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Study of the Effect of NaOH Type Alkaline Catalyst on the Physicochemical Characteristics of Used Cooking Oil Biodiesel

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Abstract

Dependence on fossil fuels causes significant environmental damage and increases costs and scarcity in the future. To overcome this problem, a transition to renewable energy is needed, one of which is biodiesel which can be obtained from used cooking oil. This study aims to convert used cooking oil that cannot be reused into biodiesel products. Biodiesel synthesis can be carried out by the transesterification process, using NaOH catalyst with concentration variations of 0.5%, 0.75%, 1%, 1.25%, and 1.5% of the total mass of oil. The test parameters are calorific value, density, viscosity and flash point as well as yield on used cooking oil biodiesel products. The test results show that the use of a catalyst concentration of 1% produces more optimal density, viscosity, calorific value and flash point. Each value is 0.859 g / cm^3, 2.34 cSt, 10,356 cal / g, and 139°C. However, the use of a catalyst concentration of 0.5% shows that the biodiesel product is less than optimal. This can be shown by the highest density, viscosity and flash point values of the catalyst concentration variations used. Each value is 0.88 g/cm^3, 3.16 cSt and 178°C, while the calorific value is also low with a value of 9,689 cal/g. However, when viewed from the yield, the catalyst concentration of 0.5% produces the highest value of all catalyst concentration variations used with a value of 88%.

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1. Introduction

The world's population's dependence on technology has resulted in increasing energy needs (Zhao & Zhang, 2018). One example of dependence on energy is the use of transportation technology which still relies on fossil fuels (Levin, 2019). However, the use of petroleum fuel has negative impacts. The negative impacts include environmental damage, air pollution and unstable prices (Martins et al., 2019). To reduce the negative impacts, transitioning to alternative energy is the solution.

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Alternative energy is currently experiencing significant demand and is becoming a popular energy source for future use (Wu et al., 2016; Popp et al., 2021). Used cooking oil has the potential as a raw material for alternative energy. This is because used cooking oil is easily obtained from both small and medium industries and households. In general, used cooking oil cannot be reused to produce food because it can trigger health problems and reduce the quality of taste. In addition, if disposed of carelessly, it can cause environmental pollution. Therefore, used cooking oil is more appropriate to be used as alternative energy. One of the alternative energies that can be obtained from used cooking oil is biodiesel.

Biodiesel can be used as fuel in industrial engines, transportation engines and power generation engines. The use of biodiesel as fuel in these engines has positive impacts including reducing exhaust emissions and reducing dependence on fossil fuels (Pérez-Méndez et al., 2023; Amira et al., 2022). To produce biodiesel, a transesterification process is required. The transesterification process can be carried out if the free fatty acid content is less than two point five percent (Said et al., 2020). The transesterification process is carried out by mixing raw materials with the addition of ethanol and catalysts. The transesterification products are ethyl ester and glycerol (Supriyadi et al., 2022).

The catalyst has a role, namely lowering the activation energy during the biodiesel synthesis process. Catalysts for biodiesel synthesis can use base catalysts or acid catalysts. Alkali catalysts are more widely used for biodiesel synthesis, this is because alkali catalysts are faster during the biodiesel synthesis process (Ayetor et al., 2015). According to research by Mohamed et al (2020) which was carried out by varying the types of NaOH and KOH catalysts showed that the NaOH catalyst was more effective than KOH.

Several studies on biodiesel synthesis have been conducted by Supriyadi et al (2022). The study was conducted on Philippine tung seed oil as a raw material for biodiesel. The study was conducted by varying NaOH and reaction temperature. The results showed that the use of catalysts had an impact on the yield value, acid number, saponification number, density and viscosity. Synthesis research was also conducted by Mukminin et al (2023) by conducting research on biodiesel synthesis from used cooking oil. The research was conducted by varying the transesterification process time by 30 to 45 minutes. The results showed that the parameters that met the SNI standard were only the flash point, distillation and color parameters. Amira et al (2022) also conducted research on biodiesel synthesis from used cooking oil. The research was conducted by varying the catalyst and temperature. The ratio of catalysts used was 1:3, 1:1, 3:1. During the transesterification process, methanol was used as a residue binder. The results showed that the catalyst influenced the characteristics of the biodiesel produced. The parameters studied included yield, density and viscosity.

From the background and previous research, further research is needed on the amount of catalyst concentration for biodiesel synthesis. The catalyst used is an alkali catalyst type, namely NaOH. This

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is done as an effort to obtain information on the right amount of catalyst concentration so that it has an impact on the resulting biodiesel product.

2. Materials and Methods

This study was conducted by direct testing on used cooking oil as raw material. Raw materials were obtained from food vendors or UMKM entrepreneurs. The characteristics of used cooking oil used as raw material consist of a density of 0.92 g/cm³ and a viscosity of 36.8 cSt. Testing was carried out by varying the raw material catalyst. The catalyst used in this study was a base type of catalyst, namely NaOH. The variation used was the variation of the catalyst mass concentration. The catalyst mass was varied by 0.5%; 0.75%; 1%; 1.25% and 1.5% of the total mass of oil. The study was conducted to determine the effect of mass concentration on the density, viscosity, calorific value, and flash point values as well as the yield of biodiesel products from used cooking oil. Figure 1 shows the flow of the biodiesel production process from used cooking oil.

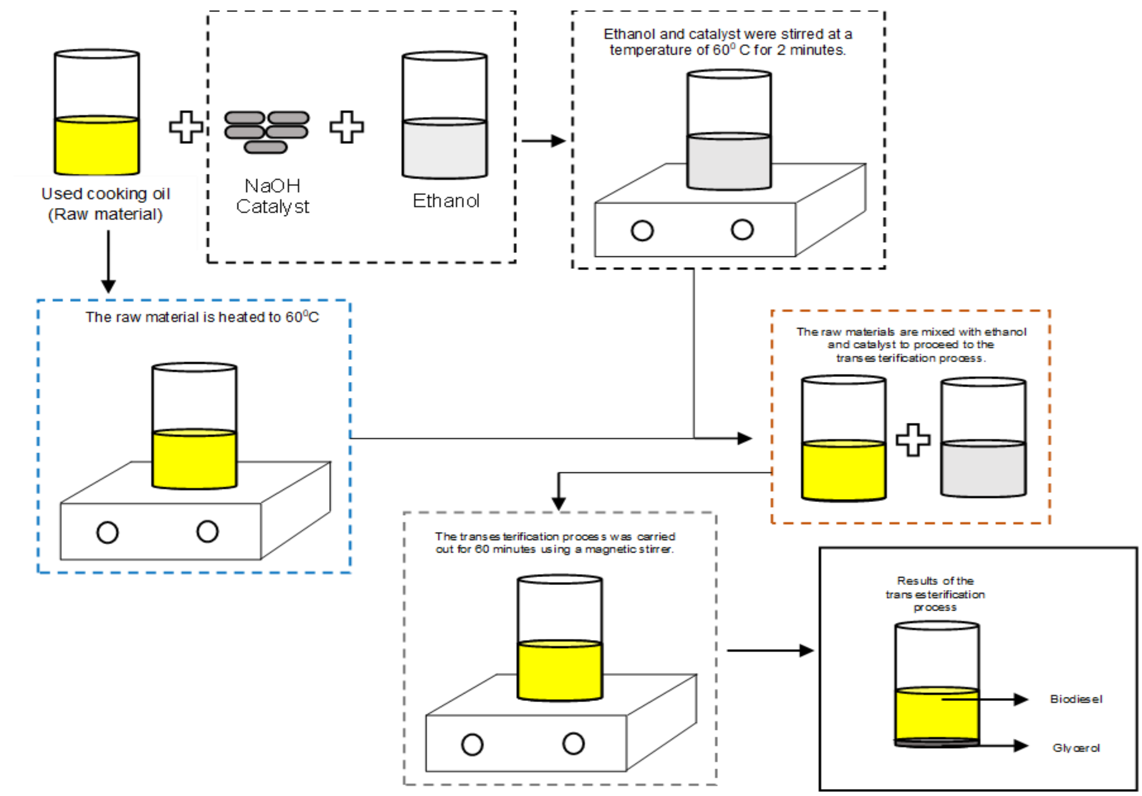

Figure 1. Biodiesel Production Process.

In Figure 1, the biodiesel production flow begins with preparing raw materials, namely used cooking oil, NaOH catalyst and ethanol. NaOH and ethanol are mixed homogeneously with the help

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of a magnetic stirrer at a temperature of 60^0C for two minutes. Used cooking oil is also heated to a temperature of 60^0C. After the ethanol and catalyst are mixed and the raw materials reach a temperature of 60^0C, the mixing process is carried out. Used cooking oil, catalyst and ethanol are mixed and the transesterification process is carried out with the help of a magnetic stirrer for the stirring process. The stirring process is carried out for sixty minutes at a temperature of 60^0C. The results of the transesterification process are biodiesel and glycerol. The next step is the purification process to clean the remaining catalyst, ethanol and water. The use of ethanol during the transesterification process is because ethanol can be obtained from vegetable raw materials through a fermentation process so that it is environmentally friendly and sustainable. The use of ethanol supports the principle of sustainability and reduces dependence on fossil resources.

2.1 Denisity Testing

Density test is a testing method carried out to determine the density level of a molecule. The molecules used during testing is spent cooking biodiesel. Density testing is carried out with a hydrometer. Hydrometers are used for density measurements. Hydrometer scale 8.00-9.00. The test is carried out by inserting a hydrometer into the test material. The hydrometer scale will show the fuel density value. Each ingredient will be tested one by one. The test results are recorded as the density value of each synthesized fuel.

2.2 Viscosity Testing

Viscosity testing was carried out using a Herzog Saybolt model ABR NL 90212. The electrical power of the tool was 5,000 watts. The test was carried out using a 50 ml fuel sample. Testing begins by inserting the sample into a tube. Then water at 40^0C is put into the heating tube. Water has a function as a heat transfer medium for the sample. A stopwatch was also prepared to calculate the time it took for the ball to go from tip to bottom in a measuring cup containing fuel. Once the materials and equipment are ready to use, the ball is put into a measuring cup containing fuel. Pay attention to the time it takes the ball from start to bottom. After completing recording the time, the recorded time is processed using equation (1) as follows.

$$V = 0.0026t - 1.175/t \quad (1)$$

Where: V: Kinematic viscosity at stoke (cSt), t: Time until the ball reaches the bottom of the tube.

2.3 Yield

Yield testing is carried out by measuring the volume produced. So, the volume of crude oil before being synthesized into biodiesel products is measured. The initial volume will be compared with the final volume after it becomes biodiesel. The equation used to calculate the yield value for each test is based on catalyst variations according to equation (2) as follows:

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$$Yield = \frac{mass\ of\ biodiesel\ produced}{mass\ of\ oil\ used} \quad (2)$$

2.4 Calorific Value

One test to determine the characteristics of fuel is the calorific value contained in the fuel. Calorific value is the thermal value contained in fuel. In this research the calorific value was tested using the Bomb Calorimeter System. The Bomb Calorimeter system used is PAAR brand with model PAAR 1241 EF. The device voltage is 220 Volts. Before use, the test equipment is calibrated using benzoic acid. The bomb calorimeter working system is used to calculate the calorific value of fuel using the combustion process. When a combustion reaction occurs, the temperature will increase. The increase in temperature is recorded to determine the calorific value of the fuel being tested.

2.5 Flash Point

Flash point is the lowest temperature value until the fuel evaporates and burns. In this research, flash point testing was carried out on all synthetic fuels by varying the catalyst during the transesterification process. The tool used to test the flash point is LEYBOLD brand. This tool has a voltage of 220 V, with a power of 420 watts. Testing on the sample is carried out by inserting 70 ml of the sample so that it fills the volume on the cup line. The cup lies in the testing medium. The stirrer is connected to the flash point cup lid. The burner is lit with a distance between the flame and the cup of 3 mm. At each temperature increase of one degree Celsius the burner is rotated above the fuel cup. Record the fuel temperature when the fuel explodes as the flash point value.

3. Results and Discussions

The test results for biodiesel synthesis from used cooking oil with varying catalyst percentages are discussed in subchapters a to e. Transesterification is carried out by adding alcohol (ethanol) with a catalyst (NaOH) to the raw material (used cooking oil) to produce ethyl ester (biodiesel) and glycerol. The transesterification process molecules can be seen in Figure 2, while the scheme for the formation of biodiesel using a NaOH base catalyst can be seen in Figure 3, while the biodiesel standards permitted according to SNI 7182:2015 can be seen in Table 1.

Table 1. Standard characteristics of biodiesel according to SNI 7182:2015.

Characteristics	SNI standards
Calorific value (cal/g)	-
Flash point (^oC)	100
Density (g/cm^3)	0,85-0,89
Viscosity (cSt)	2,3-6,0

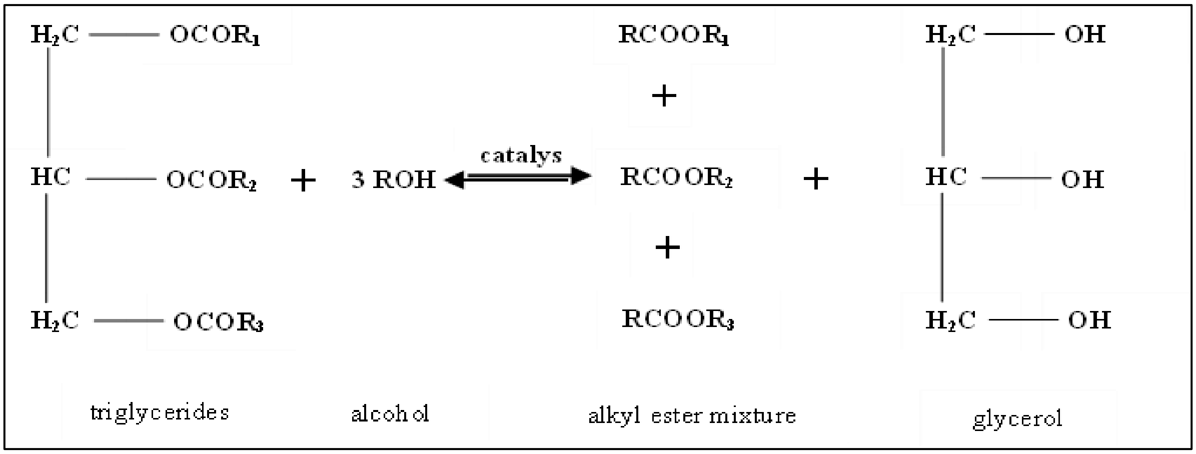

Figure 2. Transesterification process.

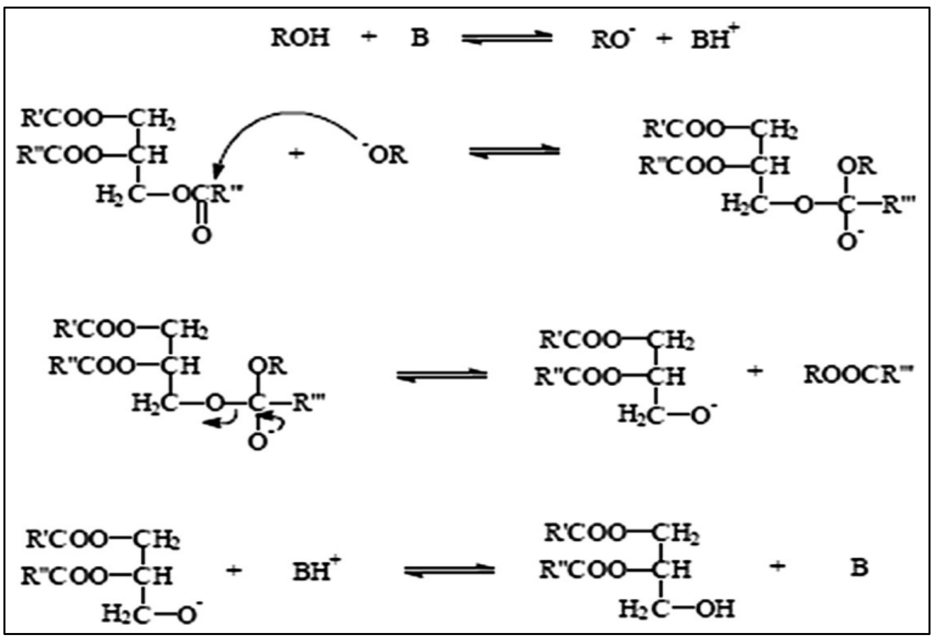

Figure 3. Transesterification Process with Base Catalyst (Mumtaz et al., 2017).

3.1 Denisity

Density is a comparison between the mass and volume of a substance (Sinaga et al., 2014). Fuel density, especially diesel, is an important parameter. This can be seen from the biodiesel standards specified in the SNI 7182:2015 standard. In addition, density that exceeds a predetermined threshold can affect engine performance. The higher the density value of biodiesel, the more difficult it is for the fuel to be injected into the combustion chamber. The results of testing the effect of catalysts on biodiesel synthesis can be seen in Figure 4 below.

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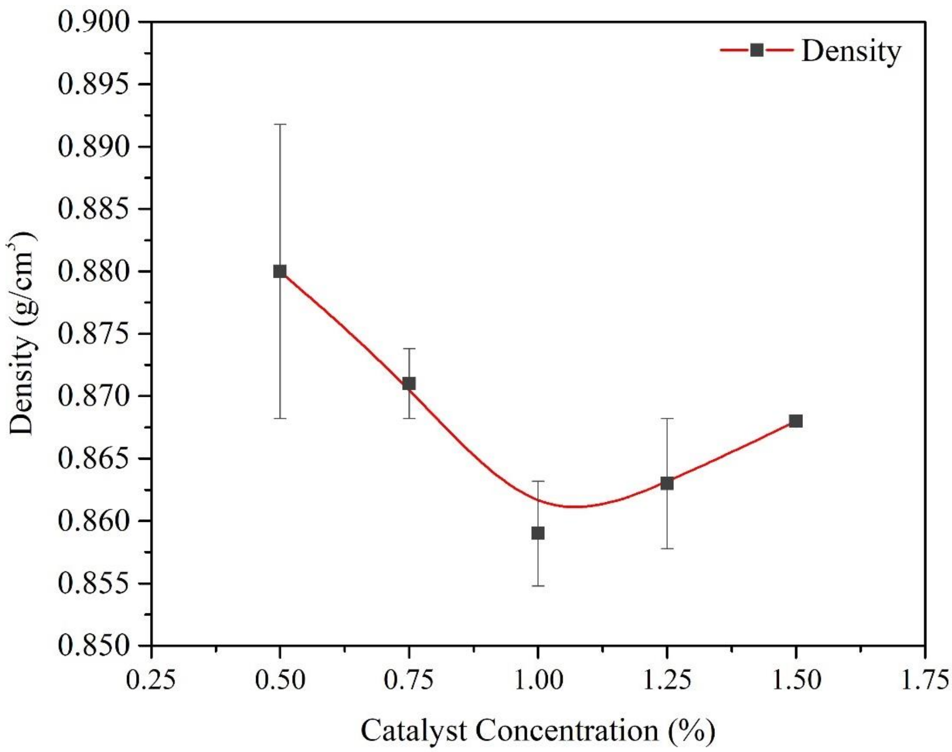

Figure 4. Fuel Density Value.

The test results showed that the density value decreased with increasing catalyst concentration. The highest density value was 0.88 g/cm³ using 0.5% catalyst. The lowest density value was 0.859 g/cm³ using 1% catalyst mass. The average value of the standard deviation of the density test results is 0.00484 g/cm3. The density of biodiesel according to SNI 7182:2015 is 0.85 g/cm3 - 0.89 g/cm³. The results of the density test on each biodiesel according to the variation of the catalyst concentration used have met the established standards. The standard deviation value of density (in g/cm³) for the variation of catalyst concentration is 0.00484 g/cm³.

Catalysts have a role in reducing barriers to kinetic reactions. Because kinetic reaction barriers are reduced, biodiesel synthesis is easier to produce. The effect of the catalyst on the density value is that the results can be observed in Figure 4. In Figure 4 it can be seen that the density value continues to decrease as the mass percentage of the catalyst increases. Catalysts have a role in increasing reaction speed. Optimal reaction speed will bind glycerol which is a residue in oil more quickly. Apart from that, a greater percentage of the mass of the catalyst, namely NaOH, increases the efficiency of the synthesis process from oil to ethyl ester.

The composition of the fuel is one of the determinants of the resulting density value. The density value is influenced by the fatty acid content. High density is more dominant in saturated fatty acids.

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Saturated fatty acids include palmitic acid and stearic acid. However, biodiesel which has a low density value tends to be composed of unsaturated fatty acids. Unsaturated fatty acids can be oleic acid. Saturated fatty acids which are residues in oil must be reduced so that the oil can be used as biodiesel. The use of a base catalyst, namely NaOH, during the transesterification process contributes to a decrease in the density value of biodiesel. The presence of a catalyst can help separate compounds in oil to be converted into by-products. The by-product will be separated with ethyl ester so it is easier to separate.

When using a catalyst with a percentage of 0.5% to 1%, the density value decreases. However, at a percentage above 1% there is an increase in the density value. Increasing the density value causes the catalyst to exceed the threshold. This causes the formation of some of the ethyl ester into soap and the residue mixed with the ethyl ester. In addition, the binding of fatty acids by ethanol is higher. The fewer free fatty acids that are bound produce a low density value. The comparison results with other studies can be observed in Table 2 as follows.

Table 2. Results Compared with Other Research on Density Values.

Raw material	Catalyst Concentration (%)					
	0,25	0,5	0,75	1	1,25	1,5
	Density Value (g/cm^3)					
Used cooking oil (*)	-	0,88	0,871	0,859	0,863	0,869
Philippine Tung oil(**)	0,889	0,876	0,881	0,875	-	-
Coconut oil (***)	0,864	0,862	-	0,860	-	-
Crude palm oil (****)	-	0,885	0,880	0,875	0,874	0,873

Source: Research result (*), (Supriyadi et al., 2022) (**), (Prayanto et al., 2016) (***), (Yunsari et al., 2019) (****)

3.2 Viscosity

Biodiesel viscosity in Indonesia must meet specified standards. The standard value for biodiesel viscosity in Indonesia is determined in SNI 7182:2015. The permissible viscosity value is 2 to 6 cSt. The results of research on biodiesel synthesis from used cooking oil with varying catalyst percentages can be seen in Figure 5 as follows.

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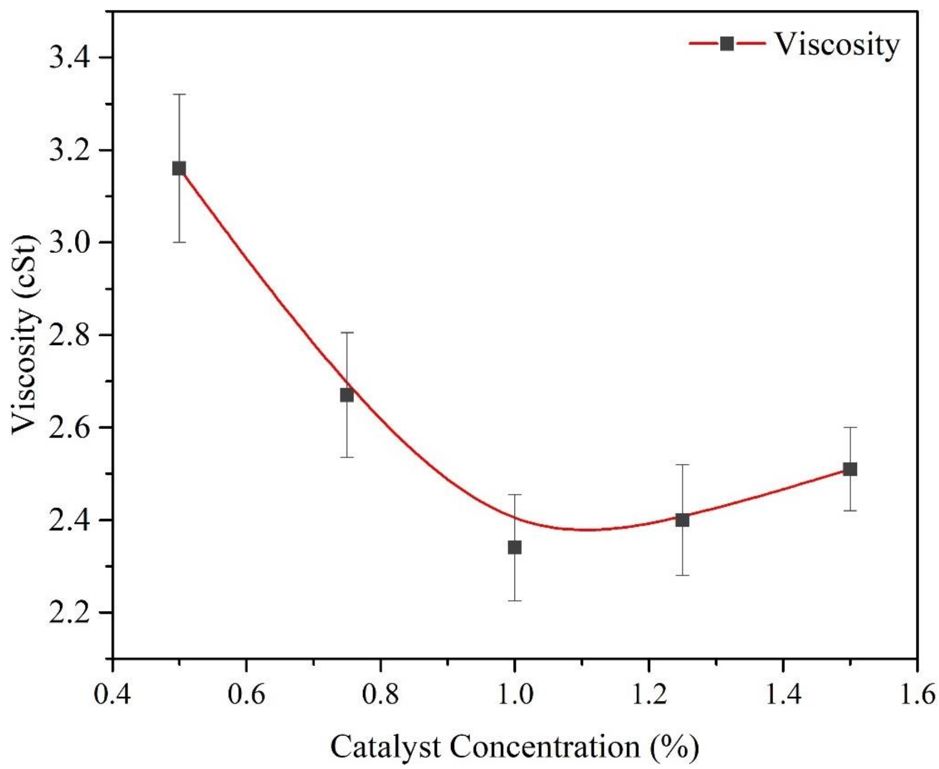

Figure 5. Relationship between Catalyst Percentage and Viscosity Value.

If we look at Figure 5, there is no significant difference in value. The highest viscosity value from the test results is found in the use of a catalyst with a percentage of 0.5% of 3.16 cSt. However, the lowest viscosity value is found when using a 1% catalyst with a value of 2.34 cSt. The viscosity value continues to decrease as the percentage of catalyst increases. This can be seen in Figure 5 which forms a decreasing graph. The average standard deviation value for viscosity is 0.124 cSt. There is a relationship between the percentage of catalyst and the viscosity value. The decrease in the viscosity value of biodiesel fuel can be observed from the effect of the catalyst percentage. The higher the catalyst value, the lower the viscosity value of biodiesel. The decrease in viscosity is influenced by the chemical structure of the raw material, namely used cooking oil. The fat content in the oil is converted into ethyl esters which are molecularly lighter. When used cooking oil is converted into ethyl esters, the product tends to have a low viscosity value. Therefore, the viscosity value decreases with increasing catalyst percentage.

A low viscosity value makes the fuel flow more easily. Low viscosity values can also improve engine performance. This is because the fuel is more easily atomized in the combustion chamber even though the surrounding air is at a low temperature. In addition, the greater the percentage of NaOH causes the higher the percentage of conversion rate of fat to ethyl ester.

3.3 Yield

The yield of research results on NaOH catalyst variations on biodiesel yield can be seen in Figure 6.

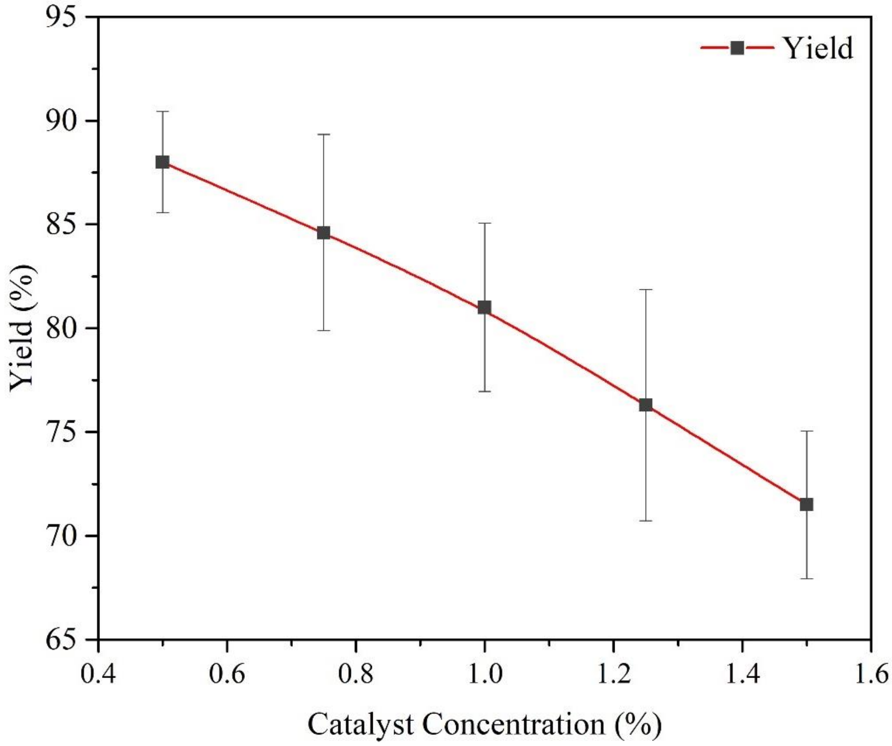

Figure 6. Effect of Catalyst Percentage on Yield.

In Figure 6, it can be observed that the higher the percentage of catalyst, the lower the yield. The use of catalyst percentage needs to be studied. This is done so that the right concentration can be determined to be used in the transesterification process and can be considered. The highest yield value was produced at a catalyst percentage of 0.5% of 88%, while the lowest yield value was at a catalyst percentage of 1.5% of 71.5%. The average value of standard deviation in this study is 4.068%. This study shows that the catalyst concentration affects the conversion of oil to ethyl ester.

The highest biodiesel yield was produced at a catalyst concentration of 0.5%, then the yield decreased along with the addition of catalyst concentration. The decrease was caused by the increasing saponification rate, so that the volume of soap and by-products became high. This caused the yield of ethyl ester to decrease. The decrease in yield value caused the formation of soap and inhibited the conversion of triglycerides. High NaOH concentrations not only reduced biodiesel products but also produced unwanted by-products (Efavi et al., 2018).

A catalyst percentage above 0.5% produces a lower yield when compared to the use of other catalysts. During the transesterification process NaOH reacts with hydroxide ions or OH-. NaOH and

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OH- have a role in replacing ester groups with alkyl groups found in ethanol. The conversion or replacement of the ester group with an alkyl group causes a change from fatty acid to ethyl ester. However, an inappropriate amount of NaOH catalyst can cause the reaction to be less than optimal. The molecular reaction scheme during saponification can be observed in Figure 7.

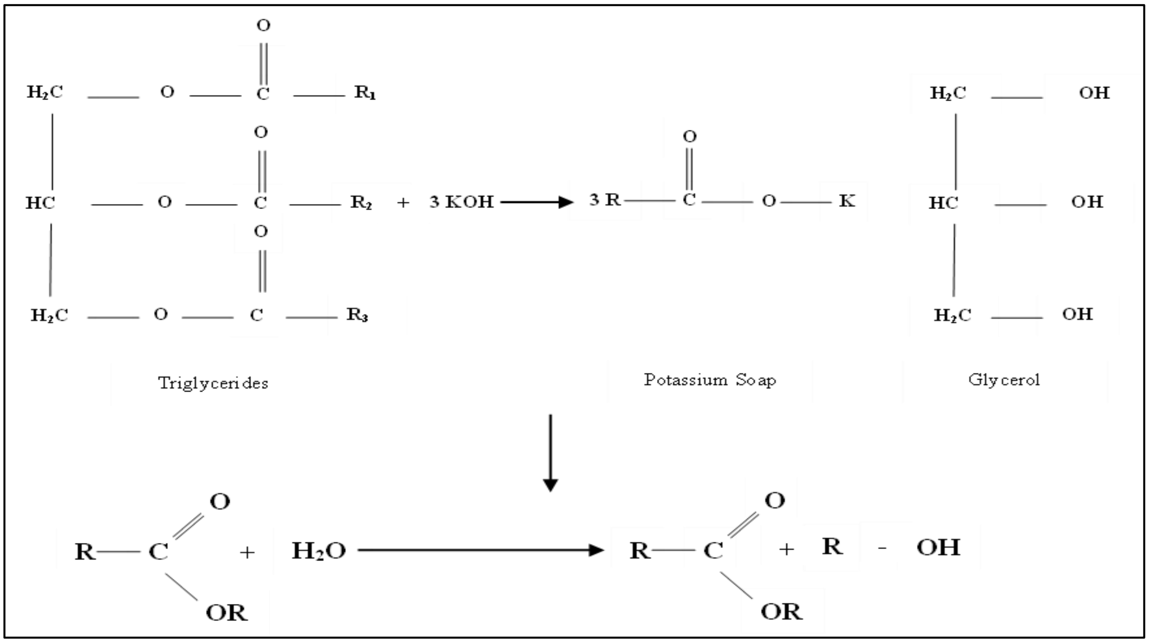

Figure 7. Saponification reaction.

Figure 7 illustrates the saponification reaction scheme. Saponification reaction, also known as soap formation reaction. During the washing process, soap emulsifies the product and mixture, making separation difficult. In addition, water can react with ethyl ester to form fatty acids, which reduces the yield and triggers further saponification of ethyl ester. The results of similar studies on biodiesel synthesis with various concentrations can be seen in Table 3.

Table 3. Results Compared with Other Research on Yield.

Raw material	Yield (%)				
	0,5	0,75	1	1,25	1,5
Used cooking oil (*)	84,50	89,70	91,54	81,30	78,20
Used cooking oil (**)	-	-	87,30	87	86,60
Used cooking oil (***)	-	82,50	85	-	73
Soybean oil (****)	64,36	-	74	-	66

Source: Research result (*), (Prihanto & Irawan, 2018)(**), (Suherman et al., 2022) (***), (Oko et al., 2021) (****)

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3.4 Caloric Value

Research into the effect of the number of catalysts in the synthesis of used cooking oil biodiesel on the calorific value needs to be carried out. This is due to the need for the right composition to use a catalyst to produce optimal heating value. The calorific value produced in this research can be observed in Figure 8.

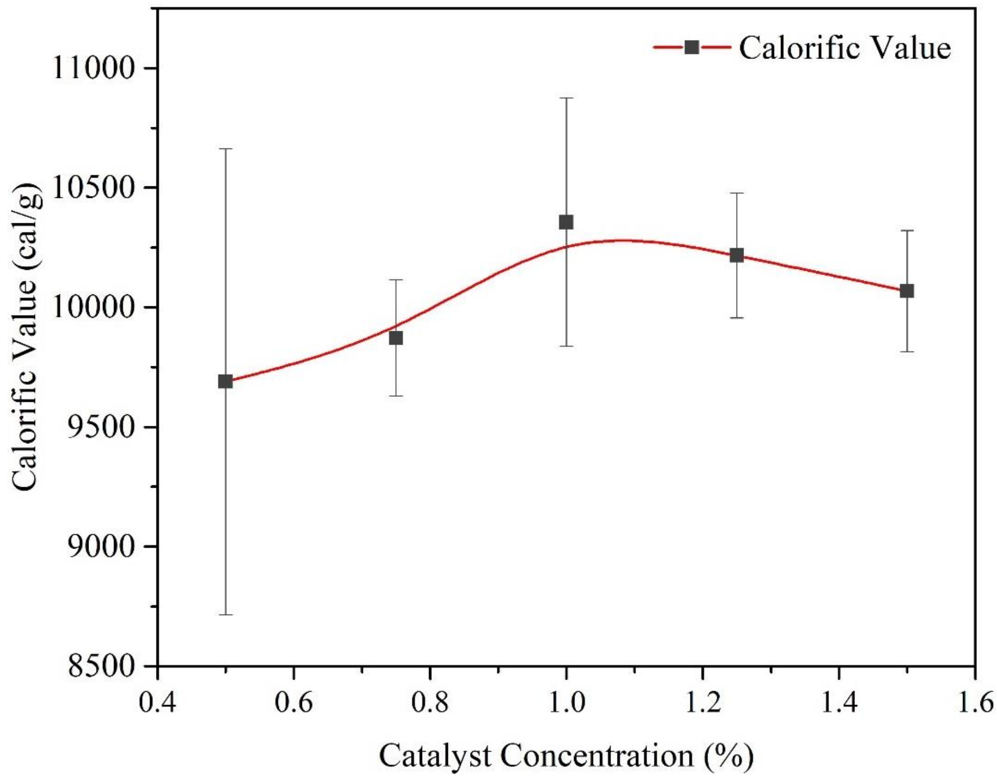

Figure 8. Relationship between Catalyst Percentage and Calorific Value.

In Figure 8 the heating value appears to increase with increasing catalyst percentage. The increase in heating value can be observed at a percentage of 1%. However, the calorific value above seems to be slowly decreasing. The lowest heating value was 10,068 cal/g at a catalyst percentage of 0.5%, while the highest heating value was 10,356 at a catalyst percentage of 1%. The standard deviation value of the calorific value of used cooking oil biodiesel synthesis is 449.97 cal/g.

The use of a catalyst appears to influence the calorific value produced in this research. As explained in the research method, the catalyst percentage lies in the transesterification process. The catalyst used is a base catalyst (NaOH). The higher the catalyst percentage appears to be effective as shown in the calorific value produced. Even though the catalyst percentage is above 1%, there appears to be a decrease. The increase in heating value is due to the large amount of oil being converted into

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ethyl ester. The more ethyl esters produced, the residue contained in the oil before it is synthesized is trapped in the ethanol and combined in the residue collection. Excessive catalyst causes the washing process to take longer. The longer the washing process causes compounds that have or produce high calorific value to be lost.

Using an inappropriate catalyst also affects the heating value. The word "low" indicates that the calorific value produced by the oil is also low. This is caused by by-products still remaining in the biodiesel produced. However, too much catalyst also affects the heating value. This is because the catalyst still remains in the biodiesel. NaOH as a catalyst left in biodiesel contains residues such as heavy metals or compounds that can reduce the heating value. One compound that can affect the calorific value is alkaline salt. Alkaline salts are compounds that have a low calorific value. Therefore, the calorific value produced is also low.

3.5 Flash Point

Flash point is the value of the fire when it first ignites with the lowest fuel temperature. The flash point value in this study can be observed in Figure 9.

In Figure 9 you can see the flash point value produced in biodiesel synthesis by varying the catalyst used. The highest flash point value is found in a catalyst composition of 1.5% at a temperature of 1490C. However, the lowest flashpoint value is located at 1% catalyst composition with a temperature of 1390C. The flash point value of a fuel is an important characteristic. The higher the flash point value, the more difficult it is for the fuel to burn. The permitted flash point value according to SNI 7182:2015 is a minimum of 1000C. In this research, the synthesis of biodiesel with various catalysts has met the permitted standards. The average value of the standard deviation of the flash point (in °C) for the given catalyst concentration variations is about 7.125 °C.

The effect of the percentage of catalyst used turns out to have varying values. The diversity of the resulting values can be seen in Figure 9. It can be seen that by using a catalyst above 1%, the flashpoint value shows an increase in flow. The flash point values using a catalyst (1.25; 1.5)% are 142^0C and 1490C respectively. The use of a catalyst provides an illustration of the resulting flash point value. It seems that the use of too little or too much catalyst affects the resulting flash point value. The higher the percentage of catalyst has an influence on the compounds that contribute to the flash point value.

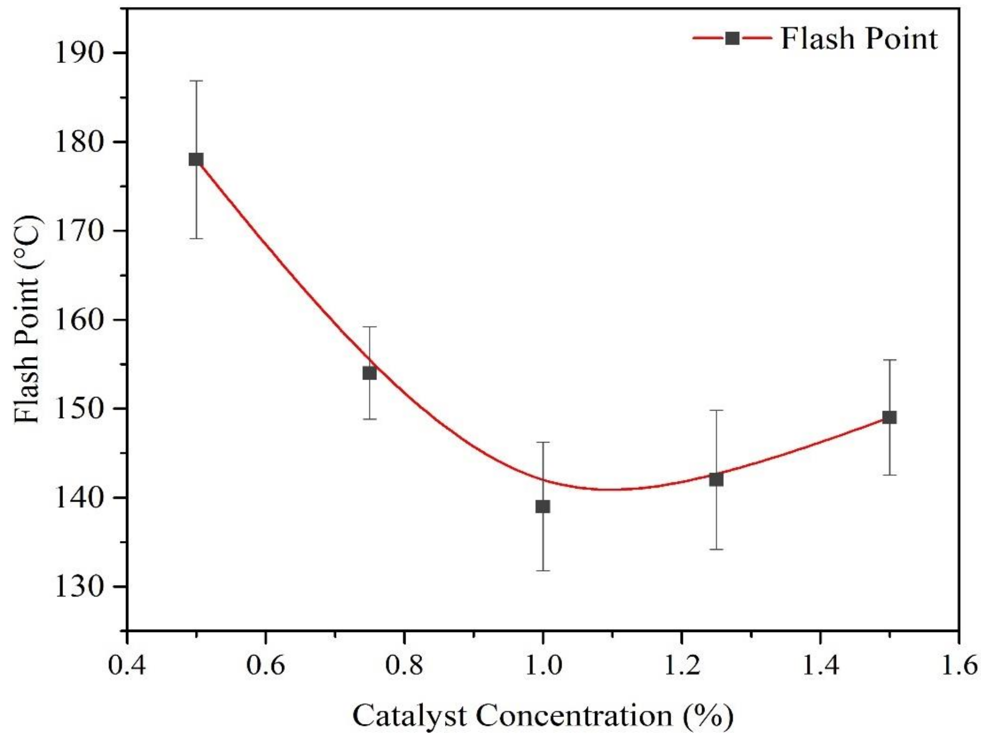

Figure 9. Flash Point Value Against Catalyst Percentage.

4. Conclusion

The conclusion of the research results is that the highest density value is found at a catalyst percentage of 0.5% with a value of 0.88 g/cm^3. However, the increasing number of catalysts, the density value decreases. The optimal density value is at a percentage of 1% with a density value of 0.859 g/cm^3. The highest viscosity is found at a catalyst percentage of 0.5% with a value of 3.16 cSt, while the lowest density is found at a catalyst percentage of 2.34 cSt. The optimal density value decreases at a percentage of 1% but increases at percentages of 1.25% and 1.5%. The highest yield value is produced using a catalyst of 0.5% with a yield value of 88%, while the lowest yield is obtained using a catalyst concentration of 1.5% with a yield value of 71.5%. For the highest calorific value, the research results are 10.356 cal/g using a catalyst concentration of 1%. The highest flash point is 178^{0}C using a catalyst concentration of 0.5%. The concentration of NaOH catalyst in the transesterification process affects the calorific value, density, viscosity, flash point and yield. A catalyst concentration of one percent produces the most optimal parameter values in this study. These parameters include density, viscosity, calorific value and flash point.

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