

# Glycerol Conversion from Biodiesel of Cooking Oil with Recydu Catalytic Cracking Catalyst At Pertamina

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## ABSTRACT

*Glycerol is a by-product of the biodiesel manufacturing process, glycerol is obtained by reacting triglycerides with alcohol. The raw materials used are used cooking oil and methanol. The purpose of this research To determine the effect of Recydu Catalytic Cracking catalyst on glycerol conversion. In this study to obtain the conversion of glycerol, namely through 2 reaction processes, namely the esterification reaction and the trans-esterification reaction, by using 4 variables, namely Stirring Speed and Reaction Comparison and Stirring Time and Amount of Catalyst. The glycerol conversion was obtained from the analysis data for the determination of the total fatty acid equivalent and the analysis of the acetin method. From the results of the study, it was found that the variation of stirring time on the amount of RCC catalyst obtained the largest conversion, namely 52.3% at optimum conditions at 105 minutes and the amount of catalyst was 2.1 grams. Meanwhile, in the variation of Stirring Speed and Reaction Comparison, the largest conversion was obtained, namely 54.51% at the optimum condition at a reaction ratio of 1:3 and the optimum condition at a speed of 90 rpm.*

**Keywords: Glycerol, Crystalline Zeolite, Cooking Oil**

## 1. INTRODUCTION

Glycerol (glycerol, glycerin, glycerine) is the simplest glyceride compound, with the hydroxyl being hydrophilic and hygroscopic. Glycerol is a component that composes various kinds of lipids, including triglycerides. Glycerol is widely used as a raw material for the chemical, pharmaceutical, and cosmetic industries. Synthetic glycerol from petrochemical hydrocarbons fulfills 40% of market demand, while the rest is obtained from glycerol recovery as a by-product of liquid soap by distillation and as the main raw material for other oleochemical products that use natural fats and oils. (Aziz et al, 2013).

Glycerol is a by-product of biodiesel production from the transesterification reaction and is an alcohol compound with three hydroxyl groups. Glycerol (1,2,3 propanetriol) is a colorless, odorless and viscous liquid that has a sweet taste. Glycerol can be purified by the distillation process so that it can be used in the food, pharmaceutical industry or can also be used for water treatment. As a by-product of the biodiesel industry, glycerol has

not been processed much so that its selling value is still low (Prasetyo, 2012).

The main raw materials used in the manufacture of glycerol are oils including palm oil, kapok seed oil and rubber seed oil. Used cooking oil (food industry and household waste) can also be used as raw material for the manufacture of glycerol. The use of used cooking oil is expected to reduce waste production and increase the selling value of used cooking oil itself.

Glycerol is a by-product of biodiesel. Biodiesel is one type of fuel that is produced using vegetable oil or animal fat through a transesterification process or an esterification process with the help of alcohol and a catalyst (Setiawati, et al, 2012).

Transesterification produces fatty acid methyl esters (FAME) or biodiesel and glycerol (glycerin) as by-products. The catalyst used in the transesterification process is base/alkali (Hikmah, 2010).

In the research on the reaction kinetics of making biodiesel from used coconut oil, the conversion calculation is carried out by analyzing the glycerol content in the reaction product. The analysis of glycerol in the material can be carried out by the acetin method (Griffin, 1958).

From the research reference (Endah Pratiwi & Fristita Mauliana Sinaga, 2017), the glycerol conversion to be carried out is different in the catalyst used, namely the RCC (Residu Catalytic Cracking) catalyst of the crystalline Zeolite type, namely from the waste of the Pertamina Petroleum Cracking process.

## **1. RESEARCH METHODOLOGY**

In this study, the reaction for making biodiesel was carried out in two stages of reaction,

namely the esterification reaction of used coconut oil (cooking) with methanol using an H<sub>2</sub>SO<sub>4</sub> catalyst to remove free fatty acids, and the transesterification reaction of used coconut oil (cooking) methanol using a KOH catalyst (Prasetyo, 2006 and 2005). Winoto 2006). In this study, the catalyst used was a crystalline zeolite type RCC (Residu Catalytic Cracking) catalyst.

### **a. Esterification Reaction Stage**

Endah Pratiwi & Fristita Mauliana Sinaga, 2017 testing the Esterification Reaction Stage that has been done using 150 ml of used cooking oil was put into a three neck flask then added 0.375 ml of concentrated H<sub>2</sub>SO<sub>4</sub> (0.25% by weight of oil), then heated to a temperature of 60°C. 185 ml of methanol was put into an Erlenmeyer which has been equipped with a back cooler and then heated to a boiling point of 64.7 °C. Next, the methanol is fed into the stirred reactor through the return cooling channel. The temperature in the reactor was maintained at 60°C for 30 minutes. After 30 minutes the reaction was stopped and then put into a separating funnel and allowed to stand until it reached room temperature (left for 24 hours). There are 2 layers, namely the top of the biodiesel and the bottom of the glycerol.

### **b. Transesterification Reaction Stage**

Endah Pratiwi & Fristita Mauliana Sinaga, 2017 did a test the Transesterification Reaction Stage that has been done using 1.7 g of RCC catalyst was dissolved with 200 ml of methanol into a beaker and stirred with a magnetic stirrer. 100 ml of oil was put into a glass beaker containing methanol-RCC. The mixture was then stirred with a magnetic stirrer at a speed of 150 rpm for 75 minutes. The mixture was fatty acid equivalent

(EALT). allowed to stand for 24 hours. The same process was carried out for variations in the amount of RCC catalyst, namely 1,9: 2,1: 2,3: 2,5. and time variations of 90 and 105 minutes. The reaction mixture obtained was filtered using filter paper to separate the RCC catalyst. The reaction mixture was put into vacuum distillation to separate the methanol. After the ester and glycerol formed into 2 layers, the distillation was stopped. The bottom layer is glycerol and the top layer is ester and residual oil.

### c. Determination of total fatty acid equivalent (EALT)

Endah Pratiwi & Fristita Mauliana Sinaga, 2017 once conducted a test for Determination of total fatty acid equivalent (EALT). Glycerol esterified as much as 5gr was put into an Erlenmeyer then added 50 ml of 0.5 N NaOH and then heated for 30 minutes. Furthermore, after being cooled this solution was added 3 drops of pp indicator and titrated with 0.5 N HCl solution until the color changed to clear.

$$EALT = \frac{(V_{blank} - V_{titration})N_{HCl}}{M_{glycerol}} \quad (1)$$

EALT = total fatty acid equivalent, mgrek/gram.

Vblanko = blank titration HCl volume, ml

Vtitration = volume of titrated HCl sample, ml

NHCl = normality HCl, grek/L

### d. Analysis of Acetine Method Results

Tests conducted by Endah Pratiwi & Fristita Mauliana Sinaga, 2017 got the Analysis of Acetine Method Results Weighing 1.5 g of glycerol then put into a 250 mL erlenmeyer, and added 3 grams

of sodium acetate and 7.5 mL of acetic anhydride, then boiled for 1 hour. Furthermore, the mixture reaches approximately 80 °C, the addition of 50 ml of distilled water at a temperature of 80 °C through a back funnel. After that it was fused to room temperature, removable cooling back, then added 2 mL of phenolphthalein indicator. After that, the liquid was neutralized with 6 N NaOH solution, a pink color appeared, then 10 mL of 1 N NaOH solution was added. After the cooling was installed, the mixture was boiled again for 15 minutes. the liquid as soon as possible and then titrated with 0.5 N HCl until the pink color disappears. Then the volume of HCl used for the titration is recorded. In addition, a blank titration was carried out in the same way, but without snippets. Then HCl for analysis was also recorded. The amount of free glycerol at the end of the reaction is expressed by the equation:

$$G = \frac{W1W3(Vb - Vs)NHCl}{W4} \quad (2)$$

Because the weight of the mixture and the weight of the sample are the same, the equation becomes:

$$G = \frac{W3(Vb - Vs)NHCl}{W4} \quad (3)$$

G = amount of glycerol produced, mgrek

W1 = weight of mixture. gr

W2 = weight of sample taken, gr

W3= weight of glycerol after heating, gr

W4= weight of glycerol after analysis, gr

Vb = volume of HCl blank titration, ml

Vs = volume of titrated HCl sample, ml

NHCl = normality of HCl solution, mgrek/l

Conversion (Xm) is calculated by the equation:

$$Xm = \frac{C}{G0} \tag{4}$$

G0 = initial total glycerol, mgrek

G0 = S x A

S = Total fatty acid equivalent mgrek/gr

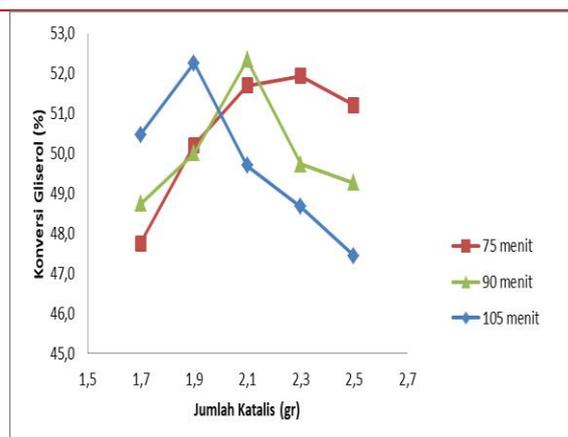
A = weight of oil

## 2. RESULTS AND DISCUSSIONS

**Table 1** Glycerol Conversion Results based on variations in stirring time to the amount of catalyst

Stirring Time (minutes)	Catalyst (grams)	Glycerol Conversion (%)
75	1.7	47.75
	1.9	50.20
	2.1	51.69
	2.3	51.94
	2.5	51.20
90	1.7	48.74
	1.9	50.00
	2.1	52.33
	2.3	49.72
	2.5	49.26
105	1.7	50.46
	1.9	52.26
	2.1	49.69
	2.3	48.66
	2.5	47.44

From these data, a graph of the relationship between the stirring time and the amount of catalyst obtained can be made.



**Figure 1** Glycerol Conversion Results based on variations in stirring time to the amount of catalyst

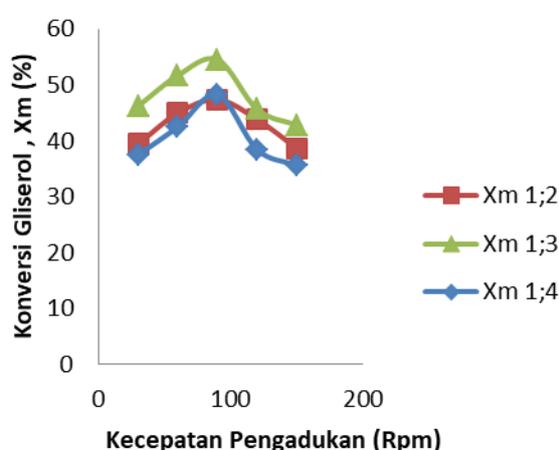
Based on these results, the stirring time and the amount of catalyst affect the glycerol content obtained. From the data above, it can be seen that the greatest increase in glycerol conversion occurred at a stirring time of 105 minutes. As for the variation in the amount of catalyst, in this study the most optimum amount of catalyst was found in the amount of catalyst 1.9 grams, the resulting glycerol conversion value was 52.3%. The longer the time used for the reaction, the greater the conversion achieved, this indicates that the longer the reaction time, the greater the opportunity to react.

**Table 2** Glycerol Conversion Results based on variations in Stirring Speed and Comparison of Reagents

Comparison (Glycerol:Methanol)	Stirring Speed (rpm)	Glycerol Conversion (%)
1:2	30	39.41
	60	44.98
	90	47.37
	120	43.81
	150	38.63
1:3	30	46.21
	60	51.69
	90	54.51

1:4	120	45.61
	150	42.77
	30	37.50
	60	42.50
	90	48.26
	120	38,40
	150	35.59

From the research data above, a graph of the relationship between Stirring Speed and Comparison of Mixers can be made



**Figure 2** Glycerol Conversion Results based on variations in Stirring Speed and Reagent Comparison

Based on the graph above, it was found that the most optimal increase in glycerol conversion occurred at a stirring speed of 90 rpm. As for the variation in the ratio of the most optimum reagents occurred in the ratio of 1:3 reagents between glycerol and methanol. The glycerol conversion value produced at a stirring speed of 90 rpm with a 1:3 ratio of reagents was 54.51%.

### 3. CONCLUSIONS

1. From the results of the research on the variation of stirring time on the amount of RCC catalyst, the largest conversion was 52.3% at optimum

conditions at 105 minutes and the amount of catalyst was 2.1 grams.

2. From the results of the research on variations in stirring speed and reaction ratio, the largest conversion was obtained, namely 54.51% at the optimum condition at a reaction ratio of 1:3 and the optimum condition at a speed of 90 rpm.

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