MANUFACTURING NI-CARBON CATALYST PRODUCED FROM PALM FIBER USING HYDROTHERMAL METHOD WITH 1.5% NICKEL SOLUTION CONCENTRATION WITHIN 48 HOURS OF CATALYST SYNTHESIS

Alif Maulana *1), Nazarudin 2), Hadistya Suryadri 3)

*Corresponding author
*ORCHID IDs: https://orcid.org/0000-0002-8404-5999

[1)]*Alif Maulana (Chemical Engineering Department, University of Jambi, Indonesia)*
[2)]*Nazarudin (Chemical Engineering Department, University of Jambi, Indonesia)*
[3)]*Hadistya Suryadri (Chemical Engineering Department, University of Jambi, Indonesia)*

email: alifmaulana15.am15@gmail.com

Abstract

This research aims to determine the potential for using palm fiber as a catalyst obtained from palm oil factory waste. The catalyst was made by carbonizing for 1 hour at a temperature of 300^{o}C. Then activation was carried out using NaOH solution for 2 hours with a ratio of 1: 10. In this research there are two methods for making catalysts which have differences in the stages of catalyst synthesis. Activated carbon is synthesized as a catalyst using a nickel (Ni) solution with the addition of a nickel solution with a concentration of 1,5% through a hydrothermal process which is heating in a closed system using an autoclave. To determine the characteristics of the catalyst produced, Scanning Electron Microscope (SEM) analysis was carried out and to determine the elements contained therein, Energy Dispersive X-Ray (EDX) analysis was carried out. The best catalyst is the method I catalyst as seen from the EDX test results and the morphology of the method I catalyst looks better in its pore structure.

Keywords: *palm fiber, catalyst, hydrothermal, activated carbon*

1. INTRODUCTION

Activated carbon is widely known as the most effective adsorbent used in industrial applications. This is because the nature of activated carbon has a large active surface area so it can provide a high adsorption capacity (Ernesto et al., 2017). Most active carbon is produced through a two-stage process, namely carbonization at high temperatures followed by physical and chemical activation to produce active carbon with a high surface area.

Commercially available activated carbon is usually made from non-renewable and expensive materials. Initially, most commercial activated carbon came from petroleum residues, wood, coal, lignite and peat. Therefore, the production of activated carbon from agricultural product waste has been explored since 1996 (Hardi et al., 2020). There are many agricultural wastes that can be recycled and commercialized to become active carbon, one of which is palm oil waste.

Indonesia is an agricultural country whose development is supported by the agricultural sector. Many oil palm plantations are being developed outside Java, such as Sumatra, Sulawesi and Kalimantan. In 2014, Indonesia produced around 35 (thirty five) million tons of crude palm oil and Indonesia became the largest producer of palm oil in the world (Maryam et al., 2022). Processing palm oil produces a lot of waste, one of which is palm fiber.

Palm fiber is palm oil waste produced from processing palm oil presses during the pressing process which is short in shape like thread and has a brownish yellow color. According to Nazarova et al., (2021). Palm fiber waste can be used as raw material for making catalysts. Processing palm fiber into a catalyst can be done using the hydrothermal method of nickel compounds in activated carbon. The nickel solution can be incorporated into activated carbon as a carrier using a hydrothermal or heating process. Processing biomass into a catalyst through a hydrothermal process is heating in a closed system using*autoclave* so that the added solution does not evaporate.

In this research, there are two methods for making catalysts which have differences in the stages of catalyst synthesis, namely in the first method the catalyst synthesis is carried out on activated carbon that has been printed, while in the second method the catalyst synthesis is carried out on activated carbon which is still in powder form.

This research aims to increase the use value of palm oil factory waste, in this case waste in the form of fiber which is usually unused and thrown away at palm oil factories. This research also needs to be carried out to innovate waste to make products that are more environmentally friendly.

2. RESEARCH METHODS

2.1 Tools and Materials

The materials used in this research were palm fiber, NaOH, nickel (Ni) solution, fox glue, distilled water, nitrogen and hydrogen.

The tools used in this research were a horizontal reactor, a vertical reactor, a 355 µm (45*mesh*), oven, *magnetic stirrer*, funnel*buncher*, *autoclave*, *hot plate*, measuring cup,*beaker glass*, erlenmeyer, separating funnel, analytical balance, porcelain cup, filter paper, pH meter paper, grinder (*mortar* and*pestle*), vernier caliper, vacuum pump, hydraulic press and SEM analyzer (*Scanning Electron Mikroscope*) and EDX (*Energy Dispersive X-Ray*).

2.2 Application

2.2.1 Research matrix

The research matrix in this study can be seen in table 1 below:

Table.1 Matrix for Making Ni-1.5%/Carbon Catalyst Methods I and II

No	Carbon:NaOH Ratio	Nickel:Carbon Solution Ratio	Nickel Solution Concentration (%)	Synthetic Time (hours)	Volume (cm^3)
1.	10:1	10:1	1,5	48	0,5
2.	10:1	10:1	1,5	48	1
3.	10:1	10:1	1,5	48	1,5

2.2.2 Research Method I

There are two methods for making catalysts in this research, the following method I flow diagram can be seen in **Figure.1** below this:

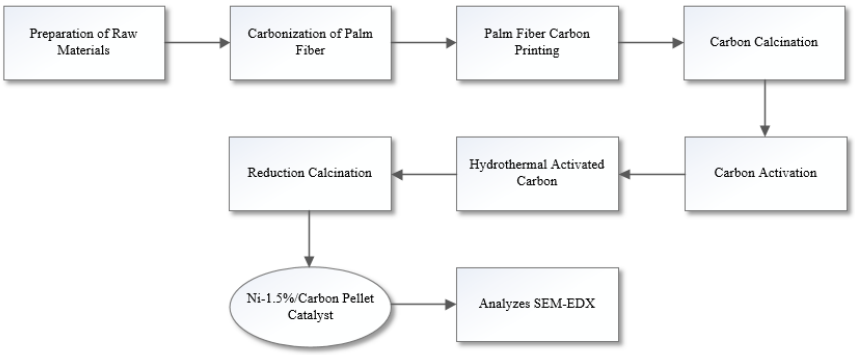


Figure.1 Flow diagram for making catalyst method I

First of all, the raw material is prepared, namely palm oil fiber, which is first carried out by a washing process which aims to remove dirt attached to the palm oil fiber. Once clean, the palm oil fibers are dried in the hot sun, then proceed by putting the palm oil fibers that have been dried in the hot sun into the oven at a temperature of 105^{o}C to ensure that the palm oil fiber is dry.

After preparing the raw materials, the next stage is carbonization of palm fiber. This stage is carried out by weighing 110 grams of dry palm fiber, then putting it in*furnace* to carry out the carbonization process at a temperature of 300^{o}C within 1 hour. The results of the carbonization are then cooled, then ground using*chopper* and then sieving with a sieve size of 355 µm (45*mesh*).

Then the palm fiber carbon printing stage is done by mixing the glue*fox* with a ratio of 1:2 with details namely 30 grams of palm oil carbon fiber and 60 grams of fox glue. This mixing process is carried out for 10 minutes above*hot plate* while stirring using a stir stick. Next, printing was carried out using a hydraulic press for 30 minutes and dried for 20 minutes

using an oven at 105°C to form a plate with the aim of making it easy to measure. Then, cutting is carried out with variations in thickness, namely 0.5 cm^3, 1 cm^3 and 1.5 cm^3. Finally, the results of the cutting are then placed in the oven for 12 hours at a temperature of 105℃.

Next, after the carbon printing stage, it will go through the calcination stage using a temperature of 300°C for 1 hour by supplying oxygen gas. According to Nazarova*et al*., (2015), The calcination function is intended to clean samples from organic impurities, nitrates and water. Because if the sample contains water, unexpected crystal growth will occur. And this calcination process also aims to remove the glue content in the carbon.

The activated carbon that has been calcined will then be activated by mixing the palm oil fiber carbon with NaOH solution for the activation process using 10 grams of palm fiber carbon and 4 grams of NaOH and 100 ml of distilled water. Then the palm oil fiber carbon is put into the NaOH (Sodium Hydroxide) solution which is then continued with the stirring process using*hot plate* and*magnetic stirrer* for 2 hours. The aim of stirring is to make the solution homogeneous. Then wash using filter paper*distilled water*until you get pH= 7 (neutral). Next, drying is carried out for 12 hours at a temperature of 105℃ to obtain activated carbon.

2.2.2.1 Process for Making Ni-1.5%/Carbon Catalyst Using The Hydrothermal Method

The hydrothermal process was carried out first by making a Nickel (Ni) solution with a concentration of 1.5%. Then the activated carbon is mixed with a Nickel (Ni) solution, namely 70 grams of Nickel solution and 7 grams of activated carbon (ratio 10:1) which is then put into*autoclave* and closed tightly so that the solution does not evaporate. Heating process*autoclave* occurs in the oven at 150°C for 2 days or 48 hours. Next, filtering is carried out to separate the filtrate (nickel solution) and residue (Ni-1.5%/Carbon catalyst). After filtering, the catalyst was obtained and dried using an oven at a temperature of 105°C for 12 hours, a Ni-1.5%/Carbon catalyst will be obtained.

After the hydrothermal process, the catalyst reduction calcination stage is carried out to reduce the catalyst that has been produced from the hydrothermal process with a flow of hydrogen and nitrogen aimed at removing the oxygen content, this process takes place at a temperature of 400℃ for 2 hours, the catalyst resulting from reduction calcination is then analyzed using SEM- EDX.

2.2.3 Research Method II

In this second research method, the process of making the catalyst is not much different from the first method, the only difference is that the activation is carried out directly after carbonization of palm fiber, to see it more clearly in Figure 2 below:

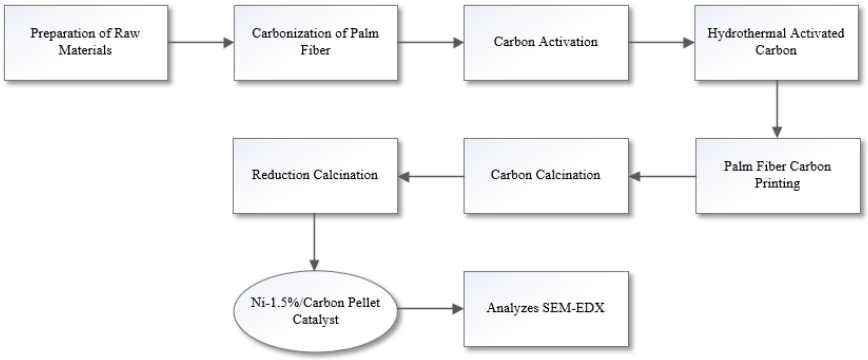


Fig.2 Flow diagram for making catalyst method II

This second research method begins with preparing the raw materials to be used by separating them from the dirt attached to the palm oil fiber. Then proceed with making active carbon, namely by carbonizing palm fiber to get palm fiber carbon and activating palm fiber using an activator, namely NaOH (Sodium Hydroxide). The next stage is making a catalyst using the hydrothermal method, this method uses activated carbon and a nickel (Ni) solution with a concentration of 1.5%. Next, the carbon printing process is carried out with a thickness variation of 0.5 cm^3, 1 cm^3 and 1.5 cm^3. Then, the catalyst calcination process is carried out by flowing with oxygen gas at a temperature of 300^OC for 1 hour then continued with reduction calcination by flowing hydrogen and nitrogen gas using a temperature of 400^OC for 2 hours, to obtain a Ni-1.5%/Carbon pellet catalyst. Finally, analysis is carried out using analysis*Scanning Electron Mikroscope* (SEM) and*Energy Dispersive X-Ray* (EDX).

3. RESULTS AND DISCUSSION

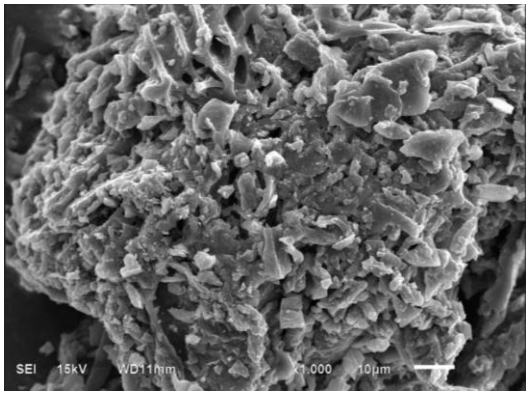
3.1 Size Variation of Ni-Carbon Catalysts

According to Norsamsi (2014), catalysts have various methods and each catalyst can be produced via a different route. The catalyst size variation data obtained from the image below can produce the desired product under operating conditions that are not too extreme. To produce an effective catalyst, the role of the support as a place for the active core to spread can increase the overall catalyst effectiveness, the size of the catalyst area. Ni carbon catalyst with a size of 0.5 cm^3, 1 cm^3, and 1.5 cm^3 Method I and Method II can be seen in Figure 3 below:

Fig.3 Activated carbon measuring 0.5 cm^3, 1 cm^3, and 1.5 cm^3

3.2 SEM Characterization of Activated Ni-Carbon Catalyst

Activated carbon is amorphous carbon whose surface area ranges from 300-3500 m2 /g and has been treated with steam and heat until it has a very strong affinity for absorbing various materials with a great ability, namely 25-100% of the weight of activated carbon. (Handika Gewa et al, 2017). This is related to the internal pore structure which causes activated carbon to have properties as an adsorbent. Activated carbon can adsorb gases and certain chemical compounds or its adsorption properties are selective, depending on the size or volume of the pores and surface area. To determine the characteristics of activated carbon, SEM analysis was carried out which aims to determine the characteristic morphology of activated carbon. The SEM test results can be seen in Figure 4 and Figure 5 below:



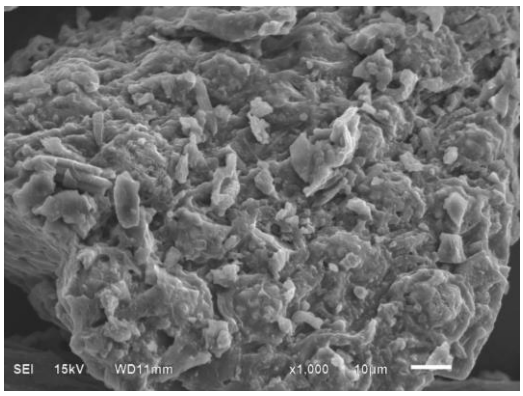


Figure.4 SEM Method I results

Figure.5 SEM Method II results

In the image above is the morphology of the active Ni-carbon catalyst from the results of SEM analysis. Based on the analysis results with a magnification of 1,000 on both catalysts and the length scale of the white thick line is 10 μm (10,000 nm). It can be seen from the picture the condition of the nickel crystal molecules that have been embedded in the pores of the active Ni-carbon catalyst. This shows that the hydrothermal process conditions

help carry nickel metal dispersed on the carbon surface. The content of constituent elements in the Ni-carbon catalyst can be determined through the EDX analysis test.

3.3 EDX Characterization of Activated Ni-Carbon Catalyst

To determine the elemental content of activated carbon, EDX testing is carried out. In samples analyzed by X-rays it is possible to determine each element that responds to the light waves. The results of the analysis test for the composition of the elements in activated carbon can be seen in Table 3 below:

Research result			
Element		**Weight (%)**	
Rumus	**Name**	**Metode I**	**Metode II**
C	Carbon	64,81	56,23
N	Nitrogen	17,10	15,11
O	Oksigen	8,17	19,95
F	Fluorin	0,41	0,52
Na	Natrium	0,30	0,15
Mg	Magnesium	0,11	0,08
Al	Alumunium	-	0,06
Si	Silikon	1,09	3,84
S	Sulfur	0,14	0,00
K	Kalium	0,13	-
Ca	Kalsium	0,10	0,18
Ni	Nikel	7,64	3,89

Based on the table above, it can be seen that the carbon produced is slightly more in the first method, namely 64.81%, while for the second method it is 56.23%. The high carbon content is due to the fact that many water molecules and volatile metres evaporate during carbonization. There are certain provisions on industrial scale catalysts for the amount of carbon detected, when compared with catalysts sold for marketing according to industry

standards which have a carbon amount of 29.5%. When compared with the data obtained above from method I and method II, the amount of carbon contained in the catalyst has met the standard, where this result is much higher when compared to catalysts that are sold on the market.

Based on the analysis data, there is still quite a lot of oxygen content, namely 8.17% in method I and 19.95% in method II. According to Aditya (2021), a combustion process requires oxygen so that it can take place easily. So the presence of oxygen in biomass can speed up the combustion process.

The addition of nickel during the hydrothermal process also affects the catalyst content. Where according to EDX analysis the nickel metal element in the catalyst absorbed in method I is 7.64% and for method II it is 3.89%. Due to the difference in treatment between method I and method II, it can affect the results of nickel absorption.

According to Wardani (2019), a low activation temperature and an activation time that is not long enough will result in the carbon not being activated properly, and allowing the Na obtained to be quite low. Based on the analysis results obtained, the Na element content value obtained in this study was 0.30% in method I and 0.15% in method II, this amount is quite low because the activation process uses thermodynamic principles where the higher the activation temperature used, the it doesn't take a long time and if you use a low temperature then the activation time needed is quite long for the activation process. For graphs of analysis results, see Figure 4 and Figure 5 below:

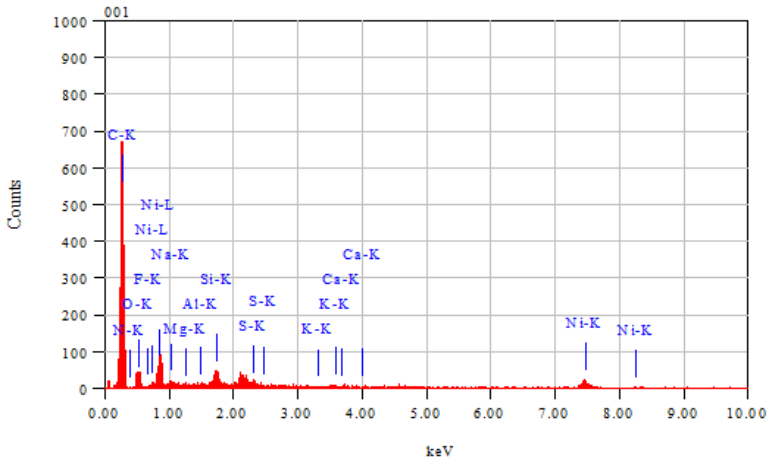


Fig.6 Spectrum of EDX Analysis Results Method I

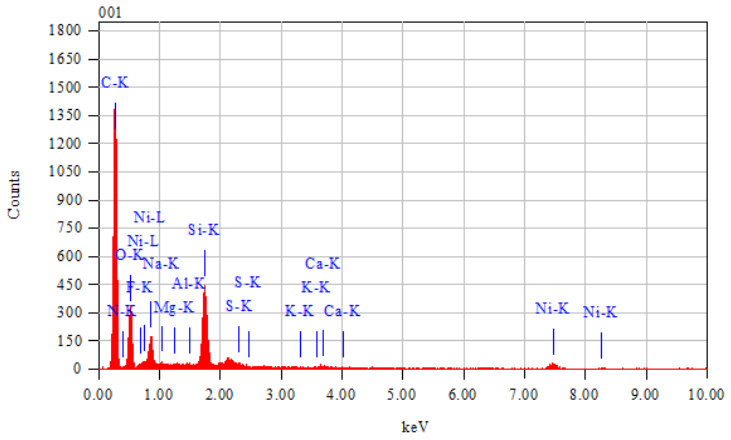


Fig.7 Spectrum of EDX Analysis Results Method II

3.4 Robust Test Method

Compressive strength is one of the parameters used to determine the strength or ability of a material or object to withstand pressure or load. For example, in the chemical industry in reactors, there are provisions that apply to the durability of catalysts which will be used in every industry both before and after introducing packing to see the durability of catalysts that are not easily brittle to withstand pressure. The methods used, such as chemical reduction or thermal decomposition, can produce different particle structures and distributions, which ultimately affect the compressive strength. Two types of printing procedures are compared here.

Method 1 is printing before hydrothermal, while method 2 is printing after hydrothermal. Procedure 1 can be used in industry, because the compressive strength for crushing is average and also the higher modulus, which means a better sample. Cracks in brittle solids are highly destructive to particle strength. It is therefore important to estimate the quantity of defects in the sample and analyze their effect on the strength of the particles. The compressive strength value of the catalyst is needed to determine the maximum strength. The following are the results of the compressive strength test on the Ni-carbon catalyst using method 1 and method II as follows:

(a) Method I (b) Method II

Figure.8 Compressive Strength Analysis Test Results

From the results obtained from the compressive strength test on the Ni-Carbon catalyst using method I, the result was 45.93 Newtons. Meanwhile, for method II, the test result was 17.63 Newtons. If the value is greater, the pressure test results will be better due to the influence of the pores in the catalyst. The influence of the pore content, where the catalyst pores contain nickel and other metals, can affect the material's ability to withstand pressure. The size of the pores also affects the compressive strength, larger and uneven pores can weaken the material structure and reduce the overall compressive strength.

4. CONCLUSION

Based on the research and discussion above, it can be concluded from the EDX test that method I produces a Ni-Carbon catalyst, the carbon element produced is 64.81%, while the nickel absorption is 7.64%. For method II the carbon produced is 56.23% and the nickel absorption is 3.89%. From the results, it is clear that method I results in greater data than method II where the nickel absorption capacity is quite small.

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