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# Non-Catalytic Biodiesel Synthesis from Kemiri Sunan (*Reutealis trisperma*) Oil in a Semi-Batch Bubble Column Reactor

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## Abstract

*The bubble column reactor (BCR) is intensive equipment used as a multiphase contactor and reactor in the chemical, biochemical, and petrochemical industries. BCR has advantages, including high mass and heat transfer rates, compactness, and low operating and maintenance costs. This research applied BCR to produce biodiesel from Kemiri Sunan (Reutealis trisperma) oil through non-catalytic and simultaneous esterification and transesterification reactions. Kemiri Sunan oil was fed into the reactor and heated to a specific temperature. During the heating process, nitrogen gas was flown to prevent oxidation. Liquid methanol was flown at various flow rates through a vaporizer and superheater so that it was delivered into the reactor in the form of the saturated condition (1 atm, 240 ºC). Experiments with variations in the methanol flow rate were carried out at a reaction temperature of 250 ºC, while experiments with various temperatures were carried out at a methanol flow rate of 2.5 mL/min. The best conditions were obtained at a 2.5 mL/min methanol flow rate with a reaction temperature of 290 ºC. The production rate was 3.47 g/min with an acid number of 0.69 mg KOH/g sample, and FAME content was 78.2% wt. These results indicate the occurrence of simultaneous esterification and transesterification reactions in one reactor.*

**Keywords:** *Kemiri Sunan; biodiesel; non-catalytic simultaneous reaction; esterification; transesterification*

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## INTRODUCTION

Biodiesel is an alternative diesel fuel derived from vegetable oils and animal fats consisting of monoalkyl esters formed from a catalyzed triglyceride or fat reaction with simple monohydric alcohol (Krawczyk, 1996; Mittlebach and Remschmit, 2004; Knothe et al., 2015). Biodiesel can be produced catalytically and non-catalytically. The process of making biodiesel without catalyst has advantages including: (i) it is not necessary to remove free fatty acids by refining or pre-esterification, (ii) esterification and transesterification reactions can take place simultaneously in one reactor so that oil with high free fatty acid levels can be directly used, (iii) simpler product separation and purification process conditions, and (iv) environmentally friendly. On the other hand, slower reactions, higher reaction temperatures than catalytic reactions, and typically working at elevated or supercritical pressures are drawbacks of this process.

Several researchers have reported the process of biodiesel production in a non-catalytic way. Among them in the supercritical conditions of methanol by

Demirbas (2003). Kusdiana and Saka (2000) produced biodiesel with reaction conditions of 350 °C, 20 mPa (200 bar), with a ratio of methanol to the oil of 42 moles. The reaction can occur very quickly, with a perfect reaction time of only 4 minutes. However, in addition to high investment and manufacturing costs, the usage of high-pressure reactors risks risking operator safety and protection. As a result, it must also be considered if used commercially.

One of the biodiesel production process technologies that have been developed previously is the production of non-catalytic biodiesel in a bubble column reactor (BCR). This research studies the effect of reaction temperature and methanol flow rate on the conversion, yield, and product quality (purity) of biodiesel at 1 atm pressure, both for esterification reactions with fatty acid raw materials (Joelianingsih et al., 2008) and transesterification with raw materials of pure vegetable oil or triglyceride (Joelianingsih et al., 2012). The advantages of BCR are simple construction, low operating costs, high energy efficiency, heat transfer, and mass transfer occur well (Mouza et al., 2004). However, research on the performance of BCR for the simultaneous esterification and transesterification reaction using raw materials of vegetable oil, especially Kemiri Sunan oil, which has high free fatty acids, has never been reported before. Usually, the processing steps depend on the free fatty acid content of the oil, whether it requires esterification or directly transesterification without esterification.

Kemiri Sunan (Reutealis trisperma (Blanco) airy shaw) is a poisonous candlenut plant. This native tropical plant has become one of the most potent plants for producing non-food vegetable oils. Kemiri Sunan fruit is rounded with three angles with a diameter of 3-6 cm, contains three loci, each containing one seed; young fruit is green and becomes brownish after ripe, as shown in Figure 1. Fruit consists of fruit skin (62-68 %), seed coat (11-16%), and core / seed flesh (16-27%). The kernel of Kemiri Sunan contains 38.7-50% crude oil. Initial water content in the core of Kemiri Sunan seeds is around 19-34%, which can cause hydrolysis reaction in oil, causing high levels of FFA (Pranowo et al., 2014).

Figure 1. a) Fruit, b) Fruit Skin, c) Seeds and d) Kernel of Kemiri Sunan

In the use of BCR for non-catalytic simultaneous esterification and transesterification reactions, the role of the catalyst is replaced by a higher reaction temperature (250-290 ºC). In the process with a homogeneous catalyst, the reaction temperature is around 65 ºC at 1 atm pressure (boiling temperature of methanol) and using a stirred tank reactor. In the non-catalytic reaction, the stirring role is replaced by methanol vapor bubbles. Methanol is kept in a liquid phase in the process with a homogeneous catalyst. Therefore, efforts to speed up the reaction by raising the temperature must always be followed by an increase in pressure. This weakness is overcome by utilizing the liquid methanol changes to vapor at temperatures above 65 °C so that it can be bubbled. The reaction between oil and methanol takes place in the interface layer of the oil and the methanol bubble. So that the more the number of bubbles and the smaller the diameter of the bubble, the higher the contact area of the interface, and the reaction will be faster, in other words, the effort to speed up the chemical reaction that was originally carried out with the help of catalysts that are chemical and conventional stirring is changed by physical and mechanical treatment following the characteristics of the methanol reagent.

In this study, the efficiency of BCR for simultaneous esterification and transesterification reactions using Kemiri Sunan oil as a raw material was investigated by observing the effect of methanol flow rate and reaction temperature in a semi-batch operation. In the future, BCR will be used to produce biodiesel without catalyst at ambient pressure, using vegetable oils as raw materials with high FFA levels, making it environmentally safe. This procedure is also safer since the BCR is a reactive-distillation, allowing for 100% conversion. The reactor is not only a place for the reaction but also a place for product separation.

## MATERIALS AND METHOD

### Materials

As a source of raw materials for vegetable oils (triglycerides), Kemiri Sunan oil was obtained from PT. Darma Prema Bioenergy (West Bandung, Indonesia). The water content of Kemiri Sunan oil was 0.11% wt. and acid number 59.10 mg KOH/g. Methanol as a reagent with a purity of 99.8% wt. was obtained from PT. Smart - Lab, Indonesia. Nitrogen Gas was supplied by CV. Mahesa Gas Pusaka-Jakarta. With a purity of 99.5%, Toluene was used as a solvent in the FAME content test was obtained from PT. Smart - Lab, Indonesia. Whereas Methyl Nonadecanoate for internal standards on the FAME test was obtained from Sigma Aldrich.

### Equipment

The equipment used in this study was a set of equipment consisting of a vaporizer, superheater, BCR, and condenser. That equipment was equipped with pumps, indicators, temperature control, pressure control, and flow rate control, as shown in Figure 2. The BCR dimension has a H/D ratio of 5, the sparger was a 10 cm hollow plate made of SS-316 material with a hole diameter of 1 mm, and the number of holes was 119. As a result, the hole area covers about 1.2% of the total sparger plate area.

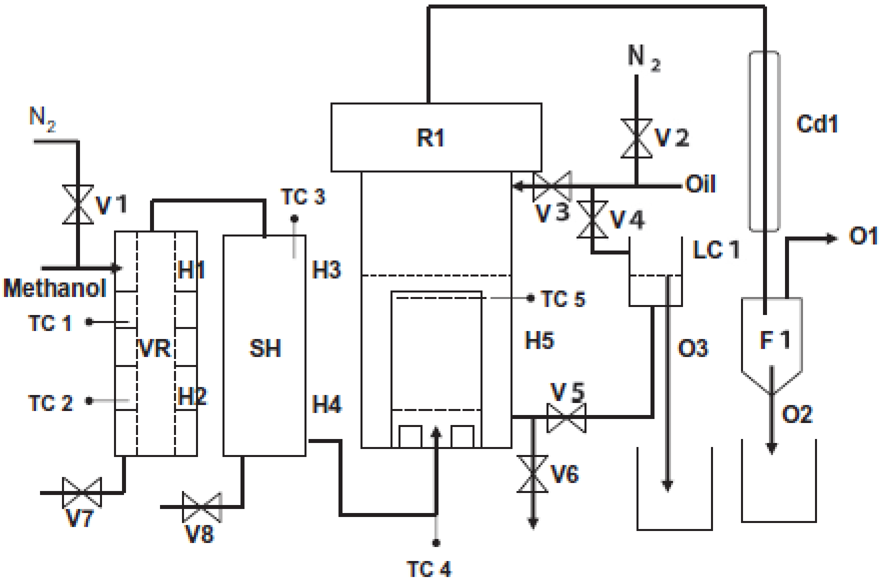

Figure 2. Schematic of Bubble Column Reactor Test Equipment. VR: vaporizer, SH: superheater, R1: bubble column reactor, LC1: level controller, Cd1: condenser, F1: glass container for sample collector, V1–V8: valves, H1–H5: electrical heaters, TC1–TC5: temperature controllers, O1: pipe on the top of F1, O2: pipe on the bottom of F1, O3: inner pipe in the LC1 (Joelianingsih et al., 2012)

### Methods

A total of 1 L of Kemiri Sunan oil was fed into the reactor and heated to a specific temperature. Nitrogen gas was fed to the reactor during the heating process to prevent oxidation. Liquid methanol was delivered at a flow rate of 2.5, 5.0, and 7.5 mL/min via a vaporizer and superheater so that the methanol turned into saturated vapor (1 atm, 240 ºC). Experiments with variations of methanol flow rate were carried out at 250 ºC, while experiments with temperature variations (250, 270, and 290 ºC) were carried out at a methanol flow rate of 2.5 mL/min. The reaction product in the vapor phase was condensed and stored every 20 minutes for 140 minutes (7 samples). Methanol in the reaction mixture was evaporated to obtain biodiesel products. Products were weighed and tested for acid number and FAME content. The production rate was calculated by measuring the products produced over a specified time, 20 minutes. The mass of FAME is then divided by the sampling time.

### Product Analysis

Analysis of acid numbers in Kemiri Sunan oil and biodiesel follows the ASTM D.664 method and according to SNI 7182: 2012 by titrimetric method employing 0.1 N KOH against Kemiri Sunan oil or biodiesel vegetable oil that has been dissolved with neutral ethanol, with the addition of a few drops of phenolphthalein. The fatty acid content of methyl esters in biodiesel was analyzed using gas Chromatography Shimadzu GC-2010 (Kyoto, Japan) with a double column according to EN 14105 using standard internal methods. This analysis aims to determine the content and composition of the amount of fatty acid methyl esters found in biodiesel.

## RESULTS AND DISCUSSION

### Effect of Methanol Flow Rate on Biodiesel Production Rate

The effect of the methanol flow rate on the biodiesel production rate is shown in Table 1. The methanol flow rate affects the amount and uniformity of bubbles formed in the BCR. The more uniform and even the bubbles, the higher the rate of biodiesel production (Kantarci et al., 2005).

Based on Table 1, the results of the most significant production rate at a flow rate of 2.5 mL/min were obtained at 120 minutes, which is 1.94 g/min. Whereas for the flow rate of 5 mL/min and 7.5 mL/min, the highest biodiesel production rate was obtained in 20 and 140 minutes, respectively, in which the value was 3.41 g/min and 2.88 g/min. Based on the average biodiesel production rate, the yield of biodiesel at a temperature of 250 °C with a methanol flow rate of 2.5

mL/min has an average biodiesel production rate of 1.43 g/min. The 5 mL/min methanol flow rate has an average biodiesel production rate of 1.48 g/min. In contrast, the 7.5 mL/min methanol flow rate has an average of 1.84 g/min biodiesel production rate.

The high biodiesel production rate at the 7.5 mL/min methanol flow rate was determined as the best flow rate. Methanol vapor efficiently made contact with Kemiri Sunan oil so that the biodiesel production process runs optimally. The number of bubbles produced was also more numerous, uniform, and evenly distributed. The contact interface with the reactants became more prolonged, forming the reaction towards the product. The higher the methanol flow rate, the more oil that comes into contact with the methanol vapor, so that more methanol vapor reacts with the oil to become biodiesel. Table 1 shows the flow rate of methanol at 250 °C.

The effect of reaction time on the rate of biodiesel production also can be shown in Table 1, which has fluctuated. The results of the rate of biodiesel production at a temperature of 250 °C with a methanol flow rate of 2.5 mL/min from 20 minutes to 140 minutes have increased and decreased regularly. However, the results of the rate of biodiesel production at temperatures of 250  C with a flow rate of methanol 5 mL/min from time to time has decreased. As for the results of the rate of biodiesel production at temperatures of 250 °C with a flow rate of 7.5 mL/min of methanol has increased and decreased irregularly.

Table 1. Effect of Methanol Flow Rate on Biodiesel Production Rate at 250 °C

| Methanol Flow Rate (mL/min) | Time (min) | Product Mass (g) | Production Rate (g/min) |
|---|---|---|---|
| 2.5 | 20 | 18.91 | 0.95 |
| | 40 | 25.30 | 1.27 |
| | 60 | 19.51 | 0.96 |
| | 80 | 31.40 | 1.57 |
| | 100 | 29.67 | 1.48 |
| | 120 | 38.81 | 1.94 |
| | 140 | 36.73 | 1.84 |
| 5.0 | 20 | 68.21 | 3.41 |
| | 40 | 27.47 | 1.37 |
| | 60 | 23.92 | 1.20 |
| | 80 | 22.66 | 1.13 |
| | 100 | 22.12 | 1.11 |
| | 120 | 22.03 | 1.10 |
| | 140 | 21.80 | 1.09 |
| 7.5 | 20 | 32.33 | 1.62 |
| | 40 | 30.23 | 1.51 |
| | 60 | 30.89 | 1.55 |
| | 80 | 30.27 | 1.51 |
| | 100 | 34.74 | 1.74 |
| | 120 | 41.23 | 2.06 |
| | 140 | 57.51 | 2.88 |

The production rate results from 20 minutes to 80 minutes have increased and decreased, and for the next time up to 140 minutes has always increased. In the first 20 minutes, oil generally has not reacted thoroughly with methanol. At a later time, oil and methanol begin to respond comprehensively. At the same time, in the following minutes, the rate of biodiesel production is lower because the oil in the reactor has decreased. This is because the operating conditions used in the reactor are semi-batch, so the longer the reaction time, the volume of oil in the reactor decreases, causing contact between the reactants to decrease, and the rate of production decreases.

### Effect of Methanol Flow Rate on Acid Number of Biodiesel Products

Simultaneous esterification and transesterification reactions at BCR for reactant flow rate variables are operated at different methanol flow rates. The reactant flow rate is one factor that influences the value of the acid number in the esterification and transesterification process of Kemiri Sunan oil. The acid number is one of the most critical parameters in determining the success of the process in the biodiesel synthesis process. The higher the acid number shows that the higher the levels of FFA and delivers the quality of the oil, which decreases. The effect of the methanol flow rate on the acid number of biodiesel products is presented in Table 2.

Table 2 reveals that the lowest acid number is obtained at a 2.5 mL/min methanol flow rate. The more homogeneous and equally dispersed amounts of methanol vapor bubbles formed. Interface contact between the vapors of methanol and the oil of Kemiri Sunan rises more often. The reaction time is more effective on the surface of the vapors of methanol (Abdulrazzaq et al., 2016). The product has obtained a lot of reactants that aim to decrease the value of the acid number (Zimmerman & Kokoo, 2018). This decrease in acid numbers suggests an effective and efficient esterification reaction in the BCR.

Based on Table 2, the smallest acid value for the flow rate of 2.5 mL/min, 5 mL/min, and 7.5 mL/min was at the reaction time of 140 minutes, with the value of 1.25, 4.16, and 4.16 mg-KOH/gram of sample, respectively. This is because of the simultaneous esterification and transesterification reactions that occur in biodiesel synthesis from time to time getting better. The impurities in Kemiri Sunan oil and organic compounds in short-chain double bonds were already carried away by the initial product with little reaction time. Thus, from time to time, the acid number produced decreases.

The flow rate of methanol vapor of 2.5 mL/min was the lowest acid number compared to 5 mL/min methanols and 7.5 mL/min. Thus the flow rate of 2.5 mL/min is the reference for the next component, namely the effect of temperatures 270 and 290°C, as the strongest methanol flow rate.

Table 2. Effect of Methanol Flow Rate on Acid Number at 250 °C

| Methanol Flow Rate (mL/min) | Time (min) | Acid Number (mg-KOH/g of sample) |
|---|---|---|
| 2.5 | 20 | 55.76 |
| | 40 | 32.65 |
| | 60 | 15.54 |
| | 80 | 5.77 |
| | 100 | 4.13 |
| | 120 | 2.36 |
| | 140 | **1.25** |
| 5.0 | 20 | 78.16 |
| | 40 | 49.99 |
| | 60 | 38.85 |
| | 80 | 25.75 |
| | 100 | 18.73 |
| | 120 | 9.55 |
| | 140 | 4.16 |
| 7.5 | 20 | 84.22 |
| | 40 | 47.42 |
| | 60 | 30.00 |
| | 80 | 18.79 |
| | 100 | 12.72 |
| | 120 | 8.95 |
| | 140 | 4.16 |

The method is more concerned with the importance of the acid number, even though biodiesel production rate also determines the best vapor flow rate of methanol (Zimmerman & Kokoo, 2018). The acid number at 2.5 mL/min, 5 mL/min, and 7.5 mL/min differed slightly, although the production rate was not significantly different. The findings of this experiment are consistent with previous research (Ahmad et al., 2019; Stacy et al., 2014; Nabetani et al., 2012).

### Effect of Reaction Temperature on the Biodiesel Production Rate

The effect of reaction temperature on the rate of biodiesel production can be seen from the comparison of the results of the production rate obtained in Table 3. The reaction temperature is vital to note. In this case, the reaction temperature affects the rate of production of biodiesel produced. Table 3 shows that the higher the temperature, the reaction will proceed quickly so that the production rate will also increase. The production rate at temperatures of 290 °C was higher than that of products with reaction temperatures of 250 °C and 270 °C. The reaction speed will increase with increasing reaction temperature. The difference in the production rate results is also obtained based on the reaction time seen in Table 3.

Table 3 shows the reaction time of the biodiesel synthesis process fluctuating. The biggest reaction rate results obtained at 120 minutes with a temperature condition of 250 ° C that is equal to 1.94 g/min, while at conditions of temperature 270 °C and 290 °C, the largest production rate was obtained at a reaction time of 120 minutes and 140 minutes respectively with a reaction rate of 3.52 g/min and 3.47 g/min. In this reaction temperature variable, there is an uncertain increase and decrease in the rate of biodiesel production. This is because, at 20 minutes, the oil generally has not reacted thoroughly with methanol. At a later time, the oil and methanol start reacting as a whole, while in the following minutes, the rate of biodiesel production is lower because the oil in the reactor has decreased.

Table 3. Effect of Reaction Temperature on Production Rate on Methanol Flow Rate 2.5 mL/min

| Reaction Temperature (°C) | Time (min) | Product Mass (g) | Production Rate (g/min) |
|---|---|---|---|
| 250 | 20 | 18.91 | 0.95 |
| | 40 | 25.30 | 1.27 |
| | 60 | 19.51 | 0.96 |
| | 80 | 31.40 | 1.57 |
| | 100 | 29.67 | 1.48 |
| | 120 | 38.81 | 1.94 |
| | 140 | 36.73 | 1.84 |
| 270 | 20 | 17.26 | 0.86 |
| | 40 | 22.10 | 1.11 |
| | 60 | 25.62 | 1.28 |
| | 80 | 35.22 | 1.76 |
| | 100 | 55.27 | 2.76 |
| | 120 | 70.36 | 3.52 |
| | 140 | 54.39 | 2.72 |
| 290 | 20 | 42.83 | 2.14 |
| | 40 | 43.64 | 2.18 |
| | 60 | 51.69 | 2.59 |
| | 80 | 56.99 | 2.85 |
| | 100 | 68.69 | 3.44 |
| | 120 | 44.70 | 2.24 |
| | 140 | 69.32 | 3.47 |

Based on the average biodiesel production rate, the yield of biodiesel at a methanol flow rate of 2.5 mL/min with a temperature of 250 °C had an average biodiesel production rate of 1.43 g/min, at a temperature of 270 °C had an average rate biodiesel production is 2.00 g/min, while at a temperature of 290 °C had an average biodiesel production rate of 2.70 g/min. Based on the results, it can be concluded that biodiesel at a reaction temperature of 290 °C is the best reaction temperature in the process of biodiesel synthesis so that the reaction runs faster and more products are produced. This is consistent with the Arrhenius equation (Yamazaki et al., 2007).

### Effect of reaction temperature on acid numbers

The reaction temperature is essential to note and is one factor that influences the esterification and transesterification reaction in the biodiesel synthesis process. In this case, the reaction temperature affects the acid number value contained in the biodiesel produced. This acid number testing needs to be done to determine the quality of Kemiri Sunan oil. The higher the acid number indicates, the worse the oil quality. The effect of reaction temperature on acid number values can be seen in Table 4.

Table 4 shows that the acid number in biodiesel produced tends to decrease with increasing reaction temperature. The smallest acid number was at the flow rate of methanol 2.5 mL/min with a reaction temperature of 250 °C, and the reaction time of 140 minutes was equal to 1.25 mg-KOH/g sample. Whereas for the reaction temperature of 270 °C and reaction temperature of 290 °C, the smallest acid number was obtained at the reaction time of 100 minutes and 80 minutes, namely 1.60 mg-KOH/g sample and 0.69 mg-KOH/g sample.

Based on Table 4, the acid number at a temperature of 290 °C is relatively lower than at temperatures of 250 °C and 270 °C, so that biodiesel produced at 290 °C is better. This shows that the reaction temperature of 290 °C is the optimum temperature for the reaction of the biodiesel synthesis process. So, if converted to FFA, it will produce a small FFA value, so that the quality of FAME levels increases.

The higher the reaction temperature caused a decrease in the value of acid numbers. This occurs because the simultaneous reaction process of esterification and transesterification at high temperatures results in better production of FAME levels. Impurities, which affect the acid number present in the oil and organic compounds in short-chain double bonds, are retained in the bottom product so that it does not carry over to the top product.

Table 4. Effect of Reaction Temperature on Acid Figures on Methanol Flow Rate 2.5 mL/min

| **Reaction Temperature (°C)** | **Time (min)** | **Acid Number (mg-KOH/g-sample)** |
|---|---|---|
| 250 | 20 | 55.76 |
| | 40 | 32.65 |
| | 60 | 15.54 |
| | 80 | 5.77 |
| | 100 | 4.13 |
| | 120 | 2.36 |
| | 140 | 1.25 |
| 270 | 20 | 85.52 |
| | 40 | 34.14 |
| | 60 | 13.01 |
| | 80 | 1.83 |
| | 100 | 1.60 |
| | 120 | 2.78 |
| | 140 | 2.49 |
| 290 | 20 | 18.22 |
| | 40 | 5.77 |
| | 60 | 2.16 |
| | 80 | 0.69 |
| | 100 | 0.70 |
| | 120 | 4.79 |
| | 140 | 2.59 |

The acid number indicates the presence of free fatty acids in biodiesel. High free fatty acids can cause corrosion and cause soot or crust in the engine injector. The acid number is also used as a parameter of oil unsaturation, or in other words, the presence of double bonds in biodiesel. Besides, acid numbers indicate the tendency of unsaturated molecules to react with oxygen in the atmosphere and turn into peroxide (Yamazaki et al., 2007).

### Effects of Methanol Flow Rate and Reaction Temperature on FAME Levels

Identification of the results of the biodiesel synthesis process with GC was carried out to prove the formation of FAME and to know the type of FAME contained in biodiesel and the levels of FAME formed. The biodiesel samples chosen to measure their FAME levels are biodiesel with good color (clear yellowish) and good acid number. Biodiesel samples used for measurement are biodiesel results on the 7th sampling or with a reaction time of 140 minutes. Data on the analysis of methyl ester levels in various variables are shown in Table 5.

Based on Table 5, it can be seen that the methanol flow rate and reaction temperature affect the levels of FAME contained in biodiesel. The results obtained show that the higher the methanol flow rate, the FAME levels will tend to be more and more. Although the results of FAME levels at the reaction temperature of 250 °C with a methanol flow rate of 2.5 mL/min towards the methanol flow rate of 5 mL/min have increased and then decreased FAME levels again at a flow rate of 7.5 mL/min, but it is not too far away. This is because, in order to encourage the reaction to take place in a perfect conversion or shift towards the product, the excess methanol must have flowed. With the higher flow rate, the more methanol flowed, so the conversion of FAME levels will be higher. The use of methanol vapor flow rate affects the amount and uniformity of bubbles formed. The more uniform and even the bubbles, the higher the level of methyl ester. Based on Table 5, it can be seen that the reaction temperature affects the FAME levels contained in biodiesel. The results obtained indicate that the higher the reaction temperature, the reaction moves to the right or the reaction is more perfect so that the FAME level produced is more and more. This is because the vapor conditions will be more easily decomposed, or the number of carbon chain bonds will be smaller when compared to liquid or solid conditions.

Table 5. Results of Methyl Esters in Various Variables

| **Variable** | **FAME Content (%-mass)** | **SNI 7182:2012 (%-mass)** |
|---|---|---|
| 250 °C; 2.5 mL/min | 60.50 | 96.50 |
| 250 °C; 5.0 mL/min | 70.80 | |
| 250 °C; 7.5 mL/min | 69.50 | |
| 270 °C; 2.5 mL/min | 68.50 | |
| 290 °C; 2.5 mL/min | 78.20 | |

Following the Arrhenius equation, the increase in reaction temperature also increases the conversion and FAME levels in oil's esterification and transesterification process. That is because the higher the temperature, the faster the molecular movement or the kinetic energy of the reagent molecules is higher so that collisions between the reacting molecules also increase. Although the results of FAME levels with a methanol flow rate of 2.5 mL/min at the reaction temperature of 250 °C towards reaction temperature of 270 °C have increased and then decreased FAME levels again when the reaction temperature is 290 °C but not too far away. So it can be concluded that the higher the reaction temperature, the FAME levels tend to increase. The results of the methyl ester fatty acid analysis in biodiesel samples on various variables are attached.

From the analysis of FAME levels of biodiesel from Kemiri Sunan oil on various variables, the chromatogram results showed that the highest fatty acid content in biodiesel produced from Kemiri Sunan oil is C18:1 compound namely oleic acid. From the difference in composition between Kemiri Sunan oil and Kemiri Sunan biodiesel that has been produced, it can be seen that the composition of methyl oleostearate in Kemiri Sunan biodiesel has decreased significantly compared with the composition in Kemiri Sunan oil. On the other hand, the composition of methyl oleate and metal palmitate in biodiesel of Kemiri Sunan significantly increased compared to that of Kemiri Sunan oil. Kemiri Sunan oil contains very high oleostearic acid. During biodiesel synthesis, the oleostearic acid underwent a cracking process at high temperatures and reacted with an alcohol. Alcohol acted as a carrier for oleic acid towards the final product, and some stearic acid also went to the final product. The nature of this stearic acid itself is easily lumpy and condensed due to low oxidation stability. Therefore, when Kemiri Sunan oil was processed into biodiesel, the oleostearate content decreased significantly while the oleate content increased.

Table 6. The Best Condition of Research Results

| Variable | Value / Information |
|---|---|
| Methanol Vapor Flow Rate | 2.5 mL/min |
| Reaction Temperature | 290 °C |
| Biodiesel Production Rate | 3.47 g/min |
| Acid Number | 0.69 mg-KOH/g sample |
| Methyl Ester Content | 78.2 % wt. |
| Organoleptic Properties | Clear yellow and not compact |

**Analysis of the Best Operating Conditions**

After biodiesel synthesis was performed for all the methanol vapor flow rate variables (2.5; 5; 7.5) mL/min and reaction temperature (250; 270; 290 °C), the best results for biodiesel synthesis were found at the flow rate methanol vapor 2.5 mL/min, and reaction temperature 290 °C so that the operating conditions for the production of biodiesel can be continued to be developed in subsequent studies using a series reactor.

The level of FAME that did not meet the requirements was due to the depreciation of the GC analysis tool. Whereas, based on SNI 7182-2012 quality requirements, the maximum allowable acid number is 0.6 mg-KOH / g-sample. The small content of methyl esters and very high acid number values are thought to be due to oxidation reactions inside the reactor, even though nitrogen has been drained before. The high acid content in Kemiri Sunan oil is caused by continuous heating at high temperatures and contact with oxygen from the outside air, which facilitates oxidation reactions. Also, the high number of acids can be caused by the presence of water content in methanol, which causes hydrolysis reactions. Kemiri Sunan biodiesel with the best synthesis conditions as presented in Table 6. This best condition still needs to be improved in order to obtain results that meet SNI standards, especially FAME levels and acid numbers. To achieve the SNI, the reactor configuration (H/D ratio) must be optimized, or the reactor can be designed with a series system.

## CONCLUSION

In a continuous-flow BCR for ME production through non-catalytic transesterification, the lower the methanol vapor flow rate, the higher the rate of biodiesel production achieved. Furthermore, as the amount of methanol vapor bubbles produced becomes more homogeneous and uniformly dispersed, the acid number had decreased. The best condition of biodiesel production using BCR is at a methanol flow rate of 2.5 mL/min with a reaction temperature of 290 ºC, the production rate was 3.47 g/min with an acid number of 0.69 mg KOH/g sample, and FAME content was 78.2% wt.

For further study, experimental work may be performed by re-designing the reactor system to achieve higher efficiency, to follow quality requirements while preserving high productivity. The BCR will use raw materials with high FFA levels to produce biodiesel with no catalyst at environmental demand in the future.

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