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B. ENG (HONS) CHEMICAL ENGINEERING

SEPTEMBER 2015

**DEVELOPMENT OF GENUINE REACTION KINETICS OF  
TRANSESTERIFICATION BASED ON TWO-PHASED  
MODEL**

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**CHEMICAL ENGINEERING  
UNIVERSITI TEKNOLOGI PETRONAS  
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Two-Phase Model**

by

Mohammad Faizal Bin Suhaimi  
15675

Dissertation submitted in partial fulfillment  
of the requirement for the  
Bachelor of Engineering (Hons)  
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SEPTEMBER 2015

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# CERTIFICATION OF APPROVAL

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Chemical Engineering Programme  
Universiti Teknologi PETRONAS  
in partial fulfilment of the requirement for the  
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Approved by,

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(Dr Yoshimitsu Uemura)

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September 2015

## CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgment, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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MOHAMMAD FAIZAL BIN SUHAIMI

## ABSTRACT

A kinetic study of KOH-catalyzed transesterification of palm oil with methanol was carried out. Overall transesterification reaction consists of three consecutive second order reversible reaction steps. Experiments were performed to estimate the saturated concentration of methanol in oil, average methanol droplet size, reaction constant,  $k$  and diffusion coefficient of methanol in oil. The experiments were performed with reaction mixture that had methanol to oil molar ratio of 6:1 and the catalyst was potassium hydroxide (2 wt. % of methanol). The reaction were conducted at room temperature of 24°C for 180 minutes. Analysis of FAME will be carry out by high performance liquid chromatography (HPLC) to get the result for oil conversion over reaction time. The overall reaction rate,  $r_{\text{overall}}$  of the palm oil-methanol transesterification was calculated, consequently, prove the kinetic model

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## **LIST OF ABBREVIATIONS**

DG	Diglycerides
FAME	Fatty Acid Methyl Ester
FYP	Final Year Project
HPLC	High Performance Liquid Chromatography
MG	Monoglyceride
TG	Triglycerides
UTP	Universiti Teknologi PETRONAS

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# CHAPTER 1

## INTRODUCTION

### 1.1 Background Study

For a long time, crude oil has been the world's solely energy source. The limited fossil fuels resources along with the need to reduce the greenhouse gas emissions from the combustion of these fuels resulted interest in finding renewable raw material (biomass) as a fuel alternatives. With the development of biomass based power, this would ensure the society's need for new renewable power alternatives for the future. On other hand, its production from resources, such as vegetable oils and animal fats make it biodegradable and non-toxic. Consequently, contributes to the reduction of emission such as carbon dioxide (CO<sub>2</sub>), carbon monoxide (CO), and Sulphur dioxide which cause the global warming.

In 1912, during a demonstration at the World's Fair, Rudolf Diesel had stated "the use of vegetable oils for engine fuels may seem insignificant today. But such oils may become, in the course of time, as important as the petroleum and coal tar products of the present time."(Zeman, 2009). His words have taken a significance by today as we can see the use of biodiesel have significantly increased over the past decade in industry and automobiles. Generally, transesterification is the most common method for the conversion of triglycerides in oil into glycerol (byproduct) and fatty acid alkyl esters. In principle, the reaction of triglycerides with alcohol such as methanol or ethanol referred as alcoholysis.

The development of reaction kinetics of transesterification of liquid-liquid phase is at its initial stages. Several studies have been carried out using different oils, alcohols and catalyst (based-catalyst and acid-catalyst) with various parameters, thus a numbers of reasons for transesterification reaction have been also discussed. The transesterification reaction involves two immiscible phase (oil and alcohol). The alcoholysis process occurs via pseudo-homogeneous regime where the mass transfer and chemical reaction between these liquids can occur near the interfacial region of alcohol droplets.

## 1.2 Problem Statement

Since the past few decades, biofuels such as biodiesel have rapidly transitioned from university laboratories into commercial production. High conversion of oil in short time periods of transesterification process is very important for a large production of biodiesel in industries. Different parameters have been investigated in this reaction including alcohol-to-oil molar ratio, catalyst concentration, and temperature (Reyero, Arzamendi, Zabala, & Gandía, 2015). The overall reaction rate is affected by chemical kinetics, mass transfer and component solubility (De Boer & Bahri, 2009). Thus, the reaction kinetic model is required to be developed for further research on the transesterification reaction.

With these consideration, the author will carry out a study on the two-phase (heterogeneous) transesterification process by using palm oil-methanol system in the presence of potassium hydroxide as a catalyst of the reaction. Consequently, the author need to find the methanol droplet size, reaction constant,  $k$  and diffusion coefficient of methanol in oil to estimate overall reaction rate,  $r_{\text{overall}}$  of the palm oil-methanol transesterification from the formulated diffusion-reaction intrinsic kinetic model.

### **1.3 Objective**

The main objectives of this project is to study a diffusion-reaction kinetic model of transesterification based on the two-phase model by using palm oil-methanol system.

### **1.4 Scope of Study**

The scope of study for this research are:

- i. To study the diffusion-reaction kinetic model of transesterification empirically.
- ii. Obtain overall reaction rate from the diffusion-reaction kinetic model by formulating the oil concentration, methanol droplet size and reaction kinetic constant.
- iii. Obtain methanol droplet size, solubility of oil in methanol, and kinetic constant empirically.
- iv. Estimate the diffusion coefficient of oil in methanol by a theoretical equation.

## CHAPTER 2

### LITERATURE REVIEW AND THEORY

#### 1.5 Biodiesel and Transesterification Reaction

Biodiesel, as defined by the U.S Department of Energy, is a form of diesel fuel manufactured from vegetable oils, animal fats, or cooking oils. It is clean, biodegradable, and produce less air pollutants than petroleum-based diesel from the combustion. The biodiesel satisfied the requirement of a diesel engine as there were molecular similarities between the petroleum (diesel) and biodiesel (Ataya, Dubé, & Ternan, 2006). The major production of biodiesel process, namely transesterification, is a catalyzed chemical reaction that convert triglyceride (TG) to yield fatty acid alkyl ester (FAAE) and a co-product, glycerol (De Boer & Bahri, 2009). Methanol is the most commonly used alcohol compare to others because of its low cost. In addition, the conversion of pure triglycerides to fatty acid methyl ester (FAME) is high, and the reaction time is relatively short (Wang, Ou, & Zhang, 2007).

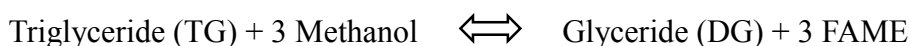
According to De Boer and Bahri (2009)

It is widely recognized that the transesterification reaction of triglyceride proceeds via three consecutive reversible reactions:

##### Stepwise Reactions:



##### Overall Reaction:



The intermediate reactants (diglycerides and monoglycerides) appear in small concentration and are considered as contaminants in the final product.

Different catalysts have been examined in the transesterification reaction. Wang (2007) says that the transesterification reactions can be alkali catalyzed, acid catalyzed or enzyme catalyzed, but the most common used and received more attention were the first two types because of short reaction time and lower cost. An acid catalyzed process required more excess of alcohol, high pressure (170-180 kPa) and high cost stainless steel equipment but give low product yield. To overcome the disadvantages of the acid catalyzed process, several experiment has been conducted. Meanwhile, an alkali catalyzed process can achieve high purity and yield of biodiesel product in a short time and easy to perform when the majority of the free fatty acid in waste cooking oil was methanolysised (Wang et al., 2007).

## 1.6 Transesterification Kinetic Model

Study of transesterification reaction have shown that its evolution from a multiphase (liquid-liquid) mixture (oil-methanol) to another biphasic mixture (glycerol and FAME) via pseudo-homogeneous phase reaction mixture. Throughout the kinetic model formulation by Reyero et al., a continuous non-polar phase (oil, biodiesel, intermediate reactants) and a polar phase (methanol, glycerol and catalyst) are presented with the composition of the phases constantly changing.

According to Karmee and Chandna (2006)

The reaction steps are in the equation as shown:



The general form of the governing set of differential equation characterizing the stepwise reactions involved in the transesterification is:



$$-\frac{d[TG]}{dt} = k_1[TG][A] - k_2[DG][E] \quad (4)$$

$$-\frac{d[DG]}{dt} = k_3[DG][A] - k_4[MG][E] - k_1[TG][A] + k_2[DG][E] \quad (5)$$

$$-\frac{d[MG]}{dt} = k_5[MG][A] - k_6[GL][E] - k_3[DG][A] + k_4[MG][E] \quad (6)$$

$$-\frac{d[E]}{dt} = k_1[TG][A] - k_2[DG][E] + k_3[DG][A] - k_4[MG][E] + k_5[MG][A] - k_6[GL][E] \quad (7)$$

where [] refer to the concentration of the corresponding species and A, E and GL refer to alcohol, ester and glycerol, respectively.

The reaction kinetic constant,  $k_{1-6}$  are estimated based on the data from experimental plot. The number of parameters are reduced to three equilibrium constants,  $K_1$ ,  $K_2$ , and  $K_3$  which defined as

$$K_1 = \frac{k_1}{k_2}, K_2 = \frac{k_3}{k_4}, K_3 = \frac{k_5}{k_6} \quad (8)$$

## 1.7 Diffusion-reaction Model

The theory of simultaneous diffusion and reaction of the liquid-liquid phase in a catalyst system is a new approach in engineering practice. A model have been developed as shown in FIGURE 2.1 to illustrate the approach of mass transfer and chemical reaction.

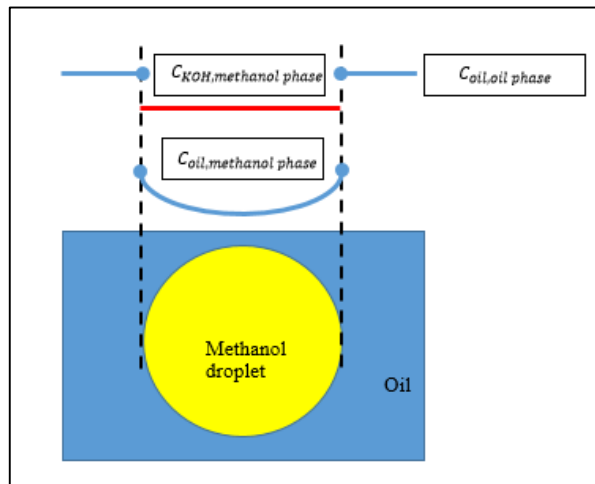


FIGURE 2.1 Diffusion-reaction model

The mass balance equation of the oil in methanol droplet is described by the relation:

$$D_{oil,m} \left( \frac{d^2 C_{oil,m}}{dr^2} + \frac{2}{r} \frac{dC_{oil,m}}{dr} \right) = k C_{oil,m} \quad (9)$$

where  $D_{oil,m}$  is the diffusion coefficient;  $C_{oil,m}$  is concentration of oil in methanol phase;  $r$  and  $k$  is the radius of methanol droplet and intrinsic reaction-rate constant, respectively.

Solving the mass balance equation, the effectiveness factor,  $y$  is derived as

$$y = \frac{1}{\phi_s} \left( \frac{1}{\tanh(3\phi_s)} - \frac{1}{3\phi_s} \right) \quad (10)$$

$$\phi_s = \frac{d_p}{6} \sqrt{\frac{k}{D_{oil,m}}} \quad (11)$$

$$y = \frac{\text{real reaction rate}}{\text{reaction rate when oil conc.in the droplet is the same as saturated concentration}} \quad (12)$$

Hence, the overall reaction rate is expressed as follows:

$$-r_{overall} = ykC_{oil,saturated} = k \frac{6}{d_p} \left( \frac{D_{oil,m}}{k} \right)^{1/2} C_{oil,saturated} \quad (13)$$

The right most form is obtained when the diffusion control is predominant.

## CHAPTER 3

### METHODOLOGY

#### 2.1 Identify Rate Constant

Experiments are to be conducted in order to measure the conversion of triglycerides in palm based-cooking oil over reaction time and then estimate the reaction-rate constant,  $k$ . The reaction mixture had a methanol to oil molar ratio of 6:1 and the catalyst was potassium hydroxide (2 wt. % of methanol). The reaction are conducted at room temperature of 25°C for 180 minutes.

##### 2.1.1 Chemical and Apparatus

Palm based-cooking oil is purchased from a local market. Methanol and potassium hydroxide are purchased through request from lab technologist. The basic equipment used during the experimentation were:

- i.glass reactor (500 ml capacity three-necked flask)
- ii.magnetic stirrer
- iii.condenser
- iv.electronic magnetic hot plate
- v.electronic weight balance
- vi.thermocouple

### 2.1.2 Experimental Setup and Procedures

Figure 1 shows the experiment setup for biodiesel production through transesterification.

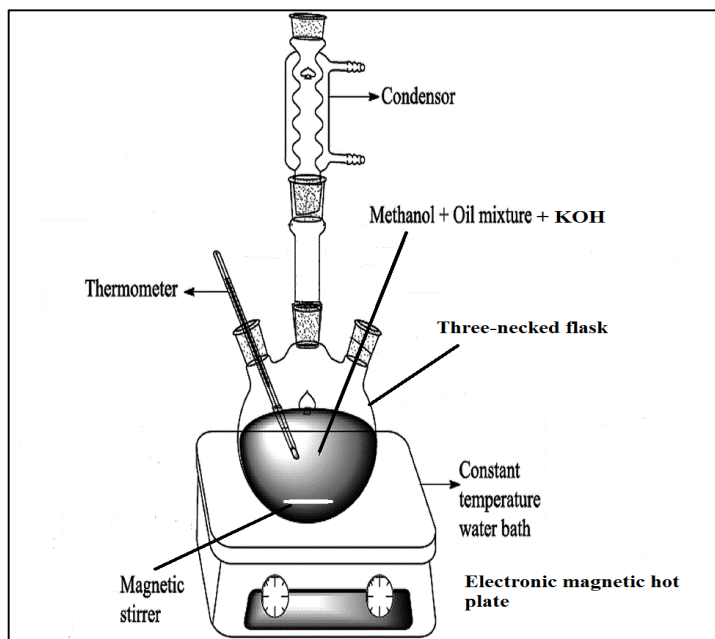


FIGURE 3.1 Batch transesterification reactor system

The expected run is conducted as follows:

- i. Initially, the weight of cooking oil is measured using electronic weight balanced before the amount of methanol and potassium hydroxide needed for reaction can be calculated based on molar ratio and catalyst weight percentage. The cooking oil is poured into three neck flask and the temperature is to be set at 25°C using electronic magnetic hot plate and thermometer. The methanol is placed in a beaker.
- ii. Potassium hydroxide is added quickly into the methanol and the mixture's beaker is closed and tighten securely to minimize the exposure to air.
- iii. With enough time, potassium hydroxide will dissolve in the methanol and the temperature of oil reached at constant 25°C. Next, the catalyst and methanol

solution is added into the cooking oil.

- iv. The reaction sample (3 mL) is withdrawn by means of a syringe during the experiments in proposed periods until the end of the experiment.
- v. 2 mL of Acetic acid is added quickly into each sample taken to stop the reaction of methanol and TG.
- vi. Each sample is then centrifuged for 10 minutes to ensure a thorough separation of biodiesel and others component.

### 2.1.3 Analysis of Triglyceride

Analysis of TG for each sample was performed by dissolving 100  $\mu\text{L}$  of biodiesel sample into 3 mL n-hexane and injecting 60  $\mu\text{L}$  of this solution in the high performance liquid chromatography (HPLC). A chromatographic peak area for TG is obtained and the TG conversion is calculated by following equation:

$$TG \text{ Conversion} = \left(1 - \frac{x}{w}\right) \times 100 \quad (14)$$

Where,

x is peak area of TG in sample

W is peak area of TG in oil

### 2.2 Measurement of Methanol Droplet Size

A sample from transesterification reaction experiment is taken to observe the methanol droplet size in the oil by using a microscope and calculate the average droplet size.

### 2.2.1 Chemical and Apparatus

The basic chemical equipment used during the experimentation were:

- i. 5 ml of methanol and oil mixture sample
- ii. Microscope

### 2.2.2 Experimental Setup and Procedures

FIGURE 3.2 shows the experiment setup to observe the sample from experiment 1 and then calculate the average methanol droplet size.

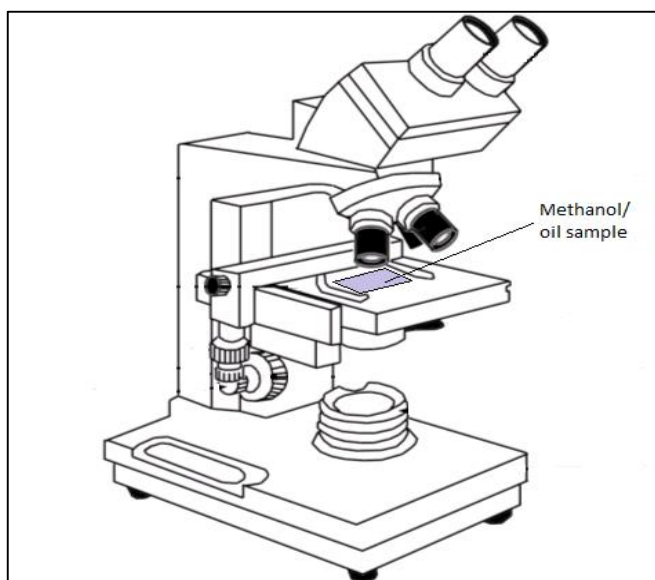


FIGURE 3.2 Observing methanol droplet in oil using microscope

The expected run is conducted as follows:

- i. A 5 ml sample is taken from the methanol and oil mixture by using a pipette.
- ii. The sample is taken to a microscope and the methanol droplets in oil are to be measured.

- iii. The methanol droplets size are record and average droplet size is to be calculated.

### **2.3 Solubility Measurement of Palm Oil in Methanol**

Experiments are to be conducted in order to estimate the saturated concentration of methanol and cooking oil solution by using titration method.

#### **2.3.1 Chemical and Apparatus**

The basic chemical and equipment used during the experimentation were:

- i. 10 ml of methanol
- ii. 50 ml cooking oil-palm based
- iii. Burette
- iv. Beaker
- v. Electronic magnetic heater
- vi. Stirrer
- vii. Retort stand

#### **2.3.2 Experimental Setup and Procedures**

FIGURE 3.3 shows the experiment setup to measure the amount of oil needed to make the solution of methanol and oil become saturated.



FIGURE 3.3 Methanol-oil titration setup

The expected run is conducted as follows:

- i. Take an initial volume reading of burette containing cooking oil and record the value.
- ii. Prepare the 10 ml of methanol in the flask.
- iii. Use the burette to deliver drops of oil and observe the solution until it become turbid.
- iv. After the solution become turbid, read the final volume in the burette and record the value.
- v. Subtract the initial volume to determine the amount of oil delivered. Use this to calculate the saturated concentration of the oil in methanol solution.



## 2.4 Diffusivity of Palm Oil into Methanol

A method of correlating diffusion coefficient was proposed by Wilke (1955) on basis of Stokes-Einstein equation. The correlation for diffusion in methanol may be expressed by the equation:

$$D = \text{const.} \frac{(xM)^{0.5} \cdot T}{\eta V^{0.6}} \quad (15)$$

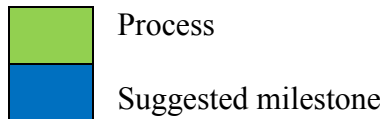
Where,

- $D$  is diffusion coefficient,  $cm^2/sec$
- $x$  is association parameter, (methanol =1.9)
- $M$  is molecular weight of solvent,  $gmol^{-1}$
- $T$  is temperature,  $K$
- $\eta$  is viscosity of solution,  $cp$
- $V$  is molar volume of solute,  $cm^3/gmol^{-1}$

## 2.5 Gantt Chart and Key Milestone

TABLE 3.1 Gantt chart

No.	Detailed work	Week														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	
1.	Project work continue	Process	Process	Process	Process	Process	Process	Process								
2.	Submission of progress report							Milestone								
3.	Project work continues								Process	Process	Process	Process	Process			
4.	Pre-SEDEX										Milestone					
5.	Submission of Draft Final Report											Milestone				
6.	Submission of Dissertation												Milestone			
7.	Submission of Technical paper													Milestone		
8.	Viva														Milestone	
9.	Submission of Project Dissertation															Milestone



## CHAPTER 4

### RESULT & DISCUSSION

#### 3.1 Reaction Rate Constant

Transesterification reaction was carried out at 25°C, 2 wt. % of KOH in methanol and initial methanol to cooking oil molar ratio of 6:1. The conversion of TG in the presence catalyst, KOH were measured as a function of time for the transesterification of palm based cooking oil. Result are shown in TABLE 4.1 and FIGURE 4.1.

TABLE 4.1 Peak area of triglyceride & TG Conversion

Time	Peak Area	TG Conversion
Feedstock	10 548 759	-
10 min	2 195 296	0.79
20 min	2 027 176	0.81
30 min	2 395 265	0.77
40 min	2 195 640	0.79
50 min	2 232 918	0.78
60 min	2 190 699	0.79
90 min	2 182 954	0.79
120 min	2 589 592	0.75
150 min	2 476 464	0.77
180 min	2 660 972	0.75

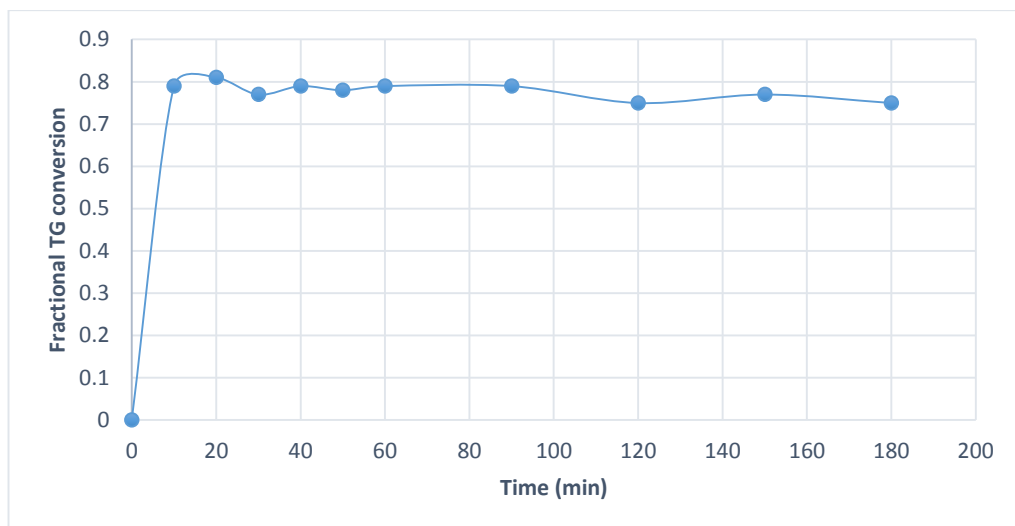


FIGURE 4.1 TG conversion versus time

From FIGURE 4.1, it shows that the increase of TG conversion during early 10 minutes. It is expected for the conversion of TG to increase with time. The effect of sample taken during mixing for each period may cause the data obtained is not really good in accuracy since the conversion should be increase over time. During analysis, the injection of sample method into HPLC also may affect the peak area obtained from chromatogram that may affect the plot in FIGURE 4.1.

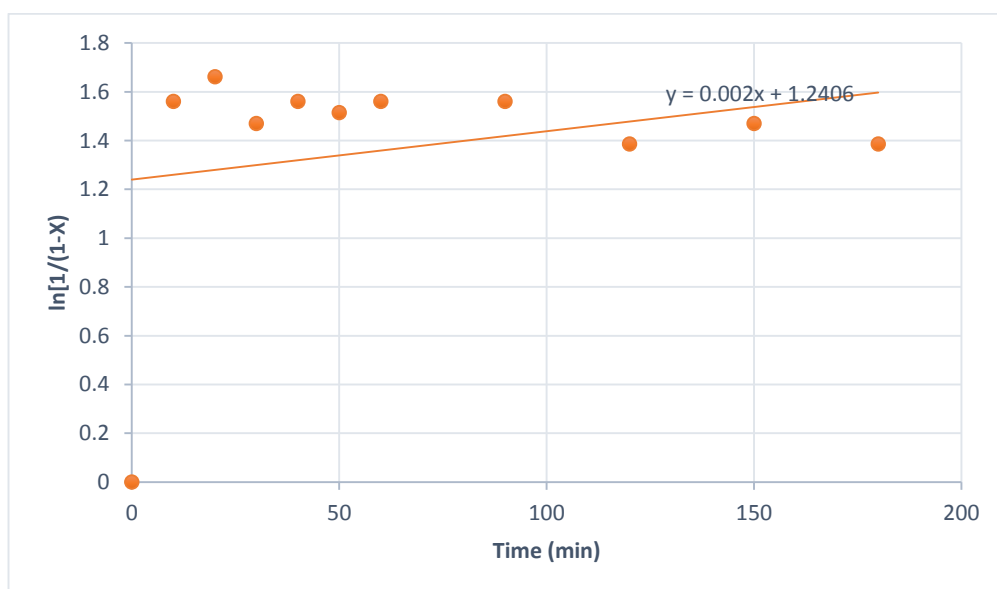


FIGURE 4.2  $\ln(1/(1-X))$  versus time

For a first-order reaction, a plot of  $\ln(1/(1-X))$  as a function of time will be linear with a slope equal to the reaction rate constant,  $k$ . From the linear line it is found that rate constant,  $k$  is  $0.002 \text{ min}^{-1}$ .

### 3.2 Measurement of Methanol Droplet Size

Samples of transesterification reaction is taken to measure their respective droplet size and thus find the average methanol droplet size in the solution.

The size of methanol droplet is at 0.005 mm.

### 3.3 Solubility Measurement of Palm Oil in Methanol

The saturated concentration of palm oil in methanol mixture,  $C_{\text{sat}}$  is measured at room temperature,  $25^\circ\text{C}$ . In this experiment the amount of cooking oil is measured to find when the solution will become turbid after the titration. The appearance of turbid/cloudy solution show that the solution is already achieved saturated condition.

TABLE 4.2 Palm oil saturated concentration in methanol

Experiment	Amount of methanol, mL	Amount of palm oil, mL	Palm oil concentration in methanol, M
1	14.76	0.20	0.0143
2	14.23	0.15	0.0111
3	48.79	0.50	0.0110
4	48.94	0.55	0.0119

Based on the table, the solubility of oil in methanol mixture was found to be at 0.014 mol/L (1.52 wt. %).

### 3.4 Calculation of Diffusion Coefficient

Diffusion coefficient's is calculated by the equation (15). The equation constant is determined from the figure below.

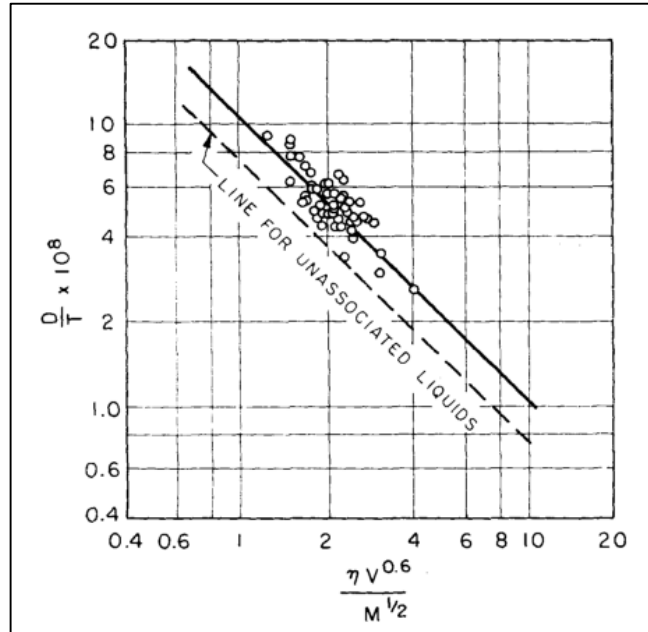


FIGURE 4.3 Diffusion in methyl alcohol

Therefore, the equation to calculate diffusion coefficient of oil into methanol is:

$$D = 10.5 \times 10^{-8} \cdot \frac{(xM)^{0.5} \cdot T}{\eta V^{0.6}} \quad (16)$$

By use of the given association parameter and data for diffusion in methanol, the diffusion coefficient, D was calculated.

#### Data:

$$X_{\text{MeOH}} = 1.9 \quad M_{\text{MeOH}} = 32.04 \text{ gmol}^{-1}$$

$$T = 25^\circ\text{C} \quad V = 965.02 \text{ cm}^3/\text{gmol}^{-1}$$

$$\eta_{\text{sol}} = 2.92 \text{ cp}$$

The diffusion coefficient of oil into methanol was found to be  $1.354 \times 10^{-6} \text{ cm}^2/\text{sec}$ .

### 3.5 Overall reaction rate

Overall reaction rate is expressed as follows:

$$-r_{overall} = ykC_{oil,saturated} = k \frac{6}{d_p} \left( \frac{D_{oil,m}}{k} \right)^{1/2} C_{oil,saturated}$$

$$-r_{overall} = (0.002 \text{ min}^{-1}) \frac{6}{0.0055 \text{ cm}} \left( \frac{8.124 \times 10^{-5} \text{ cm}^2/\text{min}}{0.002 \text{ min}^{-1}} \right)^{1/2} \left( 0.014 \frac{\text{mol}}{\text{L}} \right)$$

$$-r_{overall} = 6.156 \times 10^{-3} \frac{\text{mol}}{\text{L} \cdot \text{min}}$$

## **CHAPTER 5**

### **CONCLUSION**

This project is useful as the need of higher production and quality of biodiesel are increased. A kinetic model proposed to predict the TG conversion and is used for the design of the methanolysis reactor for transesterification reaction. This paper is to prove the reaction kinetic model in a biodiesel transesterification by using the diffusion-reaction system. The diffusion-reaction kinetic model developed describes the overall reaction rate based on the plot from oil conversion over reaction time of the methanolysis in two phase mixture. The methanolysis of triglycerides has potential for improving through inclusion of another extension such are temperature effect, the amount of catalyst, type of oil and alcohol to oil molar ratio in order to develop the kinetic model in heterogeneous transesterification for future reference.

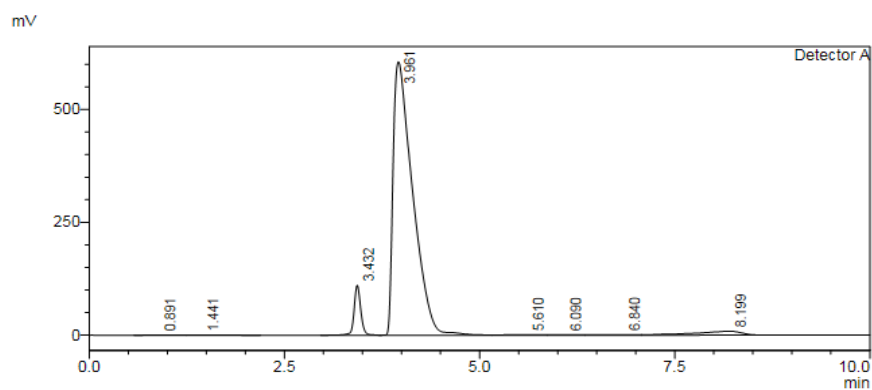


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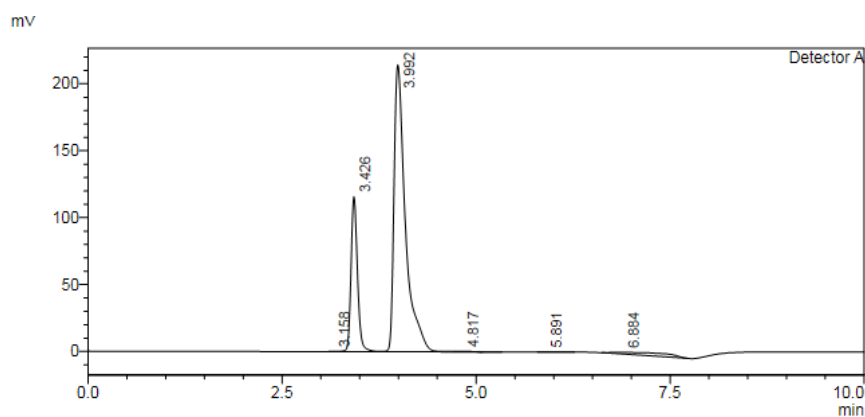
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# APPENDICES

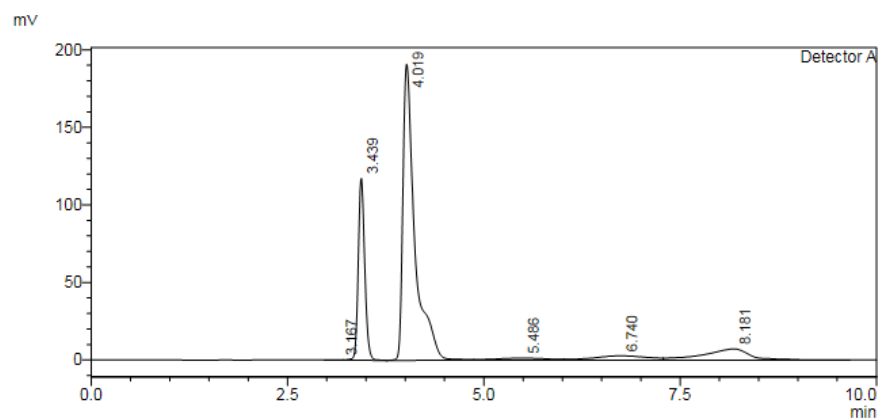
## A. Feedstock HPLC chromatogram



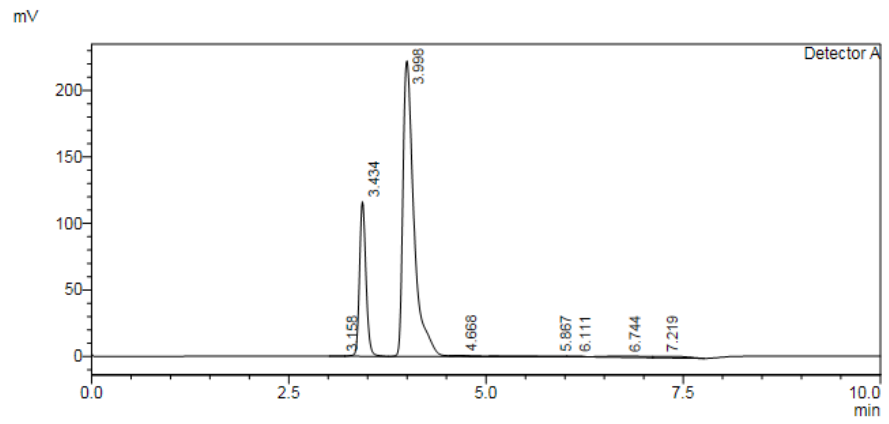
## B. 10 minutes sample HPLC chromatogram



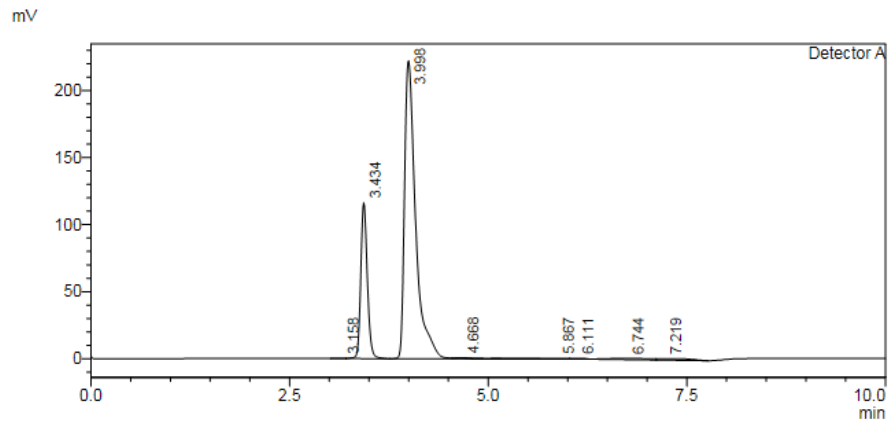
## C. 20 minutes sample HPLC chromatogram



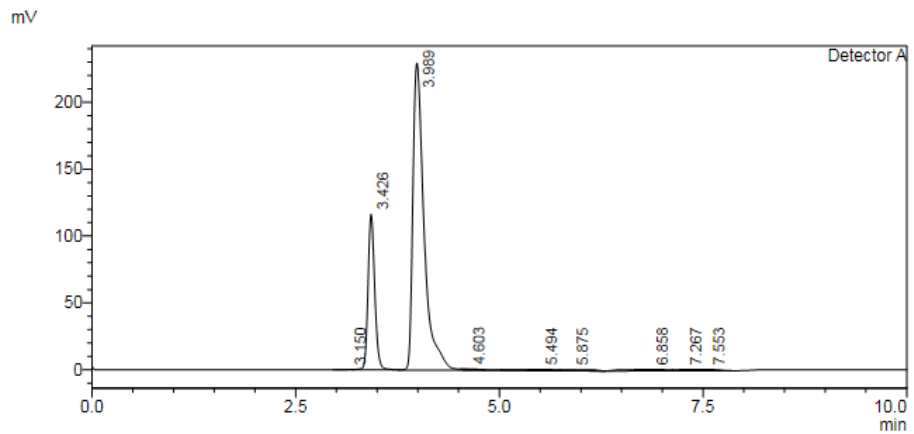
D. 30 minutes sample HPLC chromatogram



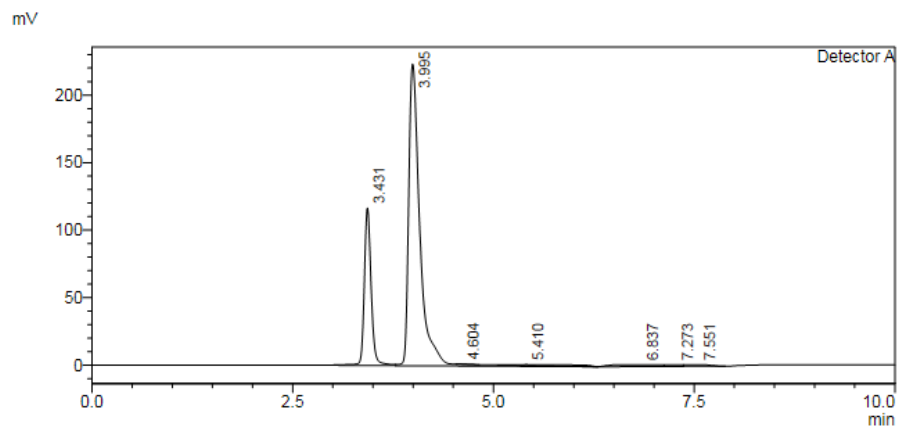
E. 40 minutes sample HPLC chromatogram



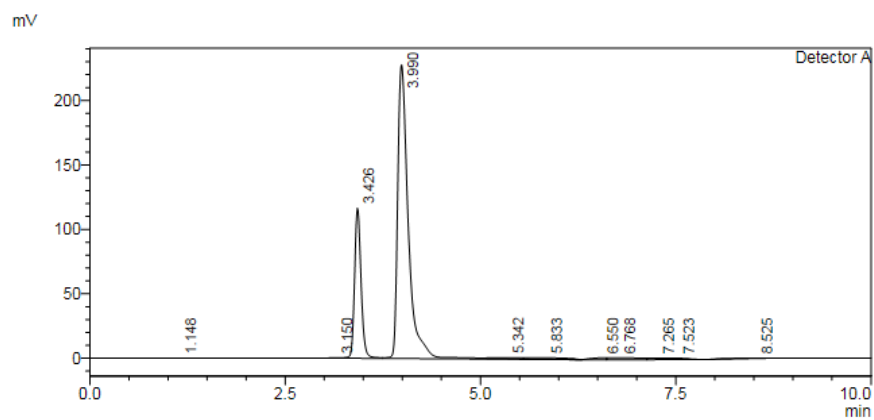
F. 50 minutes sample HPLC chromatogram



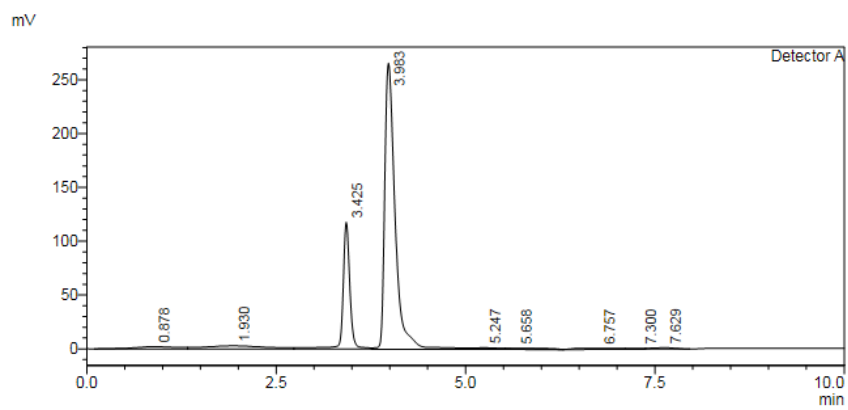
### G. 60 minutes sample HPLC chromatogram



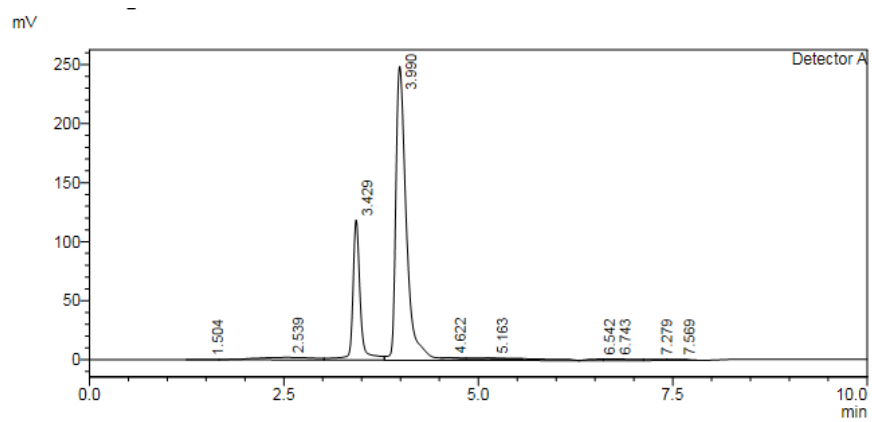
### H. 90 minutes sample HPLC chromatogram



### I. 120 minutes sample HPLC chromatogram



J. 150 minutes sample HPLC chromatogram



K. 180 minutes sample HPLC chromatogram

