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Temperature Effect on the Transesterification Process of Used Cooking Oil with a CaO-MgO Catalyst in the Biodiesel Production

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ABSTRACT

This study investigates the effect of temperature on the transesterification process of used cooking oil using CaO-MgO catalysts for biodiesel production. Temperature is a crucial factor in determining the efficiency of the transesterification reaction. The research examines a range of reaction temperatures from 50°C to 80°C to identify the optimal temperature that achieves the highest biodiesel conversion rate. The findings reveal that as the reaction temperature increases, biodiesel conversion also increases, reaching an optimum point. Beyond this optimum temperature, a decrease in conversion efficiency is observed, likely due to the reverse reaction. The use of a heterogeneous CaO-MgO catalyst, activated at 600°C for 5 hours, proves to be effective in the transesterification process, producing biodiesel that complies with the SNI 7182:2015 standards. The optimal reaction temperature identified in this study is 60°C, resulting in a biodiesel yield of 85.53%. The produced biodiesel exhibits desirable properties, including a density of 875 kg/m³, a viscosity of 4.2 cSt, a flash point of 183°C, a calorific value of 8001 cal/g, and a cetane number of 48.3. These results underscore the importance of controlling reaction temperature and using an efficient catalyst to maximize biodiesel production quality and yield.

Keywords: *Biodiesel, CaO-MgO, Heterogeneous Catalyst, Transesterification, Used Cooking Oil.*

1. INTRODUCTION

Indonesia is a country rich in natural resources, including palm oil. The excessive use of palm oil results in waste in the form of used cooking oil, known as minyak jelantah [1]. *Waste Cooking Oil* is a waste product from previously used cooking oil. Reusing minyak jelantah is not recommended as it contains carcinogenic compounds that can cause cancer.

The use of used cooking oil can produce acrolein, a compound that causes throat irritation and triggers coughing. Moreover, the disposal of used cooking oil into the environment can lead to pollution. To address this issue, efforts are needed to process *Waste Cooking Oil* into more valuable products, such as biodiesel, which is considered an ideal alternative fuel for the future. Biodiesel is a mono-alkyl ester of long-chain fatty acids derived from vegetable oils or animal fats and used as fuel in diesel engines [2].

Atated that the production of biodiesel through the transesterification of vegetable oil with short-chain alcohols and catalysts has drawbacks, such as sensitivity to FFA, soap formation as a by-product, difficulties in separating the product from the catalyst, and the need for complex and energy-intensive alkali waste treatment. Nevertheless, biodiesel has advantages such as being biodegradable and non-toxic.

The esterification of free fatty acids (FFA) and the transesterification of triglycerides (TG) are the two main processes in biodiesel production. Transesterification is carried out using short-chain alcohols, usually methanol, and a catalyst. Throughout several stages, TG is converted into FAME (fatty acid methyl ester), with triglycerides, monoglycerides, and diglycerides forming FAME, and glycerol as a by-product. Each mole of TG produces moles of FAME and glycerol. Homogeneous catalysts such as NaOH or KOH are often used in the transesterification process.

However, in biodiesel synthesis, homogeneous catalysts are difficult to separate and cannot be reused, making heterogeneous catalysts more appealing. Researchers typically utilize metal oxides from alkali (Li, Na, K, Rb, Cs) and alkaline earth (Mg, Ca, Sr, Ba) groups, with CaO being commonly used as a catalyst in transesterification. Therefore, in this study, CaO-MgO was chosen as the catalyst for the transesterification of used cooking oil.

Research by [3] and [4] has shown significant gaps in studies on temperature and catalyst use for biodiesel production. [3] found an optimal

temperature of 65°C using a NaOH catalyst, while [4] indicated the best temperature was 50°C. However, no studies have explored a broader temperature range or comprehensively compared the use of CaO-MgO catalysts. Furthermore, research by [5] using a 5% CaO-MgO catalyst and ethanol resulted in an 85.72% yield, but faced challenges in forming fatty acid ethyl esters. This study aims to fill these gaps by evaluating a broader temperature range and the use of CaO-MgO catalysts, as well as using methanol for the transesterification of bulk oil and used cooking oil.

2. MATERIAL AND METHODS

2.1 Materials

The materials used in this study were used cooking oil, Calcium Oxide (CaO), Magnesium Oxide (MgO), Methanol, Sulfuric Acid (H_2SO_4) , and aquadest.

2.2 Method

CaO and MgO catalysts, in a 1:1 ratio, were activated through calcination in a furnace for 2 hours at 600ºC. The activated CaO and MgO were then mixed to be used as a catalyst in the transesterification process. The free fatty acid (FFA) content of the raw material was analyzed by weighing 5 g of the material and placing it into an Erlenmeyer flask, followed by the addition of 50 ml of methanol and 3 drops of phenolphthalein (pp) indicator. The mixture was then titrated with 0.1 N NaOH until a pink color was observed In the esterification process, waste cooking oil was first heated to 50ºC in a stirred reactor, then 0.5% H2SO4 (98%) by weight of the oil and 10% methanol by volume of the oil were added. The reaction was maintained at 65ºC for 60 minutes with continuous stirring.

After the reaction, the mixture was allowed to settle in a separator, forming two layers. The top layer, which is the result of the esterification process, was separated and further subjected to transesterification. In the transesterification process, the oil was heated to temperatures of 50ºC, 55ºC, 60ºC, 65ºC, 70ºC, 75ºC, and 80ºC, then mixed with methanol at a 1:3 ratio and catalyst 5%, that had been activated, while stirring at 400 rpm for 80 minutes. Glycerol was separated from the methyl ester using a separator and left to stand for 24 hours. The methyl ester was then washed by heating distilled water to 65ºC, adding the methyl ester, and stirring. After mixing, the solution was allowed to settle until two layers formed. This washing process was repeated three times. Finally, the top layer was purified through distillation to separate biodiesel from methanol and water.

3. RESULTS AND DISCUSSIONS

The results of the analysis were as follows: There are key factors that greatly influence the results of the biodiesel produced, namely; analysis of density, llash point, yield of product, calorific value and cetane number.

3.1 The Effect of Temperature on Biodiesel Density

The effect of temperature on density analysis is shown in the Figure 1. As the temperature increases from 50°C to 65°C, the density of biodiesel tends to decrease but then rises again at 70°C and beyond. This is due to the evaporation of some methanol used as a reactant, which causes an increase in volume and density of the biodiesel [4].

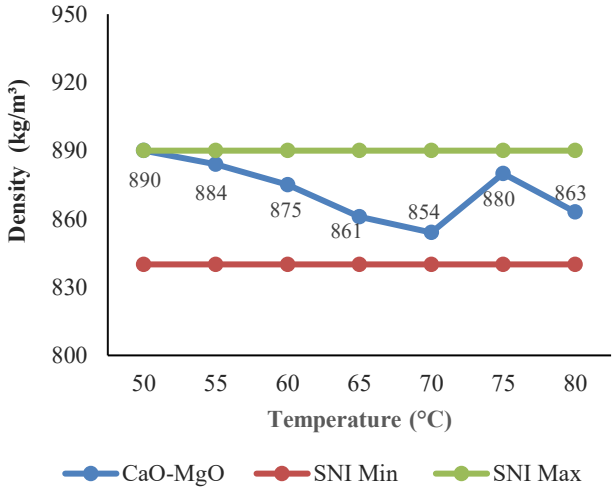

Figure 1. The Effect of Temperature on Biodiesel Density

This study indicates that the density of biodiesel decreases at temperatures from 50°C to 60°C and then increases again at 70°C due to methanol evaporation. Although temperature variations do not significantly affect the density of methyl ester, temperature still impacts the reaction rate and the frequency of molecular collisions among reactants. As the transesterification temperature increases, molecular collisions occur more rapidly, enhancing the chances of forming methyl ester [3].

3.2 The Effect of Temperature on Biodiesel Viscosity

The following shows the effect of temperature on the den of viscosity the produced biodiesel, as illustrated in the Figure 2.

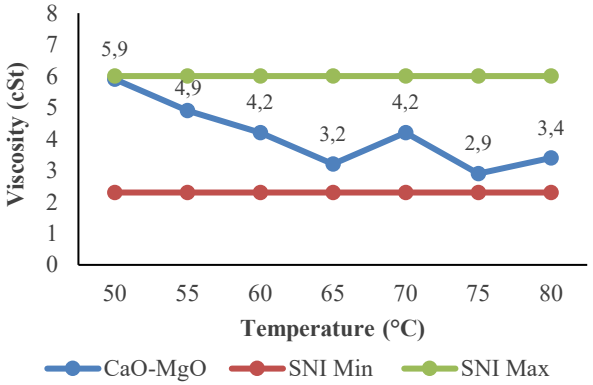

Figure 2. The Effect of Temperature on Biodiesel Viscosity

Based on the results obtained, the viscosity of biodiesel is compared with the SNI standard, which ranges from 2.3 to 6.0 cSt. Temperature variations affect the fluctuations in viscosity of each biodiesel sample tested. Research by [4] viscosity of biodiesel tends to decrease, though the decrease is only about one hundredth. At 80°C, viscosity actually increases due to incomplete biodiesel production. Methanol has a boiling point of 65°C, so it evaporates quickly before biodiesel formation is complete. A mixture with high methanol content causes rapid evaporation, resulting in limited reaction between the remaining methoxide solution and waste cooking oil.

The highest viscosity was found at 50°C, which is associated with the lowest biodiesel conversion. The more biodiesel that is converted, the lower the resulting kinematic viscosity. This may be due to a lower free fatty acid content in the produced biodiesel or due to residual water from the washing process [6]

3.3 The Effect of Temperature on Biodiesel Flash Point

Increasing the reaction temperature in biodiesel synthesis can lead to a higher flash point of the produced biodiesel. This increase is due to the higher volatility and flammability of the biodiesel. A higher flash point indicates that the biodiesel is more difficult to ignite at room temperature, making it safer for storage and use. To ensure the quality and safety of biodiesel, quality standards such as SNI set flash point limits that must be met.

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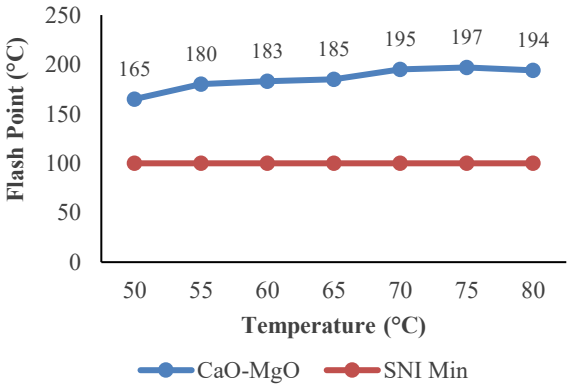

Figure 3. The Effect of Temperature on Biodiesel Flash Point

Based on the data results in Figure 3, the flash point of biodiesel ranging from 165–194°C can increase due to the water content in the biodiesel, which is closely related to the increased catalyst concentration during production. High water content requires more energy to evaporate, which reduces the heat available from combustion and consequently raises the flash point [6]. The flash point indicates the lowest temperature at which the material starts to ignite, and a higher flash point means the fuel is less easily ignited [7] The research shows that higher heating temperatures result in higher flash points, with the highest temperature at the 80°C heating variation, meeting the SNI standard, which is above 100°C.

3.4 The Effect of Temperature on Biodiesel Cetane Number & Calorific Value

The calorific value represents the amount of energy produced from the combustion of biodiesel, indicating the fuel's efficiency in generating power. Meanwhile, the cetane number is an important parameter in assessing diesel fuel performance, as it indicates the quality of fuel ignition. Analyzing both calorific value and cetane number is crucial for evaluating biodiesel quality as a fuel and understanding how temperature affects the physical properties and performance of biodiesel [8]

Table 1. Calorific Value and Cetane Number of Biodiesel Product

Catalyst	Temperature (°C)	Calorific Value (Cal/g)	Cetane Number
CaO-MgO	60	8001	48.3

The study results show a cetane number of 48.3 and a calorific value of 8001 cal/g for the biodiesel, which are still below the SNI 7182:2015 standards, which require a cetane number of 51 and a calorific value of 9062 cal/g. The calorific value measures the energy produced from the combustion of biodiesel, while the cetane number indicates fuel ignition quality. A high cetane number can lead to excessively fast combustion in a diesel engine, whereas a low cetane number can worsen engine performance and combustion efficiency. High free fatty acid (FFA) content in biodiesel can lower the cetane number, causing incomplete combustion and increased emissions [8]. Additionally, high water content in biodiesel requires more energy for evaporation, reducing combustion efficiency as some energy is wasted evaporating water rather than generating heat [9].

3.5 The Effect of Temperature on Biodiesel Cetane Number & Calorific Value

This study was conducted with a constant reaction time of 80 minutes to evaluate the effect of temperature on conversion and product yield. The results showed that a temperature of 60°C provided the best conversion and output. Lower temperatures, such as 50°C, resulted in lower conversion, while higher temperatures, such as 80°C, could lead to catalyst decomposition. This indicates that 60°C is the most optimal temperature, as shown in Figure 4.

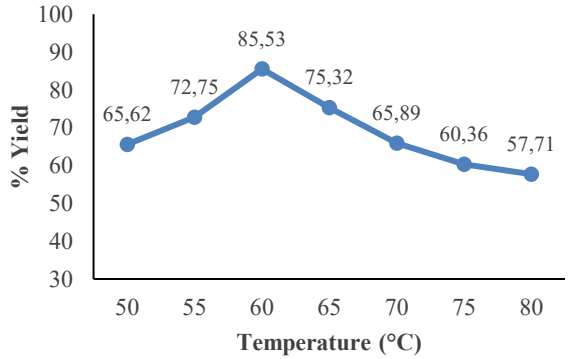

Figure 4. The Effect of Temperature on Biodiesel Yield

Each temperature variation results in different yields. At 60°C, the highest biodiesel yield reaches 85.53%, as shown in Figure 5. According to [4] this difference in yield is due to factors such as temperature and the precision in homogenizing the mixture of raw materials, catalysts, and alcohol. At 60°C, waste cooking oil receives optimal heat for the reaction. Research by [9] indicates that transesterification is an endothermic reaction that requires energy. Increasing the temperature enhances the kinetic energy of the reactants, accelerates the reaction rate, and improves conversion and yield. A temperature of 50°C may be too low to achieve maximum conversion, resulting in lower yields. According to [10] increasing the temperature increases the amount of oil converted to biodiesel, thereby improving the yield [9] found that the optimal temperature for biodiesel production is between 30°C and 60°C, while temperatures above 65°C can cause methanol to evaporate, reducing polarity, the amount of methoxide ions, and accelerating triglyceride saponification, which negatively impacts the yield. Research by [11] shows that optimal catalytic activity occurs between 30°C and 60°C, where oxide species remain stable. Higher temperatures can cause agglomeration and a decrease in catalyst activity, preventing an increase in yield conversion.

4. CONCLUSIONS

The optimal temperature for the transesterification process using the heterogeneous catalyst CaO-MgO was found to be 60°C and 65°C, yielding 85.53% and 75.32%, respectively, from 1250 ml of used cooking oil. The highest yield in this study was obtained at 60°C, with a yield percentage of 85.53%, compared to 57.71% at 80°C. The quality of the biodiesel product obtained in this study that meets SNI standards includes a density of 854 – 880 kg/m³, viscosity of 2.9 – 5.9 cSt, and a flash point of 165 – 194°C. However, the calorific value of 8001 cal/g and the cetane number of 48.3 do not meet SNI standards.

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