

Transesterification Process of Biodiesel with Potassium Glycerolate Catalyst

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Abstract

The synthesis of potassium glycerolate catalyst from DES (Deep Eutectic Solvent – K_2CO_3 -glycerol) catalyst was carried out by heating process with various temperatures. The resulting catalyst product was analyzed and the characteristics of the best potassium glycerolate catalyst had conductivity 4482 $\mu S/cm$, density 1.4858 g/cm^3 , Viscosity 121.574 cP, Freezing Point $-8^\circ C$ and pH 14. The best temperature in the manufacture of potassium glycerolate catalyst was at $150^\circ C$. Alkaline pH is the main requirement to be a catalyst in the transesterification reaction. The trial results for the conversion of RBDPO to biodiesel with the optimum weight percent ratio of potassium glycerolate catalyst to RBDPO were 3.5%w, methanol 30%w, reaction temperature $65^\circ C$, reaction time 4 hours resulted in total glycerol content in biodiesel 0.2285%, acid value 0.15%, density 0.8705 gr/cm^3 , viscosity 5.22 Cst, conversion 96.77% and 97.81% methyl ester content and all of these parameters all meet the biodiesel standards set by SNI-7182: 2015.

Keywords: Potassium Glycerolate, Transesterification, Biodiesel

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Abstrak

Sintesis katalis kalium gliserolat dari katalis DES (Deep Eutectic Solvent – K_2CO_3 -glycerol) telah dilakukan dengan proses pemanasan dengan variasi suhu. Produk katalis yang dihasilkan dianalisa dan karakteristik katalis kalium gliserolat yang terbaik memiliki konduktivitas 4482 $\mu S/cm$, densitas 1.4858 g/cm^3 , viskositas 121.574 cP, Freezing Point $-8^\circ C$ dan pH 14. Suhu terbaik dalam pembuatan katalis kalium gliserolat adalah pada $150^\circ C$. pH alkali menjadi syarat utama untuk menjadi katalis pada reaksi transesterifikasi. Hasil uji coba konversi RBDPO menjadi biodiesel dengan perbandingan persen berat optimum katalis kalium gliserolat terhadap RBDPO adalah 3,5%w, methanol 30%w, suhu reaksi $65^\circ C$, waktu reaksi 4 jam menghasilkan kandungan total gliserol dalam biodiesel 0,2285%, angka asam 0,15%, densitas 0,8705 gr/cm^3 , viskositas 5,223 Cst, konversi 96,77% dan kandungan Metil ester 97,81% dan semua parameter tersebut semua memenuhi standar biodiesel yang telah ditetapkan SNI-7182:2015.

Kata Kunci : Kalium Gliserolat, Transesterifikasi, Biodiesel

INTRODUCTION

Generally, biodiesel can be obtained by reacting triglycerides in oil with short chain alcohols to form ester compounds with the help of a catalyst. The

catalyst that is commonly used in biodiesel production today is the Potassium Methylate (MeOK) catalyst because it has a high catalytic ability [1]. The most important content contained in oil or fat that will be

used in making biodiesel is the triglyceride content. Triglycerides are known to be the main constituent compounds of vegetable oils and animal fats with a composition of up to 98% in vegetable oils [2].

However, this type of catalyst has the disadvantage that the catalyst is difficult to separate from the product mixture and will eventually be wasted as waste which can pollute the environment. For this reason, an alternative catalyst that is environmentally friendly with capabilities similar to conventional catalysts is needed, one of which is the glycerolate catalyst.

Several studies regarding the production of glycerolate catalysts have been carried out. In the previous study [3], potassium glyceroxide was prepared by reacting KOH and glycerol solutions by varying the composition of the KOH solution: Glycerol, temperature, and vacuum pressure which gave the result that the potassium glyceroxide catalyst produced succeeded in increasing the yield of biodiesel to a similar value (74.3 – 78.9%) with conventional biodiesel catalyst, potassium methoxylate (77.9%).

Glycerolate or potassium glyceroxide or potassium glyceroxide is an anhydrous metal alkoxide compound with a white color in its solid form. Potassium glycerokides are generally obtained by adding a compound with potassium (KOH) to glycerol at high temperature and vacuum conditions accompanied by stirring for a certain period of time [4]. Glycerolate in this study was produced by utilizing DES (Deep Eutectic Solvent – K_2CO_3 -glycerol) so it also has low toxicity properties and can be recycled so it is friendly to the environment [5].

Glycerolate also has the ability to catalyze chemical reactions such as transesterification. Glyceroxide can produce similar results (74.3–78.9%) with conventional catalysts, Potassium methoxylate (77.9%) [3]. Four types of catalysts including KOH as a homogeneous alkali, CaO as a heterogeneous alkali, H_2SO_4 as a homogeneous acid, and lipase as a biocatalyst was evaluated as biodiesel catalyst [6]. Glycerol is also known as the main component in the preparation of fats and oils in the form of glycerides, both monoglycerides, diglycerides and triglycerides. Glycerol can be obtained in various ways, such as transesterification reactions as a side product in low purity [7]. Calcium-based glyceroxide was applied as a catalyst in the transesterification process with vegetable oil as raw material [8]. The glyceroxide catalyst was prepared by adding a mixture of CaO and glycerol to methanol under reflux conditions under atmospheric pressure for 2 hours and then dried at 353 K for analysis. The performance of the catalyst was

tested by adding the catalyst to the transesterification reaction at a temperature of 298K at atmospheric pressure accompanied by stirring at 500 rpm. Research shows that the addition of a catalyst increases FAME conversion by more than 95%. Another research using glyceroxide as a catalyst in the transesterification reaction was carried out by [9] applying lithium glyceroxide which was made by adding lithium to glycerol in a ratio of (1:1, 2, 3) accompanied by heating at 120-140 °C accompanied by stirring. The transesterification process is carried out using canola oil as raw material at a temperature of 60 °C. In addition, during transesterification sampling is carried out at predetermined time intervals to see the performance of the catalyst. Research shows that the application of a catalyst can provide a 99% conversion for each catalyst variation in 1.5 hours of reaction

The reaction for each catalyst was optimized using response surface statistical methodology, taking into account the main parameters: substrate molar ratio, catalyst percentage and reaction time, while for enzymatic catalysis the main parameter was adjusted to be the amount of water content. The final result stated that a homogeneous alkaline catalyst gave better parameters than other catalysts. However, the use of biocatalysts provides cleaner products with lower energy use compared to other catalysts.

The potassium glycerolate catalyst in this study is a compound derived from Deep Eutectic Solvent (DES) glycerol- K_2CO_3 which undergoes heating at high temperatures accompanied by stirring for a certain period of time. DES is a solvent consisting of two components (quaternary ammonium salt and hydrogen bond donor) which are mixed in the right ratio so that the eutectic point is reached [6]. In addition, after the reaction is complete, K_2CO_3 can be separated from glycerol and used as organic fertilizer. So it does not cause waste piles and is safe for the environment.

The research focuses on the characteristics of the glycerolate catalyst with variations in production temperature. In addition, another focus is the characteristics of biodiesel using potassium glycerolate as a catalyst in the transesterification reaction. The purpose of this study was to study the effect of reaction temperature on the characteristics of the resulting potassium glycerolate catalyst and to study the effect of using potassium glycerolate catalyst on the characteristics of the biodiesel produced.

MATERIALS AND METHODS

Materials

The materials used in this study were RBDPO waste, methanol, DES catalyst and tools used,

analytical balance, magnetic stirrer, thermometer, condenser, separating funnel, beaker glass, measuring cup, Erlenmeyer, three neck flask, hot plate.

Method

The DES K_2CO_3 -glycerol catalyst [10] was put into an Erlenmeyer with a certain mass and then heated at 110, 130 and 150 °C with a certain stirring speed to get a potassium glycerolate catalyst. Analysis of biodiesel characteristics was following the FBI method. The acid value was determined using FBI-A01-03, total glycerol using FBI-A02-03, and the methyl ester content using FBI-A03-03. The density and viscosity was measured at 40 °C.

RESULTS AND DISCUSSION

The potassium glycerolate catalyst is a homogeneous catalyst which is a catalyst that has the same phase as the reactants so that it will mix with the reactants when the reaction occurs [11]. The potassium glycerolate catalyst in this study was prepared at 110, 130 and 150 °C at a certain speed for 5 hours. A photo of the potassium glycerolate catalyst is shown in **Figure 1**.

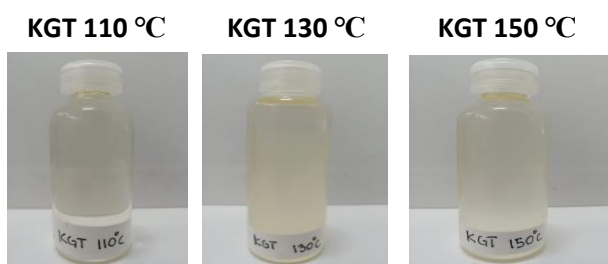


Figure 1. Physical Photo of Potassium Glycerolate Catalyst

As shown in **Figure 1**. The resulting glycerolate color tends to be cloudy white to clear which has a viscous nature. The KGT110 °C catalyst is clearer in color than the other catalyst samples. During the KGT130°C catalyst process, the color of the catalyst becomes more turbid and slightly yellowish. Furthermore, the KGT150 °C potassium glycerolate catalyst has a slightly yellowish color and tends to be clear, but not as clear as the KGT110 °C catalyst. The color of the resulting potassium glycerolate catalyst tends to be slightly yellowish in color, almost close to clear which has a viscous nature.

In this research, technical K_2CO_3 and technical glycerol were used to prepare potassium glycerolate catalyst which contains most of potassium glyceroxide. According to research conducted by Pradhan et al (2016), that potassium glyceroxide catalyst can be used for transesterification reactions of methyl esters from

fatty acids. It is known that the rate of the transesterification reaction using this catalyst is higher than that of other basic catalysts and the optimal conditions for the manufacture of potassium glyceroxide catalysts are at a temperature of 130 °C and a pressure of 113 mbar with a free water loss percentage of 84.3% [12].

Characteristics of Glycerolate Catalyst

The characteristics of the glycerolate catalyst can be seen from the conductivity, density, viscosity, freezing point and pH values. The higher the temperature of the catalyst, the higher the conductivity, but at KGT 150 °C there was a slight decrease which was not too significant. This catalyst has a pH value of 14, which indicates that it is alkaline so that it can be used as a catalyst in the transesterification reaction. The viscosity of the catalyst shows that the higher the temperature, the viscosity will also increase. This is because more and more impurities evaporate, both from glycerol and which causes the viscosity to increase. The characteristics of the potassium glycerolate catalyst are shown in **Table 1**.

Based on the data in Table 1. It is known that the highest conductivity value was obtained by the 130 °C KGT sample, amounting to 5131 $\mu S/cm$ and the lowest conductivity was obtained by the 150 °C KGT sample, which was 4482 $\mu S/cm$. The lowest density value was obtained by the 110 °C KGT sample of 1.4602 g/cm^3 and the highest was 1.4858 g/cm^3 by the 150 °C KGT sample. Density is a measure of the density of a compound. The higher the density value, the higher the density of an object, the greater the mass of each volume. The density value affects the viscosity value, when the density value is low, the viscosity value is also low, and vice versa. The highest viscosity was obtained by the same sample as the highest density value, namely KGT 150 °C of 121.57 cP. Viscosity shows a measure of the thickness of a fluid.

Parameter next is the freezing point value of the resulting potassium glycerolate catalyst. Freezing point is the freezing point of a liquid which indicates a situation where there is no movement or change of the compound. It is known that the highest freezing point value is the 150 °C KGT sample at -8 °C and the lowest freezing point value is -10 °C by 110 °C KGT sample. The freezing points of the resulting potassium glycerolate were all below the freezing points of glycerol and potassium carbonate (K_2CO_3), indicating that this catalyst was not a solution of potassium carbonate (K_2CO_3) in glycerol but had changed into other compounds, namely potassium glyceroxide and/or glycerol carbonate.

Table 1. Characteristic of Potassium Glycerolate Catalyst

Test Parameters	KGT 110°C	KGT 130°C	KGT 150°C
Conductivity ($\mu\text{S}/\text{cm}$)	4564	5131	4482
Density at 25°C (g/cm^3)	1.4602	1.4604	1.4858
Viscosity at 25°C (cP)	26,844	111,376	121,574
Freezing Point (°C)	- 10	- 9	- 8
pH	13.91	14.00	14.00

The last parameter to be measured is the degree of acidity or pH. Two (2) out of three (3) samples were known to have a pH value of 14 and the rest 13.91. This value indicates that all samples are strong bases which are good catalysts for transesterification reactions in biodiesel production.

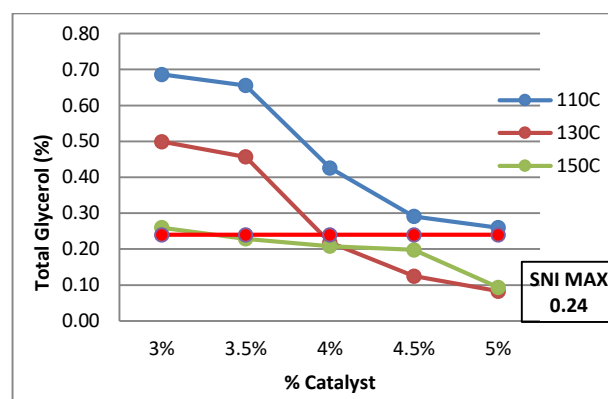
Performance test of Potassium Glycerolate Catalyst in biodiesel transesterification process

The potassium glycerolate catalyst that has been made can be said to be successful as a catalyst for biodiesel production if the biodiesel produced meets the standards set by SNI-7182:2015. The process of making biodiesel in this study used a one-step transesterification reaction for 4 hours. Transesterification is a reaction process of triglycerides, lipids, fats, with short chain alcohols using the help of an alkaline catalyst [10]. Based on the characteristic test of glycerolate catalyst as presented in **Figure 2** to **Figure 7**.

The Effect of Type and Amount of Catalyst on Total Biodiesel Glycerol Value

In this research, three types of potassium glycerolate catalysts were studied which were made at 110, 130, and 150 °C with variations in the percentage of catalyst, respectively 3%, 3.5%, 4%, 4.5%, and 5%. The performance of the potassium glycerolate catalyst can be seen from several test parameters, one of which is total glycerol. Total glycerol shows the amount of remaining monoglyceride and diglyceride compounds, as well as triglycerides that are not converted to biodiesel. The total glycerol value is one of the parameters in determining the quality of biodiesel. The maximum total glycerol value of biodiesel according to the standardization of SNI 7182-2015 is 0.24% by mass. The effect of the type and amount of catalyst on

the total glycerol content in biodiesel can be seen in **Figure 2**. All samples made using potassium glycerolate catalyst samples at 110 °C did not meet the specified total glycerol value standard. This could happen because the catalyst used is not optimal to convert triglycerides into methyl esters. Potassium glycerolate catalyst sample at 110 °C as much as 5% potassium glycerolate content is low so that the total unreacted glycerol is still above 0.24%. Samples prepared using a potassium glycerolate catalyst at 130 °C began to meet the standard total glycerol value when the addition of catalyst was greater than or equal to 4%, namely 0.2185%. As for the samples using potassium glycerolate catalyst at 150°C. Based on the figure, it is known that samples made using potassium glycerolate catalyst at 150 °C KGT50 K3 with 3% catalyst of 0.2601% did not meet the specified total glycerol value standard. This could happen because the catalyst used is not optimal to convert triglycerides into methyl esters.

**Figure 2.** The Effect of Type and Amount of Catalyst on Total Glycerol

Samples using a potassium glycerolate catalyst at 150 °C KGT50 K3.5 began to meet the standard total glycerol value when the catalyst was added as much as 3.5%. Based on SNI standards, the total value of glycerol produced by potassium glycerolate catalyst at 150 °C KGT50 K3.5 with a catalyst amount of 3.5% is 0.2285%.

Effect of Type and Amount of Catalyst on Biodiesel Acid Value

The acid value is one of the parameters used to determine the quality of the biodiesel produced. The acid value represents the corrosive potential of biodiesel, which can reduce the life of the biodiesel storage tank [13]. Determination of the acid value in biodiesel is used to see the content of acid groups in the resulting biodiesel compound. The acid value shows

the amount of free fatty acids contained in biodiesel [1]. The acid value content of biodiesel can be seen in **Figure 3**.

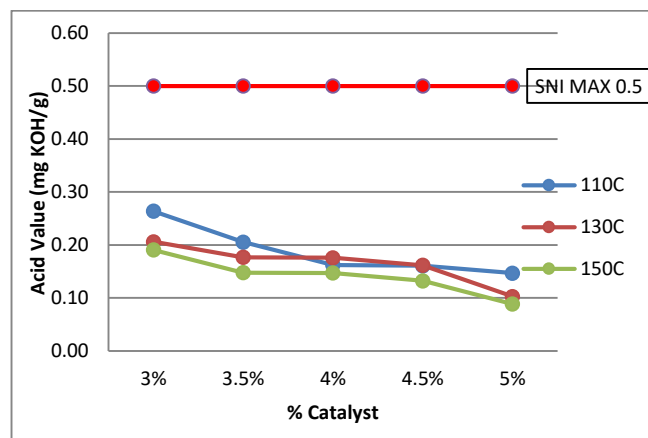


Figure 3. Effect of Type and Amount of Catalyst on Biodiesel Acid value

Based on **Figure 3**, it is known that the acid value values of all biodiesel samples meet the standards set by SNI-7182: 2015, which are below 0.5 Mg-KOH/g biodiesel. According to [10] the best acid value value is the one with the lowest acid value. Obtained the lowest acid value value of 0.09 mg KOH/g by using a glycerolate catalyst at 150 °C KGT50 K5 with 5% catalyst. The sample with the highest acid value value was the sample with potassium glycerolate catalyst at 110 °C KGT10 K3 as much as 3% catalyst which was 0.26 mg KOH/g. This shows that the greater the amount of catalyst used, the lower the value of the acid value in the biodiesel produced. Fatty acids react with bases to form soap. So, increasing catalysts would increase conversion.

Effect of Type and Amount of Catalyst on Biodiesel Density

The density of the biodiesel produced can be affected by the use of a catalyst in the transesterification reaction. The density of biodiesel will affect the process of fuel consumption. The higher the density value, the higher the fuel consumption. If the density of biodiesel meets the SNI-7182: 2015 standard, then its use can result in complete combustion, conversely if the density of biodiesel exceeds the SNI-7182: 2015 standard it will cause incomplete combustion reactions, which can cause emissions. and wear on the engine [15]. The effect of the type and amount of catalyst on the density of the biodiesel produced can be seen in **Figure 4**.

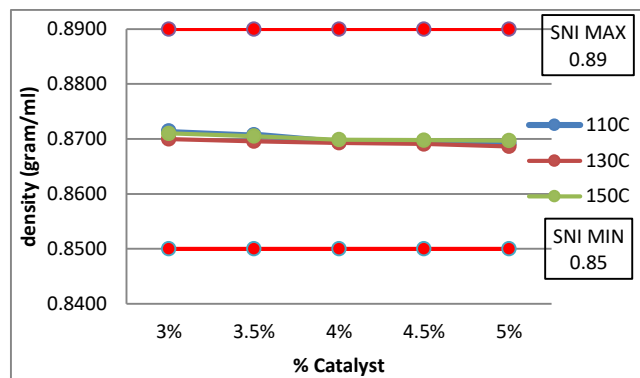


Figure 4. Effect of Type and Amount of Catalyst on Biodiesel Density

As shown in **Figure 4**, the sample has met the standard biodiesel range, which is between 0.85-0.89 g/mL. Density tends to decrease as the amount of catalyst used increases. So, it can be concluded that the greater the amount of catalyst used, the lower the density of the biodiesel produced, this is because it is influenced by the molar ratio of its constituent components so that the presence of ammonium salts and Hydrogen Bond Donors greatly affects the density value. The higher the density value, the higher the density. fuel consumption, this is because more fuel will be injected into the combustion chamber to get the same engine power [16]. The highest density value is 0.8714 g/mL obtained by a sample of potassium glycerolate catalyst at 110 °C KGT10 K3 as much as 3% catalyst. Meanwhile, the lowest density value was obtained by a sample of potassium glycerolate catalyst at 150 °C KGT50 K5 with 5% catalyst, which was 0.8697 g/mL.

Effect of Type and Amount of Catalyst on Biodiesel Viscosity

The viscosity of biodiesel is in accordance with the SNI 7128: 2015 standard, which is in the range of 2.3-6.0 cSt. The higher the viscosity value of biodiesel indicates the thicker the biodiesel is, conversely the lower the viscosity value, the more liquid the biodiesel is. The use of an alkaline catalyst can reduce the viscosity value of biodiesel [16]. The viscosity value will influence the performance of the injectors in diesel engines. High viscosity can result in liquid having better lubricating properties. The viscosity value of the biodiesel produced can be seen in **Figure 5**. Almost all biodiesel meets the specified viscosity value, which is around 2.3 – 6.0 cSt.

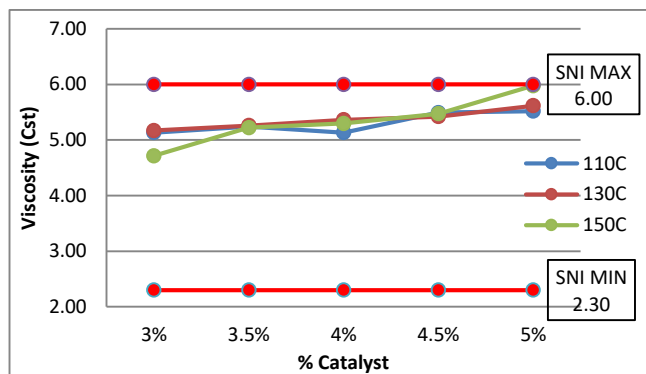


Figure 5. Effect of Amount of Catalyst on Biodiesel Viscosity

Effect of Type and Amount of Catalyst on Biodiesel Conversion

The performance of the potassium glycerolate catalyst can be seen from several test parameters, one of which is the yield of biodiesel conversion which is calculated based on the formula. Conversion is defined as the amount of substance that turns into products compared to the amount of reactants. The greater the resulting conversion means that more raw materials are turned into products and it can be said that the reaction is going well. Based on the stoichiometry of the chemical reaction, one mole of triglyceride requires three moles of alcohol to form three moles of alkyl esters and one mole of glycerol. This causes the more alcohol compounds to be added, the greater the number of alkyl esters produced [17]. The percentage of biodiesel conversion can be seen in **Figure 6**.

Based on **Figure 6**, the highest conversion was obtained by using a potassium glycerolate catalyst at 150 °C KGT50 K5 as much as 5% catalyst reaching 97.45%. The lowest conversion value was obtained by using a potassium glycerolate catalyst at 110 °C KGT10 K3 as much as 3% catalyst which is equal to 94.44%. This indicates that the conversion is higher with the increase in the amount of catalyst. The more catalyst, the larger the surface and the more active sides of the catalyst. So, more and more triglyceride molecules will react. Potassium glycerolate catalyst made from 110 °C to 150 °C showed that an increase in temperature causes an increase in the conversion formed. This is because the higher the temperature of the catalyst, the more potassium glycerolate will be formed.

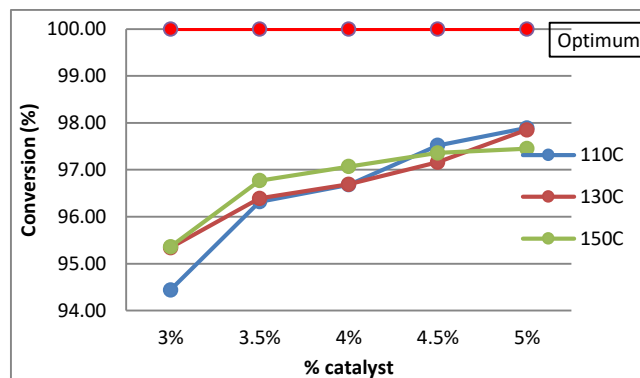


Figure 6. Effect of Type and Amount of Catalyst on Biodiesel Conversion

Effect of Type and Amount of Catalyst on Biodiesel Methyl Ester Content

Based on the standards set in SNI-7182: 2015 that the minimum methyl ester content in the biodiesel produced is 96.5%. The level of methyl ester is an indicator of the success of the biodiesel formation reaction. Data on methyl ester levels in this study can be seen in **Figure 7**. Where almost all of the biodiesel samples met the SNI7-182: 2015 standard.

Methyl ester content has an inverse relationship with the total glycerol value. The lower the total glycerol value of biodiesel, the higher the methyl ester content and vice versa. Based on **Figure 7**, it is known that 5 out of 15 samples did not meet the specified minimum standard for methyl ester content. All samples made using potassium glycerolate catalyst at 150 °C KGT50 met the standard for methyl ester levels. The highest methyl ester content was obtained by the potassium glycerolate catalyst sample at 150 °C KGT50 K5 as much as 5% catalyst by 99.0514% and the lowest by 93.5824% by the glycerolate catalyst sample at 110 °C KGT10 K3 by 3% catalyst. High levels of methyl esters indicate that the performance of the catalyst in the process of converting triglycerides to methyl esters is running optimally.

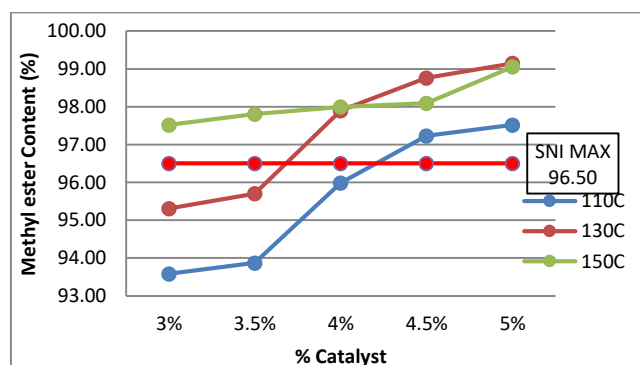


Figure 7. Effect of Type and Amount of Catalyst on Methyl Ester Content

CONCLUSION

The higher the temperature of glycerolate formation, the higher the conductivity, density, viscosity, pH of the formed potassium glycerolate catalyst, while the lower the freezing point. The greater the amount of catalyst used, the lower the acid value, total glycerol from the biodiesel produced, while the higher the methyl ester content. The higher the temperature of the formation of the potassium glycerolate catalyst, the lower the acid value, total glycerol from the biodiesel produced, while the methyl ester content will be greater. The most optimal catalyst is a sample of potassium glycerolate catalyst at 150 °C KGT50 with specifications of conductivity 4482 $\mu\text{S}/\text{cm}$, density 1.4858 g/cm^3 , viscosity 121.574 cP, freezing point -8 °C and pH 14,

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