JURNAL KIMIA (*JOURNAL OF CHEMISTRY*) 19 (2), JULI 2025 p-ISSN 1907-9850
DOI: https://doi.org/10.24843/JCHEM.2025.v19.i02.p20 e-ISSN 2599-2740

UTILIZATION OF NA_2O/LAPINDO MUD NANOCATALYST IN THE TRANSESTERIFICATION REACTION OF USED COOKING OIL INTO BIODIESEL

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Article Received on: 10th December 2024 Revised on: 14th July 2025 Accepted on: 21th July 2025

ABSTRAK

Penelitian ini mendemonstrasikan preparasi Lumpur Lapindo yang diimpregnasi dengan NaOH untuk digunakan sebagai katalis heterogen dalam reaksi transesterifikasi minyak jelantah menjadi biodiesel. Katalis dibuat dengan cara impregnasi NaOH pada Lumpur Lapindo, kemudian dikalsinasi pada suhu 550 °C selama 3 jam. Setelah itu, dilanjutkan proses *ball milling* dengan kecepatan 4000 rpm selama 1 jam untuk mengubahnya menjadi nanokatalis Na_2O/LM. Penggunaan nanokatalis ini meningkatkan luas permukaan yang memungkinkan interaksi lebih efektif dengan reaktan, sehingga mempercepat proses katalisis. Karakterisasi nanokatalis dilakukan menggunakan XRF, XRD, FTIR, dan PSA. Mekanisme reaksi transesterifikasi melibatkan minyak jelantah dan metanol dengan rasio 1:9, nanokatalis Na_2O/LM 5 %wt, reaksi ini berlangsung selama 2 jam pada suhu 65°C. Penggunaan nanokatalis $Na_2O_{(0)}$/LM, $Na_2O_{(20)}$/LM, $Na_2O_{(40)}$/LM dan $Na_2O_{(60)}$/LM menghasilkan yield berturut-turut 77,36%; 83,27%; 86,51%; dan 96,68%. Uji kualitas biodiesel mencakup bilangan asam, kadar air, titik nyala, densitas, viskositas, dan GC-MS. Pengujian GC-MS dilakukan pada yield biodiesel terendah $Na_2O_{(0)}$/LM dan tertinggi $Na_2O_{(60)}$/LM, menghasilkan FAME masing-masing sebesar 99,26% dan 99,99%.

Kata kunci: nanokatalis; Na_2O/LM; minyak jelantah; transesterifikasi; biodiesel

ABSTRACT

This research demonstrates the preparation and characterization of NaOH-impregnated Lapindo Mud nanocatalysts for biodiesel production from waste cooking oil through transesterification. The nanocatalysts were synthesized by wet impregnation of NaOH into Lapindo Mud, calcination at 550°C for 3 hours, and ball milling at 4000 rpm for 1 hour to achieve a nanoscale particle size. Enhanced surface area significantly improves reactant-catalyst interaction, accelerating the catalysis process. Characterization using XRF, XRD, FTIR, and PSA revealed systematic changes in chemical composition and structure with increasing NaOH content. XRF analysis showed decreased SiO_2 and increased Fe_2O_3 levels with higher NaOH percentages, while XRD identified four phases: SiO_2, Na_2O, Na_2SiO_3, and Al_2SiO_5, with Na_2SiO_3 formed from NaOH-SiO_2 reactions. PSA analysis indicated particle size increases from 514.3 nm [Na_2O(0)/Lapindo Mud] to 577.7 nm [Na_2O(60)/Lapindo Mud] due to aggregation. Transesterification reactions were conducted using waste cooking oil and methanol at a 1:9 ratio with 5 wt% nanocatalyst for 2 hours at 65°C. Catalytic performance demonstrated a strong correlation between NaOH content and biodiesel yield: NaOH(0)/Lapindo Mud (77.36%), NaOH(20)/Lapindo Mud (83.27%), NaOH(40)/Lapindo Mud (86.51%), and NaOH(60)/Lapindo Mud (96.68%). GC-MS analysis confirmed excellent biodiesel quality, with FAME content of 99.26% and 99.99% for the lowest and highest performing catalysts, respectively. Results demonstrate that NaOH-impregnated Lapindo Mud nanocatalysts offer a cost-effective and environmentally sustainable solution for converting waste cooking oil into high-quality biodiesel.

Keywords: nanocatalysts; Na_2O/Lapindo Mud; waste cooking oil; transesterification; biodiesel

INTRODUCTION

Biodiesel is a promising alternative fuel because it is environmentally friendly, does not harm health, and can be used as a fuel for motor vehicles so that it can reduce CO_2 emissions compared to diesel oil (Dinanti *et al*., 2024). Biodiesel processing from used cooking oil is one of the alternatives to reduce the selling price of biodiesel because of the cheap raw materials, it can also reduce environmental pollution (Okechukwu *et al*., 2022; Xie *et al.,* 2021). If the FFA content exceeds the tolerance, it is necessary to carry out pre-treatment in the form of an esterification reaction before carrying out a transesterification reaction to reduce FFA (Sutanto *et al*., 2021).

The transesterification reaction is one of the methods of making biodiesel from used cooking oil raw materials assisted by homogeneous catalysts and heterogeneous catalysts, both acids and bases. The transesterification reaction is a chain reaction that changes the triglycerides, diglycerides, and monoglyceride compound groups that are reacted with alcohol using the help of a catalyst. The reaction will produce methyl ester and glycerol (Khoiruummah *et al*., 2020). The molar ratio of oil to methanol in the transesterification process plays an important role in determining the yield of biodiesel. This ratio represents the proportion of oil molecules to methanol molecules involved in the reaction. According to the principle of stoichiometry, one mole of triglycerides (the main component of oil) reacts with three moles of methanol to produce biodiesel (methyl ester) and glycerol (Kurniati *et al*., 2020). Mulana (2011) conducted research by varying the mole ratio to determine the optimal ratio of moles and methanol to oil of 8:1; 9:1; 10:1; and 11:1 for 90 minutes with a temperature of 60 ^oC and a catalyst used by 2% weight, obtaining a biodiesel yield of 81.7% which is the optimum result at a molar ratio of methanol and oil of 9:1. In addition to the molar ratio, the reaction temperature during transesterification significantly affects the production of biodiesel. Susanti *et al*., (2023), conducted a study by varying the temperature to determine the optimum temperature in the transesterification reaction using a 1% CaO-NaOH catalyst weight. The temperature variation used is 50 °C, 60 °C, and 70 °C for 90 minutes at a speed of 650 rpm resulting in 75%, 85%, and 80% biodiesel respectively, with a ratio of 6:1 oil to methanol used.

The transesterification reaction is slow, so a catalyst is needed to accelerate the rate of reaction. In this study, a heterogeneous catalyst was used to help accelerate the reaction. Heterogeneous catalysts are considered to be more active as compared to homogeneous catalysts. The larger surface area and even distribution of active sites on heterogeneous catalysts allow more reactant molecules to come into contact with active sites at the same time (Mahmudah, 2023). Lapindo mud is used as a catalyst in transesterification reactions because it is found to be composed of a wide variety of oxide metals such as SiO_2, Fe_2O_3, Al_2O_3, and K_2O (Budiarti *et al*., 2017). In research conducted by Talib *et al*., (2016) using pure Lapindo mud as a catalyst, the biodiesel yield was obtained at 51.65% under the reaction condition of a molar ratio of oil and methanol of 1:9, catalyst weight of 3% with a temperature of 60°C for 1 hour. However, the results obtained have not been maximized, so it is necessary to add heterogeneous base active sites such as Na_2O to increase their catalytic activity.

Efforts to increase the catalytic activity of the catalyst can be carried out by inserting active metal components into the supporting material using the wet impregnation method. Ibrahim *et al*., (2020) researched wet impregnation of NaOH in CNTs *(Carbon Nanotubes*) to obtain Na_2O/CNTs using $Na_2O_{(20)}$/CNTs and $Na_2O_{(40)}$/CNTs catalysts in the transesterification reaction of used cooking oil, resulting in the conversion of biodiesel respectively of 97.3% and 97.6% at optimal conditions with a calcination temperature of 500 for 2 hours, a molar ratio of methanol to oil of 20:1, a reaction temperature of 90 ^oC, reaction time 4 hours, and catalyst count 5% oil weight. Na_2O/CNTs catalysts are obtained through a wet impregnation process, where sodium hydroxide (NaOH) is the source of Na_2O. In addition to incorporating active metal components into supporting materials, the utilization of nanoparticles also increases catalytic activity due to their large surface area. The vast surface area of the nanoparticles effectively maximizes exposure to the active catalytic site, facilitating optimal contact between the reactant and the catalyst. This significantly increases the reaction rate and increases catalytic activity in less time to achieve full conversion (Narayan *et al*., 2019).

MATERIALS AND METHODS

Materials

The materials that will be used in this study are palm waste cooking oil, Lapindo Mud, methanol (Merck), NaOH 0.1N (Merck), ethanol (Merck), distilled water, and PP indicators.

Tools

The equipment to be used in this study is glass bottles, furnaces, 500 mL triple neck flasks, analytical balances (Precision Balance

TP-4101), porcelain cups, 80 mesh sieve, mortar and pestle, hotplate magnetic strirrer, stopwatch, spatula, blender, desiccator, filter paper, separator funnel, condenser, thermometer, water hose, clamps, statics, XRF instruments (PANalytical Minipal 4), FT-IR (Shimadzu IR Prestige 21), XRD (PANalytical X'Pert PRO), GC-MS (Shimadzu QP2010 Plus) and various glassware used in laboratories.

Methods

Preparation of Lapindo Mud as a Catalyst Support Material

Lapindo mud is washed with aqueducts with the aim of removing mixed impurity elements. Then it is dried in direct sunlight until dry, then baked at a temperature of 100 ^oC until dry. Lapindo mud that has been in the oven is mashed using a blender and sifted using an 80 mesh sieve to obtain a homogeneous particle size (Ciptawati *et al*., 2022). The sifted Lapindo mud is placed on a plate and in the oven at a temperature of 100 ^oC for 2 hours, then the Lapindo mud is stored in the desiccant (Trisunaryanti *et al*., 2022).

Catalyst Impregnation

A total of 1.25 grams of NaOH is put into a beaker and dissolved with 30 mL of distilled water. Lapindo mud was weighed as much as 3.75 grams and put into the NaOH solution (Oko *et al*., 2021). The mixture is stirred using a stirrer at a speed of 250 rpm at a temperature of 30 ^oC for 14 hours, then dried in an oven at a temperature of 120 ^oC until dry. The impregnation catalyst is calcined at 550 ^oC for 3 hours (Ibrahim *et al*., 2020). After the completion of calcination, the catalyst is cooled to room temperature and stored in a vial bottle tightly closed. The procedure was performed equally on a weight of NaOH 40%wt and NaOH 60%wt.

Nanocatalyst Synthesis

The synthesis of Na_2O/Lapindo mud catalyst was carried out using *ball milling* for 1 hour at a speed of 4000 rpm to obtain a nano-sized catalyst (Sirait & Bukit, 2014) so that Na_2O/Lapindo mud nanocatalysts were obtained.

Characterization of Nanocatalysts

The Na_2O/Lapindo mud nanocatalyst was analyzed using X-ray diffraction (XRD) of the Panalytical X'Pert Pro. XRD was used to analyze (solids type and crystal phase) on the sample. The Na_2O/Lapindo mud nanocatalyst was analyzed FT-IR or Fourier Transform Infra-Red Shimadzu Prestige 21 model. FT-IR analysis was used to analyze the functional groups contained in the Na_2O/Lapindo mud nanocatalyst. Na2O/Lapindo mud catalyst was analyzed by X-ray fluorescence (XRF) PANalytical Minipal 4. XRF analysis is used to determine the elements or materials in the sample. Na_2O/Lapindo mud nanocatalysts with various variations were analyzed using the SALD-7500 nano type PSA Shimadzu instrument which can measure in the range of 7-800 nm to measure the particle size and particle size distribution of Na_2O/Lapindo mud nanocatalysts.

Biodiesel Synthesis

The synthesis of biodiesel from used cooking oil using methanol and Na_2O/Lapindo mud nanocatalyst was carried out through the transesterification method. This process begins with the activation of the catalyst at 120°C for 2 hours, then cooled in a desiccator. A total of 5%wt of activated catalysts were mixed with 42.78 grams of methanol in a triple neck flask and stirred using a magnetic stirrer at 60°C for about 20 minutes. After that, 100 grams of used cooking oil is added to the mixture, which is then heated to a temperature between 60-65°C while stirring at 500 rpm for 2 hours. Once the reaction is complete, the product is cooled and transferred to a separation funnel to separate the biodiesel formed in the upper layer from the glycerol underneath. To neutralize the pH, the mixture is washed with distilled water to neutral pH, aiming to remove any residual catalysts, glycerol, and methanol. The resulting biodiesel is then baked at 100°C to remove the remaining water. This process produces biodiesel which can be calculated based on the *yield* from the transesterification reaction. Then biodiesel is tested for quality, namely testing moisture content, acid number, viscosity, density, flash point, and GC-MS.

RESULTS AND DISCUSSION

This research is focused on the activity of Na_2O/Lapindo mud nanocatalyst in the production of biodiesel from used cooking oil which is household waste, using experimental methods in the Physics and Chemistry laboratory, State University of Surabaya, because the data used was taken directly from the research object.

Waste Cooking Oil Quality Test

The quality test of used cooking oil involves two parameters, namely FFA content and moisture content, to be analyzed. This testing process follows the procedures that have been established in ASTM D5555. The quality of used cooking oil can determine the right processing method and have an impact on the quality of the biodiesel produced. The data in Table 1. is the result of testing the quality of used cooking oil which is the main ingredient in the manufacture of biodiesel in this study.

Table 1. Results of Waste Cooking Oil Quality Test

Parameters	Results	Requirements for Making Biodiesel
FFA	0,2%	0,5% (Okechukwu *et al*., 2022)
Water content	0,04%	0,1% (Xie *et al*., 2021)

The FFA content test showed a result of 0.2% for used cooking oil, which indicates that the FFA content of used cooking oil is very low, so it is ready to be used for biodiesel transesterification reactions without esterification. The moisture content test showed a result of 0.04%, indicating that the used cooking oil has a very low water content, so it is considered good quality and ready to be used in biodiesel production.

Nanocatalyst Synthesis

Nanocatalyst synthesis was carried out by weighing 1.25 grams of NaOH into a beaker and dissolving it with 30 mL of distilled water using a magnetic stirrer hotplate. Then, 3.75 grams of Lapindo mud was weighed and put into the NaOH solution. The mixture is stirred using a stirrer at a speed of 250 rpm at a temperature of 30^{o}C for 14 hours, then dried in an oven at a temperature of 120^{o}C until dry and calcined at a temperature of 550^{o}C for 3 hours. This process is also carried out at NaOH concentrations of 40% and 60%. After calcination, the catalyst will form into Na_2O/Lapindo mud. Lapindo mud will be formed with various oxide metals. NaOH will form Na_2O, as follows reaction:

$$2NaOH_{(aq)} \rightarrow Na_2O_{(s)} + H_2O_{(l)} \quad (1)$$

Na_2O/Lapindo mud catalysts are ball milling to form nanocatalysts. The use of nanocatalysts because they have a high surface area, so they can improve catalytic performance. This allows more active sites to be available for the reaction, thus accelerating the rate of the transesterification reaction (Hawazin *et al*., 2024).

Characterization of Catalysts

XRF (X-ray Fluorescence)

Testing with XRF instruments is an analytical method to determine the composition of elements and metal oxides in a sample. XRF instrument testing was performed on $Na_2O_{(0)}$/Lapindo Mud, $Na_2O_{(20)}$/Lapindo Mud, $Na_2O_{(40)}$/Lapindo Mud, and $Na_2O_{(60)}$/Lapindo Mud nanocatalysts. Table 2 shows the results of XRF (X-ray fluorescence) testing on Na_2O/Lapindo mud nanocatalysts.

$Na_2O_{(0)}$/Lapindo Mud nanocatalyst, shows the dominance of compound SiO_2 (47%), Fe_2O_3 (22,3%), and Al_2O_3 (15%) components. This indicates that most of the composition of Lapindo Mud is silicate. On nanocatalysts of $Na_2O_{(20)}$/Lapindo Mud, SiO_2 decreased to 43.5%, Al_2O_3 to 12%, while Fe_2O_3 increased to 26.3%.

Furthermore, in the $Na_2O_{(40)}$/Lapindo mud nanocatalyst, SiO_2 decreased further to 40.6%, with Fe_2O_3 relatively stable at 26.1% and Al_2O_3 remaining at 12%. At the nanocatalyst $Na_2O_{(60)}$/Lapindo mud, the SiO_2 showed stability at 40.5%, Fe_2O_3 slightly increased to 26.9%, dan Al_2O_3 remained consistent at 12%. The addition of NaOH to the Lapindo sludge aims to modify the properties of the catalyst. NaOH can interact with sludge components, forming new active sites or modifying the formation of new compounds.

Table 2 shows that Na_2O was not detected at all, leading to several possible explanations. First, Na_2O is a compound that contains the element sodium, which is a light element with atomic number 11. One possibility is that XRF instruments have a lower sensitivity to detect light elements, such as sodium if the analysis conditions are not optimized (Putra, 2018). Second, Na_2O can react with other components in the sample and form new compounds that may not be detected as Na_2O in XRF analysis.

For example, sodium can be bound in the form of SiO_2 compounds and form Na_2SiO_3 (Meilani *et al*., 2023), As shown in the reaction:

$2NaOH_{(aq)} + SiO_{2(s)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(aq)}$.(2)

Third, the Na_2O levels in the sample may be very low, so they are below the detection limits of XRF instruments. This is often the case in mineral or rock samples where sodium concentrations are relatively small compared to other oxides (Putri *et al*., 2023).

FTIR (*Fourier Transform Infra-Red*)

The analyzed FTIR spectrum showed significant changes in the chemical structure of the Lapindo Mud due to the addition of NaOH. Characteristic peaks in the FTIR wave numbers for the Na_2O/Lapindo mud nanocatalysts were detected at 3350, 3441, 3431, and 3413 cm^{-1}, respectively, representing the vibrations of the -OH group. This is in line with research by Ramadani et al., (2023), which indicates the number of waves in the 3400-4000 cm^{-1} for the vibration of the -OH group.

Si-OH bending vibrations on the $Na2O_{(0)}$/Lapindo mud nanocatalyst were detected at the absorption peak of 1643 cm^{-1}, which was consistent with the results of Trisunaryanti *et al*. (2020) which shows the bending vibration of Si-OH at a wave number of 1636 cm^{-1}. On nanocatalysts $Na_2O_{(20)}$/Lapindo mud, $Na_2O_{(40)}$/Lapindo mud, and $Na_2O_{(60)}$/Lapindo mud peaks detected at 1454, 1437, dan 1442 cm^{-1}, which shows the O-C-O stretching vibrations due to the formation of Na_2CO_3 from the reaction between Na_2O and CO_2 (Ryu & Lee, 2018).

Table 2. Results of Na_2O /Lapindo Mud Nanocatalyst XRF test

Metal oxides	Nanocatalyst			
	$Na_2O_{(0)}$/Lapindo Mud	**$Na_2O_{(20)}$/Lapindo Mud**	**$Na_2O_{(40)}$/Lapindo Mud**	**$Na_2O_{(60)}$/Lapindo Mud**
SiO_2	47%	43,5%	40,6%	40,5%
Fe_2O_3	22,3%	26,3%	26,1%	26,9%
Al_2O_3	15%	12%	12%	12%
CaO	6,53%	7,27%	7,72%	7,78%
MoO_3	2,0%	3,7%	6,32%	5,7%

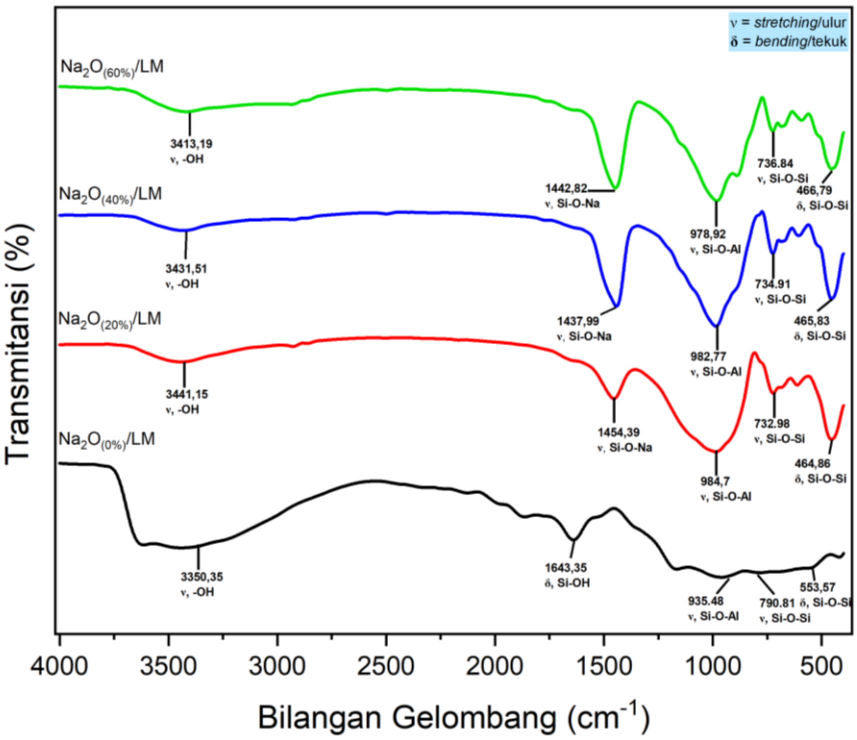


Figure 1. Nanocatalyst of Na_2O/Lapindo Mud FTIR Test Results

The asymmetric stretching vibration of Si-O-Si in the nanocatalyst $Na_2O_{(0)}$/Lapindo mud was detected at the peak of absorption of 1168 cm^{-1}, then shifted to Si-O-Al tendril vibration at the concentrations of $Na_2O_{(20)}$/Lapindo mud, $Na_2O_{(40)}$/Lapindo mud and $Na_2O_{(60)}$/Lapindo mud with absorption peaks of 935, 982, and 978 cm^{-1} respectively. Research by Tkachenko & Niedzielski, (2022) absorb at 1121, 1077, 1026, 992, and 957 cm^{-1}, which corresponds to the change in the position of the Si-O-Si stretching vibration at 914 cm^{-1} Si-O-Al stretching vibration.

The absorption peaks for the symmetrical stretching vibrations of Si-O-Si were detected at the respective nanocatalyst Na_2O/Lapindo Mud 790, 732, 734, and 736 cm^{-1}. Meanwhile, the peak absorption for Si-O-Si bending vibrations was detected at wave numbers 553, 464, 465, and 466 cm^{-1}. These findings are in line with research Priya *et al*., (2019), which recorded absorption bands at 799.4 and 471.2 cm^{-1} for symmetrical stretching vibration and Si-O-Si bending.

XRD (*X-Ray Diffraction*)

Lapindo Mud nanocatalyst and Na_2O/Lapindo Mud were analyzed using XRD at an angle of 2θ between 10-90° to identify phases, crystallinity levels, and the formation of new compounds. The optimal level of crystallinity is indicated by diffractograms that display a clear pattern of peaks and a sharp intensity. The results of the diffractogram analysis of each catalyst can be seen in Figure 2.

Based on the results of the XRD analysis presented in Figure 2, the peak of 2θ of the nanocatalyst variation $Na_2O_{(0)}$/Lapindo Mud, $Na_2O_{(20)}$/Lapindo Mud, $Na_2O_{(40)}$/Lapindo Mud, and $Na_2O_{(60)}$/Lapindo Mud compared to the ICDD database, it shows that the Lapindo Mud sample contains four main phases: SiO_2, Na_2O, Na_2SiO_3, dan Al_2SiO_5. The main peak of diffraction SiO_2 was detected on 2θ = 26,64° (ICDD 01-085-0797), while the main peak for Na_2O appears on 2θ = 27,76° (ICDD 00-023-0528). Na_2SiO_3, which is a new compound resulting from the reaction between NaOH and SiO_2, shows the main peak on 2θ = 29,47° (ICDD 01-072-0079). In addition, the peak of diffraction Al_2SiO_5 detected on 2θ = 24,36° according to ICDD reference (00-037-1461), which may be formed from the reaction between SiO_2 and Al_2O_3, as shown in the reaction equation:

$$Al_2O_{3(s)} + SiO_{2(s)} \longrightarrow Al_2SiO_{5(s)} \quad (3)$$

Diffraction peaks that appear in the results of the XRD nanocatalyst test Na_2O/Lapindo Mud are shown in Table 3.

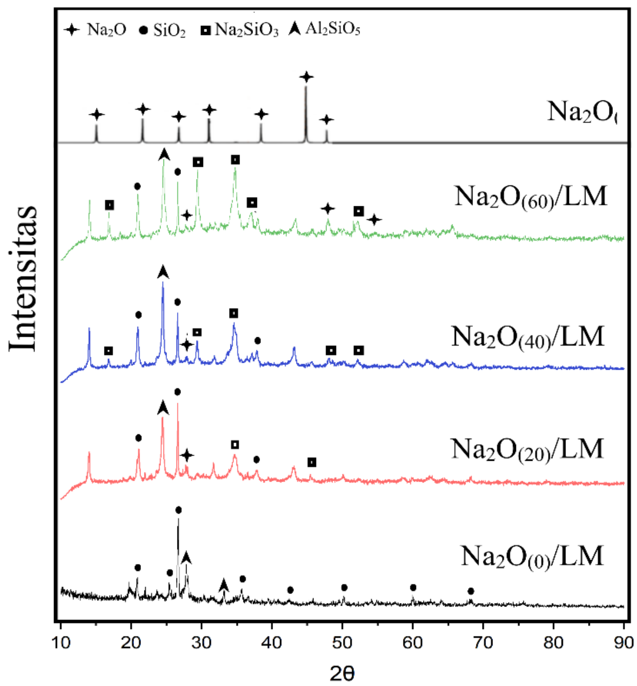


Figure 2. Results of XRD Characterization of Nanocatalysts Na_2O/Lapindo Mud and Na_2O

Table 3. Comparison of XRD Peaks with ICDD Database

Nanocatalyst				ICDD
$Na_2O_{(0)}$/Lapindo Mud	$Na_2O_{(20)}$/Lapindo Mud	$Na_2O_{(40)}$/Lapindo Mud	$Na_2O_{(60)}$/Lapindo Mud	
20,89; 25,47; 26.68; 36,59; 50,13; 59,97; 68,16	21,12; 26,61; 36,52; 50,09	20,98; 26,60; 37,20	20,98; 26,62	(01-085-0797) SiO_2
-	27,77	27,78	27,81; 49,79; 54,49	(00-023-0528) Na_2O
-	34,82; 45,52	16,82; 29,35; 34,94; 48,06; 52,12	16,84; 29,41; 34,80; 37,09; 47,85; 52,22	(01-072-0079) Na_2SiO_3
28,83; 33,19	24,44	24,51	24,59	(00-037-1461) Al_2SiO_5

Based on Figure 3, the crystallinity of the nanocatalysts $Na_2O_{(0)}$/Lapindo Mud to $Na_2O_{(60)}$/Lapindo Mud showed quite good results. Nanocatalyst $Na_2O_{(0)}$/Lapindo Mud has phase crystallinity amorph the highest is around 45.89%, However, in nanocatalysts $Na_2O_{(20)}$/Lapindo Mud, there was a decrease in the crystallinity of the amorphous phase to 36.93%. This decrease indicates that the addition of NaOH reduces the amorphous phase. Meanwhile, in nanocatalysts $Na_2O_{(40)}$/Lapindo Mud dan $Na_2O_{(60)}$/Lapindo Mud, the crystallinity increased to 38.78% and 39.99%, which may be due to an increase in NaOH concentrations that produce new compounds or alter existing structures. Figure 3 shows the gradual transition from amorphous to crystalline structure as the NaOH content increases, indicating that the addition of NaOH promotes the formation and growth of crystalline phases in the nanocatalyst material.

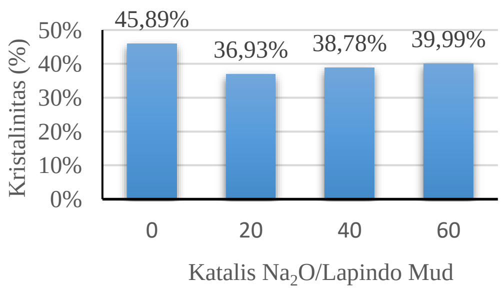


Figure 3. Crystallinity of Nanokatalis Na_2O/Lapindo Mud

PSA (Particle Size Analyzer)

PSA is used to measure the particle size distribution on the catalyst. The nanocatalyst tested using the PSA instrument is the nanocatalyst $Na_2O_{(60)}$/Lapindo Mud dan $Na_2O_{(0)}$/Lapindo Mud which is the use of nanocatalysts for transesterification reactions with the highest and lowest yields. The results of the catalyst characterization are shown in Figure 4.

The characterization results are shown in Figure 4. (a) indicates that the nanocatalyst $Na_2O_{(0)}$/Lapindo Mud has an average diameter of 514.3 nm. Further analysis revealed a peak distribution at 307.8 nm, with the smallest size reaching 105.7 nm and the largest size being 1106 nm. Meanwhile, Figure 4. (b) indicates that the nanocatalyst $Na_2O_{(0)}$/Lapindo Mud has an average diameter of 577.7 nm, with two distribution peaks detected at 243.9 nm and 339.2 nm, as well as the smallest size of 68.06 nm and the largest size of 1281 nm, a high PSA yield that may be due to aggregation and particle deposition (El-sherif *et al.*, 2023).

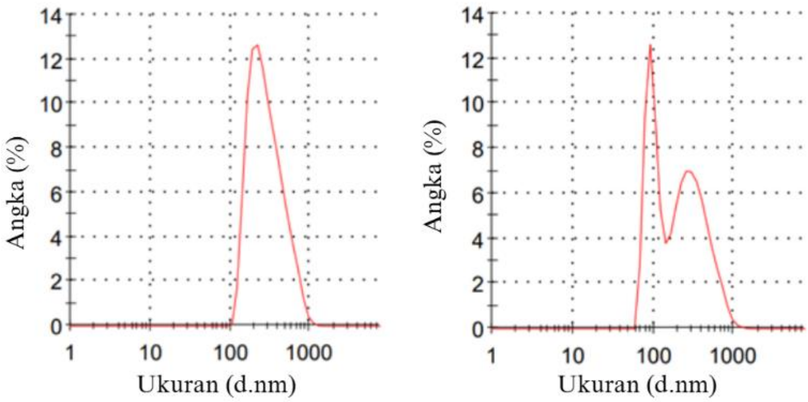
Synthesis of Biodiesel

The synthesis of biodiesel from used cooking oil is carried out through a transesterification reaction by mixing triglycerides, methanol, and heterogeneous catalysts in a triple neck flask at 65°C for 2 hours. The ratio of catalyst to methanol used is 1:9, with nanocatalyst variation $Na_2O_{(0)}$/Lapindo Mud, $Na_2O_{(20)}$/Lapindo Mud, $Na_2O_{(40)}$/Lapindo Mud, dan $Na_2O_{(60)}$/Lapindo

Mud which produces biodiesel yields of 77.36%, 83.27%, 86.51%, and 96.68%, respectively. The addition of methanol in the transesterification reaction is to function as a reagent that reacts with triglycerides to form methyl esters (biodiesel) and glycerol and is chosen because of its high reactivity. This procedure begins by mixing methanol and catalyst first to increase the homogeneity of the mixture before adding used cooking oil. The reaction results in three separate layers: the top layer of biodiesel, the middle layer of glycerol, and the bottom layer of the catalyst, which can be separated due to their insoluble properties. After the separation of biodiesel and glycerol, the next step is to wash the biodiesel to remove the remaining impurities. Washing is carried out using distilled water, in which the solution is shaken and left until two layers are formed. The top layer is made up of biodiesel, while the bottom layer contains impurities. This washing process is carried out repeatedly until the pH of biodiesel is close to a neutral value.

Biodiesel Quality Test

The Biodiesel Quality Test is adapted to ASTM standard testing. Table 4 shows the results of the biodiesel quality test.



(a) **(b)**

Figure 4. PSA results on Nanocatalysts (a) $Na_2O_{(0)}$/Lapindo Mud and (b) $Na_2O_{(0)}$/Lapindo Mud

Table 4. Biodiesel Quality Test Results

Parameters	**Nanocatalysis**				**Standard ASTM D6751-09**
	$Na_2O_{(0)}$/Lapindo Mud	**$Na_2O_{(20)}$/Lapindo Mud**	**$Na_2O_{(40)}$/Lapindo Mud**	**$Na_2O_{(60)}$/Lapindo Mud**	
Yield (%)	77,36	83,27	86,51	96,68	-
Acid number (mg-KOH/g)	0,50	1,17	0,55	0,33	0,50, max
Water content (%)	0,096	0,072	0,039	0,042	0,05%, min
Density (kg/m^3)	1031,6	936,9	911,1	891,1	860-900
Viscosity (mm^2/s)	3,69	3,25	2,30	2,50	1,9-6,0
Flash point (°C)	-	-	-	171	93°C
FAME GC-MS (%)	99,26%	-	-	99,99	96,5 %, min

Biodiesel yield is the percentage of biodiesel produced compared to the amount of oil or fat used in the production process, reflecting the efficiency of transesterification. Yields increase along with the increase in NaOH, with the lowest score of 77.36% on results of using nanocatalysts $Na_2O_{(0)}$/Lapindo Mud and the highest 96,68% on $Na_2O_{(60)}$/Lapindo Mud. This shows that the addition of NaOH significantly improves the efficiency of biodiesel production. High yields indicate an efficient process, while low yields indicate the presence of unconverted feedstocks, which can be affected by catalyst quality, temperature, or reaction time.

Uses of nanocatalysts $Na_2O_{(20)}$/Lapindo Mud produced the highest acid number of 1.17 mg-KOH/g, while $Na_2O_{(60)}$/Lapindo Mud has the lowest acid number of 0.33 mg-KOH/g. Based on the ASTM D6751 standard, the maximum acid number for biodiesel is 0.50 mg KOH/g. Acid numbers that exceed this limit can potentially cause problems at high temperatures because FFA can react with metals, accelerating the deterioration of diesel engine components and decreasing the oxidation stability of biodiesel (Deli & Veronika, 2023).

The water content in biodiesel comes from the purification process by washing using distilled water. Based on the ASTM D6751 standard, the maximum moisture content for biodiesel is 0.05%. Nanocatalyst of $Na_2O_{(0)}$/Lapindo Mud produces the highest moisture content of 0.096%, while $Na_2O_{(20)}$/Lapindo Mud, $Na_2O_{(40)}$/Lapindo Mud, dan $Na_2O_{(60)}$/Lapindo Mud each produces a moisture content of 0.072%; 0,039%; and 0.042%. High water content can slow down combustion reduce combustion efficiency and cause corrosion in the engine.

Biodiesel density decreases along with the increase in NaOH. $Na_2O_{(0)}$/Lapindo Mud produces the highest density of 1031.6 kg/m³, while $Na_2O_{(60)}$/Lapindo Mud results in the lowest density of 891.1 kg/m³. Based on the ASTM D6751-09 standard, the density specification of biodiesel is between 850-890 kg/m³. Hanya $Na_2O_{(60)}$/Lapindo Mud which is close to the maximum value of ASTM with a density of 891.1 kg/m³. The high density can be caused by oil with high-fat content and water contamination from washing.

The viscosity parameter showed the highest value of 3.6924 mm²/s at nanocatalyst use $Na_2O_{(0)}$/Lapindo Mud and the lowest 2,3087 mm²/s at $Na_2O_{(40)}$/Lapindo Mud. All biodiesel samples meet ASTM D6751 viscosity specifications ranging from 1.9-6.0 mm²/s.

The flash point is the minimum temperature at which a substance can burn when exposed to a fire source. The flash point value for biodiesel from the nanocatalyst $Na2O_{(60)}$/Lapindo Mud is 171°C, well above the ASTM D6751 minimum standard of 93°C. This indicates that the biodiesel is non-flammable and reduces the risk of fire and explosion (Kurniasih *et al*., 2017).

GC-MS is an analytical method used to identify and measure the quantity of various compounds in a sample, such as the composition in biodiesel, including FAME (*Fatty Acid Methyl Esters*) which is the result of a transesterification reaction between triglycerides and methanol. FAME was obtained from GC-MS which was tested at the lowest yield results, namely $Na_2O_{(0)}$/Lapindo Mud nanocatalyst, and the highest at $Na_2O_{(60)}$/Lapindo Mud.

Based on Table 5, the results of %FAME biodiesel in $Na_2O_{(0)}$/Lapindo Mud nanocatalyst are 99.26% and $Na_2O_{(60)}$/Lapindo Mud nanocatalyst is 99.99%. The addition of NaOH to the Na_2O/Lapindo Mud catalyst has a considerable effect on yield and FAME results. The percentage of biodiesel produced by $Na_2O_{(60)}$/Lapindo Mud nanocatalyst is 96.67%, this yield is greater than $Na_2O_{(0)}$/Lapindo Mud, which is 76.78%.

The results of the analysis of biodiesel composition using GC-MS showed that the biodiesel produced contained methyl esters that corresponded to the fatty acids in used cooking oil. Figure 5. displaying biodiesel chromatograms of $Na_2O_{(60)}$/Lapindo Mud and $Na_2O_{(0)}$/Lapindo Mud nanocatalysts, where there are two highest peaks, namely in methyl palmitate (*Hexadecanoic acid, methyl ester*) and methyl oleate (*9-Octadecenoic acid, methyl ester*, (E)). In the $Na_2O_{(60)}$/Lapindo Mud nanocatalyst biodiesel, palmitic acid was detected at 34.80% and oleic acid at 45.71%. Meanwhile, in the nanocatalyst, $Na_2O_{(0)}$/Lapindo Mud, palmitic acid reached 37.01% and oleic acid 50.87%. This GC-MS analysis confirmed the presence of methyl ester in biodiesel, proving that used cooking oil can be converted into biodiesel in the form of methyl ester.

Table 5. FAME Test Results on GC-MS Instrument

Compounds	**$Na_2O_{(60)}$/Lapindo Mud**	**$Na_2O_{(0)}$/Lapindo Mud**
Dodecanoic acid, methyl ester	0,10	-
Undecanoic acid, methyl ester	-	0,23
Tridecanoic acid, 12-methyl-, methyl ester	0,84	-
9-Hexadecenoic acid, methyl ester, (Z)-	0,07	-
Hexadecanoic acid, methyl ester	34,80	37,01
Tridecanoic acid, methyl ester	0,03	0,53
9,12-Octadecadienoic acid (Z,Z)-, methyl ester	11,43	7,44
9-Octadecenoic acid, methyl ester, (E)-	45,71	50,87
Heptadecanoic acid, 16-methyl-, methyl ester	6,72	3,18
9-Octadecenoic acid (Z)-, methyl ester	0,04	-
Eicosanoic acid, methyl ester	0,25	-
Total area % FAME	99,99%	99,26%

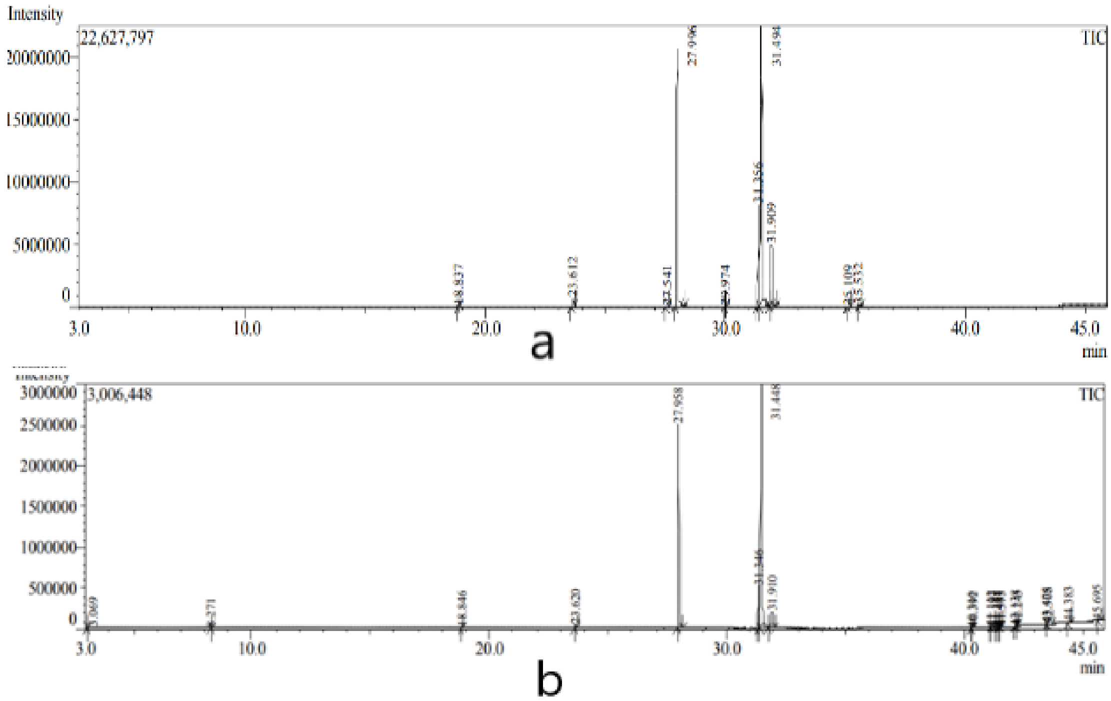


Figure 5. Chromatogram GC-MS Biodiesel a. Katalis $Na_2O_{(60)}$/Lapindo Mud and $Na_2O_{(0)}$/Lapindo Mud

CONCLUSION

The results of XRF characterization analysis showed that the addition of NaOH to $Na_2O_{(0)}$/Lapindo Mud caused significant changes in the chemical composition of the nanocatalyst, where the SiO_2 decreased and the Fe_2O_3 level increased along with the increase in NaOH percentage. FTIR analysis revealed changes in the chemical structure of the Lapindo Mud, including changes in the peak of stretching vibrations -OH, Si-OH, O-C-O, and

Si-O-Si, as well as the formation of new compounds. XRD characterization identifies four main phases in the nanocatalyst: SiO_2, Na_2O, Na_2SiO_3, and Al_2SiO_5, with Na_2SiO_3 as a compound resulting from the reaction between NaOH and SiO_2. PSA characterization showed that the average diameter of the $Na_2O_{(0)}$/Lapindo Mud nanocatalyst was 514.3 nm, while for $Na_2O_{(60)}$/Lapindo Mud it increased to 577.7 nm due to particle aggregation. Biodiesel catalyst activity is measured based on yield and percentage of FAME produced. *The highest yield* was obtained from the nanocatalyst $Na_2O_{(60)}$/Lapindo Mud at 96.68%, followed by $Na_2O_{(40)}$/Lapindo Mud (86.51%), $Na_2O_{(20)}$/Lapindo Mud (83.27%), and the lowest at $Na_2O_{(0)}$/Lapindo Mud (77.36%). GC-MS testing showed that the nanocatalyst $Na_2O_{(60)}$/Lapindo Mud produced FAME of 99.99%, while $Na_2O_{(0)}$/Lapindo Mud produced FAME of 99.26%.

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