Biodiesel Production from Used Cooking Oil Using Coal Low Rating as Environmentally Friendly Heterogeneous Catalyst

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Abstract: The increasing need for fossil fuels encourages efforts to meet these needs, by developing alternative fuels made from renewable raw materials, such as cooking oil from palm oil. Cooking oil is an important commodity at this time which is quite widespread in Indonesia. The repeated use of cooking oil can produce used cooking oil that cannot be re-consumed and has a negative impact on the environment. The purpose of this study was to identify the characteristics of low rank coal with thermal assistance as a heterogeneous catalyst to produce biodiesel and to study the effect of biodiesel product quality parameters on the esterification and transesterification processes. Therefore, in this study the production of biodiesel from used cooking oil was carried out using a low rank coal catalyst, to determine the effect of the amount of catalyst on the yield and quality of the biodiesel produced. Biodiesel is obtained by reacting oil and methanol, then low rank coal catalyst is added with various mass additions of 3, 5, 7, 9, and 11 grams. Based on this study, the best results were obtained with the addition of 7 grams of catalyst, with biodiesel yield reaching 82.20%, density 864.43 kg/m3, kinematic viscosity 3.60 cSt, water content 0.20%. , the acid number is 0.99 mgKOH/g, and the methyl ester content is 97.48%. In general, the biodiesel produced has met the requirements of SNI 7182:2015, except for the parameters of water content and acid number.

1 INTRODUCTION

Indonesia is a country with various natural resources, one of which is oil palm. Based on statistics from the Directorate General of Indonesian Plantations, the Volume and Value of Palm Oil Exports (CPO) 2015-2017 showed a decline from 26,467,564 tons in 2015 to 24,150,232 tons in 2016. It is estimated that this is due to the large consumption of domestic palm oil. from the excessive use of palm oil is the production of waste palm oil or what is known as used cooking oil or used cooking oil. To overcome this problem, efforts are needed to convert used cooking oil into more viable products, such as biodiesel (Efendi et al, 2018).

Used cooking oil is used cooking oil for frying which is used repeatedly, with the production and consumption of cooking oil, the availability of cooking oil is abundant. Cooking oil is a waste and when viewed from its chemical composition, used cooking oil contains carcinogenic compounds that occur during the frying process. The use of cooking oil in a sustainable manner can damage human health, cause cancer, and further reduce the intelligence of the next generation (Siswani et al, 2012).

For this reason, proper handling is needed so that this used cooking oil waste can be useful and not cause harm from aspects of human health and the environment through a conversion from used cooking oil into biodiesel (Darmawan, 2013).

Biodiesel is a fuel made from vegetable oil or animal fat. Biodiesel is a fuel consisting of a mixture of mono-alkyl esters derived from long-chain fatty acids, which are renewable sources from nature. Biodiesel is also known as an environmentally friendly fuel because it produces relatively cleaner

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exhaust emissions than diesel. In addition, the use of biodiesel is generally easy, because there is no need to modify the diesel engine (Aziz et al, 2012).

Biodiesel has advantages over petroleum diesel. Biodiesel fuel is renewable. In addition, it can also strengthen the country's economy and create jobs. Biodiesel is an ideal fuel for the transportation industry because it can be used in various diesel engines, including agricultural machinery.

According to the Indonesian Palm Oil Association (Gapki), domestic palm oil consumption in 2020 was 17.35 million tons or grew 3.6% compared to 2019 of 16.75 million tons. The development of biodiesel based on used cooking oil has the opportunity to be marketed, both domestically and for export. Throughout 2020, domestic biodiesel consumption increased by about 24% from 5.83 million tons in 2019 to 7.23 million tons.

In addition, the use of biodiesel as a fuel has many advantages, including being renewable and environmentally friendly (reducing vehicle emissions), being able to lubricate the engine as well as being a fuel so as to increase the life of the vehicle, it is safe to store and transport because this fuel does not toxic and biodegradable and can reduce dependence on fossil fuels (Balat et al, 2010).

The use of biodiesel as a renewable and environmentally friendly fuel (reducing vehicle emissions) is a treatment in the biodiesel manufacturing process by adding a catalyst and with ultrasonic assistance in order to increase the conversion and yield produced.

To make it as a catalyst, thermal assistance is needed so that the binding mechanism becomes a simpler hydrocarbon. In line with that, in improving the quality of biodiesel by using a catalyst, it is also possible to break the bonds in this case using an ultrasonic device. Ultrasonic waves can increase the rate of transesterification of used cooking oil into biodiesel so that the conversion of used cooking oil into biodiesel with the use of ultrasonic waves is higher than other uses.

In the research of Pasae et al., (2019), biodiesel was produced from used cooking oil using a heterogeneous catalyst in the form of shells, with variations in temperature in the calcination and transesterification processes. The amount of oil and methanol used was 200 mL of oil and 600 mL of methanol (1:3 v/v), with the addition of 2 grams of clam shells as a catalyst. The best results in this study were transesterification for 3 hours, with a yield of 66.09%, a density of 853 kg/m^2, a kinematic viscosity of 2.77 mm^2/s, an acid number of 0.56 mgKOH/g, and a saponification rate of 201 mgKOH/g. These results have met SNI Biodiesel 7182: 2015 except for the number of acids produced.

In the research of Saputri et al., (2016), biodiesel methyl esters were made from used cooking oil and methanol using a heterogeneous catalyst in the form of rubbing ash with variations of 2, 4, 6, 8, and 10 grams. The best results in this study were achieved using 10 grams of rubbing ash as a catalyst, with an acid number of 0.71 mg KOH/g, a total glycerol content of 0.01%, a density of 0.88 g/cm3, a viscosity of 47.94 cSt, an iodine number. 66.83 g I_2/100, water content 0.06%, flash point 249.33ºC, saponification number 154.84 mg KOH/g, and methyl ester content 99.5%. The results obtained have met the biodiesel standard SNI 7182: 2015, except for the viscosity and water content of the biodiesel produced.

In the research of Khoiruummah et al., (2020), activated carbon from acacia wood was impregnated using KOH and NaOH, then used as a heterogeneous catalyst in the manufacture of biodiesel from used cooking oil. This study used variations in the amount of KOH catalyst/activated carbon, namely 1,3, and 5% w/w oil, as well as variations in temperature, namely 45, 55, 65, and 75ºC, with an oil-methanol molar ratio of 1:6. In addition, this study also used variations in the amount of NaOH catalyst/activated carbon, namely 3, 4, and 5% w/w oil, with variations in transesterification temperature of 50, 55, 60, and 65ºC.

The best results on the use of KOH/activated carbon catalyst is the catalyst variation of 3% w/w with a transesterification temperature of 65ºC, which produces a yield of 87.51%. The use of KOH catalyst/activated carbon produces biodiesel with a density of 0.7724-0.8585 g/mL, viscosity 4.5485-5.3672 cSt, acid number 1.1222-2.2444 mgKOH/g, water content 0.034-0.246% , and a flash point of 150-170ºC.

Meanwhile, the best results on the use of NaOH/activated carbon catalyst are catalyst variations of 3% w/w with a transesterification temperature of 60ºC. In this variation, a yield of 88.35% was obtained. Overall, variations in the amount of NaOH catalyst/activated carbon produced biodiesel with a density of 0.89282-0.90722 g/mL, kinematic viscosity 2.3439-4.1601 cSt, acid number 0.84165-2.2444 mgKOH/g, content water 0.000592-0.071963%, and a flash point of 128-153ºC. In general, the biodiesel produced has complied with SNI 7182:2015 on the parameters of viscosity and flash point. As for the water content and density parameters, some do not meet SNI

7182:2015. While for the acid number parameter, all of them do not meet SNI 7182:2015.

Meanwhile, in the research of Oko et al., (2021) the process of making biodiesel from used cooking oil was carried out through two reaction stages, namely esterification and transesterification reactions. The esterification reaction was carried out for 1 hour with 100 grams of used cooking oil as raw material, then 52 grams of methanol and 1 gram of H_2SO_4 catalyst were added. Then, in the transesterification reaction, the mass ratio of CaO and C was varied in the NaOH/CaO/C catalyst. The best result of this research is the mass ratio of CaO/C 1:1 with a catalyst of 3% (w/w), with a yield of 83.45% (w/w), kinematic viscosity 2.3 cSt, density 0.8612 g/ mL, water content 0.0273% (w/w), and acid number 0.2516 mgNaOH/g. All of these results have met the biodiesel standard of SNI 7182:2015.

In this research, the process of making biodiesel is carried out through two reaction stages, namely the esterification reaction which refers to the research of Oko et al., (2021), and the transesterification reaction, with a fairly large oil:methanol ratio of 1:3 (v/v). referring to the research of Pasae et al., (2019). The addition of large amounts of methanol is expected to increase the yield of biodiesel and prevent the kinematic viscosity from being too high in the final product. In addition, in this study a different heterogeneous catalyst was used, namely activated carbon catalyst from low rank coal impregnated with NaOH. By using this catalyst, it is expected that the biodiesel produced is able to meet several criteria in SNI 7182:2015.

2 METHODOLOGY

There are two stages in this research, namely the production of catalysts from low rank coal and the production of biodiesel.

- For the production of catalysts from low rank coal, first prepare low rank coal, then analyze coal samples with analysis of proximate, total sulfur, and calorific value, then carry out the carbonization process in a furnace at a temperature of 600 °C for 2 hours. After that the carbon is cleaned and then cooled in a desiccator. Then do physical activation on the catalyst with a temperature of 950°C for 2 hours. Then cool the catalyst in a desiccator. Then puree the catalyst using a blender, then filter the catalyst with a size of -100 +120 mesh. After that, a proximate analysis was carried out on the low rank coal catalyst.

- Biodiesel production is the first collected used cooking oil. Then analyzed the used cooking oil which includes water content, density at 40 °C, kinematic viscosity at 40°C, FFA content, and acid number. If the FFA level exceeds 1%, then an esterification reaction is carried out (Hadrah et al., 2018), which is to react 100 grams of used cooking oil with 52 g of methanol and 1 g of H2SO4 (Oko et al., 2021). The reaction was maintained at a temperature of 50-60°C for 1 hour (Arifin et al., 2016). Then separate the esterification product in a separatory funnel for 1 hour, then take the bottom layer for use in the transesterification reaction (Oko et al., 2021).

Then carry out a transesterification reaction by reacting the esterified oil with a catalyst-methanol mixture at a temperature of 60-70°C for 1 hour 30 minutes, where the variations of the added catalyst are 3, 5, 7, 9, and 11 grams (Saputri et al., 2016) . The ratio of oil:methanol used was 1:3 v/v (Pasae et al., 2019). Then filtering the catalyst while separating the transesterification results in a separating funnel for 12 hours (Pasae et al., 2019). Then take the top layer and heat it on a hot plate at a temperature of 60-70°C for ± 1 hour to remove the remaining methanol (Khoiruummah et al., 2020). Then wash the biodiesel produced using aquadest at a temperature of 70-80°C until the water becomes clear (Khoiruummah et al., 2020). After that, the biodiesel is put in the oven for ± 3 hours to reduce the water content (Khoiruummah et al., 2020). Then save the obtained biodiesel.

The procedure of the process can be described as shown in the figure below, namely in Figure 1 and Figure 2.

Research Procedure

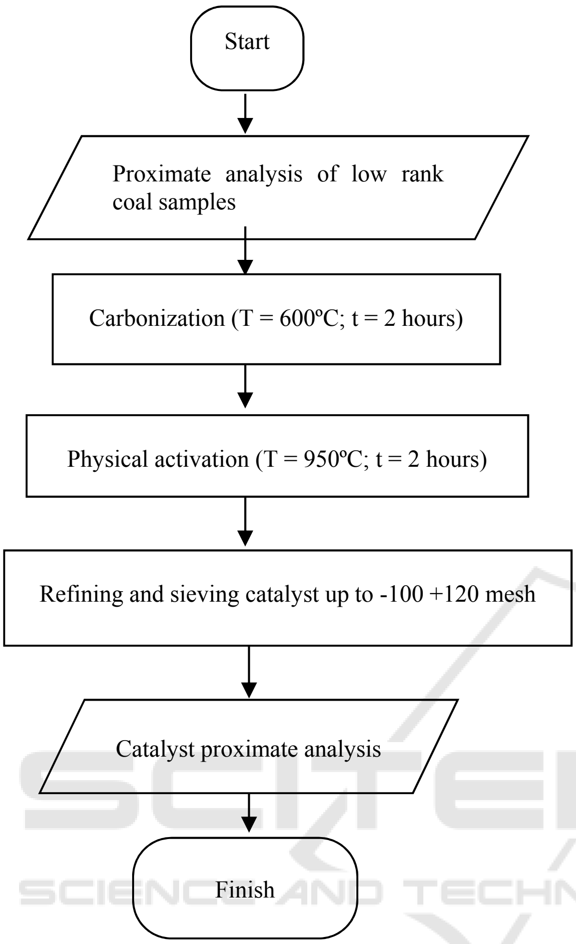

Figure 1: Catalyst Production from Low Rank Coal.

3 RESULT AND DISCUSSION

3.1 Analysis Results

In this study, biodiesel was made from used cooking oil using a low rank coal activated carbon catalyst, with variations in the addition of a catalyst of 3, 5, 7, 9, and 11 grams which aims to determine the effect of the catalyst on the yield of biodiesel produced.

The initial stage of making biodiesel is collecting used cooking oil, then analyzing the oil with the parameters of free fatty acid content, color, density at 40ºC, kinematic viscosity at 40ºC, and acid number. This analysis aims to determine the condition of used cooking oil before it is used as raw material, as well as to determine the reaction stage used in the manufacture of biodiesel.

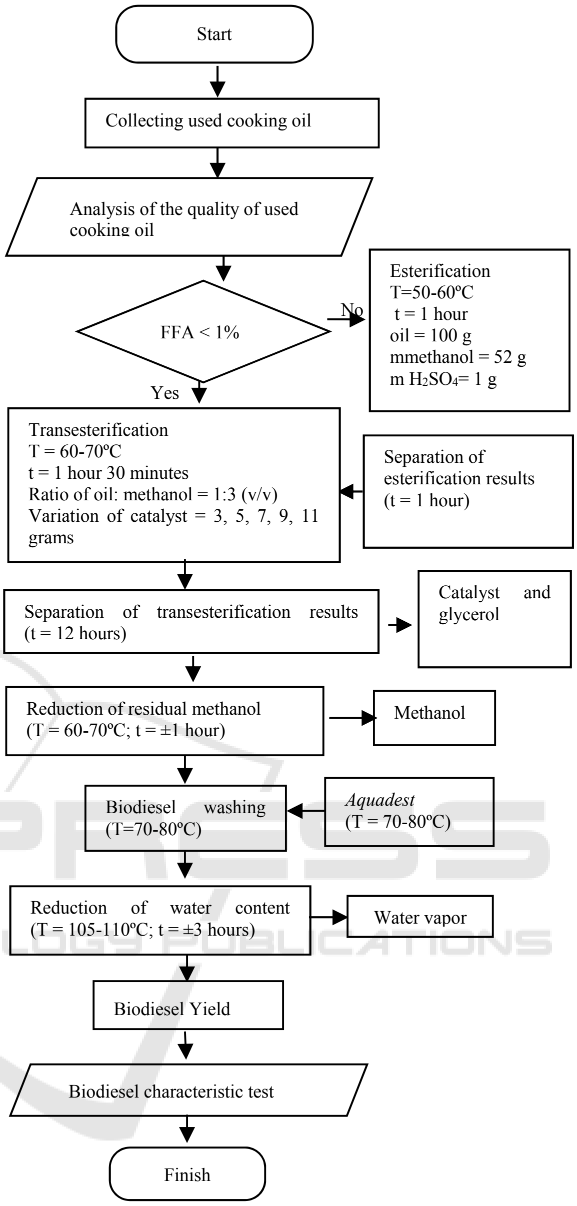

Figure 2: Biodiesel Production.

Table 1: Results of Low Rank Coal Analysis.

Parameter	Analysis Results
Total Moisture, %	37.40
Inherent Moisture, %	14.02
Ash Content, %	2.86
Volatile Matter, %	45.61
Fixed Carbon, %	32.51
Total Sulphur, %	0.98
Calorific Value, kcal/kg	4714

Table 2: Results of Low Rank Coal Analysis (Physical Activation).

Parameter	**Analysis Results**
Inherent Moisture, %	3.13
Ash Content, %	38.23
Volatile Matter, %	6.64
Fixed Carbon, %	55.13

Table 3: Results of Analysis of Used Cooking Oil Characteristics.

Parameter	Analysis Results
Free fatty acid content, %	4.34
Warna Color	Abnormal (clear brown)
Density, 40ºC, kg/m3	896.04
Kinematic viscosity, 40ºC, cSt	35.39
Acid number, mgKOH/g	12.57

Table 3 above shows that with the use of 7 grams of catalyst, the highest yield was obtained at 82.20%. The quality of the biodiesel produced is as follows: density: 864.43 kg/m3, kinematic viscosity: 3.60 cSt, water content: 0.20%, and acid number: 0.99 mgKOH/g.

Table 4: Results of Biodiesel Analysis.

Parameter	Analysis Results					SNI 7182:2015
	Catalyst 3 grams	Catalyst 5 grams	Catalyst 7 grams	Catalyst 9 grams	Catalyst 11 grams	
Density, 40ºC, kg/m3	865.18	865.91	864.43	859.42	858.07	850-890
Kinematic viscosity, 40ºC, cSt	3.59	3.64	3.60	3.58	3.34	2.3-6,0
Moisture content, % (v/v)	0.94	0.04	0.20	0.18	0.17	0.05
Acid number, mgKOH/g	1.22	1.23	0.99	1.00	0.89	0.5
Yield, %	21.97	35.22	82.20	61.46	69.13	-

4 DISCUSSION

- **Effect of Catalyst Amount on Biodiesel Yield**

Variations in the addition of catalysts as much as 3, 5, 7, 9 and 11 grams in the transesterification reaction have a significant effect on the amount of biodiesel produced, as shown in the figure below:

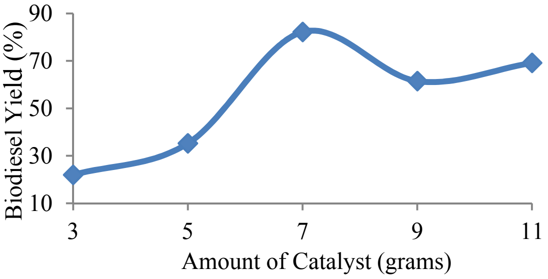

Figure 2: Graph of the Effect of Catalyst Amount on Biodiesel Yield.

Based on Figure 2 above, the addition of catalyst in the range of 3 to 7 grams showed a significant increase in yield, where the highest yield was obtained at the addition of 7 grams of catalyst, which was 82.04%. The addition of a catalyst can reduce the activation energy, where the more catalyst is added, the activation energy decreases, so the reaction rate will increase. With the increase in the reaction rate, the conversion of used cooking oil into biodiesel is also getting bigger (Prihanto & Irawan, 2018).

Meanwhile, the addition of 9 and 11 grams of catalyst actually reduced the amount of biodiesel produced. This is caused by the formation of soap that occurs through a saponification reaction, where the formation of soap can hinder the conversion of oil into methyl esters. In addition, with the addition of 9 grams of catalyst, the yield of biodiesel produced is actually lower than that of 11 grams of catalyst.

This is due to imperfect filtering of the catalyst, so that the catalyst is carried into the product and increases the amount of impurities. This increase in the amount of impurities makes the washing process more difficult, so that a lot of biodiesel is wasted during the washing process.

According to Bintang et al., (2015), the washing process and water separation after washing can reduce the amount of biodiesel produced. The purification process needs to be carried out properly because the remaining impurities can affect the quality of biodiesel, especially on the density parameter (Faizal et al., 2013)

- **Biodiesel Quality**

The quality of biodiesel produced can be seen in table 4 with analytical parameters including: density at 40ºC, kinematic viscosity at 40ºC, water content, and acid number. For the variation of the catalyst with the highest yield (7 grams), additional analysis was carried out in the form of methyl ester content to determine the purity level of the biodiesel produced.

Based on table 4, the obtained biodiesel density ranges from 858.07-865.91 kg/m3. These results have met the density standard of SNI 7182: 2015 which is between 850-890 kg/m3.

According to Hadrah et al., (2018), biodiesel with a density according to SNI standards is able to produce perfect combustion. In addition to density, the kinematic viscosity parameter also meets SNI 7182:2015, where the results obtained are in the range of 3.55-3.87 cSt. These results are within the range allowed by SNI, namely 2.3-6.0 cSt. The water content parameters in general do not meet SNI 7182:2015, except for the 5 gram catalyst variation. High water content can be caused by an incomplete evaporation process (Kusumaningtyas & Bachtiar, 2012).

Meanwhile, the acid number parameter in general does not meet SNI 7182:2015. The high acid number in biodiesel indicates the presence of free fatty acids in biodiesel produced by the hydrolysis reaction between oil and water (Zamhari et al., 2021). In addition, the acid number is also closely related to the pH of biodiesel (Faizal et al., 2013).

Therefore, to reduce the acid number, the evaporation process of the water content can be carried out to prevent the hydrolysis reaction, as well as carry out neutralization until the biodiesel pH is close to 7. The parameters of the methyl ester content were only reviewed for biodiesel with the highest yield of 82.20% (7 grams of catalyst variation).

Methyl ester analysis was carried out using an FT-IR instrument, with the resulting IR spectrum as follows:

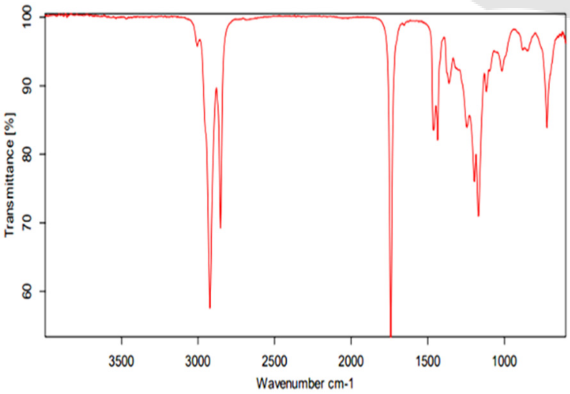

Figure 3: IR Spectrum on Biodiesel.

In the spectrum above, it is seen that there is a sharp absorption at wavelengths 1169.46, 1741.85, 2853.07 and 2922.45 cm-1. Based on the book Dachriyanus, (2004), the absorption at 1169.46 cm-1 indicates the presence of C-O bonds (1300-1000 cm-1). Meanwhile, the absorption at 1741.85 cm-1 indicated the presence of an ester group, namely C=O (1900-1650 cm-1). Then the absorption at 2922.45 cm-1 and 2853.07 cm-1 indicated the presence of C-H bonds (3000-2700 cm-1). The C=O, C-H and C-O bonds in biodiesel prove the presence of methyl ester compounds contained in it. The types of methyl esters in biodiesel can be identified using the IR spectrum comparison as follows:

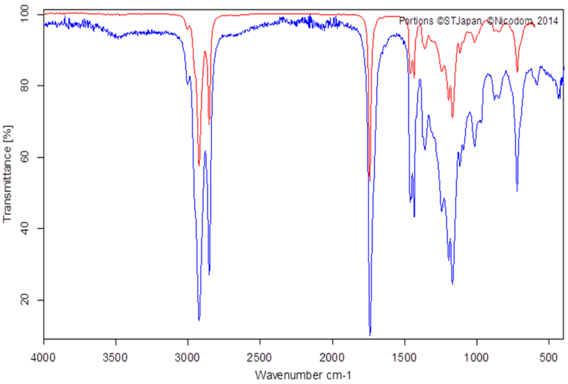

Figure 4: Comparison of the IR Spectrum of Methyl Oleate (Blue) with Biodiesel (Red).

In Figure 4 above, it can be seen that there is a comparison between the IR spectrum of biodiesel and the IR spectrum of methyl oleate under the same conditions. The IR spectrum of methyl oleate is the standard spectrum used as a comparison.

According to Siswani et al., (2012), to identify an unknown compound, a comparison can be made between the IR spectrum of the compound and the IR spectrum of a standard compound under the same conditions. The similarity between the two spectra being compared indicates that the compound is identical. The results of the analysis show that biodiesel has an IR spectrum that is identical to the IR spectrum of methyl oleate. Therefore, it can be seen that the methyl ester compound in the biodiesel produced is predominantly methyl oleate.

Meanwhile, to determine the methyl ester content in detail, additional analysis was performed using the FT-IR ATR-PLS-FAME Quantification method, which refers to the ASTM D7371 and EN 14078 methods. From this analysis, the methyl ester content was obtained with an estimate of 97.48%. This level has exceeded the minimum limit determined by SNI 7182:2015 which is 96.5%. Thus, the methyl ester content in biodiesel has met the SNI 7182:2015 standard.

5 CONCLUSION

1. The more catalyst added to the reaction, the greater the yield of biodiesel produced, but the yield of biodiesel will decrease if the addition of catalyst has reached the optimum condition.

2. The best result of this research is the use of 7 grams of catalyst, with a yield of 82.20%. The quality of the biodiesel produced is as follows:

- density: 864.43 kg/m3
- kinematic viscosity: 3.60 cSt
- water content: 0.20%
- acid number: 0.99 mgKOH/g
- methyl ester content: 97.48%.

3. Parameters of density, kinematic viscosity, and methyl ester content in the biodiesel produced have complied with SNI 7182:2015. Meanwhile, the parameters of water content and acid number do not meet SNI 7182:2015.

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