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SYNTHESIS OF RESIDUAL OIL FROM SPENT BLEACHING EARTH (SBE) INTO BIODIESEL USING MICROWAVE REACTOR

SINTESIS RESIDU OIL DARI SPENT BLEACHING EARTH (SBE) MENJADI BIODIESEL MENGGUNAKAN MICROWAVE REACTOR

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ABSTRAK

Spent bleaching earth (SBE) merupakan limbah dari proses pemurnian CPO yang mengandung kadar minyak tinggi, sekitar 20-30%. Terdapat beberapa metode untuk menurunkan kadar minyak SBE, salah satunya metode ekstraksi pelarut. SBE residual oil (R-oil) dapat dimanfaatkan sebagai bahan baku biodiesel. Penelitian ini bertujuan untuk mengetahui perbandingan massa SBE, pelarut, dan suhu yang optimal dalam mengekstrak R-oil, serta potensi SBE R-oil untuk dijadikan biodiesel dengan penggunaan microwave reactor. Ekstraksi R-oil dilakukan pada variasi perbandingan massa SBE dan pelarut 1:2, 1:4, dan 1:6 pada varisi suhu 26, 40, dan 50°C. R-oil yang telah ditreatment kemudian diesterifikasi dan dilanjutkan ke reaksi transesterifikasi dengan metanol 15% dan KOH 1% selama 10 menit pada suhu 60°C menggunakan microwave reactor. Hasil ekstraksi R-oil paling optimal ada pada perbandingan 1:4 suhu 26°C dengan yield 19,12% dan terdapat perbesaran luas permukaan pada deoiled-SBE. Nilai rendemen dari R-oil SBE yang dikonversi menjadi biodiesel adalah sebesar 33,53% dengan nilai konversi 45,28% dan kadar FAME 92,97%. Karakterisitik biodiesel yang dihasilkan memenuhi SNI Biodiesel dengan densitas biodiesel sebesar 0,8674 g/cm^3 dan viskositas biodiesel sebesar 5,69 mm^2/s.

Kata kunci: asam lemak, ekstraksi, transesterifikasi, yield

ABSTRACT

Spent bleaching earth (SBE) is a waste from the crude palm oil CPO refining process which contains high oil content, around 20-30%. There are several methods to reduce SBE oil content, one of which is the solvent extraction method. SBE residual oil can be utilized as a raw material for biodiesel. This research aimed to determine the mass ratio of SBE and solvent, the optimal temperature for extracting residual oil from SBE, and the potential of residual oil from SBE to be used as biodiesel using a microwave reactor. Residual oil extraction was carried out at varying mass ratios of SBE and solvent (1:2, 1:4, and 1:6) and varying temperatures (26, 40, and 50°C). The treated residual oil was esterified and subjected to transesterification reaction with 15% methanol and 1% KOH for 10 minutes at 60°C using a microwave reactor. The optimal extraction result for residual oil was at a 1:4 ratio and 26°C with a yield of 19.12%. There was also an increase in surface area in deoiled-SBE. The conversion of residual oil from SBE into biodiesel yielded 33.53% with a conversion value of 45.28% and a FAME content of 92.97%. The characteristics of the produced biodiesel meet the Indonesian National Standard (SNI) for biodiesel with density of 0.8674 g/cm^3 and viscosity of 5.69 mm^2/s.

Keywords: extraction, fatty acid, transesterification, yield

INTRODUCTION

Indonesia's crude palm oil (CPO) production in 2023 reached 50.07 million tons (GAPKI, 2024). In the palm oil industry, there are several processess for refining CPO, including the bleaching process using bleaching earth (Hasballah and Siregar, 2020). The use of bleaching earth is aimed at removing substances such as color pigments (α and β-*carotenes*), phospholipids, fatty acids, oxidizing compounds, heavy metals, and residual latex from the oil (Arpornpong *et al*., 2018). One of the waste in CPO refining process using bleaching earth is called spent bleaching earth (SBE). SBE contains high oil content, around 20-30% (Musa *et al.*, 2018).

Based on the Indonesian Government Regulation number 22 of 2021, SBE is categorized as non-hazardous waste if its oil content was less than or equal to 3%. There are several processess used to reduce the oil content in SBE, with one of the most commonly used methods being solvent extraction, as it is relatively inexpensive and effective *(Paunovic et al.*, 2014). The result of SBE extraction is commonly referred to SBE residual oil (R-oil), which has been utilized for various industrial application such as biofuel feedstock, bio-lubricants, animal feed, and fertilizers (Arpornpong e*t al.*, 2018).

**Coressponding Author*

Biodiesel is one type of biofuel made from vegetable oil, animal fat, or other substances containing triglycerides. It is formed through a transesterification reaction, which involves the reaction between an alcohol and a triglyceride to produce alkyl esters and glycerol. The transesterification process is used to extract glycerin from the oil and convert free fatty acids with alcohol into methyl ester or biodiesel. In biodiesel production, the typical method involves direct heating using a hot plate or oil bath to react triglycerides with alcohol (Leung *et al.*, 2010). Another method to produce biodiesel is using a microwave reactor. The reactor utilizes electromagnetic microwaves to emit radiation directly at the molecular level, enabling heat to distribute more evenly and yield higher results (Rahkadima and Yulia, 2019). The efficiency in transesterification reactions using microwaves comes from the dielectric properties of polar mixtures and ionic components in oil, solvent, and catalyst. The rapid and efficient heating through microwaves radiation is caused by the microwaves interaction with the sample at the molecular level, creating intermolecular mixtures and agitation that enhance the chances for alcohol and oil molecule interactions (Terigar *et al.*, 2010).

The previous research on biodiesel production from SBE was conducted by Sugiharto *et al.* (2019), who achieved a yield of 21.45% (w/w, biodiesel/SBE) with a reaction time of 2.32 hours, while research by (Suryani *et al.*, 2017) achieved a yield of 84.5% (w/w, biodiesel/SBE) with a reaction time of 90 minutes. Both studies utilized conventional heating methods with two-stage reactions in their biodiesel production. Dimawarnita *et al.* (2023) used a microwave reactor to shorten the reaction time in just 10 minutes. Therefore, this research integrates the process of extracting SBE R-oil and utilizing it for biodiesel using a microwave reactor to shorten the biodiesel production time.

MATERIALS AND METHODS

Materials

The materials used in this research are SBE, technical acetone, H_2SO_4 (Merck No. 1.00371.2500), methanol (Merck No. 1.06009.2500) and KOH (Merck No. 1.05033.1000). The equipment used includes a digital balance, hot plate, Erlenmeyer flask, magnetic stirrer, centrifuge, Falcon tube, condenser, microwave reactor, separatory funnel, temperature controller, rotary evaporator, thermocouple, and three-necked flask.

Spent Bleaching Earth Residual Oil extraction (SBE Deoilization)

The extraction process of SBE was conducted based on the method by Low *et al.*, 2022 with modifications. This extraction process can also be said the deoilization of SBE. SBE was extracted using acetone as the solvent with variations at solvent ratios of 1:2, 1:4, and 1:6 (w/w) and temperatures of 26°C, 40°C, and 50°C. A total of 100 g of SBE and 200 g of acetone were macerated at 26°C and stirred using a magnetic stirrer at 250 rpm for 1 hour. The resulting extraction solution underwent centrifugation for 5 minutes at 5000 rpm to separate the solution from the supernatant. The filtrate from centrifugation, which contains acetone and SBE R-oil, was then separated using a rotary evaporator at 56°C and 50 rpm. The maceration process was also conducted for each solvent ratio and temperature variation. The schematic of the SBE R-oil extraction process can be seen in Figure 1. Extraction process yield two outputs: SBE residual oil (R-oil) and deoiled SBE (dSBE).

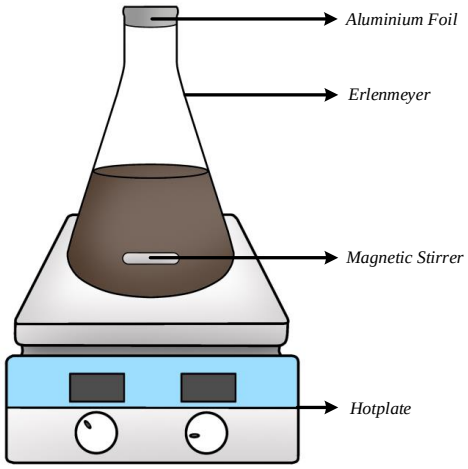


Figure 1. SBE R-oil extraction equipment setup

SBE R-oil Pretreatment Followed by Esterification Process

SBE R-oil was centrifuged for 5 minutes at 8000 rpm to separate impurities that were carried over from the previous process. The centrifuged SBE R-oil then underwent esterification which was carried out based on the method by Dimawarnita *et al.*, 2023 with modifications. In this step, 100 mL of SBE R-oil was reacted with methanol at a ratio of 225% v/v and H_2SO_4 at 5% v/v. 225 mL of methanol was mixed with 5 mL of H_2SO_4 until they fully dissolved. The SBE R-oil was heated in a three-necked flask to a temperature of 60°C using a microwave reactor. The methanol-H_2SO_4 mixture was added to the three-necked flask. The esterification reaction was carried out for 10 minutes using a microwave reactor with a power of 200-300W and stirred using a magnetic stirrer.

The solution from the esterification reaction was transferred to a 1000 ml separatory funnel and allowed to stand for approximately 24 hours until two layers was formed. The upper layer consisted of methanol and water, while the lower layer contained methyl esters from the esterification, H_2SO_4, and SBE R-oil with a low FFA content. The lower layer was

then washed with hot water at 60°C until the pH became neutral. The esterified SBE R-oil was then oven-dried for 2 hours to reduce its water content.

SBE R-oil Transesterification

The transesterification of SBE R-oil was conducted based on the method by Dimawarnita *et al.*, 2023 with modifications. 15% v/v methanol and 1% (w/v) KOH were used. 15 mL of methanol was mixed with 1 g of KOH until it fully dissolved. 100 mL of esterified SBE R-oil was heated to 60°C in a three-necked flask using a microwave reactor followed by the addition of methanol-KOH mixture. The transesterification reaction was conducted in a microwave reactor at a power of 200-300W and stirred with a magnetic stirrer for 10 minutes.

The solution was then transferred to a separation flask and allowed to stand for approximately 24 hours until two layers was formed. The upper layer consisted of fatty acid methyl esters (FAME), remaining FFA, and KOH; glycerol was amidst the lower layer. The upper layer was then washed with deionized water at 60°C until FAME layer was clean and the washed water became clear. The FAME from the reaction was then heated in an oven at 105°C for approximately 2 hours. The equipment setup for producing SBE R-oil into biodiesel using a microwave reactor can be seen in Figure 2.

SBE R-oil Yield Calculation

The yield of SBE R-oil from the extraction process is calculated to determine the amount of SBE R-oil produced. The yield calculation of SBE R-oil is calculated using the following equation (Adiandasari *et al.*, 2021) :

$$Residual\ Oil\ SBE\ Yield = \frac{Residual\ oil\ mass}{SBE\ mass} \times 100\%$$

SBE R-oil Characterization

The characterization of SBE R-oil is conducted through several tests, including density testing at 40°C using the ASTM D 4052-18a method, kinematic viscosity testing at 40°C using the ASTM D 445-21 method, water content testing using the ASTM D 6304-20 method, and free fatty acid content testing using titration method.

Deoiled SBE Characterization

SBE which its oil has been extracted is called deoiled SBE (dSBE). The characterization of dSBE is conducted through several tests, including the Brunauer-Emmet-Teller (BET) test and Scanning Electron Microscopy (SEM). The BET test is conducted to determine the pore size and surface area of de-oiled SBE. The SEM test is conducted to observe the morphological structure on the surface of de-oiled SBE.

Biodiesel Yield Calculation

Biodiesel yield was calculated quantitatively to determine the amount of biodiesel produced from the SBE R-oil used using the following equation (Singh *et al.*, 2019) :

$$Yield\ Biodiesel = \frac{Volume\ biodiesel}{Volume\ residual\ oil} \times 100\%$$

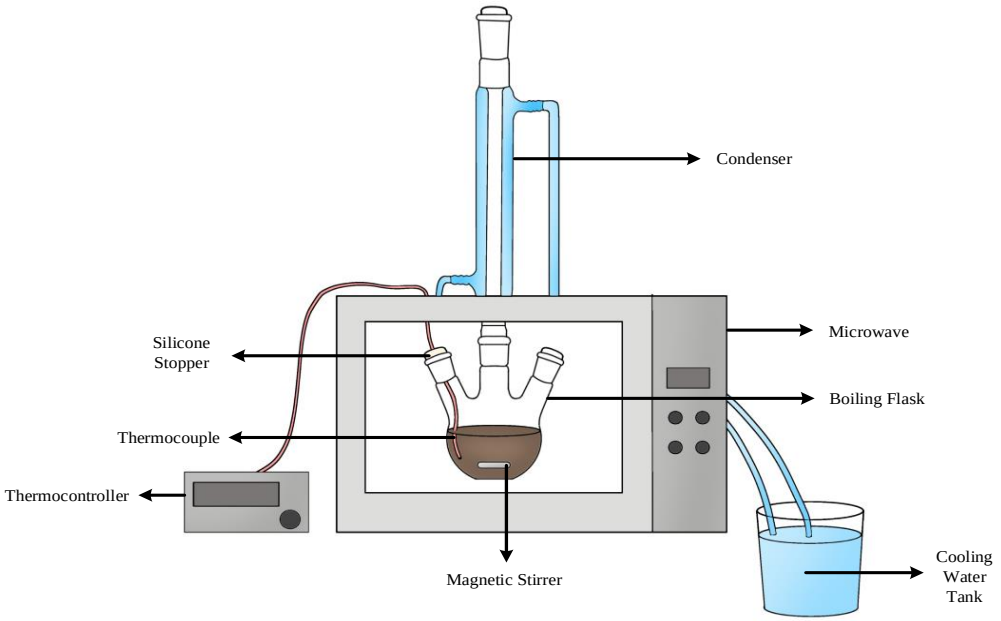


Figure 2. Microwave reactor equipment setup

Biodiesel Conversion Calculation

The conversion in biodiesel production was calculated to determine the amount of triglyceride compounds converted into FAME. The conversion calculation is based on the percentage of FAME obtained from GC-MS analysis. The biodiesel conversion can be calculated using the following equation (Singh *et al.*, 2019) :

$$Conversion = \frac{Moles\ of\ FAME}{3 \times Moles\ of\ Triglyceride} \times 100\%$$

Biodiesel Characterization

The characterization of biodiesel is conducted through several tests, including FAME content testing using GC-MS, density testing at 40°C using a DMA 4500M density meter, and kinematic viscosity testing at 40°C using a DV3T Brookfield viscometer method.

RESULTS AND DISCUSSION

R-oil Yield from Spent Bleaching Earth in Relation to Solvent Ratio and Extraction Temperature

In this study, SBE was extracted with acetone solvent using a maceration method and stirring it by a magnetic stirrer. In extraction process, the amount of solvent is a critical factor for optimal extraction performance because the more solvent used, the greater the amount of compounds that will dissolve. Therefore, three variations of the mass ratio of SBE to solvent were conducted to observe their effect on the yield percentage (%yield) of residual oil. The influence of the SBE to solvent mass ratio on the yield percentage of residual oil can be seen in Figure 3.

Figure 3 shows the extraction results of R-oil from SBE with varying solvent ratios and extraction temperatures. An increase in %yield is observed between the 1:2 and 1:4 solvent ratios. This increase is attributed to the greater amount of solvent used in the extraction process, which enhances the extraction of dissolved substances. Acetone, being a semi-polar solvent, can attract polar, semi-polar, and non-polar compounds, allowing for the maximum extraction of all compounds in SBE, including oil (Low *et al.*, 2022). In this extraction comparison, the optimal results were obtained with a mass ratio of SBE to solvent of 1:4. This finding aligns with the research by Low *et al.*, 2022, which varied the types of solvents and extraction methods on SBE. The solid-liquid extraction process, known as leaching, involves the diffusion of oil into the liquid phase, which is the acetone solvent, until equilibrium is reached. Once equilibrium is achieved, the oil in the SBE can no longer diffuse into the solvent. The advantages of using acetone as a solvent include its renewability compared to hexane and its reusability. (Garcia *et al.*, 2024).

However, this differs in the comparison between the 1:4 and 1:6 ratios, where a decrease in % yield is observed. This is likely due to the extraction equilibrium being reached at the 1:4 ratio. Consequently, at the 1:6 ratio, the extraction process is not optimal because the excessive amount of solvent does not enhance the yield further. Instead, it may lead to inefficient use of the solvent, as the equilibrium state prevents additional oil from diffusing into the solvent. According to Pratama *et al.*, 2019, the sample-to-solvent ratio significantly affects extraction efficiency. There is an optimal amount of solvent where the extraction works most efficiently; however, an excessive amount of solvent does not necessarily result in extracting more solute. This finding is supported by Yuliani *et al.*, 2018 who stated that solid-liquid extraction is heavily influenced by the extraction equilibrium, including both the volume of solvent and the amount of substance being extracted.

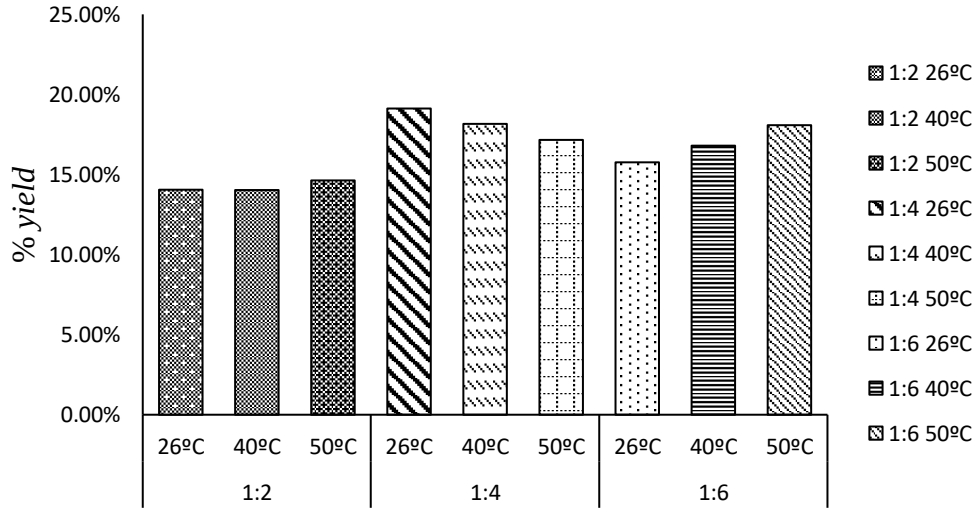


Figure 3. SBE R-oil extraction yield

Based on the data (Figure 3), the highest %yield is observed at a 1:4 ratio with an extraction temperature of 26°C, yielding 19.12%. The effect of different extraction temperatures at the 1:2 ratio does not result in significant changes. However, as the extraction temperature increases to 40°C and 50°C at the 1:4 ratio, there is a decrease in %yield, while at the 1:6 ratio, there is an increase in %yield. This is because the extraction temperatures of 40°C and 50°C are close to the boiling point of acetone (56°C), whereas the boiling point of oil is higher at 149°C. As a result, the solvent evaporates before effectively extracting the oil. (Low *et al.*, 2022; Wu *et al.*, 2022).

Moreover, high temperatures can affect the solubility of substances in the sample, through reduction of the extracts selectivity and may cause the decomposition of the extracted substances, which then can interfere with the extraction result (Abdullah *et al*., 2017). At the 1:4 ratio, using a temperature of 26°C is sufficient to achieve the highest %yield. Increasing the temperature at this ratio reduces the amount of acetone in contact with the SBE, leading to a decrease in %yield. Conversely, at the 1:6 ratio, increasing the temperature results in a higher yield. This is because the higher temperature reduces the amount of acetone to a level closer to the optimal amount, matching the extraction equilibrium point and thus yielding a higher %yield.

Physico-Chemical Characteristics of SBE R-Oil

In this study, R-oil from SBE is utilized as a raw material for biodiesel production. Therefore, several physico-chemical characterization tests were conducted on the R-oil. The results of these characterizations are presented in Table 1.

Table 1. Characteristics of SBE R-oil

No.	Characteristics	Test Result	SNI 7182:2015
1	Density at 40ºC	907.3 kg/m^3	850-890 kg/m^3
2	Kinematic Viscosity at 40ºC	39.318 mm^2/s	2.3-6 mm^2/s
3	Water Content	2500 ppm	Maximal 400 ppm
4	Free Fatty Acid	11.4%	Maximal 1 %

Based on Table 1, the characterization results of the R-oil from SBE were compared with the SNI Biodiesel standards. According to the SNI 7182:2015 on biodiesel quality requirements, the density, viscosity, water content, and free fatty acid content of the residual oil do not meet the standards. The high density value could be due to impurities in the residual oil, such as mono- and diglycerides, free fatty acids, phosphatides, and metals (Arpornpong *et al.*, 2018). Meanwhile, the high viscosity value may result from the presence of stearin fractions and high levels of saturated fatty acids in the residual oil (Muslich *et al.*, 2020). After esterification, there was a decrease in free fatty acid content to 0.97%, indicating that the esterification process successfully reduced contaminants that impact viscosity and density. However, to meet the SNI Biodiesel standards, further processing or purification of the residual oil may be necessary before the transesterification process into biodiesel.

The Fatty Acid Content of SBE R-oil

The fatty acid content of SBE R-oil was tested after esterification, and the total fatty acid content was found to be 70.17%, with saturated fatty acids accounting for 32.56% and unsaturated fatty acids for 38.09%. The results of the fatty acid content of SBE R-oil are shown in Table 2.

Table 2. Fatty acid content on SBE R-oil

No	Compound		After esterification
	Saturated Fatty Acid		**%**
1	Caprylic acid	C8:0	0.01
2	Capric acid	C10:0	0.02
3	Lauric acid	C12:0	0.16
4	Myristic acid	C14:0	0.87
5	Palmitic acid	C16:0	28.19
6	Stearic acid	C18:0	3.31
	Unsaturated Fatty Acid		
5	Oleic acid	C18:1	30.13
6	Linoleic acid	C18:2	7.25
7	Linolenic acid	C18:3	0.53
	Total		70.17

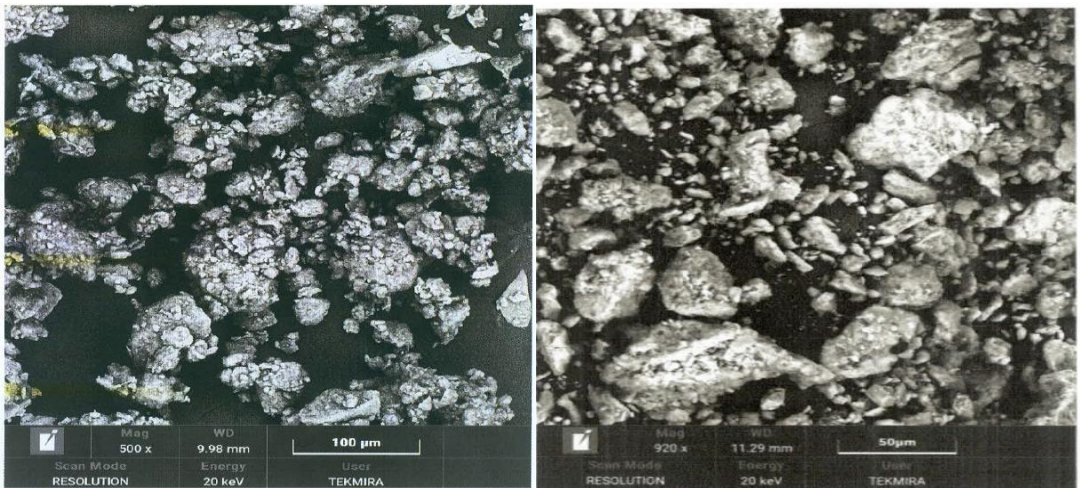
Characterization Results of SBE on BET and SEM Before and After Deoilization

Based on the BET analysis (Table 3), it can be seen that the surface area value of deoiled SBE (dSBE) in this study (40.654 m²/g) is larger compared to the surface area value of SBE (32.980 m²/g). This increase in surface area value is attributed to the use of solvents during the SBE R-oil extraction (deoilization) process. It is possible because during the extraction process of SBE R-oil, the oil is extracted, leaving the pore diameter initially filled with oil empty and thus increasing the surface area value.

Table 3. BET Analysis of SBE and dSBE

Keterangan	dSBE	SBE
Surface Area (m^2/g)	40.654	32.980
Total pore volume (cc/g)	0.084	-
Average pore diameter (Å)	45.01	-

The SEM results of SBE and dSBE are shown in Figure 4. The SEM results of SBE (a) magnified at 500x and the SEM results of dSBE (b) magnified at 920x appear to have similar surface morphology.



(a) (b)

Figure 4. SEM results of SBE (a) and dSBE (b)

This indicates that a higher magnification is needed for dSBE to observe surface morphology that is almost identical to SBE, suggesting the presence of more cavities on the surface of dSBE. The increased number of cavities on the surface of SBE after deoiling is due to the oil that previously covered the surface/cavities of SBE being extracted *(Saputro et al.,* 2020). The abundance of cavities on the surface of dSBE is supported by the BET test data, where dSBE has a larger surface area than SBE.

Biodiesel Production from SBE R-oil

In this study, biodiesel was produced through transesterification reaction using a ratio of residual oil to methanol of 1:4.7, operated at a temperature of 60°C for 10 minutes using a microwave reactor and 1% KOH catalyst. Based on calculations, the biodiesel yield obtained in this study was 33.53% (v/v, biodiesel/R-Oil), and the amount of triglycerides converted to fatty acid methyl ester (FAME) was 45.28%. The biodiesel was subjected to several characteristic tests, which were chosen based on Laksono and Oktavia (2021) study, and compared the characteristics to SNI Biodiesel standards (SNI 7182:2015) as shown in Table 4.

Table 4. Biodiesel characteristics

No	Characteristic	Unit	Test result	SNI 7182:2015
1	FAME Content	%	92.97	min. 96.5
2	Density at 40ºC	g/cm^3	0.8674	0.85-0.89
3	Kinematic Viscosity at 40ºC	mm^2/s	5.69	2.3-6

Based on the results, SBE R-oil shows potential to be produced as biodiesel using a microwave reactor. The use of a microwave reactor results in high FAME yield in a short amount of time. This occurs due to an increase in intermolecular collisions leading to a decrease in activation energy. A low activation energy of a reaction can increase the reaction rate, allowing the reaction to proceed faster (Poerwadi *et al*., 2019). Based on GC-MS results, the produced biodiesel contains three major FAME compounds: 9-octadecenoic acid methyl ester (Oleic acid methyl ester, $C_{19}H_{36}O_2$) at 48.84%, Hexadecanoic acid methyl ester (Palmitic acid methyl ester, $C_{17}H_{34}O$) at 26.78%, and Octadecanoic acid methyl ester (Linoleic acid methyl ester, $C_{19}H_{34}O_2$) at 7.91%.

The reaction time needed in this study was 20 minutes resulting from 10 minutes for each esterification and transesterification process. Compared to Sugiharto et al (2019) and Suryani *et al.*, (2017), whose reaction time were 2.32 hours and 90 minutes, respectively, reaction time in this study is faster than those of the two. However, the biodiesel yield in this study was lower.

The results in Table 4 show that the density and viscosity values of the produced biodiesel conform with SNI Biodiesel standards, although they are close to the upper limits of the standard. This is because the density and viscosity values are influenced by the composition of FAME in biodiesel (Jekayinfa *et al.,* 2019). According to Pratas *et al.*, 2010, the density values of FAME compounds at 40°C for 9-octadecenoic acid methyl ester, hexadecanoic acid methyl ester and octadecanoic acid methyl ester are 0.8595 g/cm^3, 0.8508 g/cm^3, and 0.8498 g/cm^3, respectively. Similarly, the viscosity values of FAME composition at 40°C for 9-octadecenoic acid methyl ester, hexadecanoic acid methyl ester, and octadecanoic acid methyl ester are 3.9303 mm^2/s, 3.7551 mm^2/s, and 4.9862 mm^2/s. Therefore, the high density and viscosity values of the FAME compounds can also increase the density and viscosity values of biodiesel.

CONCLUSIONS AND RECOMENDATIONS

Conclusions

The extraction of SBE R-oil with the highest %yield is demonstrated using a solvent ratio of 1:4 at 26°C, resulting in a yield of 19.12% and there is an increase in surface area in dSBE. SBE R-oil has the potential to be converted into biodiesel using a microwave reactor, producing biodiesel with yield of 33.53%, density of 0.8674 g/cm^3, and viscosity of 5.69 mm^2/s that conform with SNI Biodiesel standards. The reaction time of R-oil into biodiesel was 20 minutes. The FAME content of the produced biodiesel is 92.97% with the largest FAME content being 9-octadecenoic acid methyl ester (Oleic acid methyl ester, $C_{19}H_{36}O_2$) at 48.84%, hexadecanoic acid methyl ester (Palmitic acid methyl ester, $C_{17}H_{34}O$) at 26.78%, and Octadecanoic acid methyl ester (Linoleic acid methyl ester, $C_{19}H_{34}O_2$) at 7.91%.

Recommendations

The extraction method using acetone in further study should require the use of a condenser to prevent solvent evaporation during the extraction process that might interfere to biodiesel production. Further investigation is needed regarding the use of methanol ratio, catalyst, temperature, and reaction time variation to produce biodiesel from acetone-extracted SBE R-oil.

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