



# Properties of Biodiesel Purified by Membrane Technology

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## **Authors' contributions**

*This work was carried out in collaboration among all authors. Author YP designs studies, analyzes data, wrote the protocol, wrote the first draft of the manuscript and completes final manuscripts. Author YS conducted data collection in the laboratory, authors LB analyzed physical and chemical properties. All authors have agreed the final manuscript.*

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## **ABSTRACT**

Membrane technology is the most effective technology in the process of separation and purification because the separation of components can occur to the molecular level. Therefore the application of membrane technology in the biodiesel production process can provide high-purity biodiesel quality. In this research, the process of separating and refining palm oil biodiesel does not use the washing process, but it uses membrane separation technology. The membrane used is the ceramic ultrafiltration membrane 0.02  $\mu\text{m}$ . The purification process was carried out at temperature 70°C and pressure 12 Psi (0.86 bar), flow rate of 39.53 L/min, circulation time of 3 hours with a feed of 10 L. After purification, an obtained biodiesel has physical properties as follows: Purity level 97.63% mass (total ester content) and 97.02% mass (methyl ester content), kinematic viscosity at 40°C is 5.70 (cSt), density 0.86 (g/cm<sup>3</sup>), acid number 0.45 (mg KOH/g) and the saponification number 206.45 mg KOH/g. The values of the physicochemical properties have met Indonesian National Standard (SNI).

**Keywords:** Biodiesel; purification; membran separation.

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## 1. INTRODUCTION

Biodiesel is a type of alternative diesel fuel that can be synthesized from various types of vegetable oils such as jatropha oil, palm oil, used cooking oil, coconut oil, palm oil, other vegetable oils, and animal fats [1]. The use of biodiesel as an alternative diesel fuel has a positive impact on reducing the use of fossil energy and reducing exhaust emissions. In addition, combustion residues are easily degraded and renewable so they are more environmentally friendly [2].

Generally, biodiesel is produced through the transesterification reaction process, which is an ester formation reaction with the help of an acid, base, or enzyme catalyst. To accelerate the process of transesterification, catalysts derived from alkali group elements such as NaOH, KOH,  $\text{CH}_3\text{ONa}$ , and  $\text{CH}_3\text{OK}$  catalysts can provide high conversion and reaction rates [3]. Besides, several other catalysts that have also been developed for the use are super base CaO and zeolite which can be synthesized from materials that are available in the abundance of nature [4]. Transesterification reaction between triglycerides and methanol produces a mixture consisting of biodiesel, impurity components such as soap, catalyst, glycerol, the remaining methanol, and unreacted triglycerides.

After the transesterification process, the purification process is needed to obtain biodiesel products that are suitable for use as fuel in diesel engines. The common biodiesel purification processes used are the wet washing process and dry washing process [5]. Wet washing uses water, acid solutions, and organic solvents as the main variables in the process of biodiesel purification from impurities [6]. While dry washing is the development of biodiesel purification technology without the use of water. The removal of impurities from biodiesel is carried out using adsorbents and acid resins (cation exchangers). The most common adsorbent used in the process of purifying dry washing is Magnesium (Magnesium Silicate) with cation exchanger resin from polymer matrix, counter cation ( $\text{H}^+$  or  $\text{Na}^+$ ) [7].

The wet and dry washing purification methods can produce high quality diverse results. But both of these methods have weaknesses. The use of large amounts of water in the wet washing method will result in large liquid waste, higher energy consumption, and higher production

costs. It can reduce the yield value of methyl esters produced due to repeated dumping. For the dry washing method, the use of adsorbents that cannot be regenerated, the use of supporting chemicals such as cation exchanger resins will increase production costs. Apart from that, some intermediate reactions can reduce the yield of biodiesel produced [3]. In general, the constraints of biodiesel production for plant scale are the method of separation and purification processes which are very complex, so that it requires high production costs. Thus the biodiesel production process is considered ineffective and not economical.

Advances in membrane technology provide a very promising solution for the biodiesel purification process. Membrane technology has attracted the interest of researchers because it can produce high purity and quality of biodiesel fuel, the process of separating and purifying biodiesel does not condense water so that the process does not produce liquid waste, easier process operations, and environmentally friendly [8].

For the biodiesel purification process, the most popular type of membrane developed is the Micro-Filtration (MF) and Ultra Filtration (UF) ceramics membranes. The advantages of ceramic membranes compared to polymer membranes are the level of mechanical strength and better structural rigidity, corrosion and thermal resistance, the stability of operating characteristics, can be regenerated gradually and resistance to bacterial attack [9].

Several researchers [3,9,10,11] have evaluated the results of the purification of biodiesel using microfiltration and ultrafiltration ceramic membranes. The results of their research concluded that the success of the biodiesel purification process was strongly influenced by several things including the type of membrane used, trans membrane pressure, flow rate, temperature and quality of raw materials. The use of an ultrafiltration ceramic membrane with a pore size of  $0.02 \mu\text{m}$  with a trans membrane pressure of 2 bar, a temperature of  $40^\circ\text{C}$  and a flow rate of 150 L/min produces biodiesel which has a free glycerol content of 0.007% wt with a permeate flux of  $9.08 \text{ kg/m}^2\text{h}$  [5].

The focus of this research is to find out the properties of biodiesel made from palm oil with high acid numbers, through the transesterification reaction, which is followed by

the purification process of biodiesel using an ultrafiltration ceramic membrane.

## 2. MATERIALS AND METHODS

Stages of research that have been carried out consist of, production of biodiesel from palm oil using a 10 L/batch capacity Biodiesel Pilot Plant, Purification Stage using ultrafiltration ceramic membranes arranged as in Fig. 1 and the analysis stage of biodiesel characteristics.

### 2.1 Biodiesel Production

The process of biodiesel production is carried out through a palm oil transesterification reaction with a mole ratio between oil and methanol 1: 6, using KOH catalyst as much as 0.2% by weight of oil, the reaction process is carried out at 60°C for 2 hours. The transesterification reaction takes place according to the reaction equation as shown in Fig. 2. Palm oil with water content <0.5 and FFA >2% as much as 4000.89 mL is poured into the reactor. 999.11 mL methanol and 0.2% by weight KOH catalyst were also added to the reactor. The transesterification reaction is carried

out at 60°C and the reaction time is 2 hours. After the reaction process is complete, continue with the recovery process of methanol at 80°C and 1 hour. Methanol is collected in a storage container. The reaction product is then passed into the separation column to separate the glycerol phase from the biodiesel phase. The biodiesel phase is flowed into the storage tank to be purified using membranes. Biodiesel production in this study was repeated up to eleven batches to meet the needs of raw materials in the purification process using membrane.

### 2.2 Purification Using Membrane

Crude biodiesel from storage tanks, flowed into the membrane to conduct the purification process. Membrane process conditions are set at 70°C as operating temperature with a flow rate of 39.53 L/m and a pressure of 12 psi (0.83 bar) with a circulation time of 3 hours. Pure biodiesel products are stored as permeates in pure biodiesel storage containers and residues are stored as retentates in residual storage containers.

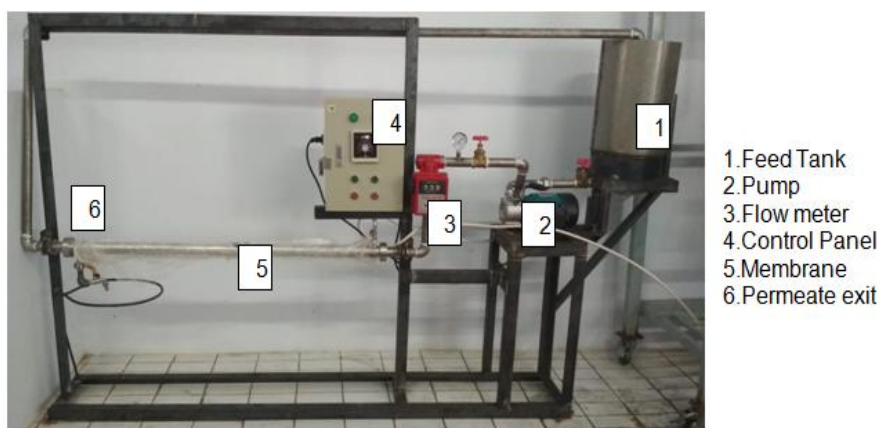


Fig. 1. Series of membrane circulation equipment for biodiesel purification

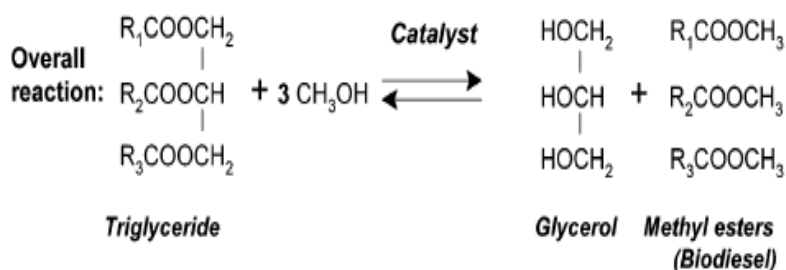


Fig. 2. Transesterification reaction

### 2.3 Analysis of Physical Properties of Biodiesel

Biodiesel product analysis is carried out to determine the acid number, saponification number, viscosity and density. GCMS analysis is carried out to determine the total methyl ester content in crude biodiesel and in purified biodiesel.

### 3. RESULTS AND DISCUSSION

The characterization of raw materials is carried out before the reaction process. Palm oil is analyzed to determine water content and levels of Free Fatty Acid (FFA). The results of the characterization of raw materials showed the value of water content 0.04% and FFA levels > 0.5. Based on the results of this characterization, the biodiesel synthesis process should be carried out by an esterification reaction and then a transesterification reaction [12,13,14]. But in this research the esterification process was not carried out, to determine changes in acid numbers and other properties of biodiesel produced from the purification process using membranes. In addition to determining the water content and FFA levels, acid number analysis, density saponification number and viscosity were also analyzed. The analysis results for each of these parameters are presented in Table 1.

#### 3.1 Biodiesel Production

The process of biodiesel production begins with preheating the mixture of oil, methanol and catalyst. Initial heating is done in stages by raising the process temperature from 30°C to 60°C. This gradual heating process is accompanied by a mixing process. After the reaction temperature reaches 60°C, the process temperature is maintained and the reaction is carried out for 2 hours. After the reaction process, proceed with the recovery process of methanol at 80°C for 1 hour. The reaction product is flowed into the separation column to separate the biodiesel phase from the glycerol phase. Methanol which has been successfully recovered, biodiesel and glycerol products are accommodated in storage containers. The results of biodiesel production are presented in Fig. 3.

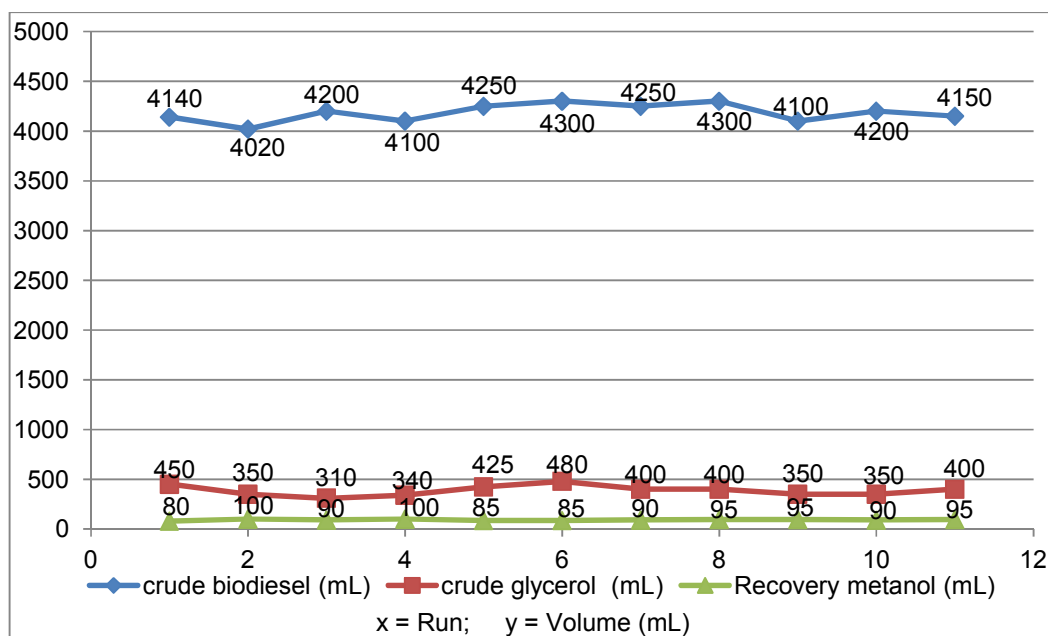
Based on the data in Fig. 3 it is known that the product from the transesterification process is crude biodiesel with an average yield of 4183 mL (yield 84%), crude glycerol with an average yield

of 387 mL and methanol recovery with an average yield of 91.36 mL. The maximum yield of crude biodiesel obtained at run 6 and 8 is 4300 mL (86% yield), however there are differences in the acquisition of by-products and methanol recovery. The difference in methanol recovery is caused by the evaporation of methanol during the recovery process [15,16]. The minimum yield of eleven experiments occurred on run 2 with the results of 4020 mL crude biodiesel (80% yield), 350 mL crude glycerol and 100 mL recovery methanol. Crude biodiesel produced was analyzed by acid number, saponification number, viscosity, density and chemical composition analysis. Chemical composition analysis uses GCMS to determine levels of ester and methyl esters components. The results of the analysis of the parameters mentioned above are presented in Table 2.

**Table 1. Characteristics of palm oil**

No	Properties	Value
1	Water content (% mass)	0.04
2	FFA (% mass)	1.28
3	Acid number (mg KOH/g)	2.81
4	Saponification number (mg KOH/g)	217.67
5	Kinematic Viscosity 40°C (sCt)	18.55
6	Density (g/mL)	0.88

The data in Table 1 and Table 2 show the change in acid number from oil to raw biodiesel is 2.81 mg KOH / g to 1.57 mg KOH / g. This means that there has been a decrease in acid number of 1.24 mg KOH / g. Even so, acid decline did not meet the acid number determined by the Indonesian National Standard (SNI), which is <0.5. The results of the analysis of the viscosity of raw biodiesel also showed a significant decrease of 13.75 sCt. This can be seen in Table 1 where the viscosity of palm oil is 18.55 sCt and after the transesterification process it becomes 5.70 sCt. This decrease in viscosity is one indicator of the ongoing transesterification reaction [12]. In addition, a decrease also occurred in the amount of saponification of 4.49 mg KOH / g and a density of 0.02 g / mL. The results of the GCMS analysis showed that the total esters and methyl esters in crude biodiesel were 92.75% and 89.67%, respectively. When compared with the total methyl ester required in SNI, which is ≥96.5%, the total value of methyl ester from crude biodiesel does not meet the requirements.



**Fig. 3. Result of the transesterification process**

**Table 2. Results of analysis of acid numbers, saponification numbers, viscosity, density and content of esters and methyl esters in crude biodiesel**

No	Properties	Value
1	Acid number (mg KOH/g)	1.57
2	Saponification number (mg KOH/g)	213.18
3	Kinematic viscosity 40°C (sCt)	4.80
4	Density (g/mL)	0.86
5	Total Ester Content (%)	92,75
6	Total Methyl Ester Content (%)	89,67

### 3.2 Biodiesel Purification

Crude biodiesel as much as 10 Liters is put into the process storage tank, then the membrane operating conditions are set at 70°C and a pressure of 0.86 bar. The purification process is carried out by circulating crude biodiesel in circulation through the membrane at a speed of 39.53 L/min. Pure biodiesel products are stored as permeates and the circulation process is carried out for 3 hours. Then the product is analyzed its physical and chemical properties, as has been done with crude biodiesel. The results of the analysis of the characteristics of pure biodiesel are presented in Table 3.

The value of the biodiesel acid number of the purified product with a membrane of 0.45 mg KOH/g as presented in Table 3 shows a

significant decrease with the value of the crude biodiesel acid value in Table 2 which is 1.57 mg KOH/g. This value is already lower than the value required in SNI. The high acid number in crude biodiesel is caused by high palm oil acidity. Generally for high acidic vegetable oils, esterification reaction is needed before transesterification [14]. But in this study no esterification reaction was carried out. The process of purification of biodiesel by using a membrane in this study was proven to reduce the acid number to smaller than the acid number required by SNI [3]. Membrane selectivity in the separation of biodiesel components from other components including free fatty acids is very good.

**Table 3. Properties of biodiesel resulting from the membrane purification process**

No	Properties	Value
1	Acid number (mg KOH/g)	0.45
2	Saponification number (mg KOH/g)	206.45
3	Kinematic viscosity 40°C (sCt)	4.80
4	Density (g/mL)	0.86
5	Total Ester Content (% mass)	97.63
6	Total Methyl Ester Content (% mass)	97.02

The effectiveness of the purification process using a membrane also appears in the increase in the total content of methyl ester in the purified biodiesel which is 97.02% (previously only 89.67%), the result is already higher than the

requirements in SNI. When compared with the total ester content of 97.63%, it can be seen that the total ester content is almost equal to the total content of methyl ester (biodiesel). The value of kinematic viscosity and the density of biodiesel from purification did not change with the value of biodiesel before purification.

Based on the facts in the analysis of the characteristics of biodiesel purification results with the membrane, it can be seen that the use of membranes in the process of biodiesel purification can improve the quality of biodiesel to meet the requirements set by SNI [5]. Another advantage of using membranes in this study is that the process is shorter because it does not require washing and evaporation stages. Thus the process becomes more effective and economical.

#### 4. CONCLUSION

Based on the results of research that has been done it can be concluded that:

1. The Properties of Biodiesel obtained through the purification process using membranes in this study consist of acid number 0.45 mg KOH/g, saponification number 206.45 mg KOH/g, kinematic viscosity 4.80 sCt, density 0.86 g/ml. These properties meet the quality standards specified in the Indonesian National Standard.
2. The use of membranes in the purification process can increase the total content of methyl esters in biodiesel from 89,67% to 97.02%.
3. The use of membranes in the process of biodiesel production can reduce the stages of the process in biodiesel production, namely the esterification stage, the washing stage and the evaporation stage.

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#### COMPETING INTERESTS

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