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Multifeedstock Biodiesel Production from a Blend of Five Oils through Transesterification with Variation of Moles Ratio of Oil: Methanol

Yoyon Wahyono^{1,3}, Hadiyanto^{1,2,3*}, Mochamad Arief Budihardjo⁴, Yogi Hariyono⁵, Rifqi Ahmad Baihaqi⁵

¹Doctoral Program of Environmental Science, School of Postgraduate Studies, Diponegoro University, Jl. Imam Bardjo SH, Pleburan, Semarang, Central Java 50241, Indonesia

²Department of Chemical Engineering, Faculty of Engineering, Diponegoro University, Jl. Prof. Soedharto, SH, Tembalang, Semarang, Central Java 50275, Indonesia

³Center of Biomass and Renewable Energy (C-BIORE), Diponegoro University, Jl. Prof. Soedharto, SH, Tembalang, Semarang, Central Java 50275, Indonesia

⁴Department of Environmental Engineering, Faculty of Engineering, Diponegoro University, Jl. Prof. Soedharto, SH, Tembalang, Semarang, Central Java 50275, Indonesia

⁵Department of Physics, Faculty of Science and Mathematics, Diponegoro University, Jl. Prof. Soedharto, SH, Tembalang, Semarang, Central Java 50275, Indonesia

Abstract. This study was conducted to produce biodiesel from a mixture of 5 different oils i.e, palm oil, used cooking oil, soybean oil, canola oil, and sunflower oil, through transesterification under mole ratio variations of oil: methanol. The oils were mixed at a total volume of 300 mL with the same amount of each oil used. The transesterification of blended oils was conducted at 60°C for 1 h, and the mole ratios of oil: methanol were set to 1:3, 1:6, 1:9, 1:12, and 1:15. The results demonstrated that the mole ratios of 1:6 resulted in the highest yield of 92.99% with the conversion of 99.58% mass. The gas chromatography-mass spectrometry (GCMS) results showed that all mole variations had a methyl ester percentage of more than 98% area. The FTIR analysis revealed peaks that indicated the presence of a methyl ester functional group and its long-chain (–R) for all variations. The methyl ester content, Density, acid value, and total glycerol test parameters were in accordance with the quality standards of ASTM D 6751, EN 14214, and SNI 7182–2015. Therefore, multi-feedstock biodiesel suitable for industrial-scale applications was successfully produced in this study.

Keywords: Biodiesel; Moles; Multifeedstock; Production; Transesterification

1. Introduction

The development of renewable energy is supported by the Indonesia government. For instance, biofuel from palm oil is developed in compliance with Government Regulation No. 79, 2014 (Republic of Indonesia government, 2014), which stipulates that the national renewable energy consumption must increase to 23% of the total energy use by 2025. In 2020, the production of biodiesel B30 (30% biodiesel and 70% diesel) for consumption was initiated. Biodiesel production that relies on vegetable oil as the sole feedstock is

^{*}Corresponding author's email: hadiyanto@live.undip.ac.id, Tel.: +62-82329771232 doi: 10.14716/ijtech.v13i3.4804

disadvantageous and can result in shortages of vegetable oil (Hadiyanto et al., 2020). Many countries, including Indonesia, do not sufficiently produce vegetable oil to sustain its use as a raw material for biodiesel production (Hadiyanto et al., 2018).

In Indonesia, the palm oil industry promotes aggressive deforestation to clear land for oil palm plantations, leading to shortages of raw materials (Soraya et al., 2014; Siregar et al., 2015; Wahyono & Hadiyanto, 2019). Therefore, biodiesel production using raw materials from a mixture of several vegetable oils and used cooking oil can be valuable for countries such as Indonesia with an increasing demand for biodiesel (Hadiyanto et al., 2018; Hadiyanto et al., 2020). Biodiesel produced from multiple raw materials can be called multi-feedstock biodiesel (Flood et al., 2016; Hadiyanto et al., 2020). Furthermore, raw materials from vegetable oil and used cooking oil can be a solution to the scarcity of raw materials.

Numerous studies on biodiesel production have proposed blends of oils, such as on canola oil mixed with cooking oil (Issariyakul et al., 2008); castor oil mixed with soybean oil (Barbosa et al., 2010); soybean oil mixed with rapeseed oil (Qiu et al., 2011); soybean mixed with tallow and canola (Flood et al., 2016); non-edible oils mixed with castor seed oil and waste fish oil (Fadhil et al., 2017a); Calophyllum inophyllum mixed with Jatropha curcas and Pongamia pinnata (Miraculas et al., 2018); waste cooking oil mixed with castor oil (Hadiyanto et al., 2018); and Calophyllum inophyllum oil mixed with castor oil, palm oil, and waste cooking oil (Hadiyanto et al., 2020). These studies have high success rates in producing multi-feedstock biodiesel with methyl ester contents higher than 80% mass. However, further studies should be performed on multi-feedstock biodiesel production (Flood et al., 2016; Hadiyanto et al., 2018; Hadiyanto et al., 2020). Research on a mixture of palm oil, used cooking oil, soybean oil, canola oil, and sunflower oil has yet to be conducted. Palm oil (Saksono et al., 2019), used cooking oil (Yusuff et al., 2018; Ani et al., 2018), soybean oil (Qiu et al., 2011), canola oil (Flood et al., 2016), and sunflower oil (Salmasi et al., 2020) contain triglyceride compounds that can be reacted with methanol to produce methyl ester (biodiesel) and glycerol.

High free fatty acids (FFA) and water content in used cooking oils (UCO) cannot be directly transesterified using an alkaline catalyst, which gives low yield and low quality of biodiesel. This is because the side saponification reaction consumes catalyst and generates soap which causes problems in producing high-quality biodiesel. Transesterification of used cooking oils with an alkaline catalyst can be done only when the FFA and water content have been removed through different pre-treatment processes (Canakci & Gerpen, 2001; Cvengros & Cvengrosova, 2004). Alternatively, the acid catalyst can be used instead to prevent the emergence of this saponification (Obibuzor et al., 2008). However, this approach requires a longer reaction time, a higher operating temperature, and an acid-resistible reactor. It is obvious that the exploitation of used cooking oils requires more sophisticated technology and a more complicated process, which increases the cost of the biodiesel production process (Issariyakul et al., 2008). A mixture of five raw materials, including four fresh vegetable oil and one used cooking oil, provides benefits. Adding fresh vegetable oil to used cooking oil would improve the yield and quality of biodiesel produced from direct alkali-catalyzed transesterification (Issariyakul et al., 2008). This study could provide an alternative means to make use of UCO for a low-cost biodiesel production process.

Methanol is an essential material in transesterification for biodiesel production. An environmental life cycle assessment study on biodiesel production has shown that methanol is one of the causes of the high environmental pollution of transesterification (Soraya et al., 2014; Siregar et al., 2015; Wahyono et al., 2020). Methanol is a volatile organic compound (VOC); as it evaporates into the atmosphere, it reacts with NOx, water vapor, and sunlight radiation, thereby forming photochemical oxidants (Zou et al., 2015; Wahyono et al., 2020). In Indonesia, the production of 1 ton of biodiesel from palm oil requires 1.28 tons of palm

oil and 0.64 tons of methanol (Siregar et al., 2015). This is equivalent to using 15 moles of methanol. Meanwhile, the yield of biodiesel produced is 78% (Sorava et al., 2014). The use of methanol should be reduced to make biodiesel production more environmentally friendly. Methanol emissions can be reduced by producing biodiesel with low methanol moles, namely, 3 and 6 moles. Minimizing the use of methanol moles in the biodiesel production process is an effort to reduce the amount of methanol used in biodiesel production. The target is to obtain biodiesel with high vield and quality with a more environmentally friendly production process. Therefore, this study is expected to produce multi-feedstock biodiesel with a methyl ester content of >80% mass through transesterification with only 3 and 6 moles of methanol. This study was performed to produce multi-feedstock biodiesel from a mixture of five raw materials, namely, palm oil, used cooking oil, soybean oil, canola oil, and sunflower oil, through transesterification with various moles of methanol and quality testing. The quality test consisted of determining the Density, kinematic viscosity, acid value, saponification value, total glycerol, methyl ester content, and yield and included Fourier transform infrared spectroscopy (FTIR) and gas chromatography-mass spectrometry (GCMS).

2. Methods

2.1. Materials

Used cooking oil, palm oil, soybean oil, canola oil, and sunflower oil were utilized in this study. Used cooking oil was purchased from a fried food seller. Palm oil, soybean oil, canola oil, and sunflower oil were obtained from local supermarkets. Methanol (99%; Merck), ethanol (95%; Merck), KOH (Merck), chloroform (99%; Merck), acetic acid glacial (Brightchem), sodium thiosulfate (Merck), potato starch solution, phenolphthalein (PP), hydrochloric acid (HCl; Merck), periodic acid (Merck), sodium hydroxide (NaOH; Merck), and potassium iodide (KI) were also used.

2.2. Characterization of Raw Materials

The Density, viscosity, and acid value of the raw materials were identified before transesterification. Raw materials with low acid values, i.e., not exceeding 2.0 mg-KOH/g, were ready for use in transesterification (Qiu et al., 2011).

2.3. Transesterification

In transesterification, used cooking oil, palm oil, soybean oil, canola oil, and sunflower oil (60 mL each) were mixed to obtain a total volume of 300 mL of raw materials. KOH (1% of the mass of the oil mixture [1% wt]) was used as a catalyst. Subsequently, transesterification was conducted at 60°C and a stirring speed of 300 rpm for one h. The mole ratios of oil to methanol were set to 1:3, 1:6, 1:9, 1:12, and 1:15. These variations of oil: methanol mole ratios were chosen from the previous works conducted by Al-dobouni et al. (2016); and Fadhil et al. (2017b). They found that the optimum value for the oil: methanol ratio will be in the range value of 1:3 to 1:15. After transesterification was completed, the remaining substances were placed in a separating funnel for 24 h to allow them to separate into two layers. The top layer was the multi-feedstock biodiesel, and the bottom layer was glycerol as a byproduct. Glycerol was separated and stored in bottles. Multifeedstock biodiesel was washed three times with distilled water at 60°C to remove residual methanol, soap, catalyst, and glycerol. Then, it was heated to 100°C for 20 min to remove any remaining distilled water. The final product obtained was pure multi-feedstock biodiesel. Figure 1 shows a flow chart of the transesterification process, and Figure 2 shows the experimental set-up and apparatus for the transesterification reaction of multi feedstock.





2.4. Testing Methods

2.4.1. Density

The mass of an empty 5 mL pycnometer was weighed. The multi-feedstock biodiesel sample was then preheated to 40°C and inserted into the 5 mL pycnometer. The filled pycnometer was weighed using a Pioneer Ohaus scale with an accuracy of 0.0001 g. The mass of the multi-feedstock biodiesel was obtained by subtracting the mass of the empty

pycnometer from the total mass of the filled pycnometer. The Density of the multi feedstock biodiesel sample was calculated using the following equation 1:

$$\rho = \frac{m}{\nu},\tag{1}$$

where ρ , *m*, and *v* are the density (g/mL), mass (g), and volume (5 mL) of biodiesel, respectively.

2.4.2. Kinematic Viscosity

Kinematic viscosity was tested using an NDJ-5S digital rotary viscometer in accordance with standard operating procedures. Before the kinematic viscosity test, 30 mL of multi-feedstock biodiesel was heated to 40°C. The viscometer was set at 60 rpm, and its rotor was allowed to rotate until the measured values were stable. The kinematic viscosities displayed on the viscometer were recorded.

2.4.3. Acid Value

The acid value was examined in accordance with the American Oil Chemists' Society (AOCS) Cd 3d-63 (AOCS, 2009a), which was ratified in the Indonesian National Standard (SNI) 01-3555 (BSN, 1998) and SNI 7182 (BSN, 2015).

2.4.4. Saponification Value

A saponification value test was conducted in accordance with AOCS (2009b) Cd 3-25, which were ratified in the SNI 01-3555 (BSN,1998) and SNI 7182 (BSN, 2015).

2.4.5. Total Glycerol

Total glycerol content was determined in accordance with AOCS (2011) Ca 14-56, which was ratified in the SNI 7182 (BSN, 2015).

2.4.6. Fatty Acid Methyl Esters and Yield

Fatty acid methyl esters (FAMEs) and yields were tested in accordance with SNI 7182 (BSN, 2015). FAMEs were calculated with the equation 2 (Hadiyanto et al., 2020).

$$FAME(\% - mass) = \frac{100(As - Aa - 18.27Gttl)}{As}$$
 (2)

where *As* is the saponification value (mg-KOH/g), *Aa* is the acid value (mg-KOH/g), and *Gttl* is the total glycerol (%-mass). The yield was calculated with the equation 3 (Hadiyanto et al., 2020).

$$Yield = \frac{Biodisel(g)}{Oil(g)} \times \% FAME.$$
 (3)

2.4.7. GCMS

GCMS is an analytical technique that combines the features of gas chromatography and mass spectrometry to identify substances contained in a test sample (Sparkman et al., 2011). In the present study, GCMS was performed to determine the percentage–the area of methyl ester compounds and their names found in multi-feedstock biodiesel.

2.4.8. FTIR

FTIR is a technique used to obtain the infrared spectrum of the absorption or emission of solids, liquids, or gases. An FTIR spectrometer simultaneously collects high-resolution spectral data over a wide spectrum range (Griffiths & de Hasseth, 2007). In the present study, FTIR was conducted to determine the methyl ester functional groups in multi-feedstock biodiesel.

3. Results and Discussion

3.1. Characteristics of Raw Materials

The characteristics of raw materials should be studied to determine whether transesterification alone or transesterification with esterification is required for multi feedstock biodiesel production. Table 1 shows the characteristics of the raw materials.

	Raw material samples							
Test parameters	Palm oil (A)	Used cooking oil (B)	Canola oil (C)	Soybean oil (D)	Sunflower oil (E)			
Density (kg/m ³)	912	904	902	900	914			
Kinematic viscosity (mm ² /s)	61.20	49.10	35.60	52.30	42.20			
Acid value (mg-KOH/g)	0.561	1.010	0.494	0.606	0.561			

Table 1 Characteristics of raw materials

Acid value, an important parameter, represents the amount of free fatty acids in raw materials (Issariyakul et al., 2008). The higher the acid value, the higher the free fatty acid content. The acid value of high-value raw materials should not exceed 2.0 mg-KOH/g (Qiu et al., 2011). However, the transesterification of raw materials with a high acid value is inefficient; most of them form soap, while some of them produce methyl esters (Barbosa et al., 2010). An acid value exceeding 2.0 mg-KOH/g should be reduced through esterification (Qiu et al., 2011). In Table 1, all five raw materials are below the acid value limit, so esterification is not required. Therefore, transesterification is sufficient for the efficient production of multi-feedstock biodiesel, producing methyl esters and forming a small amount of soap (Barbosa et al., 2010).

3.2. Characteristics of Multifeedstock Biodiesel

The characteristics of biodiesel were tested in terms of seven parameters (Table 2), namely, Density, kinematic viscosity, acid value, saponification value, total glycerol, methyl ester content, and yield. The characteristics of multi feedstock biodiesel were compared with the biodiesel quality standards of the American Society for Testing and Materials (ASTM) D 6751, Europäische Norm (EN) 14214 (ECN, 2002), and SNI 7182 (BSN, 2015).

Test parameters	Multifeedstock biodiesel sample					USA,	Europe,	Indonesia
	1:3	1:6	1:9	1:12	1:15	ASTM D 6751	EN 14214	SNI 7182
Density at 40°C (kg/m ³)	886.10	847.32	861.14	865.78	876.42	900	860-900	850-890
Kinematic viscosity at 40°C (mm²/s)	5.14	5.19	5.64	5.24	5.20	5.0	3.50-5.0	2.3-6.0
Acid value (mg-KOH/g)	0.27	0.36	0.45	0.31	0.36	0.50	0.50	Max 0.50
Saponification value (mg-KOH/g)	143.05	228.61	238.42	141.65	164.09	-	-	-
Total glycerol (% mass)	0.05	0.03	0.02	0.02	0.03	0.24	0.25	Max 0.24
Methyl ester (% mass)	99.12	99.58	99.68	99.52	99.44	-	96.5	Min 96.5
Yield (%)	81.96	92.99	83.39	85.24	82.75	-	-	-

Table 2 Characteristics of multi feedstock biodiesel

The standard parameter of methyl ester set by EN 14214 (ECN, 2002) and SNI 7182-2015 (BSN, 2015) is 96.5% mass. However, a standard methyl ester parameter has yet to be established in ASTM D 6751. At mole ratios of oil to methanol of 1:3, 1:6, 1:9, 1:12, and 1:15, methyl esters of 99.12% mass, 99.58% mass, 99.68% mass, 99.52% mass, and 99.44%

mass were produced, respectively. The methyl ester contents of the five multi-feedstock biodiesel samples exceeded the standard. With 3 moles of methanol per mole of oil, multi-feedstock biodiesel with an 81.96% yield could be produced at 1:3. Similarly, 6 moles of methanol per mole of oil could be used to obtain a 92.99% yield at 1:6. The biodiesel yield was suitable for industrial-scale application because it exceeded the 78% yield of Indonesian palm oil biodiesel plants (Soraya et al., 2014). Applying these findings on an industrial scale could provide benefits such as reduced use of methanol in the biodiesel production life cycle. As a result, production costs and environmental impact could be minimized as methanol causes considerable environmental pollution (Soraya et al., 2014; Siregar et al., 2015; Wahyono et al., 2020).

The Density of multi-feedstock biodiesel at various mole ratios of oil to methanol met the quality standards of the ASTM D 6751, EN 14214 (ECN, 2002), and SNI 7182-2015 (BSN, 2015). The kinematic viscosities at 1:3, 1:6, 1:9, 1:12, and 1:5 satisfied the SNI 7182-2015 (BSN, 2015) quality standard. The acid values at different ratios also fulfilled the quality standards of ASTM D 6751, EN 14214 (ECN, 2002), and SNI 7182-2015 (BSN, 2015). The total glycerol contents at various ratios complied with the quality standards of ASTM D 6751, EN 14214 (ECN, 2002), and SNI 7182-2015 (BSN, 2015). However, saponification value and yield standards are not specified in ASTM D 6751, EN 14214 (ECN, 2002), and SNI 7182-2015 (BSN, 2015). Multifeedstock biodiesel with a ratio of 1:6 had the highest yield (92.99%). More moles of methanol, such as at ratios 1:9, 1:12, and 1:15, led to low yields. This finding was consistent with those of Nurhayati et al. (2020). The reaction between triglycerides and alcohol to produce biodiesel or fatty acid alkyl esters (FAAE) involves three sequential reactions (Chongkhong et al., 2009):

$$Triglyceride + R'OH \rightleftharpoons Diglyceride + FAAE,$$
(4)

$$Diglyceride + R'OH \rightleftharpoons Monoglyceride + FAAE,$$
(5)

Monoglyceride + R'OH
$$\rightleftharpoons$$
 Glycerol + FAAE. (6)

A high mole ratio of oil to methanol can cause an equilibrium reaction to reverse and recombine with a methyl ester to form monoglycerides, which decrease the biodiesel yield (Fadhil & Abdulahad, 2014; Al-dobouni et al., 2016; Fadhil et al., 2017b). Therefore, 1:6 was determined as the optimal ratio, which was supported by Al-dobouni et al. (2016) and Fadhil et al. (2017b). Multifeedstock biodiesel at 1:3 and 1:6 had yields of 81.96% and 92.99%, respectively. Therefore, they should be considered as alternatives to limit the use of methanol in biodiesel production. These yields are greater than those reported by Fadhil et al. (2017a), who produced 76% and 92% biodiesel from an equivalent blend of castor seed oil and waste fish oil at 1:3 and 1:6, respectively.

3.3. GCMS Test Results

GCMS aimed to determine the percentage–area and names of methyl esters in multifeedstock biodiesel. Figure 3 shows the profile of the chemical compounds in multifeedstock biodiesel.



Figure 3 Profile of chemical compounds in multi-feedstock biodiesel

The GCMS results (Figure 3) showed that multi-feedstock biodiesel with ratios of 1:3, 1:6, 1:9, 1:12, and 1:15 has an increase of high methyl ester contents by 98.42% area, 99.46% area, 99.76% area, 99.87% area, 100% area, respectively. The higher the moles of methanol, the higher the content of methyl ester in biodiesel (Musa, 2016). This finding is consistent with Encinar et al. (2007) and related to stoichiometry during transesterification for biofuel production, which is presented as follows.



In this reaction, 1 mole of triglycerides reacts with 3 moles of methanol to produce 3 moles of methyl ester and 1 mole of glycerol. Therefore, transesterification requires at least 3 moles of methanol. Transesterification stoichiometry was the basis of the threefold variation in the moles of methanol in this study, namely, 3, 6, 9, 12, and 15. Excess methanol is needed to break the glycerin–fatty acid relationship during the transesterification of triglycerides into biodiesel (Miao & Wu, 2006). Therefore, a high mole ratio of alcohol and oil increases the conversion of large alkyl esters in a short period (Helwani et al., 2009). Furthermore, the purity of biodiesel can be increased by increasing the amount of alcohol in oil. This observation is consistent with that of Eevera et al. (2009), who studied the transesterification of pure vegetable oil.

The methyl esters with the highest percentages in multi-feedstock biodiesel were 9,12octadecadienoic acid (Z,Z)-methyl ester (54.77% area, 57.25% area, 58.03% area, 57.69% area, and 59.64% area) hexadecanoic acid methyl ester (18.95% area, 19.02% area, 19.67% area, 19.06% area, and 19.33% area), and 9-octadecenoic acid (Z)-methyl ester (15.48% area, 14.13% area, 12.85% area, 13.30% area, and 11.43% area). 9,12-Octadecadienoic acid (Z, Z)-methyl ester, hexadecanoic acid methyl ester, and 9-octadecenoic acid (Z)-methyl ester is types of linoleic acid methyl ester, palmitic acid methyl ester, and oleic acid methyl ester, respectively. Similarly, Issariyakul et al. (2008) and Hadiyanto et al. (2020) produced the largest percentage of methyl ester that consisted of these three types of FAMEs. The chemical formula of Hexadecenoic acid methyl ester, 9,12-Octadecadienoic acid (Z,Z)methyl ester, and 9-Octadecenoic acid (Z)-methyl ester, are shown below.



3.4. FTIR Test Results

FTIR aimed to determine the methyl ester functional groups in multi-feedstock biodiesel. Figure 4 shows the FTIR spectrum of multi-feedstock biodiesel.



Figure 4 FTIR spectrum of multi-feedstock biodiesel under different oil: ethanol mole ratio

In Figure 4, the FTIR spectra of the optimal methyl esters in multi-feedstock biodiesel with ratios 1:3, 1:6, 1:9, 1:12, and 1:15 showed some characteristic bands. The absorption band at 3008–2854 cm⁻¹ refers to asymmetric and symmetric CH₃ stretching vibrations (– $CO-O-CH_3$). The characteristic absorption band at 1742 cm⁻¹ corresponds to the ester carbonyl group (C=O). The stretching mode characteristics of olefins denoted by (C=C) appear at 1655 cm⁻¹. The band at 1460–1438 cm⁻¹ is due to the (–C–H) bending in alkane, whereas 1195–1016 cm⁻¹ is attributed to the stretching vibration of (–C–O–) ester groups. This finding is consistent with Fadhil and Abdulahad (2014) and Al-dobouni et al. (2016). Al-dobouni et al. (2016) and Fadhil et al. (2017b) provided evidence supporting the conversion of oil to its corresponding esters; in particular, the area under the peak of each of C=O band, stretching C–H band, and C–H bonding band is smaller in methyl esters than in oil. This difference is attributed to the substitution of glycerol with a methoxy radical (Fadhil, 2013a; Fadhil, 2013b). The chemical formula of methyl esters is as follows:

$$H_{3}C \longrightarrow O \\ C \longrightarrow R_{1}.$$
(11)
Methyl ester

A methyl ester is composed of functional groups -C=0, $-CH_3$, and C-O-C. Further details on the profile of methyl ester compounds in multi-feedstock biodiesel are presented in chemical formulas (8), (9), and (10). Our results indicate that transesterification is a successful means to alter the fuel properties of oil mixtures and convert them into more valuable fuel.

4. Conclusions

This study successfully produced multi-feedstock biodiesel from a mixture of five oils suitable for industrial-scale applications. All the testing parameters of methyl ester content, Density, acid value, and total glycerol in the multi-feedstock biodiesel met the quality standards of ASTM D 6751, EN 14214, and SNI 7182-2015. The multi-feedstock biodiesel of all the moles of methanol variations contained >98% mass of methyl ester. This study also found that 3 moles of methanol per mole of oil could be used to produce multifeedstock biodiesel with a methyl ester content of 99.12% mass and 81.96% yield at a 1:3 ratio. Similarly, 6 moles of methanol per mole of oil could be utilized to obtain multifeedstock biodiesel with a methyl ester content of 99.58% mass and 92.99% yield at a 1:6 ratio. Multifeedstock biodiesel at 1:3 and 1:6 could be an alternative that should be considered to reduce methanol use for industrial-scale biodiesel production. The ratio of 1:6 corresponded to the highest yield. GCMS results demonstrated that all mole variations had methyl ester percentages that exceeded 98% area. The methyl ester with the highest percentage in multifeedstock biodiesel was 9,12-octadecadienoic acid (Z,Z)-methyl ester. FTIR results revealed peaks that indicated the presence of a methyl ester functional group and its long-chain (-R). Therefore, this study produced multi-feedstock biodiesel that showed potential for industrial-scale applications. Future works should be done on the kinetics of the multi-feedstock biodiesel production. Actually, each oil has a different reaction time, and therefore the yield and conversion of biodiesel will be determined by each reaction of the oil. By evaluating each kinetic of oil, we will be able to decide on which oil is the most determinant in the process. Moreover, the stability of biodiesel will be affected by the storage period. Therefore, the stability test is also crucial for future studies.

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