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Active Charcoal from Palm Kernel Shells as a Catalyst in The Production of Biodiesel

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Abstract

Palm kernel shells are one of the main wastes for processing of palm oil. Palm kernel shells are waste that can be used as a catalyst in the biodiesel production. Besides many natural ingredients, the price of catalyst prepared from these materials is also relatively cheap compared to other catalysts, make the biodiesel production more sustainable, and environmentally friendly. This study aims to make activated carbon from palm shell through the process of carbonization and chemical activation. The carbonization process was carried out at 550°C for 3 hours until charcoal was formed. While the activation process was carried out using $ZnCl_2$ activators with concentrations of 0.1 M, 0.25 M, 0.5 M and 1 M which were activated for 4 hours at 90°C. Based on the results of the FTIR analysis the presence of O-H and C-O bonds indicates that the carbon produced from the palm kernel shell tends to be polar (volatile). Thus the charcoal produced can be used as catalyst in the biodiesel production. For the SEM test results, it can be seen morphologically that more pore crystals are added and are still brittle at a $ZnCl_2$ concentration of 0.1 M. Based on FTIR and SEM analysis, the best activated charcoal was activated charcoal with a concentration of 0.25 M $ZnCl_2$.

INTRODUCTION

The production of biodiesel can be done with various types of catalysts: homogeneous catalysts, heterogeneous alkaline catalysts, and ion exchange resins. Astuti & Mufrodi (2019) have made biodiesel from used cooking oil by a continuous process with a homogeneous catalyst. Biodiesel was also made with a homogeneous catalyst in a batch system (Agus and Zahrul, 2019; Soegiantoro et al., 2019). The disadvantage of homogeneous catalysts is the use of catalysts that cause the reactor to corrosive, separation catalysts require cost and this process produces chemical waste (Gohain et al., 2020). In operation, heterogeneous catalysts tend to be easier to separate because the phase used is different from the reaction product and reusability (Shan et al., 2018; Semwal et al., 2011). Another advantage of using solid catalysts is that the amount of catalyst required is much less than homogeneous catalysts (Dossin et al., 2006; Mbaraka & Shanks, 2006). Synthesis with enzyme catalysts can be carried out at room temperature with high yield (Semwal et al., 2011) but this process requires high costs and a more complicated purification process. (Chen et al., 2017). Commonly, the catalysts used as heterogeneous catalysts are synthetic catalyst which are relatively expensive. Therefore, it is necessary to has a material that can be made into a catalyst at a low price, and noticeable reusability (Dhawanea et al., 2018). Heterogeneous catalysts can be derived from renewable sources like clays and rocks (Doyle et al., 2017), shale rock (Alismaeela et al, 2018), zeolite (Mufrodi et al., 2020), shells, bones, ashes from plant/tree, natural sources, and large-scale industrial wastes (Shan et al., 2018). Catalysts prepared from these materials could make the biodiesel product more sustainable, environmentally friendly and cost-effective. In

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addition to the many natural ingredients, the price is also relatively cheap compared to other catalysts. This paper proposes palm kernel shells as material to make catalyst.Activated carbon is often used in several heterogeneous catalytic processes (Lazaro et al., 2015). Activated carbon has many advantages such as availability, which is mostly waste management, which costs low and stability at low pressures and temperatures. Excellent surface properties like large specific surface area and high porosity make carbon an exceptional contender for catalyst support (Dhawanea et al., 2018). In addition, activated carbon has a good catalyst buffer because it is inert, the surface can be modified, activated carbon is used as a buffer because of its nature (Lazaro et al., 2015).

Palm kernel shells are one of the major palm oil processing wastes. The amount of palm oil shell waste in Indonesia is around 8.7 million tons / year. Palm oil shells have a composition of cellulose (26.27%), hemicellulose (12.61%), lignin (42.96%) and have a higher density than wood, reaching 1.4 g / mL. The greater the density of the raw material, the greater the absorption of activated charcoal will be so that it is good to be used as activated charcoal (Dejean et al, 2017). Activated charcoal has a surface area of pore volume, a pore size that is spread on the surface of active charcoal. Chemical compounds that can be used as activating materials include KCl, NaCl, $ZnCl_2$, $CaCl_2$, $MgCl_2$, H_3PO_4, Na_2CO_3 and other mineral salts (Dejean et al, 2017; Tang et al, 2018).. This study aims to get the characteristics of palm kernel shell as catalyst. The benefit of this study is to obtain the best palm oil shell size data and make activated palm oil shells.

MATERIALS AND METHODS

Research Procedure

Palm kernel shells were cleaned from unwanted impurities, then the water content was removed with dehydration using an oven at 100°C for 1 hour, and physical activation was conducted by inserting palm kernel shells into the Muffle for the process of drying at 550°C for 3 hours until the charcoal is formed. For sieving, the charcoal that has been removed from the muffle was mashed using porcelain mortar, then it was screened using screening to get the results of 200 mesh sieves. Then 500 ml of 0.1 M zinc chloride solution was made. For chemical activation, 50 grams of palm kernel shell charcoal was weighed and put in 250 ml of 0.1 M $ZnCl_2$ solution in three neck flask, then a cooling ball, stirrer and thermometer were installed. The mixture was then stirred at 90°C for 4 hours. After 4 hours, the solution as cooled and then filtered with Whatman 42 filter paper, after that solids and filtrate were then separated. The filtrate was then thrown out and the solids was neutralized by dripping the aquadest into solids until pH is neutral. After the pH is neutral, the filtrate was tested by dripping an NaOH. If a cloudy solution is formed after the NaOH drops, the neutralization was continued. Neutralization was stopped if no cloudy solution was formed on the filtrate after NaOH drops, this indicates sulfate is already free of solids. The solid was then dried in an oven at 130°C for 24 hours. All steps were repeated for a concentration of 0.25M; 0.5M; 1M.

Research Analysis

Samples were analysed using Fourier Transform Infrared (FTIR) scanning electron microscopy (SEM). The FTIR is MB3000 type with specification: resolution >0.7%, short term stability of 0.09%, frequency repeatability of 0.001 cm^{-1} and frequency accuracy of 0.06 cm^{-1}. The type of SEM is Thermo Scientific Nicolet iS10 that has spectral range of 7800 -350 cm^{-1} and equipped with fast recovery deuterated triglycine sulfate (DTGS) as detector.

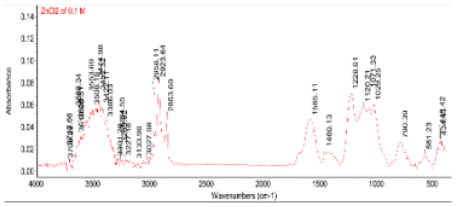
RESULTS AND DISCUSSION

Analysis of Fourier Transform Infrared (FTIR)

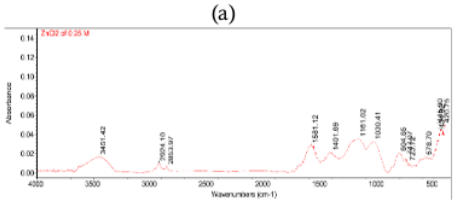
To find out the microstructure in the form of a functional group from the four activated carbons obtained, microstructure analysis was carried out with the FTIR instrument. The FTIR test results show the vibration of each group formed. The various types of wavelengths produced at each peak occur so that the microstructure can be known in the palm kernel shell carbon in each variation. After analyzing the graph results of the tool, an indication of the functional group is performed. The FTIR test results are presented in Figure 1.

Based on Figures 1 and the difference in charcoal wave peaks from activated palm shells, information is obtained that the functional groups contained in each sample are listed in Table 1.

Based on the results, although there are observed peak differences, the overall spectrum



(a)



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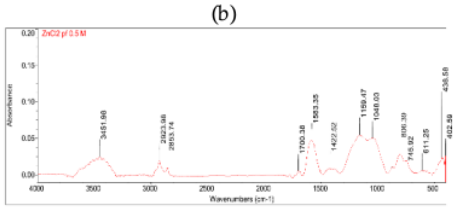
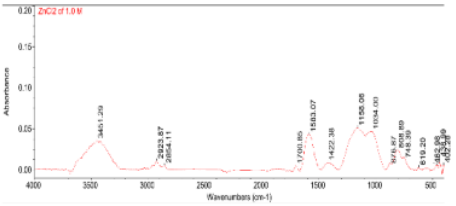


(c)



(d)

Figure 1. FTIR Test Results of Charcoal from Activated Palm Shells by $ZnCl_2$ (a) 0.1 M (b) 0.25 M $ZnCl_2$ (c) 0.5 M (d) 1 M.

obtained for activated palm kernel shell carbon is not too different. From the spectrum, information is obtained that the functional groups contained in palm oil activated carbon are as follows. In the absorption band 2853.69 cm^{-1}, 2853.97 cm^{-1}, 2853.74 cm^{-1} and 2854.11 cm^{-1} there is a vibration of C-H. Uptake of 1029.25 cm^{-1}, 1030.41 cm^{-1}, 1048.57 cm^{-1} and 1034.00 cm^{-1} showed the presence of aliphatic C-O indicating that the sample was activated carbon.

The activation process on palm oil shell carbon has also formed C = C (Aromatic) bonds at wave numbers 1585.11 cm^{-1}, 1581.12 cm^{-1}, 1583.35 cm^{-1} and 1583.07 cm^{-1}. This indicates that carbonization and activation into carbon will increase carbon group bonds. These compounds are active compounds of hexagonal structure of charcoal.

The presence of water vapor in the activated charcoal activation process and in the preparation of samples, apparently still plays a role with the identification of OH groups in activated charcoal namely at wave numbers 3508.18 cm^{-1}, 3451.42 cm^{-1}, 3451.98 cm^{-1} and 3451, 29 cm^{-1}. The cluster can be derived from the reaction between water vapor and free compounds on the activated carbon / carbon surface and not from the raw material of oil palm shell charcoal. Furthermore, the absorption band obtained at wave number 581.23 cm^{-1}, 578.70 cm^{-1}, 611.25 cm^{-1} and 619.20 cm^{-1} which informs the presence of C-Cl. The C-Cl bond is formed because carbon binds to the remaining Cl from the activator, $ZnCl_2$.

Analysis of Scanning Electron Microscopy (SEM)

In the activation process there are three processes in the development effort, namely to open pores that previously cannot be entered, form new pores and expand existing pores. In this study several analyzes were carried out to determine the characteristics and morphology of the activated charcoal surface of oil palm shells. Catalyst Surface Morphology. To find out the shape of the surface morphology of the catalyst before and after activation, it was done using Scanning Electron Microscope (SEM).

The results of identification of oil palm shell charcoal using Scanning Electron Microscope (SEM) can be seen in Figures 2(a) – (d) with a magnification of 10000. From the morphology in Figures 2(a) – (d), it can be seen that where the constituent particles are not arranged regularly, although there are also a small portion arranged regularly. The resulting particles vary, some are small and large on the submicron scale. The arrangement of irregular amorphous particles does not appear in all parts such as crystalline solids. So that Figures 2(a) – (d) are solids that are predominantly amorphous.

The morphology in Figures 2(a) – (d), shows a non-uniform particle size. The oil palm shell charcoal powder used was to pass the 200 mesh sieve (particle size = 74 microns = 0.0740 mm). This is due to the effect of even grinding, so that the difference in pressure during grinding causes different grain sizes. In Figure 2(a), morphologically there are more uniform pores with a diameter of ± 1 micrometer. So that it can be concluded that in Figure 2(a), the concentration of

Table 1. Data on FTIR Analysis Results

Functional Groups	Wave Number of Activated Palm Shell Charcoal $ZnCl_2$ (cm^{-1})			
	0.1 M	0.25 M	0.5 M	1 M
O-H (Alcohol OH Stretch)	3508.18 (strong sharp)	3451.42 (strong broad)	3451.98 (strong broad)	3451.29 (strong broad)
Carboxylic Acid OH Stretch	3508.18 (strong)	3451.42 (strong)	3451.98 (strong)	3451.29 (strong)
C-H (Alkana)	2923.64 (strong)	2924.10 (strong)	2923.98 (strong)	2923.87 (strong)
C=C (Aromatik)	1585.11 (weak)	1581.12 (weak)	1583.35 (weak)	1583.07 (weak)
NO2	1585.11 (strong)	1581.12 (strong)	1583.35 (strong)	1583.07 (strong)
C-H (Strecth)	2853,69 (weak)	2853,97 (weak)	2853.74 (weak)	2854.11 (weak)
C-O Eter	1120.21	1161.02	1159.16	1158.06
C-O Alifatik	1029.25	1030.41	1048.57	1034.00
C-Cl	581.23	578.70	611.25	619.20

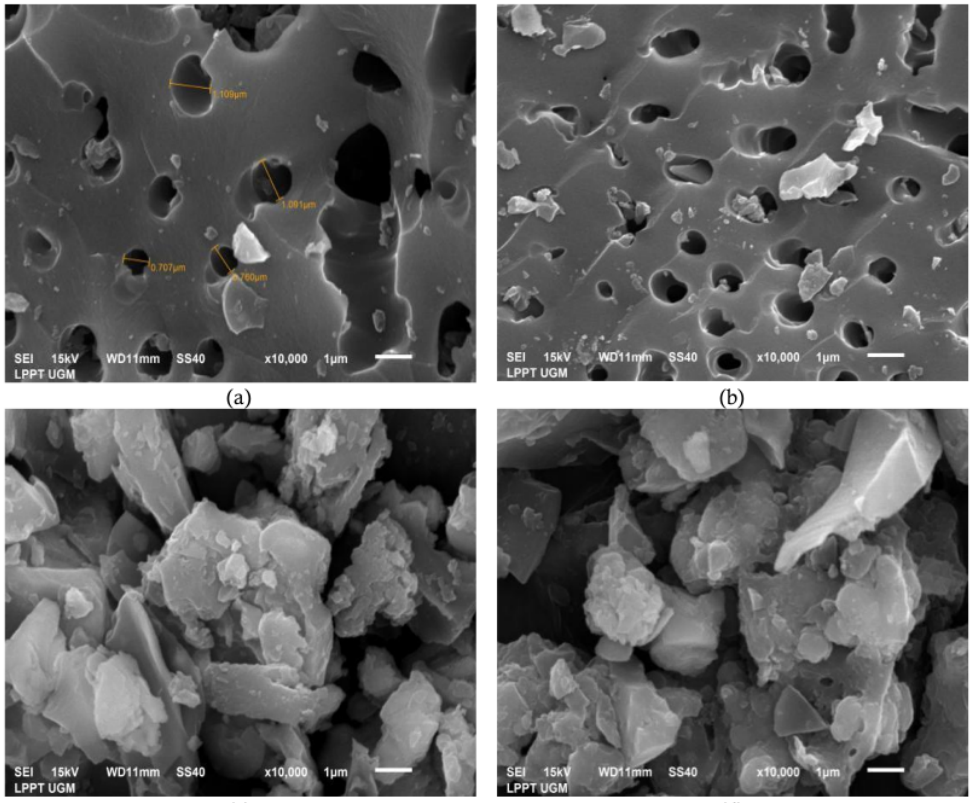


Figure 2. SEM analysis of oil palm shell charcoal activated $ZnCl_2$ (a) 0.1 M (b) 0.25 M $ZnCl_2$ (c) 0.5 M (d) 1 M.

ZnCl2 0.1 M seen morphologically has a greater number of porous crystals, uniform, evenly distributed and still fragile compared to the number of pores in Figures 2(b), (c), and (d).

In carbonization on charcoal using a temperature of 550° C for 3 hours and the activated charcoal activation process using a temperature of 130° C for 24 hours, the difference in temperature used causes the formation of impurities which are thought to be ash. Ash is the result of degradation of inorganic compounds or minerals by high temperatures. The formation of ash in activated charcoal and charcoal can be seen in Figures 2(c) and (d). The image that is white is suspected the content of ash attached to the material. In Figures 2(a) and (b), it can be seen that the ash content of

the charcoal is lower than the Figures 2(c) and (d). Whereas, in Figures 2(c) and (d) the formation of ash tends to be more so that it causes the structure of the pores to be formed. The use of high temperatures and longer activation times causes more degradation of inorganic compounds. Degradation by high temperatures causes inorganic deposits which attach more to the material. Therefore, the higher the temperature used, the higher the ash formed. The morphology in Figure 2 shows that there are contents in the pore after being given an activation treatment. Although it is known that the active charcoal pore is filled after the activation treatment is carried out, further testing is needed to find out the elements that fill the activated charcoal.

CONCLUSION

Function groups as OH (Alcohol OH Stretch), Carboxyloic Acid OH Stretch, CH (Alkanes), C = C (Aromatic), NO 2 , CH (Stretch), CO Ether, Aliphatic CO and C-Cl in charcoal were obtained from FTIR analysis. Activated palm shells are activated by 0.1 M, 0.25 M, 0.50 M and 1 M. Based on the results of the FTIR analysis the presence of O-H and C-O bonds indicates that the carbon produced from the palm kernel shell tends to be polar (volatile). Thus the charcoal produced can be used as catalyst in the biodiesel production. For the SEM test results, it can be seen morphologically that more pore crystals are added and are still brittle at a $ZnCl_2$ concentration of 0.1 M. Activated charcoal forms a brittle crystalline solid structure.

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