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Determination of Optimum Conditions for Biodiesel Synthesis from Bulk Palm Oil Using Red Mud-CaO Catalyst

Sri Nurdianty, Thamrin Usman*, Winda Rahmalia

Department of Chemistry, Faculty of Mathematics and Natural Sciences, Tanjungpura University,
Jl. Ahmad Yani Pontianak, West Kalimantan 79124, Indonesia
*Corresponding author: Thamrin Usman, Email: thamrin_usman@chemistry.untan.ac.id

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ABSTRACT

The synthesis of biodiesel from bulk palm oil using methanol and a heterogeneous catalyst, red mud-CaO, has been carried out. This research aimed to investigate the effect of the ratio of red mud-CaO, percent catalyst, and reaction time on the conversion of methyl ester. Red mud and CaO catalysts were obtained from calcining red mud and clay bath $CaCO_3$ (calcium carbonate) waste at 200°C and 900°C for 5 and 4 hours, respectively. Biodiesel synthesis was carried out by adding bulk palm oil:methanol (1:10 v/v) and red mud-CaO heated for 1, 2, and 3 hours at 65 °C. In addition, the reaction was followed using thin-layer chromatography (TLC) and Gas Chromatography-Mass Spectrophotometry (GC-MS). Optimum reaction conditions were obtained at the ratio of red mud-CaO (1:1 w/w), catalyst 5%, and time of 2 hours indicated by the total formation of methyl ester on TLC plate. Meanwhile, red mud-CaO (1:1 w/w) catalyst was characterized by x-ray diffraction (XRD) and x-ray fluorescence (XRF) to determine the crystal structure and element composition. The analysis was carried out using Fourier transform infra-red (FTIR) to determine the functional groups of catalyst. XRD characterization results showed that there were 4 highest peaks, namely 29.29° ($CaCO_3$), 33.99° and 17.93° ($Ca(OH)_2$), and 11.59° (aluminum silicate mineral). The 4 highest elemental contents were Ca, Fe, Al, and Si. FTIR characterization showed the peak wavenumbers 3641.60 cm^{-1} ($Ca(OH)_2$),1423.47 cm^{-1} (O-C-O bond of carbonate), 981.77 cm^{-1} and 986.69 cm^{-1} (Si-O), 875.68 cm^{-1} and 713.66 cm^{-1} (Ca-O), 366.48 cm^{-1} (Al-O), and 426.27 cm^{-1} (Fe-O). According to GC-MS analysis, the most formed methyl ester compositions were methyl oleate (47.35%) and methyl palmitate (40.13%).

Keywords: Biodiesel; bulk palm oil; catalyst; transesterification

INTRODUCTION

The need for fuel oil is increasing and this condition is inversely proportional to the dwindling energy reserves causing an energy crisis. Currently, renewable alternative energy is needed to anticipate dwindling oil reserves such as biodiesel. The Indonesian government responded to biodiesel policy as a new and renewable energy to reduce global warming (Hakim, 2017). Based on the 2018 Presidential Regulation, the Indonesian government implemented the use of sustainable Biodiesel 30 (B30) to B40 which is mandatory for public transportation and industry.

Biodiesel is an alternative fuel produced from the transesterification reaction of vegetable oil (triglycerides) with methanol. Vegetable oil commonly used as raw materials for biodiesel varies, including soybean oil, coconut oil, sunflower oil, or palm oil (CPO). In this

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context, palm oil (CPO) has great potential as a raw material for biodiesel due to the abundant availability of raw materials (Yustira et al., 2018).

The use of CPO through transesterification reactions is inhibited by the presence of free fatty acids (FFA) with a fairly high percentage above 1%. The presence of FFA can cause soap formation when the transesterification reaction takes place using a base catalyst (Wendi et al., 2015). Therefore, a refining process is needed for CPO to remove impurities such as FFA ranging from 4-5%, β-carotene, phospholipids, and water content. CPO refining produces palm oil with the specified quality (Mahmud, 2019) and this process is carried out to remove impurities affecting biodiesel formation. The treatment requires a fairly long time in biodiesel synthesis, hence, this research uses another alternative in the form of bulk palm oil (Lempang et al., 2016).

Biodiesel synthesis carried out through transesterification reactions requires basic catalysts such as NaOH and KOH. However, these catalysts are less efficient because of the homogeneous nature, difficulty in separating from the reaction results, and inability to be regenerated or reused. The weaknesses have led to the development of heterogeneous catalysts with advantages including easy separation from the product, reusability, and the potential to reduce biodiesel production costs. Heterogeneous catalysts used include red mud, CaO (calcium oxide), MgO (magnesium oxide), and $CaCO_3$ (calcium carbonate) (Julianti, 2014) developed for transesterification reactions. Red mud is solid-phase waste produced from the processing of bauxite into alumina (Senthil et al., 2016). The amount can reach 40-50% of the weight of bauxite ore processed using the Bayer method (Sipayung, 2019). Red mud has a hydroxide content with a pH ranging from 10-13 and is classified as B3 waste (hazardous and toxic materials) which can damage the environment (Liu et al., 2013; Senthil et al., 2016). The high pH content can be an added value as a heterogeneous base catalyst in biodiesel synthesis. However, Liu et al. (2013) reported that the use of catalyst was not maximized in producing methyl esters with a relatively long time and a fairly large catalyst percentage.

CaO is a material with the potential to be developed as a heterogeneous catalyst in biodiesel synthesis. This compound is very effective because the base strength is relatively high, less soluble in methanol, and environmentally friendly (Zabeti et al., 2009). Usman et al. (2022) conducted research using CaO from claybath waste in synthesizing biodiesel under reaction conditions with 5% catalyst, 1 hour reaction time, 60°C temperature with total methyl ester conversion. CaO was obtained from claybath waste in the process of separating palm shells and kernels in the form of $CaCO_3$ suspension solution. $CaCO_3$ has the potential to be used because of the abundant availability in Indonesia. Therefore, research related to biodiesel synthesis using catalyst raw materials from alumina industrial waste and claybath waste was conducted. The parameters were the mass ratio of red mud-CaO, variations in the percentage of red mud-CaO catalyst, and reaction time to determine the optimum conditions for the synthesis. Catalyst was characterized using x-ray diffraction (XRD) and x-ray fluorescence (XRF) to determine the crystal structure and element composition, while Fourier transform infra-red (FTIR) was used to determine the functional groups.

METHODS

Materials

The raw materials were bulk palm oil purchased from the Flamboyan Market in Pontianak City, $CaCO_3$ taken from claybath waste from CPO processing at PT Pundi Lahan Khatulistiwa (PLK) Kubu Raya, and red mud from PT. Indonesia Chemical Alumina (ICA) Tayan.

The tools included a set of reflux tools, FTIR IRPrestige-21 SHIMADZU (Shimadzu Corporation, Kyoto, Japan), Gas Chromatography-Mass Spectrophotometry (GC-MS) QP2010S SHIMADZU (Shimadzu Corporation, Kyoto, Japan), XRD Philips Xpert MPD (Philips, Amsterdam, Netherlands), and XRF PANAlitical Epsilon 3 (Malvern Panalytical Ltd, Malvern, United Kingdom), and other laboratory equipments.

Sample Preparation

Red mud sample from PT. ICA was prepared by heating in an oven at a temperature of 100 °C for 24 hours. The dried red mud was ground until smooth and sieved with a 100-mesh sieve. Subsequently, the sample was calcined in a furnace at 200 °C for 5 hours and the prepared red mud was analyzed using XRD (Liu et al., 2013).

$CaCO_3$ from claybath waste was filtered, centrifuged for 15 minutes and heated in an oven at 105°C for 24 hours. The dried $CaCO_3$ waste was ground and sieved using a mortar and 100-mesh sieve. $CaCO_3$ sample was calcined at 900°C for 4 hours to produce CaO and the resulting product was analyzed using XRD (Haryono, 2018).

Preparation of Red Mud-CaO Catalyst

The previously prepared red mud and CaO sample was mixed with variations in the mass ratio of red mud to CaO of 1:1 and 1:2 w/w. In addition, red mud-CaO was stirred, ground until homogeneous, and sieved with a 100-mesh sieve. Catalyst with maximum biodiesel yield was characterized by XRD, XRF, and FTIR. Identification and characterization of catalysts using XRD were carried out at the Materials and Metallurgy Laboratory, Sepuluh November Institute of Technology. The characterization was performed by placing the sample into XRD machine, and scanning was carried out to identify the type of compound formed from the material. The crystal structure of catalyst was also identified with variations in the mass ratio of red mud-CaO. Meanwhile, the diffraction pattern provided information about the typical angles of the material atoms (2θ).

Identification and characterization of catalysts using XRF were carried out at the Instrumental Chemistry Laboratory, Padang State University, by preparing the sample to be tested. XRF test tool was directed at the sample and shot with X-ray before the appearance of the material composition data on the monitor layer. In addition, identification and characterization of catalysts using FTIR were performed at the Chemistry Laboratory, Hassanudin University, where the sample was ground with KBr (potassium bromide) powder and pressed in a plate to form pellets. In this context, the pellets were inserted into the sample holder and analyzed at wavenumbers 4000-400 cm^{-1}.

Transesterification Reaction of Bulk Palm Oil Using Red Mud-CaO Catalyst

Transesterification reaction was carried out by inserting red mud-CaO in a three-necked flask with variations in the ratio of red mud-CaO at 1:1 and 1:2 w/w with catalyst percentages of 1%, 3%, and 5%. Methanol was added at a molar ratio of oil/methanol of 1:10 w/w with magnetic stirring in a three-necked flask for variations of 1, 2, and 3 hours at a temperature of 65 °C. After the reaction was complete, the mixture was centrifuged for 15 minutes and 3 layers were formed at the bottom (catalyst), middle (methyl ester), and top (methanol). The reaction was monitored through TLC with a silica gel 60 F$_{254}$ plate and cyclohexane:dichloromethane as the stationary and mobile phase with a ratio of 2:1 v/v. After 3 hours of reaction, the products in the form of methyl ester and methanol were separated using a separating funnel. Methyl ester product was characterized using GC-MS (Yustira et al., 2016). Identification and characterization of the phase using GC-MS were carried out at the Organic Chemistry Laboratory, Gajah Mada University. GC-MS autosampler used Thermo Scientific TriPlus RSH as well as HP-5MS and TG-WAXMS columns. The sample was injected into GC through an injector, and heated at a certain temperature until vapor was formed. The sample in the form of vapor would be carried by the carrier gas to enter the column heated at a certain temperature. Based on the chemical and physical interactions between the sample, carrier gas, and the material making up the column, the sample would be diffracted into single compounds.

RESULTS AND DISCUSSION

Catalyst Characterization

Red mud powder calcined at a temperature of 200 °C was not in the form of oxide but amorphous. Liu et

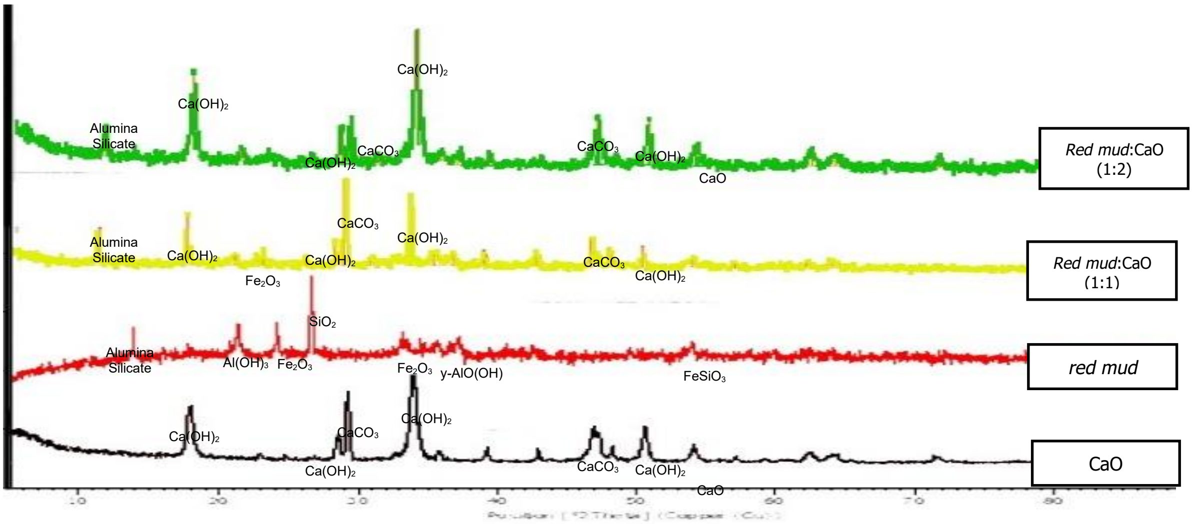

Figure 1. XRD characterization

al. (2013) stated that red mud calcination at 200 °C was the best temperature and affected biodiesel yield by 94%. Calcination or thermal decomposition of $CaCO_3$ suspension from claybath was conducted in a furnace at 900°C for 4 hours. The selection of the temperature is based on Haryono et al. (2018), where the thermal decomposition of $CaCO_3$ has reached an equilibrium condition at 850°C as indicated by a decrease in the weight of the material due to the release of CO_2 gas.

XRD characterization results of CaO, red mud, and variations of red mud-CaO are presented in Figure 1. CaO catalyst diffractogram at an angle of 2θ = 54.37° in accordance with JCPDS 82-1691 is a characteristic of CaO. $Ca(OH)_2$ also appear in the diffraction pattern 2θ = 18.15; 28.71; 34.00; and 50.87° in JCPDS 01-073-5492 database. Similarly, diffraction peaks appear at angles 2θ = 29.40; 47.21; and 47.55° which are typical peaks of $CaCO_3$ crystals in JCPDS 47-1743 database. The presence of peaks 47.21 and 47.55° is an indication that the conversion of $CaCO_3$ to CaO is not perfect. These are consistent with Nurhayati (2016) where there are diffraction peaks at 2θ = 17.87°, 28.55°, 29.35°, 34.18°, 47.12°, 50.71°, and 53.88°.

Identification of XRD data of red mud shows the presence of hematite mineral phase (Fe_2O_3), reported by diffraction peaks at angles 2θ= 24.10, 33.07, and 35.62° according to JCPDS 00-033-0664 database. The diffraction at angle 2θ= 21.32° is the peak of gibbsite mineral corresponding to (JCPDS 00-007-0324). Boehmite, quartz, ferrosilite ($FeSiO_3$), and aluminium silicate minerals (mullite) have a typical diffraction peak at angle 2θ= 37.08° (JCPDS 00-0211307), 2θ= 26.53° (JCPDS 00-005-0490), 2θ= 53.94°, and 2θ = 13.79°, respectively. The diffraction peaks of red mud are related to Sushil and Batra (2012) at 2θ = 12.82, 20.50, 24.81, 26.49, 38.40, and 35.94° as well as by Wanda's research (2021) where there are diffraction peaks at 2θ are 33.04 and 54.26°.

The pattern results of red mud-CaO variation (1:1 w/w) show a combination of diffraction peaks from red mud and CaO. In the variation (1:1 w/w), 6 peaks originate from CaO, namely 17.93, 28.60, 29.29, 33.99, 47.02, and 50.71° while 2 are from red mud, including 11.59 and 23.45°. However, the peaks experience a slight shift in the 2θ angle at red mud-CaO ratio (1:2 w/w) due to the addition of CaO. The highest diffraction peaks are at angles 2θ = 11.67, 18.02, 28.68, 29.36, 34.11, 47.15, 50.83, and 54.35°. In red mud-CaO with a variation (1:2 w/w), 7 peaks originate from CaO, namely 18.02, 28.68, 29.36, 34.11, 47.15, 50.83, and 54.35° as well as 1 peak from red mud, namely 11.67°. The peaks from the variations of red mud and CaO show that catalyst does not experience bonding but only mixing.

Catalyst analysis is also strengthened by the results of XRF characterization. This shows the presence of a more dominant Ca (calcium) element content of 63.648% which is followed by Fe (iron) (19.24%), Al (Aluminum) (7.771%), and Si (Silicon) (5.963%). The content of red mud:CaO (1:2 w/w) catalyst experienced an increase in Ca elements, but Fe, Al, and Si elements decreased.

The elements are supported by FTIR data presented in Figure 2, where the spectrum of red mud-CaO 1:1 w/w catalyst shows a peak at wavenumber 3641.60 cm^{-1} indicating the presence of hydroxyl groups (O-H) from $Ca(OH)_2$. CaO is hygroscopic since the compound easily absorbs water vapor and air (Granados et al., 2007; Fatmawati et al., 2018). The peak observed at wavenumber 1423.47 cm^{-1} is a characteristic of O-C-O carbonate bond, showing that there is $CaCO_3$ yet to be converted into CaO. These results are in line with Granados (2007) where the peaks at wavenumbers

Table 1. Catalyst composition from XRF characterization results

Metal	Red mud-CaO (1:1 w/w)	Red mud-CaO (1:2 w/w)	Oxide	Red mud-CaO (1:1 w/w)	Red mud-CaO (1:2 w/w)
	Abundance (%)	Abundance (%)		Abundance (%)	Abundance (%)
Al	7.771	5.058	Al_2O_3	11.523	7.744
Si	5.963	3.784	SiO_2	9.788	6.452
P	0,.566	0.481	P_2O_5	0.978	0.866
Ca	63.648	76.182	CaO	58.764	72.112
Ti	1.494	0.923	TiO_2	1.512	0.956
Fe	19.24	12.49	Fe_2O_3	16.387	10.961
Ag	0.489	0.807	Ag_2O	0.368	0.625

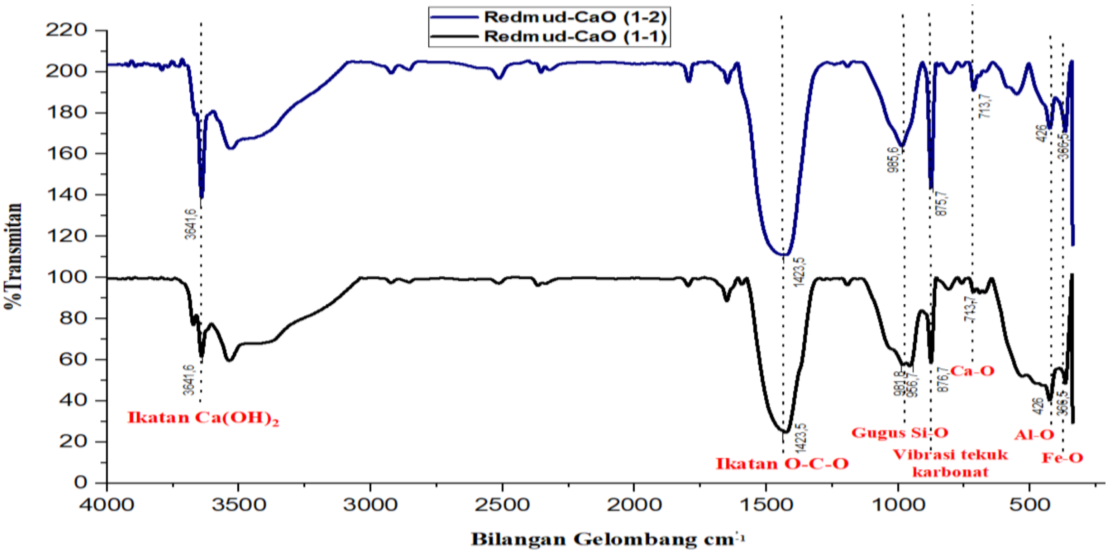

Figure 2. FTIR spectrum of red mud-CaO

1479 and 1419 cm^{-1} are typical for O-C-O bond from CaO surface. The peaks at wavenumbers 981.77 and 986.69 cm^{-1} indicate the presence of Si-O groups. Meanwhile, wavenumbers 875.68 cm^{-1} and 713.66 cm^{-1} are the bending vibrations of the carbonate group and Ca-O bonds, respectively (Granados et al., 2007). The peak at wavenumbers 366.48 cm^{-1} and 426.27 cm^{-1} shows the presence of Al-O and Fe-O groups, respectively. Based on FTIR analysis, red mud-CaO has several functional groups such as OH, Si-O, Al-O, and Fe-O.

Bulk Palm Oil Transesterification

Transesterification reaction uses bulk palm oil as the base material with FFA content of 0.49%. The process starts by weighing red mud-CaO at various ratios, namely 1:1 and 1:2 w/w. Subsequently, red mud-CaO is reacted with methanol and bulk palm oil at an oil:methanol ratio of 1:10 w/w. Previous research conducted by Wahyudi (2016) stated that the optimum oil:methanol ratio was 1:10 w/w. An increased amount of methanol causes a back reaction (reaction) and reduces the methyl ester conversion results. The high methyl ester conversion results with the mole ratio of 1:10 w/w show that the reaction has proceeded (shifting towards the product). Methanol is used as an alcohol in the reaction due to the short chain and higher reactivity than other alcohols. The use also produces purer methyl ester products when compared to other types of alcohol such as ethanol, propanol, or butanol (Hass et al., 2004).

Transesterification reaction uses the reflux method with the help of a magnetic stirrer and the heating process is at a temperature of 65 °C. The temperature is directly proportional to the fastness of the collisions between molecules and energy (Aziz, 2003). The reaction was carried out for 3 hours and monitored through TLC every hour. TLC was carried out to determine the progress of the reaction and the formation of methyl esters qualitatively. The conversion of methyl esters in the variation of red mud-CaO 1:1 w/w with catalyst concentration of 5% for 2 hours is the best condition. The total conversion can be seen through TLC elution results in Figure 3.

Based on TLC test in Figure 3, red mud-CaO can convert bulk palm oil into biodiesel at 1%, 3%, and 5%. Red mud-CaO (1:1 w/w) with a concentration of 5% at hours 2 and 3 showed the complete conversion of methyl esters, which was reported by the presence of ester stains and no residual oil stains. However, the use of catalyst concentrations of 1% and 3% until hour 3 still showed oil stains. The use of 1% and 3% red mud-CaO has not been able to convert biodiesel completely due to the direct relationship between the percentage of catalyst and the reaction results. The longer the contact time between the reactants, the greater the product obtained. In the variation of red mud-CaO (1:2 w/w) with catalyst concentrations of 1%, 3%, and 5%, methyl ester has been formed, but only partial part is formed.

The conversion ability of methyl ester has been converted completely at a ratio of 1:1 and 1:2 in relation to XRD characterization. XRD diffractogram at a ratio of 1:2 shows an increase in CaO diffraction peak and a slight shift in others. A ratio of 1:1 has an optimum pH of 12.83 and the best catalyst in the variation of red mud-CaO (1:1 w/w) with catalyst percentage of

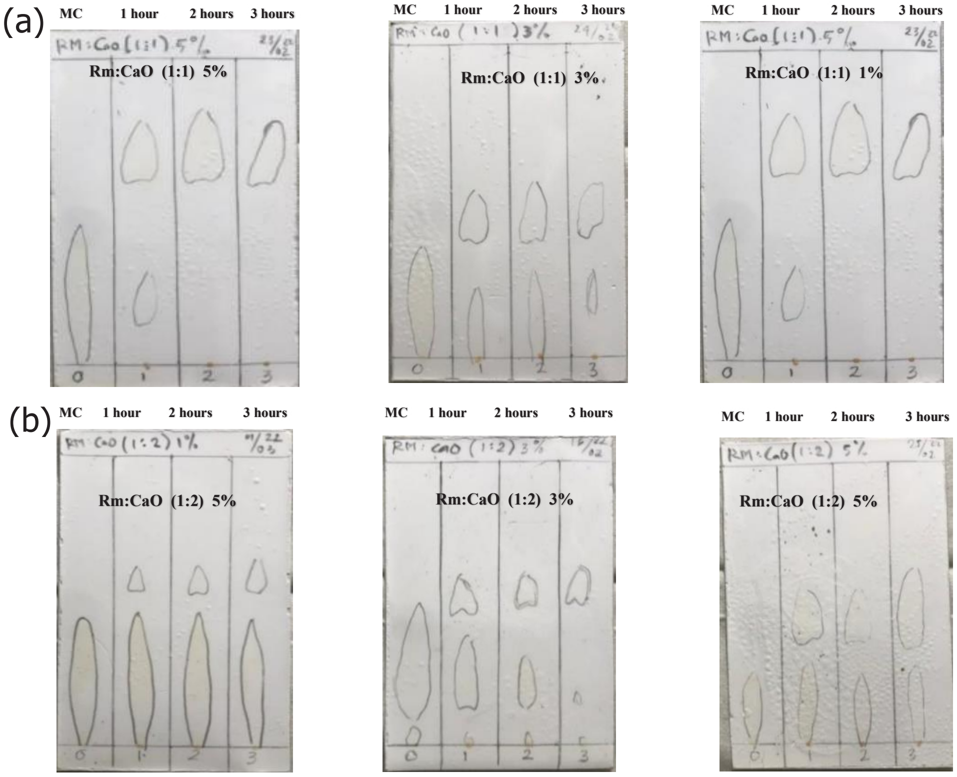

Figure 3. TLC results of transesterification synthesis using red mud-CaO with variations in the ratio of red mud-CaO (1:1 w/w) (a), red mud-CaO (1:2 w/w) (b) at catalyst concentrations of 5%, 3%, and 1%

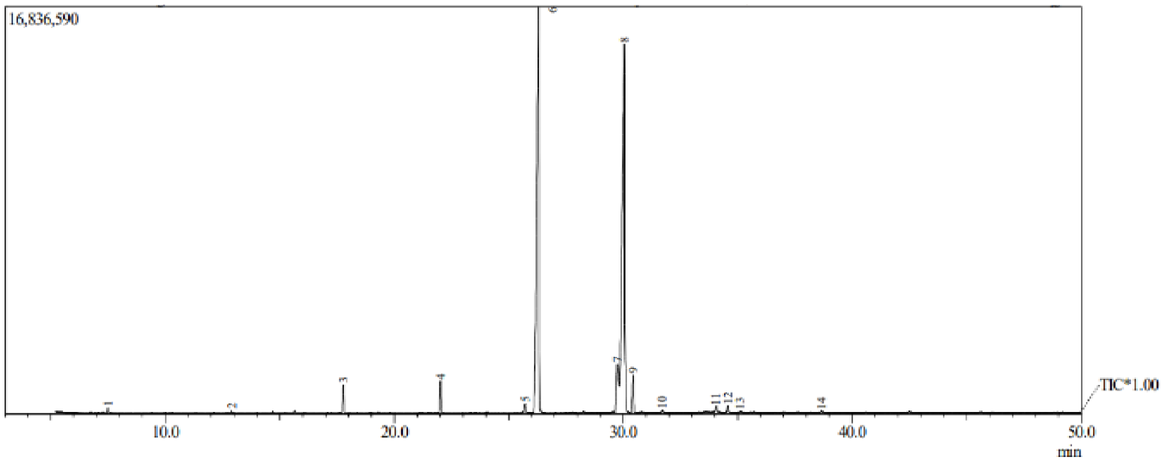

Figure 4. Chromatogram of methyl ester from transesterification results

5% for 2 hours. Subsequently, the best conversion results were subjected to GC-MS characterization tests to identify the type of fatty acids in the form of methyl esters as well as the abundance of each fatty acid composition.

Based on the chromatogram of Figure 4, 14 peaks were detected, but the compound formula in line with the standard spectrum is only 9. Table 2 shows the component of methyl ester from transesterification results of bulk palm oil.

The highest peak at number 8 and 6 with an area of 47.35% and 40.13% of the total area is chromatogram in the form of methyl oleate and palmitate compounds, respectively. Therefore, the most abundant ester

Table 2. Components of methyl ester compounds from transesterification results of bulk palm oil

No. Peak	Retention Time	Name	Area (%)
2	12.891	Methyl decanoate	0.14
3	17.776	Methyl laurate	1.29
4	22.022	Methyl tetradecanoate	1.55
5	25.711	Methyl palmitoleate	0.55
6	26.310	Methyl palmitate	40.13
7	29.737	Methyl linoleate	4.90
8	30.049	Methyl oleate	47.35
9	30.439	Methyl stearate	2.35
12	34.575	Methyl arachidonate	0.48

compound components in palm oil-based methyl esters are oleate and palmitate compounds.

CONCLUSION

In conclusion, the optimum conditions for biodiesel synthesis were obtained by using catalyst with variations in the ratio of red mud-CaO (1:1 w/w) at catalyst percentage of 5% in 2 hours. This was shown by the characterization of methyl esters using TLC plate that formed a total ester spots accompanied by XRD, XRF, FTIR, and GC-MS characterization. In addition, XRD characterization reported the presence of 4 highest peaks, namely 29.29°, 33.99°, 17.93°, and 11.59° which were characteristics of $CaCO_3$, $Ca(OH)_2$, $Ca(OH)_2$, and aluminium silicate minerals (*mullite*). Ca, Fe, Al, and Si contents were also reported at 63.65%, 19.24%, 7.77%, and 5.63%, respectively. FTIR characterization showed peaks at wavenumbers 3641.60 cm^{-1} ($Ca(OH)_2$), 1423.47 cm^{-1} (carbonate O-C-O bond), 981.77 cm^{-1} and 986.69 cm^{-1} (Si-O), 875.68 cm^{-1} and 713.66 cm^{-1} (Ca-O), 366.48 cm^{-1} (Al-O), and 426.27 cm^{-1} (Fe-O).

CONFLICT OF INTEREST

The authors declare no conflict of interest with other parties.

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