



Concentration Effect of MgO/K₂O Catalyst on Transesterification of Castor Seed (*Ricinus communis*) Oil

Pengaruh Konsentrasi Katalis MgO/K₂O pada Transesterifikasi Minyak Biji Jarak (*Ricinus communis*)

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Received: November 30, 2024 Accepted: July 18, 2024 Published: October 31, 2024

ABSTRACT

Fatty acid methyl esters can be resulted from the transesterification of castor seed oil with methanol using a base catalyst. To the improving of the methyl ester quality, a catalyst was developed by modification of MgO/K₂O. The aims of this study were: to determine the concentration effect of MgO/K₂O on the yield of the castor seed oil-based methyl ester, and to determine their characteristics. The procedures included: (1) isolation of castor seed oil, (2) determination of free fatty acid (FFA) levels of castor seed oil, (3) synthesis of MgO/K₂O (4) esterification of castor seed oil with H₂SO₄ catalyst (5) transesterification of castor oil with methanol and various concentration of the MgO/K₂O (1%, 2%, 3%), and (6) the characterization included: density, viscosity, acid number, refractive index and water content. The results showed that the concentration of MgO/K₂O showed an effect on the synthesis of methyl esters from castor seed oil, where the best condition was achieved at 2% of MgO/K₂O with the yield of 97.99%. The methyl ester characters were density of 1.0244 g/mL, viscosity of 22.531 cSt, acid number of 2.81 g KOH/g methyl ester, refractive index of 1.456, and water content. 0.035%.

Keywords: castor seed oil, esterification, heterogeneous catalyst, methyl ester, transesterification

INTRODUCTION

Crude oil is a non-renewable fossil fuel which is available after thousands of years, while the need for fuel oil is currently very high (Qasimi, Isazade, & Toomanian, 2022). As an alternative policy, many fuels are produced from biomass, so the development of this renewable energy fuel is quite rapid. This relates to the commitment of world citizens in

terms of reducing gas emissions, pollutants and the greenhouse effect (Perea-Moreno, Samerón-Manzano, & Perea-Moreno, 2019). So, renewable energy as fuel appears in the form of bio-fuel, bio-avtur, and biodiesel (Zou, Zhao, Zhang, & Xiong, 2016).

Biodiesel is a fuel consisting of a mixture of mono-alkyl esters from fatty acids as a substitute for diesel. Biodiesel is a renewable raw material and is also more environmentally

friendly (Wahyudiantoa, Nisa'Alfikrya, & Taufik, 2020). Biodiesel is a renewable fuel processing from renewable sources, generally plant oils and animal fats (Zhang et al., 2019). Biodiesel has a high lubricating effect that can extend engine life and has a high cetane number (> 50). The higher the cetane number, the safer the exhaust emissions will be (Aziz, Aisyah, & Ilyas, 2016).

Processing of biodiesel from plant materials has been widely used, but the price of plant materials is expensive. The available materials have a higher production cost than biodiesel and most of it is for food purposes. One material that has bright prospects to be biodiesel raw materials is castor plant (Oko & Feri, 2019) because there is no need for food.

Biodiesel can be produced through transesterification reaction, namely the reaction between triglycerides from vegetable oils and alcohol and converts them into methyl esters (biodiesel). The trans-esterification can be effective if the raw material of vegetable oil has a free fatty acid (FFA) content below 2% (Rhofita, 2015). Castor seed (*Ricinus communis*) oil is one of the potential vegetable oils because it is non-edible oil (Setiadji et al., 2017).

In industrial processes, a catalyst is very important where it can accelerate a reaction. Currently, homogeneous catalysts have several drawbacks, which are difficult to separate from the resulting solution. The catalyst cannot be reused and will become a hazardous waste if disposed of directly. Besides, the catalyst can react with Free Fatty Acid (FFA) to form soap, which will complicate the separation of glycerol and reduce biodiesel yield (Santoso, Sumari, & Asrori, 2022). To reduce the weakness, a heterogeneous catalyst is applied which are more environmentally friendly, stable at high temperatures, have large pores and are relatively inexpensive, so that the process can take place more quickly and can minimize production costs. In the transesterification reaction, heterogeneous catalysts can reduce wasted water and the catalyst can be reused for further processes (Kurniasih & Pardi, 2018).

Research on the synthesis of biodiesel using castor seed oil as raw material needs to be carried out as the development of biodiesel following the rapid development of science. Previous studies showed that castor seed oil-

derived biodiesel from transesterification catalyzed by MgO/ γ -Al₂O₃ (Navas, Lick, Bolla, Casella, & Ruggera, 2018), MgO/CaO nanorods (Abukhadra, Mohamed, El-Sherbeeny, Soliman, & Abd Elgawad, 2020), carbon-based MgO (Du et al., 2019), CaO/K₂O (Santoso, Sumari, Asrori, & Januarti, 2023). These studies resulted a standard biodiesel, but the catalysts need to be modified. To the best knowledge of authors, there is no report using MgO/K₂O in transesterification of castor seed oil. This study aims to determine the effect of MgO/K₂O catalyst concentration on the yield of methyl ester resulting from trans-esterification of castor seed oil, and to determine the character of biodiesel trans-esterified from castor seed oil with MgO/K₂O catalyst.

METHODOLOGY

Tools

The tools in this study were a set of reflux instruments (Sumari, Santoso, & Asrori, 2021), thermometers, glass tubes (pyrex), burettes (pyrex), separatory funnels (pyrex), Beakers (pyrex), measuring cups (pyrex), three-neck flasks (pyrex), Erlenmeyer (pyrex), pipettes, volume pipettes (pyrex), and desiccators. The non-glass tools in this study were hoses, aluminum foil, label paper, filter paper, pycnometer (pyrex), stopwatch, universal indicator paper, mortar, clamps, staves, filler, evaporating cups and bottles. The instruments used in this study included the Oswald viscometer (pyrex), hot plate stirrer (IKA C-MAG HS7), furnace (Thermolyne Benchtop Muffle Furnaces FB1410M-33), refractometer Abbe (Abbe 2WA) monochromatic Refractometer) and oven (memmert).

Materials

The materials in this study were distilled water, diethyl ether (Merck), KOH p.a. (Merck), phenolphthalein indicator, 96% methanol (Merck), H₂SO₄ p.a. (Merck), MgO powder (Sigma Aldrich), and castor seed oil.

Procedures

The procedure for determining free fatty acid levels was carried out by: ± 1 gram of oil and 10 mL of methanol p.a were put in Erlenmeyer. It was then heated until boiling. After cooling, the mixture was titrated with 0.008 N KOH solution with 3 drops of phenolphthalein indicator. The amount of KOH

used for the titration was recorded. This test was in duplicate (Santoso, Wijaya, et al., 2023).

The procedure for preparing the MgO/K₂O catalyst was carried out by: MgO powder (100 grams) was heated at 300°C for 2 hours. Next, the powder was cooled for 24 hours. After cooling, the MgO powder was dissolved with 10% alcoholic KOH with a mass and volume ratio being 2:1. Then, this mixture was heated while stirring for 60 minutes. After that, the mixture was evaporated. Furthermore, the mixture was in a furnace for 90 minutes at 300 °C.

The esterification process was carried out by: castor seed oil (200 grams), 1 mL of H₂SO₄ p.a solution, and 15 mL of methanol were put into a three-neck flask with heating at 70 °C. The mixture was then refluxed for 2 hours. After that, 7.5 mL of methanol and 0.6 mL of H₂SO₄ p.a were added to continue the reflux process for 1 hour. The results were cooled and allowed to stand in a separatory funnel until they separated into two layers. The top layer was a mixture of triglycerides (oil) and fatty acid methyl esters, while the bottom layer contains concentrated H₂SO₄ and unreacted methanol. The top layer was separated, washed with a little distilled water, and heated on a hot plate at ±90°C. This procedure was carried out three times (Santoso, Wijaya, et al., 2023).

The transesterification procedure was carried out with a mole ratio of oil: methanol of 1:6. A total of 20 g of esterified castor seed oil samples were put into a three-neck flask. The oil was heated at ± 65°C. After the temperature reached 65°C, 5 mL of methanol was added to the three-neck flask for each addition of MgO/K₂O catalyst with a concentration of 1%, 2%, 3% w/w of MgO/K₂O. Furthermore, the transesterification was carried for 120 minutes. When finished, the result was put into a separatory funnel until two layers formed for 24 hours. The top layer was methyl ester while the bottom layer was glycerol, oil and catalyst. The top layer was separated and washed with warm water to remove residual glycerol and MgO/K₂O catalyst until a neutral pH. The methyl ester obtained was characterized according to quality standards as biodiesel fuel (Santoso, Sumari, Asrori, & Wele, 2024).

The characterization carried out on the results of this study included: acid number, density, and viscosity which were compared to the Indonesian National Standard (SNI) for

biodiesel issued by BSN with SNI 7182:2015 (Santoso et al., 2024).

The density test was carried out by weighing the empty pycnometer and recording the weight. Next, the methyl ester sample was added until it was full, weighed and the weight was recorded. This test was carried out twice. Density values can be determined by the following equation:

$$\rho \left(\frac{g}{mL} \right) = \frac{M_{\text{picnometre + methyl ester}} - M_{\text{picnometre}}}{\text{Volume methyl ester}}$$

The viscosity test was carried out by placing distilled water in the Ostwald viscometer tube and recording the flow time to pass the distance between the two marks on the viscometer using a stopwatch as a comparison. The same treatment was carried out on the methyl ester and the flow time was also recorded. This test was carried out twice. The value of viscosity can be determined by the following equation:

$$\frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2}$$

Information: η_1 = viscosity of methyl ester (cSt), η_2 = viscosity of distilled water (cSt), ρ_1 = density of methyl ester (g/mL), ρ_2 = density of distilled water (g/mL), t_1 = flow time of methyl ester (s), t_2 = time aquadest flow (s).

The acid number test was carried out by weighing ±1 g of the methyl ester sample and 10 methanol p.a. was put into Erlenmeyer. Next, the mixture was heated on a hot plate until boiling. After cooling, this solution was added 3 drops of phenolphthalein indicator and titrated using 0.01 N KOH solution (which has been standardized with oxalic acid) until the color changes from colorless to pink. This test was carried out twice. The value of the acid number can be determined by the following equation:

$$\text{Acid number} = \frac{V \times N \times \text{Molar mass KOH}}{\text{sample mass}}$$

Note: V = volume of KOH (mL) N = normality of KOH (N) Molar mass of KOH = 56.1 (g/mol) Sample mass = sample weight (g)

Before determining the refractive index, it is necessary to clean the refractometer glass with alcohol. Then the synthesized methyl ester was dripped on the refractometer glass and closed.

The reading of the refractive index value was carried out by adjusting the light and dark lines to the exact cross position of the observation lens. Furthermore, the temperature and refractive index values were recorded as data to determine the refractive index value at 25°C which follows the following equation:

$$n = n' + k(T' - T)$$

Note: n = value of refractive index at 25°C, n' = value of refractive index at temperature at observation, k = correction factor (0.00045) T' = temperature at observation, T = temperature at 25°C. This test was carried out twice.

The water content test was carried out by weighing 3 grams of the methyl ester sample and placing it in a cup (the mass was known). Next, the sample and cup were dried in oven at 105 °C for 3 hours. After it, the cup and sample were cooled and weighed and dried again until a constant mass was obtained. The water content was calculated using the following formula:

$$\text{water content} = \frac{W - (W_1 - W_2)}{W} \times 100\%$$

Information: W = sample weight (g), W₁ = sample weight + cup after heating (g), W₂ = empty cup weight (g). This test was carried out twice.

RESULTS AND DISCUSSION

Characterizations of castor seed oil can be seen in Table 1.

Table 1. Characteriation of castor seed oil

Characterization	Value
Free Fatty Acid (%)	4.97
Density (g/mL)	1.074
Viskosity (cSt)	679.7

Based on Table 1, the free fatty acid content does not meet the requirements for the transesterification process because it is above 2%, so it must be esterified first (Sumari et al., 2021). Reducing the free fatty acid content was carried out in order to meet the 2015 SNI biodiesel methyl acid number (maximum 0.5%). The density and viscosity value of castor seed oil is still too high to be used as biodiesel.

Besides, the high viscosity must be reduced by trans-esterification.

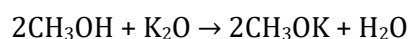
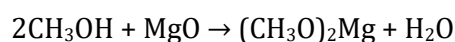
The esterification results can be observed in Figure 1.



Figure 1. Castor seed oil after esterification

In the synthesis of biodiesel, the using of a catalyst is highly recommended because the catalyst plays a role in accelerating the reaction. Synthesis of methyl ester from castor seed oil was carried out at 65°C. The mole ratio of oil is 1 : 6 to the mole of methanol with a catalyst ratio of 1%, 2%, 3% w/w oil MgO/K₂O, and the reaction time is 2 hours. While the oil used is castor seed oil resulting from the esterification process 3 times.

The transesterification process begins by heating 20 g of oil at 65 °C, then adding 1%, 2%, 3% MgO/K₂O catalyst, followed by the addition of 5 mL of methanol. This reaction takes place in equilibrium. The use of excess methanol in this reaction is intended so that the reaction proceeds towards the product, namely methoxide. The MgO/K₂O catalyst will react with methanol to form methoxide in the following reaction (Oko & Feri, 2019).



The synthesis will produce methyl esters. The results of the trans-esterification reaction can be seen in Figure 2.

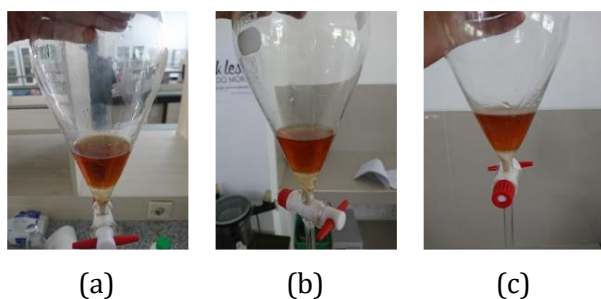


Figure 2. Results of Transesterification of Castor Seed Oil with (a) 1% MgO/K₂O, (b) 2% MgO/K₂O, (c) 3% MgO/K₂O

Based Figure 2. the results of the reaction form two layers. So, the separation was success. The top layer (methyl ester) was taken and washed using distilled water. The purpose of this washing is to remove the remnants of the catalyst, glycerol, oil and methanol which are still contained in the synthesized methyl ester. The yield of transesterification results of castor seed oil is shown in Table 2.

Table 2. Characterization of Methyl Esters from Castor Seed Oil

Charac ter	Oil and methanol mole ratio (1:6), temperature 65°C, 2 hours			
	MgO/ K ₂ O 1%	MgO/ K ₂ O 2%	MgO/ K ₂ O 3%	SNI 7182:2 015
yield (%)	96.57	97.99	96.62	
Acid numbe r (mg KOH/g)	2.95	2.81	3.00	Max 0.5
Density (g/mL)	1.0303	1.0244	1.0245	0.850 – 0.890
Viscosi ty (cSt, 26°C)	32.244	22.531	24.401	2.30 – 6.00
Refract ive index	1.461	1.456	1.456	Max 1.45
Water content (%)	0.08	0.035	0.04	Max 0.05

Based on Table 1, The acid number is an indicator of biodiesel quality. A high acid number value indicates damage or a decrease in the quality of biodiesel due to oxidation. The low acid number indicates that the fuel does not easily corrode the metals through which the fuel passes when used (Santoso, Agustin, et al., 2022). The biodiesel quality standard according to SNI 2015 requires the acid number of biodiesel not to exceed 0.5 mgKOH/g biodiesel while the acid value of the methyl ester produced from synthesis does not meet SNI 2015 because it is more than 0.5 mgKOH/g, but the values is highly reduced.

The density is affected by the fatty acid composition and purity. Density according to SNI 2015 is in the range of 0.850-0.890 g/mL. Methyl ester which has a density value exceeding the quality standard causes engine wear, so that it can accelerate the occurrence of damage to the engine. In this study, the densities are higher than SNI's density.

Viscosity (thickness) is one of the main parameters in determining the quality of methyl ester, because it has a major influence on the effectiveness of methyl ester as fuel. The higher the molecular weight, the greater the viscosity (Nasrudin, 2018). The viscosity of the methyl ester was measured at 26°C. The value of the viscosity of the methyl ester did not meet the SNI, but the value has been highly reduced. If the viscosity is too high, it will be difficult for the methyl ester to flow which will cause damage to the engine. The higher the molecular weight, the greater the viscosity and the greater the viscosity (Hanif, Nisar, Akhtar, Nisar, & Rashid, 2018).

The temperature factor can affect the value of the refractive index. The double bond in the oil broken by oxidation process causes the oil to become saturated and will lower the refractive index value of the methyl ester (Fitriana & Fitri, 2019). The Refractive Index of the methyl ester with 1% MgO/K₂O catalyst was measured at 28.9°C, the Refractive Index of methyl ester with 2% MgO/K₂O catalyst was measured at 29.1°C and the Refractive Index of methyl ester with 1% MgO/K₂O catalyst was measured at 30°C. The refractive index of the synthesized methyl ester that meets SNI 2015 is methyl ester with 2% and 3% MgO/K₂O catalyst with maximum limit (Santoso et al., 2024).

The maximum water content based on SNI is a maximum of 0.05%. High water content in

biodiesel will cause a decrease in heat during combustion, and be corrosive if it reacts with sulfur and will become a pollutant for biodiesel (Hoang, Tabatabaei, & Aghbashlo, 2020). The water content of the synthesized methyl ester that meets SNI is methyl ester with 2% and 3% MgO/K₂O catalyst, namely 0.035% and 0.04%.

Based on Tabel 1, the transesterification process may undergo a side reaction and a non-optimal reactor process affected the characteristics of the resulted biodiesel (Asrori, Santoso, Sumari, & Prakasa, 2023). Therefore, the methyl ester of this study cannot obtained the good quality, but the best condition was achieved at 2% of MgO/K₂O with the yield of 97.99%. For further study, optimalization process is necessary, such as process intensification, response surface methodology, optimalized MgO/K₂O, and optimalized the product separation.

CONCLUSION

Based on the results, The concentration of MgO/K₂O catalyst affects the synthesis of methyl esters from castor seed oil, where optimization conditions are achieved at a catalyst concentration of 2% MgO/K₂O with a yield of 97.99%, The character of the methyl ester synthesized from castor seed oil with the catalyst MgO/K₂O 1% has a density of 1.0303 g/mL, viscosity of 32.244 cSt, acid number of 2.95 g KOH/g methyl ester, a refractive index of 1.461, and a water content of 0.08%. The character of the methyl ester synthesized from castor seed oil catalyzed MgO/K₂O 2% has a density of 1.0244 g/mL, a viscosity of 22.531 cSt, an acid number of 2.81 g KOH/g methyl ester, an index of refraction of 1.456, and water content of 0.035%. While the character of the methyl ester synthesized from castor seed oil catalyzed MgO/K₂O 3% has a density of 1.0245 g/mL, a viscosity of 24.401 cSt, an acid number of 3.0 g KOH/g methyl ester, an index of refraction of 1.456, and water content of 0.04%.

ACKNOWLEDGMENT

Thanks to Universitas Negeri Malang for supporting this study and funding.

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