

Synthesis and Characterization of Catalyst Nickel/Activated Carbon From Palm Oil Fiber Using the Hydrothermal Method

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ABSTRACT – The Research has been carried out on the Nickel/Activated Carbon catalyst as a heterogeneous catalyst using the hydrothermal method using methods 1 and 2. Carbon from palm fiber is carbonized in a furnace at a temperature of 300°C for 1 hour and then activated using a NaOH solution for 2 hours. After that, the activated carbon was hydrothermalized in a nickel solution with a concentration of 1.5% for 48 hours with a ratio of 1:5. The catalyst was analyzed using SEM-EDX to see the characteristics of the catalyst and the elements contained in the catalyst and also tested for compressive strength using a texture analyzer. The analysis results show that the catalyst morphology of method 1 has a larger pore size than method 2, and the best method which produces a catalyst with the highest nickel content after hydrothermal at 7.59% is obtained using method 1, while method 2 has a nickel content of 4.07 %. For the compressive power of the catalyst, it was found that the method 2 catalyst was better than the method 1 catalyst because it was influenced by the pore size

Keywords: *Catalyst, Activated Carbon, Palm Fiber, Hydrothermal*

1. INTRODUCTION

Palm fiber is one of the wastes produced from the process of making palm oil. Palm fiber itself can only be used as personal fuel from palm oil processing factories. From 100% of fresh palm fruit that is processed, 12% of palm fiber will be produced (Juliantoni, Mucra and Febrina, 2018). This amount is a large figure if it is only used as personal fuel, so efforts need to be made to increase the economic value by making activated carbon from palm fiber.

Activated carbon can be defined as a carbon material with an amorphous structure and a large internal surface area with a high priority level. Activated carbon is categorized as non-graphitic carbon because it has a low density and porous structure. Activated carbon can be produced from materials containing carbon, one of which is palm fiber. When viewed from the chemical elements, palm fiber contains 28.28% cellulose, 27.86% lignin, and 34.78% hemicellulose, where these compounds include hydrocarbons which can be used as active carbon.

Activated carbon is effective as a catalyst in gas phase and liquid phase reactions. The process of making active carbon from palm fiber is by means of a carbonization process which is then followed by an activation process. The activation process can be carried out through two methods, namely chemically as well as physically. The choice of activator type will affect the quality of the activated carbon. Each type of activator will have a different effect or influence on the surface area and pore

volume of the activated charcoal produced. Chemicals that can be used as activators are acids, bases or salts including H_3PO_4 , HCl , NaOH , and ZnCl_2 (Sahara, Dahliani and Manuaba, 2017).

Based on the description above, research was carried out regarding the manufacture of activated carbon as a catalyst from palm fiber using nickel metal. This research was carried out using 2 methods which have differences in the catalyst synthesis stage. The method used is making carbon from palm fiber through a carbonization process followed by molding to get the desired size. The resulting printed palm fiber carbon is then calcined to remove the adhesive in the printing process and continues with the activation process using the NaOH activator. The resulting activated carbon is continued to the catalyst synthesis stage using an autoclave. The resulting Ni-Carbon catalyst will be subjected to reduction calcination which will then be analyzed. The Ni-Carbon catalyst produced is 1 cm^3 in size with a concentration of 1.5% which will be analyzed using SEM-EDX.

2. RESEARCH METHODS

2.1 Materials and Tools

The materials used in this research include palm fiber, glue, NaOH , Nickel Nitrate, Nitrogen, Hydrogen, aquadest, filter paper, and pH paper.

The tools used in this research include beakers, reactors, furnace, oven, autoclave, hot plate, cutters, magnetic stirrers, grinder, analytical balance, buchner funnel, hydraulic press, and porcelain cup.

2.2 Implementation of Research

2.2.1 Experimental Design

This research was carried out using 2 methods, where the difference between these two methods occurred at the catalyst synthesis stage. In method 1, catalyst synthesis is carried out when the activated carbon is in solid form. whereas in method 2, catalyst synthesis is carried out when the activated carbon is in powder form.

The research matrix used in conducting this research includes:

Table 1. Catalyst Manufacturing Matrix

Treatment	Ratio of Nickel solution: activated carbon	Ratio of NaOH solution: carbon	Nickel solution concentration	Synthesis time	Catalyst size
Method 1	5:1	5:1	1,5%	48 hours	1 cm^3
Method 2	5:1	5:1	1,5%	48 hours	1 cm^3

2.2.2 Preparation of Raw Materials

The raw material used is palm fiber obtained from waste palm oil industry in Jambi province. Palm fiber is washed first to remove the dirt on the palm fiber then dry it in the sun and then in the oven at 105°C .

2.2.3 Palm Fiber Carbonization

The dried palm fiber is put into the furnace for the carbonization process was carried out using a temperature of 300°C over time for 1 hour which will produce palm fiber carbon. The resulting product is then ground and sifted using sieve $355 \mu\text{m}$ (45 mesh).

2.2.4 Preparation for Making Carbon 1 cm^3

Palm fiber carbon printing uses Fox glue adhesive, with a ratio of 1:2, namely 30 grams of palm fiber carbon and 60 grams of Fox glue. Mix and stir for 10 minutes. The results of mixing palm fiber

carbon and fox glue were then printed using a hydraulic press for 30 minutes. The result of pressing using a hydraulic press is palm fiber carbon plates which will then be dried for 20 minutes using an oven at a temperature of 105°C.

Printed palm fiber carbon that has been dried for 20 minutes will then be cut to a size of 1cm³. After cutting, the printed palm fiber carbon will be dried for 12 hours using a temperature of 105°C.

2.2.5 Carbon Calcination.

The palm fiber carbon calcination process is carried out for 1 hour at a temperature of 300°C using nitrogen. The calcination process aims to remove the glue content in the carbon. Nitrogen gas is used as a driving force that enters the carbon pores to help remove the glue contained in the carbon.

2.2.6 Carbon Activation

Palm fiber carbon is mixed with NaOH solution for activation with a ratio of carbon to NaOH solution, namely 5:1. After mixing the NaOH solution with carbon, stir it using a temperature of 30°C for 2 hours on a hot plate with using a magnetic stirrer. The activated carbon will then be washed using distilled water until it reaches neutral pH = 7. The washed activated carbon will then be dried in an oven at a temperature of 105°C for 12 hours.

2.2.7 Synthesis of Ni-1.5%/Carbon Catalyst (Hydrothermal Process)

The nickel solution is first made with a concentration of 1.5%. The 1.5% Nickel solution that has been made will be mixed with activated carbon in a ratio of 5:1, namely 35 grams of 1.5% Nickel solution and 7 grams of activated carbon which is then put into an autoclave and closed tightly. Then, for the heating process, the autoclave is placed in the oven with temperature 150°C for 48 hours. After the hydrothermal process for 48 hours, filtering was carried out to separate the filtrate and residue, the catalyst was then obtained dried in the oven at 105°C for 12 hours.

2.2.8 Reduction Calcination

The catalyst produced after the hydrothermal process will then be subjected to reduction calcination using hydrogen gas. The reduction calcination process lasts for 2 hours using a temperature of 400°C. The result obtained is a Ni-1.5%/Carbon catalyst which will then be carried out by SEM-EDX analysis.

2.3 Analysis of Research Parameters

2.3.1 SEM-EDX analysis

SEM is a type of electron microscope that uses electron beams to image the surface profile of a sample. The work of the SEM itself is that the surface of the sample that is hit by the beam reflects the beam back or produces secondary electrons. Then the detector in the SEM will detect the reflected electrons. The sample surface area hit by the beam is scanned throughout the observation area.

Energy Dispersive X-ray Spectroscopy (EDX) is an analytical technique for analyzing elements or chemical characteristics of the specimen. This characterization is based on studies of the interaction of multiple X-ray excitations with the specimen. The ability to characterize is similar to most of the basic principles that state that each element has a unique atomic structure. and is a characteristic feature of the atomic structure of an element, thus supporting X-rays to identify it.

2.3.2 Compressive Strength Test

According to Knudsen (2017), the compressive strength test is used to determine the capacity or strength and to determine the durability of the catalyst, for example in the chemical industry where there are provisions that apply to the durability of the catalyst which will be used in every industry both before and after introducing the packing to see the durability of the catalyst. which is 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. The tool used in the durability test is a Texture Analyzer. Texture Analyzer is used to determine the physical properties of catalysts which are related to the durability or strength of a material under pressure (Smewing, 1999).

3. RESULTS AND DISCUSSION

3.1 SEM-EDX analysis

In this research, the sample analyzed using SEM was a 1 cm³ Ni-1.5%/Carbon catalyst which had 2 different treatment methods. Therefore, it can be seen from the analysis results how the two methods influence the characteristics of the catalyst produced. The catalyst image resulting from SEM analysis can be seen in **figure 1**.

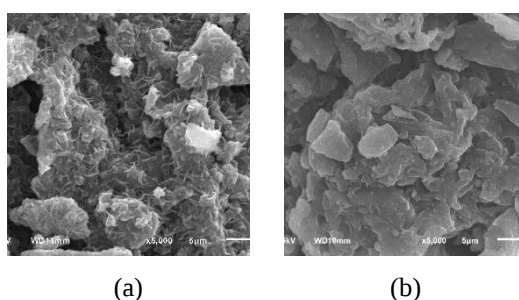


Figure 1. Characteristics of the Ni-1.5%/Carbon catalyst (a) Method 1, and (b) Method 2

The image above is morphological appearance of the Ni-1.5%/Carbon catalyst using SEM analysis test with a magnification of 5 µm. It can be seen from the morphology of the Ni-1.5%/Carbon catalyst method 1, image (a) that the catalyst surface has large pores and has a rather rough particle shape and has a distance between each other, whereas in the Ni-1.5%/Carbon catalyst method 2 image (b) the catalyst surface has quite small pores, and has particles with a flat or smooth surface but are close together or close to each other

Activated carbon from palm fiber carbonized using NaOH. aims to raise the level acidity on the surface of the activated carbon so that the surface of the activated carbon will have more positive ion charge. The carbon activation process will cause its absorption capacity to increase compared to carbon without activation. The activation process will dissolve impurities so that the pores open more. In this way the absorption process will be maximized. This is what causes an increase in the absorption ability of Nickel metal on the surface of activated carbon. After activation, the activated carbon is washed using distilled water and then dried again using an oven. This aims to remove water content from the activation process. Then, the activated carbon goes through a hydrothermal process using a nickel solution. The hydrothermal process aims to develop nickel metal in the pores of active carbon by dissolving nickel in activated carbon using high temperature and pressure so that nickel enters and sticks to the active carbon. The following are the Energy Dispersive X-Ray (EDX) results which explain the catalyst element content Ni-1.5%/Carbon in method 1 and method 2.

Table 2. Element Content in Ni-1.5%/Carbon Catalyst Methods 1 and 2

Element Number	Element Symbol	Element Name	Weight (%) Method 1	Weight (%) Method 2
6	C	Carbon	54,39	64,76
7	N	Nitrogen	15,47	18,99

8	O	Oxygen	20,18	10,02
9	F	Fluorine	0,41	0,49
11	Na	Sodium	0,07	0,14
12	Mg	Magnesium	0,26	0,10
13	Al	Alumunium	0,13	0,20
14	Si	Silicon	0,93	0,38
19	K	Potassium	0,22	0,10
20	Ca	Calcium	0,35	0,74
28	Ni	Nickel	7,59	4,07

The EDX analysis results on the Ni-1.5%/Carbon catalyst using method 1 to obtain the Carbon content(C) is (54.39%), where carbon is the carrier medium in this research, in method 1 the nickel (Ni) that is carried is 7.59% of the total content in the Ni-1.5%/Carbon catalyst method I. This embedded nickel affects the surface area of the active site of the catalyst, becoming wider and also affects the activity and selectivity of the catalyst. The embedded nickel is expected to be able to be evenly dispersed throughout the surface and pores of the support material. Based on research conducted by Rasidi et al (2015), selectivity and catalyst activity generally depend on catalyst preparation and support characterization. The active metal components are distributed evenly throughout the pores of the support material so that a good and homogeneous dispersion system is formed.

Meanwhile, the results of EDX analysis on the Ni-1.5%/Carbon catalyst using method 2, the Carbon (C) content was (64.76%) and the nickel (Ni) contained is 4.07% of the total content in the Ni-1.5%/Carbon catalyst. It can be seen that method I and method II in making the Ni-1%/Carbon catalyst have results that are not much different. However, from these two methods there is still little nickel, so it is still not effective for use in industry. According to Sondari (1999), generally commercial catalysts used by the oleochemical industry in Indonesia have a Ni content of 20-25% of the total catalyst components.

The process of making the Ni-1%/Carbon catalyst is influenced by the processes that have been carried out, namely pre-treatment of raw materials, carbonization, activation, hydrothermal and calcination. At the pretreatment stage, the raw materials are washed with running water and then dried in the sun using rice sacks which allow the raw materials to be contaminated by other substances. In fact, according to Winata et al (2020), pretreatment really determines the quantity and quality that will be produced, this can help reduce or eliminate various materials or compounds that can hinder the next stage.

Then in the carbonization stage, the raw material is carbonized at a temperature of 300°C for 1 hour to obtain carbon, but it turns out that the operating conditions used are less than optimal. Choosing the right carbonization temperature needs to be done to produce carbon with a high surface area, only carbon remains, however Sometimes results in the form of ash are also obtained if the carbonization process does not take place in an oxygen-limited environment. Carbon formation can occur at temperatures of 400-600°C.

In the chemical activation process which uses room temperature and is carried out for 2 hours, it can reduce the carbon adsorption process, where in research conducted by Rispiandi et al (2016), it was stated that activated charcoal activated with 2.5% NaOH had water content. lowest at 28 hours activation time, namely 8.40 ± 0.72 , this was done to determine the hygroscopic properties of activated charcoal, because the higher the water content bound to the activated charcoal, the pores of the activated charcoal will close and result in reduced adsorption ability.

The hydrothermal process was carried out at a temperature of 150 °C and a time of 48 hours, resulting in less than optimal characteristics of the synthesis results. Apart from that, it used nickel

with a concentration of 1.5%, which is said to have a low nickel content so that the nickel could not be absorbed properly, based on research by Devianti et al. (2014), that hydrothermal temperature and time will affect growth crystals on the surface of the supporting material. Then calcination was carried out at a temperature of 400 °C for 2 hours which can be said to be less than optimal, because the calcination temperature can have an effect on the formation of a wider pore structure, this is said by Arlofa (2016), that a high calcination temperature causes the formation of a wider pore structure. area in activated carbon, so that the absorption process is more optimal. The optimum calcination temperature obtained in this experiment was 800 °C with an absorption efficiency of 89.467%. So it can be concluded that in making the Ni-1.5%/Carbon catalyst using method I and method 2 are less than optimal because there are many inappropriate processes which affect the resulting catalyst results

3.2 Compressive Strength Test

The compressive strength test results of a 1 cm³ Ni-1.5%/Carbon catalyst using 2 different methods were tested using a texture analyzer as follows. From the compressive strength test carried out on the Ni-1.5%/Carbon catalyst using method 1, a result of 29,73 Newtons was obtained, while on the Ni-1.5%/Carbon catalyst using method 2, a result of 37,41 Newtons was obtained.

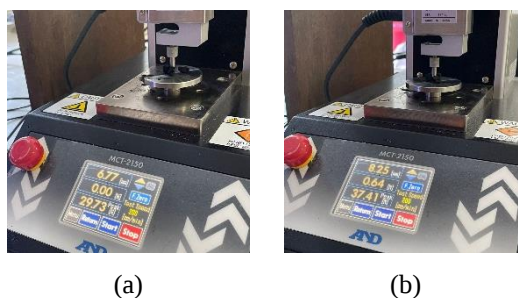


Figure 2. Compressive Strength Test Results for Ni-1.5%/Carbon Catalyst (a) Method 1, and (b) Method 2

According to Liku et al (2021), the higher the value of the pressure test, the better it is, this is related to the higher pressure the catalyst can accept. The factors that influence the compressive strength of the catalyst are the pores on the catalyst. A catalyst that has a small pore size will distribute pressure and prevent damage to the catalyst. Meanwhile, large pore sizes and uneven distribution will cause a weak structure of the catalyst. In the SEM-EDX test results, the Ni-1.5%/Carbon catalyst method 1 has particles that are far from each other. so it has quite large pores, while the Ni-1.5%/Carbon catalyst method 2 has particles that are close to each other so they have small pores

This is influenced by different treatment methods, in method 1 the catalyst synthesis stage is carried out using activated carbon that has been printed first and then synthesized and continued with the next process stage. Meanwhile, in method 2, catalyst synthesis is carried out using activated carbon powder, which later after the synthesis stage is carried out by a printing process. So it can be concluded that the Ni-1.5%/Carbon catalyst method 2 has a stronger catalyst structure compared to the Ni-1.5%/Carbon catalyst method 1.

4. CONCLUSION

Based on the results of the research that has been carried out, it can be concluded that the effect of using 2 methods in making the catalyst does not really have an influence on the characteristics of the Ni-1.5%/Carbon catalyst. In the SEM-EDX analysis that has been carried out, it can be seen that nickel can be embedded or absorbed in the pores of the catalyst during the hydrothermal process but

it is not very effective, where the nickel contained in the catalyst in method 1 is only 7.59% and in method 2 it is 4.07%, but method 1 is recommended for use in further research.

For the effect of using methods 1 and 2 in making catalysts on the compressive power of the catalyst, it provides a different compressive power, where the method 1 catalyst has a compressive power of 29.73 N, while the method 2 catalyst has a compressive power of 37.41 N. It can be seen that the method catalyst 2 has stronger compressive power than catalyst method 1.

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