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Preparation and Characterization of Catalyst Cr/Activated Carbonfrom Palm Empty Fruit Bunch

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Abstract

Preparation and characterization of catalyst Cr/Activated Carbon from palm empty fruit bunch had been done. The research purpose isdetermining the effect of carbonization temperature towards adsorption of ammonia, iodine number, methylene blue number, and porosity of Activated Carbon and catalyst Cr/Activated Carbon. The determination of porosity include surface area, microspore volume and total pore volume. The results showed the best carbonization temperature Activated Carbon and catalyst Cr/Activated Carbon at 700°C. The adsorption ammonia of Activated Carbon and catalyst Cr/Activated Carbonwas 6.379 mmol/g and 8.1624 mmol/g. The iodine number of Activated Carbon and catalyst Cr/Activated Carbonwas 1520.16 mg/g and 1535.67 mg/g. The methylene blue number of Activated Carbon and catalyst Cr/Activated Carbonwas 281.71 mg/g and 319.18 mg/g. The surface area of Activated Carbon and catalyst Cr/Activated Carbonwas 1527.80 m^2/g and 1652.58 m^2/g. The microspore volume of Activated Carbon and catalyst Cr/Activated Carbonwas 0.7460 cm^3/g and 0.8670 cm^3/g. The total pore volume of Activated Carbon and catalyst Cr/Activated Carbonwas 0.8243 cm^3/g and 0.8970 cm^3/g.

Keywords: Activated Carbon, palm empty fruit bunch, porosity, catalyst, chromium

Abstrak (Indonesian)

Pembuatan dan karakterisasi katalis Cr/Karbon aktif dari tandan kosong kelapa sawit telah dilakukan. Penelitian ini bertujuan untuk menentukan pengaruh temperatur karbonisasi terhadap penyerapan Amoniak, bilangan iodin, bilangan metilen biru dan porositas Karbon aktif dan katalis Cr/Karbon aktif. Penentuan porositas mencakup luas permukaan, volume mikropori dan total volume pori-pori. Hasil menunjukkan bahwa temperatur karbonisasi terbaik dari Karbon aktif dan katalis Cr/karbon aktif adalah 700°C. Penyerapan amoniak oleh Karbon aktif dan katalis Cr/karbon aktif adalah 6.379 mmol/g and 8.1624 mmol/g. Bilangan iodin Karbon aktif dan katalis Cr/karbon aktif adalah1520.16 mg/g dan 1535.67 mg/g. Bilangan methylene blue Karbon aktif dan katalis Cr/karbon aktif adalah 281.71 mg/g dan 319.18 mg/g. Luas permukaan Karbon aktif dan katalis Cr/karbon aktif adalah 1527.80 m^2/g dan 1652.58 m^2/g. Volume mikropori Karbon aktif dan katalis Cr/karbon aktif adalah 0.7460 cm^3/g dan 0.8670 cm^3/g. Total volume pori-pori Karbon aktif dan katalis Cr/karbon aktif adalah 0.8243 cm^3/g dan 0.8970 cm^3/g.

Keywords: Karbon aktif, tandan kosong kelapa sawit, porositas, katalis, Kromium

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INTRODUCTION

The catalyst is a substance that can increase the reaction rate without altering the enthalpy of the product [1]. The catalyst has been widely used, especially in the chemical industry. For example, a catalyst is used to solve the air pollution problems such as reducing emissions of carbon monoxide gas in motor vehicles and hydrocracking process in biodiesel. In the hydrocracking reaction, acidic catalyst is needed [2]. Guan, et.all.[3] reported the total acidity of catalyst increased with the total metal content. Currently, the researchers are using a metal transition as catalyst such as Cu, Ni, Pb, and Cr [4].
Metal Chromium (Cr) is a third-period transition metal that can form complexes compounds and easily experience to oxidation. Cr has a catalytic activity, either in a state of pure metal or oxidized. Cr metal catalytic ability and decided to form a double bond or diatomic molecules like H_2. The reaction of hydrocarbons- Cr act as a dehydrogenation process and it can increase the cracking number because the dehydrogenation mechanism allows the termination of the C-C bonds [3]. In contrary, the metal catalyst has a weakness where the thermal stability resulted was low and the surface area also decreased. Metal catalyst efficiency can be improved by impregnating or adding support or carrier substance such as silica, alumina, zeolite[5].

Some researchers have been used metal catalyst such as Cr to modify Akhtar and Amin [6] converts used cookingwasteoil into liquid fuels by modifying zeolite catalyst-Cr. Lu et al. [7] use a catalyst Cr/HZSM-5 in cracking reactions of isobutene while the catalyst Ni-Cr/zeolite in converting plastic waste to gasoline until 86.91% [3]. Besides zeolite and HZSM-5, there are supporting substances that can be converted as a catalyst i.e. Activated Carbon.

Activated Carbon (AC) is the famous supporting material in the adsorption-catalytic process because it can increase micro porosity and surface area. Several studies add AC from coconut shell in the manufacture of metal catalysts. Bash[8] adding AC into Ni metal catalyst to enhance the conversion of benzene into cyclohexane amounted to 25.36%. Blanch-Raga et.al.[9] did the preparation of Cu catalyst and zeolit tomodified beta zeolite catalysts for the trichloroethylene oxidation. Preparation and characterization of activated carbon from pineapple waste biomass for dye removal has been done by Muhamad et.al.[10]. One of the constituent materials of AC is the oil palm empty fruit bunches. Oil palm empty fruit bunches (OPEFB) are remnants of oil palm crops that are not included in the main product or a byproduct of the processing of palm oil [11].
This study will make a catalyst from metal Cr and CA from OPEFB as a supporter. In this study the catalyst preparation Cr/AC catalyst activity test will be conducted as well as the characterization of such absorption of ammonia, iodine determination, and determination of the number of methylene blue, determination of porosity covering a surface area, microspore volume, and total pore volume.

EXPERIMENTAL SECTION

Preparation OPEFB and AC

OPEFB was taken from the oil palm plantation PT. Earth Sawindo Permai,Tanjung Enim. Furthermore, weighed with a certain weight and then dried. OPEFB dried chopped and mashed size of 60 meshes. Then, total of 250 g of dry OPEFB size of 60 meshes is inserted into furnace reactor. The OPEFB was heated in various temperatures of 500, 600, 700, 750 and 800°C.

Characterization of AC Pore

1. Ammonia Absorption

After heated in the oven for 24 hours at a temperature of 110°C, AC was cooled in a desiccator and then weighed as much as 1 g and placed in the cup of the crucible. Grail crucible containing AC was placed in a vacuum desiccator together with 5 mL of NH3 that placed in the cup of crucible for 24 hours. Grail issued and cooled moment. Weight of AC was weighed again until the value constant.

2. Determination of total iodine and Methylene Blue

The procedure of determination of total Iodine and Methylene Blue numbers follow the procedure initiated by Nunes and Guerriro[12]. For the measurement of Iodine, 0.01 g of AC was reacted with 5 mL of HCl 5% and then heated at a temperature of 60°C for 30 seconds. Next step, 50 mL of 0.1 N Iodine solutions is added to the mixture, stirred for 2 minutes and then filtered. 25 mL of filtrate was added with starch as the indicator and then titrated with 0.1 N $Na_2S_2O_3$.

For Methylene Blue, 0.01 g of AC was reacted with 10 mL of methylene blue solution with varying concentrations of methylene blue such as 10, 25, 50, 100, 250, 500, and 1000 mg/L for 24 hours at room temperature. Then the remaining concentration of methylene blue was analyzed using UV-VIS spectrophotometer at a wavelength of 600-700 nm.

3. Preparation of Catalyst Cr / AC

number of 40 g of AC was refluxxed in 150 mL 1 N H_2SO_4 solution at 60°C for 6 hours. The precipitate was filtered and washed with distilled water until the pH neutral. Next, AC is washed and dried in an oven at 110°C for 3 hours.

A number of 3 g of AC was added to 76 g of Cr $(NO_3)_3.9H_2O$ and stirred with 500 mL of distilled water for 24 hours. At 4 hours, mixture was added drop by drop of ammonia. Once the stirrer process is complete, Cr_2O_3/AC was dried at a temperature of 130°C for 3 hours until it becomes a paste and then calcinated at a temperature of 450°C for 1 hour. Next, catalysts Cr2O3/AC was reduced to H_2 gas with a temperature of 400° C for 2 hours.

The final step is charactization of catalysts Cr2O3/AC 3 includes the absorption of ammonia, iodine determination and the determination of methylene blue number.

4. Data analysis

Analysis of the data includes:

a. Carbon absorption capacity of the ammonia is calculated by determining the amount of NH_3 (mmol/gram) which is absorbed by the following formula:

$$Amonia\ Absorption = \frac{w_{NH_3}\ x\ 1000}{BM_{NH_3}\ x\ w_{KA}} \qquad (2.1)$$

where$w_{NH_3}$is the weight of absorbed NH_3, , $BM_{NH_3}$is a relative molecular weight is the weight of NH_3 and w_{KA} active carbon used.

b. Iodine number (IN) is calculated using the equation:

$$IN = \frac{Eq_{I_2}\ x\ V_{Na_2S_2O_3}\ x\ N_{Na_2S_2O_3}\ x\ 100\ x\ 10^{-3}}{w} \qquad (2.2)$$

Where Eq_{I_2} is the equivalent weight of iodine, $V_{Na_2S_2O_3}$ is the volume of $Na_2S_2O_3$ in mL, $N_{Na_2S_2O_3}$ is the normality of $Na_2S_2O_3$ and w is the weight of active carbon used.

c. The amount of methylene blue adsorbed to each solution can be calculated using the equation:

$$Qe = \frac{(C_0 - C_e)\ x\ V}{w} \qquad (2.3)$$

where C_0 is the initial concentration of methylene blue solution (mg / L), C_e is the concentration of methylene blue at equilibrium (mg / L), V is the volume of the solution (L), and w is the weight of AC (g).

d. After the results obtained and then find the value Qe MBN or ymax using the following equation:

$$Y_{max} = Slope \ln(x) - Intersept \qquad (2.4)$$

whereymax is the value of MBN, x is the highest value of the initial concentration.

e. From the result of the determination of iodine and methylene blue numbers, then the surface area (S), micropore volume (Vm), and total pore volume (Vt) to be determined using the following mathematical relationship [12]:

$$S\ (m^2/g) = 2.28 \times 10^2 - 1.01 \times 10^{-1}\ MBN + 3.00 \times 10^{-1}\ IN + 1.05 \times 10^{-4}\ MBN^2 + 2.00 \times 10^{-4}\ IN^2 + 9.38 \times 10^{-4}\ MBN\ IN \qquad (2.5)$$

$$Vm\ (cm^3/g) = 5.60 \times 10^{-2} - 1.00 \times 10^{-3}\ MBN + 1.55 \times 10^{-4}\ IN + 7.00 \times 10^{-6}\ MBN^2 + 1.00 \times 10^{-7}\ IN^2 - 1.18 \times 10^{-7}\ MBN\ IN \qquad (2.6)$$

$$Vt\ (cm^3/g) = 1.37 \times 10^{-1} + 1.90 \times 10^{-3}\ MBN + 1.00 \times 10^{-4}\ IN \qquad (2.7)$$

RESULT AND DISCUSSION

Activation of Catalyst Cr/AC

Carbon usually still contains substances that cover the surface pores. Before impregnated with Cr, carbon was activated by chemical activation method using sulfuric acid reflux. This method is more profitable than the immersion method using multiple stages of acidification. In addition, the chemical activation method require shorter time than reflux method. Theacid was used as an activator in chemical activation which aims to remove the impurities and residual carbonization tar to make more porous [13].

Effect of temperature carbonization Against Ammonia Absorption

The determination of ammonia absorption of AC and catalysts Cr /ACas the effect of temperature has been done with ammonia gas as a base absorbate. It is expected that the size of a small ammonia allowed to enter into the pores of AC and Cr catalysts/AC so the amount of ammonia absorption per gram sample can be measured. The magnitude of the effect of

temperature on the ammonia absorption of AC and catalysts Cr /AC can be seen in Figure 1.

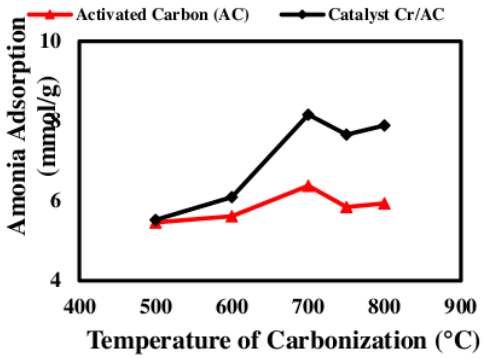

Figure 1.Effect of carbonization temperature on Ammonia Absorption

In Figure 1, it can be seen that the absorption of AC and catalysts Cr /AC start from temperature of 500°C carbonization while on the others carbonization temperature, absorption of ammonia is higher and the maximum temperature of 700°C. This phenomenon due to the pore structure of AC and catalysts Cr /AC. Higher temperature makes bigger pore so more ammonia gas can be absorbed. Further, the absorptionof Ammonia indicates AC has the ability as adsorbent. Absorptive capacity of the catalyst Cr/AC tends to be higher than the AC. This phenomenom is causedby the chemical activationof AC resulted in bigger pore and better surface area; and the presence of Cr, impregnated on the surface of AC,have d orbitals that still a shortage of electrons can capture an electron from the reactant.

Effect of carbonization temperature on Iodine Number

The result of measurements of iodine is an important parameter used to characterize AC, where iodine is a measure of the level of activity of AC. The number of iodine can be used as an approach for surface area and micro porous AC with good precision [14]. Carbonization temperature has a significant influence on the formation of porous AC [15]. The effect of temperature on the Iodinenumber of AC and catalysts Cr/AC can be seen in Figure 2.

In figure 2 it can be seen that the iodinenumber ofAC and catalysts Cr/AC increased from temperature of 500°C to 700°C. The maximum iodine number reached at temperature of 700°C and decline at higher temperature. It can be concluded that the higher temperature, the carbonization of AC iodine increases. When the carbonization temperature reached a maximum of cavities produced,AC is capable to absorbing iodine molecules as well. This indicates that the structure of micro porous AC has been widely established and adsorbs the iodine molecule that has a small molecular size. But after reaching the maximum carbonization temperature, iodine number will decrease again. It happens because the cavities of pores generated above the maximum carbonization temperature will wider due to erosion of carbon. According to Jain et al.[16], as the high temperature carbonization made of carbon structure becomes brittle and this makes cavity surface pores in AC becomes shallower.

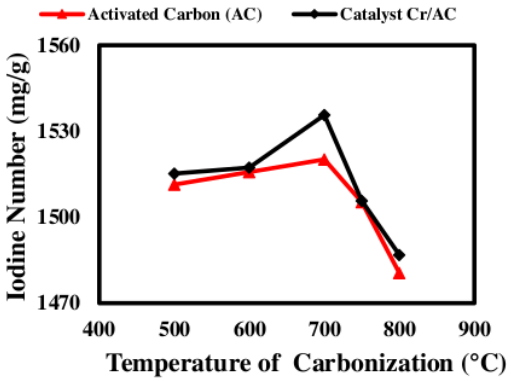

Figure 2.Effect of Temperature Carbonization of Iodine Number

Number iodine of catalyst Cr/AC tend to be higher than iodine number of AC. This is not only caused by more formed of microporous structure of carbon, but also the surface of catalyst Cr/AC more activated than the AC. According to Wang et al. [17], the higher the carbonization temperature, the higher the number of iodine, because more activated surface formed. In addition, the chemical activation treatment which serves to larger the absorption area resulting more microporous carbon structures so the carbon Iodine absorption capacity will rise.

Effect of temperature carbonization on Methylene Blue number

The methylene blue numbers is used to see the ability of carbon to absorb dye and big molecules; and determine the pore surface area. The magnitude of the effect of temperature on the methylene numberblue by active carbon and Cr catalysts / AC can be seen in Figure 3.

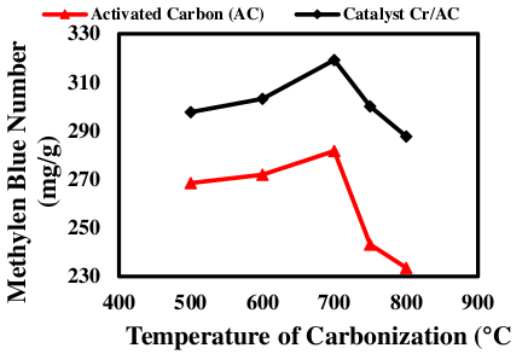

Figure 3. Effect of Temperature carbonization on the Methylene Blue number

In Figure 3, it can be seen the maximum number of methylene blue active carbon and catalysts Cr/AC is at 700°C and a decline at the carbonization temperature between 750°C to 800°C. It can be concluded that the higher the temperature the greater the number of methylene blue. When the carbonization temperature reach maximum, the cavities of ACcapableto absorb the molecules of methylene blue. This indicates that the macropore structure to AC has been widely established. But after reaching the maximum, temperature carbonization of methylene blue numbers will decline because there are still lots of impurities and carbon-covered surface erosion due to carbon at high temperatures and affect the absorption of active carbon to the methylene blue [16]. Methylene blue number of catalysts Cr/AC is greater than the number of methylene blue of active carbon only. This is possible because the physical activation process long enough so that the process of opening the pores is not complete and the carbon surface is covered by a lot of impurities.

Effect of Temperature Carbonization on Porosity

In this section, determination of porosity of AC and catalysts Cr/ACincluding determine surface area, microspore volume and total pore volume using iodine value data and the number of methylene blue, respectively. The surface area, microspore volume and total pore volume is calculated using the equation 2.5, 2.6 and 2.7. The magnitude of the effect of temperature on the surface area, microspore volume, and total pore volume of AC and Cr catalysts / AC can be seen in Figures 4, 5, and 6.

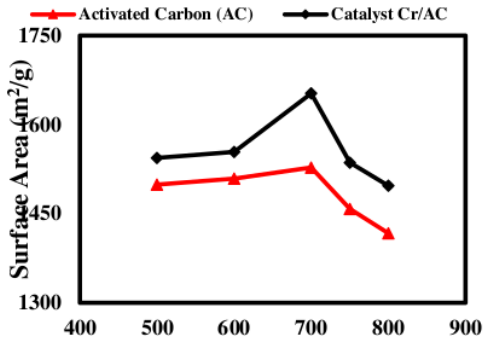

Figure 4. Effect of Temperature carbonization on the Surface Area

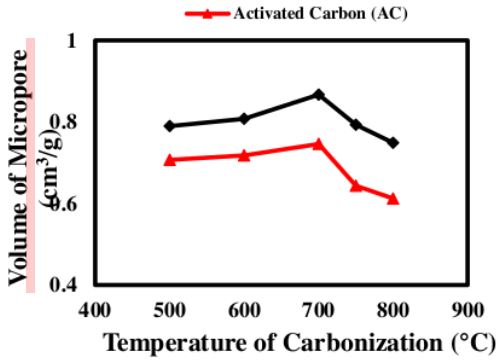

Figure 5. Effect of Temperature carbonization on the volumes of micro porous

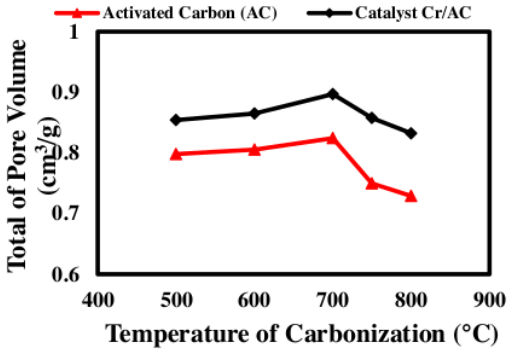

Figure 6. Effect of Temperaturecarbonization on Total Pore Volume

In figure 4, it can be seen the maximum surface area of AC and catalyst Cr/AC at the carbonization

temperature of 700°C and a decline in the carbonization temperature of 750°C to 800°C. It can be concluded that the higher the carbonization temperature increased the pore surface area. This is because the pore cavities produced AC has been widely established. But after passing the maximum temperature, the pore surface area is reduced due to the fragility of the carbon and covers the surface of AC. Similar results were obtained in studies Foo and Lee [18].

The surface area of catalyst Cr/AC is greater than the AC. This is because the pore cavity is more open and clear after chemical activation and metal impregnated Cr to make catalyst surface greater. According to Ródenas et al. [19]the benefit the chemical activation process allows to obtain very large surface area of AC. It is caused by the chemical compound open the closed surface that full of impurities and other chemical compounds.

In Figure 5, it can be seen that the volume of micro porous AC and catalysts Cr/AC increased from carbonization temperature of 500°C to 700°C and the maximum volume of micro porous at 700°C and then will decline as temperature increase. This is relating to the carbon absorption capacity of the small molecular size larger than the molecule size. It can be seen from iodine value of AC has a value greater than the number of methylene blue active carbon. In addition, the results confirmed that the active carbon material such as empty fruit bunches of oil palm will have dominant pore structure on the micro porous up to 95% of the total internal surface of the AC[20]. The volume of microporous catalyst Cr/AC is greater than the AC. This is because the more carbon micro porous structure is formed after the metal impregnated Cr, which previously conducted chemical activation to clear the pores of the carbon impurities.

Porosity, surface area and total pore volume are interrelated, in which a carbon surface area is proportional to the total pore volume. Based on Figure 6, carbonization temperature has an influence on the total pore volume. The results obtained show total pore volume of AC and catalysts Cr/AC increased from carbonization temperature of 500°C to 700°C. The maximum total pore volume of AC and catalyst Cr/AC is at the carbonization temperature of 700°C and a decline in the carbonization temperature of 750°C and 800°C. This occurs because the carbonization temperature will increase the surface area and allows pore AC. But when it has passed the maximum carbonization temperature, total pore volume decreased with the carbon surface area decreases due to the fragility of the carbon structure at high temperatures.

Quality AC of oil palm empty fruit bunch

Pore characterization of AC conducted in this research include the absorption of ammonia, iodine determination, determination of the number of methylene blue and the porosity of the AC are listed in Table 1.

Table 1. Results of Analysis of AC

Temperature of Carbonization (°C)	Iodine Number (mg/g)	Methylene Blue Number (mg/g)
500°C	1511,34	268,45
600°C	1515,75	271,99
700°C	1520,16	281,71
750°C	1505,30	243,22
800°C	1480,50	233,59

Based on Quality Requirements of AC according to IEC No.06-3730-1995 which is the Iodineand methylene blue number in powderform were minimum 750 mg/g and 120 mg/g, respectively. From theTable 1 show the AC from oil palm empty fruit bunches has met the requirements.Thebest characterization of AC from oil palm empty fruit bunches are on carbonization temperature of 700°C where the iodine number is 1520.16 mg/g and the methylene blue is 281.71 mg/g. The greater iodine and methylene blue number indicate the better quality of the AC.

CONCLUSION

From the research could be taken some conclusion i.e:

1. The best temperature AC to the absorption of ammonia and the porosity is at a temperature of 700°C, with the ammonia absorption capacity of 6.379 mmol/g, a surface area of 1527.80 m^2/g, microspore volume of 0.746 cm^3/g, and total pore volume of 0.8243 cm^3/g.
2. The best temperature catalyst Cr/AC to the absorption of ammonia and the porosity is at a temperature of 700°C, with the absorption of ammonia by 8.1624 mmol/g, a surface area of 1652.58 m^2/g, the micro pore volume of 0,8670 cm^3/g, and a total pore volume of 0.8970 cm^3/g.

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