MOLAR RATIO VARIATION AND CATALYST TYPE ON BIODIESEL PRODUCTION FROM TRANSESTERIFICATION METHOD

Ajeng Anisa Dewi[1], Dessy Agustina Sari[2*], Muhammad Fahmi Hakim[3]
[1-3]Chemical Engineering Program, Universitas Singaperbangsa Karawang
[2]Department of Chemical Engineering, Universitas Diponegoro

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ABSTRACT

Biodiesel, a renewable fuel derived from vegetable oils, has gained attention as a sustainable alternative to fossil fuels. The study's goals are to find the best conditions for making biodiesel from corn oil using the transesterification method and to look at how different molar ratios and catalyst types affect the quality and yield of biodiesel. This study conducted the transesterification process using methanol, NaOH, and CaO as catalysts. The molar ratios of oil to methanol were 1:12, 1:15, and 1:18. The study conducted the reaction at 60°C for 120 minutes. The characteristics of the produced biodiesel were density, kinematic viscosity, and methyl ester content, and we compared the results to biodiesel quality standards. This study found that a molar ratio of 1:12 with CaO as a catalyst resulted in optimal biodiesel properties, including a density of 862.87±2.01 kg/m³, a viscosity of 2.77±0.12 cSt, and a methyl ester content of 96.85±0.91%. These results show that the molar ratio and type of catalyst have a big impact on the production of biodiesel. CaO worked better than NaOH at meeting quality standards.

Kata Kunci:

Bahan Bakar Terbaharukan

Biodiesel

CaO

Minyak Jagung

NaOH

INTISARI

Biodiesel, bahan bakar terbarukan yang berasal dari minyak nabati, telah menarik perhatian sebagai alternatif yang berkelanjutan untuk bahan bakar fosil. Tujuan dari penelitian ini adalah untuk menemukan kondisi terbaik dalam pembuatan biodiesel dari minyak jagung dengan menggunakan metode transesterifikasi dan untuk melihat pengaruh rasio molar dan jenis katalis yang berbeda terhadap kualitas dan yield biodiesel. Penelitian ini melakukan proses transesterifikasi dengan menggunakan metanol, NaOH, dan CaO sebagai katalis. Rasio molar minyak dan metanol yang digunakan adalah 1:12, 1:15, dan 1:18. Peneliti melakukan reaksi pada suhu 60°C selama 120 menit. Kami mengkarakterisasi biodiesel yang dihasilkan untuk mengetahui densitas, viskositas kinematik, dan kandungan metil ester dan membandingkan hasilnya dengan standar kualitas biodiesel. Hasil penelitian menunjukkan bahwa rasio molar 1:12 dengan katalis CaO menghasilkan biodiesel yang optimal, yaitu densitas 862,87±2.01 kg/m³, viskositas 2,77±0,12 cSt, dan kandungan metil ester 96,85±0,91%. Hasil ini menunjukkan bahwa rasio molar dan jenis katalis berpengaruh besar terhadap produksi biodiesel. CaO bekerja lebih baik daripada NaOH dalam memenuhi standar kualitas.

Korespondensi Penulis:

Dessy Agustina Sari
Chemical Engineering Program, Universitas Singaperbangsa Karawang; and Department of Chemical Engineering, Universitas Diponegoro
Jl. HS Ronggowaluyo, Karawang, Jawa Barat 41361; and Jl. Prof. Soedarto, Tembalang, Semarang 50275, Indonesia
Email: dessy.agustina8@staff.unsika.ac.id

1. INTRODUCTION

The renewable energy alternatives are facing significant challenges due to the increasing global demand for power, the depletion of fossil fuel reserves, and the consequent search for alternative renewable energy sources. Currently, traditional fossil fuels are non-renewable and present environmental challenges such as greenhouse gas emissions, which significantly contribute to climate change [1]. As a result, several countries have begun to embrace renewable energy sources such as wind power and hydrogen biodiesel. In this regard, biofuels have emerged as a favorable and eco-friendly alternative to conventional diesel due to their reusability and non-toxicity, making them biodegradable [2].

The usual method for biodiesel production involves the trans-esterification of vegetable oils or animal fats with methanol. This process turns oils in the system into fuel and glycerol using a catalyst (commonly methanol) along with an alcohol. Having said that, the most widely used vegetable oil as a biodiesel feedstock is palm oil [3,4]. Nevertheless, other feedstocks. However, other feedstocks rich in unsaturated fatty acids, such as corn oil, are gaining interest due to their high triglyceride content and the availability of improved and optimized technologies [5,6]. Corn oil underperforms as a feedstock for biodiesel production.

Corn production represents around 88% of the total agricultural crop output in Indonesia (2016–2020) annually, yielding approximately 22.9 million tons. The extensive scope of corn serves as a valuable basis for the production of biodiesel, as it presents potential economic and environmental benefits [7]. The high level of unsaturated fatty acids in corn oil also plays a role in why it is so well suited for making biodiesel [5].

The researchers can use heterogeneous catalysts like CaO or homogeneous catalysts like NaOH to catalyze the transesterification process. NaOH's high activity and stability in reaction conditions make it a commonly used substance. This is attributed to the complex separation process, which typically yields low yields, thereby increasing its cost [8,9]. CaO could be directly suitable due to its nature as a heterogeneous catalyst [10]. Reports also suggest that BSC, despite its drawbacks such as its solubility in methanol and inability to recycle, is fairly useful for producing biodiesel [11-13].

The molar ratio of oil to methanol, reaction time, and temperature are also controlling factors for biodiesel quality [14-16]. Prior studies have documented that the molar composition for biodiesel synthesis depends on feed fragments and catalytic systems used. For instance, the researchers [17] discovered that a temperature of 60°C is optimal for biodiesel conversion yield and reaction time (120 min). Additionally, researchers [18] conducted a study on the maximum yield of biodiesel production at an optimal molar ratio of 1:12 using CaO catalysts.

The aim of this study was to evaluate the quality reduction in corn oil biodiesel, which depends on types and molar ratio catalyst effects. The main goal of this paper, which was to get a better understanding of these kinds of systems by comparing NaOH and CaO at different molar ratios, was to look into different ways to help make biodiesel processes that are both technically and economically viable.

2. METHODS

2.1. Materials and Equipment

The tests used packaged corn oil, calcium oxide (CaO), sodium hydroxide (NaOH), methanol (96%), potassium hydroxide (KOH), phenolphthalein indicator, starch indicator, distilled water (H_2O), hydrochloric acid (HCl) 0.5 N, periodic acid ($HIO_4 \cdot 2H_2O$), potassium dichromate, sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$), potassium iodide (KI), ethanol (95%), chloroform, and glacial acetic acid.

This study used a complete transesterification setup, which included a hot plate, a 500-mL three-necked flask, thermometer, magnetic stirrer, condenser, water pump, burette, volumetric pipette, separatory funnel, pycnometer, capillary viscometer, and analytical balance.

2.2. Experimental Procedure

This research used corn oil as its feedstock and developed biodiesel by transesterifying it with methanol and two different catalysts: NaOH (as homogeneous) and CaO (as heterogeneous). Figure 1 illustrates the transesterification process. This study conducted experiments using oil to methanol molar ratios of 1:12, 1:15, and 1:18. The study conducted transesterification reactions under stirring conditions at 60°C for a duration of 120 minutes.

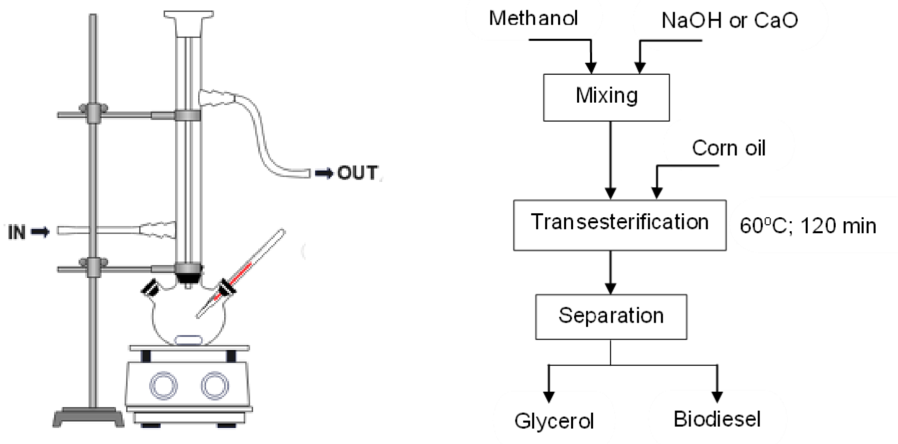

Figure 1. Experimental Set-Up and Procedure for Transesterification

This study prepared the catalyst, either NaOH or CaO, by dissolving them in methanol. The process added the resulting catalyst solution to the corn oil in a three-necked flask. The mixture was heated to 60°C and agitated using magnetic stirring at room temperature for 120 minutes. This reaction resulted in the separation of the biodiesel from the glycerol. A biodiesel phase sample was analyzed.

2.3. Analytical Methods

The tests on the samples followed the steps in SNI 7812:2015 [19]. The tests looked at the sample's density, kinematic viscosity at 40°C, saponification number, acid number, and total glycerol (methyl ester content). **Density measurement**. In this research, a photometer measured the density of the produced biodiesel. The researchers warmed a sample to 40°C and weighed the photometer before filling it with the biodiesel. The researcher used equation (1) to determine the density.

$$\rho = \frac{m}{V} \tag{1}$$

where: ρ = density of the sample (kg/m^3), m = mass of the sample (g), and V = volume of the pycnometer (m^3).

Kinematic viscosity. This study used a capillary viscometer to study kinematic viscosity and determine its behavior with temperature. The researchers recorded the time it took for the sample to flow through the viscometer at 40°C and used equation (2) to calculate the kinematic viscosity of the sample [19].

$$v = C \times t \tag{2}$$

where: v = kinematic viscosity (cSt or mm^2/s), C = viscometer constant (cSt/s), and t = time to flow the sample through the gravity of capillary viscometer (s).

Methyl ester content. The researchers determined the methyl ester content by calculating the saponification value, acid value, and total glycerol. The researchers then determined the methyl ester content using equations (3) up to 7, respectively.

a. The saponification value quantified the amount of base required for the hydrolysis and breakdown of this biodiesel into its distinct triglyceride forms. In this process, the researcher titrated the biodiesel sample against HCl after treating it with KOH and heating it.

$$A_S = \frac{56.1(B - C)N}{m} \tag{3}$$

where: A_S= saponification value (mg/g). The variables B and C represent the volume of 0.5 N HCl consumed in the blank titration (ml) and sample titration (ml), respectively, while N represents the exact concentration of the 0.5 HCl solution (N), and m represents the weight of the sample (g).

b. The study used titration with a standard alkali, KOH solution to determine the acid value of biodiesel, which indicates the amount of free fatty acids present. Acid value gives an inference to the extent of corrosive effects on engine parts due to biodiesel.

$$A_a = \frac{56.1(A - B)N}{W} \tag{4}$$

where: A_a= acid value in mg KOH/ g sample, A and B are the volumes of KOH solution used in the titration (ml) and the titration blank (ml), respectively, and W= weight of the sample (mg).

c. In this study, glycerol content is an unreacted component remaining in the biodiesel analysis. Maintaining fuel quality is crucial, as excessive glycerol can result in engine deposits and decreased combustion efficiency. The researchers titrated the biodiesel sample with periodic acid and sodium thiosulfate to figure out how much of that was free glycerol, bound glycerol, or something else.

$$G_{total} = \frac{2.302(B-C)N}{W} \quad (5)$$

$$W = \frac{\text{mass of the sample}^{a} \times \text{ml of the sample}^{b}}{900} \quad (6)$$

where: G_{total}= total glycerol in % mass. The variables *B* and *C* display the milliliters of sodium thiosulfate solution used in both the blank and sample titrations. The variable *N* shows the exact concentration of the sodium thiosulfate solution, and the variable *W* indicates the sample's weight.
The following equation (7) then calculates the ester content of alkyl ester biodiesel (% mass).

$$\text{The ester content} = \frac{100(A_S - A_a - 18.27G_{total})}{A_S} \quad (7)$$

3. RESULTS AND DISCUSSION

Effect of molar ratio and catalyst on biodiesel density. Density is a crucial quality parameter for fuel injection, atomization, and combustion of biodiesel. Table 1 compares densities of the biodisel samples prepared out of different catalysts (NaOH and CaO) with respect to molar ratios (1:12, 1:15, and 1:18). However, as the molar ratio increased to 1:18, the density of biodiesel dropped significantly, particularly for CaO (812.67±78.46 kg/m³), indicating that higher molar ratios could reduce biodiesel quality. This result can attribute the density reduction to incomplete transesterification or excess methanol diluting the biodiesel.

Table 1. Effect of molar ratio and catalyst type on produced biodiesel quality

Specification for Biodiesel	Molar Ratio (Oil:Methanol) and Catalyst					
	NaOH, 1:12	1:15	1:18	CaO, 1:12	1:15	1:18
Density, kg/m³	851.27 ± 2.01	842.73 ± 15.37	839.33 ± 23.93	862.87 ± 2.01	845.87 ± 10.80	812.67 ± 78.46
Kinematic viscocity, cSt	2.81 ± 0.11	2.15 ± 0.05	2.05 ± 0.18	2.77 ± 0.12	2.19 ± 0.07	2.06 ± 0.18
Saponification Value (mg/g)	125.06 ± 2.66	NG	NG	119.21 ± 1.74	NG	NG
Acid value, mg/g	0.14 ± 0.03	NG	NG	0.41 ± 0.02	NG	NG
Glycerol total, % mass	0.21 ± 0.037	NG	NG	0.18 ± 0.056	NG	NG
The ester content, % mass	96.85 ± 0.55	NG	NG	96.85 ± 0.91	NG	NG

The molar ratio of oil to methanol plays a crucial role in the biodiesel production process. Setting the molar ratio to 1:12 allowed both the NaOH and CaO catalysts to produce biodiesel with densities within the SNI 7182:2015 standard range (850–890 kg/m³). The densities were 851.27±2.01 kg/m³ for the NaOH catalyst and 862.87±2.01 kg/m³ for the CaO catalyst. This confirms that the 1:12 molar ratio is optimal for producing biodiesel of suitable density, with CaO yielding a slightly higher density compared to NaOH.

The density of the biofuels produced by CaO reduced gradually when increasing the molar ratio from 1:12 to 1:18, except in biodiesel, which was prepared using a high amount of catalyst. The biodiesel that was made with CaO at a 1:18 molar ratio had a low density (812.67±78.46 kg/m³), which might be because there was too much methanol in the reaction. This already demonstrated that according to the fact that the increase in molar ratio improves the transesterification reaction, an excess of methanol can promote a dilution effect where part remains without reacting, decreasing biodiesel density [20]. Then, excessive methanol can make the separation of glycerol and biodiesel more difficult, which could decrease product purity. The 1:12 molar ratio ensures enough methanol for a complete reaction and not too much to dilute the result. This result is also in line with [21,22], who also reported similar ideal molar ratios during biodiesel production investigations.

The choice of catalyst greatly influences the density of biodiesel. As a heterogeneous catalyst, CaO typically produces biodiesel with a slightly higher density than NaOH initially. The result attributes this to the improved glycerol separation in CaO-catalyzed reactions. Calcium oxide, along with its CaO, facilitates the easier separation of the glycerol by-product, leading to the production of pure biodiesel. Studies by [12,23] agree and provide further findings that CaO-catalyzed biodiesel has fewer impurities, meaning increased density.

For example, homogeneous catalysts such as NaOH can saponify and produce soapy products due to the presence of water. This leads to a slightly lowered density and purity of biodiesel. When examining the quality of biodiesel and its compatibility with the molar ratio, it is evident that NaOH, when used in a 1:12 molar kit, also meets the specifications. However, when considering these as by-products, CaO provides better density properties, leading to a higher cetane number.

Effect of molar ratio and catalyst on kinematic viscosity. When it comes to biodiesel, kinematic viscosity is a key parameter that ultimately affects its flow behavior as well as fuel injection in engines. Biodiesel viscosity has to be within specifications or may suffer poor engine performance. Table 1 shows biodiesel viscosity for various molar ratios and catalysts.

The kinematic viscosity of biodiesel decreased with increasing molar ratios of oil to methanol, from 1:12 to 1:18 for both catalysts (NaOH and CaO molar ratio of 1:12 gave the kinematic viscosities of NaOH and CaO 2.81±0.11 cSt, which was in the standard range (2.3–6.0 cSt) set by SNI 7182:2015. These values are ideal for biodiesel as they ensure low soot emissions and efficient fuel injection and combustion in diesel engines.

When the molar ratio increased to 1:18, viscosities decreased (NaOH: 2.05±0.18 cSt and CaO: 2.06±0.18 cSt). Most likely, the excess methanol present in the reaction mixture causes a dilution effect, leading to a decrease in viscosity. When this research uses excess methanol, it becomes an unreacted component in the final biodiesel, thereby reducing viscosity. According to [20], biodiesel primarily consists of lower melting-point methyl esters due to the excess of methanol in the reaction mixture, which leads to a higher purity level. The researchers [21] confirm that too much methanol may reduce product flow properties, slowing down the conversion time of mol ratio changes.

Lower molar ratios, like 1:12, complete the transesterification reaction, resulting in higher methyl ester contents that contribute to favorable viscosity values. Therefore, the 1:12 molar ratio will perform a tradeoff in catalysis needed for viscosity control and fuel efficiency of biodiesel.

Biodiesel maximum viscosity values under NaOH or CaO catalysts reached the limits prescribed by standards at 1:12 molar ratio. However, the two catalysts had minor differences. The NaOH-catalyzed biodiesel had a slightly higher viscosity (2.81±0.11 cSt compared to 2.77±0.12 cSt) than the CaO-catalyzed biodiesel at an equal molar ratio. The NaOH-catalyzed reaction may have produced soap-like by-products, which could have an in-situ viscosifying effect [12].

Biodiesel produced by CaO (as widely seen from heterogeneous catalysts) has fewer impurities and thus slightly lower viscosity. However, better glycerol separation in the CaO-catalyzed reaction makes for better biodiesel with more consistent viscosity [23]. This result matched what [24] found in other studies: biodiesel made with CaO had better flow properties than NaOH because it had a higher purity content.

Moreover, NaOH, a homogenous catalyst, can trigger saponification reactions when water is present in the reaction mixture, leading to increased viscosity due to soap by-products. In contrast, this phenomenon is much less common with CaO, and it results in improved product stability (lower viscosity) at an optimized molar ratio.

Effect of molar ratio and catalyst on methyl ester content. Methyl ester content (Table 1) is one of the most important parameters for biodiesel quality analysis. A higher methyl ester content means a more thorough transesterification reaction has taken place and leads to superior combustion characteristics and fuel quality.

Biodiesel's methyl ester content goes down because the molar ratio of oil to methanol goes above the 1:12 ideal molar ratio for NaOH or CaO catalysts. The obtained ratio of 1:12 at metil content resulted in NaOH (96.85±0.55%) and CaO (96.85±0.91%), meeting the SNI 7182:2015 standard, which requires a minimum methyl ester content of 96.50% or higher. This indicates that the transesterification reaction, using a molar ratio of 1:12, is 90% complete, leading to the production of highly pure biodiesel.

When the molar ratio was raised to 1:18, only 93.64% of methyl ester was produced for NaOH and 92.98% for CaO, which means that the requirements were not met. Too much methanol diluting could be the reason for the drop in methyl ester content because it stops glycerol and unbonded methanol from completely separating from biodiesel [20]. However, higher concentrations of methanol can reversibly shift the equilibrium in the reactants favor and thus lead to lower conversion rates, as demonstrated in [21,22].

A molar ratio of 1:12 yields the best methyl ester content. This is because a little more methanol is available to move the reaction forward, while the larger amounts of inhibitory effort work against it. This finding is consistent with the results of other studies, such as this study [25].

Using both NaOH and CaO as catalysts to make biodiesel worked at a 1:12 molar ratio, which met the standard for methyl ester content. However, at the given ratio, CaO-catalyzed biodiesel has a similiar higher methyl ester content (96.85±0.91%) than NaOH-catalyzed biodiesel (96.85±0.55%). The difference can be attributed to the improved glycerol segregation in the CaO-catalyzed reaction, resulting in fewer residual contaminants in the biodiesel product [12].

Both catalysts with higher molar ratios saw a decrease in the methyl ester content, with NaOH biodiesel showing a slight increase over CaO. Additionally, NaOH-catalyzed reactions may form soap, acting as a proinhibitor in the transesterification reaction and potentially reducing the total methyl ester contribution [23]. Thus, using CaO as a heterogeneous catalyst has led to low production of by-products and enhanced methyl ester yield at different molar ratios [26].

Effect of molar ratio and catalyst on biodiesel quality. Numerous parameters, including density, viscosity, and methyl esters content, evaluate the quality of biodiesels. Results from the various molar ratios and catalysts suggest that both have a considerable effect on biodiesel quality. For both types of catalysts, the optimal molar ratio for achieving the best biodiesel quality is 1:12, where NaOH and CaO result in high-quality methyl esters based on density, viscosity, and a percentage of methyl ester properties.

The Table 1 indicate that a molar ratio of 1:12 has produced biodiesel with optimum quality, as evidenced by its compliance with SNI 7182:2015 for density and methyl ester content in all observed parameters. The quality parameters begin to decrease when the molar ratio is greater than 1:18. The dilution effects of high methanol, which reduce density, viscosity, and methyl ester content, are one of the major factors [20]. It simply shows that molar ratio optimization is key for good-quality biodiesel.

The ratio 1:12 ensures enough methanol is present to drive the transesterification reaction to completion but not too much unreacted methanol, which can dilute the final product and reduce quality. The researchers [27] found that this ratio yields the best biodiesel quality.

When compared to NaOH, the CaO catalyst produced biodiesel of slightly higher quality, with significantly more methyl ester and a simpler product; however, both could meet the specifications at a 1:12 molar ratio. A reason for that may be the higher selectivity in glycerol separation of CaO-catalyzed reactions [12]. Furthermore, the use of much alkaline CaO does not generate too many impurities (such as soap) that might disturb biodiesel flow property and combustion efficiency [23].

In contrast, NaOH is a homogeneous catalyst that will promote saponification reactions even in the presence of relatively low levels of water within the reaction medium. Such reactions form by-products of a soapy nature, and these might diminish the quality of biodiesel collectively [28].

4. CONCLUSION

The study results revealed that the ideal conditions for producing biodiesel from corn oil through transesterification were a 1:12 molar ratio of oil to methanol, with a CaO catalyst present. This ratio must also meet the requirements of the Indonesian National Standard (SNI 7182-2015), which includes density, viscosity, and methyl ester content. Exceeding a molar ratio of more than 1:12 results in a deterioration of the biodiesel specification, as excess methanol disrupts the transesterification process by inhibiting the separation of glycerol. Out of all the catalysts tested, CaO outperformed NaOH by producing biodiesel with a similar higher percentage of methyl esters (CaO 96.85±0.91% vs. NaOH 96.85±0.55%), thereby simplifying the separation process and reducing the environmental impact of the disposal phase. The studies show that using CaO as a green catalyst can enhance production efficiency and reduce pollution associated with biodiesel synthesis.

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