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Effect of Time Variation and Mole Ratio on Biodiesel Production from Coconut Oil Using CaO Catalyst via Transesterification Method

Pengaruh Variasi Waktu dan Rasio Mol pada Produksi Biodiesel dari Minyak Kelapa Menggunakan Katalis CaO melalui Metode Transesterifikasi

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ABSTRACT

The use of fossil fuels by the world resulted in an impulse for the use of renewable energy sources including biodiesel. In the present investigation, the objective is to find out the best conditions of coconut oil transesterification into biodiesel for transesterification source of the homogeneous basic catalyst of CaO. The experiments were conducted by varying the reaction time (100, 120 and 150 min), the molar ratio of coconut oil to methanol (1:12, 1:15 and 1:18), and temperature (60°C) as a fixed level in the experimental plan The optimum condition of reaction was at 1:15 molar ratio and a time of 120 min producing biodiesel that meets the (SNI 7182:2015) Indonesian National Standard with properties; density was 858.20±26.95 kg/m^3, viscosity was 2.64±0.22 cSt, methyl ester content was 96.14±0.73%. Furthermore, the utilization of CaO catalyst not only improved the quality of biodiesel, but also possessed economic and environmental benefits compared with traditional homogeneous catalysts. These results confirm the feasibility of using coconut oil as a cheap and sustainable raw materials for biodiesel production as well as their effective use in larger scale production of renewable energy.

Keywords: *Biodiesel; CaO Catalyst; Coconut Oil; Renewable Energy; Transesterification*

ABSTRAK

Penggunaan bahan bakar fosil di seluruh dunia telah mendorong pemanfaatan sumber energi terbarukan, termasuk biodiesel. Penelitian ini bertujuan untuk menentukan kondisi terbaik dalam proses transesterifikasi minyak kelapa menjadi biodiesel dengan menggunakan katalis basa homogen CaO. Eksperimen dilakukan dengan memvariasikan waktu reaksi (100, 120, dan 150 menit), rasio mol minyak kelapa terhadap metanol (1:12, 1:15, dan 1:18), serta suhu tetap sebesar 60 °C sesuai rancangan percobaan. Kondisi optimum diperoleh pada rasio mol 1:15 dan waktu reaksi 120 menit, yang menghasilkan biodiesel sesuai dengan Standar Nasional Indonesia (SNI 7182:2015) dengan karakteristik: densitas sebesar 858,20 ± 26,95 kg/m^3, viskositas 2,64 ± 0,22 cSt, dan kandungan metil ester sebesar 96,14 ± 0,73%. Selain meningkatkan kualitas biodiesel, penggunaan katalis CaO juga memberikan keuntungan ekonomi dan lingkungan dibandingkan dengan katalis homogen konvensional. Hasil ini menegaskan bahwa minyak kelapa merupakan bahan baku yang murah dan berkelanjutan untuk produksi biodiesel serta berpotensi digunakan secara efektif dalam produksi energi terbarukan skala besar.

Kata Kunci: Biodiesel; Energi Terbaharukan; Katalis Cao; Minyak Kelapa; Transesterifikasi

1. INTRODUCTION

Today, energy demand is a major focus around the world. The decreasing availability of energy resources does not match the ever-increasing demand for energy (Fattaheian-Dehkordi et al., 2023; Laugs et al., 2020). Fossil fuels, as the main source of energy, have become a problem due to their non-renewable nature and negative effects on the environment.

Population growth and the development of the industrial sector have also exacerbated the global energy crisis, prompting many countries to seek more sustainable alternative energy sources. Biodiesel, among the developing renewable energy sources, is emerging as a potential solution due to its production from renewable feedstocks, environmental friendliness, and reduced harmful emissions (Güney, 2019; Kataya et al., 2023; Zuhri et al., 2024).

Biodiesel is a diesel fuel that has advantages over petroleum-based diesel, such as producing fewer emissions, having a higher cetane number, and being non-toxic and biodegradable (Neupane, 2022). The transesterification process typically produces biodiesel by reacting triglycerides in vegetable oils or animal fats with short-chain alcohols like methanol or ethanol, resulting in the production of methyl esters (FAME) as biodiesel and the by-product of glycerol.

One source of vegetable oil that has great potential as a biodiesel feedstock in Indonesia is coconut oil, given the vast area of coconut land in Indonesia, which reaches 3.34 million hectares with an annual production of 2.80 million tons (Firdous et al., 2016). Coconut oil, especially virgin coconut oil, has a high content of saturated fatty acids, such as palmitic acid and stearic acid, making it suitable as a biodiesel feedstock.

The coconut oil transesterification process commonly uses homogeneous base catalysts like NaOH or KOH because of their high catalytic activity and reasonably low cost. On the other hand, these homogeneous catalysts had some drawbacks, such as difficulty in separating from the final product and generating process-unmanageable waste (Fadhillah & Sari, 2023; Govan & Gun'ko, 2014; Ling et al., 2019).

Thus, this has prompted the researchers to investigate a relatively more efficient heterogeneous catalyst such as CaO. CaO works better in the biodiesel purification process because it doesn't dissolve easily in methanol and is easy to separate after the reaction (Kesić et al., 2016; Marinković et al., 2016).

The goal of this research is to identify the ideal conditions for the transformation of coconut oil into biodiesel. To achieve this, the researchers manipulate the reaction time and mole ratio between coconut oil and methanol at 60 °C using a CaO catalyst. The various times were 100, 120, and 150 minutes, while the ratio of mole coconut oil to methanol was 1:12, 1:15, and 1:18.

The researchers tested the quality parameters of biodiesel, specifically density, viscosity, and methyl ester content, using SNI 7182:2015 as a reference (Standar Nasional Indonesia, 2015). The study expects this research to significantly contribute to the development of environmentally friendly and cost-effective biodiesel from coconut oil in Indonesia.

The other aim of this study is to show that CaO catalysts can work as heterogeneous catalysts, which can improve the quality and efficiency of biodiesel production and do so at a lower cost than traditional catalysts.

2. RESEARCH METHOD

2.1 Materials

Support materials used were technical 95% ethanol, 0.5N technical KOH, phenolphthalein indicator, amylum/tapioca indicator, distilled water, technical hydrochloric acid, periodic acid ($HIO_4.2H_2O$) as a pro-analyst, glacial acetic acid as a reagent analyst, potassium dichromate ($K_2Cr_2O_7$), sodium thiosulfate ($Na_2S_2O_3.5H_2O$), and potassium iodide (KI) as catalysts and reagent analysts.

The equipment used was a hotplate with a magnetic stirrer, a 500-mL triple-neck flask (as a batch reactor), a thermometer, a condenser, a burette, a glass Erlenmeyer, a stative and clamp, a beaker, and a laboratory watch glass.

2.2 Biodiesel Production

Figure 1 illustrates the research process for biodiesel production. This research uses Barco brand coconut oil as the main raw material, technical methanol as a reactant, and calcium oxide as a heterogeneous catalyst.

2.3 Biodiesel Characterization

This research used a pycnometer and a capillary viscometer for density measurement, and a capillary viscometer for viscosity measurement, respectively. The study checked the quality of the biodiesel that was made by measuring its density, viscosity, and methyl ester content in line with the SNI 7182:2015 (Standar Nasional Indonesia, 2015).

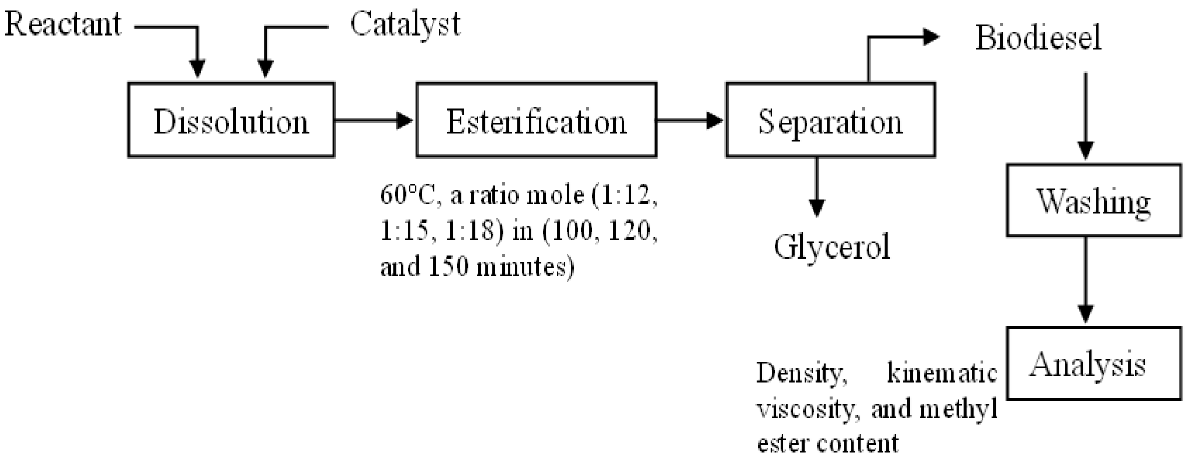

Figure 1. Biodiesel Production from Coconut Oil using CaO Catalysts

A pycnometer at 40°C measures density, which must fall within the standard range of 850–890 kg/m^3. Using a capillary viscometer, the researchers measured the kinematic viscosity at 40°C, which should match the biodiesel viscosity standard of 2.3-6.0 cSt. The study used the saponification number, acid number, and total glycerol content tests to figure out the methyl ester content. This was done to make sure that the biodiesel met the standard requirements for methyl ester content, which are at least 96.5% by mass.

2.4 Data Analysis

The experiments were repeated for three times (n = 3) under each combination of variables to confirm the reproductivity and the reliability of the results. Results were presented as mean ± standard deviation and analyzed descriptively, compared to Indonesian National Standard (SNI 7182:2015) of biodiesel quality. Measurement protocols for each parameter are outlined in the following:

1. **Density**

The sample was heated above 40°C and density measured using pycnometer. The empty pycnometer was first weighed (a), then filled with sample and reweighed (b). The pycnometer volume was 50 mL. The density (ρ, kg/m^3) was determined by Equation (1) as follows:

$$\rho = \frac{b - a}{V} \tag{1}$$

2. **Kinematic viscosity**

The kinematic viscosity was determined using an Ostwald capillary viscometer. The sample had been preheated to 40°C, obtained, and allowed to flow through the viscometer until the duration of flow (t) was recorded. The viscosity was determined by equation (2):

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

where v is viscosity in cSt (mm^2/s) and C is the viscometer constant.

3. **Saponification value (A_S)**

The refluxed volume with 50 mL alcoholic KOH (1 hr) was done with 4–5 g sample. Titration with phenolphthalein indicator with 0.5 N HCl was performed after cooling. The saponification value was determined according to the method of Equation (3):

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

where B, C, N, and m are the titrant volumes (blank and sample), the exact concentration of HCl, and the sample weight in grams.

4. **Acid value (A_a)**

Eight grams of sample (W) were mixed with 125 ml of neutralized solvent (B) and 0.1 N KOH was titrated the sample until the first stable pink color persist for 30 seconds (A). The acid value (mg KOH/g sample) was determined by the following Equation (4):

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

5. **Glycerol total (G_{total})**

Approximately 10 g of the sample (W) was saponified with alcoholic KOH and extracted with chloroform. The aqueous phase was treated with periodic acid and KI and titrated with standardized sodium thiosulfate solution. The total amount of glycerol was determined by the following equation (5)-(6):

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

For glycerol total

$$W = \frac{m_{sample} \times V_{sample}}{900} \tag{6}$$

6. Methyl ester content

Methyl ester was measured indirectly through saponification value (A_S), acid value (A_a) and glycerol total (G_{total}) according to the following equation (7):

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

Table 1. Variation of Reaction Time and Mole Ratio with CaO Catalyst on Density and Kinematic Viscosity

Mole Ratio (Oil:Methanol) and Time	Specification for Biodiesel	
	Density, kg/m³	Kinematic viscosity, cSt
1:12, 100 min	878.33 ± 4.23	2.19 ± 0.13
120	866.60 ± 17.28	2.90 ± 0.10
150	847.67 ± 124.24	1.90 ± 0.35
1:15, 100 min	788.33 ± 18.67	1.74 ± 0.16
120	858.20 ± 26.95	2.64 ± 0.22
150	792.13 ± 8.08	1.94 ± 0.28
1:18, 100 min	784.27 ± 12.34	2.00 ± 0.06
120	851.07 ± 2.50	2.48 ± 0.13
150	838.53 ± 14.59	2.23 ± 0.21

Table 2. Chemical Properties and Biodiesel Quality for the Optimum Condition

Mole Ratio and Time	Biodiesel Quality			
	Saponification value (mg/g)	Acid value, mg/g	Glycerol total, % mass	The ester content, % mass
1:12, 120 min	112.20 ± 3.48	0.28 ± 0.04	0.24 ± 0.037	95.88 ± 0.71
1:15, 120 min	123.42 ± 1.74	0.42 ± 0.04	0.24 ± 0.044	96.14 ± 0.73
1:18, 120 min	105.19 ± 0.17	0.14 ± 0.04	0.15 ± 0.098	97.29 ± 1.73

3. RESULTS AND DISCUSSIONS

The study looked at the properties of biodiesel made from coconut oil using the transesterification method and a CaO catalyst. The main quality parameters used were density, viscosity, and methyl ester content. The test results in **Table 1** and **Table 2** show that variations in time and mole ratio affect the quality of biodiesel produced.

Biodiesel density is one of the vital parameters to determine the suitability of biodiesel for diesel engines since very low or very high biodiesel density can influence fuel consumption and combustion. The density of biodiesel in accordance with SNI 7182:2015 should be in the range of 850–890 kg/m³. Under a mole ratio of 1:15 and a decoration time of 120 min, the density of biodiesel in this study was 858.20±26.95 kg/m³, which is within the range of standards.

This suggests that these conditions are the best for achieving the biodiesel density criteria. However, after 150 minutes, the density was 838.53±14.59 kg/m³ at a mole ratio of 1:18, which was almost outside the standard. A high dose of methanol can reduce the molecular density of biodiesel (Darwin et al., 2021; Kawashima et al., 2008). The sluggish flow of the fuel due to its high kinematic viscosity may be an impediment to combustion.

According to my SNI 7182:2015 standard, the viscosity of biodiesel should be in the range of 2.3-6.0 cSt. The viscosity touches the standard value in 120 minutes with a mole ratio of 1:15, where the viscosity value of biodiesel is 2.64±0.22 cSt. The low viscosity of 2.19±0.13 cSt at 100 minutes (1:12 mole ratio) indicates that too low a mole ratio or a short reaction time does not yield the ideal viscosity (Gülüm et al., 2017) Methyl ester content in **Table 2** is an indication of the purity of the biodiesel obtained. The methyl ester content shall be at least 96.5% (SNI 7182:2015). The maximum result at 120 minutes was obtained in this study with a mole ratio of 1:15 and a methyl ester content of 96.14±0.73%. The maximum content of methyl ester was higher at a mole ratio of 1:18 and 120 minutes, with a value of 97.29±1.73%, as reported by the researchers.

The researcher can explain this by noting that excessive methanol increased the reaction time without significantly refining the biodiesel process. This result is in consonance (in agreement) with that of other researchers (Anjana et al., 2016; Balki, 2024; Salehi et al., 2019) who discovered that excess methanol can increase the methyl ester content but does not always lead to effective biodiesel transformation.

CaO catalysts are better than homogeneous base catalysts like NaOH or KOH in many ways, such as making separation easier and allowing them to be used again and again without contaminating the end products. In this study, CaO in particular presented favorable results, with a utilization time of 120 min and a mole ratio of 1:15.

The researcher could use catalysts like CaO to reduce production costs and unwanted residues in the final product. CaO catalysts demonstrate excellent stability during transesterification reactions, making them an environmentally friendly alternative to traditional homogeneous catalysts (Pattiasina et al., 2023; Santos et al., 2019; Temu, 2013).

The results of this study align with previous studies that focused on achieving the optimal mole ratio and time for biodiesel production. A reaction with castor oil and NaOH catalysts at 120 minutes of time and 60 °C of temperature yielded a maximum biodiesel conversion of 96% (Sipahutar & Tobing, 2013).

Additionally, data from (Pasae et al., 2019), revealed that the effective mole ratio of caustic and oil used to produce biodiesel from palm oil ranged from 1:12 to 1:15 when using the CaO catalyst. This work, therefore, further confirms CaO's effectiveness as a catalyst, which can be applied to most types of vegetable oils and creates practical situations (Mohamad et al., 2015; Primadi et al., 2020).

4. CONCLUSIONS

This study produced biodiesel using transesterification with a CaO catalyst at 60°C for 120 minutes, using a 1:15 mole ratio of coconut oil to methanol, yielding the most effective results. By following these steps, biodiesel was made that had a density of 858.20±26.95 kg/m^3, a viscosity of 2.64±0.22 cSt, and a methyl ester content of 96.14±0.73%, all of which were in line with SNI 7182:2015 standards.

The CaO catalyst was better than NaOH or KOH (homogeneous catalysts) because it was easy to separate from the final product, didn't dissolve in methanol, and could be used again and again. The quality of the biodiesel changed a lot depending on the mole ratio and reaction time. The highest-content methyl esters were made with high mole ratios and long reaction times.

However, excess methanol could lead to a reduction in the density of the final biodiesel, thereby decreasing the process's effectiveness. The results of this study align with previous research demonstrating the effectiveness of the CaO catalyst in catalyzing various vegetable oils. The results also lend support to the idea of using Indonesia's abundant coconut oil as a feedstock for biodiesel.

Hence, using CaO as a catalyst for the production of biodiesel from coconut oil on an industrial scale is highly promising, as it can support renewable energy and reduce the use of fossil fuels.

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