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Characterization of Lapindo Mud-Based Nanocatalyst and Quality Testing of Biodiesel from Used Cooking Oil via Transesterification Reaction

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Abstract

This study explores the transesterification of used cooking oil using a Lapindo mud-based nanocatalyst and evaluates the resulting biodiesel quality. The reaction was carried out via the reflux method for 2 hours at 65°C, with a methanol-to-oil molar ratio of 9:1 and a catalyst-to-oil weight ratio of 3:100. The high SiO_2 metal oxide content in Lapindo mud functions as a heterogeneous catalyst. Nanocatalyst synthesis involved calcination followed by ball milling. Characterization confirmed the presence of SiO_2: FTIR analysis identified Si-O-Si functional groups at 1168 cm^{-1}, XRD revealed SiO_2 crystals at 2θ values of 20.18°, 26.68°, 42.40°, and others, while XRF showed 47.0% SiO_2 content. PSA results indicated a particle size distribution of 514.3 ± 361.8 nm. Biodiesel analysis using GC-MS showed a methyl ester content of 99.26%. Other quality parameters included density (929.4 kg/m³), viscosity (4.52 mm²/s), water content (0.097%), and acid number (0.50 mg-KOH/g). These findings suggest that the Lapindo mud-based nanocatalyst is highly effective in promoting transesterification and presents a promising, eco-friendly alternative for biodiesel production from used cooking oil.

Keywords: Biodiesel, Nanocatalyst, Lapindo Mud, Used Cooking Oil

INTRODUCTION

Biodiesel is an alternative, renewable, and environmentally friendly fuel produced through the transesterification process of triglycerides in vegetable oils or used cooking oils with alcohols such as methanol (Kathumbi et al., 2023). The transesterification reaction proceeds at a relatively slow rate (Tahya et al., 2019). Therefore, catalysts are required to accelerate the reaction and enhance the biodiesel yield (Santoso et al., 2024). Heterogeneous catalysts are an attractive option because they are easy to separate from the final product and can be reused, making them more efficient and economical (Ahranjani et al., 2024).

Lapindo mud, produced from a hot mud eruption in Sidoarjo, Indonesia, contains numerous minerals such as silica (SiO_2), alumina (Al_2O_3), and iron oxide (Fe_2O_3) that can be utilized as raw materials for catalysts (Rokhim et al., 2022). By using nanotechnology, this mud can be processed into nanocatalysts with very small particle sizes and large surface areas, enhancing their effectiveness in accelerating the transesterification reaction (Wahyuningsih et al., 2018).

Nanotechnology plays a crucial role in the production of these nanocatalysts. Nanoparticles, defined as particles with dimensions between 1 and 1000 nanometers (nm) (Samudra et al., 2021), possess unique properties that differ from bulk materials. The synthesis process involves steps such as calcination to enhance the crystallinity of the material and milling using a ball mill to reduce particle size to the nanoscale. The resulting nanoparticles have a uniform particle size distribution and increased active surface area, all contributing to enhanced catalytic activity (Rahim et al., 2023).

Characterization of Lapindo mud-based nanocatalysts is necessary to ensure their effectiveness in the transesterification reaction. Techniques such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), X-ray fluorescence (XRF), and particle size analysis (PSA) are used to identify the composition, crystal structure, and particle size distribution of the nanocatalysts. PSA testing helps determine particle size distribution, which is a crucial factor in assessing the catalyst's effectiveness in chemical reaction processes. Nano-sized particles are expected to provide a larger surface area, improving contact between the catalyst and reactants and accelerating the reaction rate.

In addition to evaluating catalyst effectiveness, it is also important to test the quality of the produced biodiesel. Parameters such as density, viscosity, water content, and acid number need to be measured to ensure the produced biodiesel meets international quality standards.

This research aims to study the use of Lapindo mud-based nanocatalysts in the transesterification process of used cooking oil and assess the quality of the produced biodiesel, hoping to provide a more environmentally friendly solution for biodiesel production.

METHODOLOGY

Materials and Instrumentals

Lapindo mud comes from Porong, Sidoarjo Regency, East Java, 600 m away from the center of the burst (Figure 1). Used cooking oil (UCO) comes from food production or restaurants in the Surabaya area. The reagents employed in this study comprised distilled water, methanol (Merck), ethanol (Merck), 0.5 N potassium hydroxide solution, and phenolphthalein as an indicator.

Figure 1. Lapindo mud sampling location

The instruments utilized in this study included GC-MS (Shimadzu QP2010 Plus), XRF (PANalytical Minipal 4), XRD (PANalytical X'Pert PRO), FTIR (IR Prestige 21), and PSA (Malvern Nano ZS). Additional equipment comprised a stopwatch, oven, furnace, analytical balance, hotplate magnetic stirrer, condenser, 500 mL three-neck flask, separating funnel, desiccator, thermometer, and various other standard glassware.

Methods

Pharmacokinetic Evaluation CLM Nanocatalyst

Before the synthesis process, Lapindo Mud (LM) was prepared by washing using distilled water to remove impurities, then dried under direct sunlight. After drying, LM was pulverized and sieved with 80 mesh to produce uniform particles. The synthesis of LM was carried out through calcination for 3 hours at 550^{o}C (Talib et al., 2016), produce CLM. The CLM was then ball-milled for 1 hour at 4000 rpm at a catalyst-to-ball mass ratio of 1:12, so CLM nanocatalysts are obtained, as shown in Figure 2.

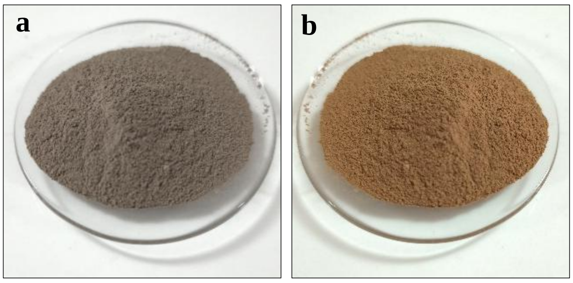

Figure 2. Lapindo Mud before and after calcination (a, b), in the form of nano-sized particles.

The nanocatalyst was characterized based on X-ray florescence (PANalytical Minipal 4), Fourier Transform Infrared Spectroscopy (Shimadzu IR Prestige 21) with wavenumbers in the range of 4000-400 cm^{-1}, X-ray diffraction (PANalytical X'pert PRO Analytical diffractometer) with XRD pattern with 2θ measurement, and. PSA (Malvern Nano ZS) to determine the particle size distribution of nanocatalysts.

Syntesis and Characterization Biodiesel

The activity of Lapindo Mud-based nanocatalysts was evaluated for biodiesel production using used cooking oil (UCO) as feedstock. The prepared catalyst was homogenized for two hours at 65 °C in a three-neck flask following the addition of oil and methanol at a 1:9 molar ratio (Talib et al., 2016). The excess methanol was used to shift the reaction equilibrium toward product formation (Tahya et al., 2019). The mixture was then placed in a separatory funnel and left to stand until two layers formed. After being separated, the top layer was washed with water. The top and bottom layers were separated, and any remaining water in the upper layer was evaporated at 105°C for 1 hour (Sitanggang & Aras, 2024).

The biodiesel was characterized by GCMS, to determine the methyl ester derivative compounds contained. In addition, biodiesel quality was analyzed based on several parameters, such as yield of biodiesel, density, viscosity, acid number, and moisture content.

a Yield of biodiesel

The yield is the ratio between the weight of biodiesel obtained and the weight of used cooking oil used. The yield can be calculated by the formula in Equation (1) (Sitanggang & Aras, 2024).

$$\text{Yield} = \frac{\text{mass biodiesel (g)}}{\text{mass of oil (g)}} \times 100\% \quad (1)$$

b Density

Density was measured using a pycnometer by comparing the weight of the biodiesel to its volume. The density value was then calculated using the formula in Equation (2) (Santoso et al., 2024).

$$\rho = \frac{m_1 - m_2 (g)}{v\ (cm^3)} \quad (2)$$

where m_1 is mass of pycnometer containing sample and m_2 is mass of empty pycnometer.

c Viscosity

Viscosity was measured using an Ostwald viscometer, with the measurement performed three times. The average of the measured data was taken as the final result (Maleki & Talesh, 2021). Biodiesel was drawn into the viscometer by suction until it reached the upper limit line, and the time for the biodiesel to flow to the lower limit line was recorded. The viscosity value was then calculated using the formula in Equation (3) (Mujiyanti et al., 2020),

$$V = \frac{(C_1 \times t_1) + (C_2 \times t_2)}{2} \quad (3)$$

where V is viscosity (mm^2/s), C_1 and C_2 is constanta of viscosity, and t_1 is initial flow time while t_2 is final flow time (s).

d Acid number

The acid number was measured by mixing biodiesel with warm ethanol and phenolphthalein (PP) indicator to signal the endpoint of the titration. The titration was performed using 0.5 N KOH (Akhtar et al., 2023). The acid number value was calculated using the formula in Equation (4) (Maarani et al., 2023).

$$\text{Acid number} = \frac{\text{FW KOH} \times \text{mL KOH} \times 0{,}1\ \text{N}}{\text{g sample}} \quad (4)$$

e Moisture content

Moisture content was measured by comparing the weight of the sample before and after heating. The biodiesel was heated at 105°C for 30 minutes (Daryono et al., 2020). W_1 represents the weight of the cup containing the sample before heating, W_2 represents the weight of the cup containing the sample after heating, and W_0 represents the weight of the empty cup. The moisture content value was calculated using the formula in Equation (5).

$$\text{Moisture content} = \frac{W_1 - W_2}{W_1 - W_0} \times 100\% \quad (5)$$

RESULTS AND DISCUSSION

Analysis of CLM Nanocatalyst

Table 1 shows the XRF data for CLM nanocatalyst. According to Shinde et al. (2021), The presence of silica in Lapindo Mud makes it suitable for use as a catalyst material because of its broad micropore and mesopore structure, which enables reactants to adsorb and react on the catalyst surface (Shinde et al., 2021). Trisunaryanti et al. (2022) used lapindo mud as a catalyst to study the manufacture of biofuel. According to the study, the percentage compositions of silica (SiO_2), ferric oxide (Fe_2O_3), and alumina (Al_2O_3) in Lapindo Mud (LM) and Lapindo Mud after Calcination (CLM) were 41.16, 18.24, and 12.84% in LM and 39.09, 19.24, and 12.22% in CLM, respectively. In that study, CLM was used without further modification into nanoscale form, whereas in the present study, CLM was utilized as a nanocatalyst. Consequently, the study strengthened the argument for using Lapindo Mud, which has greater potential as a catalyst material because of its higher silica and alumina content, as well as the LM and CLM content in Table 1.

Figure 3 shows the XRD data obtained from CLM nanocatalyst. The SiO_2 diffraction peaks on the CLM nanocatalyst appear at 2θ = 20.89°, 26.69°, 36.59°, 42.40°, 45.57°, 50.14°, 59.98°, and 68.17°, which corresponds to the ICDD 01-081-0065 database, where the SiO_2 peak has a quartz phase. The number of SiO_2 diffraction peaks that appear indicates the number of SiO_2 crystals contained in the catalyst. In addition to the detected SiO_2 peak, there are other

Table 1. XRF result on LM, CLM, and CLM nanocatalyst

Compound	Content (%)		
	LM*	CLM *	CLM nanocatalyst
SiO_2	41.16	39.09	47.0
Fe_2O_3	18.24	19.24	22.3
Al_2O_3	12.84	12.22	15

diffraction peaks such as Al_2SiO_5, Al_2O_3, and Fe_2O_3 peaks. The Al_2SiO_5 diffraction peaks appeared at 2θ = 19.75°, 23.69°, and 27.83°, which corresponds to the ICDD 01-074-1827 database. The Al_2O_3 peak appears at 2θ = 33.20°, which corresponds to the ICDD 00-001-1304 database. The Fe_2O_3 peak appears at 2θ = 35.66°, which corresponds to the ICDD 01-073-0603 database.

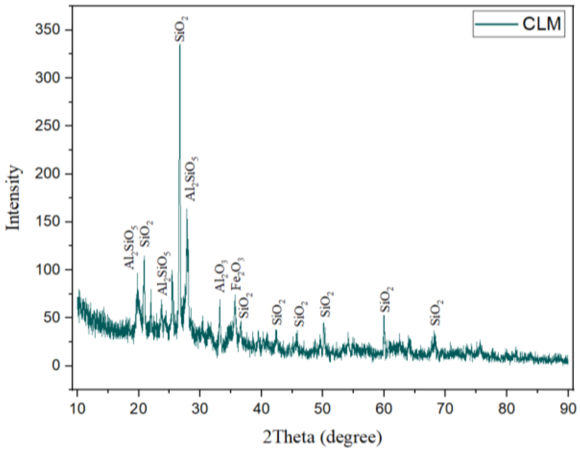

Figure 3. Peak XRD of CLM nanocatalyst

Figure 4 shows the FTIR spectra of CLM nanocatalysts. The peak CLM nanocatalyst at 1650 cm^{-1} was Si-OH bending functional group vibrations, which indicate the presence of silanol groups that may act as Brønsted acid sites and contribute to the catalytic activity in the transesterification reaction (Wijaya et al., 2024). Meanwhile, the peak CLM nanocatalyst at 3350 cm^{-1} was -OH stretching vibrations. This -OH stretching vibration appears to be sourced from H_2O and created by the physical adsorption of H_2O molecules on the surface of the nanocatalyst (Trisunaryanti et al., 2022). Sahu et al., (2023) identified a peak at 1047 cm^{-1}, indicating a Si-O-Si stretching vibration (Sahu et al., 2023) and CLM nanocatalyst indicates asymmetrical Si-O-Si stretching vibrations at 1169 cm^{-1}. Talib et al., (2016) identified a peak at 455 cm^{-1}, indicating a Si-O-Si bending vibration and CLM nanocatalyst indicates Si-O-Si bending vibration at 553 cm^{-1} (Talib et al., 2016).

Figure 5 shows the particle size distribution for CLM nanocatalysts. According to DLS analysis, the CLM nanocatalyst has an average particle size of 514.3 ± 361.8 nm, with the smallest size being 156 nm and the dominating size being 307.8 nm. El-Sheriff et al. (2023) identified a nanocatalyst with a particle size of 506 ± 42.69 nm, likely due to particle aggregation (El-sherif et al., 2023).

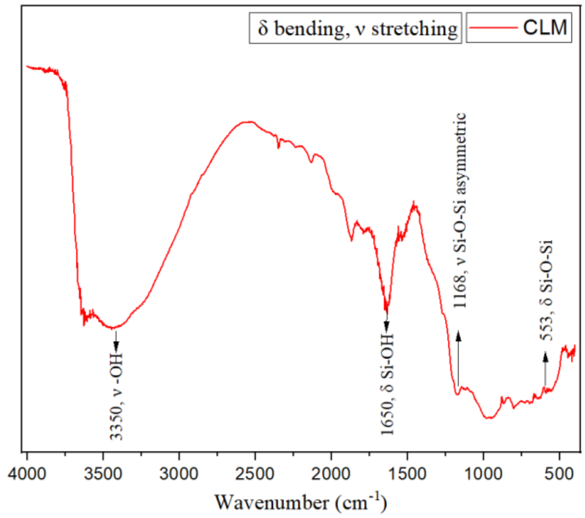

Figure 4. FTIR spectra of CLM nanocatalyst

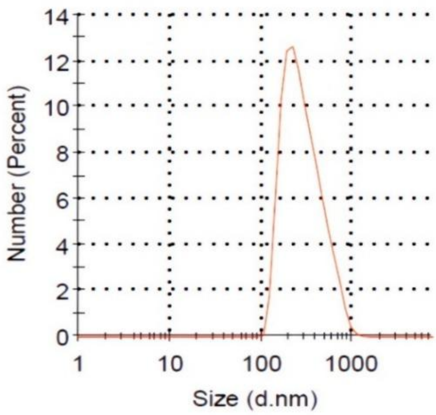

Figure 5. Size distribution by particle number CLM nanocatalyst

Analysis Quality of Biodiesel

Table 2 shows the yield of biodiesel produced during the transesterification reaction with the CLM catalyst and nanocatalyst. An analysis of biodiesel yield from the usage of a CLM catalyst was used to measure the effectiveness of the catalyst's size in contributing with product production. The yields from the transesterification reaction with CLM catalyst and CLM nanocatalyst were 45.32% and 77.36%, respectively. According to this, the CLM nanocatalyst works better because it has a larger surface area, enhancing catalytic activity in the transesterification step.

Table 2. Percentage yield of biodiesel

Type of CLM	Yield (%)
Catalyst	45,32
Nanocatalyst	77,36

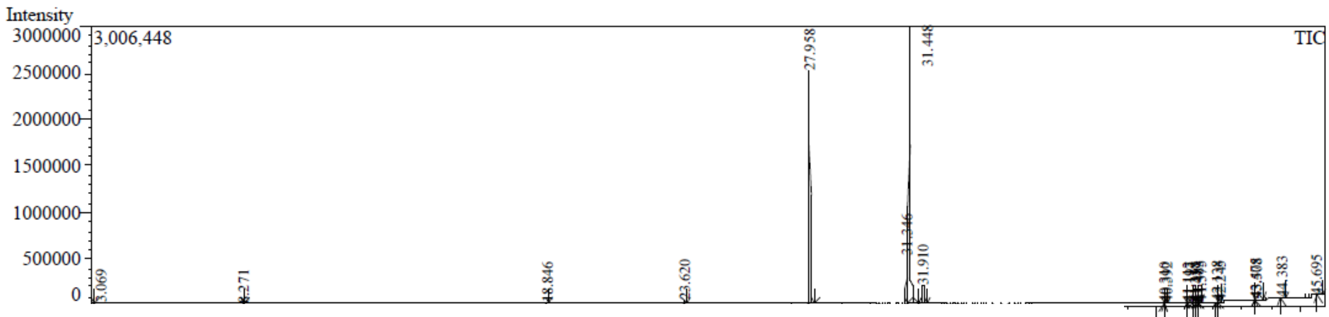

Figure 6. GCMS chromatogram of biodiesel CLM nanocatalyst

The yield of biodiesel produced using CLM nanocatalysts was then analyzed using GCMS to determine the levels of methyl ester derivative compounds contained therein, as shown in Table 3. The methyl ester content in CLM nanocatalyst biodiesel reached 99.26%. Based on the ASTM 6751-09 standard, the minimum methyl ester content of biodiesel is 96.5%, so the CLM nanocatalyst biodiesel has exceeded the minimum biodiesel standard. Among the methyl ester compounds in the nanocatalyzed biodiesel, the peaks detected in the GCMS analysis showed three peaks with the highest values, namely methyl palmitate, methyl linoleate and methyl oleate reaching 37.01%, 7.44 and 50.87%, respectively, as shown in Figure 6.

Table 3. Identification and composition of methyl esters in biodiesel catalyzed by CLM nanocatalyst based on GC-MS analysis

Methyl ester compounds	Retention time (min)	% Area
Methyl undecanoate	18.850	0.23
Methyl Tridecanoate	23.617	0.53
Methyl palmitate	27.958	37.01
Methyl linoleate	31.350	7.44
Methyl oleate	31.450	50.87
Methyl Heptadecanoate	31.908	3.18
Total % area		99.26

The three highest methyl esters identified in the biodiesel catalyzed by CLM nanocatalyst are methyl oleate, methyl palmitate, and methyl linoleate, which contain 16 or more carbon atoms. The molecular formulas of the three methyl ester compounds are methyl palmitate $C_{17}H_{34}O_2$, methyl oleate $C_{19}H_{36}O_2$, and methyl linoleate $C_{19}H_{34}O_2$. This shows that methyl palmitate has the shortest carbon chain length compared to other methyl esters so that it appears first from methyl oleate and methyl linoleate (Prastyo et al., 2022). The peak of methyl linoleate appears first, then methyl oleate, because the molecular weight of methyl oleate is greater than that of methyl linoleate (Prastyo et al., 2021).

Table 4 presents the quality parameters of biodiesel catalyzed by the CLM nanocatalyst—density, viscosity, moisture content, and acid number—compared with the international biodiesel standard ASTM D6751-09. Table 4 shows that the viscosity and acid number of biodiesel catalyzed by the CLM nanocatalyst satisfied the specifications, but the density value and moisture content was incompatible with the biodiesel quality standards. The density exceeding the maximum standard may indicate an incomplete washing and purification process, while the high moisture content suggests insufficient drying (Sari et al., 2016). This condition may result in reduced combustion efficiency of the biodiesel (Amalia et al., 2020). The acceptable viscosity ensures proper fuel flow and atomization during combustion (Laila & Oktavia, 2017), while the low acid number is favorable as it reduces the potential for engine corrosion and degradation (Oko et al., 2021).

Table 4. Physicochemical quality parameters of biodiesel catalyzed by CLM nanocatalyst

Parameter	**This study**	**ASTM 6751-09**
Density (kg/m^3)	929.4	860-890
Viscosity (mm^2/s)	4.52	1.9-6
Moisture content (%)	0.097	Maximum 0.05
Acid number (mg-KOH/g)	0.50	Maximum 0.50

CONCLUSION

This study demonstrates that Lapindo mud works as a heterogeneous catalyst, with improved effectiveness in nanoparticle form. This is demonstrated by the increased biodiesel production achieved with CLM nanocatalyst compared to standard CLM catalyst. XRD data of the CLM nanocatalyst shows the presence of SiO_2, Al_2O_3, and Fe_2O_3, which correlates to the CLM content revealed in the XRF data. The transesterification process of used cooking oil with CLM nanocatalyst gave 99.26% methyl ester at a reaction temperature of 65°C for 2 hours, a methanol-to-oil molar ratio of 9:1 and a catalyst concentration of 3% (w/w). The resulting biodiesel exhibited physicochemical properties such as viscosity and acid number that met the ASTM D6751-09 standard, while density and moisture content did not meet the standard. These results indicate that the CLM nanocatalyst effectively promoted the transesterification reaction, but further optimization of the purification and drying processes is necessary to improve overall fuel quality and ensure compatibility with combustion systems

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