

Quantity and Physicochemical Analysis of Biodiesel from CPO Processed by Electrolysis Method

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ABSTRACT

Biodiesel is produced by esterification-transesterification, using alcohol as a reactant and acid and base catalysts. Electrocatalyst is a method to increase production yield by utilizing H^+ and OH^- ions as a catalyst. This study aims to investigate the biodiesel production process through the esterification-transesterification reaction and analyze the physicochemistry of biodiesel produced from CPO by electrolysis using platinum electrodes. CPO was esterified-transesterified and then analyzed the production yield and physicochemistry of biodiesel. Results showed that the highest biodiesel yield was produced at a voltage of 6 V, which is 88.67%, the lowest acid value and moisture content of biodiesel was produced from a voltage of 12 V are 0.99 mgKOH/g and 0.75%, the kinematic viscosity that met the SNI biodiesel standards resulted in a voltage of 6 V and without the use of electrolysis, namely 3.53 cSt and 3.63 cSt, and the highest flash point value for biodiesel is produced from a voltage of 12 V, which is 109 °C.

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1. INTRODUCTION

Crude palm oil (CPO) is the largest vegetable oil in Indonesia produced from oil palm, the largest vegetable oil-producing plant. CPO production in Indonesia reached 44.8 million ton (BPS, 2020). This is a big opportunity in producing biodiesel as an alternative fuel for diesel. Biodiesel is a renewable fuel that can be used in diesel engines. Biodiesel can be produced from living thing (vegetable oil or animal oil). One example of vegetable oil that can be used as biodiesel is CPO.

Biodiesel is produced by a process called esterification-transesterification with the help of acid and base catalysts which function to speed up the reaction. If the feedstock has high free fatty acid (FFA), that is greater than 2%, the biodiesel conversion process goes through a two-step reaction process, namely esterification with an acid catalyst and then transesterification with an alkaline catalyst. The catalysts that are generally used including KOH, NaOH, H_2SO_4 , and HCl. Actually a catalyst is a substance functioning to boost the rate of reaction in the esterification-transesterification process. Without the use of a

catalyst, the biodiesel production process will take a very long time and must use a high temperature of 250 °C (Hadrah *et al.*, 2018).

CPO is a vegetable oil with a high FFA value (> 2%), so it needs to be esterified with an acid catalyst first to reduce the FFA value (Laila, 2017). After the esterification, the next step, namely transesterification, will be carried out with the addition of a base catalyst. This reaction will produce two products, namely biodiesel and glycerol.

One way to increase the yield of biodiesel is by utilizing the electrolysis process which produces OH^- and H^+ ions which are used as catalysts in the transesterification process to produce biodiesel. The advantage of using electrolysis in the transesterification process is that it can be an acid and base catalyst at the same time (Putra *et al.*, 2016). Research conducted by Moeksin *et al.* (2017) electrocatalyze used cooking oil using a series of electrodes with graphite bars as cathodes and anodes and flowing several variations of electric current of 6 V to 12 V for 4 hours and stirred at a speed of 30 rpm. From these experiments, the highest yield of biodiesel (38.3%) was obtained at voltage of 12 V.

Based on research conducted by Asl *et al.* (2020) who electrolyze used cooking oil using graphite electrodes, the voltage used ranged from the smallest 0 V to the largest 50 V. It showed that the effect of voltage on biodiesel based on the yield was quite significant as the voltage increased during the transesterification reaction process. This shows that the amount of voltage applied during the transesterification process greatly affects the yield produced. Meanwhile, research by (Sinaga *et al.*, 2014) shows that the longer the time and the higher the temperature of the transesterification reaction, the higher the yield of biodiesel obtained and the better characteristics of biodiesel will be. According to (Wahyono *et al.*, 2017) the material of the electrodes is one of the factors that affect electrolysis, inert metals will not oxidize (react) in the electrolysis process. Metal electrodes such as platinum are often used as electrodes in the biodiesel electrolysis process because platinum is an inert metal that has good catalytic properties in the formation of biodiesel in the electrolysis process (Ramadhan, 2020). Many factors affect the quantity and quality of biodiesel production. Therefore we need standards that will be used as a reference to determine the biodiesel quality as shown in Table 1.

Table 1. SNI biodiesel quality requirements

No	Test Parameters	Units, min/max	SNI 7182:2015
1.	Density at 40 °C	kg/m ³	850 – 890
2.	Kinematic viscosity at 40 °C	mm ² /s (cSt)	2.3 – 6.0
3.	Flash point (covered bowl)	°C, min	100
4.	Water content	%-vol, max	0.05
5.	Acid number	mg-KOH/g, max	0.5

Source : (BSN, 2015)

This study aims to carry out the biodiesel production process through the esterification-transesterification reaction and analyze the production yield, acid number, kinematic viscosity, density, moisture content, and flash point of biodiesel produced from CPO by using electrolysis in the transesterification process using platinum electrodes.

2. MATERIALS AND METHODS

2.1. Equipment and Materials

The tools used in this study were platinum electrodes, DC power supply, beaker, measuring cup, stirrer, thermometer, digital scale, syringe, viscometer, pan, pH meter, oven, thermometer, Erlenmeyer tube, magnetic stirrer and hot plate. The materials used in this study were crude palm oil (CPO) provided from PT. Syaukath Sejahtera, Bireuen District, Aceh Province. Ethanol, methanol, KOH, aquades, and fenolftalein (PP) were obtained from Post Harvest Engineering Laboratory, Faculty of Agriculture, Syiah Kuala University.

2.2. Work Procedures

2.2.1. FFA Determination

The initial step taken in this study was to analyze the free fatty acid (FFA) value in CPO which was carried out by the titration method. CPO was weighed with a mass of 5 grams and mixed with 25 ml of ethanol and heated to boiling, then allowed to stand until normal temperature (room temperature). After that, 3 drops of PP indicator were added, then titrated using 0.1 N KOH until the color of the sample turned pink and the amount of KOH (ml) used was recorded (Suaniti *et al.*, 2015). The FFA value was calculated using Equation (1).

$$FFA (\%) = \left(\frac{V \times N \times M}{10 w} \right) \quad (1)$$

where V is titration volume (ml), N is the normality of KOH (N), w is the oil mass (g), and M is molecular weight of predominant fatty acid (g/mol).

2.2.2. Esterification Step

CPO esterification was carried out by placing 300 mL of CPO into a beaker glass and heating it to a temperature of 60 °C. Then HCl catalyst was added as much as 1% (w/w) of CPO, and methanol was added with a mole ratio of 1:12 (CPO : methanol). After that, it was stirred using a magnetic stirrer for approximately 60 min (Ramadhanti *et al.*, 2020). Then leave it for 60 min which will produce water and biodiesel and unreacted triglycerides.

2.2.3. Esterification Synthesis

CPO that has been esterified and if the FFA value is below 2% then transesterification is carried out using methanol which has been added with KOH (1% by weight of CPO). The mixture was then electrolyzed using a series of platinum electrodes using a DC power supply and stirred using a magnetic stirrer for 1 hour (Ikram *et al.*, 2021). This research was conducted with different voltages (6 V, 9 V, 12 V) as presented in Figure 1. One experiment without electrolysis was also performed as a control. After that it was left for 24 h which will produce two products, namely methyl ester (biodiesel) and glycerol. All experiments were carried out with three replicates.

Transesterification produces two products, namely biodiesel and glycerol. Biodiesel is then taken and separated from glycerol. After separation, it is then washed using water with a temperature of 60 °C several times until the color of the washed water is clean. The final stage is drying through heating with the aim of cleaning the remaining water that is still contained in the biodiesel.



Figure 1. Electrolysis process for biodiesel synthesis

2.3. Measurement and Analysis

Some parameters to be evaluated included biodiesel yield, acid number, kinematic viscosity, density, moisture content, flash point, cloud point, and pour point contained in biodiesel. The biodiesel production was the biodiesel quantity obtained after the transesterification reaction process. The biodiesel yield was presented in % (v/v) and was calculated using the formula in Equation (2).

$$\text{Biodiesel yield (\%)} = \frac{\text{biodiesel volume}}{\text{CPO Volume}} \times 100\% \quad (2)$$

The acid number is the amount of KOH added (ml) to biodiesel, testing the acid number is the same as testing the FFA value. The value of the acid number can be calculated using the formula in equation (3).

$$\text{Acid number} = \left(\frac{B \times V \times N}{w} \right) \quad (3)$$

where B is molecular weight of KOH (g/mol).

Kinematic viscosity was measured using Ostwald viscometer. As much as 20 ml of biodiesel was heated to a temperature of 40 °C, then put it in an Ostwald viscometer and the time the biodiesel moves from top to bottom marks was recorded ($t_{\text{biodiesel}}$). Similar step was performed using water to get the value (t_{water}). The kinematic viscosity of biodiesel was calculated according to Equation (4) (Efendi *et al.*, 2018).

$$\text{Biodiesel Viscosity (cSt)} = \frac{t_{\text{biodiesel}}}{t_{\text{water}}} \times \text{water viscosity} \quad (4)$$

where kinematic viscosity is presented in cSt (centistokes = mm²/s), and kinematic viscosity of water is 0.658 cSt

The density of biodiesel was measured by taking 100 ml of biodiesel and then weigh it using a digital scale. The density of biodiesel was calculated by Equation (5) (Efendi *et al.*, 2018).

$$\rho = \frac{m}{v} \quad (5)$$

where ρ is density of biodiesel (kg/m³), m is biodiesel mass (kg), and v is biodiesel volume (m³).

The moisture content of biodiesel was measured by heating 5 grams of biodiesel in an oven at a temperature of 105 °C to a constant weight. The formula to calculate the moisture content was Equation (6) (Efendi *et al.*, 2018).

$$\text{Moisture content (\%)} = \frac{W_2 - W_3}{W_1} \times 100\% \quad (6)$$

where W_1 is biodiesel mass (gram), W_2 is mass of biodiesel + cup before heating (gram), and W_3 is mass of biodiesel + cup after heating (gram).

Flash point is the lowest temperature for biodiesel to cause a fire. The flash point was measured by heating the biodiesel in a container until it emitted smoke and the temperature was measured using an infrared thermometer (Efendi *et al.*, 2018). The complete research flow chart can be seen in Figure 2.

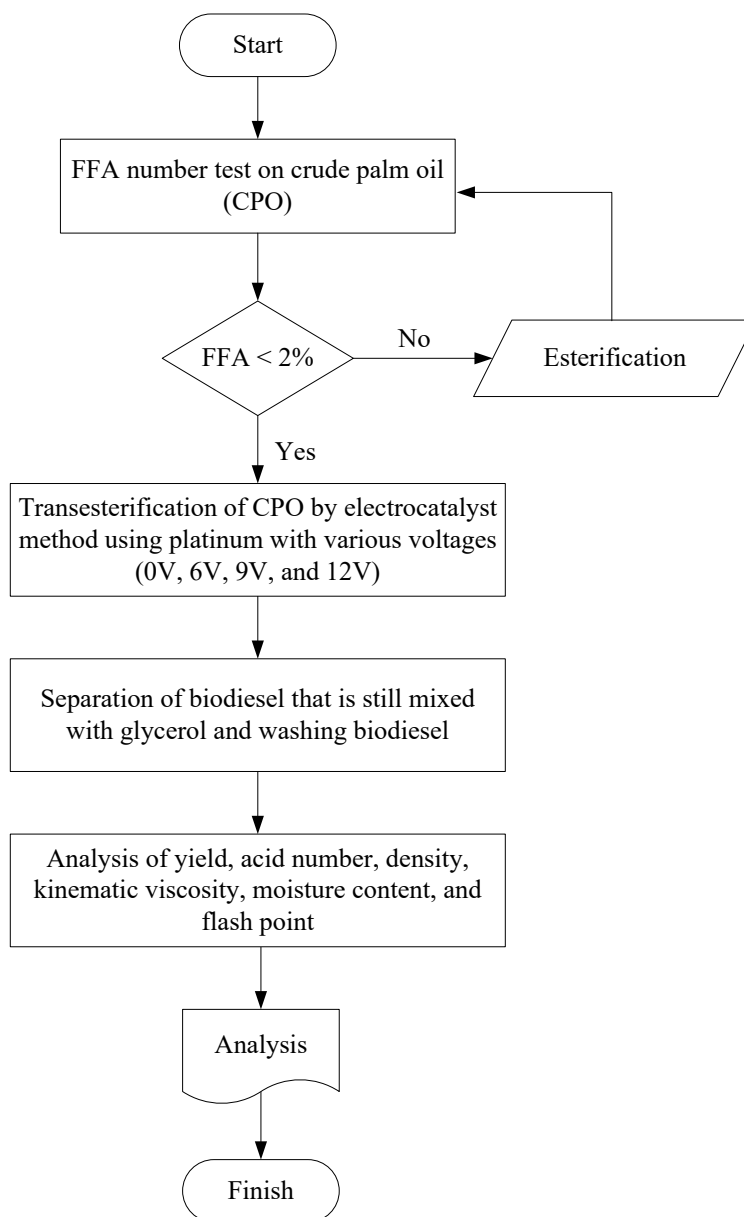


Figure 2. Research flow chart for biodiesel synthesis using electrolysis

3. RESULTS AND DISCUSSION

3.1. Esterification of CPO

Table 2 shows the analysis of FFA and acid numbers of the CPO used in this research. The results of the analysis of the FFA value of CPO in this study were not much different from the results of the analysis conducted by [Hasibuan \(2012\)](#) which was 3.94% which also said that the FFA value of some CPO production in Indonesia did not meet the standards. One of the reasons for the high FFA value in CPO is due to the lack of supervision and control measures carried out at the palm oil mills. Oil with low FFA can be synthesized to biodiesel with only one step process, namely transesterification. However, oil with high FFA value must go through two stages for biodiesel production, namely esterification-transesterification.

Table 2. Analysis of FFA value and acid number of CPO

Sample	Esterification	FFA (%)	Acid number (mgKOH/g)
Crude plam oil (CPO)	Before	3.06	6.73
	After	1.8	4

Based on the analysis results as shown in Table 2, CPO has an FFA value above 2% with an acid number exceeding 5 mgKOH/gram. Therefore, CPO must go through an esterification process before the transesterification process. The high FFA value in CPO may be due to the fact that there is still a lot of water content in it, causing oil (triglycerides) to hydrolyze into free fatty acids (FFA) ([Suroso, 2013](#)). Esterification is important to reduce soap production during the transesterification process with an alkaline catalyst which will react with the acid content in the oil which can lead to a lower yield ([Setiawan *et al.*, 2017](#)). Table 2 also shows that the FFA value and acid number of esterified CPO decreased, to 1.8% and 4 mgKOH/gram, respectively. The FFA value is lower than 2% and the acid number is lower than 5 mgKOH/g, so that CPO can be processed through transesterification.

3.2. Biodiesel Yield

Figure 3 shows the yield of biodiesel from the four voltage treatments (0, 6, 9, and 12 volts) used in electrolysis. Analysis of variance showed that voltages 6 and 9 had a significant effect on yield, but voltage 12 had no effect on yield.

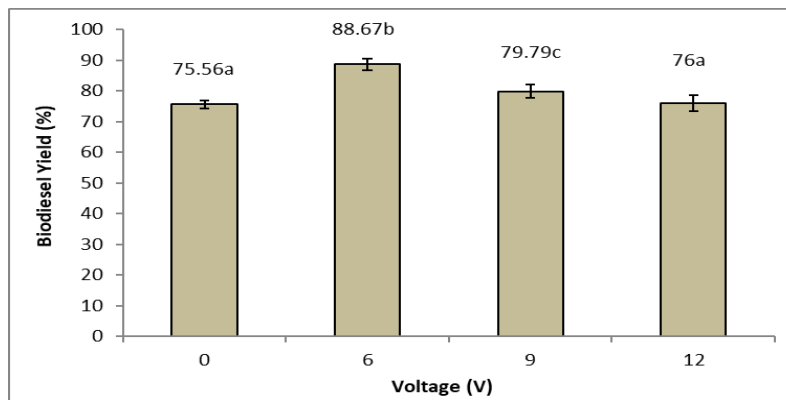


Figure 3. Effect of voltage on the biodiesel yield. (Values followed by the same letters are not significantly different at $\alpha = 5\%$).

The highest yield value occurred with electrolysis treatment at 6 V voltage, namely 88.67%, while the yield value of biodiesel without electrolysis treatment was 75.56%. The yield value increased to 13.11%. This improvement can be caused by the presence of OH^- ions produced at the cathode during the electrolysis process and can ultimately increase the yield value. The electrolysis process can also increase the conductivity so that the FFA in oil will easily decompose into biodiesel through reaction with methanol and facilitate the transesterification reaction. This is in line with (Putra & Putra, 2011). OH^- ions can also function as base catalysts during transesterification (Moeksin *et al.*, 2017). The KOH used in this study will combine with OH^- so that the transesterification process will increase thereby increasing the yield value. In this study, the yield value at 12V voltage was 76%, still higher than the results of research (Moeksin *et al.*, 2017), which was 38.3%. However, an excessive increase in voltage will actually result in an excessive number of OH^- ions resulting in a high saponification reaction and a decrease in the yield of biodiesel (Putra & Putra, 2011). The transesterification reaction of triglycerides into methyl esters or biodiesel is presented in the following sketch .

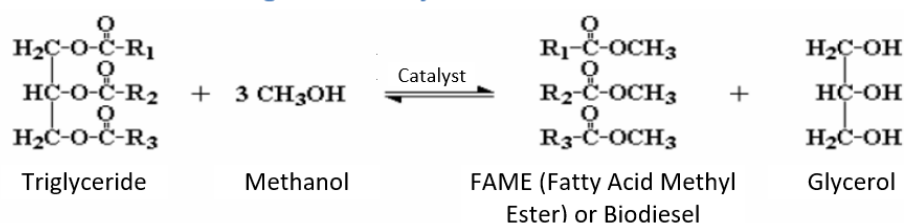


Figure 4. The process of transesterification of triglycerides into biodiesel (Hadrah *et al.*, 2018)

3.3. Biodiesel Acid Number

The effect of voltage during electrolysis-aided transesterification on the acid number of resulted biodiesel is presented in Figure 5. Analysis of variance showed that applying a voltage of 6V significantly affected the acid number, but increasing more voltage had no further effect on the acid number. The results of the analysis of the biodiesel acid number in this study showed that the acid number of biodiesel generated from the electrolysis treatment was in the range of 0.99 – 1.1 mgKOH/g, while the value of that without electrolysis was significantly higher, namely 1.93 mgKOH/g.

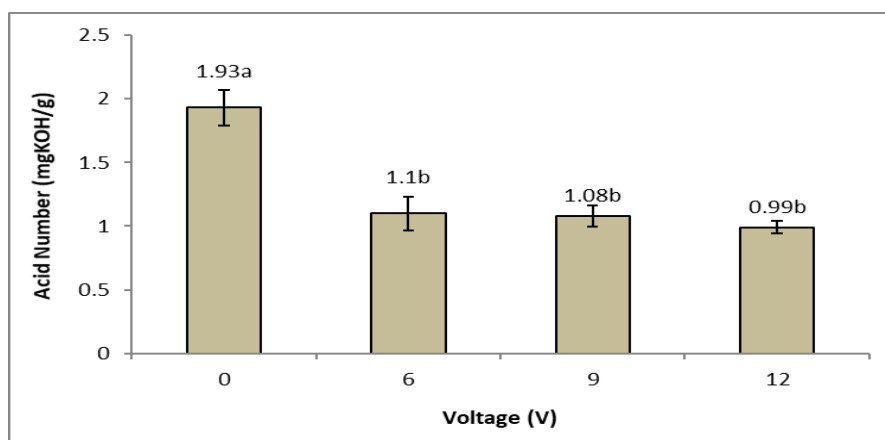


Figure 5. Effect of voltage on the acid number of biodiesel. (Values followed by the same letters are not significantly different at $\alpha = 5\%$).

The value of the acid number produced does not yet meet the biodiesel standard, which is a maximum of 0.5 mgKOH/g. Therefore, further treatment is needed to reduce acid number of biodiesel such as the use of an appropriate acid catalyst during the esterification process. Increasing the voltage seems to be able to reduce the value of the acid number. Biodiesel with low acid number is good for the use of the engine in suppressing the occurrence of corrosion in the engine, with the lower the acid number in biodiesel, the engine will have a lower risk of corrosion. The role of electrolysis in reducing the acid number in biodiesel is by producing H^+ ions at the anode which functions the same as an acid catalyst in reducing the acid number during the transesterification process (Putra *et al.*, 2015).

The raw material in the manufacture of biodiesel also affects the value of the biodiesel acid number. Raw materials having high acid number value will produce biodiesel with a high acid number as well. There are also several other factors that affect the level of biodiesel acid number, namely the type of acid catalyst during the esterification process and the biodiesel storage system (Kasim *et al.*, 2016). Biodiesel with high acid number will cause corrosion in the engine and also cause an oxidation reaction in the engine (Setiawan *et al.*, 2017).

3.4. Kinematic Viscosity of Biodiesel

The kinematic viscosity of biodiesel in relation with voltage variation was presented in Figure 6. Analysis of variance showed that applying voltage significantly affected the viscosity at voltages 9 and 12, but had no effect on voltage 6.

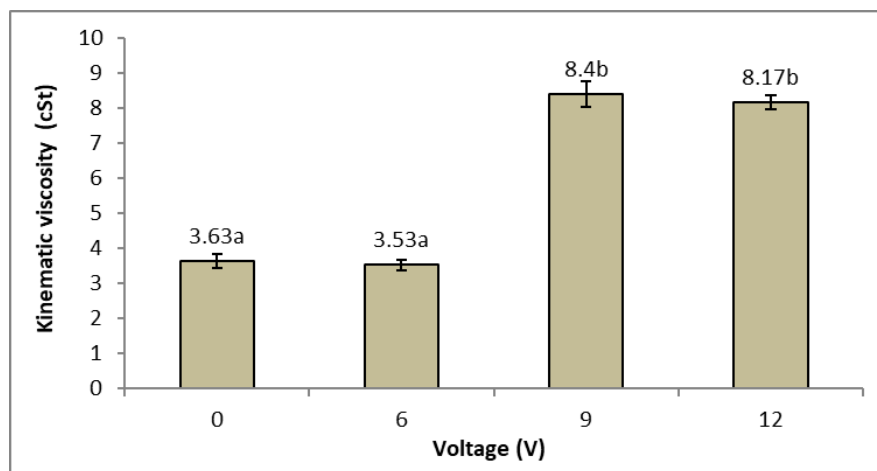


Figure 6. Effect of voltage on the kinematic viscosity of biodiesel. (Values followed by the same letters are not significantly different at $\alpha = 5\%$).

The viscosity of fuel must have appropriate value to get perfect engine combustion efficiency. The expected viscosity should not be too high or too low. A high viscosity will inhibit the pumping system resulting in the imperfect carburizing, while a low viscosity will cause the engine to heat up faster and wear out quickly because of the low level of lubrication. This is why the viscosity value is very important because it greatly affects the performance of the injector in the engine (Shimmamah, 2017).

The results of the kinematic viscosity analysis of the biodiesel showed that the highest value resulted from the use of electrolysis with a voltage of 9V, namely 8.4 cSt, while the lowest kinematic viscosity value resulted from the use of no electrolysis, namely 3.4 cSt. The value of the kinematic viscosity of biodiesel that has met the

biodiesel standard is only at the use of 6V electrolysis and without the use of electrolysis. The range of values that have been determined by the biodiesel standard is 2.3 cSt – 6.0 cSt. The value of kinematic viscosity increased greatly at the application voltage of 9V and 12V. This indicates that the use of electrolysis can increase the kinematic viscosity value of the biodiesel. The use of electrolysis can increase the saponification reaction which can actually increase the viscosity of biodiesel (Lin & Ma, 2022). Raw materials are also a factor causing the level of biodiesel viscosity. It is well known that CPO has a high kinematic viscosity value that produce biodiesel with high kinematic viscosity (Kurniawati, 2021).

The content of FFA in vegetable oil can also affect the kinematic viscosity of biodiesel. A high FFA value will produce biodiesel with a high kinematic viscosity, and vice versa. This is caused by an imperfect transesterification reaction that interferes with the biodiesel conversion process and also damages the quality of biodiesel in terms of its viscosity (Kartika *et al.*, 2012).

3.5. Density

Density is one of the important properties of biodiesel fuel. The results of the biodiesel density analysis in this study can be seen in Figure 7. The analysis of variance shows that applying a voltage has a significant effect on density, and an increase in voltage also has a significant effect on density.

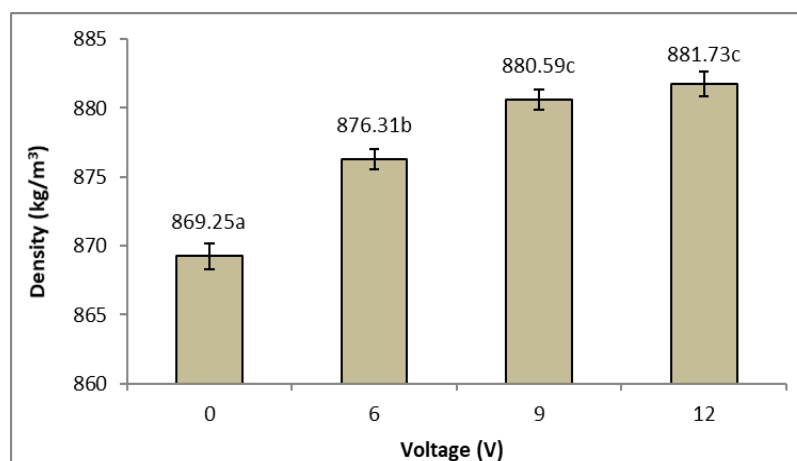


Figure 7. Effect of voltage on the density of biodiesel. (Values followed by the same letters are not significantly different at $\alpha = 5\%$).

The density with the highest value was obtained from the use of electrolysis at a voltage of 12V, which was 881.73 kg/m³, while the lowest value was found in biodiesel which was produced without the use of electrolysis, which was 869.25 kg/m³. All density values produced have met the SNI biodiesel standard, namely 850 – 890 kg/m³. The density value in biodiesel continues to increase when the amount of voltage is added, in other words the density value is directly proportional to the given voltage value. The amount of base catalyst given during transesterification can affect the density value, because a saponification reaction may occur between the base catalyst and the FFA contained in CPO during the transesterification process. Other components contained in biodiesel due to the reaction of KOH with FFA can also cause the density value of biodiesel to be high (Adhani *et al.*, 2016). The OH⁻ ion produced from the cathode during the electrolysis process can help the KOH catalyst during transesterification which will increase the saponification reaction, with this the

components contained in biodiesel such as soap will increase and the density value will also increase (Atina, 2015).

3.6. Biodiesel Moisture Content

The moisture content in biodiesel is expected to have a low value and can meet the SNI biodiesel standard, which is a maximum of 0.05%. The moisture content of biodiesel produced in this study can be seen in Figure 8. Analysis of variance showed that applying a voltage significantly affected the water content, and increasing the voltage also significantly affected the water content.

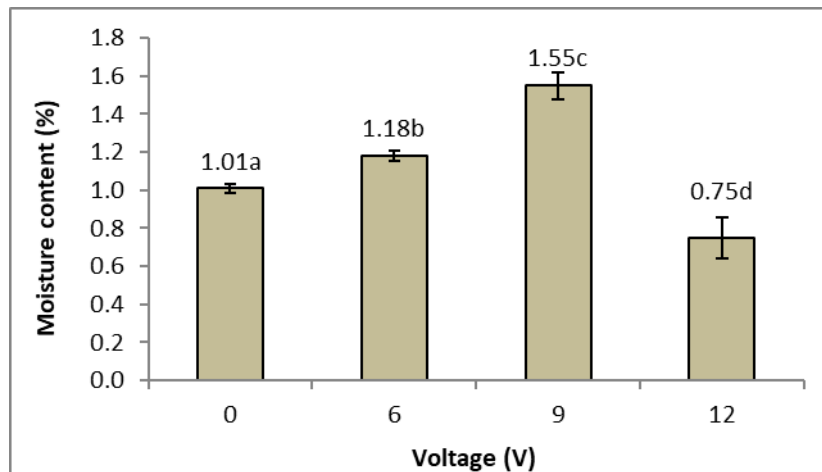


Figure 8. Effect of voltage on the moisture content of biodiesel. (Values followed by the same letters are not significantly different at $\alpha = 5\%$).

Analysis of the value of the moisture content of biodiesel in this study showed that the highest value was found in the use of electrolysis at a voltage of 9V, which was 1.55%, while the lowest moisture content resulted from the use of electrolysis at a voltage of 12V, which was 0.75%. The moisture content value obtained in this study is still too high, even for the lowest moisture content value obtained in this study and still does not meet the SNI biodiesel standard. The high moisture content can be caused by the heating process that is less than optimal. Corrosion in the engine will very easily occur if the moisture content in biodiesel is too high, this is due to the hydrolysis process that occurs so that it can increase the acid number in biodiesel (Hendra *et al.*, 2016). Therefore, further processes are needed to reduce the moisture content in biodiesel by heating the biodiesel to the maximum.

3.7. Biodiesel Flash Point

The flash point is analyzed to find out what is the lowest temperature for biodiesel to cause a fire or to burn. The results of the flash point analysis of biodiesel in this study can be seen in the graph in Figure 9. Analysis of variance showed that the applied voltage significantly affected the flash point at voltages 6 and 12, but had no effect on voltage 9. The flash point determine the resistance of biodiesel when heated. This test serves to maintain the safety of the engine, because the higher the flash point of biodiesel it will further increase the safety of the engine to use because it can minimize the explosion that occurs in the engine during heating. The high flash point of biodiesel is determined by the low level of fatty acids in biodiesel (Permana *et al.*, 2020).

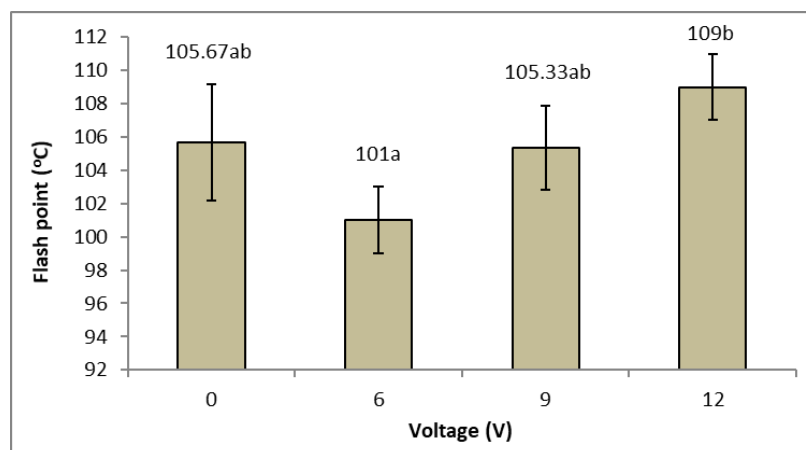


Figure 9. Effect of voltage on the flash point of biodiesel. (Values followed by the same letters are not significantly different at $\alpha = 5\%$).

The results of the flash point analysis of biodiesel in this study showed that the highest flash point value resulted from the use of electrolysis at a voltage of 12 V, which was 109 °C, while the lowest flash point value was found in biodiesel which was produced using electrolysis at a voltage of 6V, which was 101 °C. The use of electrolysis can help the transesterification process so that the biodiesel yield will be better. This can lead to an increase in the flash point value of biodiesel (Lin & Ma, 2022). All biodiesel produced from all types of methods given has a flash point value that meets the SNI biodiesel standard, which is at least 100 °C.

4. CONCLUSIONS

The electrolysis process with voltage variations 6, 9, and 12 significantly affected the biodiesel yield, acid number, kinematic viscosity, density, water content, and flash point of the biodiesel produced. The resulting biodiesel has an acid number ranging from 0.99 – 1.1 mgKOH/gram, kinematic viscosity 3.53 – 8.4 cSt, density 876.31 – 881.73 kg/m³, moisture content 0.75 – 1.18% and flash point 101 – 109 °C. None of the acid numbers and water content meet the biodiesel requirements. At voltage 6, the highest biodiesel production was obtained, namely 88.67%, and the viscosity that met the biodiesel requirements was 3.53 cSt, so it was the most recommended for use.

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