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Synthesis of Biodiesel from Kapok (*Ceiba pentandra L.*) Seed Oil through Ultrasound-Enhanced Transesterification Reaction

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Abstract. Biodiesel is among the prospective renewable energy due to its superior properties as fuel such as environment friendly, non-toxic, biodegradable, resulting low pollutant emission, having high energy value and high cetane number, as well as can be readily applied in diesel machine. Biodiesel is also among the priority in the Indonesian national blue print on new and renewable energy development 2005-2025. Currently, Biodiesel is commonly produced through alkaline-catalyzed transesterification of vegetable oil which is conducted in conventional batch reactor. However, this process shows several drawbacks for instance the low reaction conversion. To overcome this challenge, process intensification of biodiesel production using ultrasound-enhanced transesterification process in batch reactor was employed in this work. The feedstock used for biodiesel synthesis in this work was kapok (*Ceiba pentandra L.*) seed oil. The oil contains gum and high free fatty acid (FFA) as much as 8.89%. Therefore, prior to the transesterification reaction, two steps of pretreatment processes i.e. degumming process for gum removal and esterification for reducing the FFA content until it reaches the value below 2%. The main transesterification reaction was then performed by using ultrasound enhanced batch reactor which was operated at the frequency of 20 Hz and temperature of 60°C in the presence of KOH catalyst. The reaction time was fixed 60 minutes. On the other hand, the molar ratio of methanol to oil was varied at 1:4, 1:5 and 1:6, and the catalyst concentration was studied at 0.5, 1, and 1.5% w cat/ w oil. It was found that highest yield was obtained at the reaction conducted using 0.5% catalyst and the molar ratio of 1.6 with the reaction conversion of 99.82%. This result was higher than the yield resulted by the conventional process performed at the similar condition which revealed the yield of 90%. Biodiesel produced using ultrasound-enhanced reactor fulfilled the SNI standard of properties in term of viscosity and density.

INTRODUCTION

The application of biodiesel as alternative energy resources in diesel machine has gained an increasing interest recently. Biodiesel is a renewable energy derived from animal fats and vegetable oils which is biodegradable and environmentally beneficial [1]. Even biodiesel is considered prospective to be applied as a sustainable fuel for motorsport [2].

Biodiesel is commonly synthesized through the transesterification reaction of vegetable oils or animal fats in the presence of base catalyst. Various types of vegetable oils have been utilized as feedstocks of biodiesel at commercial scale, such as crude palm oil and soybean oil. However, the consumption of edible oils as fuel raw materials will cause the competition between the fuel and food demand. Therefore, the development of biodiesel production using

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non-edible oils is preferred. Among the potential non-edible oil in Indonesia is kapok seed (*Ceiba pentandra L.*) oil. Kapok seed contains 25-40% oil and 71.95% unsaturated fatty acid [3]. The exceptionally high unsaturated fatty acid content causes the rancid smell of the vegetable oil and thus it is not suitable to be consumed as edible oil [4]. Therefore, utilization of kapok seed oil as biodiesel feedstock will provide added value to this Indonesian local vegetable oil.

Conventional process of transesterification of vegetable oil to produce biodiesel encounters a challenge since it is a reversible reaction which is usually slow and results in low biodiesel yield. Hence, process intensification is urgent to improve the process efficiency and to result in higher biodiesel yield. To overwhelm this limitation, a process intensification of biodiesel production using ultrasonic technology was developed in this work. Acoustic cavitation generated by the ultrasonic transducer can potentially accelerate the transesterification reactions [5].

An ultrasound enhanced batch reactor was utilized for producing biodiesel from kapok seed oil in this work. To date, the application of ultrasonic reactor for producing biodiesel from kapok seed oil has never been found in the literature. Thus, this work is expected to contribute on the providing added value to kapok seed oil as biodiesel feedstock through the application of ultrasonic reactor, which can generate high biodiesel yield in an efficient way. The effects of main parameters on the biodiesel yield and the biodiesel properties were investigated in this research.

MATERIALS AND METHODS

Materials

The feedstock used in this work was kapok seed oil which was obtained from Pati, Central Java, Indonesia and methanol (techical grade with the density of 0.792 g/mL). The other materials were phenol phthalein indicator, oxalic acid (p.a.), potassium hydroxide and sulfuric acid (H_2SO_4) which were from Merck (Darmstadt, Germany).

Methods

Synthesis of biodiesel from kapok seed oil consisted of several steps, i.e.: feed-stocks characterization, degumming process, ultrasound-enhanced esterification reaction, ultrasound-enhanced transesterification reaction, purification and biodiesel characterization. Feedstock characterization included the analysis of acid number, saponification number, and free fatty acid (FFA) content of the kapok seed oil using titration standard method [6]. To reduce the gum content in kapok seed oil, degumming process was conducted. Initially the kapok seed oil was heated to 70°C and the phosphoric acid with the amount of 0.3% w/ w oil was then added. The mixture was stirred using magnetic stirrer to ensure a good contact of the reactants. The degumming step formed two layers, in which gum was found on the bottom layer and could be separated using a separating funnel.

The degummed kapok seed oil then underwent esterification with methanol in the presence of sulfuric acid catalyst in an 8 L batch reactor equipped with ultrasonic apparatus (Biosonic 6000) to reduce the FFA content. The reactor scheme is presented in Figure 1. The esterification reaction was conducted at the temperature of 60°C with the molar ratio of oil to methanol of 1:12, the sulfuric acid catalyst concentration of 0.5% w/w oil, the stirring speed of 1000 rpm, and the ultrasound frequency of 28 kHz for 90 minutes.

When the FFA content has reached below 2%, the main alkaline-catalyzed transesterification was then performed using methanol in batch reactor equipped with ultrasonic apparatus (Biosonic 6000) at the temperature of 60°C, stirring speed of 1000 rpm and the ultrasound frequency of 28 kHz for 60 minutes. The volume of kapok seed oil used for each process was 2 L. The reaction was carried out in the presence of potassium methoxide catalyst. Potassium methoxide catalyst can simply be prepared by solving potassium hydroxide in methanol reactant [7]. In this work, the effects of the molar ratio of oil to methanol and potassium hydroxide (KOH) catalyst concentration were studied at 1:4, 1:5, 1:6 and 0.5%, 1%, 1.5%, respectively. Biodiesel product through transesterification reaction was then washed using aquadest to neutralize the remains of catalyst and remove the glycerol byproduct. Biodiesel product was also heated up to 100 °C to remove the leftover water and methanol. Having been purified, biodiesel was subsequently tested to determine its properties (density, viscosity and methyl ester content). The result was compared to that of the conventional batch process.

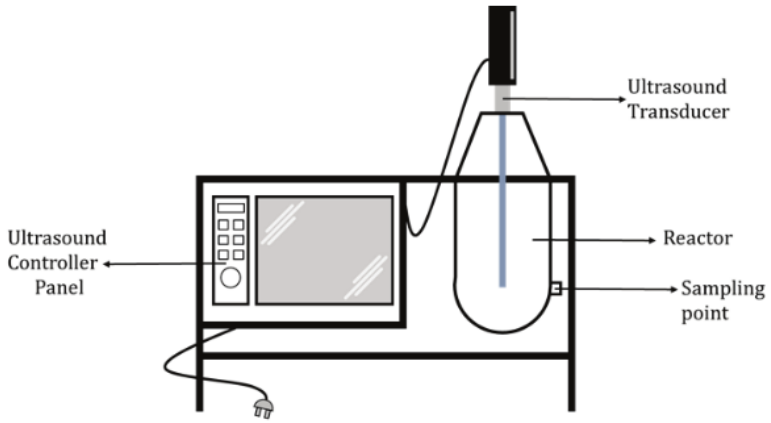

FIGURE 1. Ultrasound-Enhanced Batch Reactor for Biodiesel Production

RESULT AND DISCUSSION

Feedstock Characterization

Kapok seed oil is among the low-quality vegetable oil which has high content of FFA and gum. Hence, prior to the transesterification reaction, performing degumming and esterification processes are necessary as pretreatment of the feed-stock. Degumming process was carried out using phosphoric acid (H_3PO_4) as degumming agent to reduce gum [8]. After the degumming, kapok seed oil had to undergo the acid-catalyzed esterification reaction to decrease its FFA content until it reached below 2%. It has been well known that the high FFA vegetable oil cannot go through the alkaline-catalyzed transesterification process since the excess FFA will react with the catalyst resulting in saponification side-reaction and lowering biodiesel yield [9, 10]. The esterification reaction was conducted using batch reactor equipped with the ultrasonic tranducers. The pretreatment processes (degumming and esterification) could improve the properties of kapok seed oil as demonstrated in Table 1. It was depicted that after undergoing the pretreatment reactions, kapok seed oil has a lower density, viscosity, acid value, and FFA content. After the final pretreatment process of kapok seed oil, the FFA content decreased from 9.78% to 0.44%, which fulfilled the limitation of transesterification reaction. Fatty acid composition of kapok Seed Oil after pretreatment process is presented in Table 2.

TABLE 1. Comparison of Kapok Seed Oil Characteristics

Parameter	Crude Kapok Seed Oil	Kapok Seed Oil after Degumming	Kapok Seed Oil after Esterification
Density (g/mL)	0.942	0.941	0.87
Viscosity (cSt)	35.70	35.36	11.34
Acid Value (mg KOH/g)	19.60	17.82	0.88
FFA Content (%)	9.78	8.89	0.44

The molecular weight (MW) of kapok seed oil was determined by analyzing the fatty acid composition of kapok seed oil using Gas Chromatography-Mass Spectroscopy (GC-MS). The chromatogram of kapok seed oil is exhibited in Figure 2.

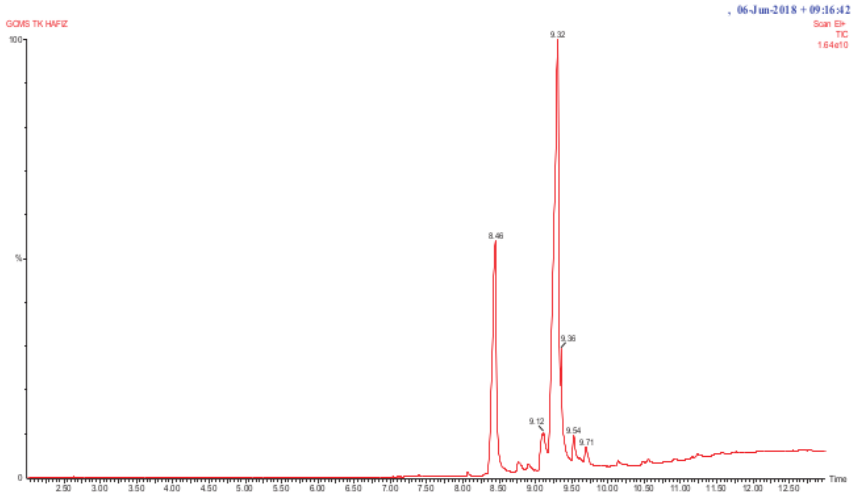

FIGURE 2. GC-MS Chromatogram of Kapok Seed Oil after Pretreatments (Degumming and Esterification)

TABLE 2. Kapok Seed Oil after Pretreatments (Degumming and Esterification)

Fatty Acid	Percentage (%)	Molecular Weight of Fatty Acid (g/mol)
Palmitic acid	29.728	256
Linoleic acid	3.678	280
Linoleic acid	57.842	280
Stearic acid	5.847	284
Linoleic acid	1.793	280
Octadecanoic acid	1.111	282

The molecular weight of vegetable oils can be estimated based on their fatty acid composition using the formula presented in Equation 1 [11].

$$MW_{V0} = \left(3 \times MW_{Avg\ of\ Fatty\ Acids} - 3 \times MW_H\right) + \left(3 \times MW_c + 5 \times MW_H\right) \quad (1)$$

MW_{VO} is molecular weight of vegetable oil and $MW_{Avg\ of\ Fatty\ Acids}$ is the average of the molecular weight of fatty acids which compose the vegetable oils. $MW_{Avg\ of\ Fatty\ Acids}$ is obtained by multiplying the molecular weight of each fatty acid with its fraction. MW_H and MW_C are the molecular weights of hydrogen (1.00794 g/gmol) and carbon (12.0107 g/gmol), respectively. Based on the calculation using Eq. 1, it was found that the molecular weight of kapok seed oil was 857.4037. It is in line with the result reported by Kapilan et al. [12], stating that vegetable oils (triglyceride molecules) commonly have molecular weights between 800 and 900 g/gmol.

Biodiesel Synthesis Using Ultrasound-Enhanced Batch Reactor

Having been pretreated, kapok seed oil feed-stock was then underwent transesterification reaction with methanol in the presence of potassium methoxide catalyst. The reaction was carried out in a batch reactor equipped with ultrasonic transducer (Biosonic 6000). During the reaction, small white bubbles appeared in the mixture which eventually spread throughout the mixture of kapok seed oil and methanol. This phenomenon is called cavitation. The high-power ultrasound produces ultrasonic cavitation, which is described as the formation, growth and collapse of bubbles regularly in the liquid exposed by ultrasound [13]. The collapse of the bubbles will cause a high local temperature and pressure, microstreaming, and shockwaves. This condition enhances the heat and mass transfer of the reactants. It will also increase the velocity gradient which generates shear stress. These facts can promote the mixing of reactants, the break of immiscible interfacial film, and enhance the mass and heat transfer in the system.

Ultrasound cavitation can create an intensive turbulence and liquid circulation at micro scale, and thus it enhances the mixing and reduce the mass transfer resistance especially in the heterogeneous system [14]. In reality, the reactants in the biodiesel production (vegetable oil and methanol) are not completely miscible. Therefore, an effective mass transfer will increase the overall reaction rate and results in higher product yield at the lower temperature and pressure.

Transesterification to produce biodiesel was conducted by reacting kapok seed oil with methanol in the presence of potassium methoxide catalyst at the temperature of 60°C and ultrasonic wave frequency of 28 kHz for 60 minutes. The effect of oil to methanol ratio was studied by varying it at 1:4, 1:5, and 1:6. On the other hand, the influence of catalyst concentration was investigated at the concentration of 0.5%, 1%, and 1.5% w catalyst/ w oil.

Transesterification will result in biodiesel as the main products, glycerol as by-product, as well as the remaining reactants and catalyst. To obtain a high purity biodiesel, purification step is necessary. Glycerol is a polar compound which can easily be separated from the biodiesel product, which is a non-polar compound, using decantation. The water is sometimes added to wash the leftover base catalyst. Meanwhile, the remaining methanol can be removed by heating biodiesel product at 100°C. The yield and properties of biodiesel were then tested.

Effect of Molar Ratio of Methanol to Oil on the Biodiesel Yield

The effect of reactant molar ratio was investigated at the molar ratio of 1:4, 1:5, and 1:6. The reactions were carried at the fixed reaction temperature of 60°C, catalyst concentration of 0.5%, and ultrasonic frequency of 28 kHz for 60 minutes. The stoichiometric ratio of tryglycerides transesterification is 1:3. However, based on the GC-MS analysis, it was found that the best biodiesel yield was 99.82%, which was achieved at the molar ratio of oil to methanol of 1:6 (Figure 3). Molar ratio of the reactants provides significant influence to the biodiesel yield since transesterification reaction is a reversible reaction, in which the excess of the reactant will shift the equilibrium the the product formation [15].

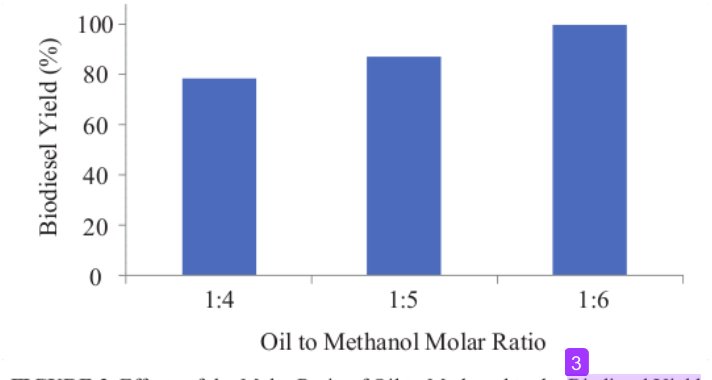

FIGURE 3. Effects of the Molar Ratio of Oil to Methanol to the Biodiesel Yield

Effect of Catalyst Concentration on the Biodiesel Yield

The effect of catalyst concentration was evaluated at the concentration of 0.5%, 1%, and 1.5%. The reactions were carried at the certain reaction temperature of 60°C, oil to methanol molar ratio of 1:6, and ultrasonic frequency of 28 kHz for 60 minutes. Transesterification is a slow and reversible reaction. Hence, the addition of catalyst is important to increase the reaction rate and speed up the reaction [16]. A catalyst gives a different reaction route with lower activation energy. Hence, the addition of catalyst will make the higher number of succesfull collision to happen, allowing the reaction to occur. It will then improve the reaction rate. In this work, it was revealed that the best biodiesel yield was provided by the reaction employing catalyst concentration of 0.5% with the biodiesel yield of 99.82% (Figure 4).

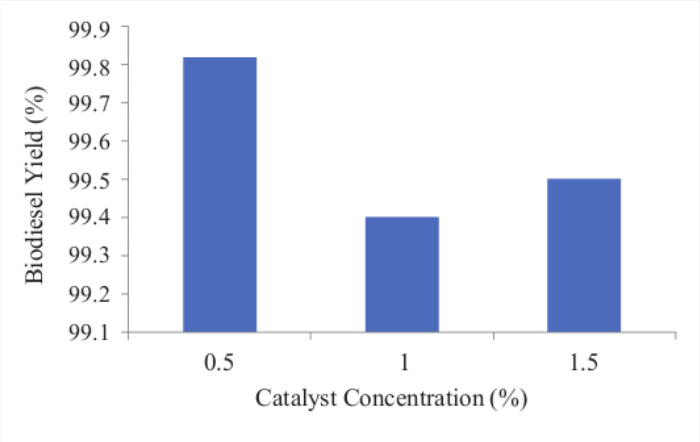

FIGURE 4. Effects of the Molar Ratio of Oil to Methanol to the Biodiesel Yield

A considerable amount of catalyst is needed to increase the reaction rate, to reduce the reaction time, and to inhibit the reversible reaction. However, too excess catalyst addition is not effective since it will cause the difficulty in the separation of biodiesel product, methanol, and catalyst. It consequently rise the separation cost and reduce the biodiesel yield, as well [15]. The excessive base catalyst will also contribute to the formation of dimethyl ether due to the reaction of methanol and KOH. Therefore, among the experiments, the optimum biodiesel yield was 99.82%, obtained at the reaction condition of: oil to methanol molar ratio of 1:6, reaction temperature of 60°C, catalyst concentration of 0.5%, and ultrasonic frequency of 28 kHz for 60 minutes reaction time.

Comparison of Biodiesel Yielded by Ultrasound Enhanced and Conventional Reactor

To examine the effectiveness of the biodiesel process intensification using ultrasonic technology, biodiesel produced using ultrasound assisted batch reactor was compared to that resulted by the conventional batch reactor at the identical reaction condition. The conventional process was accomplished at the oil to methanol molar ratio of 1:6, reaction temperature of 60°C, catalyst concentration of 0.5%, for 60 minutes reaction time. The summary is depicted in Table 3.

TABLE 3. Comparison between Biodiesel Yielded Using Conventional and Ultrasonic Reactors

Parameter	Conventional Reactor	Ultrasonic Reactor
Density (g/mL)	0.8667	0.8646
Viscosity (cSt)	4.95	4.38
Yield (%)	90.79	99.82

It was revealed that the ultrasound assisted batch reactor resulted in the higher yield of biodiesel (99.82) compared to that of the conventional process (90.79). This result was also superior compared to the transesterification of kapok seed oil conducted by Palupi and Siswani [17] which provided biodiesel yield of 89.26% for the reaction performed at 65°C for 60 minutes. Ultrasound cavitation contributes to the higher energy of the reactants molecules to overcome the activation energy, causing the higher reaction rate. In addition to giving a higher product yield, the application of ultrasonic technology for biodiesel production can also reduce the energy requirement since the cavitation of the ultrasonic wave will cause the thermal energy which increase the temperature from room temperature to the reaction temperature. Hence, the higher biodiesel yield can be achieved with the lower energy necessity. The chromatogram of biodiesel product resulted by the ultrasonic process at the optimum condition is presented in Figure 5 and the fatty acid composition is shown in Table 4.

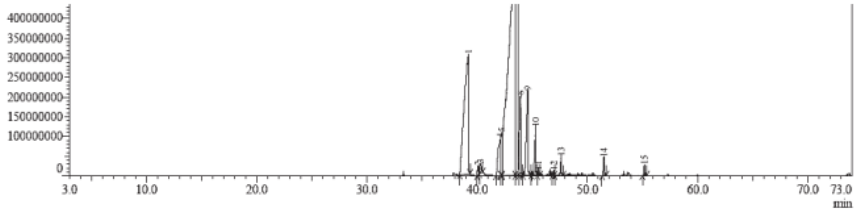

FIGURE 5. GC-MS Chromatogram of Biodiesel Produced Using Ultrasonic Reactor at the Optimum Condition

TABLE 4. Fatty Acid Composition of Biodiesel Synthesized Using Ultrasonic Reactor

Peak#	R.Time	Area%	Name
1	39.218	18.15	Hexadecanoic acid, methyl ester (CAS)
2	40.089	0.39	9,12-Octadecadienoic acid (Z,Z)-, methyl ester (CAS)
3	40.318	0.45	9-Octadecenoic acid (Z)-, methyl ester (CAS)
4	42.11	3.45	9,12-Octadecadienoic acid (Z,Z)-, methyl ester (CAS)
5	42.234	1.76	Cyclopropaneoctanoic acid, methyl ester (CAS)
6	43.339	48.66	Cyclopropanebutyric acid, methyl ester (CAS)
7	43.599	15.3	13-Octadecenoic acid, methyl ester, (Z)- (CAS)
8	43.929	3.18	Octadecanoic acid, methyl ester (CAS)
9	44.598	5	9,12-Octadecadienoic acid (Z,Z)-, methyl ester (CAS)
10	45.305	1.96	Cyclopropaneoctanoic acid, 2-octyl-, methyl ester (CAS)
11	45.55	0.18	2,5-Furandione, 3-(dodecenyl)dihydro-
12	46.944	0.14	11-Eicosenoic acid, methyl ester (CAS)
13	47.622	0.59	Eicosanoic acid, methyl ester (CAS)
14	51.505	0.53	Docosanoic acid, methyl ester (CAS)
15	55.184	0.26	Tetracosanoic acid, methyl ester (CAS)

Biodiesel Properties

To determine the quality of biodiesel produced using ultrasonic reactor, the properties of biodiesel was then assessed. Among the important properties are density, viscosity, and biodiesel purity. Density is among the parameter of the successfullnes of the transesterification reaction since it is correlated with the calor and power resulted by the diesel machine when biodiesel is applied in the machine. The lower the density of biodiesel will bring about the higher calor generated in the combustion[18]. The density of biodiesel produced at various oil to methanol molar ratio is demonstrated in Table 5. It was depicted that the higher amount of methanol used in the reaction, the density of the biodiesel was lower. It was due to the fact that some of the unreacted methanol was dissolved in the biodiesel product, lowering the density [3].

TABLE 5. Effects of Molar Ratio of Oil to Methanol on the Biodiesel Density.

No	Molar Ratio of Oil to Methanol	Density of Biodiesel (g/mL)
1	1:4	0.8687
2	1:5	0.8667
3	1:6	0.8646

The influence of catalyst concentration on the biodiesel density is shown in Table 6. It was discovered that the catalyst concentration did not significantly affect the density. The density of biodiesel for all the parameter variation has met the Indonesian standard (SNI 7182:2015) which obligate the biodiesel density of 850-890 kg/m^3 (0.850 – 0.890 g/mL).

TABLE 6. The Influence of Catalyst Concentration on the Biodiesel Density

No	Catalyst Concentration (%)	Density (g/mL)
1	0.5	0.8646
2	1	0.8646
3	1.5	0.8621

Another important parameter of the fuel combustion is viscosity. Vegetable oil cannot be readily applied as fuel in diesel machine because of its high viscosity. Vegetable oils are composed of complex stucture and they usually have high molecular weight. The particularly high viscosity of vegetable oils will cause crucial problems in term of fuel pumping and poor atomization when they are directly applied in the diesel fuel [19]. Transesterification of vegetable oil could reduce the viscosity to meet the fuel property standard. The influence of molar ratio of oil to methanol and the catalyst concentration on the biodiesel from kapok seed oil viscosity were presented in Table 7 and 8, respectively.

TABLE 7. The Effects of Oil to Methanol Molar Ratio to the Biodiesel Viscosity

No	Molar Ratio of Oil to Methanol	Viscositas (cSt)
1	1:4	4.91
2	1:5	4.63
3	1:6	4.38

TABLE 8. The Effects of Catalyst Concentration on the Biodiesel Viscosity

No	Catalyst Concentration (%)	Viscosity (cSt)
1	0.5	4.38
2	1	4.66
3	1.5	4.62

Table 7 has shown that the higher amount of methanol used in the reaction, the lower viscosity of biodiesel will be resulted. It is due to the fact that the higher ratio of methanol to oil caused the higher quantity of unreacted methanol which was dissolved in the biodiesel product, reducing the viscosity. Table 8 indicates that the catalyst concentration of 0.5% shown the lowest value of viscosity. It is because of the fact that the 0.5% catalyst concentration resulted in the highest biodiesel yield. It means that the amount of triglyceride converted to methyl ester was higher than the other, resulting in the significant decrease of the viscosity. The standard value of biodiesel viscosity based on SNI 7182:2015 is 2.3 – 6.0 cSt. Hence, the viscosity of biodiesel syntesized in this work has satisfied the SNI.

CONCLUSIONS

Based on the experimental result on the biodiesel synthesis from kapok seed oil using ultrasonic batch reactor, it can be concluded that biodiesel yield enhanced with the increase of reactants molar ratio. Furthermore, the addition of catalyst will improve the reaction rate. However, too excessive employment of catalyst will decrease the yield due to the separation problem and the formation of side-reactions. The optimum biodiesel yield was 99.82%, which was obtained at the reaction condition of: oil to methanol molar ratio of 1:6, reaction temperature of 60°C, catalyst concentration of 0.5%, and ultrasonic frequency of 28 kHz for 60 minutes reaction time. The properties of biodiesel product met the Indonesian standard (SNI) in term of density, viscosity and biodiesel purity. The yield obtained by using ultrasonic reactor (99.82%) was higher that the yield achieved by the conventional process at the identical reaction condition (90.79%).

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