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Synthesis of Nano Zinc Oxide Heterogeneous Catalyst Supported with Fly Ash (NZO/FA) for Kapok Seed Oil (*Ceiba pentandra*) Transesterification

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ABSTRACT

Biodiesel was produced through a transesterification reaction between kapok seed oil (MBK) and methanol in a laboratory-scale batch reactor using a heterogeneous nano zinc oxide catalyst supported by fly ash (NZO/FA). This catalyst is synthesized through impregnation, precipitation, coprecipitation, and calcination methods at 500°C. To enlarge the pores of fly ash (FA), activation is carried out using hydrochloric acid. Furthermore, NZO/FA catalysts were characterized using Brunauer Emmett Teller (BET) method and Fourier Transform Infra-Red (FTIR) spectroscopy. The activity of the synthesized NZO/FA catalyst has been tested through transesterification of kapok seed oil (MBK) with a variable tranacetrification time of 3-5 h with an interval of 0.5 h. The research results showed that FA pretreatment could significantly increase the activity of the ZNO/FA catalyst, with the highest biodiesel yield of 95.93% obtained at a transesterification time of 4.5 h. It has been proven that the synthesized NZO/FA catalyst has great potential for transesterifying MBK into biodiesel

Keywords: *Zinc oxide; fly ash; heterogeneous acid catalyst; kapok seed oil; biodiesel.*

ABSTRAK

Produk biodiesel dihasilkan melalui reaksi transesterifikasi antara minyak biji kapuk (MBK) dengan metanol pada reaktor batch skala laboratorium menggunakan katalis heterogen nano zinc oksida berpenyangga fly ash (NZO/FA). Katalis ini di sintesis melalui metode impregnasi, presipitasi, kopresipitasi, dan kalsinasi di temperatur 500°C. Untuk memperbesar pori-pori fly ash (FA) maka dilakukan aktivasi menggunakan asam klorida. Selanjutnya katalis NZO/FA dikarakterisasi menggunakan metode Brunauer Emmett Teller (BET) dan Spektroskopi Fourier Transform Infra-Red (FTIR). Katalis NZO/FA hasil sintesis telah diuji aktivitasnya melalui transesterifikasi minyak biji kapuk (MBK) dengan vaiable waktu trenasetrifikasi 3-5 jam dengan interval 0,5 jam. Hasil penelitian menunjukkan bahwa *pretreatmen* FA dapat meningkatkan secara signifikan aktivitas katalis ZNO/FA, dengan yield biodiesel teringgi sebesar 95,93% diperoleh pada waktu tranesterifikasi 4.5jam. Terbukti, bahwa Katalis NZO/FA hasil sintesis sangat berpotensi untuk mentransesterifikasi MBK menjadi biodiesel.

Keywords: *Seng oksida; fly ash; katalis heterogen asam; minyak bapuk, biodiesel.*

INTRODUCTION

To reduce greenhouse gas emissions, Indonesia targets biofuels to become 46% of national transportation energy sources by 2050. This was conveyed at the *Conference of Parties* (COP) 26, 2021 as *Nationally Determined Contributions* (NDC) in the *Long-term Strategy on Low Carbon and Climate Relience* 2-5- (LCCR 2050) mission [1]. The target is also shown to avoid an energy crisis

in the future as a result of the use of fossil fuels to meet energy needs in daily life, while fossil fuels are included in the limited number of non-renewable natural resources [2]. One of the widely developed biofuel products is biodiesel. Biodiesel is considered the best solution to overcome problems that arise in the use of fossil energy, such as global warming, climate change, and air pollution [3]. Biodiesel also offers several other advantages, such as being environmentally friendly, easily biodegradable, non-toxic, and containing low greenhouse gas emissions [4]. In this case, the availability of raw materials is a major aspect in the development of biodiesel in every country. One of the abundant raw materials in Indonesia is kapok seed oil (MBK). MBK is easy to find because in Indonesia there are 7,630 hectares of kapok plantations. In addition, the oil content in kapok seeds is also quite high, ranging from 25-40% so that MBK was chosen as raw material for making biodiesel in this study [5, 6]. The selection of MBK is also considered the best effort to overcome national food security problems because it does not compete with the food industry for raw materials, can be mass produced, and does not have an impact on deforestation [3, 7].

Biodiesel itself is produced through a transesterification reaction between triglycerides derived from oil or fat with short-chain alcohol using the help of catalysts. The use of catalysts is intended to accelerate the occurrence of reactions with more optimal results because transesterification reactions without the help of catalysts will run very slowly [8]. Based on the phase, catalysts are divided into 2, namely homogeneous and heterogeneous catalysts. Homogeneous catalysts have a high degree of selectivity so they are not easily poisoned by the presence of impurities, but are difficult to separate from the product, less stable at high temperatures, can form soap, reduce biodiesel yield, and produce toxic and hazardous waste [9, 10, 11]. To overcome this problem, a heterogeneous catalyst was developed that offers several advantages such as being easily separated from the product, can be used repeatedly, does not form soap and is stable at high temperatures [10]. Therefore, in this study, heterogeneous catalysts were used to accelerate the transesterification reaction that occurred.

Siswoyo and Asri (2022) have developed NZO/FA as a catalyst in making biodiesel through transesterification reactions [12]. ZnO was chosen as a heterogeneous catalyst because it is able to produce *high* biodiesel yields, is not toxic, is cheap, can be regenerated so as to reduce production costs, and is environmentally friendly [10, 12, 13]. But a ZnO particle size of about 232.48 nm can cause the product to be difficult to separate from the residue. Therefore, a catalyst support is needed to facilitate the product separation process. In this case fly ash *(*FA) was chosen as a support for ZnO catalysts because it has a high content of SiO_2 (41-55%) and Al_2O_3 (20-25%) so that it can support oxides and active metals catalytically [14, 15]. In addition, FA is also considered as the best alternative as a supported with low prices and abundant quantities in Indonesia. Because Asri, et al. in previous studies used gamma-Al_2O_3 and *Multiwalled Carbon Nanotubes* (MWCNTs) as a catalyst suppoted which are expensive [2, 3, 4, 7]. Given that in the previous study the highest biodiesel yield was around 51.01% [12], then in this study the author tried to optimize the activity of NZO/FA catalysts in order to get high biodiesel yields through modifications to make NZO/FA catalysts by adding an FA activation process using HCl solution. So that the catalyst is expected to work optimally in converting MBK into biodiesel. To test the catalytic activity of NZO/FA catalysts, this also focuses on the effect of transesterification time on the yield of biodiesel produced. While NZO/FA catalysts will be characterized through the Brunauer Emmett Teller (BET) method and Fourier Transform Infra-Red (FTIR) spectroscopy to determine the catalyst surface area and functional groups, chemical composition, along with bond types based on atomic vibrations in the molecule.

LITERATURE REVIEW

Heterogeneous Acid Catalyst

Because they can aid in esterification and transesterification reactions concurrently in the production of biodiesel without the saponification step, acid heterogeneous catalysts were chosen as an alternative to acid homogeneous catalysts. Because of this, a higher percentage of biodiesel products are created [17]. The acidic heterogeneous catalyst is additionally readily detachable from the end product and is regenerable, minimizing environmental pollution.

Active Catalyst Support

FA is a coal ash byproduct. FA processing errors may lead to environmental damage. FA has a high amount of porosity and a particular surface area, which make it an effective adsorbent for resolving these issues [18]. In order for FA to function as an adsorbent at its best, activation is required to widen the pore by breaking hydrocarbon bonds. HCl is one of the chemical activators that is regularly employed. The use of HCl as a chemical activator is thought to have the ability to expose surfaces that had previously been covered by chemical components, increasing the amount of active surface area.

Transesterification Reaction

Triglycerides are esterified into a glycerol structure during the transesterification event. Triglycerides' three fatty acid chains will be freed from the glycerol framework in the meantime, and when they interact with short-chain alcohols, they transform into biodiesel. Additionally, the transesterification event that takes place will result in the production of glycerol as a byproduct [19].

Biodiesel

A class of renewable fuels known as long-chain fatty acid acyl esters includes biodiesel. Biodiesel is currently being developed to solve a number of issues with fossil fuels, including their scarcity and potential for environmental pollution. While this is going on, biodiesel itself offers a number of benefits, including the fact that it is environmentally benign, and biodegradable [17].

METHOD

Tools and Materials

The tools used in this study are stative, clamps, sensors, thermometers, three-neck flasks, hotplates, stirrers, weighing bottles, beaker glass, porcelain cups, erlenmeyer, burettes, split funnels, measuring cups, measuring flasks, volumetric pipettes, water hyacinths, pycnometers, Ostwald viscometer, ovens, and analytical balances.

While the materials used include MBK, fly ash (FA), methanol, zinc chloride, sodium hydroxide, aquadest, 96% alcohol, oxalic acid hydrate, potassium hydroxide, sodium carbonate, hydrochloric acid, phosphoric acid, phenolphthalein indicator, and methyl red indicator.

Work Procedure

This research includes the MBK pre-treatment process, FA activation, NZO/FA catalyst synthesis, and MBK transesterification using NZO / FA catalyst. Where the MBK pre-treatment process, NZO/FA catalyst synthesis, and MBK transesterification using NZO/FA catalyst have been carried out by Siswoyo and Asri (2022) without activation of FA. However, the highest biodiesel yield obtained only of 51.01% [12]. This is possible due to the FA as catalyst support was directly use without treatment, which allows the FA pores to be inactive so that ZnO cannot stick properly to the catalyst surface. Therefore, in this study FA was activated using hydrochloric acid concentration of 0.5M by stirring using a *magnetic stirrer* to enlarge the pores of FA before zinc methoxide was impregnated into the buffer.

Meanwhile, NZO/FA catalysts were characterized using Brunauer Emmett Teller (BET) method and Fourier Transform Infra-Red (FTIR) spectroscopy. Then the catalyst activity test was carried out by carrying out the MBK transesterification process using methanol in a laboratory-scale batch reactor with a variable transesterification time of 3; 3.5; 4; 4.5; 5 hours. To increase the *yield of* biodiesel produced, the catalyst dose used is 5% with a transesterification reaction temperature of 80°C and a molar ratio between MBK and methanol 1: 15. The transesterification reaction can be written as follows:

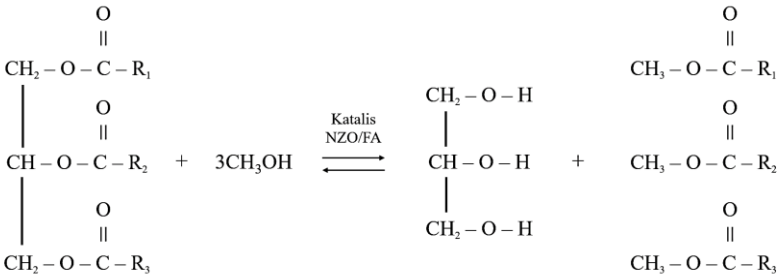

Figure 1. Transesterification reaction.

The FAME content of biodiesel can be calculated from the calculation of biodiesel *yield* in equation 1. Furthermore, biodiesel products are characterized through analysis of free fatty acid content (FFA), hoarding number (SV), iodine number (IV), density, viscosity, and moisture content based on SNI 7182:2015.

$$yield\ (\%) = \frac{W_{BD}}{W_0} \times 100\% \qquad \text{..... (1)}$$

Where W_{BD} is the weight of biodiesel (g), obtained from experiments. While W_0 is the weight of kapok seed oil (g) needed in the experiment.

RESULTS AND DISCUSSION

The fatty acid content in MBK was analyzed using GC-MS using a combination of gas chromatography analysis and mass spectrophotometry. The following is the result of the analysis of fatty acid composition in MBK:

Table 1. Results of MBK's GC-MS analysis

Fatty Acids	Composition (%)	Fatty Acids	Composition (%)
Caprylic Acid	0.10%	14-Pentadecenoic Acid	0.18%
Nanonoic Acid	0.08%	Palmitic Acid	28.51%
Capric Acid	0.08%	Heptadecanoic Acid	1.08%
Lauric Acid	0.65%	Linoleic Acid	59.10%
Myristic Acid	0.37%	Stearic Acid	9.59%
Oleic Acid	0.26%		

Table 1 shows that linoleic acid, palmitic acid, and stearic acid are the main constituent components of fatty acids in MBK. Since linoleic acid is the largest component in MBK, the molecular weight of linoleic acid is used in FFA calculations.

Oil characterization tests before and after pre-treatment are carried out to determine the characteristics of MBK used in the manufacture of biodiesel. The test results are as follows:

Table 2. Results of the MBK characteristic tests

Parameters Test	Analysis Findings	
	Before Pre-Treatment	After Pre-Treatment
Linoleic Acid as FFA (%w/w)	11.27	10.37
Iodine Number (gr I_2/100 gr)	53.52	72.94
Saponification Value	455	372
Density (gr/mL)	0.95	0.94
Viscosity (cSt)	2.11	1.82
Moisture Content (%w/w)	0.04	0.003

The high level of free fatty acids and water content is caused by a hydrolysis reaction in MBK [20]. In the hydrolysis reaction, fatty acids will be converted into free fatty acids (FFA) and glycerol. In addition, the hydrolysis reaction also causes hydrolytic rancidity which can cause a

rancid smell and taste in the oil. Rancidity in oil can also be affected due to an oxidation reaction between oil and a certain amount of oxygen [21]. While the dark color of the oil results from the oxidation process of tocopherol.

The degumming process of MBK is considered important because it can improve the quality of MBK, including reducing FFA levels, density values, viscosity, moisture content and the color of the oil produced [22]. In addition, through this degumming process, it is expected that oil yield can increase because the gum in the oil will be removed through the addition of phosphoric acid.

Catalyst Characterization

NZO/FA catalysts produced through impregnation, precipitation, and calcination processes are characterized by Brunauer Emmett Teller (BET) and Fourier Transform Infra-Red (FTIR) spectroscopy methods as follows:

Brunauer Emmett Teller (BET)

Table 3. 50-Sto NZO/FA catalysts and fly ash BET results

Name of Sample	BET Surface Area Results (m2/g)
Fly Ash	0.075
NZO/FA 50-Sto Catalysts	14.849

From the results of characterization of NZO / FA catalysts using the BET method, it is known that the surface area of fly ash is 0.075 m^2/g, while the surface area of NZO/FA catalysts produced is 14.849 m^2/g. This increase in surface area in NZO/FA catalysts indicates that ZnO has been well dispersed into the pores of the supports port. This increase in surface area is also expected to help increase yield and decrease the concentration of dyes in the biodiesel products produced. When compared with the previous research [12], which used the same material the highest surface area was found only 1.5 m^2/g. This is possible because FA as a support was not subjected to treatment before being synthesized. In contrary on this work the FA was subjected by treatment using HCl.

Fourier Infra-Red Spectroscopy (FTIR)

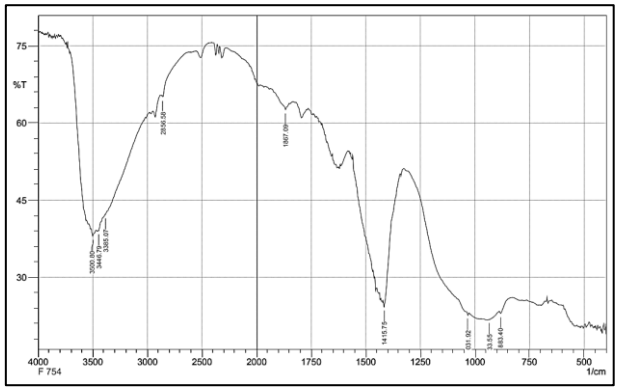

Figure 2. Fourier Infra-Red (FTIR) spectroscopy of NZO/FA catalysts.

Based on the characterization of the catalyst of the FTIR method, it can be seen that there is absorption at wavenumbers 1415.75 cm^{-1} and 933.55 cm^{-1}. The absorption peak in the wave refinery 1415.75 cm^{-1} indicates the presence of metal oxidesThis is because metal oxides have a typical absorption at wavenumbers below 1010 cm^{-1}. While the absorption shown by wavelengths 863.3 cm^{-1} and 933.55 cm^{-1} shows the deformation of Zn=O bonds and at wavelengths 620.93 cm^{-1} shows absorption due to Zn=O bond deformation [24]. On the other hand, absorption that appears at wavelengths 3500.8-3385.07 cm^{-1} is caused by strain groups O-H due to the presence of little water content due to material moisture. Then the absorption at wavelength 1867.09 cm^{-1} shows if there is

a vibration group H-O-H and the presence of a silica functional group at wavelength 3500.8-1415.75 cm^{-1} due to the bond between cyan and siloxane [25].

MBK conversion

NZO/FA catalysts that have been characterized will then be tested for catalyst activity through a transesterification process. Where in this study the parameter used to measure the catalyst, activity is the effect of transesterification time on the yield of biodiesel produced. Theoretically, the longer the contact reaction time between substances is greater, as a result the yield of biodiesel produced is greater [26]. The effect of reaction time on yield of biodiesel was shown on Figure 3. Yield of biodiesel (%) was calculated using equation 1.

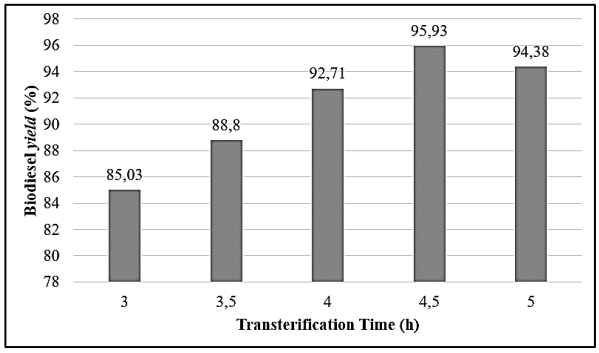

Figure 3. The yield of biodiesel product with transesterification time variation.

From figure 3 it is known that the highest yield is at the transesterification time of 4.5 hours with a percentage of 95.93% because the longer the reaction time used, the more methyl ester levels obtained [27]. But at the 5-hour transesterification time, the yield percentage decreased to 94.38% as a result of the transesterification reaction which is classified as a reversible reaction. When equilibrium is reached, the transesterification reaction must be stopped so that the energy used is more efficient [28]. Meanwhile, the 3-hour transesterification time shows the lowest yield percentage, which is 85.03%. The low yield of biodiesel is caused by the lack of time it takes methanol to convert triglycerides into biodiesel [29]. Furthermore, biodiesel products are carried out quality tests to determine the quality of the biodiesel produced. The characteristics of biodiesel produced include linoleic acid as FFA (%w/w), Iodine number (IV), saponification number, density, viscosity and moisture content are shown at table 4.

Table 4. Transesterification time variable results of quality examination of biodiesel product

Parameters Test	SNI 7182: 2015	Analysis Findings
Linoleic Acid as FFA (%w/w)	0,6, max	1,63
Iodine Number (gr I_2/100 gr)	115, max	56,26
Saponification Value	189-197	196,40
Density (gr/mL)	0,85-0,89	0,88
Viscosity (cSt)	2,3-6,0	4,48
Moisture Content (%w/w)	0,05, max	0,0013

FFA values that have not met the provisions of SNI 7182:2015 are caused by no reduction in FFA levels in the MBK used. Meanwhile, a low iodine number can prevent scale formation in the injection channel holes and pistons [30]. The high number of hoardings indicates that the MBK used has a small molecular weight due to long storage of raw material products. On the other hand, the density value obtained has met the specified SNI. High density values indicate that MBK conversion is not perfect and the process of separating products from residues is not optimal, so that the methanol content along with other impurities is still included in the biodiesel products produced. Meanwhile, the viscosity value will decrease along with the increasing conversion of biodiesel produced due to the minimal FFA content contained in biodiesel products. In addition, a high enough water content

can cause biodiesel products to become foamy and corrosive when reacting with sulfur while giving microbes room to grow into impurities in biodiesel [27].

CONCLUSION

In this study, it can be concluded that the acid solid catalyst NZO/FA was successfully synthesized using impregnation, precipitation, and calcination methods for transesterification of MBK into biodiesel with satisfactory results. This is shown by the high yield value of 95.93% obtained from the best reaction time, which is 4.5 hours with a ratio of oil to methanol 1:15, reaction temperature 80°C and NZO / FA catalyst dose 5%. In addition, the quality of biodiesel produced also almost meets all the requirements in SNI 7182:2015. Furthermore, the selection of low cost and environmentally friendly FA as a support of zinc oxide catalysts provides promising potential to convert MBK into biodiesel.

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