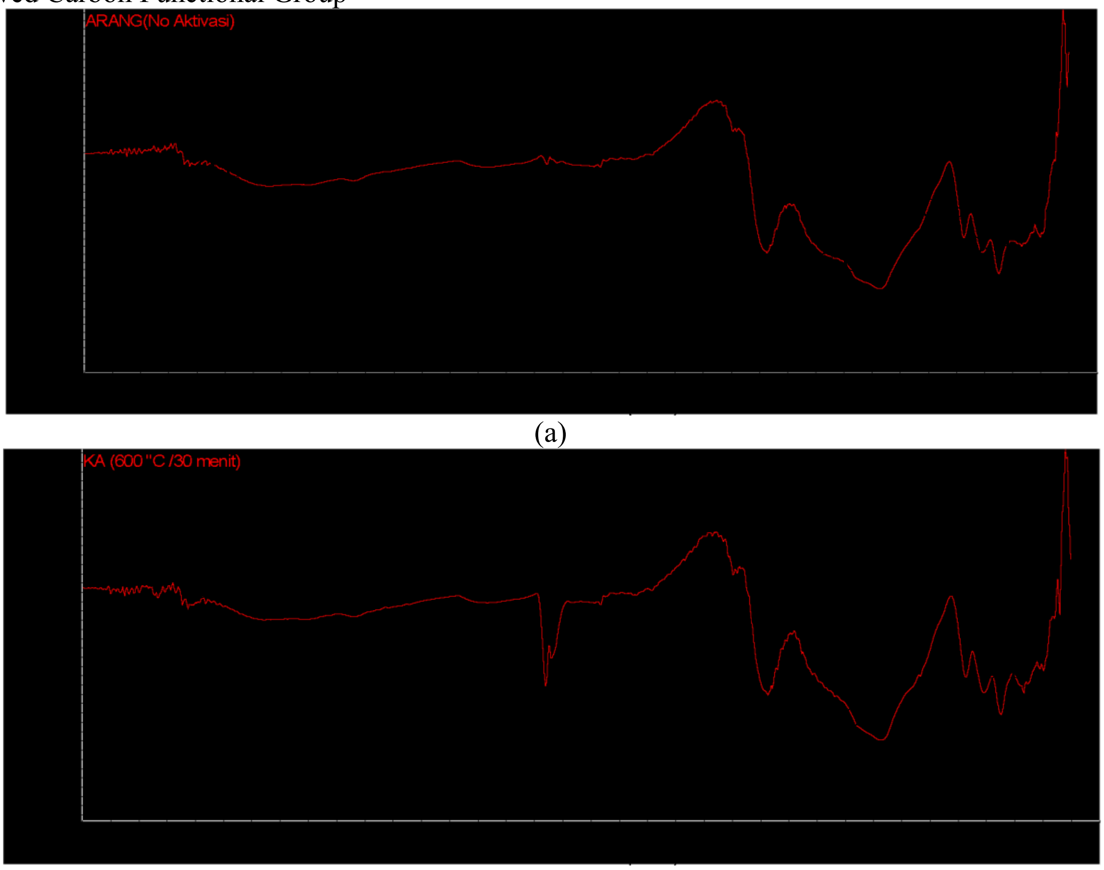


Figure 5. Levels of Activated Carbon in Activated Carbon

From Figure 5, it can be seen that the increase in carbonization temperature from 400°C to 600°C causes an increase in evaporating content from 62.51% to 76.15%. In addition to the influence of water content, ash content and *volatile matter*, the bound carbon content of activated charcoal is also strongly influenced by the carbon content of the raw materials used. But there are several other factors that influence the level of activated charcoal activated carbon, among others, the way of activation, activation temperature, cellulose content and lignin in the raw material.

Actived Carbon Functional Group



(a)

(b)

Figure 6 (a) FTIR Spectra of Rubber Seed Shell Charcoal (b) FTIR Spectra Activated Carbon from Rubber Seed Shell

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Figure 6 is a non-activated FTIR carbon spectrum (a) and activated carbon (b) . From Figure 4.5 it can be seen that in accordance with the functional groups of several compounds in Table 2.5 both activated and unactivated carbon has the same functional group at wave number 1573 cm^{-1}; 1173 cm^{-1}; 872 cm^{-1}; 808 cm^{-1}; and 748 cm^{-1} , each of which is a functional group C = C; CO; CH and OH. However, the activated carbon decreases the value *transmittance* at wave number 2359.8 cm^{-1}. According to Sahara, et al. (2017) it was caused by OH vibrations in P-OH groups because the effect of activator substances used was H_3PO_4. The results of identification with this infrared spectrophotometer indicate that the activated carbon derived from rubber seed shell waste contains functional groups P-OH, OH and CH aliphatic.

4. Conclusion

Activated carbon based on carbonized shell seed waste at a temperature of 600°C has better quality compared to activated carbon which is carbonized at a temperature of 400 and 500°C. The higher the carbonization temperature the lower the water content of the activated carbon due to carbon samples Activated carbonized at 400° C has a moisture content of 3.22% while at carbonization temperature 600° C is 2.03%. The higher the carbonization temperature, the ash content of the activated carbon will increase because the activated carbon sample is carbonized with a temperature of 400° C, ash content of 0.99% while at the carbonization temperature 600° C 2.57%. The higher the temperature carbonization of the *volatile matter* to fall further as activated carbon samples were carbonized at a temperature of 400° C resulted in *volatile matter* 32.89%, while the carbonization temperature of 600° C is 19.64%. The higher the carbonization temperature, the *fixed carbon* increases because the carbonized activated carbon sample at 400° C produces *fixed carbon* 62.51%while at the carbonization temperature 600° C is 76.15%. Activated carbon from rubber seed shell waste contains functional groups P-OH, OH and CH aliphatic.

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