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Optimization of Biodiesel Synthesis from Palm Oil Using Continuous Circulation Reactor on Fuel Characteristics

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Abstract

The ever-growing demand for energy has driven many parties to seek more sustainable alternative fuels, and biodiesel from palm oil is a promising candidate. However, the biodiesel production process still faces several significant challenges, particularly regarding reaction effectiveness and transesterification stability. These two issues are the primary focus of this research. This study investigated the effects of circulation operating time and circulation flow rate in a continuous circulation reactor on biodiesel characteristics. Biodiesel synthesis was performed by varying the circulation operating time (60, 80, and 100 min) and the circulation flow rate (100, 300, and 500 LPM). Here, the circulation operating time refers to the duration of each experimental run during which the reaction mixture was continuously recirculated through the reactor. It represents the reactor operating duration rather than the hydraulic residence time (HRT). The resulting biodiesel was then tested for viscosity, density, flash point, calorific value, and yield. The test results showed that the viscosity of biodiesel ranged from 4.625 to 7.098 cSt, while the density was between 0.850 to 0.871 g/cm³. The calorific value of biodiesel ranged from 9915.56 to 10862.22 cal/gram, while the yield obtained was between 63.3% and 84.2%. The highest biodiesel yield (84.2%) was obtained at an operating time of 80 min and a circulation flow rate of 100 LPM. Extending the operating time to 100 min did not further increase the biodiesel yield. The highest calorific value, 10862.22 cal/gram, was achieved at an operating time of 60 minutes with a flow rate of 300 LPM. Overall, the continuous circulation reactor has good potential to increase the effectiveness of biodiesel synthesis from palm oil. Overall, the continuous circulation reactor demonstrated good potential for biodiesel production from palm oil. Among the investigated operating conditions, the highest biodiesel yield was obtained at an operating time of 80 min and a circulation flow rate of 100 LPM.

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1. Introduction

Industrial activity and global population growth have led to increased energy consumption (Vijay Kumar et al., 2019). On the other hand, current technological advances in 5.0 rely on electricity sources still derived from fuel oil (Jindapon et al., 2022). However, energy reserves, particularly petroleum, are increasingly limited. Furthermore, global oil prices fluctuate. Fluctuations in world oil prices over the past few decades have shown instability per barrel. A push is needed to initiate the use of alternative energy to support fuel demand. One potential alternative fuel development strategy is biodiesel (Mulalee et al., 2020; Prasetiyo et al., 2022; Winarko et al., 2022).

Biodiesel, derived from vegetable oil, faces limitations in its implementation in terms of reaction efficiency and stable operating conditions (Acevedo-Quiroz et al., 2024). However, biodiesel availability is required to be continuously available quickly, while its characteristics must meet SNI 7182:2024. Therefore, serious steps and efforts are needed to address these challenges (Prasetiyo et al., 2024, 2025). Possible steps include optimizing reactor operating conditions, particularly circulation operating time and circulation flow rate. Biodiesel, developed as an alternative energy source to fuel oil, can certainly reduce carbon emissions and encourage the development of a palm oil-based biodiesel industry, thus having great potential to improve the country's economy (Jindapon et al., 2020).

Previous research conducted by Phromphithak et al. (Phromphithak et al., 2020), examined the transesterification process of palm oil into biodiesel using a microwave-heated continuous flow reactor. The results showed that the optimum conditions were an oil-to-methanol ratio of 1:13.2 and a flow rate of 20 mL/min, thus meeting the ASTM D6751 biodiesel standard. Furthermore, Carlucci et al. (Carlucci, 2022), also conducted research on biodiesel synthesis using a continuous flow method. The results showed that the continuous flow method can improve process control, reaction efficiency, and operational safety, and has the potential to be applied on an industrial scale through the integration of modern technologies such as microwaves and ultrasound. Bačić et al. (Bacic et al., 2021) developed an integrated biodiesel production process. The system used was a microscale simultaneous glycerol extraction system aimed at improving mass transfer efficiency and biodiesel quality. Research using microsystems has shown the ability to produce biodiesel with a free glycerol content below international standards, thus offering potential for application in sustainable biodiesel processes. Based on previous research, it is clear that the development of biodiesel synthesis technology has largely focused on continuous flow and intensification systems. However, there has been little comprehensive reporting on the optimization of continuous circulation reactors for the characteristics of palm oil biodiesel fuel.

This study aimed to optimize biodiesel synthesis from crude palm oil (CPO) using a continuous circulation reactor. The continuous circulation system was chosen to improve the mixing quality between oil, methanol, and catalyst, thus improving the transesterification process. The main focus of this study was on the effect of circulation rate on the characteristics of the resulting biodiesel, such as yield, density, viscosity, flash point, and calorific value, thus meeting SNI 7182:2024 standards. Furthermore, this research is also expected to provide an alternative for developing more efficient biodiesel production technology with the potential for sustainable application on an industrial scale.

2. Material and Methods

The research method was conducted experimentally by directly observing the biodiesel product's characteristics and yield. The synthesis process was carried out using a continuous circulation reactor operated in a closed-loop recirculation mode. A schematic of the research to optimize biodiesel synthesis from palm oil using a continuous circulation reactor for fuel characteristics can be seen in Figure 1.

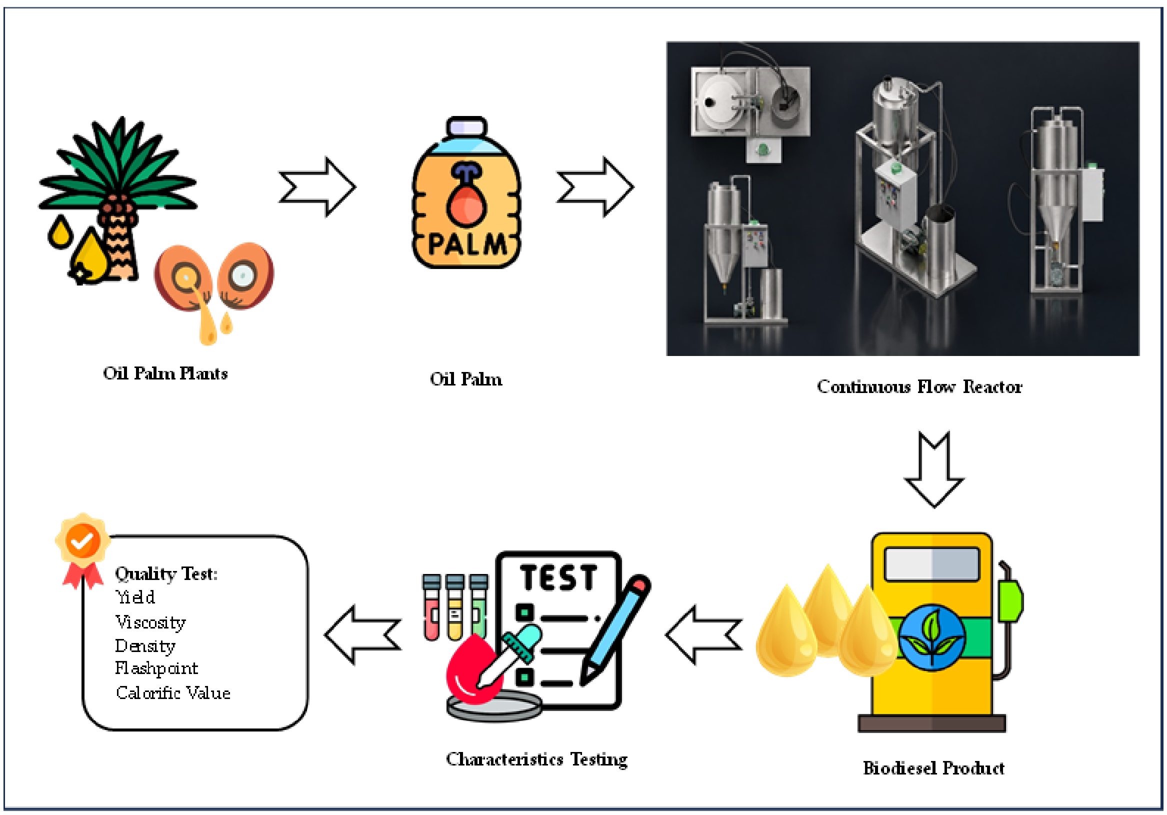

Figure 1. Research scheme for optimizing biodiesel synthesis from palm oil using a continuous circulation reactor.

The research began with degumming palm oil as the raw material. This was done to remove any remaining impurities, sap, or gum in the palm oil as the raw material. The degumming process was carried out using a mechanical stirrer accompanied by the application of heat to the palm oil. The composition in the degumming process was palm oil added with phosphoric acid (H3PO4) of one

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percent of the total mass of oil accompanied by the application of heat at 60oC and stirred for 30 minutes. The results of the degumming process were two layers between the oil and the remaining impurities. The degumming process scheme can be seen in Figure 2.

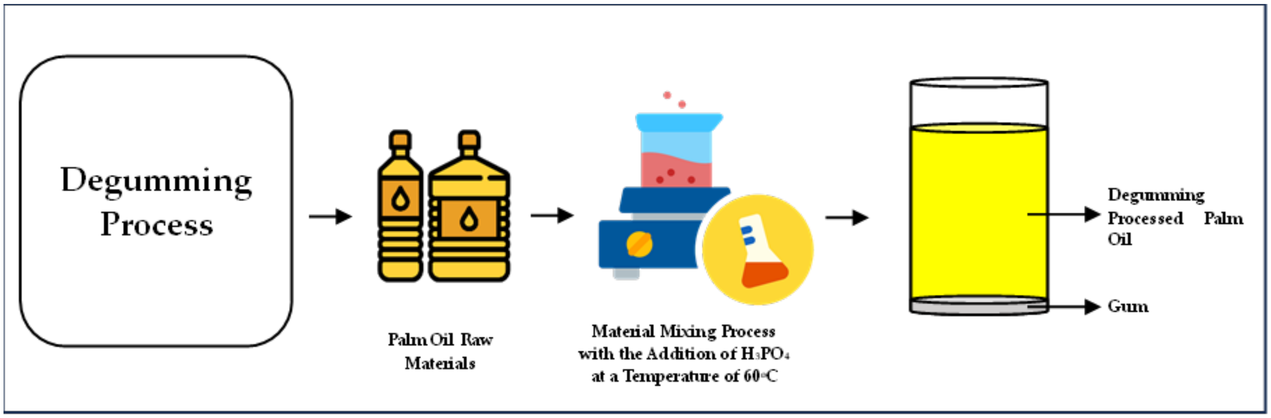

Figure 2. Schematic of the degumming process for optimizing biodiesel synthesis from palm oil.

After the free fatty acid reduction process, the transesterification process is continued. The transesterification process is carried out to convert Triglycerides into Methyl Ester (Biodiesel). This process is carried out by adding methanol and a base catalyst, namely NaOH. Methanol is added with a mole ratio of 1:6 (mol/mol), with the mass of NaOH being one percent of the total mass of the esterified oil. During the transesterification process, two operating parameters were studied, namely the circulation operating time (60, 80, and 100 minutes) and the circulation flow rate (100, 300, and 500 LPM). The circulation operating time refers to the duration of each experiment in which the reaction mixture is continuously circulated through the reactor using a recirculation pump. The image of the reactor components can be seen in Figure 3 while the transesterification process scheme can be seen in Figure 4. The reactor was operated in a closed-loop mode, where the same reaction mixture continuously passes through the loop until a predetermined operating time is reached. Therefore, the operating time studied represents the reactor's operating duration, not the hydraulic residence time (HRT). Since the reactor volume remained constant throughout the experiment, the hydraulic residence time was not varied independently. The circulation operating time was controlled by monitoring the elapsed time using a stopwatch. The recirculation pump was operated continuously throughout each experimental run and was manually stopped after the predetermined operating time (60, 80, or 100 min).

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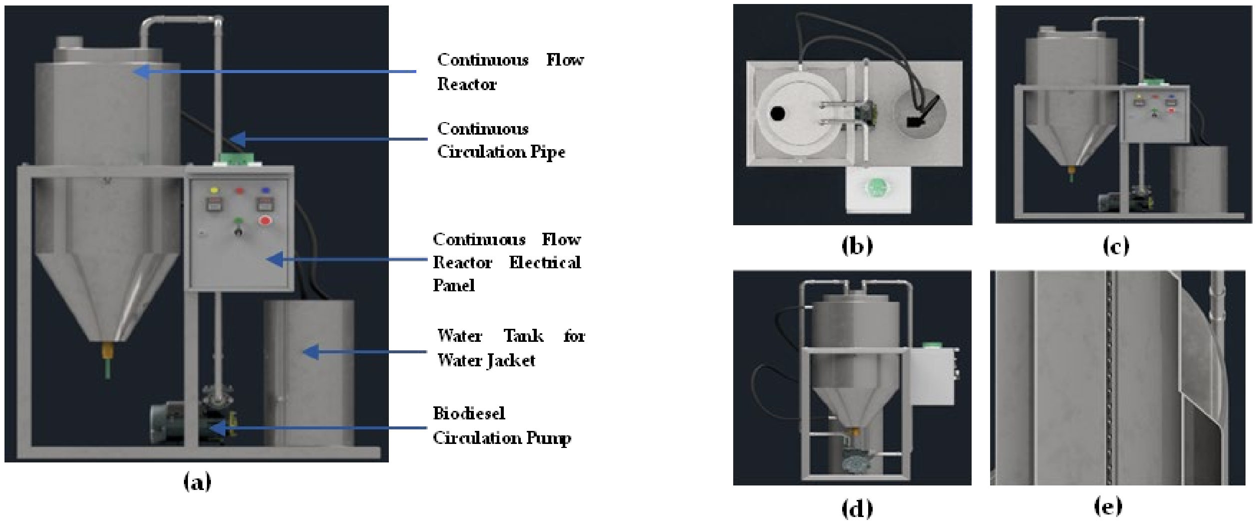

Figure 3. (a) Reactor components, (b) Top view of reactor, (c) Front view of reactor, (d) Side view of reactor, (e) Internal view of continuous circulation pipe.

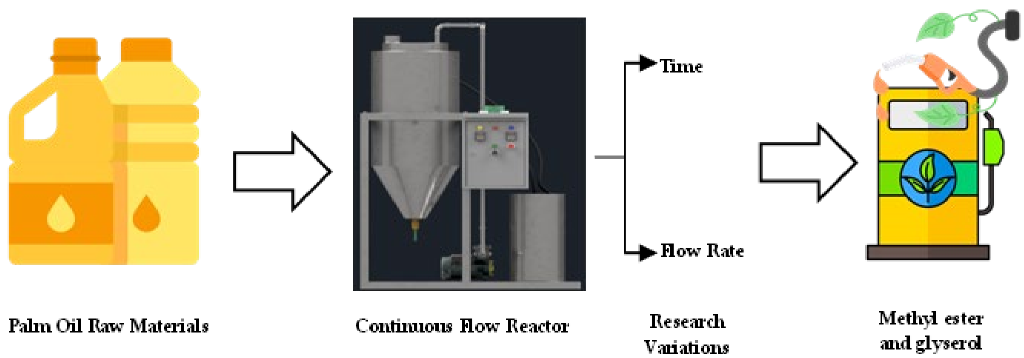

Figure 4. Schematic of the transesterification process for biodiesel synthesis from palm oil.

After the methyl ester is formed, the process continues with washing and evaporation. Washing is carried out to purify the biodiesel from residual catalyst, glycerol, and methanol residue, as well as soap, along with impurities after the transesterification reaction. At this stage, the biodiesel is washed using distilled water. The temperature used is 60oC. The washing process is repeated three times. After the washing process, the washed biodiesel product is evaporated. Evaporation is carried out to reduce water content. In addition, it reduces residual methanol and increases the storage stability of biodiesel, as well as preventing corrosion and microorganism growth during storage. The evaporation process is carried out by heating the biodiesel product to a temperature between 100oC and 110oC and stirring for five minutes. After the evaporation process, the process continues to the biodiesel quality testing stage. Biodiesel quality testing consists of testing viscosity, calorific value, flashpoint, density value, and calculating the resulting yield value. The test scheme can be seen in Figure 5.

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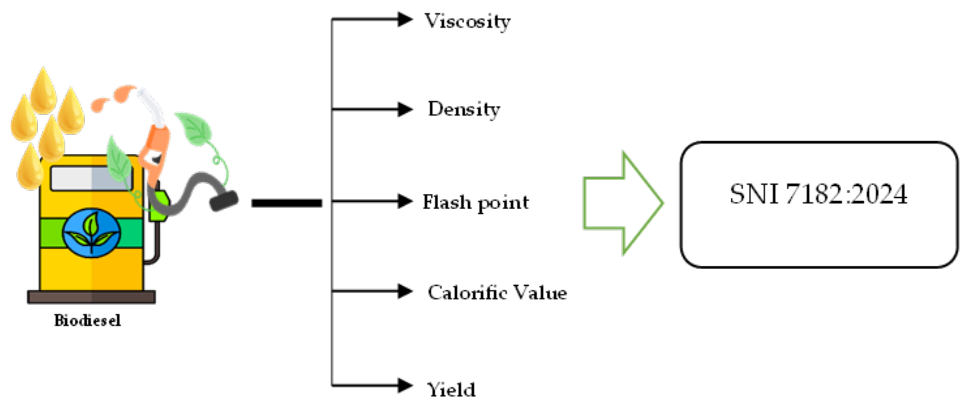

Figure 5. Scheme of quality testing of biodiesel that has been produced.

Viscosity testing was performed using an Ostwald Viscometer. Viscosity was measured by measuring the flow time of biodiesel from the upper to the lower limit of the capillary tube at a temperature of 40oC. The time required for the sample to pass both measurement marks was recorded using a stopwatch. Viscosity testing refers to the ASTM D445 standard. To obtain the viscosity value of biodiesel, calculations were carried out using equation (1).

$$\mu_b = \mu_a \times \frac{\rho_b \times t_b}{\rho_a \times t_a} \quad (1)$$

Description: μ_β: biodiesel viscosity (cP), μ_a : reference fluid/water viscosity (cP), ϱ_β : biodiesel density (g/cm^3), ϱ_a : reference fluid/water density (g/cm^3), t_β : biodiesel flow time (s), t_a : reference fluid/water flow time (s).

After obtaining the viscosity value in cP units, the unit conversion is continued to cSt units. To convert the values, use equation (2).

$$\nu = \frac{\mu}{\rho} \quad (2)$$

Description: ν: kinematic viscosity (cSt), μ: dynamic viscosity (cP), ϱ: biodiesel density (g/cm^3).

After viscosity testing, the test proceeds with density testing. Density testing is performed using a hydrometer. The test is conducted in accordance with the ASTM D1298 standard. The biodiesel sample is placed in a graduated cylinder up to 100 mL. The biodiesel being tested is at a temperature of 40°C. The hydrometer is then slowly inserted into the sample until it floats steadily. The density value is read on a scale parallel to the surface of the liquid at the test temperature. The test is conducted at room temperature to determine the density of the biodiesel produced.

The test continues with the calorific value test. The test is conducted using a Bomb Calorimeter System, with a voltage of 220 Volts. Prior to testing, the instrument is calibrated using benzoic acid. This is done as a reference standard to ensure the accuracy and stability of the measurement results, ensuring compliance with the ASTM D240 standard. During the testing process, a biodiesel sample is burned adiabatically in a closed chamber (bomb) to prevent heat from escaping to the environment. The resulting heat raises the temperature of the medium inside the calorimeter, which is then used to determine the biodiesel's calorific value.

Flash point testing aims to determine the lowest temperature at which fuel vapors begin to ignite. Measurements are performed using a closed-cup flash point tester according to standard methods such as ASTM D93. A 50 mL biodiesel sample is placed in the test cup and heated gradually with a controlled increase in temperature. During heating, a small flame is periodically applied to the sample surface to observe if a flash occurs. The temperature at which the flash first appears is recorded as the flash point. The higher this value, the safer the fuel is for storage and distribution. This parameter is important for safety in fuel storage and handling, particularly regarding volatility and fire risk.

Biodiesel yield measurements are conducted to determine the percentage of biodiesel produced. The yield value of palm oil biodiesel is obtained by comparing the mass of pure biodiesel with the initial mass of palm oil. Before measurement, the biodiesel is separated from glycerol, washed thoroughly, and heated to remove residual water and methanol. After the purification process, the biodiesel is weighed using a digital balance to obtain the final mass. To obtain the yield value of biodiesel, equation (3) is used.

$$Yield\ (\%) = \frac{m_b}{m_o} \times 100\% \tag{3}$$

Description: m_b: mass of biodiesel produced (grams),m_o: initial mass of palm oil (grams).

The test data were evaluated through standard deviation analysis, calculated based on repetitions for each parameter variation. This method is used to assess the distribution of data on observed parameters, such as yield, density, viscosity, flash point, and calorific value. A small standard deviation value indicates consistent and precise measurements, while a large value indicates significant variation in the experimental results. The equation used to calculate the standard deviation (SD) can be seen in equation (4). Once the SD value is known, the standard error (SE) is calculated, which is used to indicate the average accuracy of the data. The equation used to calculate the SE value can be seen in equation (5).

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}} \tag{4}$$

Description: s : standard deviation, x_i: i-th data value, $\bar{x}$: mean data value, n: number of data (replications).

$$SE = \frac{SD}{\sqrt{n}} \tag{5}$$

Description: SE: Standard Error, SD: Standard Deviation, n: number of data or number of replications.

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3. Results and Discussion

3.1 Effect of Circulation Operating Time and Flow Rate Variations on Biodiesel Viscosity

Viscosity is a physical parameter that plays a crucial role in determining biodiesel quality because it directly relates to the characteristics of fuel flow during the injection and combustion processes in a diesel engine. Viscosity values that are too high can hinder fuel atomization, resulting in uneven droplet distribution and incomplete combustion. This condition has the potential to increase carbon deposit formation, increase fuel consumption, and reduce overall engine performance. Therefore, viscosity control is a crucial aspect of the biodiesel synthesis process, particularly in continuous circulation reactor-based production systems that involve the interaction between operating time and flow rate. The results of the biodiesel viscosity test in this study are shown in Figure 6.

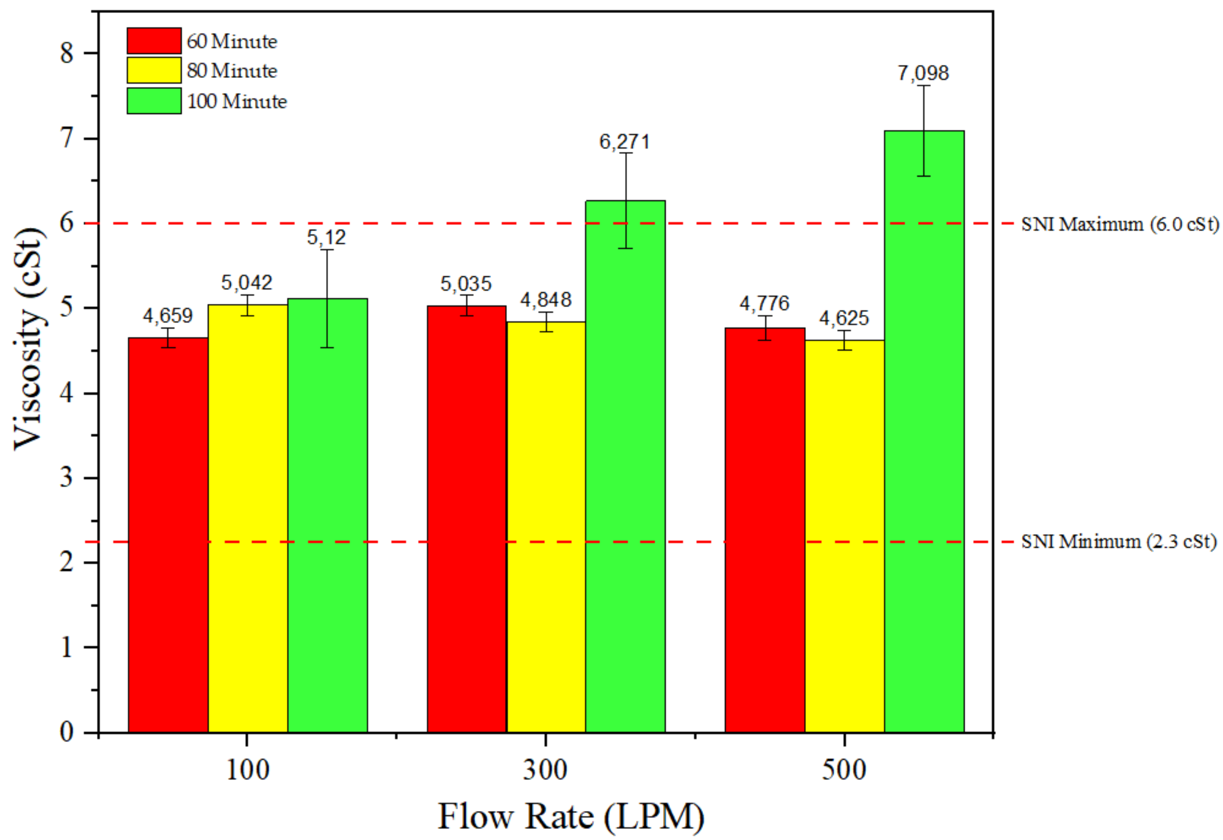

Figure 6. Effect of flow rate in continuous circulation reactor on palm oil biodiesel viscosity.

The viscosity value of the biodiesel produced is in the range of 4.625–7.098 cSt. Based on the SNI 7182:2024 value, the permissible viscosity value of biodiesel is in the range of 2.3 to 6.0 cSt. All biodiesel produced at 60 minutes and 80 minutes operating time still meets these requirements at all variations of circulation flow rates, while at 100 minutes operating time only the flow rate of 100 LPM still meets the standard with a viscosity of 5.120 cSt. Increasing the flow rate from 100 LPM to 300 LPM and 500

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LPM at 60 minutes operating time only causes a relatively small change in viscosity, namely from 4.659 cSt to 5.035 cSt and again decreasing to 4.776 cSt. A similar condition also occurred at 80 minutes of operating time, where the viscosity decreased gradually from 5.042 cSt to 4.848 cSt and 4.625 cSt as the circulation flow rate increased. Despite the change in viscosity value, all results at both operating times were still within the range required by SNI, so that the increase in circulation flow rate did not have a significant impact on the quality of biodiesel in terms of viscosity parameters. From an operational perspective, the use of a flow rate of 100 LPM is a more profitable choice because it has been able to produce biodiesel that meets the standards with lower pumping energy requirements compared to higher flow rates. In contrast to these two conditions, at 100 minutes of operating time there was a significant increase in viscosity to 6.271 cSt at a flow rate of 300 LPM and 7.098 cSt at a flow rate of 500 LPM, thus exceeding the maximum limit of SNI. Continuous mixing over a long period of time has the potential to increase the occurrence of side reactions, such as saponification, and inhibit the separation of glycerol from methyl ester, thus causing the viscosity of biodiesel to increase. Therefore, if the parameters considered are viscosity and process energy efficiency, the use of an operating time of 60 minutes with a circulation flow rate of 100 LPM is a more efficient operating condition because it has produced biodiesel that meets the requirements of SNI 7182:2024 with lower energy consumption. Overall, the results of the study indicate that the effect of operating time on biodiesel viscosity is more dominant than the effect of circulation flow rate. This can be seen from the sharp increase in viscosity at an operating time of 100 minutes, while changes in flow rate at operating times of 60 and 80 minutes only produce relatively small changes in viscosity and all still meet the requirements of SNI 7182:2024.

The results of this study align with the findings of previous studies. Kadi et al. stated that the use of a mixer reactor increases mass transfer between oil and methanol through more intensive flow mixing. This innovation increases the efficiency of transesterification of triglycerides to methyl esters and produces biodiesel that meets viscosity specifications according to international standards ASTM D6751 and EN 14214 (Kadi et al., 2019). Jindapon et al. also reported that increased mixing efficiency in a peristaltic reactor with bionic flow induction results in a more even temperature distribution and better reactant contact, thereby increasing biodiesel conversion efficiency compared to a conventional reactor (Jindapon et al., 2020). Similar findings were reported by Laziz et al., who showed that the flow recirculation pattern in a microchannel reactor accelerated mass transfer between oil and methanol. The transesterification process proceeded faster and resulted in a higher biodiesel conversion rate. These findings confirm that flow stability and mixing homogeneity are key factors in the effectiveness of methyl ester production. These results are also consistent with this study, where flow rate regulation in a continuous circulation reactor was shown to directly affect the viscosity of the biodiesel produced (Laziz et al., 2020). Therefore, optimizing operating parameters in a continuous circulation reactor is crucial to balance mixing intensity, residence time, and reaction stability.

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Based on the SNI 7182:2024 biodiesel quality standard, the required viscosity is 2.3–6.0 cSt. Most of the research specimens met this standard. However, at an operating time of 100 minutes with a flow rate of 300 LPM and 500 LPM, the viscosities were 6.271 cSt and 7.098 cSt, respectively, exceeding the maximum limit. This indicates that the combination of too long an operating time and a high flow rate can reduce the quality of biodiesel in terms of viscosity. Therefore, optimizing the operating parameters in a continuous circulation reactor is crucial to produce biodiesel with flow characteristics that meet the standards.

3.2 Effect of Circulation Operating Time and Flow Rate Variations on Biodiesel Flash Point

Flash point is an important thermal parameter in biodiesel and is closely related to fuel safety during storage, distribution, and use in diesel engines. In biodiesel, the flash point is often used as an indirect indicator of the presence of residual methanol from the transesterification process. Biodiesel with a high methanol content usually has a lower flash point because methanol evaporates easily at low temperatures. Therefore, the flash point is influenced by the methyl ester composition, reaction efficiency, glycerol separation, and the purification process after synthesis. The results of the biodiesel flash point measurements in this study are shown in Figure 7.

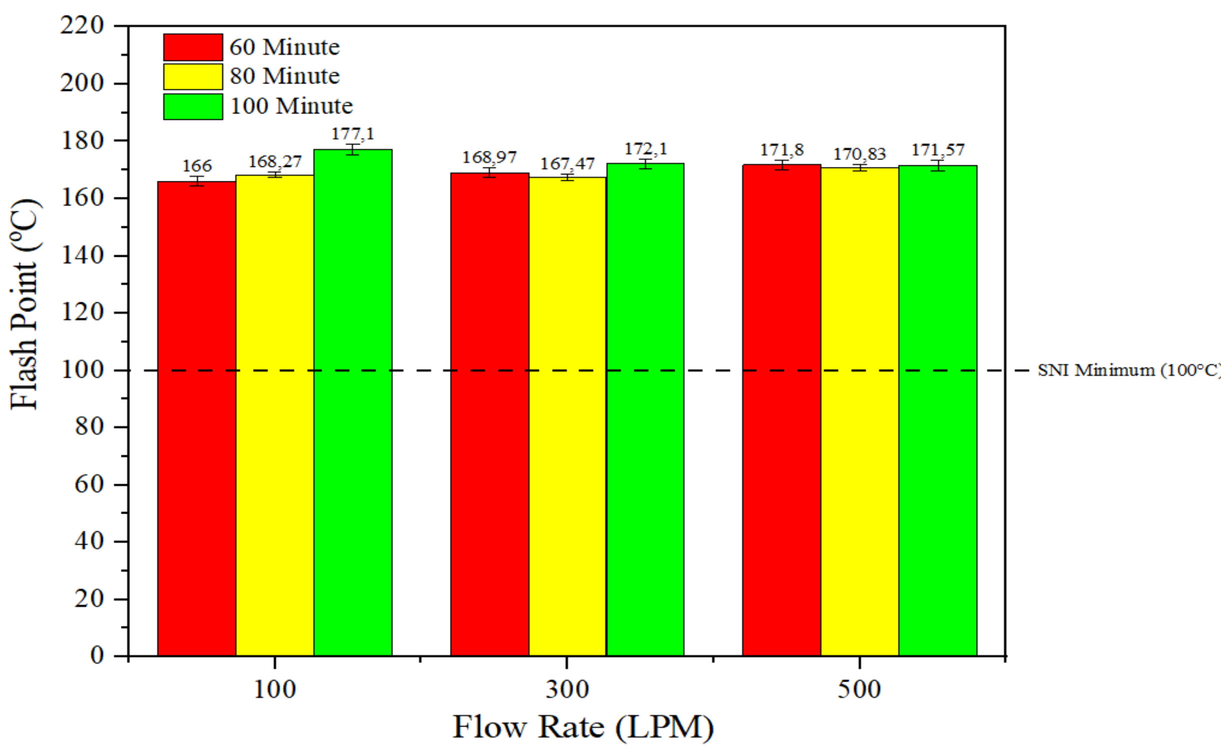

Figure 7. Effect of flow rate and circulation operating time variations on biodiesel flash point.

Figure 7 shows that the flash point of biodiesel is in the range of 166.0–177.1°C. Based on SNI 7182:2024, all biodiesel produced has met the minimum flash point requirement of 100°C. At an

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operating time of 60 minutes, increasing the flow rate from 100 LPM to 500 LPM increases the flash point gradually from 166.0°C to 171.8°C. At an operating time of 80 minutes, the change in flow rate only causes a relatively small change in the flash point, namely from 168.27°C to 167.47°C and then increasing again to 170.83°C. Meanwhile, at an operating time of 100 minutes, the highest flash point value was obtained at 177.1°C at a flow rate of 100 LPM, then decreased to 172.1°C at 300 LPM and 171.57°C at 500 LPM. These results indicate that extending the operating time to 100 minutes at a flow rate of 100 LPM produces the best thermal characteristics of biodiesel, while increasing the flow rate above 100 LPM no longer provides an increase in the flash point value. This decrease is thought to be related to changes in mixing conditions and the glycerol separation process so that the removal of residual methanol is not as effective as at lower flow rates.

These results align with several previous studies that examined the effects of flow characteristics, reactant residence time, and mixing intensity on biodiesel quality in continuous reactor systems. Subramaniam et al., found that the use of a microchannel reactor can increase heat and mass transfer between reactants through more intensive mixing, so that the transesterification process becomes faster and more efficient. This increased mass transfer makes the distribution of methanol in the oil more even and accelerates the formation of methyl esters (Sharma et al., 2021). Furthermore, Silva and Andrade, through simulation and optimization of a continuous stirred tank reactor (CSTR), demonstrated that reaction temperature, alcohol-to-oil ratio, and reactant residence time are the main parameters affecting ester conversion and biodiesel characteristics in a continuous system. The study showed that increasing the reactant residence time under optimum operating conditions can produce biodiesel with quality that meets ASTM D6751 and EN 14214 standards (Silva & Andrade, 2020). Jazie also reported that the residence time distribution of reactants in a CSTR system affects the physical characteristics of biodiesel, particularly viscosity and flash point. Biodiesel produced under optimum operating conditions has a higher flash point than conventional diesel fuel, making it safer for storage and distribution (Jazie, 2020). Sharma et al., explained that temperature and weight hourly space velocity (WHSV) in the continuous co-processing process have a large influence on the thermal characteristics of the fuel, including the flash point and boiling point distribution of the products (Sharma et al., 2021). Meanwhile, Kaisan et al. stated that biodiesel with good combustion quality usually has a high flash point value and still complies with ASTM standards, so it is safe for use in diesel engine (Kaisan et al., 2017). These findings demonstrate that mixing homogeneity, flow stability, reactant residence time, and mass transfer efficiency are crucial for producing biodiesel with acceptable thermal characteristics. This is also evident in this study, where the flow rate and operating time settings in the continuous circulation reactor directly impact the flash point characteristics of the resulting biodiesel.

When compared with the SNI 7182:2024 biodiesel quality standard, all biodiesel results from this study met the minimum flash point requirement for biodiesel, which is above 100°C. The relatively

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high flash point values indicate that the synthesized biodiesel has a low methanol residue content, making it safer for storage and use as an alternative fuel. These results also demonstrate that optimizing the operating time and flow rate in the continuous circulation reactor plays a crucial role in producing biodiesel with stable thermal characteristics that meet diesel fuel quality standards.

3.3 Effect of Circulation Operating Time and Flow Rate Variations on the Calorific Value of Biodiesel

The calorific value is a key parameter for assessing the effectiveness of biodiesel as an alternative fuel, as it directly determines the amount of heat energy released during combustion. The energy produced influences the fuel's ability to generate power in diesel engines. The calorific value of biodiesel is influenced by various factors, such as the fatty acid profile, the efficiency of triglyceride conversion to methyl ester, oxygen concentration, and the presence of contaminants such as glycerol, soap, water, and residual methanol from the transesterification process. Therefore, evaluating the calorific value is a crucial step in assessing biodiesel synthesis methods, particularly in continuous circulation reactor-based production systems that optimize mass transfer by adjusting operating time and flow rate. The results related to the calorific value of biodiesel in this study are shown in Figure 8.

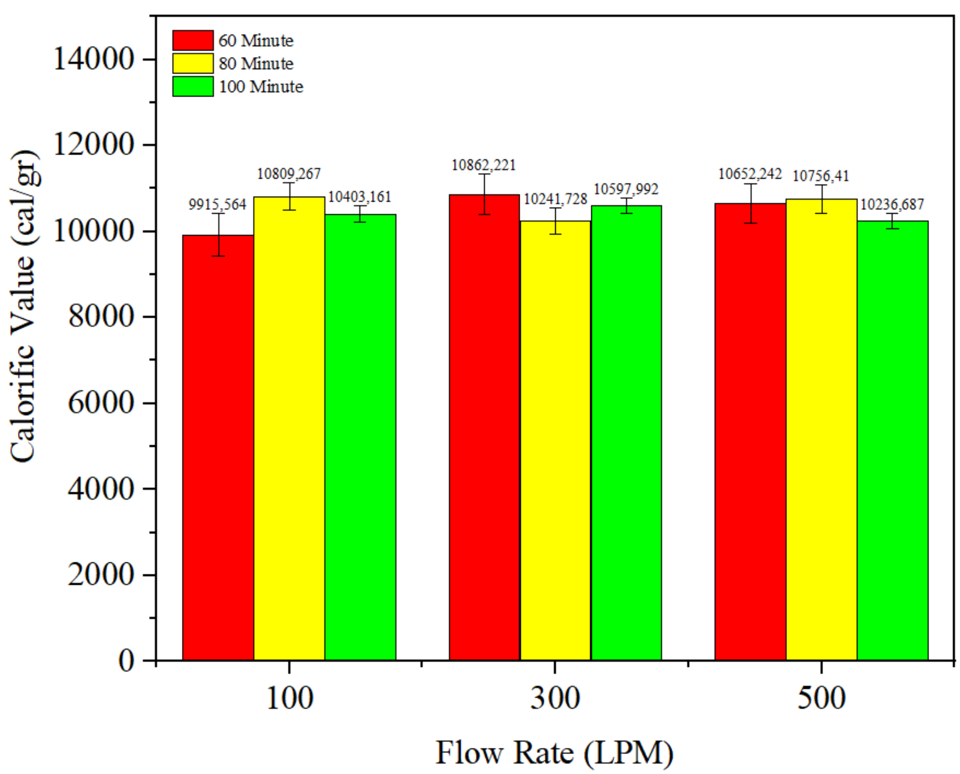

Figure 8. Effect of flow rate and circulation operating time variations on the calorific value of biodiesel.

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Figure 8 shows the test results. The test results show that biodiesel has a calorific value between 9915.56 and 10862.22 cal/gram. These results indicate that operating time and flow rate affect the energy characteristics of biodiesel. At an operating time of 60 minutes, increasing the flow rate from 100 LPM to 300 LPM increased the calorific value from 9915.56 to 10862.22 cal/gram. This may be attributed to more effective mixing between the reactants, which likely promoted a more complete transesterification process. However, when the flow rate was increased to 500 LPM, the calorific value dropped to 10652.24 cal/gram, this decrease may be associated with excessive circulation intensity, which could reduce the effectiveness of the transesterification process. At 80 minutes of operating time, the calorific value tended to stabilize at 10,809.27 cal/gram at 100 LPM, decreasing to 10,241.73 cal/gram at 300 LPM, and then increasing again to 10,756.41 cal/gram at 500 LPM. This indicates that the transesterification process is becoming more stable, so changes in flow rate do not significantly affect methyl ester formation. At 100 minutes of operating time, the calorific value decreased again, particularly at 500 LPM, reaching 10,236.69 cal/gram. This decrease is likely due to increased side reactions such as saponification, which inhibits glycerol separation and reduces biodiesel purity.

Furthermore, excessively long operating times can also potentially reduce biodiesel quality. This is due to changes in the physicochemical properties of the fuel during the process. Mota et al. reported that excessively increasing operating time can lead to a decrease in biofuel quality, resulting in the reaction process only being effective for a limited time. This condition shows that the stability of biodiesel is greatly influenced by the operating parameters during the conversion process (Mota et al., 2021). On the other hand, Hendrawan et al. stated that the reaction temperature and transesterification operating conditions directly influence the calorific value of the resulting biodiesel. This indicates that optimizing operating parameters is a crucial factor in producing biodiesel with high and stable energy characteristics (Hendrawan et al., 2021).

3.4 The Effect of Operating time and Flow Rate Variations on Biodiesel Density

Biodiesel density is an important parameter for assessing fuel quality, particularly during the injection process in diesel engines. Density affects the amount of fuel mass entering the combustion chamber during each combustion cycle, thus determining combustion efficiency and engine performance. If the biodiesel density is too high, atomization will be poor due to the formation of larger droplets. Conversely, a density that is too low can reduce the energy released during combustion due to the smaller mass of fuel injected. Therefore, density stability is an important indicator for assessing the success of the biodiesel transesterification process. The results of the biodiesel density test in this study can be seen in Figure 9.

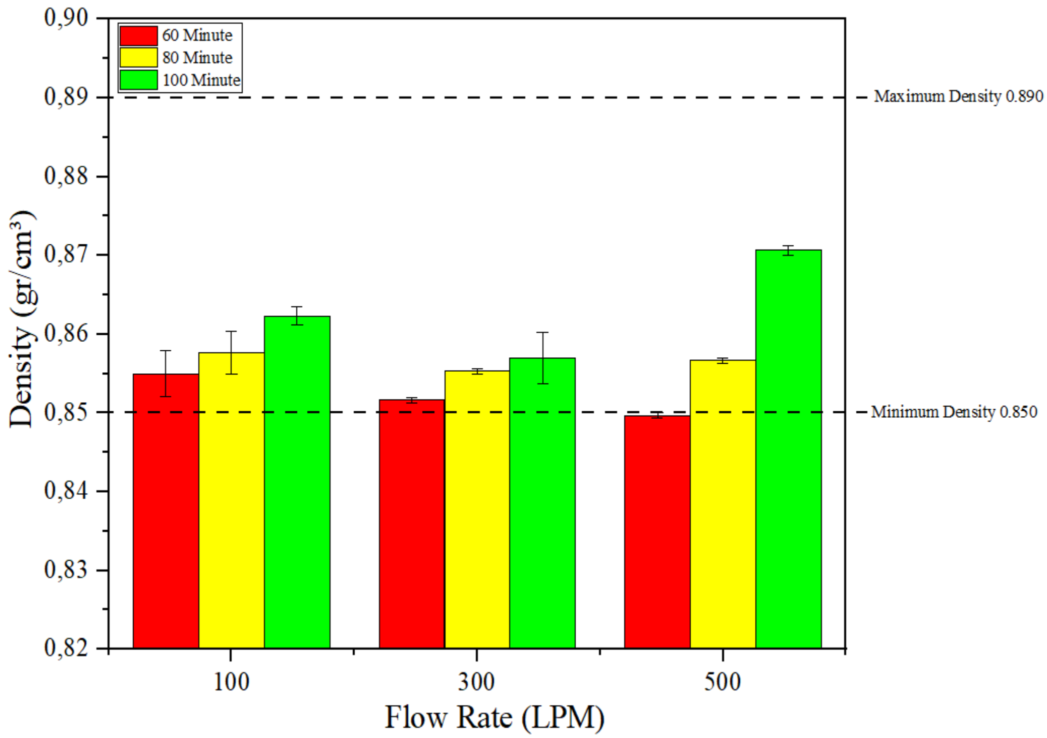

Figure 9. Effect of flow rate and circulation operating time variations on biodiesel density.

Tests showed that the resulting biodiesel had a density ranging from 0.850 to 0.871 g/cm³. This range indicates that variations in operating time and circulation flow rate produced only relatively small changes in biodiesel density. This small fluctuation indicates that the continuous circulation reactor system is capable of maintaining a stable transesterification process throughout the reaction. These relatively small density variations suggest that the continuous circulation reactor provided stable operating conditions during the transesterification process. At an operating time of 60 min, increasing the circulation flow rate from 100 LPM to 300 LPM and 500 LPM gradually decreased the biodiesel density from 0.855 g/cm³ to 0.851 g/cm³ and 0.850 g/cm³, respectively. This decrease indicates that a higher circulation rate early in the reaction can enhance mass transfer between the reactants. More intensive mixing is thought to accelerate the contact between triglycerides and methanol, thus optimizing the formation of methyl esters. As triglyceride conversion increases, the content of heavy compounds such as triglycerides and diglycerides in biodiesel decreases. Consequently, the density of biodiesel decreases because methyl esters have a lower density compared to unconverted vegetable oil. Furthermore, the decrease in density also indicates that the content of impurities such as glycerol and catalyst residues begins to decrease as the effectiveness of the transesterification process increases. However, a different trend is seen at operating times of 80 minutes and 100 minutes. At an operating time of 80 min, the biodiesel density showed only minor fluctuations with increasing circulation flow rate, changing from 0.858 g/cm³ at 100 LPM to 0.855 g/cm³ at 300 LPM and 0.857 g/cm³ at 500 LPM.

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Although these variations were relatively small, this pattern indicates a change in reaction characteristics as the system operates for longer periods and at higher flow rates. This shows that increasing the circulation flow rate only results in a relatively small change in density as long as all results remain within the SNI specification range.

The increase in density under these conditions likely occurs because the phase separation between biodiesel and glycerol is incomplete after the transesterification reaction. At high flow rates, the flow in a continuous circulation system becomes more turbulent. This makes the reactor's hydrodynamic stability more difficult to maintain. As a result, some glycerol, catalyst residue, or intermediate compounds from the reaction are still carried into the biodiesel, causing the fuel density to increase (Hoang, 2018). This is consistent with research by Mairizal et al., who stated that biodiesel density is closely related to the chemical composition of the fuel, including intermediate compounds and the purity of the resulting methyl ester (Mairizal et al., 2020). Meanwhile, Burmana et al., explained that increasing operating time does not always improve biodiesel quality because there is an optimum reaction point, where increasing operating time too long no longer significantly increases methyl ester conversion. This phenomenon indicates that the effectiveness of product separation and reaction stability are crucial in determining biodiesel density. However, the relatively small changes in density across all test variations indicate that the continuous circulation reactor system is still capable of maintaining the stability of the transesterification process well (A D Burmana et al., 2020).

When compared with the SNI 7182:2024 biodiesel quality standard, all biodiesel results from the study were still within the standard density range, namely 0.850–0.890 g/cm³. These results indicate that the biodiesel produced with the continuous circulation reactor system has met the density requirements for use as an alternative fuel in diesel engines. Overall, the results demonstrate that variations in operating time and circulation flow rate influenced biodiesel density while maintaining all density values within the SNI 7182:2024 specification. The observed changes were relatively small, indicating that the continuous circulation reactor provided stable mixing and transesterification conditions throughout the process.

3.5 Effect of Circulation Operating Time and Flow Rate Variations on Biodiesel Yield

Biodiesel yield is a key parameter for assessing the success of the transesterification process because it indicates how much palm oil is successfully converted into methyl esters. The yield value is not only related to the amount of biodiesel produced but also reflects the effectiveness of mixing, reaction stability, mass transfer, and the quality of reactor operation. The higher the yield, the more effective the triglyceride conversion during biodiesel synthesis. Therefore, analyzing yield changes is important to understand the effect of operating time and circulation rate on the performance of the continuous circulation reactor in this study. The results of the biodiesel yield test can be seen in Figure 10.

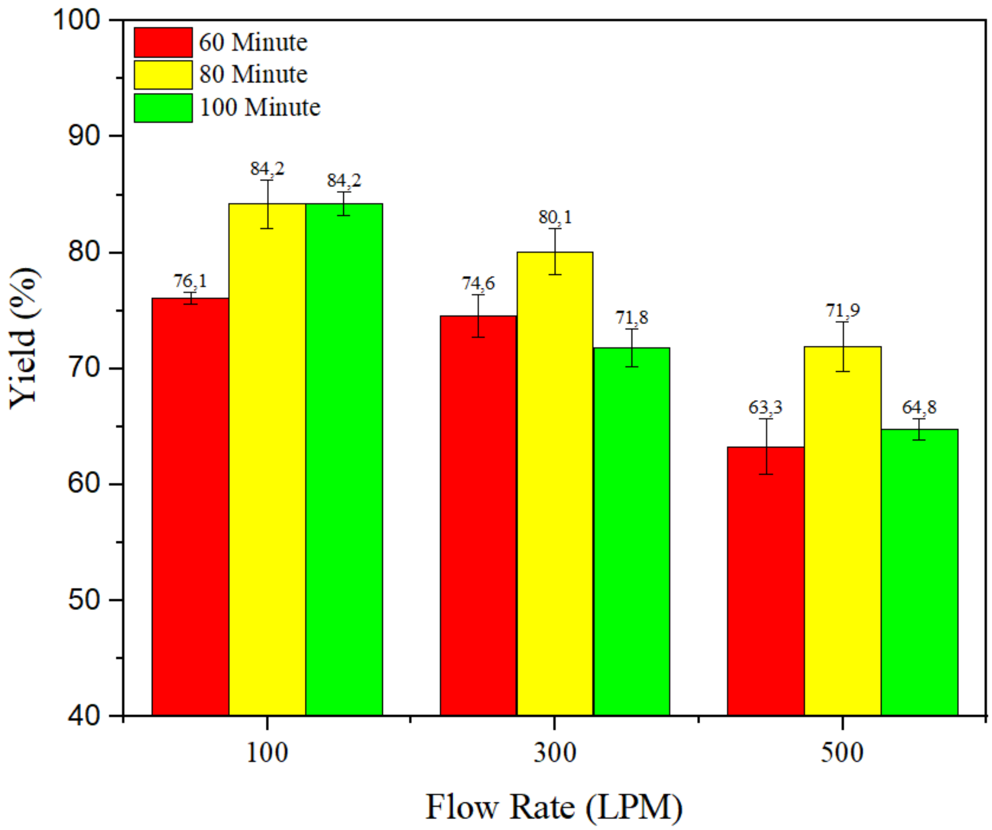

Figure 10. Effect of flow rate and circulation operating time variations on biodiesel yield.

This study found that the biodiesel yield ranged from 63.3% to 84.2%. In general, increasing the operating time from 60 to 80 min increased the biodiesel yield. However, extending the operating time to 100 min did not further improve the yield, particularly at circulation flow rates of 300 and 500 LPM. At a circulation flow rate of 100 LPM, the biodiesel yield remained constant at 84.2% for both 80 and 100 min. However, increasing the flow rate beyond a certain limit actually decreased the amount of biodiesel produced. This indicates that the success of the transesterification process in a continuous circulation reactor depends not only on the intensity of mixing, but also on the balance between reactant contact time, flow stability, and the effectiveness of product separation.

At an operating time of 60 minutes, the biodiesel yield decreased significantly as the flow rate increased. At a flow rate of 100 LPM, the biodiesel yield was 76.1%, dropping to 74.6% at 300 LPM, and then dropping again to 63.3% at 500 LPM. This decrease indicates that at short operating times, increasing the circulation rate is not sufficient to support optimal transesterification. Increasing the circulation flow rate reduces the effective contact duration between methanol and triglycerides during each circulation cycle. Under a fixed operating time, this condition may reduce the extent of transesterification (Yu et al., 2017). As a result, some of the triglycerides have not been converted into methyl esters and the amount of biodiesel produced is less (Woo & Kim, 2019). Furthermore,

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excessively strong circulation increases flow turbulence, thus reducing the stability of the reaction phase within the reactor. Despite more intensive mixing, effective contact between reactants is actually less than optimal due to excessively fast flow. This indicates that increasing the circulation flow rate alone does not necessarily improve biodiesel yield, particularly when the operating time is limited to 60 min. In contrast, extending the operating time to 80 min increased the biodiesel yield at all circulation flow rates. The highest yield was obtained at a flow rate of 100 LPM (84.2%), while at 300 LPM and 500 LPM (80.1%) and 71.9%, respectively. This increase indicates that longer operating times provide a greater opportunity for the transesterification process to proceed more completely. The longer reaction duration allows triglycerides and methanol to have sufficient contact time, thus optimizing methyl ester formation. Under these conditions, a continuous circulation system can produce more homogeneous mixing and increase the effectiveness of mass transfer between reactants. The increase in biodiesel yield at 80 min may be attributed to more effective mixing and sufficient operating time, which promoted a more complete transesterification process. However, at too high a flow rate, excessive mixing intensity can potentially trigger the formation of an emulsion between biodiesel and glycerol, resulting in incomplete phase separation. This can cause some biodiesel to be retained with the glycerol during the separation and washing processes, resulting in a lower final biodiesel yield.

At an operating time of 100 minutes, biodiesel yield decreased again as the flow rate increased. The highest yield remained at 84.2% at 100 LPM, then decreased to 71.8% at 300 LPM and 64.8% at 500 LPM. These results indicate that longer operating times do not always increase the amount of biodiesel produced. Under some conditions, excessively long operating times can trigger side reactions such as saponification due to the interaction between free fatty acids and the base catalyst. This reaction produces soap, which can inhibit the separation of biodiesel and glycerol and increase biodiesel loss during washing. Furthermore, the combination of long operating times and high circulation rates also reduces the stability of the reactor system. Continuously cycling the flow for extended periods increases the likelihood of microemulsions forming, making separation of the reaction products more difficult. As a result, some biodiesel remains mixed with glycerol or other compounds, reducing the amount of pure biodiesel successfully separated and decreasing biodiesel yield. This phenomenon indicates that biodiesel synthesis in a continuous circulation reactor is strongly influenced by the balance between operating time, mixing intensity, and the stability of the reaction phase separation.

Overall, this study demonstrates that a continuous circulation reactor can improve the effectiveness of biodiesel synthesis through mixture homogenization and enhanced mass transfer between reactants. However, increasing the operating time and circulation flow rate beyond certain operating conditions did not always result in higher biodiesel yields. Among the investigated operating conditions, the highest biodiesel yield (84.2%) was obtained at an operating time of 80 min with a circulation flow rate of 100 LPM. Extending the operating time to 100 min did not provide any additional increase in biodiesel yield under the same circulation flow rate. The consistency was quite good in each test repetition. This indicates that the continuous circulation reactor system is capable of producing biodiesel with relatively stable yield characteristics. Thus, the setting of operating time and flow rate is a very important factor in determining the success of the palm oil biodiesel synthesis process, especially in efforts to obtain biodiesel with high yields and better quality for industrial scale development.

4. Conclusion

The conclusion of the research results on the optimization of biodiesel synthesis from palm oil using a continuous circulation reactor on fuel characteristics shows that changes in operating time and flow rate in the continuous circulation reactor directly affect the characteristics of palm oil biodiesel, such as viscosity, density, calorific value, and biodiesel yield. Viscosity testing showed biodiesel viscosities ranging from 4.625 to 7.098 cSt. According to SNI 7182:2024, the acceptable viscosity range is 2.3–6.0 cSt. Most samples complied with this requirement, except those produced at an operating time of 100 min with circulation flow rates of 300 and 500 LPM, which exceeded the maximum allowable viscosity of 6.0 cSt. Density testing showed biodiesel densities ranging from 0.850 to 0.871 g/cm^3. Stable densities indicate that the continuous circulation reactor system can maintain mixing homogeneity during the transesterification process. The calorific value of the biodiesel produced ranged from 9915.56 to 10862.22 cal/gram. The highest calorific value was obtained at an operating time of 60 min and a circulation flow rate of 300 LPM (10862.22 cal/gram). These results indicate that the selected operating time and circulation flow rate influenced the calorific value of the biodiesel. The higher calorific value obtained under these operating conditions may be associated with a more effective transesterification process, resulting in improved fuel energy characteristics. The highest biodiesel yield (84.2%) was obtained at an operating time of 80 min and a circulation flow rate of 100 LPM. Extending the operating time to 100 min did not further increase the biodiesel yield under the same circulation flow rate.

Overall, the experimental results demonstrated that all biodiesel produced at an operating time of 60 min and a circulation flow rate of 100 LPM satisfied the quality requirements specified in SNI 7182:2024 while requiring the shortest processing time and the lowest circulation energy. Although higher biodiesel yields were obtained at 80 min, the selection of operating conditions should consider not only biodiesel yield but also compliance with fuel quality standards and process efficiency..

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6. AI Writing Statement

The authors used the generative AI tool Gemini solely to assist with English language editing, grammar correction, sentence restructuring, and enhancing the clarity and readability of the manuscript during the writing and revision process. The core elements of the manuscript including the research concept, experimental design, data collection, data analysis, interpretation of results, and scientific conclusions were developed entirely by the authors. Gemini was not used to generate or modify research data, conduct scientific analysis, interpret experimental results, or formulate the conclusions presented in this study. All AI assisted content was carefully reviewed, verified, and revised by the authors, who bear full responsibility for the accuracy and integrity of the final manuscript.

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