

ทรานซ์ไฮสเทอร์ฟิเคชันที่ใช้เบสเป็นตัวเร่งปฏิกิริยาของปาล์มสเตียริน
ในวัฏภาคเดียวเทียมไปเป็นไบโอดีเซล



นาย ชาทรี เกรียงยะกุล

ศูนย์วิทยทรัพยากร จุฬาลงกรณ์มหาวิทยาลัย

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต

สาขาวิชาวิศวกรรมเคมี ภาควิชาวิศวกรรมเคมี

คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย

ปีการศึกษา 2552

ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

PSEUDO SINGLE PHASE BASE-CATALYST TRANSESTERIFICATION
OF PALM STEARIN TO BIODIESEL



Mr. Chatri Kriengyakul

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Engineering Program in Chemical Engineering

Department of Chemical Engineering

Faculty of Engineering


Chulalongkorn University

Academic Year 2009

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Thesis Title PSEUDO SINGLE PHASE BASE-CATALYST
TRANSESTERIFICATION OF PALM STEARIN TO
BIODIESEL
By Mr. Chatri Kriengyakul
Field of Study Chemical Engineering
Thesis Advisor Assistant Professor Montree Wongsri

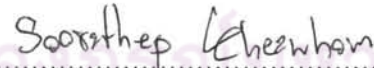
Accepted by the Faculty of Engineering, Chulalongkorn University in Partial
Fulfillment of the Requirements for the Master's Degree


..... Dean of the Faculty of Engineering
(Associate Professor Boonsom Lerdkhironwong, Dr.Ing.)

THESIS COMMITTEE


..... Chairman
(Professor Piyasan Praserttham, Dr.Ing.)


..... Thesis Advisor
(Assistant Professor Montree Wongsri, D.Sc.)


..... Examiner
(Soorathep Kheawhom, Ph.D.)


..... External Examiner
(Prakorn Ramakul, Ph.D.)

ชาติรี เกียรติยะกุล : ทรานส์เอสเทอร์ฟิเคชันที่ใช้เบสเป็นตัวเร่งปฏิกิริยาของปาล์มสเตอริน
 ในวัฏภาคเดียวเทียมไปเป็นไบโอดีเซล (PSEUDO SINGLE PHASE BASE-CATALYST
 TRANSESTERIFICATION OF PALM STEARIN TO BIODIESEL) อ.ที่ปรึกษา
 วิทยานิพนธ์หลัก : ผศ. ดร. มนต์รี วงศ์ศรี, 86หน้า.

งานวิจัยนี้เป็นการศึกษาการผลิตไบโอดีเซลจากไขปาล์มสเตอรินและเมทานอล ด้วย
 ปฏิกิริยาทรานส์เอสเทอร์ฟิเคชันทำในเครื่องปฏิกรณ์แบบกะ โดยใช้อัตราส่วนเมทานอลต่อไข
 ปาล์มสเตอริน 6:1 โพลีเอทิลีนไฮดรอกไซด์ร้อยละ 1 โดยน้ำหนักไขปาล์มสเตอรินเป็นตัวเร่ง
 ปฏิกิริยา และใช้ตัวทำละลายร่วมต่างๆเช่นเตตระไฮโดรฟูแรน, MTBEและไดเอทิลอีเทอร์เพื่อลด
 เวลาการทำปฏิกิริยาและค่าใช้จ่ายในการผลิตไบโอดีเซล พบว่าสภาวะที่สามารถผลิตไบโอดีเซล
 ได้ร้อยละเมทิลเอสเทอร์สูงสุดคือ ที่อุณหภูมิการทำปฏิกิริยา 60 องศาเซลเซียส, ความเร็วรอบกวน
 250 รอบต่อนาที, เวลาการทำปฏิกิริยา 10 นาที และใช้เตตระไฮโดรฟูแรนที่อัตราส่วนโดยโมล
 ระหว่างเตตระไฮโดรฟูแรนต่อเมทานอลเป็น 0.2 เป็นตัวทำละลายร่วม โดยให้ร้อยละเมทิลเอส
 เทอร์สูงสุด 97.6 สำหรับการทดลองที่สภาวะเดียวกันแต่ไม่ใช้ตัวทำละลายร่วมจะได้อัตราส่วนเมทิล
 เอสเทอร์ 93.5 ดังนั้นการใช้ตัวทำละลายร่วมจึงสามารถลดเวลาและพลังงานในการผลิตไบโ
 อดีเซลได้

ศูนย์วิทยทรัพยากร จุฬาลงกรณ์มหาวิทยาลัย

ภาควิชา วิศวกรรมเคมี.....
 สาขาวิชา วิศวกรรมเคมี.....
 ปีการศึกษา 2552

ลายมือชื่อ..... *Carik*
 ลายมือชื่อ อ.ที่ปรึกษาวิทยานิพนธ์หลัก..... *[Signature]*

4971414821 : MAJOR CHEMICAL ENGINEERING

KEYWORDS : METHYL ESTER / BIODIESEL / PALM STEARIN / COSOLVENT

CHATRI KRIENGYAKUL : PSEUDO SINGLE PHASE BASE-CATALYST

TRANSESTERIFICATION OF PALM STEARIN TO BIODIESEL. THESIS

ADVISOR : ASST. PROF. MONTREE WONGSRI, D.Sc., 86 pp.

The objective of this research was to study the production of biodiesel fuel from palm stearin and methanol with transesterification carried out in a batch reactor by using 6:1 methanol/ oil molar ratio, 1wt% potassium hydroxide as homogeneous catalyst and various cosolvent such as tetrahydrofuran (THF), MTBE and dimethyl ether (DEE). Cosolvents were used to reduce the reaction time and costs in biodiesel production. It has been found that the suitable condition for biodiesel production were 60 °C reaction temperature, 250 rpm stirring rate, 10 minutes reaction time with tetrahydrofuran as cosolvent (THF/methanol molar ratio 0.2). At these conditions, the maximum methyl ester content in the product was 97.6% by weight. In the same condition but absence of cosolvent case methyl ester concentration was 93.5 wt%. Therefore addition of cosolvent in the biodiesel production can reduce energy and reaction time.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

Department : Chemical Engineering.....

Student's Signature

Field of Study : Chemical Engineering.....

Advisor's Signature

Academic Year : 2009.....

Chat K

Montree

ACKNOWLEDGEMENTS

I would like to acknowledge Asst. Prof. Dr. Montree Wongsri for his guidance and support throughout the course of my graduate studies and research.

Many thanks are also extended to Oline Co., Ltd. who provide assistance on the provision of feedstock. Also, I am thankful to Scientific and Technology Research Center of Chulalongkorn University for assistance in the provision of experimental instrument.

Also, I am thankful to the advisory committee members, Prof. Dr. Piyasarn Prasertdam , Dr. Surathep Keawhom and Dr. Prakorn Ramakul on their time spending for many useful advices and suggestions.



ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

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CHAPTER I

INTRODUCTION

1.1 Background

Biodiesel is a viable source of energy alternative to petroleum-based diesel fuel in the foreseeable future. As an alternative fuel, biodiesel can be used in neat form or mixed with petroleum-based diesel. Biodiesel, as an alternative fuel, has many merits. It is derived from a renewable, domestic resource, thereby relieving reliance on petroleum fuel imports. It is biodegradable and non-toxic. Compared to petroleum-based diesel, biodiesel has a more favourable combustion emission profile, such as low emissions of carbon monoxide, particulate matter and unburned hydrocarbons. Carbon dioxide produced by combustion of biodiesel can be recycled by photosynthesis, thereby minimizing the impact of biodiesel combustion on the greenhouse effect. Biodiesel has a relatively high flash point (150 °C), which makes it less volatile and safer to transport or handle than petroleum diesel. It provides lubricating properties that can reduce engine wear and extend engine life. In brief, these merits of biodiesel make it a good alternative to petroleum based fuel and have led to its use in many countries, especially in environmentally sensitive areas.

Thailand development strategy on alternative energy from 2008 to 2022 is to achieve 20.4% proportion of alternative energy use in 2022 with 4.50 ml/ day, increasing from the existing 1.4 ml/ day in year 2008, for biodiesel equals to 1,416 ktoe against 13,684 ktoe total renewable energy demand, 112,046 ktoe total energy demand (Table 1.1). In the plan, biodiesel will be produced from oil palm, Jatropha, used vegetable oil and DME. However, most of the oil for biodiesel production will be from palm oil. The current status of palm oil plantation in Thailand (as of 2008) produces 9.264 million tons palm oil from the total producible 2.87 million rai out of the total plantation area of 3.63 million rai.

Table 1.1 Development strategy on alternative energy from 2008 to 2022

Alternative Energy Target of 20.4% in 2022								
Energy Type	Potential	Existing	Year 2008-2011		Year 2012-2016		Year 2017-2022	
	MW	MW	MW	ktoe	MW	ktoe	MW	ktoe
Electricity								
Solar Energy	50,000	32	55	6	95	11	500	56
Wind Energy	1,600	1	150	17	400	45	700	78
Hydropower	700	50	165	43	281	73	324	85
Biomass	4,400	1,597	2,800	1,463	3,235	1,682	3,700	1,933
Biogas	190	29	60	27	90	40	120	54
MSW	320	5	100	60	130	87	160	96
Hydrogen			0	0	0	0	3.5	1
Total		1,714	3,330	1,616	4,231	1,938	5,508	2,303
Thermal (Heat) Energy	ktoe	ktoe		ktoe		ktoe		ktoe
Solar Energy	154	2.3		5		17		34
Biomass	7,400	2,344		3,544		4,915		6,725
Biogas	600	79		470		540		600
MSW	78	1		16		25		35
Total		2,426.3		4,035		5,497		7,394
Biofuels	ml/day	ml/day	ml/day	ktoe	ml/day	ktoe	ml/day	ktoe
Ethanol	3.30	1.00	3.00	816	6.20	1,686	9.00	2,447
Biodiesel	3.30	1.39	3.00	944	3.64	1,145	4.50	1,416
Hydrogen			0.00	0	0.00	0	0.1 m.kg	124
Total		2.39	6.00	1,760	9.84	2,831	13.50	3,987
Total Energy Demand (ktoe)		65,420.00		72,539		88,389		112,046
Total Renewable Energy Demand		3,411.80		7,411		10,266		13,684
Proportion of Renewable Energy Use		5.2		10.2%		11.6%		12.2%
NGV (mmscfd)		91.5	345	3,045	826	7,290	1,035	9,135
Total Alternative Energy Demand (ktoe)				10,456		17,556		22,819
Proportion of Alternative Energy Use				14.4%		19.9%		20.4%

Biodiesel is usually produced by the transesterification of vegetable oil or animal fat with short chain alcohol (Zullaikah et al., 2005). The reaction is commonly carried out in the presence of homogeneous base or acid catalysts. Alkali process can achieve high purity and yield of biodiesel product in a short time but it is very sensitive to the purity of reactants, the starting material (oil or fat) must be dried (moisture level < 0.06%) and free of fatty acid (FFA) (< 0.5%). The presence of minor amount of FFA and moisture in the reaction mixture produces soap, which lower the yield of ester and renders the separation of ester and glycerol by water washing difficult. FFA also consumed the catalyst and reduced catalyst efficiency (Zullaikah et al., 2005). The alternative is the acid catalyzed process, in which acid catalyst such as H_2SO_4 and HCl is used. Acid catalyzed process does have advantages such as reduced purification costs as no soap is produced and is therefore suitable for biodiesel production from oil with high FFA, however the reaction is much slower than the alkali process (Mohamad et al., 2002, Zheng et al., 2006). A two-step transesterification process in which acid catalyzed process is followed by alkali catalyzed process, has been developed to improve the yield of biodiesel for used oil with

high FFA content (Wang et al., 2007). Although shorter reaction time is needed, no recovery of catalyst and high cost of reaction equipment were still the main disadvantages of this process. In the homogeneous transesterification with liquid catalysts, recovery of the catalyst was not possible. Heterogeneous catalyst can therefore be used, nevertheless, there still appear to be some problems with this technique and finding a suitable catalyst that is active, selective, and stable under the process conditions is the major challenge (Kiss et al., 2006).

Alternatively, transesterification using enzyme catalyst such as lipase can also convert oils and fats into methyl esters (Fukuda et al., 2001). The important barrier for using enzyme catalyst is that the price of enzyme is much higher than price of acid or base catalyst result in higher production cost of biodiesel.

However, the alkali catalyzed process with low FFA which is the widely used process for biodiesel production will be selected for the study to evaluate the potential improving in reaction time, reaction temperature by using various suitable cosolvents.

Boocock et al. (Boocock et al., 1996) have developed a novel technique for accelerating the transesterification reaction rate. During its early stages, the transesterification reaction is limited by the low solubility of the alcohol, especially methanol, in the oil. Boocock (Boocock et al., 1998) proposes the addition of a cosolvent to create a single phase and this greatly accelerates the reaction so that it reaches substantial completion in a few minutes. The technique is applicable for use with other alcohols and for acid-catalyzed pretreatment of high free fatty acid feed stocks. The primary concerns with this method are the additional complexity of recovering and recycling the cosolvent although this can be simplified by choosing a cosolvent with a boiling point near that of the alcohol being used. Additional concerns have been raised about the hazard level associated with the cosolvents most commonly proposed, tetrahydrofuran and methyl tertiary butyl ether.

1.2 Objective of the Research

To investigate the appropriate conditions and type of cosolvent required for the production of biodiesel from palm stearin via alkali catalyzed transesterification, with cosolvent, process in a laboratory scale batch reactor by:

1. Studying the effect of various cosolvents to reaction time at various reaction conditions.
2. Proposing a suitable cosolvent with potential improving reaction result for biodiesel production

1.3 Scope of the Research

1. Determine the effect of each cosolvent on conversion and reaction time for alkali catalyzed process of palm stearin – methanol tranesterification.
2. Determine the above effect by varying the reaction temperature and reaction time.
3. The study will be done at atmospheric pressure for the reaction conditions.
4. Detailed Scope
 - a. Transesterification of palm stearin with methanol and KOH in the laboratory scale.
 - b. Determine the effect of cosolvent type (THF, MTBE and DEE) on the conversion for transesterification of palm stearin to biodiesel.
 - c. Determine the effect of reaction time (5 - 15 min.) and temperature (55 – 60 °C) on the conversion for transesterification of palm stearin to biodiesel.
 - d. Determine the effect of stirring speed (200 -550 rpm) on the conversion for transesterification of palm stearin to biodiesel.

1.4 Benefit of the Research

Improvement to achieve and economy of biodiesel production process via the appropriate cosolvent in alkali catalyzed process with the optimum reaction condition which shall result in:-

1. High yield
2. Fast reaction
3. Less energy as the reaction conditions can be kept at atmospheric pressure and low temperature with less reaction time

1.5 Research Methodology

1. Study the previous researches and theory relevant to biodiesel production via transesterification.
2. Set up laboratory equipment and chemicals for the experiment.
3. Analyze chemical properties of raw material.
4. Carry out the experiments.
5. Analyze percentage of produced methyl ester using gas chromatograph.
6. Conclude the experimental result.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

CHAPTER II

THEORY AND LITERATURE REVIEW

2.1 Biodiesel as Alternative Engine Fuel

Biodiesel is defined as fatty acid methyl or ethyl esters from vegetable oil or animal fats when they are used as fuel in diesel engine and heating system. In this context, Biodiesel shows the following general advantages (Vicente et al., 2004)

1. It is biodegradable and non-toxic, assuring safe handling and transport.
2. It does not contain sulfur or aromatic compounds and thus it contributes to the reductions of the diesel engine exhaust emission level.
3. It comes from renewable source such as vegetable oil.
4. It can be produced domestically, reducing a country's dependency on foreign fuel.

2.2 Chemical Foundation of Biodiesel Making

Chemically, biodiesel is a mixture of fatty alkyl esters, or esters formed by fatty acids and an alcohol. Alkyl esters are commonly made from vegetable oil through a chemical reaction called transesterification. Although this process has had long use in making detergents, the resulting compound was first used as a diesel fuel by Austrian researchers around 1980.

Biodiesel is fuel made from fat. It can be produced from vegetable oil or animal fat. Either virgin vegetable oil or waste vegetable oil (WVO) can be used to make quality fuel. Fats are converted to biodiesel through a chemical reaction involving alcohol and a catalyst.

2.2.1 *Chemical Building Block* (Turner T. L., 2005)

It is instructive to think of the chemistry of biodiesel in terms of the building blocks that comprise the larger molecules involved in the biodiesel-making reactions.

Fatty acids are a component of both vegetable oil and biodiesel. In chemical terms, they are carboxylic acids of the form

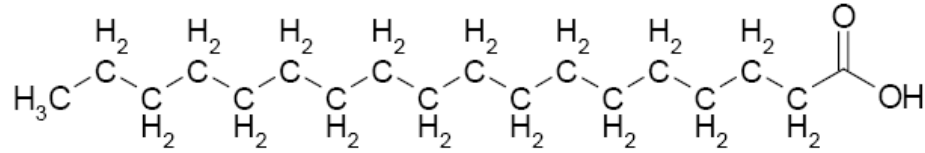


Fig. 2.1 Molecular structure of an idealized fatty acid

Fatty acids which are not bound to some other molecule are known as free fatty acids. When reacted with a base, a fatty acid loses a hydrogen atom to form soap.

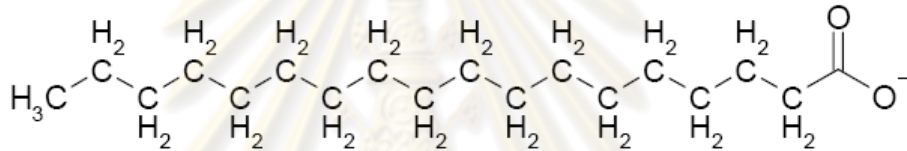


Fig. 2.2 Molecular structure of soap

Chemically, soap is the salt of a fatty acid.

The structures of fatty acids shown in this section are highly idealized. Real fatty acids vary in the number of carbon atoms, and in the number of double bonds. Glycerol, a component of vegetable oil and a by-product of biodiesel production, has the following form:

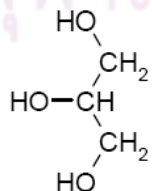


Fig. 2.3 Molecular structure of glycerol

Alcohols are organic compounds of the form $R-OH$, where R is a hydrocarbon. Typical alcohols used in biodiesel-making are methanol, ethanol, 1-propanol, and 1-butanol.

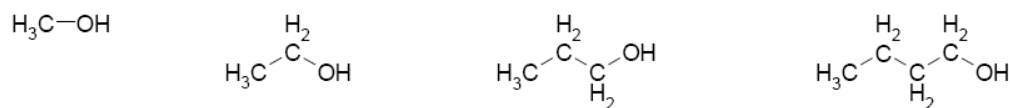


Fig. 2.4 Molecular structure of methanol, ethanol, 1-propanol, and 1-butanol.

Of these, methanol is the most commonly used to make biodiesel. Since ethanol is easily obtained from plant sugars, while methanol is commonly produced from natural gas, using ethanol makes for a more sustainable fuel. Ethanol is harder to use because it forms emulsions easily, making the separation of end products more difficult. This is especially true if the oil source is WVO.

Transesterification is sometimes called alcoholysis, or if by a specific alcohol, by corresponding names such as methanolysis or ethanolysis.

Chemically, biodiesel is a fatty acid alkyl ester:

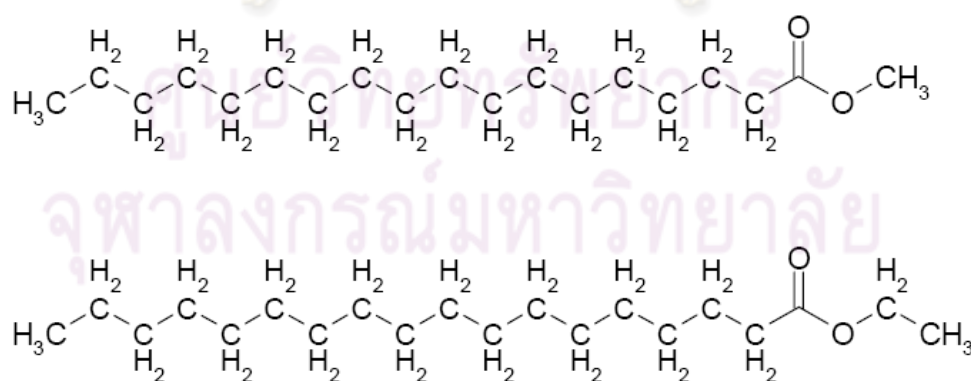


Fig. 2.5 Biodiesel molecules. Above is a methyl ester; below, an ethyl ester.

An ester is a compound of the form:

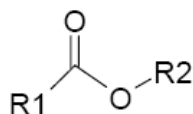


Fig. 2.6 Form of the ester compound.

The biodiesel ester contains a fatty acid chain on one side, and a hydrocarbon called an alkane on the other. Thus, biodiesel is a fatty acid alkyl ester. Usually, the form of the alkane is specified, as in “methyl ester” or “ethyl ester”.

Vegetable oil is a mixture of many compounds, primarily triglycerides and free fatty acids. A triglyceride is a tri-ester of glycerol and three fatty acids:

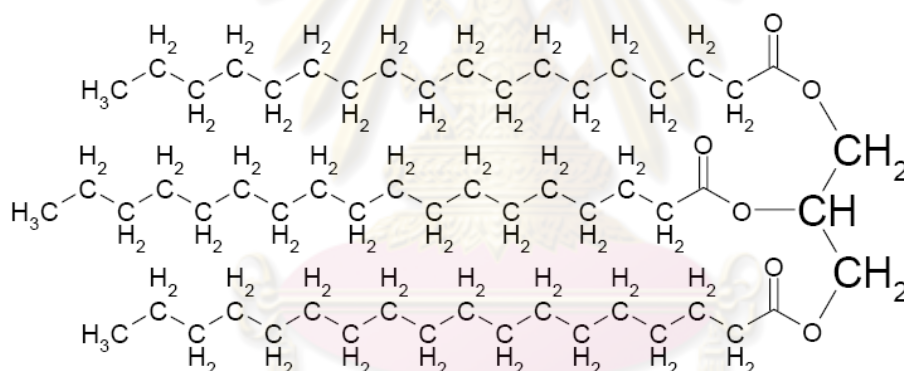


Fig. 2.7 Molecular structure of triglyceride.

Virgin oil contains a low percentage of free fatty acids. Waste vegetable oil contains a higher amount of FFA's because the frying process breaks down triglyceride molecules.

Petroleum diesel and biodiesel are both mixtures of organic compounds. The idealized petroleum molecule is cetane, a pure paraffin. Compared to cetane, alkyl esters are somewhat longer, and more importantly, contain two oxygen atoms.

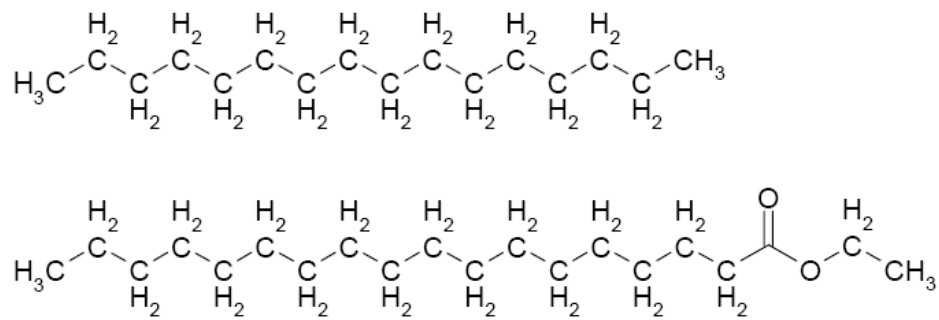


Fig. 2.8 Cetane molecule, above, versus ethyl ester, below

Since combustion is an oxidation reaction, the heating value of cetane, which contains no oxygen atoms, is higher than that of biodiesel. For this reason, diesel engines running biodiesel experience a loss of power on the order of 5%.

2.2.2 Palm Oil

Palm oil can be derived from two parts of the fruit of the oil palm, from the pulp and kernel. Palm kernel oil contains high content of saturated fatty acids (85 – 90%) which is not suitable for the use as food due to healthy problem concerns and normally it will be used as a feedstock for producing soaps, washing powders, personal care products, and biofuel. Crude palm oil or oil from pulp contains saturated fatty acids (palmitic and stearic acid) 50%, unsaturated fatty acid (oleic acid) 40%, vitamin A and E. Crude palm oil is typically in a mixed liquid and solid at room temperature. Using of crude palm oil for biodiesel production can reduce fractionation cost. However, fractionation of crude palm oil yields palm olein, which is typically used as cooking oil, and palm stearin which is normally used as a feedstock producing soap, margarine, etc. When consider their properties, palm kernel oil and palm stearin is suitable to be used as a feedstock for biodiesel as they are not suitable for food like other oils and can alleviate over supply problem.

Table 2.1 and 2.2 show properties of free fatty acids and iodine value and fatty acid composition of typical palm oil.

Table 2.1 Properties of free fatty acids (Knothe, 1996)

Fatty acid	Molecular Weight	Melting point (°C)	Boiling point (°C)	Heat of combustion (kg-cal/mol)
Caprylic acid (C8:0)	144.2	16.5	240	-
Capric acid (C10:0)	172.3	31.6	271	1453.1
Lauric acid (C12:0)	200.3	44.8	130	1763.3
Myristic acid (C14:0)	228.4	54.4	149	2073.9
Palmitic acid (C16:0)	256.4	62.9	167	2384.7
Stearic acid (C18:0)	284.5	70.1	184	2696.1
Oleic acid (C18:1)	282.5	16.3	286	2657.4
Linoleic acid (C18:2)	280.4	-5.0	229	-
Linolenic acid (C18:3)	278.4	-11.0	231	-
Erucic acid (C22:1)	338.6	33.7	265	-

Table 2.2 Iodine value and fatty acid composition of typical palm oil (Phisamai, 2001)

Oil	Iodine value	Fatty Acid Composition (%wt)						
		12:0	14:0	16:0	18:0	18:1	18:2	18:3
Crude palm oil	14.2-21	ND-0.5	0.5-2	39.3-47.5	3.5-6	36-44	9-12	ND-0.5
Palm olein	>56	0.1-0.5	0.5-1.5	38-43.5	3.5-5	39.8-46	10-13.5	ND-0.6
Palm stearin	<48	0.1-0.5	1.0-2.0	48-74	3.9-6	15.5-36	3.0-10	0.5
Palm kernel oil	50-55	45-55	14-18	6.5-10	1-3	12-19	1.0-3.5	ND-0.2

2.3 The Production of Biodiesel

The most common way to produce biodiesel is by transesterification, which refers to a catalyzed chemical reaction involving vegetable oil and an alcohol to yield fatty acid alkyl esters (i.e., biodiesel) and glycerol (Fig. 2.9). Triacylglycerols (triglycerides), as the main component of vegetable oil, consist of three long chain fatty acids esterified to a glycerol backbone. When triacylglycerols react with an alcohol (e.g., methanol), the three fatty acid chains are released from the glycerol skeleton and combine with the alcohol to yield fatty acid alkyl esters (e.g., fatty acid methyl esters or FAME). Glycerol is

produced as a by-product. Methanol is the most commonly used alcohol because of its low cost and is the alcohol of choice in the processes developed in this report. In general, a large excess of methanol is used to shift the equilibrium far to the right (Fig. 2.9).

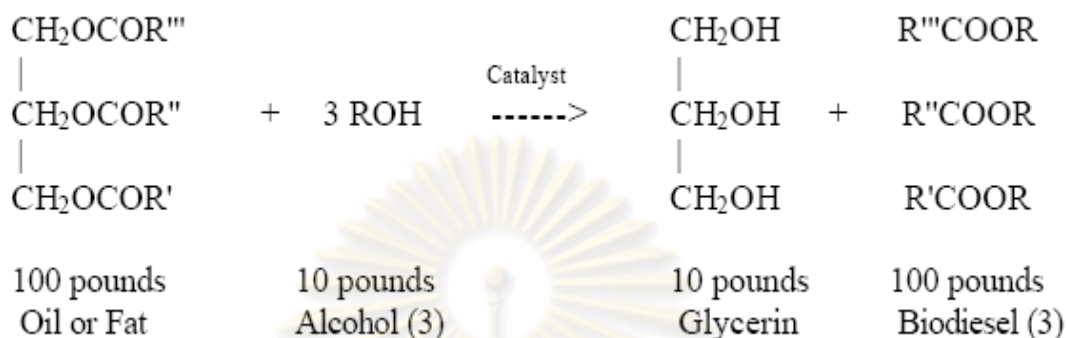


Fig. 2.9 A schematic representation of the transesterification of triglycerides (vegetable oil) with methanol to produce fatty acid methyl esters (biodiesel).

Transesterification reactions can be alkali-catalyzed, acid-catalyzed or enzyme-catalyzed. The first two types have received the greatest attention. As for the enzyme-catalyzed system, it requires a much longer reaction time than the other two systems. To date it has only been carried out on the laboratory scale.

Most current biodiesel research concentrates on the alkali-catalyzed technology carried out on a bench scale. Apart from the bench-scale research, the alkali-catalyzed process for biodiesel production has been applied industrially. A commercial continuous alkali-catalyzed transesterification process to produce methyl esters on the industrial scale under high pressure (90 bar) and at high temperature (240 °C) was demonstrated by Kreutzer (1984). However, high energy consumption, a significant increase in equipment cost and process safety issues related to, for example, high pressure and high temperature, could make this process prohibitive. Krawczyk (1996) presented a flow diagram for producing biodiesel via transesterification on the industrial scale. The process mainly consisted of a transesterification reactor, a methanol/glycerol distillation column and a methyl ester distillation column. Aside from the flowsheet, no detailed description of the process was provided. A continuous deglycerolization process to produce biodiesel from refined rapeseed oil by alkali-catalyzed transesterification at

ambient pressure and 65–70 °C was introduced by Connemann and Fischer in 1998. They noted successful commercial applications of this process in Europe. One limitation to the alkali-catalyzed process is its sensitivity to the purity of reactants; the alkali-catalyzed system is very sensitive to both water and free fatty acids. The presence of water may cause ester saponification under alkaline conditions. Also, free fatty acids can react with an alkali catalyst to produce soaps and water. Saponification not only consumes the alkali catalyst, but also the resulting soaps can cause the formation of emulsions. Emulsion formation creates difficulties in downstream recovery and purification of the biodiesel. Thus, dehydrated vegetable oil with less than 0.5 wt.% free fatty acids, an anhydrous alkali catalyst and anhydrous alcohol are necessary for commercially viable alkali-catalyzed systems. This requirement is likely to be a significant limitation to the use of waste cooking oil as a low-cost feedstock. Usually the level of free fatty acids in waste cooking oil is greater than 2 wt.%. In 1986 Lepper and Friesenhagen recommended a pretreatment step to reduce the free fatty acid content via an esterification reaction with methanol in the presence of sulfuric acid catalyst. Glycerine was employed as a liquid entraining agent to purify the refined oil. After such a treatment, the oil phase, having a low level of free fatty acids (less than 0.5 wt.%), was subjected to the alkali-catalyzed transesterification.

Acid-catalyzed system, despite its insensitivity to free fatty acids in the feedstock, acid-catalyzed transesterification has been largely ignored mainly because of its relatively slower reaction rate.

A simple production flow chart along with a short explanation of the steps involved in the process is shown below.

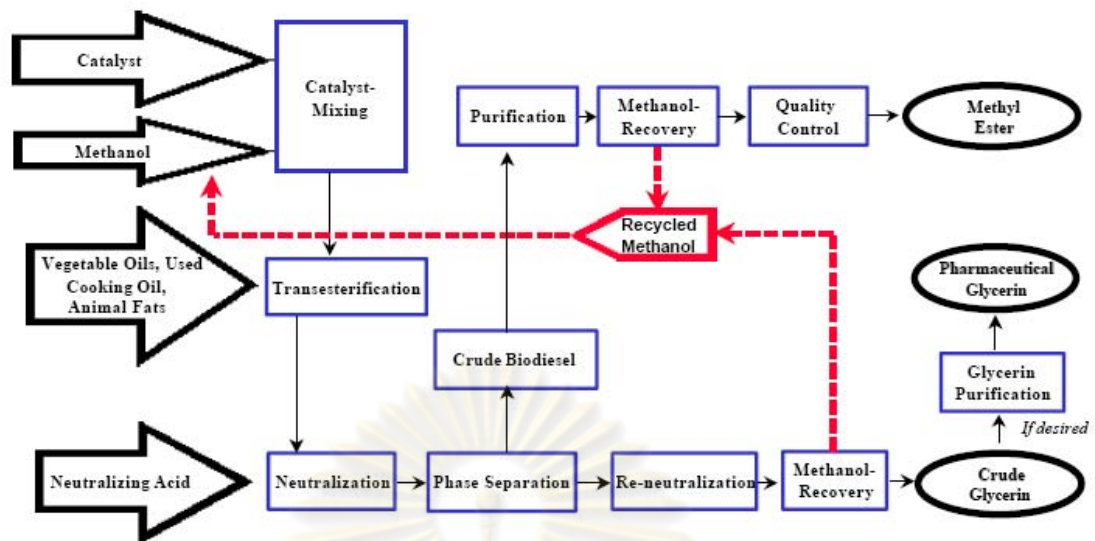


Fig. 2.10 Biodiesel production process

The base catalyzed production of biodiesel generally occurs using the following steps:

Mixing of alcohol and catalyst. The catalyst is typically sodium hydroxide (caustic soda) or potassium hydroxide (potash). It is dissolved in the alcohol using a standard agitator or mixer.

Reaction. The alcohol/catalyst mix is then charged into a closed reaction vessel and the oil or fat is added. The system from here on is totally closed to the atmosphere to prevent the loss of alcohol. The reaction mix is kept just above the boiling point of the alcohol (around 160 °F or 82 °C) to speed up the reaction and the reaction takes place. Recommended reaction time varies from 1 to 8 hours, and some systems recommend the reaction take place at room temperature. Excess alcohol is normally used to ensure total conversion of the fat or oil to its esters.

Care must be taken to monitor the amount of water and free fatty acids in the incoming oil or fat. If the free fatty acid level or water level is too high it may cause problems with soap formation and the separation of the glycerin by-product downstream.

Separation. Once the reaction is complete, two major products exist: glycerin and biodiesel. Each has a substantial amount of the excess methanol that was used in the reaction. The reacted mixture is sometimes neutralized at this step if needed. The glycerin phase is much more denser than biodiesel phase and the two can be gravity separated with glycerin simply drawn off the bottom of the settling vessel. In some cases, a centrifuge is used to separate the two materials faster.

Alcohol Removal. Once the glycerin and biodiesel phases have been separated, the excess alcohol in each phase is removed with a flash evaporation process or by distillation. In others systems, the alcohol is removed and the mixture neutralized before the glycerin and esters have been separated. In either case, the alcohol is recovered using distillation equipment and is re-used. Care must be taken to ensure no water accumulates in the recovered alcohol stream.

Glycerin Neutralization. The glycerin by-product contains unused catalyst and soaps that are neutralized with an acid and sent to storage as crude glycerin. In some cases the salt formed during this phase is recovered for use as fertilizer. In most cases the salt is left in the glycerin. Water and alcohol are removed to produce 80-88% pure glycerin that is ready to be sold as crude glycerin. In more sophisticated operations, the glycerin is distilled to 99% or higher purity and sold into the cosmetic and pharmaceutical markets.

Methyl Ester Wash. Once separated from the glycerin, the biodiesel is sometimes purified by washing gently with warm water to remove residual catalyst or soaps, dried, and sent to storage. In some processes this step is unnecessary. This is normally the end of the production process resulting in a clear amber-yellow liquid with a viscosity similar to petrodiesel. In some systems the biodiesel is distilled in an additional step to remove small amounts of color bodies to produce a colorless biodiesel.

In the case of using waste vegetable oil (yellow grease) as a feedstock, free fatty acids (FFA's) may pose a problem. A free fatty acid is one that has already separated from the glycerol molecule. This is usually the result of the oil breaking down after many cycles of use. FFA's create 3 major problems.

- More catalyst will need to be used leading to higher cost
- Soap (fatty acid salt) is formed, making washing the finished product more difficult.
- Water is formed which will retard the main reaction
- The FFA's are not converted into fuel, reducing the yield

Fig. 2.11 shows the reaction of FFA's and the catalyst NaOH.

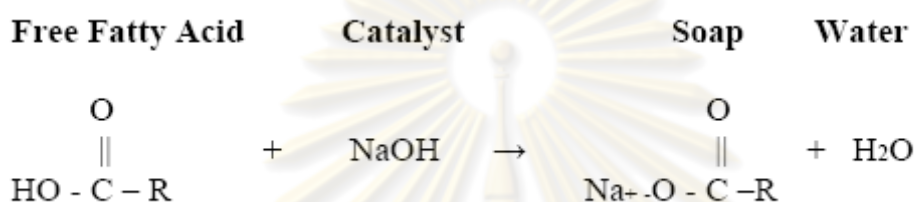


Fig. 2.11 Formation of soap

When the oil has less than 2.5% FFA, the problems listed previously are negligible by using the single step (transesterification) only. Others have reported good results up to 4% FFA.

2.4 Variable Affecting Transesterification and Esterification

The process of transesterification is affected by various factors depending upon the reaction condition used. The effects of these factors are described below (Meher et al., 2006).

1. Effect of free fatty acid and moisture

The free fatty acid and moisture content are key parameters for determining the viability of the vegetable oil transesterification process. To carry the base catalyzed reaction to completion; a free fatty acid (FFA) value lower than 3% is needed. The higher the acidity of the oil, smaller is the conversion efficiency. Both, excess as well as insufficient amount of catalyst may cause soap formation.

Ma et al. (Ma et al., 1999) studied the transesterification of beef tallow catalyzed by NaOH in presence of free fatty acids and water. Without adding FFA and water, the

apparent yield of beef tallow methyl esters (BTME) was highest. When 0.6% of FFA was added, the apparent yield of BTME reached the lowest, less than 5%, with any level of water added.

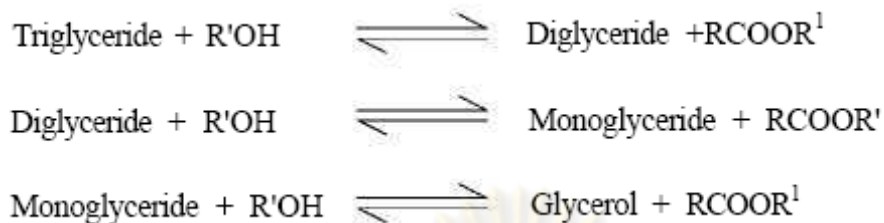
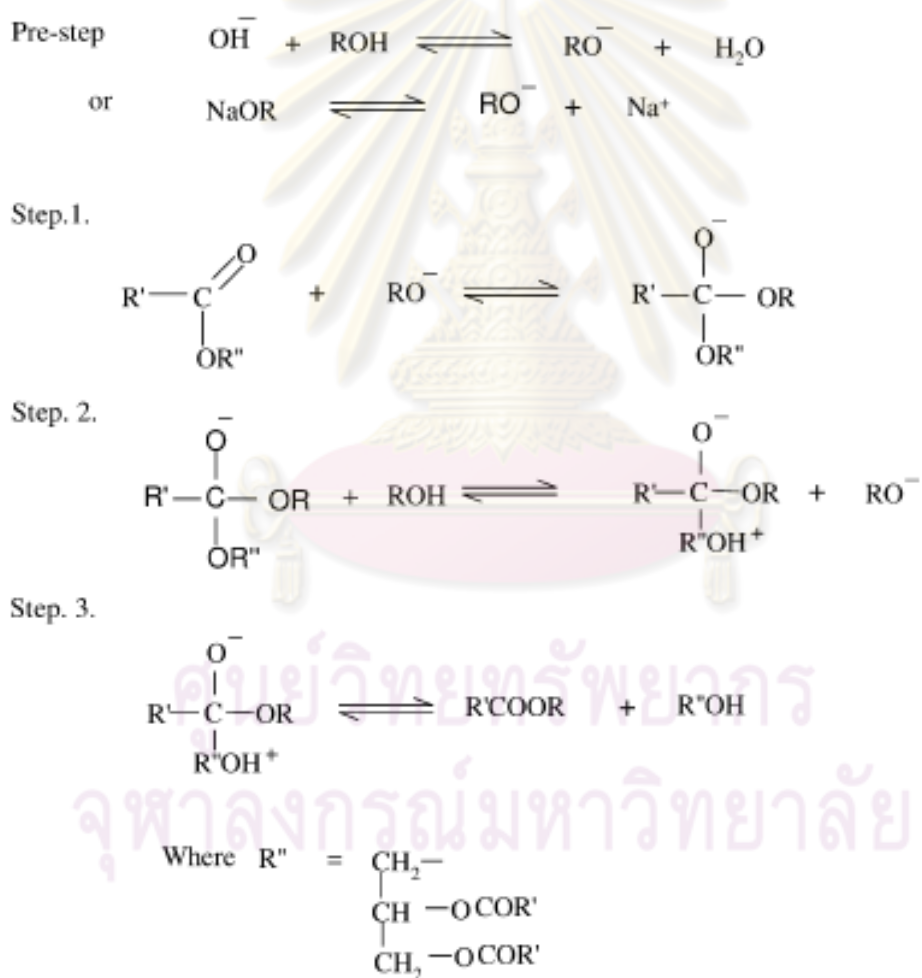


Fig. 2.12 General equation for transesterification of triglycerides.



R' = Carbon chain of fatty acid

R = Alkyl group of alcohol

Fig. 2.13 Mechanism of base catalyzed transesterification.

The products were solid at room temperature, similar to the original beef tallow. When 0.9% of water was added, without addition of FFA, the apparent yield was about 17%. If the low qualities of beef tallow or vegetable oil with high FFA are used to make biodiesel fuel, they must be refined by saponification using NaOH solution to remove free fatty acids. Conversely, the acid catalyzed process can also be used for esterification of these free fatty acids.

The starting materials used for base catalyzed alcoholysis should meet certain specifications. The triglycerides should have lower acid value and all material should be substantially anhydrous. The addition of more sodium hydroxide catalyst compensates for higher acidity, but the resulting soap causes an increase in viscosity or formation of gels that interferes in the reaction as well as with separation of glycerol. When the reaction conditions do not meet the above requirements, ester yields are significantly reduced. The methoxide and hydroxide of sodium or potassium should be maintained in anhydrous state.

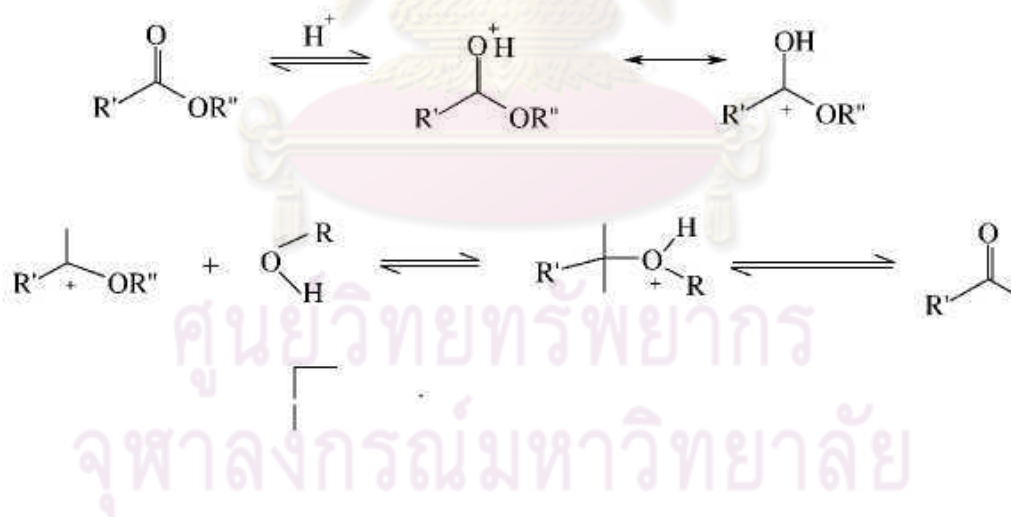


Fig. 2.14 Mechanism of acid catalyzed transesterification.

Prolonged contact with air will diminish the effectiveness of these catalysts through interaction with moisture and carbon dioxide.

Most of the biodiesel is currently made from edible oils by using methanol and alkaline catalyst. However, there are large amounts of low cost oils and fats that could

be converted to biodiesel. The problems with processing these low cost oils and fats are that they often contain large amounts of free fatty acids that cannot be converted to biodiesel using alkaline catalyst. Therefore, two-step esterification process is required for these feed stocks. Initially the FFA of these can be converted to fatty acid methyl esters by an acid catalyzed pretreatment and in the second step transesterification is completed by using alkaline catalyst to complete the reaction. Initial process development was performed with synthetic mixture containing 20 and 40% free fatty acid prepared by using palmitic acid. Process parameters such as molar ratio of alcohol to oil, type of alcohol, amount of acid catalyst, reaction time, free fatty acid level were investigated to determine the best strategy for converting the free fatty acids to usable esters. The work showed that the acid level of the high free fatty acids feed stocks could be reduced to less than 1 % with a two step pretreatment reaction. The reaction mixture was allowed to settle between steps so that the water containing phase could be removed. The two-step pretreatment reaction was demonstrated with actual feedstocks, including yellow grease with 12% free fatty acid and brown grease with 33% free fatty acids. After reducing the acid levels of these feedstocks to less than 1%, the transesterification reaction was completed with an alkaline catalyst to produce fuel grade biodiesel.

Investigation of the negative influence of base catalyzed transesterification of triglycerides containing substantial amount of free fatty acid has been carried out by Turck R. in 2002. Free fatty acids react with the basic catalyst added for the reaction and give rise to soap, as a result of which, one part of the catalyst is neutralized and is therefore no longer available for transesterification. These high FFA content oils/fats are processed with an immiscible basic glycerol phase so as to neutralize the free fatty acids and cause them to pass over into the glycerol phase by means of monovalent alcohols. The triglycerides are subjected to transesterification, using a base as catalyst, to form fatty acid alkyl esters, characterized in that after its separation, the basic glycerol phase produced during transesterification of the triglycerides is used for processing the oils/fats for removal of free fatty acids. The minimum amount of catalyst required for this process was calculated, relative to 1000 g of the oil to be processed, as a function of the acid value and the mean molar mass of the oil/fat.

2. Catalyst type and concentration

Catalysts used for the transesterification of triglycerides are classified as alkali, acid, enzyme or heterogeneous catalysts, among which alkali catalysts like sodium hydroxide, sodium methoxide, potassium hydroxide, potassium methoxide are more effective. If the oil has high free fatty acid content and more water, acid catalyzed transesterification is suitable. The acids could be sulfuric acid, phosphoric acid, hydrochloric acid or organic sulfonic acid. Methanolysis of beef tallow was studied with catalysts NaOH and NaOMe.

Comparing the two catalysts, NaOH was significantly better than NaOMe. The catalysts NaOH and NaOMe reached their maximum activity at 0.3 and 0.5% w/w of the beef tallow, respectively. Sodium methoxide causes formation of several by-products mainly sodium salts, which are to be treated as waste. In addition, high quality oil is required with this catalyst. This was different from the previous reports in which ester conversion at the 6:1 molar ratio of alcohol/oil for 1% NaOH and 0.5% NaOMe were almost the same after 60 min. Part of the difference may be attributed to the differences in the reaction system used.

As a catalyst in the process of alkaline methanolysis, mostly sodium hydroxide or potassium hydroxide have been used, both in concentration from 0.4 to 2% w/w of oil.

Refined and crude oils with 1% either sodium hydroxide or potassium hydroxide catalyst resulted successful conversion. Methanolysis of soybean oil with the catalyst 1% potassium hydroxide has given the best yields and viscosities of the esters.

Attempts have been made to use basic alkaline-earth metal compounds in the transesterification of rapeseed oil for production of fatty acid methyl esters. The reaction proceeds if methoxide ions are present in the reaction medium. The alkaline-earth metal hydroxides, alkoxides and oxides catalyzed reaction proceeds slowly as the reaction mixture constitutes a three-phase system oil-methanol-catalyst, which for diffusion reason inhibits the reaction. The catalytic activity of magnesium oxide, calcium hydroxide, calcium oxide, calcium methoxide, barium hydroxide, and for comparison, sodium hydroxide during the transesterification of rapeseed oil was investigated.

Sodium hydroxide exhibited the highest catalytic activity in this process. The degree to which the substrates were reacted reached 85% after 30 min of the process and 95% after 1.5 h, which represented a close value to the equilibrium. Barium hydroxide was slightly less active with a conversion of 75% after 30 min. Calcium methoxide was medially active.

The degree to which the substrates were reacted was 55% after 30 min. Eighty percents after 1 h and state of reaction equilibrium (93%) was reached after 2.5 h. The rate of reaction was slowest when catalyzed by CaO. Magnesium oxide and calcium hydroxide showed no catalytic activity in rapeseed oil methanolysis.

Acid catalyzed transesterification was studied with waste vegetable oil. The reaction was conducted at four different catalyst concentrations, 0.5, 1.0, 1.5 and 2.25 M HCl in presence of 100% excess alcohol and the result was compared with 2.25 M H_2SO_4 and the decrease in viscosity was observed. H_2SO_4 has superior catalytic activity in the range of 1.5–2.25 M concentration.

Although chemical transesterification using an alkaline catalysis process gives high conversion levels of triglycerides to their corresponding methyl esters in short reaction times, the reaction has several drawbacks: it is energy intensive, recovery of glycerol is difficult, the acidic or alkaline catalyst has to be removed from the product, alkaline waste water require treatment, and free fatty acid and water interfere the reaction. Enzymatic catalysts like lipases are able to effectively catalyze the transesterification of triglycerides in either aqueous or non-aqueous systems, which can overcome the problems mentioned above. In particular, the by-products, glycerol can be easily removed without any complex process, and also that free fatty acids contained in waste oils and fats can be completely converted to alkyl esters. On the other hand, in general the production cost of a lipase catalyst is significantly greater than that of an alkaline one.

3. *Molar ratio of alcohol to oil and type of alcohol*

One of the most important variables affecting the yield of ester is the molar ratio of alcohol to triglyceride. The stoichiometric ratio for transesterification requires three moles of alcohol and one mole of triglyceride to yield three moles of fatty acid alkyl

esters and one mole of glycerol. However, transesterification is an equilibrium reaction in which a large excess of alcohol is required to drive the reaction to the right. For maximum conversion to the ester, a molar ratio of 6:1 should be used. The molar ratio has no effect on acid, peroxide, saponification and iodine value of methyl esters. However, the high molar ratio of alcohol to vegetable oil interferes with the separation of glycerin because there is an increase in solubility. When glycerin remains in solution, it helps drive the equilibrium to back to the left, lowering the yield of esters. The transesterification of Cynara oil with ethanol was studied at molar ratios between 3:1 and 15:1. The ester yield increased as the molar ratio increased up to a value of 12:1. The best results were for molar ratios between 9:1 and 12:1. For molar ratios less than 6:1, the reaction was incomplete. For a molar ratio of 15:1 the separation of glycerin is difficult and the apparent yield of esters decreased because a part of the glycerol remains in the biodiesel phase. Therefore, molar ratio 9:1 seems to be the most appropriate.

The base catalyzed formation of ethyl ester is difficult compared to the formation of methyl esters. Specifically the formation of stable emulsion during ethanolysis is a problem. Methanol and ethanol are not miscible with triglycerides at ambient temperature, and the reaction mixtures are usually mechanically stirred to enhance mass transfer. During the course of reaction, emulsions usually form. In the case of methanolysis, these emulsions quickly and easily break down to form a lower glycerol rich layer and upper methyl ester rich layer. In ethanolysis, these emulsions are more stable and severely complicate the separation and purification of esters. The emulsions are caused in part by formation of the intermediates monoglycerides and diglycerides, which have both polar hydroxyl groups and non-polar hydrocarbon chains. These intermediates are strong surface active agents. In the process of alcoholysis, the catalyst, either sodium hydroxide or potassium hydroxide is dissolved in polar alcohol phase, in which triglycerides must transfer in order to react. The reaction is initially mass-transfer controlled and does not conform to expected homogeneous kinetics. When the concentrations of these intermediates reach a critical level, emulsions form. The larger non-polar group in ethanol, relative to methanol, is assumed to be the critical factor in stabilizing the emulsions. However, the concentration of mono- and di-

glycerides is very low, then the emulsions become unstable. This emphasizes the necessity for the reaction to be as complete as possible, thereby reducing the concentrations of mono- and di-glycerides.

4. *Effect of reaction time and temperature*

The conversion rate increases with reaction time. Freedman et al. transesterified peanut, cotton-seed, sunflower and soybean oil under the condition of methanol–oil molar ratio 6:1, 0.5% sodium methoxide catalyst and 60 °C. An approximate yield of 80% was observed after 1 min for soybean and sunflower oils. After 1 h, the conversion was almost the same for all four oils (93–98%). Ma et al. studied the effect of reaction time on transesterification of beef tallow with methanol. The reaction was very slow during the first minute due to mixing and dispersion of methanol into beef tallow. From one to 5 min, the reaction proceeds very fast. The production of beef tallow methyl esters reached the maximum value at about 15 min.

Transesterification can occur at different temperatures, depending on the oil used. For the transesterification of refined oil with methanol (6:1) and 1% NaOH, the reaction was studied with three different temperatures. After 0.1 h, ester yields were 94, 87 and 64% for 60, 45 and 32 °C, respectively. After 1 h, ester formation was identical for 60 and 45 °C runs and only slightly lower for the 32 °C run. Temperature clearly influenced the reaction rate and yield of esters.

5. *Mixing intensity*

Mixing is very important in the transesterification reaction, as oils or fats are immiscible with sodium hydroxide–methanol solution. Once the two phases are mixed and the reaction is started, stirring is no longer needed. Initially the effect of mixing on transesterification of beef tallow was study by Ma et al. No reaction was observed without mixing and when NaOH–MeOH was added to the melted beef tallow in the reactor while stirring, stirring speed was insignificant. Reaction time was the controlling factor in determining the yield of methyl esters. This suggested that the stirring speeds investigated exceeded the threshold requirement of mixing.

6. *Effect of using organic cosolvent*

The methoxide base catalyzed methanolysis of soybean oil at 40 °C (methanol–oil molar ratio 6:1) shows that to form methyl esters proceeds approximately more slowly than butanolysis at 30 °C. This is interpreted to be the result of a two phase reaction in which methanolysis occurs only in the methanol phase. Low oil concentration in methanol causes the slow reaction rate; a slow dissolving rate of the oil in methanol causes an initiation period. Intermediate mono- and di-glycerides preferentially remain in the methanol, and react further, thus explaining the deviation from second order kinetics. The same explanations apply for hydroxide ion catalyzed methanolysis.

In order to conduct the reaction in a single phase, cosolvents like tetrahydrofuran, 1,4-dioxane and diethyl ether were tested. Although, there are other cosolvents, initial study was conducted with tetrahydrofuran. At the 6:1 methanol–oil molar ratio the addition of 1.25 volume of tetrahydrofuran per volume of methanol produces an oil dominant one phase system in which methanolysis speeds up dramatically and occurs as fast as butanolysis. In particular, THF is chosen because its boiling point of 67 °C is only two degrees higher than that of methanol. Therefore at the end of the reaction the unreacted methanol and THF can be co-distilled and recycled.

Using tetrahydrofuran, transesterification of soybean oil was carried out with methanol at different concentrations of sodium hydroxide. The ester contents after 1 min for 1.1, 1.3, 1.4 and 2.0% sodium hydroxide were 82.5, 85, 87 and 96.2%, respectively. Results indicated that the hydroxide concentration could be increased up to 1.3 wt%, resulting in 95% methyl ester after 15 min. Similarly for transesterification of coconut oil using THF/MeOH volume ratio 0.87 with 1% NaOH catalyst, the conversion was 99% in 1 min.

A single-phase process for the esterification of a mixture of fatty acids and triglycerides were investigated. The process comprises forming a single-phase solution of fatty acids and triglyceride in an alcohol selected from methanol and ethanol, the ratio of said alcohol to triglyceride being 15:1–35:1. The solution further comprises a cosolvent in an amount to form the single phase. In a first step, an acid catalyst for the esterification of fatty acid is added. After a period of time, the acid catalyst is neutralized

and a base catalyst for the transesterification of triglycerides is added. After a further period of time, esters are separated from the solution.

An improved process was investigated for methanolysis and ethanolysis of fatty acid glycerides such as those found in naturally occurring fats and oils derived from plant and animals. The processes comprise solubilizing oil or fat in methanol or ethanol by addition of a cosolvent in order to form a one-phase reaction mixture, and adding an esterification catalyst. The processes proceed quickly, usually in less than 20 min, at ambient temperatures, atmospheric pressure and without agitation. The co-solvent increases the rate of reaction by making the oil soluble in methanol, thus increasing contact of the reactants. The lower alkyl fatty acid monoesters produced by the process can be used as biofuels and are suitable as diesel fuel replacements or additives.

2.5 Biodiesel Production Using Cosolvent

A number of studies on preparation of biodiesel from vegetable oils have been reported using a variety of oils, alcohols, different catalysts, and reaction conditions. For alkali catalyzed process, Aracil et al. (Aracil et al., 2005) used different basic catalysts (sodium methoxide, potassium methoxide, sodium hydroxide and potassium hydroxide) for transesterification of sunflower oil. The biodiesel purity near 100 wt.% was obtained for all catalysts. However, biodiesel yields near 100 wt.% were only obtained with the methoxide catalysts. Although all the transesterification reactions were quite rapid and the product achieved nearly 100% methyl ester concentrations, the reactions using sodium hydroxide turned out the fastest. Another example on alkali process is that of Meher et al., (2006), in which the transesterification of karanja oil with methanol was carried out using alkaline catalyst in a batch type reactor. At 65 °C, with a molar ratio of 1:6 of the karanja oil to methanol, using KOH as catalyst, the 97% conversion to methyl esters was obtained after 3 hrs. Although high purity and yield of biodiesel can be achieved in a short time with the alkali process. However, it is very sensitive to the purity of reactants. For example, the starting material (oil or fat) must be dried (moisture level <0.06%) and free of free fatty acid (FFA) (<0.5%). The presence of minor amount of FFA and moisture in the reaction mixture produces soap, which lower the yield of ester and

renders the separation of ester and glycerol by water washing difficult. FFA also consumed the catalyst and reduced catalyst efficiency (Zullaikah et al., 2005).

Boocock et al. (Boocock et al., 2004) have developed a novel technique for accelerating the transesterification reaction rate. During its early stages, the transesterification reaction is limited by the low solubility of the alcohol, especially methanol, in the oil. Boocock proposed the addition of a cosolvent to create a single phase, and this greatly accelerates the reaction so that it reaches substantial completion in a few minutes. The technique is applicable for use with other alcohols and for acid-catalyzed pretreatment of high free fatty acid feed stocks.

2.6 Cosolvent Selection

The first consideration of cosolvents for the transesterification shall be inert to the reaction and have a boiling point lower and near to the boiling point of methanol to simplify the solvent recycle by flashing them out of the product at the boiling point of methanol.

The second consideration is that the selected cosolvents shall be soluble in both methanol and triglyceride. Due to methanol is a polar substance while triglyceride which contains a long chain alkyl (hydrocarbon) group is non-polar, hence they are immiscible. So, the selected cosolvents which can be solute in both methanol and triglyceride shall have both polar and non-polar parts in their molecules.

Table 2.3 summarized the properties of cosolvents selected for the study.

Fig. 2.15 shows molecular structure of THF, MTBE and DEE from the left to right respectively. It can be seen that all these cosolvents have both polar part (at hydrogen atom) and non-polar part (at Oxygen atom). MTBE and diethyl ether have lower boiling point than the melting point of palm stearin which requires higher reaction temperature. However, these cosolvents were selected due to they have either lower or close boiling point to that of methanol, soluble in both methanol and triglyceride and the reaction will be carried out in a closed reactor. The boiling point of the solution will also be raised by the high content of both methanol and the non volatile triglyceride.

Table 2.3 Properties of selected cosolvent

Chemical	Formula	Molecular Wt	Density (g/ml)	Solubility in Water (20 °C)	Boiling Point (°C)
THF	C ₄ H ₈ O	72.11	0.88	Soluble	67
MTBE	C ₅ H ₁₂ O	85.15	0.74	26 g/l	55.2
DEE	C ₄ H ₁₀ O	74.12	0.71	69 g/l	34.6

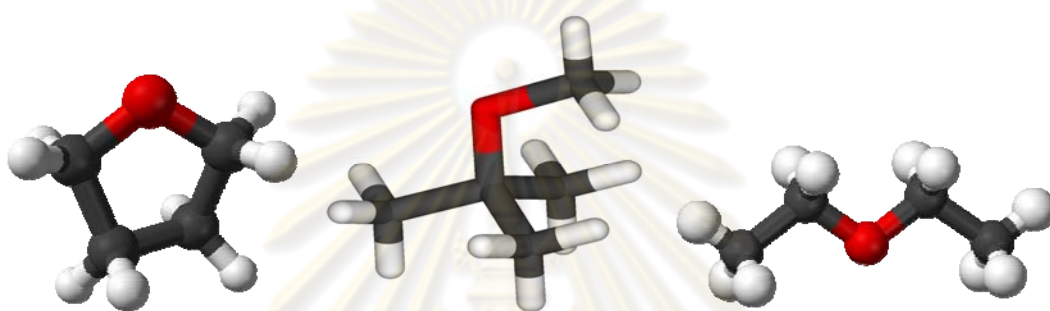


Fig. 2.15 Molecular Structure of Tetrahydrofuran, MTBE and Diethyl Ether

2.7 Reaction Conditions for the Study

KOH was the selected catalyst due to its high catalytic activity, relatively cheap price and the washing water from biodiesel product can be used as a fertilizer. Catalyst concentration will be fixed at 1 wt% KOH to oil which has given the highest yields and viscosity of esters in methanolysis as suggested in the literature review in Section 2.4.2.

Excess methanol of 6:1 mol ratio of methanol to oil was selected for the study which will yield maximum conversion for methanolysis as described in the literature review in Section 2.4.3.

From the study of biodiesel production from palm stearin using THF and hexane as a cosolvent (Veerapol et al., 2008) found that at 0.2 mol ratio of cosolvent to methanol yields the highest methyl ester content, hence in this study the mol ratio of solvent to methanol will be fixed at 0.2.

CHAPTER III

RESEARCH METHODOLOGY

3.1 Experimental

The palm stearin oil was transesterified in the presence of methanol with cosolvent using homogeneous base catalyst (Potassium hydroxide) in a laboratory scale.

3.2 Experimental Equipment

- 1 Beaker 600 ml.
- 2 Stick glass
- 3 Spatula
- 4 Water Bath
- 5 Tree blade paddle
- 6 Variable speed motor
- 7 Funnel
- 8 Thermometer
- 9 Hotplate
- 10 Weight Scale
- 11 Gas Chromatograph

3.3 Chemical

1. Methanol, Analytical grade : Merk
2. Potassium hydroxide, Analytical grade : Carlo Erba
3. Tetrahydrofuran, Analytical grade : QReC
4. MTBE, Analytical grade: Carlo Erba
5. Ether, Analytical grade: J.T.Baker
6. Sulfuric acid, Analytical grade : J.T.Baker

3.4 Raw Material

Palm stearin used in the experiment was obtained from Olene Co., Ltd.

3.5 Research Procedure

3.5.1 Analyze chemical properties of raw material for

1. Free fatty acid content
2. Iodine value
3. Moisture content
4. Slip point
5. Free fatty acid composition in accordance with AOAC969.33, AOAC991.39 (2005)

Properties of palm stearin were provided with the palm stearin by Olene Co., Ltd. except item 5 was analyzed by Thailand Institute of Scientific and Technological Research (TISTR).

3.5.2 Experimentation steps

- 1 Heat the palm stearin to a temperature of approximately 120 degree Celsius for 60 minute in order to remove the impured water (100 g).
- 2 Mix methanol with potassium hydroxide at 60 degree Celsius (23.07 g methanol with 1.0 weight percent of Potassium hydroxide to methanol).
- 3 Mix cosolvent with palm stearin at the required reaction temperature (0.2 mol percent of cosolvent to palm stearin, 55 and 60 degree Celsius)
- 4 Heat or cool the methoxide obtained from step 3 to the required reaction temperature.
- 5 Pour the methoxide obtained from step 4 into the mixed oil and cosolvent in step 3. and stir at 200,250,400 and 550 rpm. Start counting the reaction time.

- 6 Terminate the reaction using sulfuric acid to neutralize the un-reacted base catalyst and to reduce soap produced from saponification of free fatty acids and base catalyst
- 7 Pour the product into a funnel and leave it until methyl ester and glycerol is completely separated (3 hours).
- 8 Check the upper layer, methyl ester, of the separated product to remove the un-reacted excess methanol and cosolvent by distillation at a temperature approximately 100 degree Celsius.
- 9 Wash methyl ester with distilled water to remove the excess base catalyst, methanol, cosolvent and glycerol.
- 10 Pour methyl ester and water into a funnel and leave it until methyl ester and washing water is completely separated.
- 11 Wash methyl ester until the washing water is neutralize (pH7).
- 12 Remove washing water by heating the methyl ester at a temperature of approximately 120 degree Celsius for 1 hour duration.
- 13 Filter the methyl ester using filter paper no.1 and keep the sample for the analysis of ester composition by gas chromatograph.

3.5.3 Analyze percentage of produced methyl ester using gas chromatograph.

The content of fatty acid methyl ester (FAME) in product was analyzed by a GC-2010 gas chromatography (Shimadzu). Its column is SGE, BP20 GC capillary column (30m x 0.32mm.i.d. x 0.25 μ m film thickness) capable to maintain temperature in the range of 20 – 250 degree Celsius. The temperature of the injector and the flame ionization detector (FID) was 210 and 250 °C, respectively. The chromatographic conditions are summarized in Table 3.1 below:-

Table 3.1 Chromatographic conditions

Condition	Value
Carrier gas (He) flow rate	30 mL/min
Detector temperature (FID)	250 °C
Split ratio	1 : 25
Injection part temperature	220 °C
Inject volume	1 µL
Column temperature	210 °C
Holding time	30 min

The ester content of fatty acid methyl ester is determined in accordance with EN14103:2003 (Fat and Oil Derivatives – Fatty Acid Methyl Ester (FAME) – Determination of ester and linolenic acid methyl ester contents) using methyl heptadecanoate ($C_{18}H_{36}O_2$) as an internal standard and use normal heptane as a solvent. Appendix B shows how to determine FAME content from gas chromatograph in accordance with EN 14103. Appendix E contains GC result for the products. Gas chromatography of the products was carried out by Scientific and Technological Research Equipment Center Chulalongkorn University.

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CHAPTER IV

RESULTS AND DISCUSSION

4.1 Raw Material Properties

4.1.1 Physical and Chemical Properties

Table 4.1 shows the analyzed properties of palm stearin used in the experiments.

Table 4.1 Properties of palm stearin used in the experiments

Properties	Palm stearin
Free fatty acid (%) (as palmitic acid)	0.04
Moisture content (%)	0.03
Peroxide value (meq O ₂ /kg)	0.57
Slip point (°C)	54
Iodine value (g I ₂ / 100 g oil)	82.86

4.1.2 Fatty Acid Content

Table 4.2 shows the composition of palm stearin feedstock used in the experiments which were analyzed by Thailand Institute of Scientific and Technological Research using AOAC 969.33, AOAC 991.39 (2005) analytical standard with Gas Chromatography. The result shows that major fatty acid component of palm stearin is 63.7 wt% Palmitic acid (C16:0) and 24 wt% Oleic. The total percentage of saturated fatty acid is 70.7 wt% and the remaining 29,3 wt% is unsaturated fatty acid. From the analyzed fatty acid content, the average molecular weight of palm stearin can be calculated as 831.98 g/mol.

Table 4.2 Fatty acid contents in palm stearin used in the experiments

Fatty acid composition (wt%)	Molecular Weight	% By Weight
C12:0 Lauric acid	200	0.3
C14:0 Myristic acid	228	1.2
C15:0 Pentadecanoic acid	242	0.1
C16:0 Palmitic acid	256	63.7
C17:0 Margaric acid	270	0.1
C18:0 Stearic acid	284	4.9
C20:0 Arachidic acid	312	0.3
C24:0 Lignoceric acid	368	0.1
Saturated		70.7
C16:1 Palmitoleic acid	254	0.1
C18:1 Oleic acid	282	24
C18:2 Linoleic acid	280	5
C18:3 α -Linolenic acid	278	0.1
C20:1 Gondoic acid	310	0.1
Unsaturated		29.3
Total fatty acid composition		100
Molecular weight		831.98

4.2 Effect of Speed

Mixing is very important in the transesterification reaction, as oils or fats are immiscible with sodium hydroxide–methanol solution. Once the two phases are mixed and the reaction is started, stirring is no longer needed. Initially the effect of mixing on transesterification of beef tallow was study by Ma et al. (Ma et al., 1999). No reaction was observed without mixing and when NaOH–MeOH was added to the melted beef tallow in the reactor while stirring, stirring speed was insignificant. Reaction time was the controlling factor in determining the yield of methyl esters. This suggested that the stirring speeds investigated exceeded the threshold requirement of mixing.

This research studied for the optimum speed using 6:1 constant molar ratio of methanol to oil, 1 wt% of potassium to oil, 60 °C and 10 minutes reaction time with varying stirring speed of 200, 250, 400 and 550 rpm. The paddle used in the experiments is three blade paddle, 5 cm diameter and 2.2 cm blade diameter. The beager is 9 cm diameter, 13 cm height with 3.0 cm chemical level in case of the reaction without cosolvent and 4.0 cm chemical level in case of using cosolvent. Distant between blades to bottom of the beager is 1.5 cm. The experimental results are shown in figure 4.1.

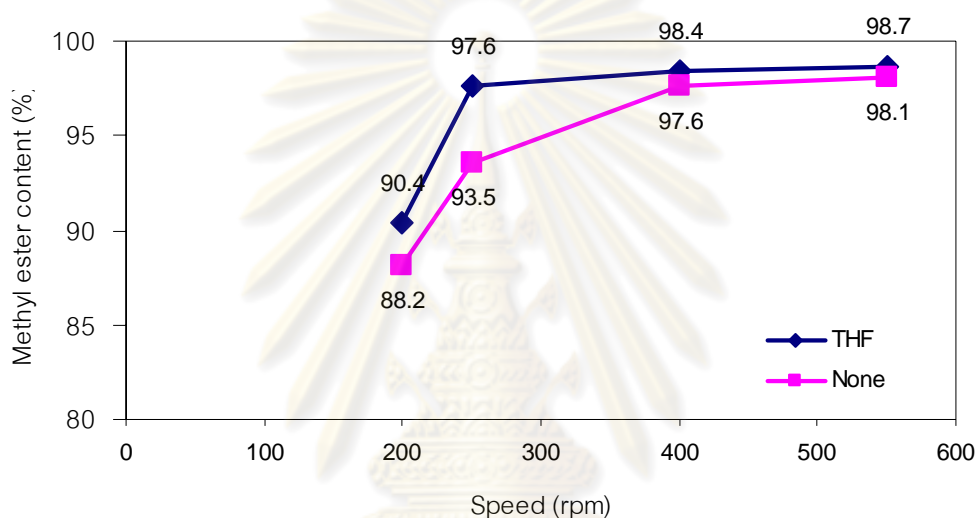


Fig. 4.1 Effects of stirring speed on wt% of methyl ester yield for the reaction with and without tetrahydrofuran cosolvent

Reaction Condition: 6:1 mol MeOH/ Oil, 1 wt% KOH, 60°C, 10 minutes, 0.2 mol% THF/ MeOH

Figure 4.1 shows that increasing of stirring speed will increase methyl ester content in the product due to increasing stirring speed resulting in smaller droplet size diameter of methanol from blade shearing. This will increase reaction surface for methanol, catalyst and oil resulting in faster reaction rate. In case of the reaction with tetrahydrofuran cosolvent, the highest content of 98.7 wt% methyl ester occurs at 550 rpm stirring speed. However, increasing of stirring speed from 250 rpm to 550 rpm enhances only 1.1 wt% additional methyl ester content for the reaction with tetrahydrofuran cosolvent whereas the reaction without cosolvent enhances 4.6 wt% methyl ester content from the same speed increasing.

It can be concluded that the optimum speed to obtain ester content meeting with biodiesel standard, i.e. 96.5 wt% minimum is 250 rpm for the reaction with THF and 400 rpm for the reaction without THF.

From accuracy/ error analysis in Appendix C shows that if the different of methyl ester content from the different experiment conditions is less than 0.81 wt% (98.96 – 98.15), the result can be considered that these conditions yield the same methyl ester content, hence it can be considered that there are no differences of methyl ester content from the reaction with and without THF at 550 rpm. So, at 550 rpm the effect of mixing overcomes the effect of cosolvent.

4.3 Effects of Reaction Temperature and Time

Transesterification reaction of vegetable oil using alkali catalyst will be performed at the temperature near to the boiling point of alcohol. Methanol has a boiling point of 64.7 °C at atmospheric pressure, so the reaction temperature should not be greater than this temperature. While the freezing point of palm stearin is 54 °C, so the experiment must be done between 55 to 60 °C.

In this research the experiments were carried out at 6:1 constant molar ratio of methanol to oil, 1 wt% of potassium hydroxide to oil, 55 and 60 °C, 5, 10 and 15 minutes reaction time and 250 rpm stirring speed.

4.3.1 Transesterification reaction without cosolvent

The experiment result for the reaction without cosolvent is shown in Figure 4.2.

From the above figure, methyl ester contents in the product from gas chromatography analysis are:-

- 1) At 55 °C reaction temperature and 5, 10, 15 min. reaction time gave 90.2%, 92.5%, 93.2% methyl ester content, respectively.
- 2) At 60 °C reaction temperature and 5, 10, 15 min. reaction time gave 92.0%, 93.5%, 94.3% methyl ester content, respectively.

Figure 4.2 shows that wt% methyl ester formed from the reaction at 60 °C is greater than at 55 °C due to higher reaction temperature giving higher reaction rate.

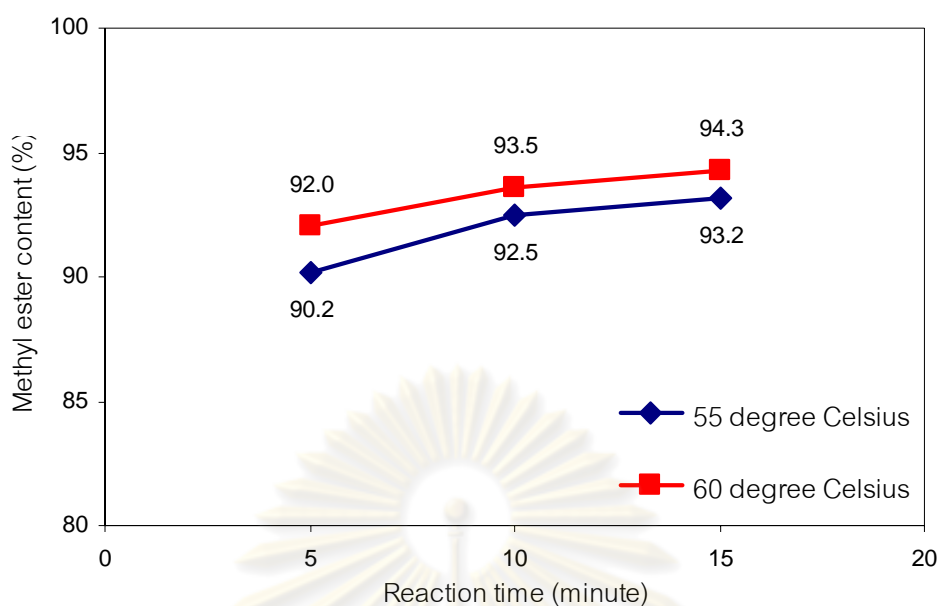


Fig. 4.2 Effects of reaction temperature and time on wt% methyl ester in the reaction without cosolvent

Reaction Condition: 6:1 mol MeOH/ Oil, 1 wt% KOH, 250 rpm

4.3.2 Transesterification reaction with tetrahydrofuran cosolvent

In case of using tetrahydrofuran as a cosolvent at the same reaction condition resulted in higher wt% methyl ester content in the product as per figure 4.3.

From Fig 4.3, methyl ester contents in the product from gas chromatography analysis are:-

- 1) At 55 °C reaction temperature and 5, 10, 15 min. reaction time gave 93.9%, 95.7%, 96.3% methyl ester content, respectively.
- 2) At 60 °C reaction temperature and 5, 10, 15 min. reaction time gave 95.2%, 97.6%, 98.3% methyl ester content, respectively.

Figure 4.3 shows that wt% methyl ester formed from the reaction at 60 °C is greater than at 55 °C due to higher reaction temperature giving higher reaction rate.

However, using THF as a cosolvent gave a higher methyl ester content in the product compared to the reaction without cosolvent at the same reaction time due to THF enhances the miscibility of methanol and oil to become a homogeneous phase resulting in a high reaction rate and the reaction will be in equilibrium faster.

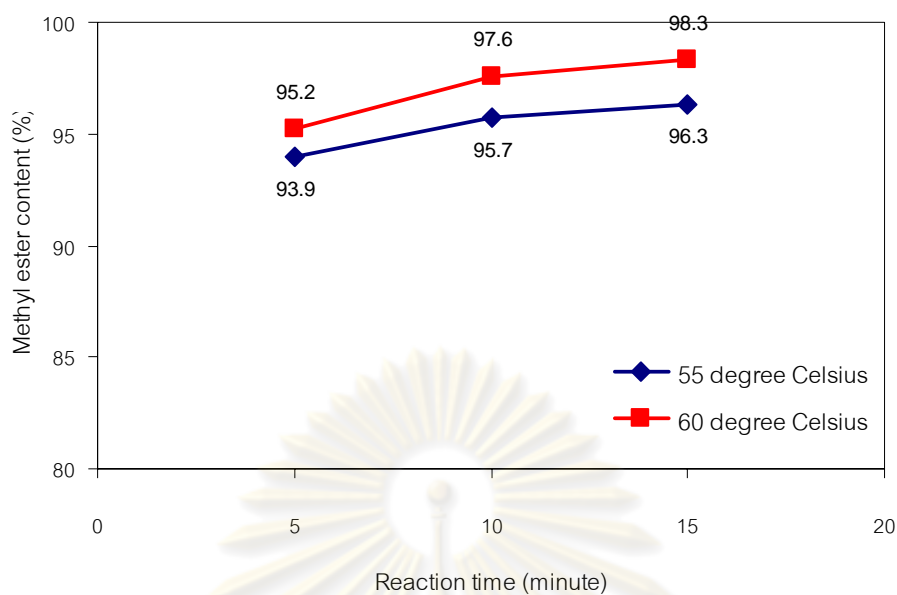


Fig. 4.3 Effects of temperature and time on wt% methyl ester in the reaction with THF cosolvent

Reaction Condition: 6:1 mol MeOH/ Oil, 1 wt% KOH, 250 rpm, 0.2 mol% THF/ MeOH

4.3.3 Transesterification with MTBE cosolvent

In case of using MTBE as a cosolvent at the same reaction condition resulted in the same trend but less effective than tetrahydrofuran as per figure 4.4.

From the above figure, methyl ester contents in the product from gas chromatography analysis are:-

- 1) At 55 °C reaction temperature and 5, 10, 15 min. reaction time gave 88.9%, 92.3%, 92.9% methyl ester content, respectively.
- 2) At 60 °C reaction temperature and 5, 10, 15 min. reaction time gave 93.0%, 94.9%, 96.1% methyl ester content, respectively.

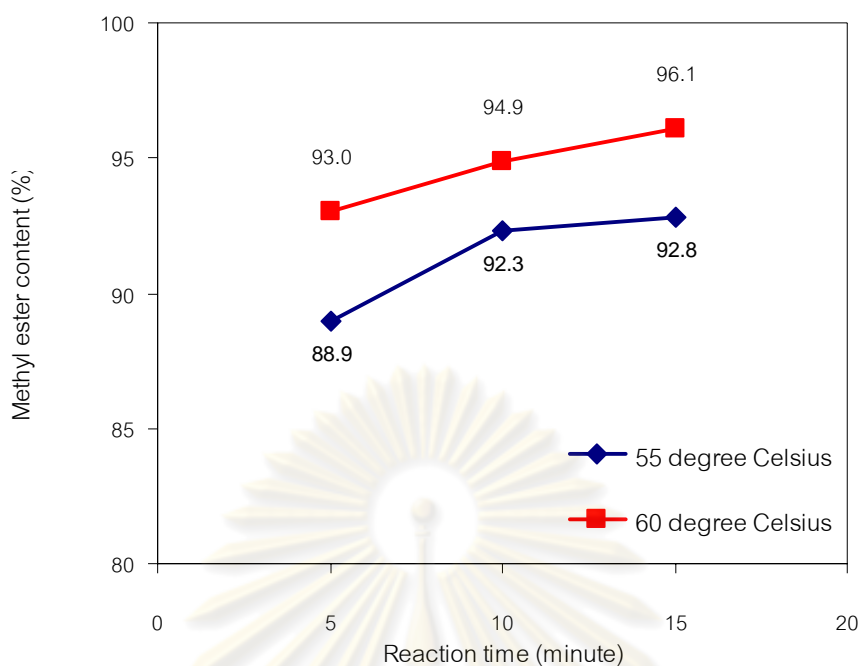


Fig. 4.4 Effects of temperature and time on wt% methyl ester in the reaction with MTBE cosolvent

Reaction Condition: 6:1 mol MeOH/ Oil, 1 wt% KOH, 250 rpm, 0.2 mol% MTBE/ MeOH

4.3.4 Transesterification with DEE cosolvent

In case of using Diethyl Ether as a cosolvent at the same reaction condition resulted in the same trend but less effective than tetrahydrofuran as per figure 4.5.

From Fig 4.5, methyl ester contents in the product from gas chromatography analysis are:-

- 1) At 55 °C reaction temperature and 5, 10, 15 min. reaction time gave 92.6%, 93.2%, 94.5% methyl ester content, respectively.
- 2) At 60 °C reaction temperature and 5, 10, 15 min. reaction time gave 92.8%, 94.0%, 96.3% methyl ester content, respectively.

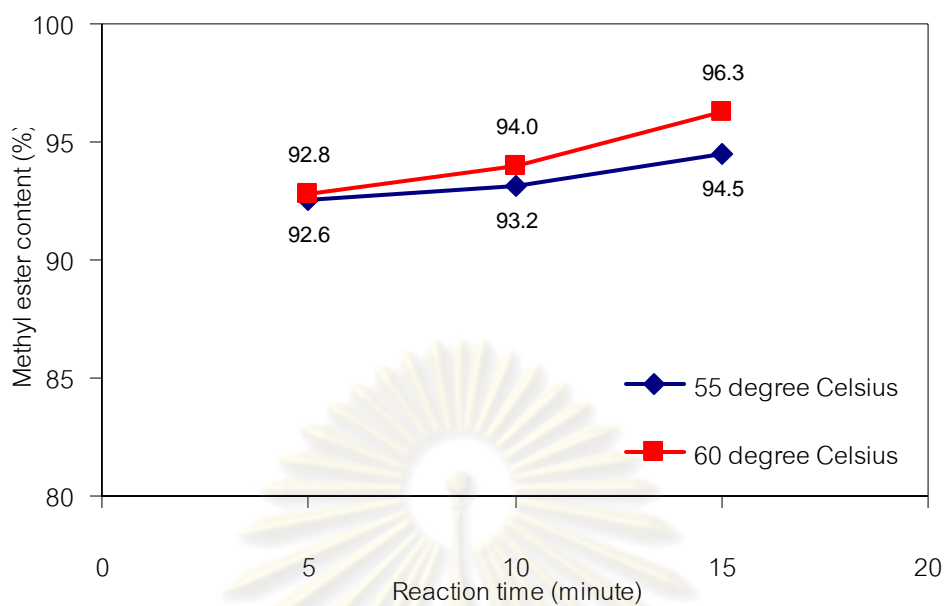


Fig. 4.5 Effects of temperature and time on wt% methyl ester in the reaction with DEE cosolvent

Reaction Condition: 6:1 mol MeOH/ Oil, 1 wt% KOH, 250 rpm, 0.2 mol% DEE/ MeOH

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CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

1. Addition of cosolvent into the reaction mixture making oil miscible with methanol. Because the mass transfer in the one-phase reaction is superior to that in the two-phase reaction, due to an increase in the contact surface, the production rate of methyl ester by the one-phase reaction increases. Therefore, the initial addition of a cosolvent enhances the miscibility of oil to methanol and reduces the time required to form the one-phase system.
2. Using tetrahydrofuran as a cosolvent resulted in higher methyl ester content than diethyl ether in which diethyl ether cosolvent gave higher methyl ester content than MTBE cosolvent. The maximum methyl ester content obtained from the alkali-catalyzed transesterification of palm stearin and methanol (1:6 molar ratio) without cosolvent using 1 wt% KOH and 250 rpm stirring speed was 94.3 wt% at 60 °C reaction temperature and 15 minutes reaction time while the reaction at the same condition with 0.2 mol % tetrahydrofuran, DEE and MTBE cosolvent gave methyl ester content 98.3, 96.3 and 96.1 wt% respectively.
3. In this study, the experiments to observe the effects of stirring speed also had been carried out by varying stirring speed from 250 to 550 rpm using 6:1 molar ratio of methanol to palm stearin oil, 1 wt% KOH, 60 °C and 10 minutes reaction time, found that the maximum methyl ester contents for the reaction with and without tetrahydrofuran cosolvent were 98.7 and 98.1 wt%, at the maximum stirring speed of 550 rpm, respectively. However, increasing of stirring speed from 250 rpm to 550 rpm enhances only 1.1 wt% additional methyl ester content for the reaction with tetrahydrofuran cosolvent whereas the reaction without cosolvent enhances 4.6 wt% methyl ester content from the same speed increasing. It can be concluded that the optimum speed to obtain ester content

meeting with biodiesel standard, i.e. 96.5 wt% minimum is 250 rpm for the reaction with THF and 400 rpm for the reaction without THF.

4. From accuracy/ error analysis in Appendix C shows that if the different of methyl ester content from the different experiment conditions is less than 0.81 wt% (98.96 – 98.15), the result can be considered that these conditions yield the same methyl ester content, hence it can be considered that there are no differences of methyl ester content from the reaction with and without THF at 550 rpm. So, at 550 rpm the effect of mixing overcomes the effect of cosolvent.
5. It also observed that increasing the reaction time (upto 15 minutes) and reaction temperature (upto 60 °C) also resulted in higher methyl ester content in the product.
6. To obtain methyl ester content meeting with biodiesel standard (96.5 wt% minimum) for the reaction without cosolvent the reaction time must be more than 15 minutes for for 250 rpm 60 °C or the stirring speed must be 400 rpm minimum for 10 minutes 60 °C.
7. The conditions to obtain methyl ester content meeting with biodiesel standard (96.5 wt% minimum) for the reaction with cosolvent at 250 rpm are
 - 7.1 10 minutes at 60 °C or more than 15 minutes at 55 °C for THF cosolvent
 - 7.2 More than 15 minutes at 60 °C DEE and MTBE cosolvent

5.2 Recommendations

1. The addition of diethyl ether to the reaction as a cosolvent can improve the reaction rate and methyl ester content but with less efficiency than tetrahydrofuran. However, due to the fact that diethyl ether is cheaper than

tetrahydrofuran, the comparative economics study between tetrahydrofuran and diethyl ether is recommended.

2. In order to obtain methyl ester content to meet with the biodiesel standard, i.e. 96.5 wt% minimum, it was found that even using tetrahydrofuran the reaction temperature shall be more than 55 °C at 15 minutes. However, the current study on the effect of cosolvent was done at fixed 250 rpm, 6:1 mol MeOH/Oil and 1 wt% KOH, so further study by increasing stirring speed, methanol to oil molar ratio or catalyst concentration is recommended.



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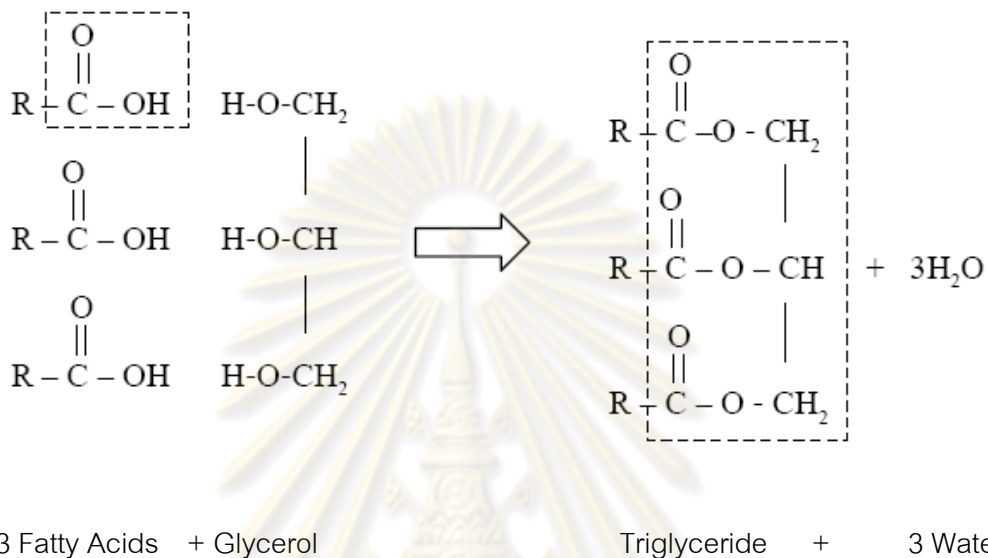
APPENDICES

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APPENDIX A

REACTION CHEMISTRY CALCULATION

Palm Stearin Molecular Weight Calculation



From Triglyceride formation reaction, the molecular weight of triglyceride can be obtained from the following equation:-

$$\begin{aligned}
 MW_{TG} &= 3 R_{avg} + 38 \\
 R_{avg} &= \sum \left[\frac{\%F_{AN}}{100} \times MW_n \right]
 \end{aligned}$$

Where

MW_{TG} is average molecular weight of triglyceride

R_{avg} is average Molecular weight of fatty acid less COOH

$\%F_{AN}$ is %wt of each fatty acid in palm stearin

MW_n is Molecular weight of each fatty acid

$$\begin{aligned}
 R_{AVG} &= (0.003 \times 200) + (0.012 \times 228) + (0.001 \times 242) + (0.637 \times 256) + (0.001 \times 270) + \\
 &+ (0.049 \times 284) + (0.003 \times 312) + (0.001 \times 368) + (0.001 \times 254) + (0.24 \times 282) + (0.05 \times 280) + \\
 &+ (0.001 \times 278) + (0.001 \times 310) = 264.66 \text{ g/mol}
 \end{aligned}$$

$$\begin{aligned}
 MW_{TG} &= (3 \times 264.66) + 38 \\
 &= 831.98 \text{ g/mol}
 \end{aligned}$$

Table A1 Chemical and physical properties

Chemical	Density (g/ml)	Molecular Weight
Palm stearin	0.86	831.98
Methanol	0.79	32.04
Tetrahydrofuran	0.88	72.11
MTBE	0.74	88.15
Diethyl Ether (DEE)	0.71	74.12

Methanol Quantity Calculation

The experiment used palm sterain 100 g

Molar ratio of methanol to oil is 6

Palm stearin 100 g equals to $100/831.98 = 0.120$ mol

Hence, methanol is $6 \times 0.120 = 0.72$ mol or $0.72 \times 32.04 = 23.07$ g

or $23.07/0.79 = 29.20$ ml

Catalyst Quantity Calculation

The experiment used 1 %wt catalyst to palm sterain

Hence, catalyst quantity used is $(1/100) \times 100 = 1$ g

Moisture Quantity Calculation

Palm sterain contains 3 %wt

or palm stearin 100 g contains moisture $(3/100) \times 100 = 3$ g

Tetrahydrofuran Quantity Calculation

Methanol used in the experiment is 0.72 mol
Used 0.2 molar ratio of tetrahydrofuran to methanol
Hence, tetrahydrofuran used is $0.2 \times 0.72 = 0.144$ mol
or $0.144 \times 72.11 = 10.38$ g or $10.38 / 0.88 = 11.80$ ml

MTBE Quantity Calculation

Methanol used in the experiment is 0.72 mol
Used 0.2 molar ratio of MTBE to methanol
Hence, MTBE used is $0.2 \times 0.72 = 0.144$ mol
or $0.144 \times 88.15 = 12.69$ g or $12.69 / 0.74 = 17.15$ ml

DEE Quantity Calculation

Methanol used in the experiment is 0.72 mol
Used 0.2 molar ratio of DEE to methanol
Hence, DEE used is $0.2 \times 0.72 = 0.144$ mol
or $0.144 \times 74.12 = 10.67$ g or $10.67 / 0.71 = 15.03$ ml



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APPENDIX B

DETERMINATION OF METHYL ESTER CONTENT

Analysis of Methyl Ester Content in the Product

Methyl ester content in the product is determined from gas chromatograph in accordance with EN 14103: 2003 using methyl heptadecanoate (C17:0) as an internal standard and uses normal heptane (n-C₇H₁₆) as a solvent. Methyl ester content is calculated using the following formula:

$$C = \frac{(\sum A) - A_{EI}}{A_{EI}} \times \frac{C_{EI} \times V_{EI}}{m} \times 100$$

Where

$\sum A$ is the total peak area from methyl ester in C14 to that in C24:1

A_{EI} is the peak area corresponding to methyl heptadecanoate

C_{EI} is the concentration, in mg/ml, of methyl heptadecanoate solution being used

V_{EI} is the volume, in ml, of the methyl heptadecanoate solution being used

m is the mass, in mg, of the sample

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APPENDIX C

ANALYSIS OF EXPERIMENTAL ERROR

In order to check the accuracy/ error of the experimental result, three samples were tested at the same reaction conditions, i.e. 6:1 mol MeOH/ Oil, 1 wt% KOH, 60 °C, 10 minutes, 550 rpm, with 0.2 mol% THF cosolvent. The results are shown in the figure below.

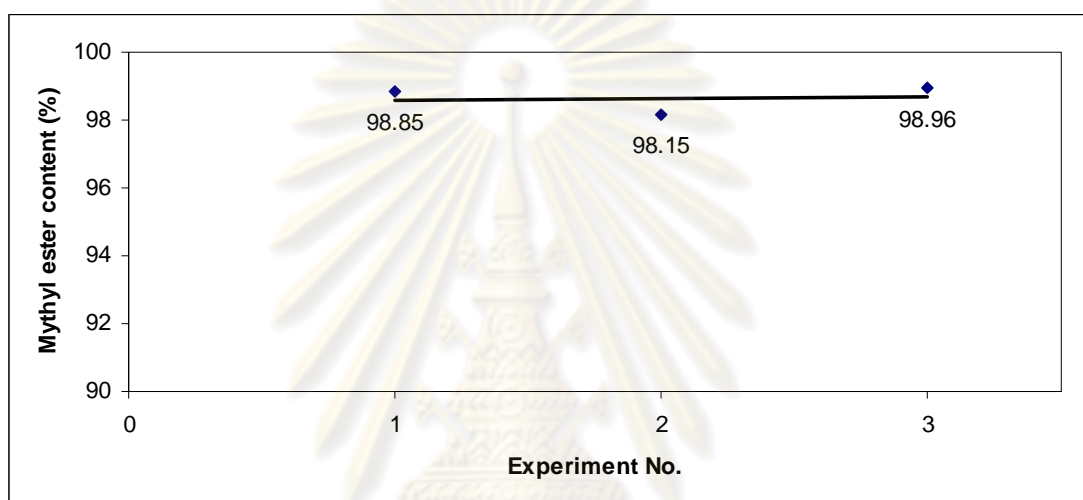


Fig. C1 Methyl ester content from the three experiments at the same reaction conditions

Reaction condition: 6:1 mol MeOH/ Oil, 1 wt% KOH, 60 °C, 10 minutes, 550 rpm, without cosolvent

From the above results

- average methyl ester content is 98.65 wt%
- maximum methyl ester content (from experiment no. 3) is 98.96 wt% or +0.31% error from the average value
- minimum methyl ester content (from experiment no. 2) is 98.15 wt% or -0.51 % error from the average value

It can be concluded that if the different of methyl ester content from the different experiment conditions is less than 0.81 wt% (98.96 – 98.15), the result can be considered that these conditions yield the same methyl ester content.

APPENDIX D

BOILING POINT ELEVATION OF MIXTURE

The solution of two or more components with different boiling point will have a bubble point which can be calculated from the thermodynamics equilibrium between vapor and liquid phases with the criteria on equality of temperature in both phases, equality of pressure in both phases and equality of fugacity of each component in both phases. In order to simplify the calculation we will apply Raoult's law which is based on the assumption that the vapor phase behaves as ideal gas and liquid phase is an ideal solution, i.e. gas component fugacity is equals to its partial pressure and liquid component fugacity equals to its mole fraction multiplied by its vapor pressure.

$$P = x_1 p_1^* + x_2 p_2^*$$

$$x_2 = 1 - x_1$$

Vapor pressure is dependent on temperature, we will use Antoine equation for the correlation of pressure and temperature.

$$p^* = 10^{(A - B/(t+C))}$$

Where p^* is vapor pressure in mm Hg

T is temperature in degree C

Upon substitution we arrive at the following expression

$$P = x_1 * 10^{(A_1 - B_1/(t_1+C_1))} + x_2 * 10^{(A_2 - B_2/(t_1+C_2))}$$

From the experiment by Joshua H. et al (Calvin College Engineering – 2008) Antoine coefficients for soy oil, which will be used for the calculation as the data for stearin is not available, are $A = 11.4785$, $B = -708.72$ and $C = -167.48$.

From Lange's Handbook of Chemistry 15th Antoine coefficients for diethyl ether are $A = 6.92032$, $B = 1064.07$ and $C = 228.80$.

In the experiment we used 0.2 mole ratio for diethyl ether to methanol, 6:1 mol ratio of methanol to stearin (0.14 mol DEE in 0.12 mol stearin) and $P = 1$ atm. By trial & error we found that bubble point of the solution is 95.1 degree C.

APPENDIX E
EXPERIMENTAL DATA

1. Experimental Data

Sample No.	Cosolvent	Reaction Condition		
		Speed (rpm)	Temperature (°C)	Time (min)
S01	-	200	60	10
S02	-	250	60	10
S03	-	400	60	10
S04	-	550	60	10
S05	THF	200	60	10
S06	THF	250	60	10
S07	THF	400	60	10
S08	THF	550	60	10
S09	-	250	60	5
S10	-	250	60	15
S11	-	250	55	5
S12	-	250	55	10
S13	-	250	55	15
S14	THF	250	60	5
S15	THF	250	60	15
S16	THF	250	55	5
S17	THF	250	55	10
S18	THF	250	55	15
S19	MTBE	250	60	5
S20	MTBE	250	60	10
S21	MTBE	250	60	15
S22	MTBE	250	60	5
S23	MTBE	250	60	10
S24	MTBE	250	60	15

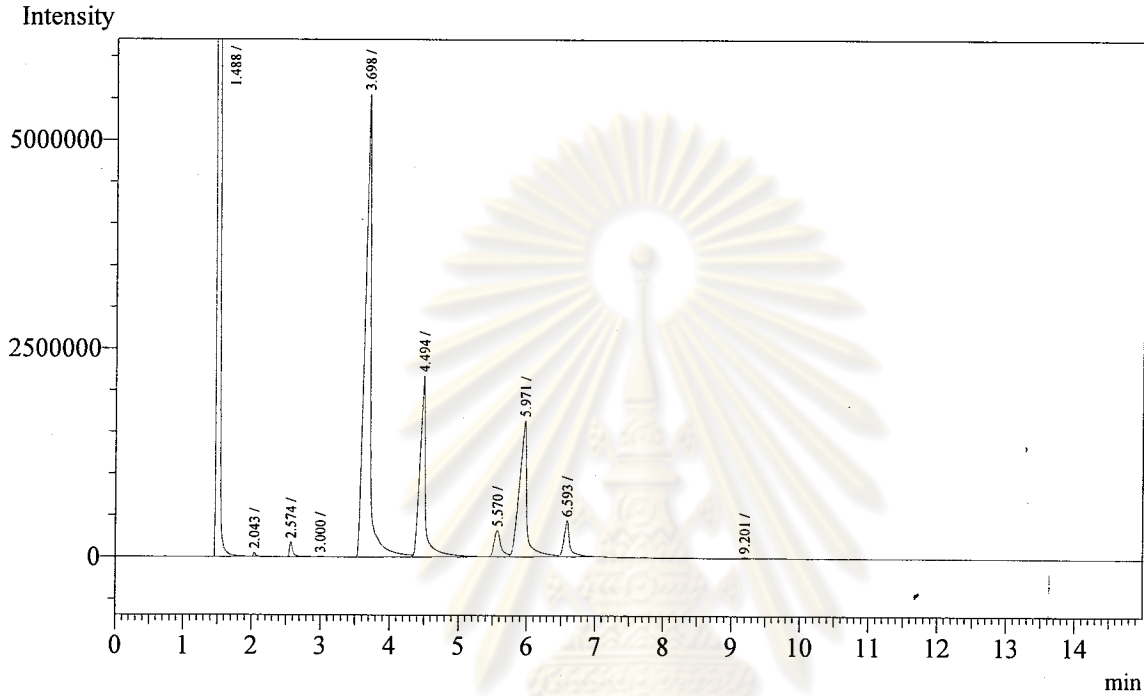
Sample No.	Cosolvent	Reaction Condition		
		Speed (rpm5)	Temperature (°C)	Time (min)
S25	ETHER	250	60	5
S25	ETHER	250	60	10
S27	ETHER	250	60	15
S28	ETHER	250	60	5
S29	ETHER	250	60	10
S30	ETHER	250	60	15
S31	THF	550	60	10
S32	THF	550	60	10

2. Gas Chromatography Analysis for Methyl Ester Content in Product

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Analysis Date & Time : 2/9/2552 13:47:58
 User Name : Admin
 Vial# : 21
 Sample Name : S01
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA110.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

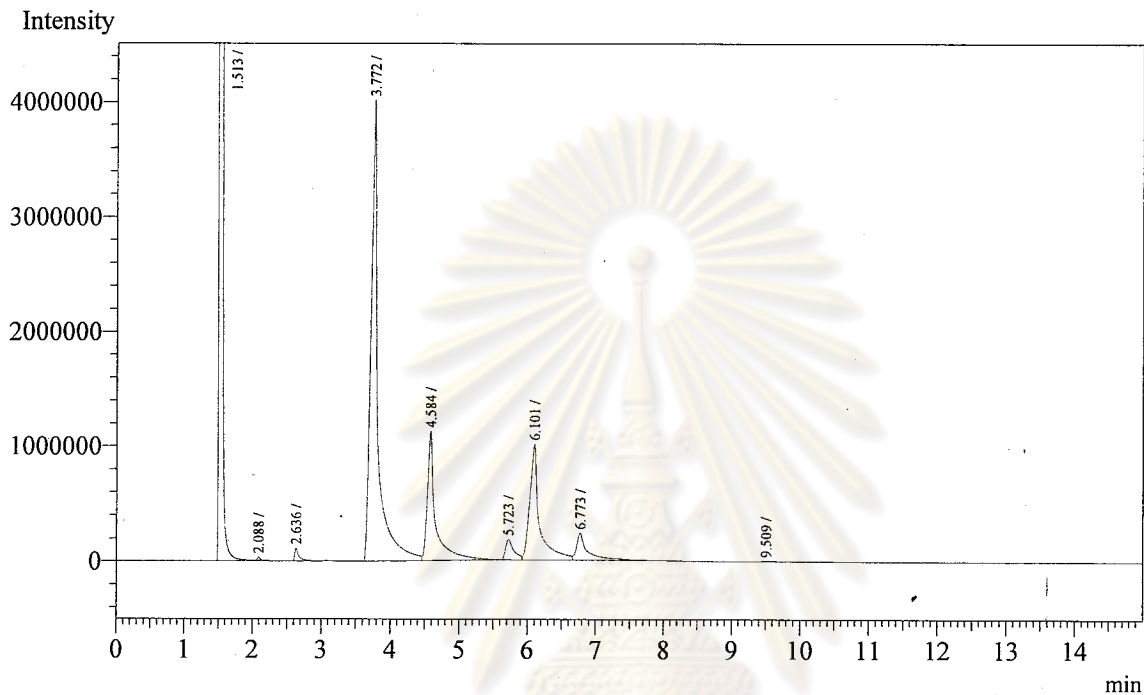


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.488	826658511	73118692	0.000	S	
2	2.043	118316	49657	0.000		
3	2.574	575832	177509	0.000		
4	3.000	30305	7263	0.000		
5	3.698	37360429	5528305	0.000		
6	4.494	12967032	2158533	0.000	V	
7	5.570	2175006	315064	0.000		
8	5.971	12801259	1630830	0.000	V	
9	6.593	2621189	433212	0.000	V	
10	9.201	113123	13895	0.000		
Total		895421002	83432960			

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 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/7/2552 10:45:52
 User Name : Admin
 Vial# : 4
 Sample Name : S02
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA8.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

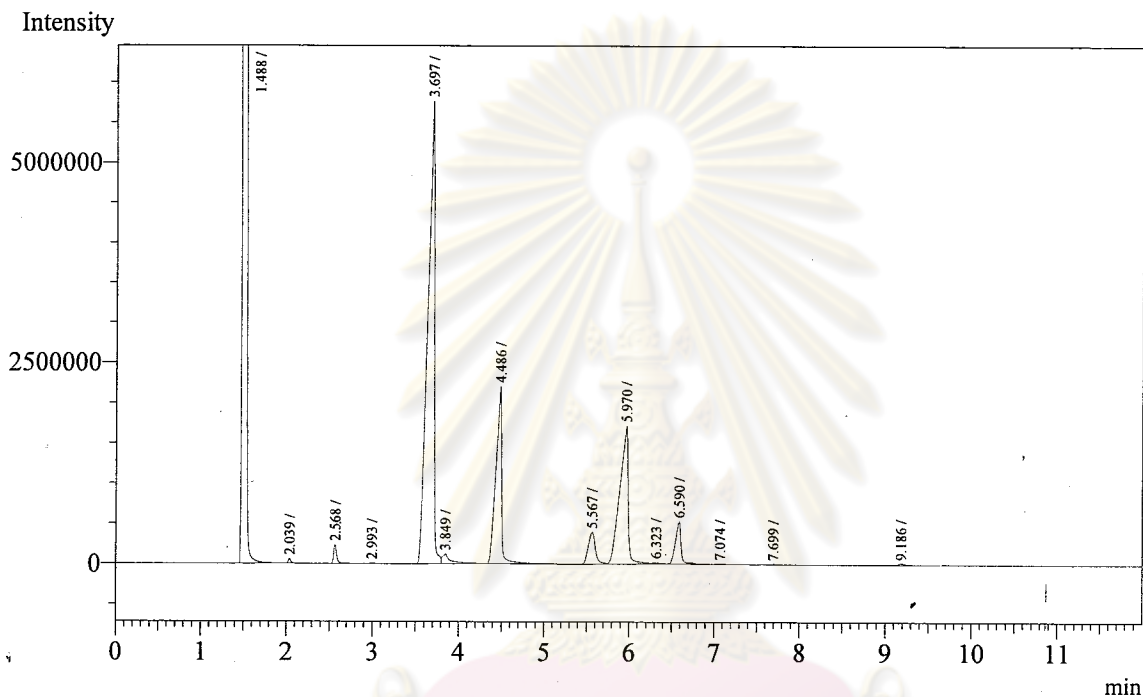


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.513	742341323	93669805	0.000			
2	2.088	91865	31060	0.000			
3	2.636	485489	110940	0.000			
4	3.772	29214648	4003598	0.000			
5	4.584	9403185	1123298	0.000	V		
6	5.723	1484895	178379	0.000			
7	6.101	10060612	1006489	0.000	V		
8	6.773	2761908	236659	0.000	V		
9	9.509	69389	6440	0.000			
Total		795913314	00366668				

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 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 3/9/2552 15:44:03
 User Name : Admin
 Vial# : 29
 Sample Name : S03
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

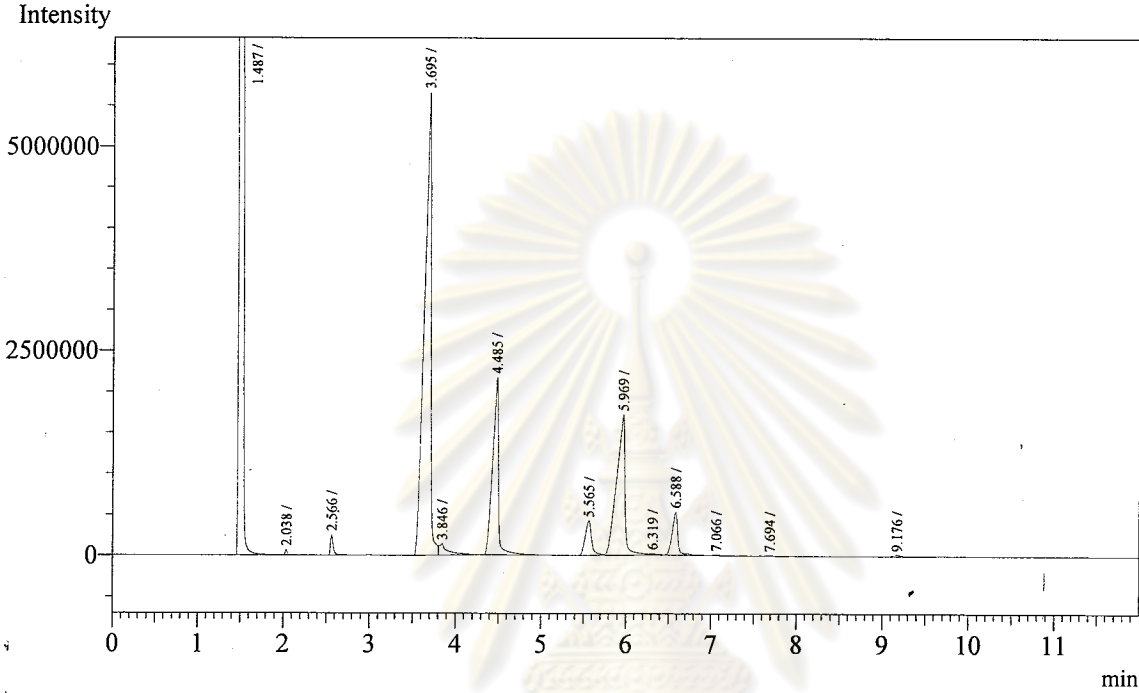
Data Name : D:\ACID\FA118.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm



Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.488	868958265	79583135	0.000	S	
2	2.039	104711	59822	0.000		
3	2.568	274963	231796	0.000	S	
4	2.993	245429	9583	0.000		
5	3.697	31999349	5708505	0.000		
6	3.849	925957	121126	0.000	V	
7	4.486	10426573	2191265	0.000		
8	5.567	2329883	394893	0.000		
9	5.970	14310576	1712107	0.000	SV	
10	6.323	58862	12238	0.000	T	
11	6.590	56702	522713	0.000	S	
12	7.074	44012	9051	0.000		
13	7.699	34138	7038	0.000		
14	9.186	134038	19146	0.000		
Total		929903458	90582418			

Analysis Date & Time : 3/9/2552 15:16:45
 User Name : Admin
 Vial# : 27
 Sample Name : S04
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA116.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

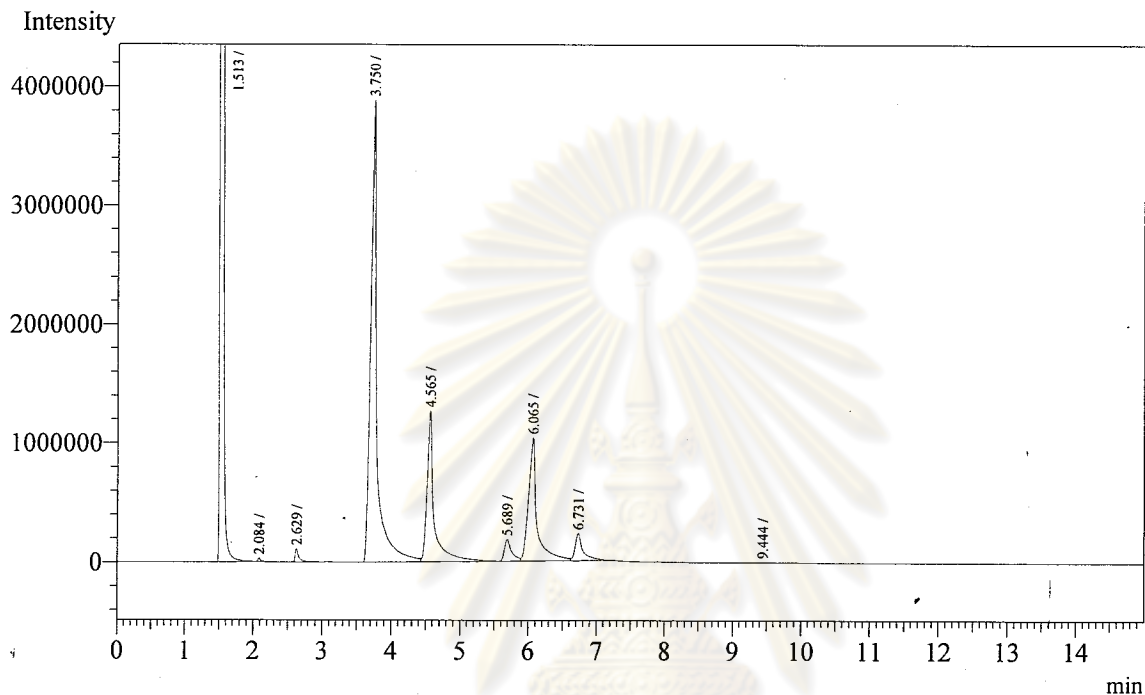


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.487	832823568	03448812	0.000	S		
2	2.038	114595	64080	0.000			
3	2.566	575173	248087	0.000			
4	3.695	30592515	5594708	0.000			
5	3.846	1025813	139163	0.000	V		
6	4.485	9877600	2147218	0.000			
7	5.565	2265248	422193	0.000			
8	5.969	9680972	1708217	0.000	SV		
9	6.319	36814	10558	0.000	T		
10	6.588	2451572	526299	0.000	S		
11	7.066	38208	8562	0.000			
12	7.694	31994	6875	0.000			
13	9.176	129182	19412	0.000			
Total		889643254	14344184				

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 วิทยาลัย
 ทรัพยากร

Analysis Date & Time : 15/7/2552 12:19:47
 User Name : Admin
 Vial# : 17
 Sample Name : S05
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA32.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

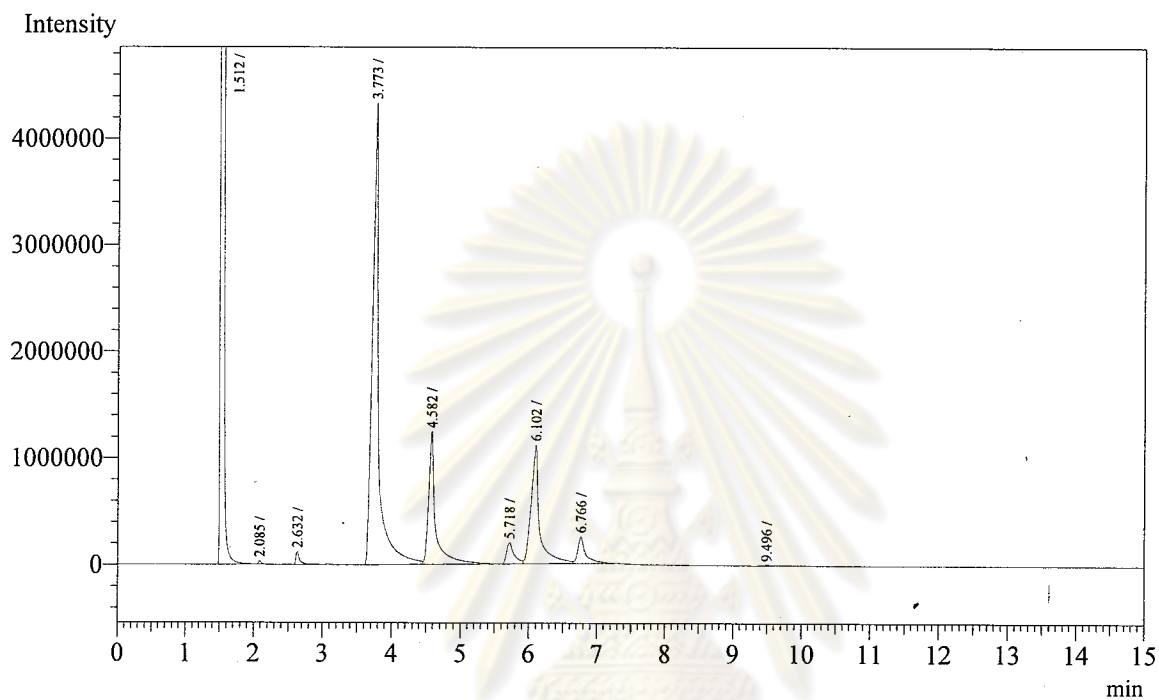


Peak#	Ret. Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.513	754523298	00356839	0.000		
2	2.084	79481	29712	0.000		
3	2.629	422132	109406	0.000		
4	3.750	24800226	3869206	0.000		
5	4.565	9082917	1264180	0.000	V	
6	5.689	1360509	182498	0.000		
7	6.065	8975315	1035502	0.000	V	
8	6.731	1852793	227969	0.000	V	
9	9.444	64247	6580	0.000		
Total		801160918	07081892			

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Analysis Date & Time : 2/7/2552 10:12:36
 User Name : Admin
 Vial# : 2
 Sample Name : S06
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA6.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

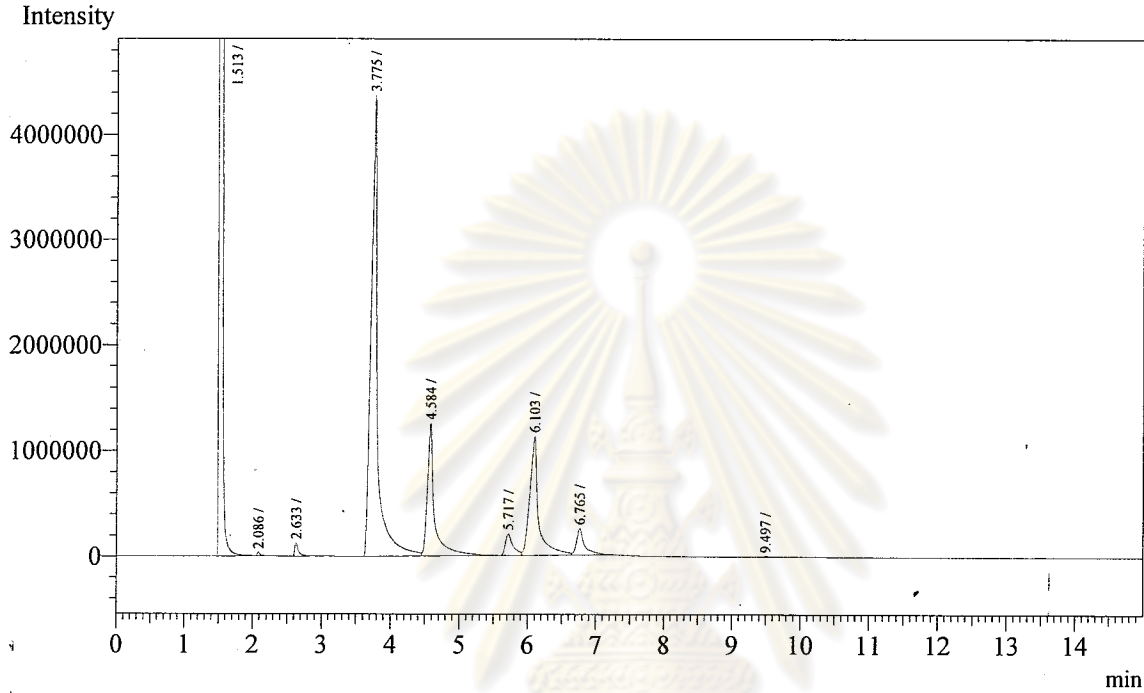


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.512	755023194	09409173	0.000	S	
2	2.085	89070	33335	0.000		
3	2.632	477013	122237	0.000		
4	3.773	28558120	4322330	0.000		
5	4.582	8953322	1243989	0.000	V	
6	5.718	1555019	199942	0.000		
7	6.102	9755963	1115167	0.000	V	
8	6.766	2031817	250841	0.000	V	
9	9.496	73553	7453	0.000		
Total		806517071	16704467			

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 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/7/2552 9:55:57
 User Name : Admin
 Vial# : 1
 Sample Name : S07
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA5.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

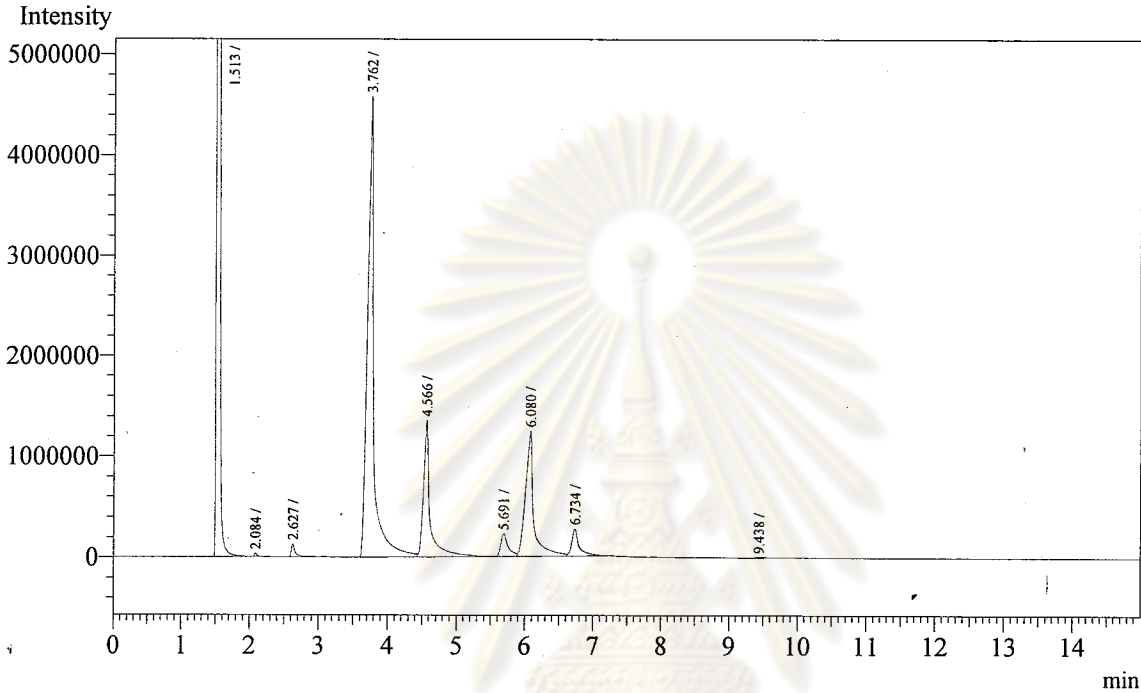


Peak#	Ret. Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.513	749587239	97562063	0.000	S		
2	2.086	93137	33965	0.000			
3	2.633	497299	124749	0.000			
4	3.775	29530266	4333071	0.000			
5	4.584	9253552	1256549	0.000	V		
6	5.717	1619443	205483	0.000			
7	6.103	10139071	1128266	0.000	V		
8	6.765	2107263	252013	0.000	V		
9	9.497	79835	7622	0.000			
Total		802907105	04903781				

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 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 15/7/2552 12:36:30
 User Name : Admin
 Vial# : 18
 Sample Name : S08
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA33.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

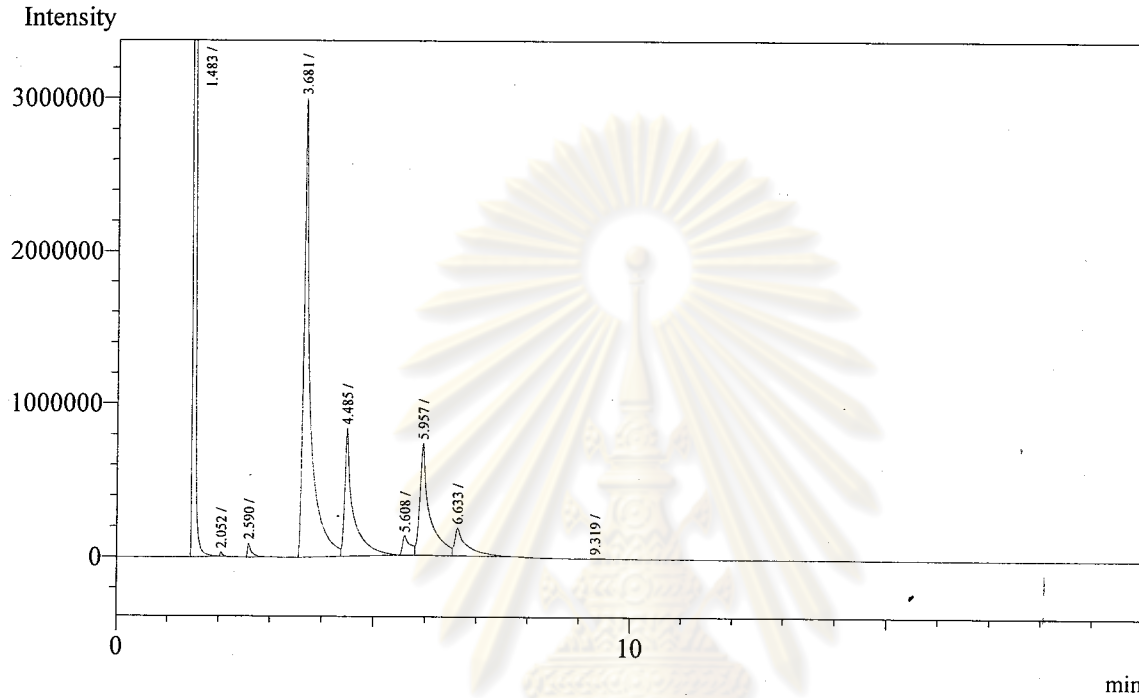


Peak#	Ret. Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.513	747796657	01081924	0.000		
2	2.084	86040	35638	0.000		
3	2.627	470913	130087	0.000		
4	3.762	29514891	4555544	0.000		
5	4.566	9210193	1356144	0.000	V	
6	5.691	1677922	224159	0.000		
7	6.080	10452503	1241472	0.000	V	
8	6.734	2036452	267500	0.000	V	
9	9.438	80672	8563	0.000		
Total		801326243	08901031			

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 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 19/6/2552 9:01:29
 User Name : Admin
 Vial# : 1
 Sample Name : S09
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA1.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

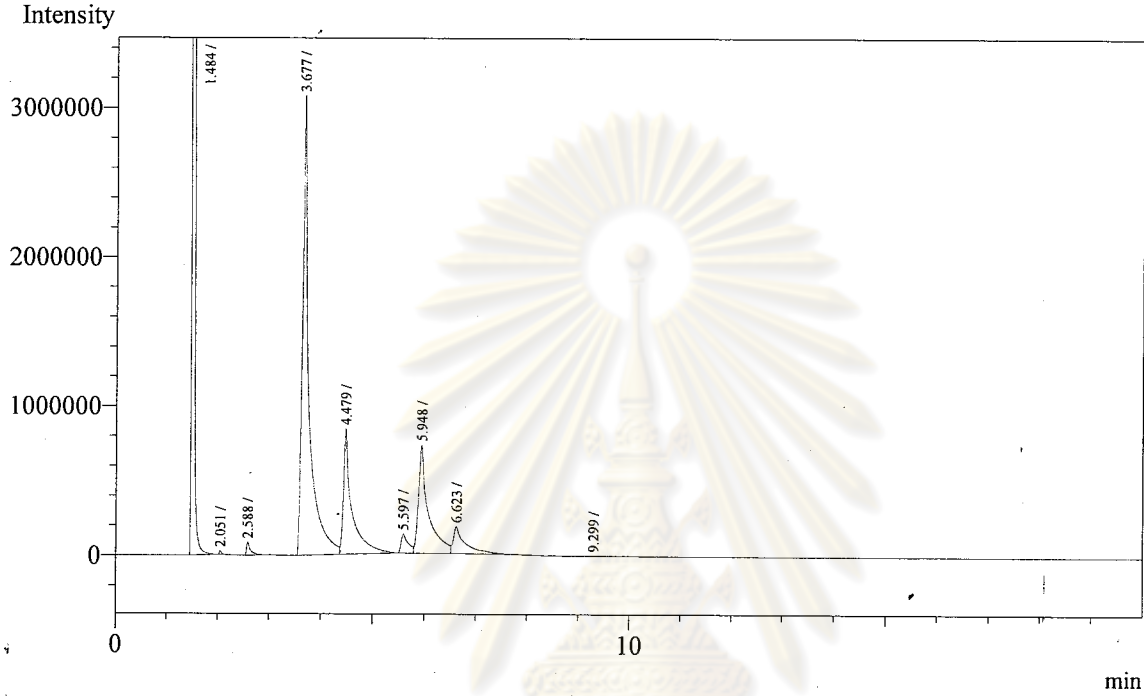


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.483	800935013	34888474	0.000		
2	2.052	96363	28891	0.000		
3	2.590	487334	91284	0.000		
4	3.681	27142500	2998516	0.000		
5	4.485	9448479	834114	0.000	V	
6	5.608	1290466	127144	0.000		
7	5.957	9697322	730511	0.000	V	
8	6.633	2997747	184072	0.000	V	
9	9.319	50770	4382	0.000		
Total		852145994	39887388			

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Analysis Date & Time : 19/6/2552 9:44:48
 User Name : Admin
 Vial# : 3
 Sample Name : S10
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA3.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

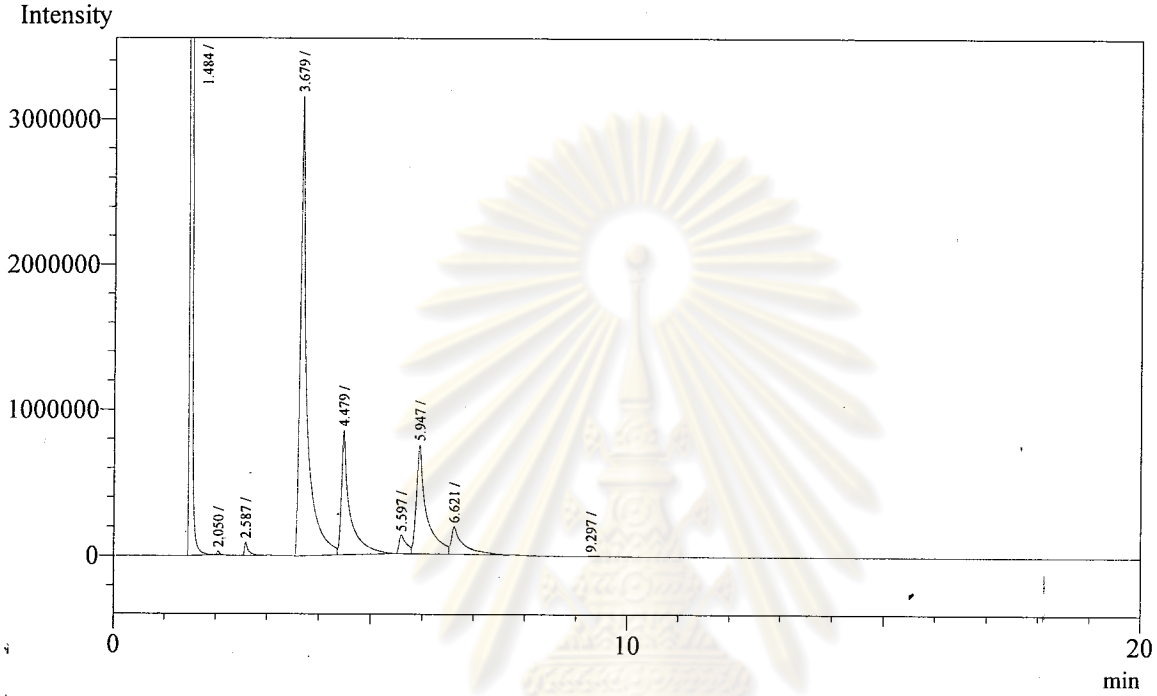


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.484	791093832	27107594	0.000		
2	2.051	86170	25455	0.000		
3	2.588	472231	88618	0.000		
4	3.677	27719643	3068812	0.000		
5	4.479	9456039	842281	0.000	V	
6	5.597	1194851	127144	0.000		
7	5.948	9608988	725775	0.000	V	
8	6.623	2956468	181528	0.000	V	
9	9.299	52263	4410	0.000		
Total		842640485	32171617			

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Analysis Date & Time : 19/6/2552 10:06:28
 User Name : Admin
 Vial# : 4
 Sample Name : S11
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA4.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

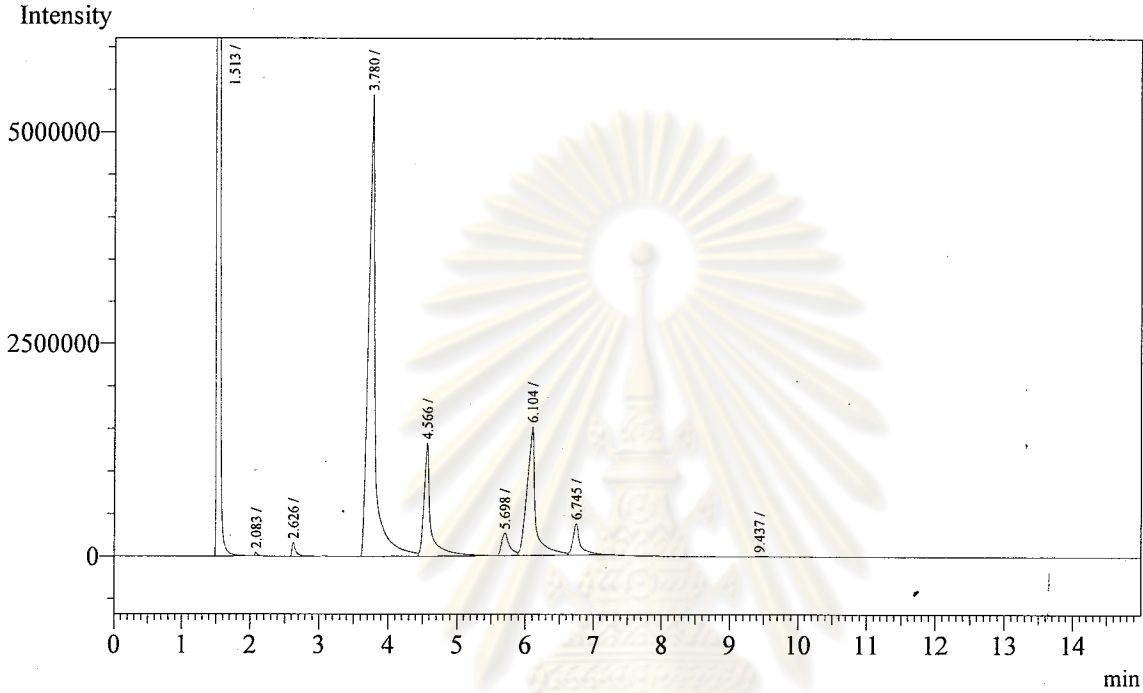


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.484	790265173	30073798	0.000		
2	2.050	95563	28374	0.000		
3	2.587	489256	92860	0.000		
4	3.679	28194837	3147689	0.000		
5	4.479	9560385	853333	0.000	V	
6	5.597	1255875	127540	0.000		
7	5.947	10037933	745573	0.000	V	
8	6.621	3009520	188684	0.000	V	
9	9.297	55118	4348	0.000		
Total		842963660	35262199			

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Analysis Date & Time : 15/7/2552 11:13:08
 User Name : Admin
 Vial# : 13
 Sample Name : S12
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA28.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

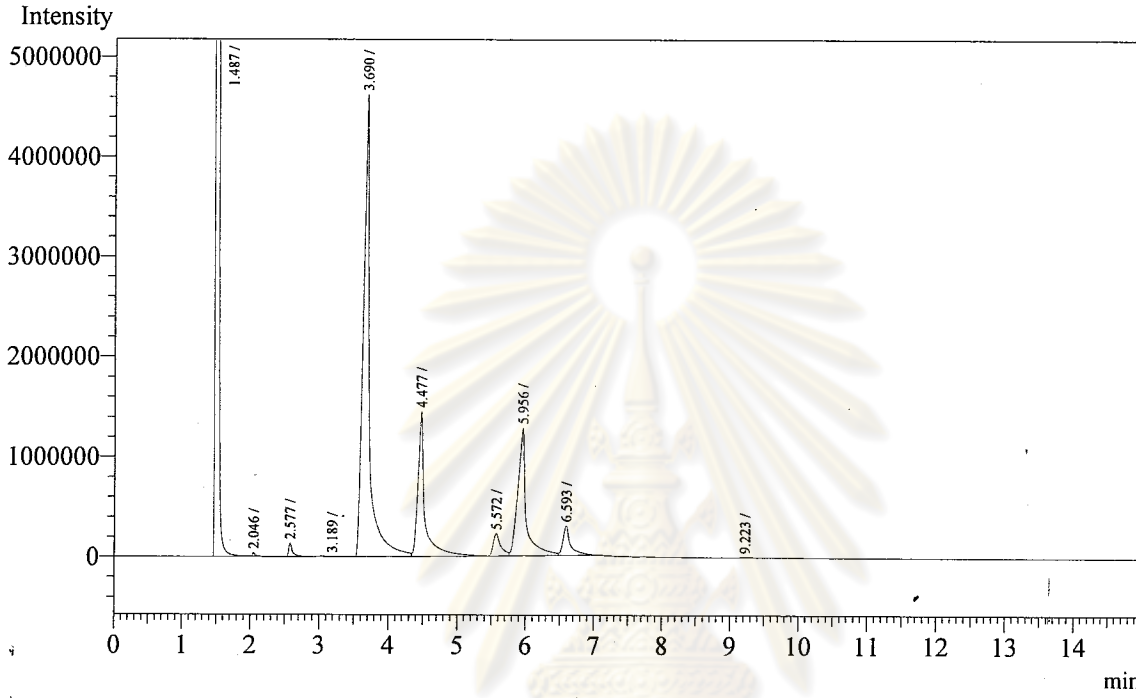


Peak#	Ret. Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.513	746380692	00787331	0.000	S		
2	2.083	124507	45296	0.000			
3	2.626	638409	163653	0.000			
4	3.780	34669883	5382429	0.000			
5	4.566	12253507	1326764	0.000	V		
6	5.698	2126162	267575	0.000			
7	6.104	13400412	1508282	0.000	V		
8	6.745	2777501	369516	0.000	V		
9	9.437	101442	10765	0.000			
Total		812472515	09861611				

ศูนย์วิทยาศาสตร์พยากร
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 6/8/2552 10:44:08
 User Name : Admin
 Vial# : 8
 Sample Name : S13
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA41.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

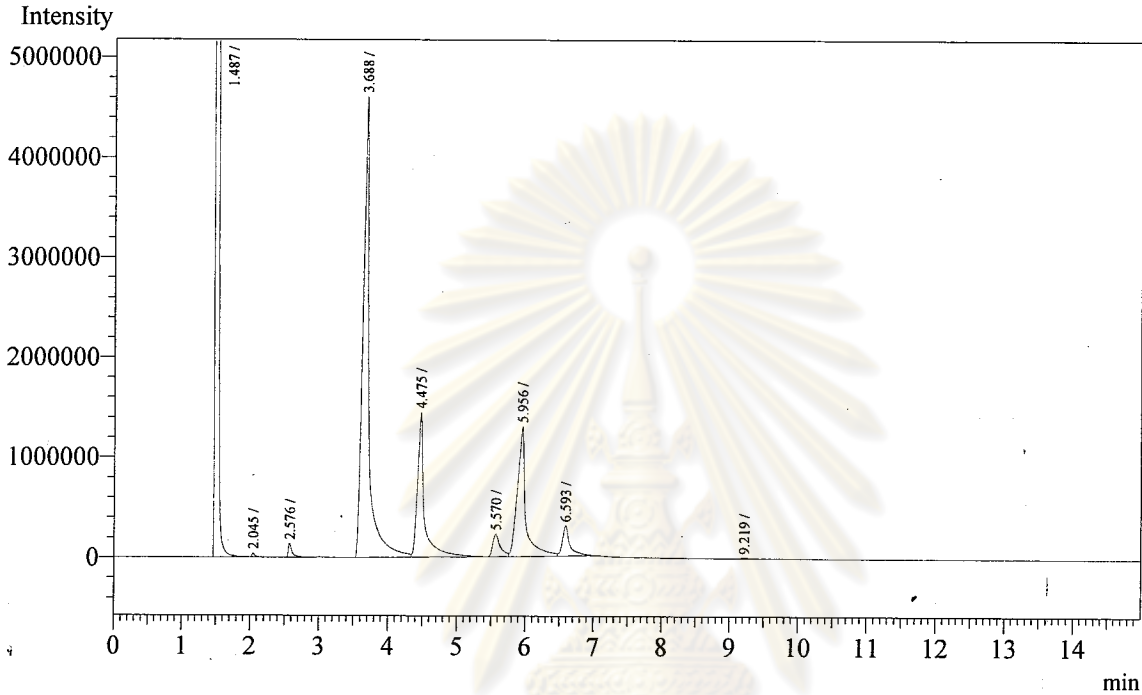


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.487	808612329	51738306	0.000	S		
2	2.046	105591	38040	0.000			
3	2.577	532408	135452	0.000			
4	3.189	86659	6027	0.000	V		
5	3.690	30775131	4602058	0.000			
6	4.477	10269785	1445506	0.000	V		
7	5.572	1706670	224856	0.000			
8	5.956	11075486	1277642	0.000	V		
9	6.593	2301722	297983	0.000	V		
10	9.223	83750	8845	0.000			
Total		865549531	59774715				

จุฬาลงกรณ์มหาวิทยาลัย
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 6/8/2552 11:00:45
 User Name : Admin
 Vial# : 9
 Sample Name : S14
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA42.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

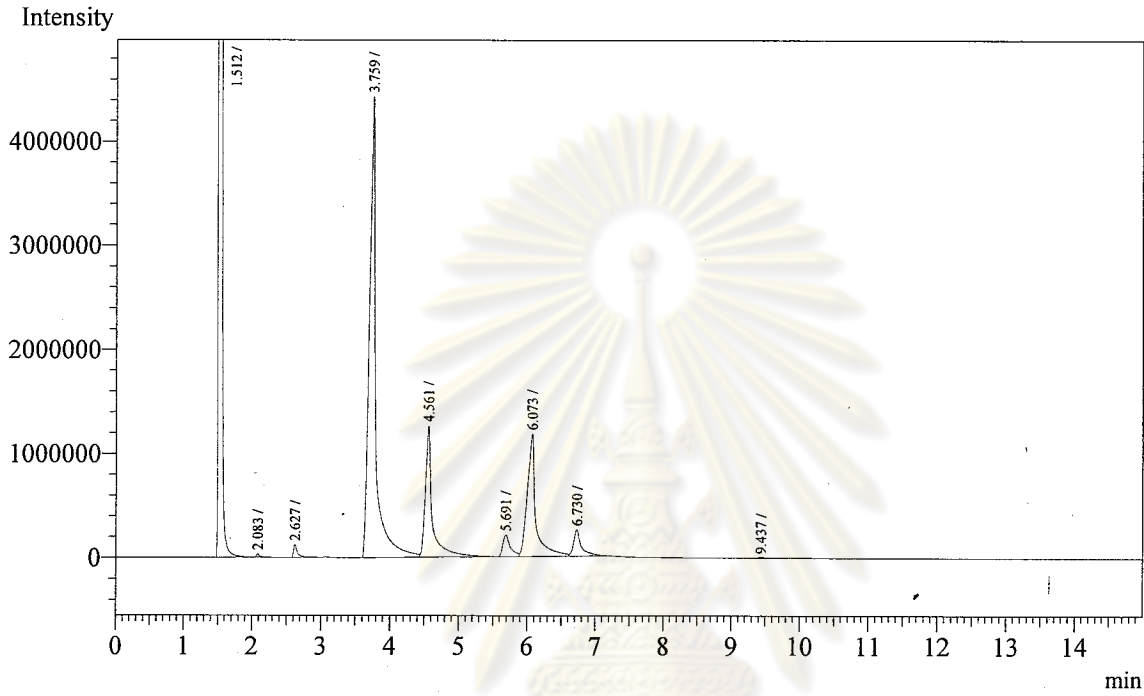


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.487	808450139	54372731	0.000	S	
2	2.045	112886	40837	0.000		
3	2.576	550587	139386	0.000		
4	3.688	30325493	4581149	0.000		
5	4.475	10267114	1444326	0.000	V	
6	5.570	1735706	225764	0.000		
7	5.956	11273745	1299075	0.000	V	
8	6.593	2374199	304689	0.000	V	
9	9.219	85957	9023	0.000		
Total		865175826	62416980			

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 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 15/7/2552 12:03:05
 User Name : Admin
 Vial# : 16
 Sample Name : S15
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA31.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

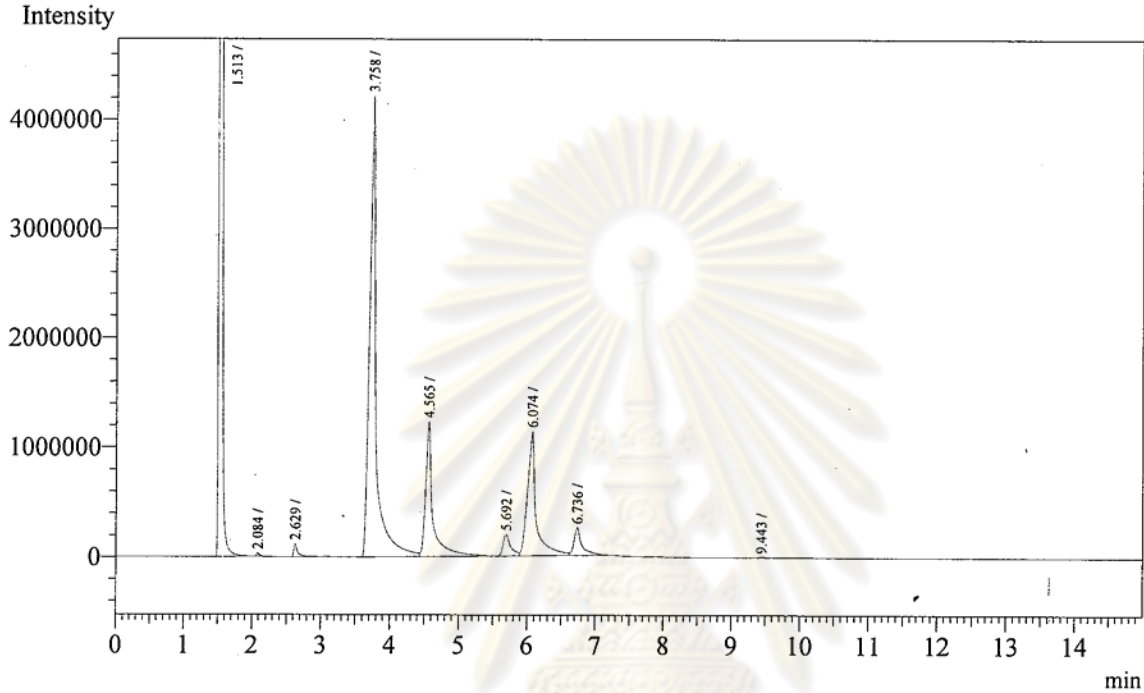


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.512	753106509	02798843	0.000	S		
2	2.083	99810	34541	0.000			
3	2.627	467569	125180	0.000			
4	3.759	29548289	4422467	0.000			
5	4.561	9134218	1264616	0.000	V		
6	5.691	1631758	209352	0.000			
7	6.073	10344322	1180576	0.000	V		
8	6.730	2057657	254688	0.000	V		
9	9.437	81064	7912	0.000			
Total		806471196	10298175				

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 15/7/2552 11:29:48
 User Name : Admin
 Vial# : 14
 Sample Name : S16
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA29.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

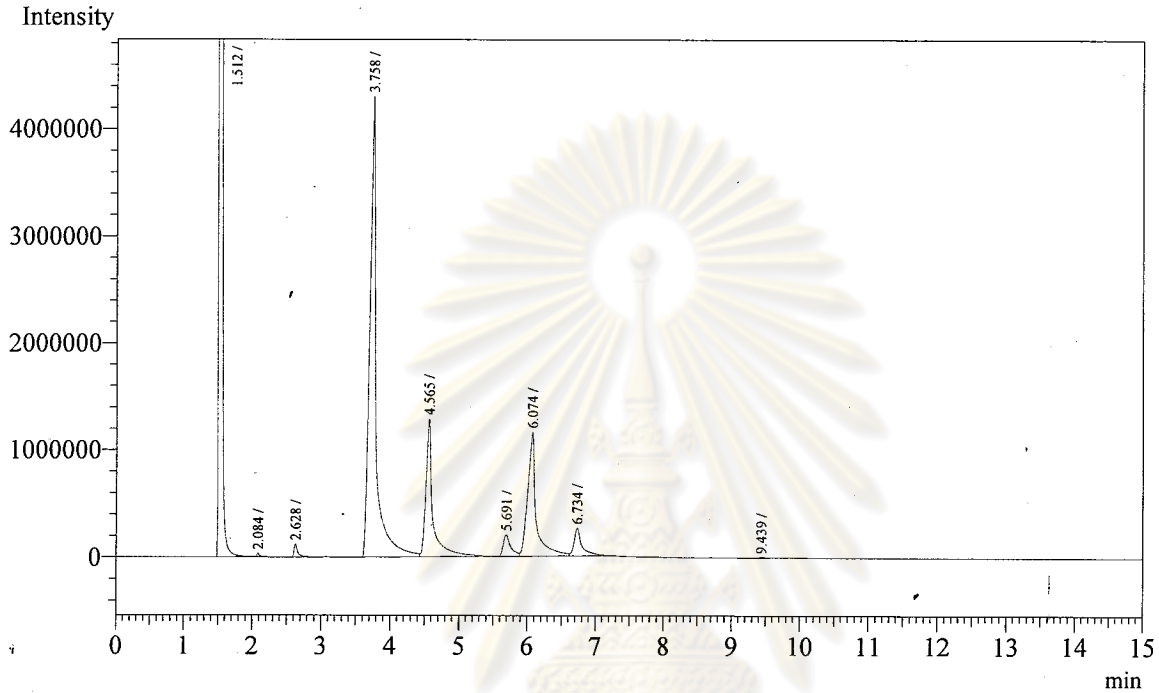


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.513	755923819	03892731	0.000		
2	2.084	90844	32911	0.000		
3	2.629	476444	119155	0.000		
4	3.758	28292780	4202860	0.000		
5	4.565	9132400	1230812	0.000	V	
6	5.692	1533375	198688	0.000		
7	6.074	10099913	1135977	0.000	V	
8	6.736	2102368	255717	0.000	V	
9	9.443	72341	7296	0.000		
Total		807724284	11076147			

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 15/7/2552 10:56:29
 User Name : Admin
 Vial# : 12
 Sample Name : S17
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA27.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

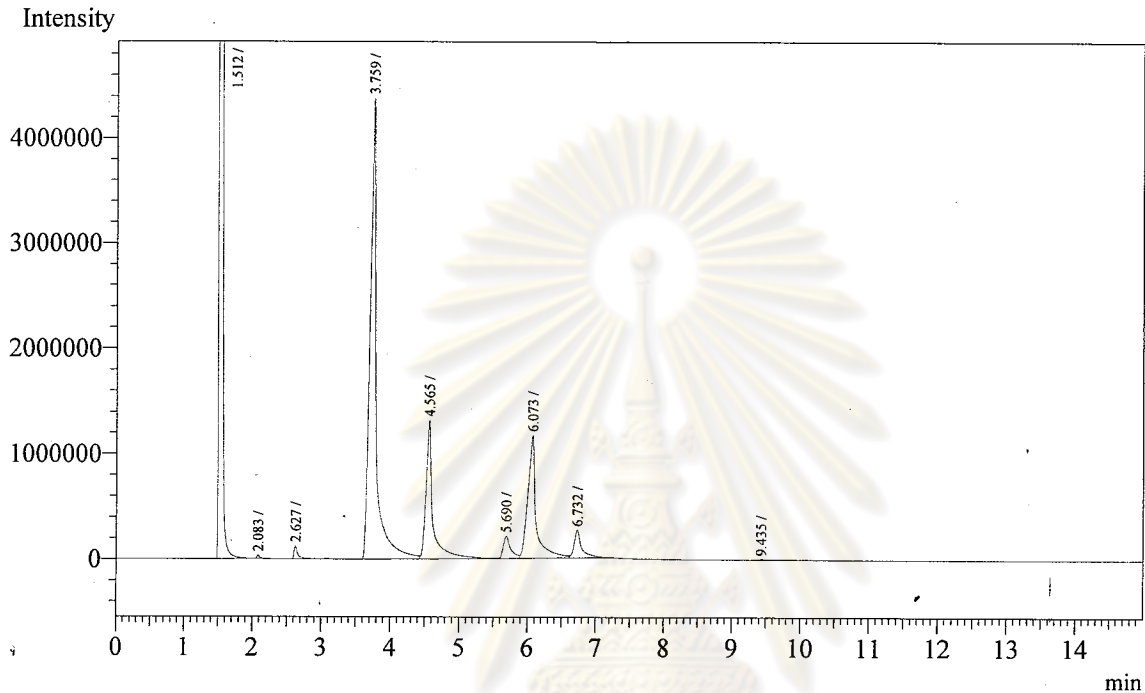


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.512	755471000	07007616	0.000		
2	2.084	92117	33818	0.000		
3	2.628	478482	122793	0.000		
4	3.758	28308578	4286612	0.000		
5	4.565	9202235	1283275	0.000	V	
6	5.691	1551850	202759	0.000		
7	6.074	10055528	1158508	0.000	V	
8	6.734	2085945	259424	0.000	V	
9	9.439	76782	7538	0.000		
Total		807322517	14362343			

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 15/7/2552 11:46:27
 User Name : Admin
 Vial# : 15
 Sample Name : S18
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA30.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

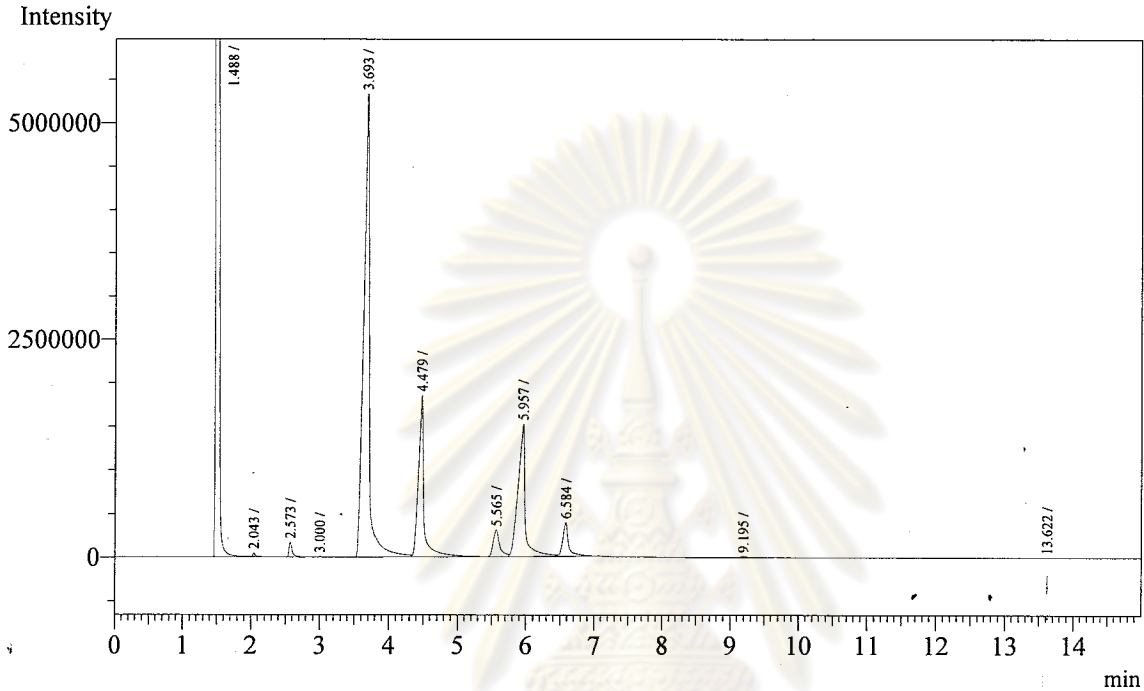


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.512	753629198	02778230	0.000	S	
2	2.083	91654	34252	0.000		
3	2.627	478187	125236	0.000		
4	3.759	28626516	4370457	0.000		
5	4.565	9207621	1315215	0.000	V	
6	5.690	1581111	210625	0.000		
7	6.073	9969249	1166469	0.000	V	
8	6.732	2067139	265963	0.000	V	
9	9.435	74127	7732	0.000		
Total		805724802	10274179			

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/9/2552 13:14:39
 User Name : Admin
 Vial# : 19
 Sample Name : S19
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA108.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

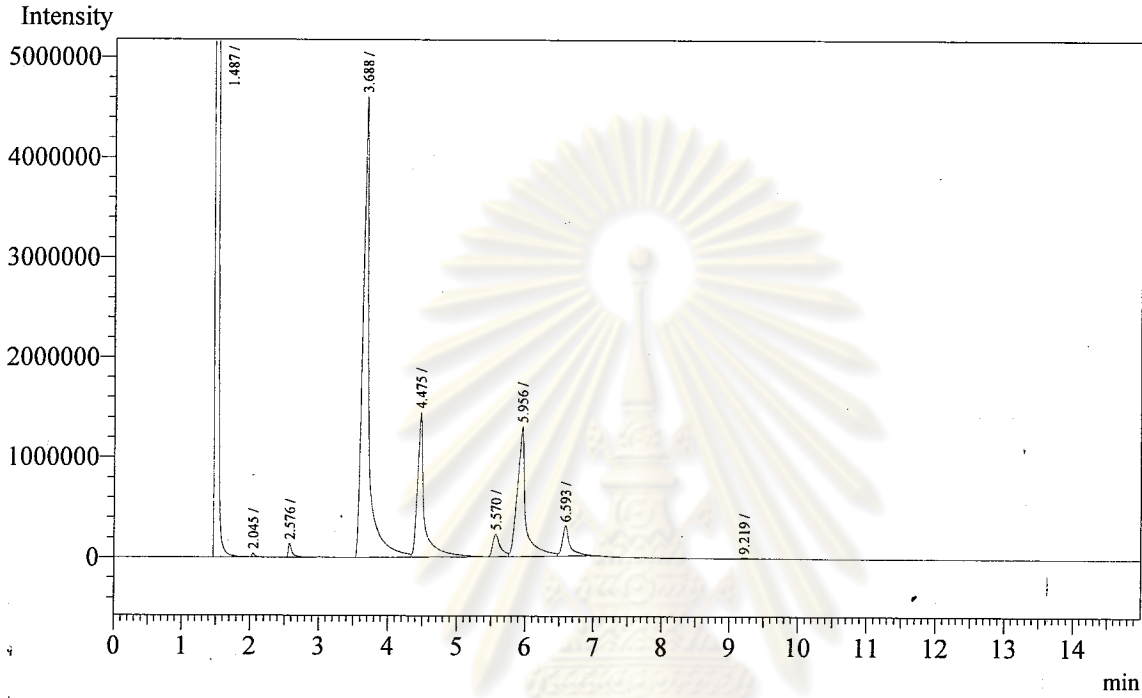


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.488	827613706	73534565	0.000	S	
2	2.043	106554	46249	0.000		
3	2.573	549144	170179	0.000		
4	3.000	34440	7382	0.000		
5	3.693	31754333	5307974	0.000		
6	4.479	10468119	1848887	0.000	V	
7	5.565	2014176	304215	0.000		
8	5.957	11324711	1518989	0.000	V	
9	6.584	2332394	387254	0.000	V	
10	9.195	103754	12777	0.000		
11	13.622	35599	1028	0.000		
Total		886336930	83139499			

จุฬาลงกรณ์มหาวิทยาลัย
 วิทยาลัยพยาบาล

Analysis Date & Time : 6/8/2552 11:00:45
 User Name : Admin
 Vial# : 9
 Sample Name : S20
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA42.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

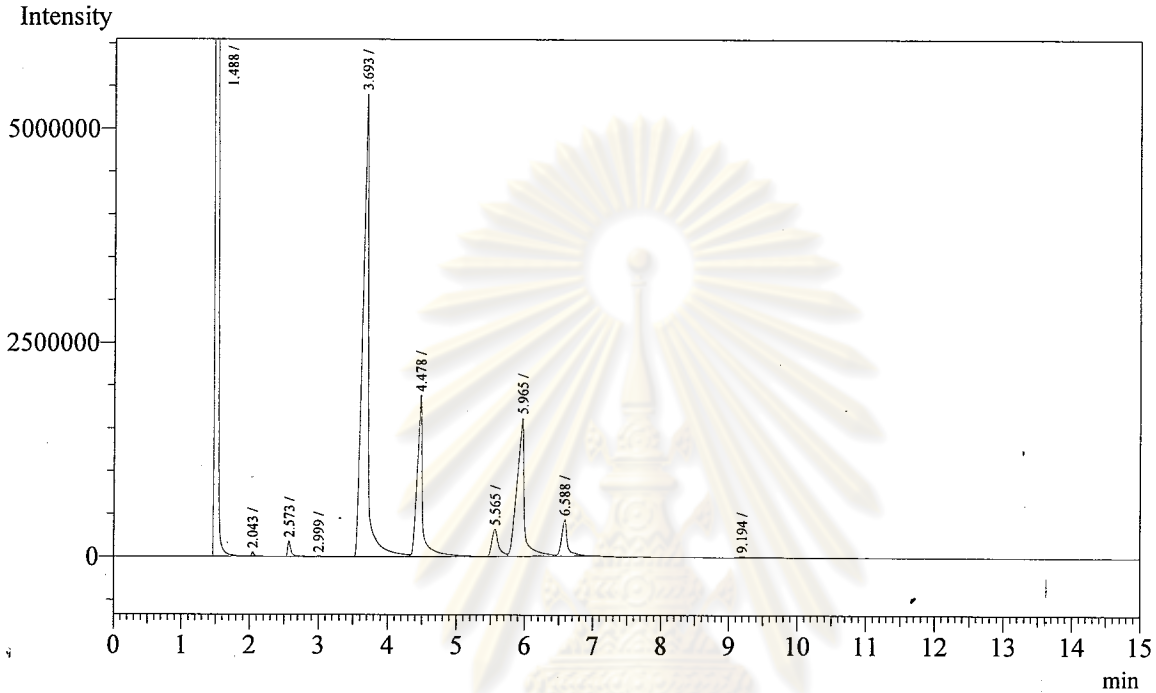


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.487	808450139	54372731	0.000	S	
2	2.045	112886	40837	0.000		
3	2.576	550587	139386	0.000		
4	3.688	30325493	4581149	0.000		
5	4.475	10267114	1444326	0.000	V	
6	5.570	1735706	225764	0.000		
7	5.956	11273745	1299075	0.000	V	
8	6.593	2374199	304689	0.000	V	
9	9.219	85957	9023	0.000		
Total		865175826	62416980			

ศูนย์วิทยาศาสตร์การ
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/9/2552 13:31:19
 User Name : Admin
 Vial# : 20
 Sample Name : S21
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA109.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

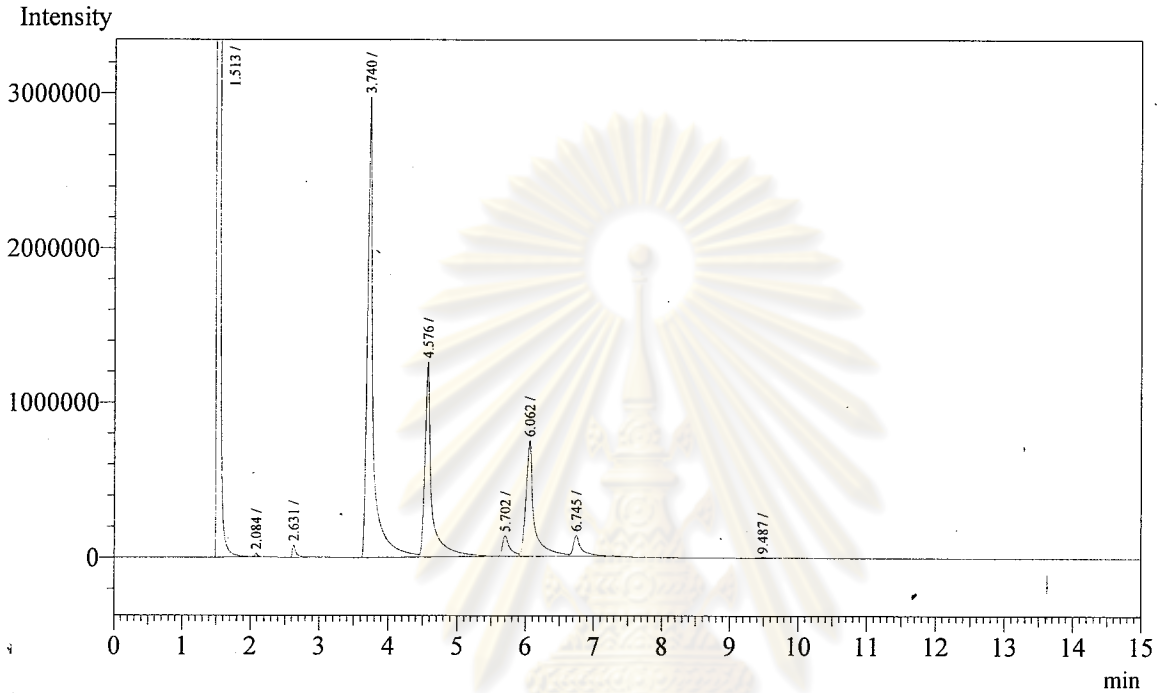


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.488	834774712	77638284	0.000	S		
2	2.043	118324	50709	0.000			
3	2.573	589372	179236	0.000			
4	2.999	30098	7341	0.000			
5	3.693	32627277	5379383	0.000			
6	4.478	10720408	1884046	0.000	V		
7	5.565	2119856	315079	0.000			
8	5.965	12507103	1607171	0.000	V		
9	6.588	2593499	426765	0.000	V		
10	9.194	109972	13612	0.000			
Total		896190621	87501626				

จุฬาลงกรณ์มหาวิทยาลัย
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/7/2552 10:29:14
 User Name : Admin
 Vial# : 3
 Sample Name : S22
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA7.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

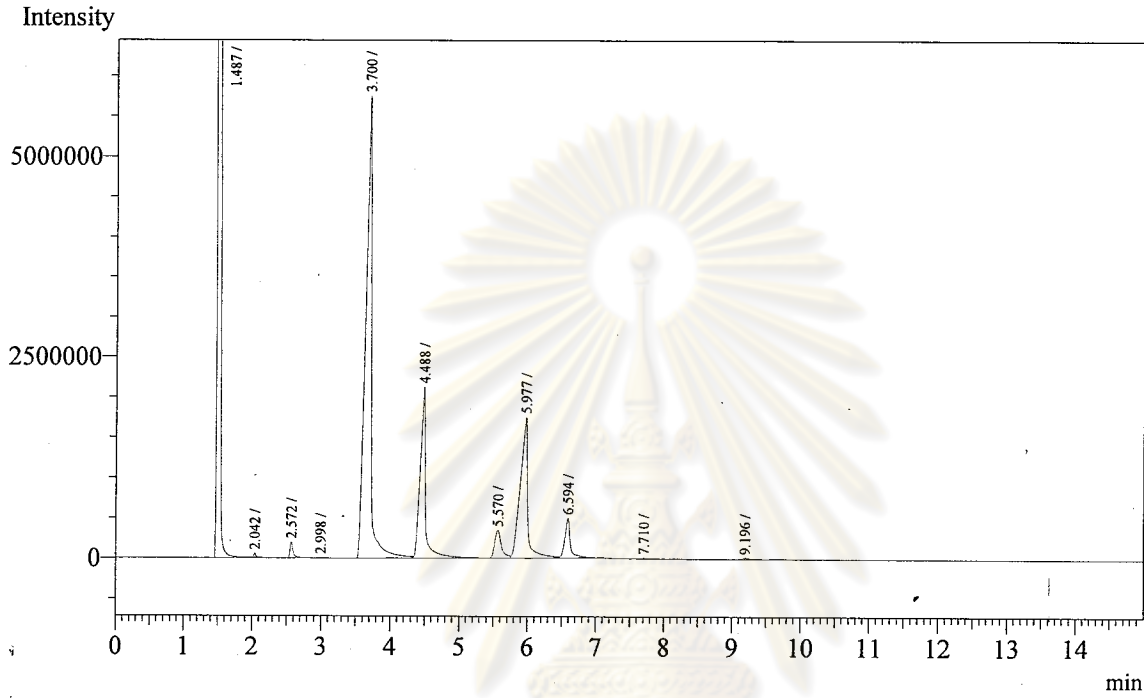


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.513	757243170	08278370	0.000			
2	2.084	54641	23188	0.000			
3	2.631	286226	79780	0.000			
4	3.740	28659112	2965309	0.000			
5	4.576	8774653	1259183	0.000	V		
6	5.702	984470	134176	0.000			
7	6.062	6176289	745633	0.000	V		
8	6.745	1103337	134139	0.000	V		
9	9.487	50596	4912	0.000			
Total		803332494	13624690				

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/9/2552 14:04:38
 User Name : Admin
 Vial# : 22
 Sample Name : S23
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA111.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

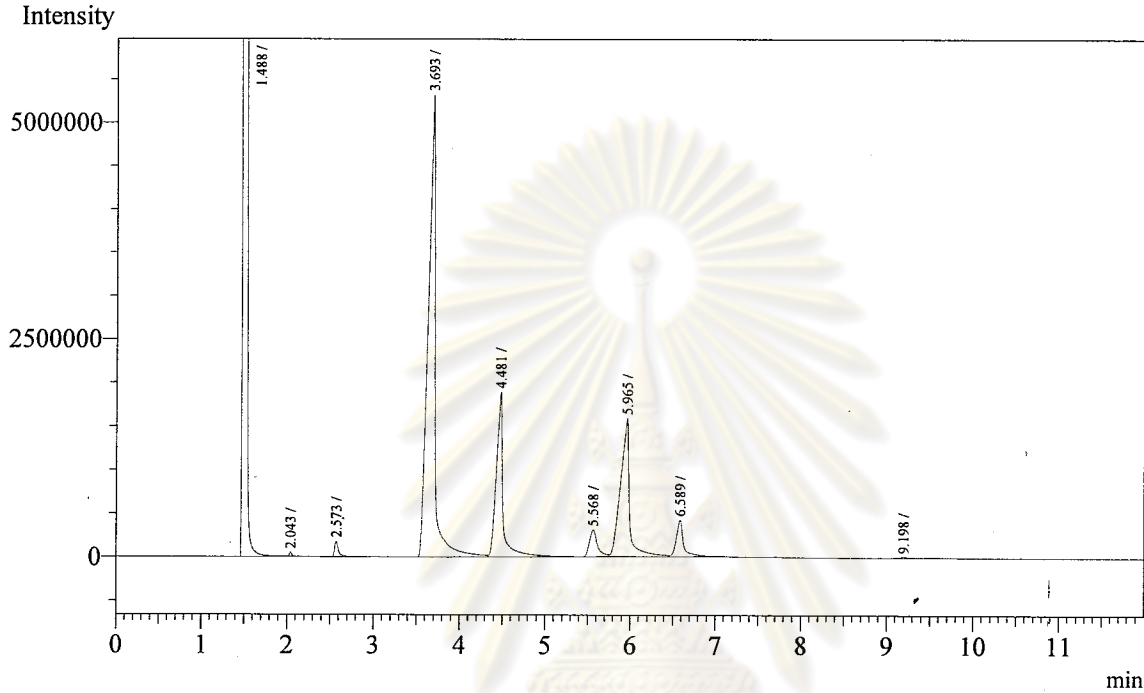


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.487	826104685	75373151	0.000	S	
2	2.042	120961	53704	0.000		
3	2.572	598854	194073	0.000		
4	2.998	35599	8202	0.000		
5	3.700	34890373	5674627	0.000		
6	4.488	11826982	2111407	0.000	V	
7	5.570	2327905	344081	0.000		
8	5.977	13522284	1738641	0.000	V	
9	6.594	2779452	488141	0.000	V	
10	7.710	35837	6872	0.000		
11	9.196	124094	15967	0.000		
Total		892367026	86008866			

จุฬาลงกรณ์มหาวิทยาลัย
 วิทยาลัย
 วิทยาการ

Analysis Date & Time : 2/9/2552 14:22:06
 User Name : Admin
 Vial# : 23
 Sample Name : S24
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA112.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

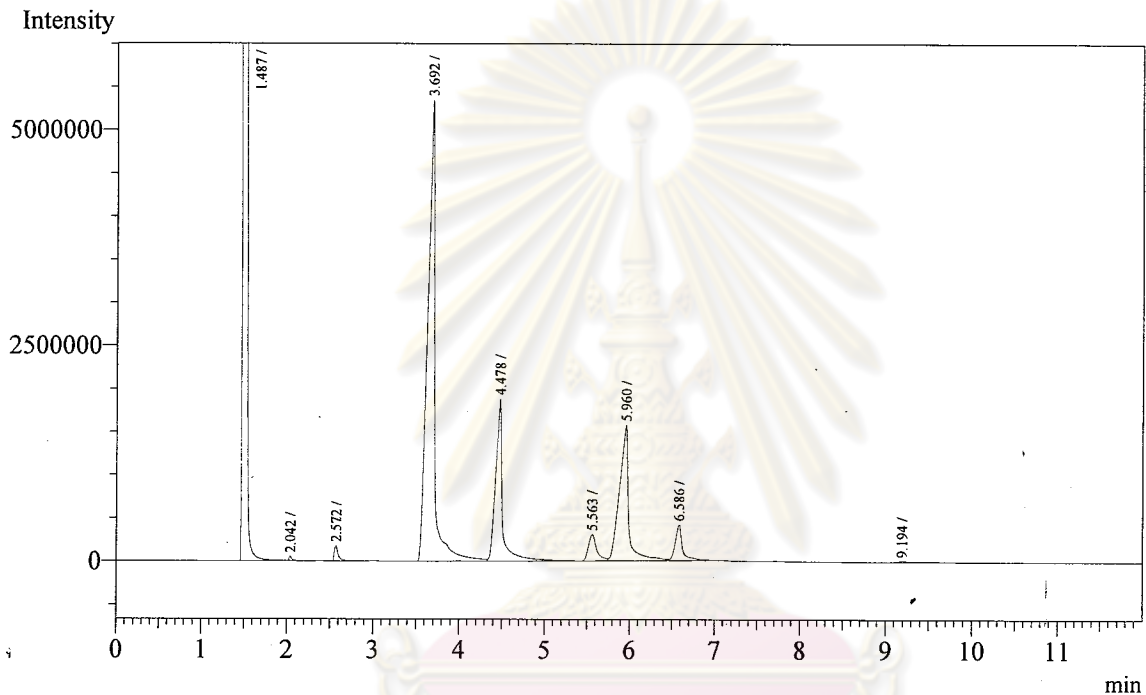


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.488	825262687	73762180	0.000	S	
2	2.043	109722	49471	0.000		
3	2.573	561478	176995	0.000		
4	3.693	31297857	5290828	0.000		
5	4.481	10527344	1876938	0.000	V	
6	5.568	2053457	307624	0.000		
7	5.965	11982817	1581221	0.000	V	
8	6.589	2483140	420079	0.000	V	
9	9.198	107880	13353	0.000		
Total		884386382	83478689			

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/9/2552 15:30:24
 User Name : Admin
 Vial# : 28
 Sample Name : S25
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

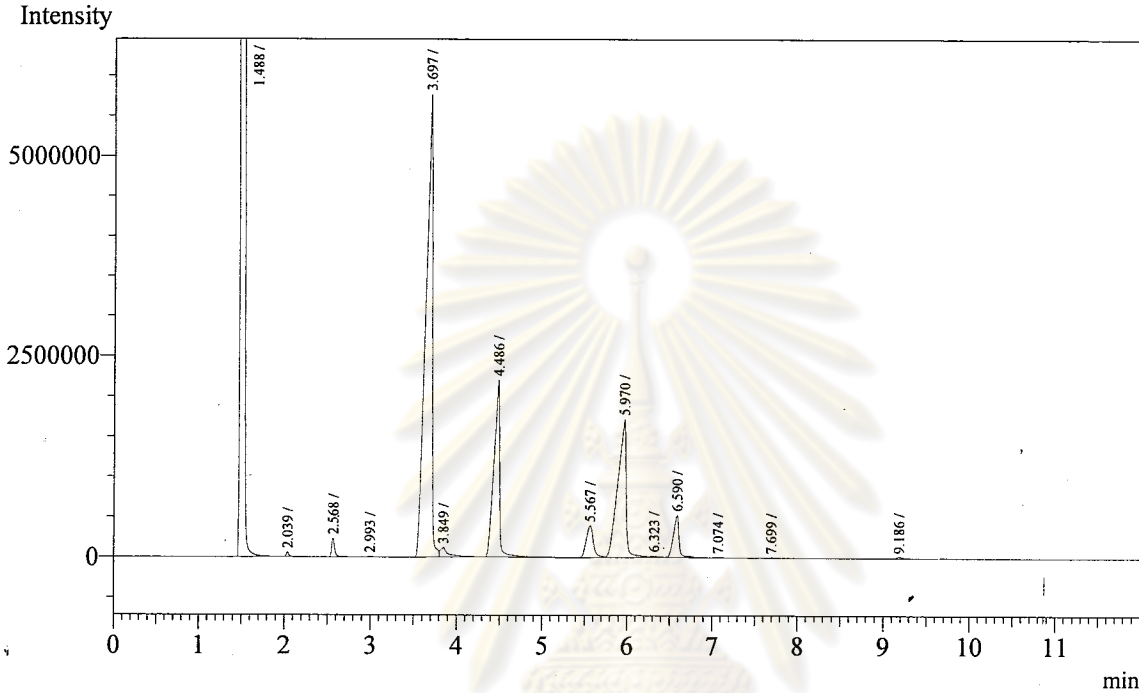
Data Name : D:\ACID\FA117.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm



Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.487	831230470	75411791	0.000	S	
2	2.042	114931	49213	0.000		
3	2.572	574195	175450	0.000		
4	3.692	32226678	5271410	0.000		
5	4.478	10746368	1881427	0.000	V	
6	5.563	2071553	306239	0.000		
7	5.960	12105930	1573316	0.000	SV	
8	6.586	2505147	416081	0.000	V	
9	9.194	107865	13389	0.000		
Total		891683137	85098316			

Analysis Date & Time : 2/9/2552 15:44:03
 User Name : Admin
 Vial# : 29
 Sample Name : S26
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA118.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

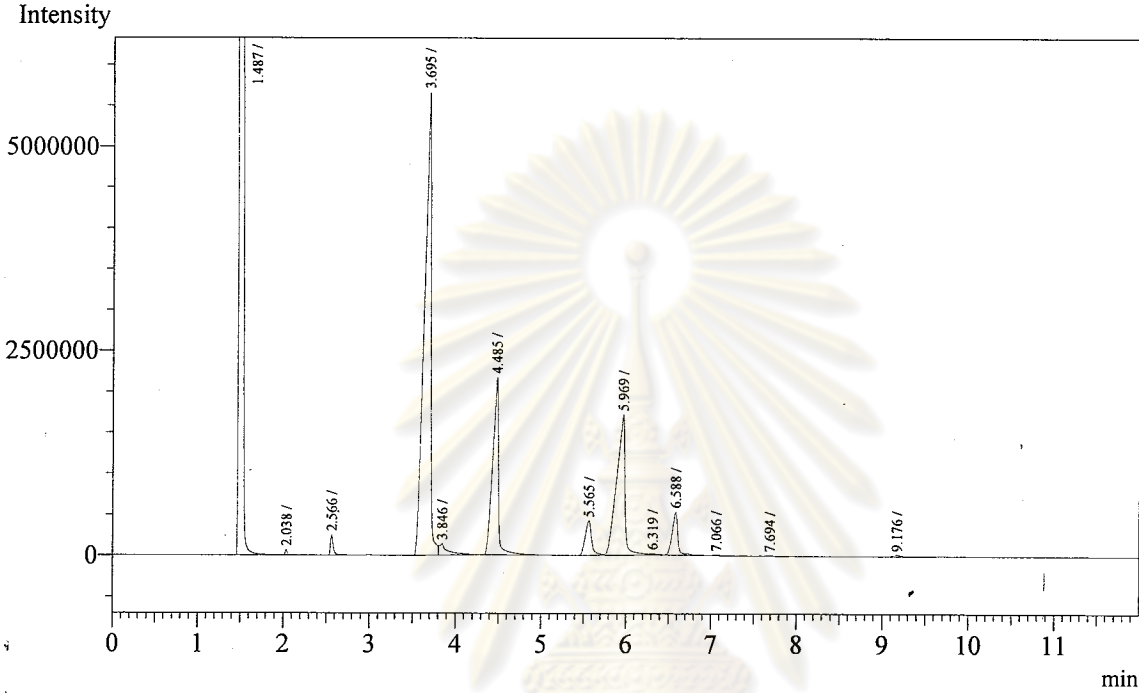


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.488	868417773	79583135	0.000	S	
2	2.039	111420	59822	0.000		
3	2.568	565020	231796	0.000	S	
4	2.993	30074	9583	0.000		
5	3.697	31024503	5708505	0.000		
6	3.849	814338	121126	0.000	V	
7	4.486	10428743	2191265	0.000		
8	5.567	2297221	394893	0.000		
9	5.970	11867109	1712107	0.000	SV	
10	6.323	46848	12238	0.000	T	
11	6.590	2422493	522713	0.000	S	
12	7.074	44012	9051	0.000		
13	7.699	34138	7038	0.000		
14	9.186	134038	19146	0.000		
Total		928237730	90582418			

จุฬาลงกรณ์มหาวิทยาลัย
 วิทยาลัยการสัตวแพทยศาสตร์
 วิทยาลัยการสัตวแพทยศาสตร์

Analysis Date & Time : 2/9/2552 15:16:45
 User Name : Admin
 Vial# : 27
 Sample Name : S27
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA116.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

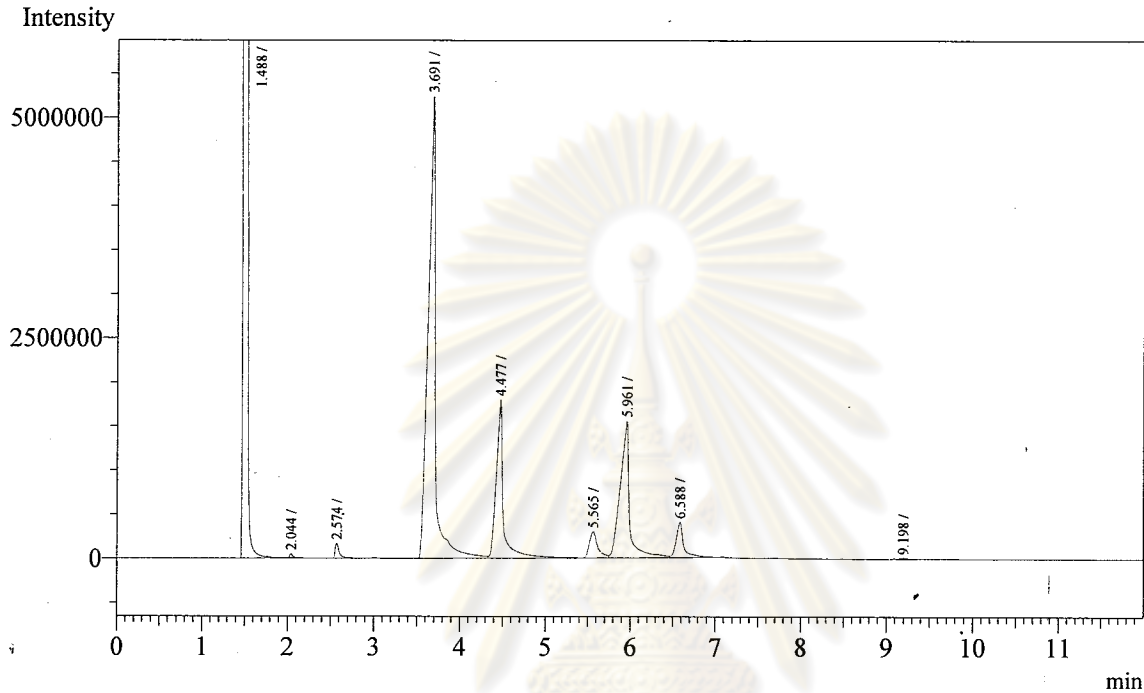


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.487	862616996	03448812	0.000	S	
2	2.038	114595	64080	0.000		
3	2.566	575173	248087	0.000		
4	3.695	30592515	5594708	0.000		
5	3.846	1025813	139163	0.000	V	
6	4.485	10412986	2147218	0.000		
7	5.565	2265248	422193	0.000		
8	5.969	12052158	1708217	0.000	SV	
9	6.319	36814	10558	0.000	T	
10	6.588	2451572	526299	0.000	S	
11	7.066	38208	8562	0.000		
12	7.694	31994	6875	0.000		
13	9.176	129182	19412	0.000		
Total		922343254	14344184			

จุฬาลงกรณ์มหาวิทยาลัย
 วิทยาลัยพยาบาล

Analysis Date & Time : 2/9/2552 14:49:29
 User Name : Admin
 Vial# : 25
 Sample Name : S28
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA114.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

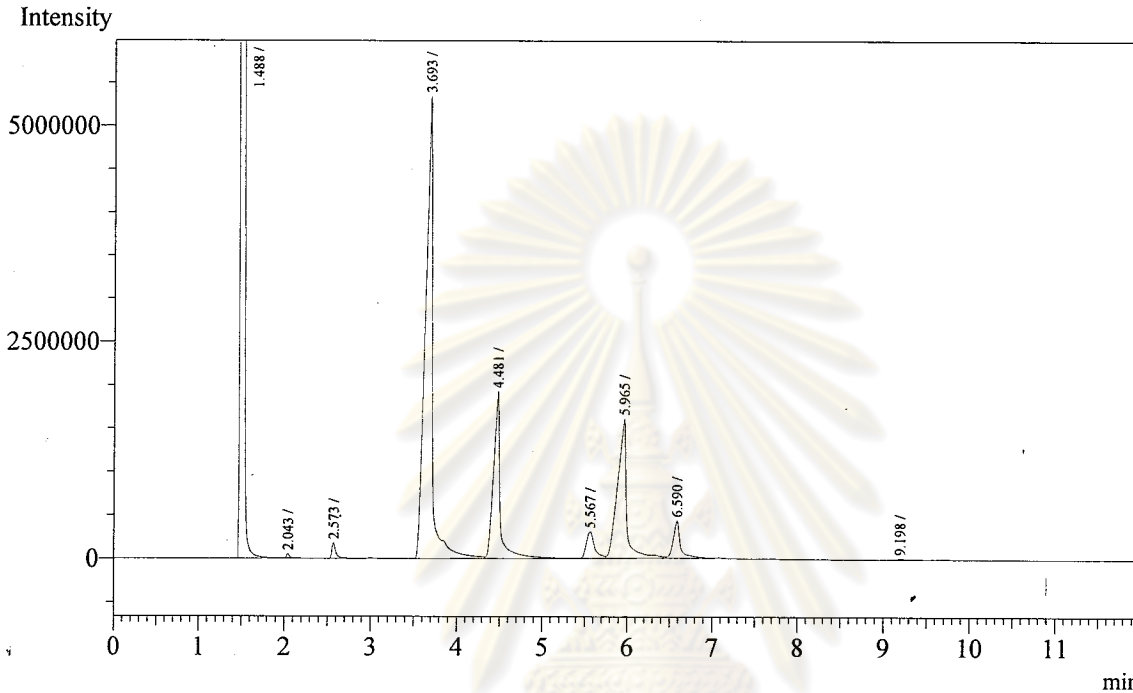


Peak#	Ret. Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.488	834726219	76752134	0.000	S		
2	2.044	114785	47949	0.000			
3	2.574	569391	170473	0.000			
4	3.691	31766455	5177077	0.000			
5	4.477	10431885	1796351	0.000	V		
6	5.565	2028979	295474	0.000			
7	5.961	12041473	1547990	0.000	SV		
8	6.588	2480936	398828	0.000	V		
9	9.198	105298	13005	0.000	V		
Total		894265421	86199281				

ศูนย์วิทยาศาสตร์พยากร
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/9/2552 15:03:07
 User Name : Admin
 Vial# : 26
 Sample Name : S29
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA115.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

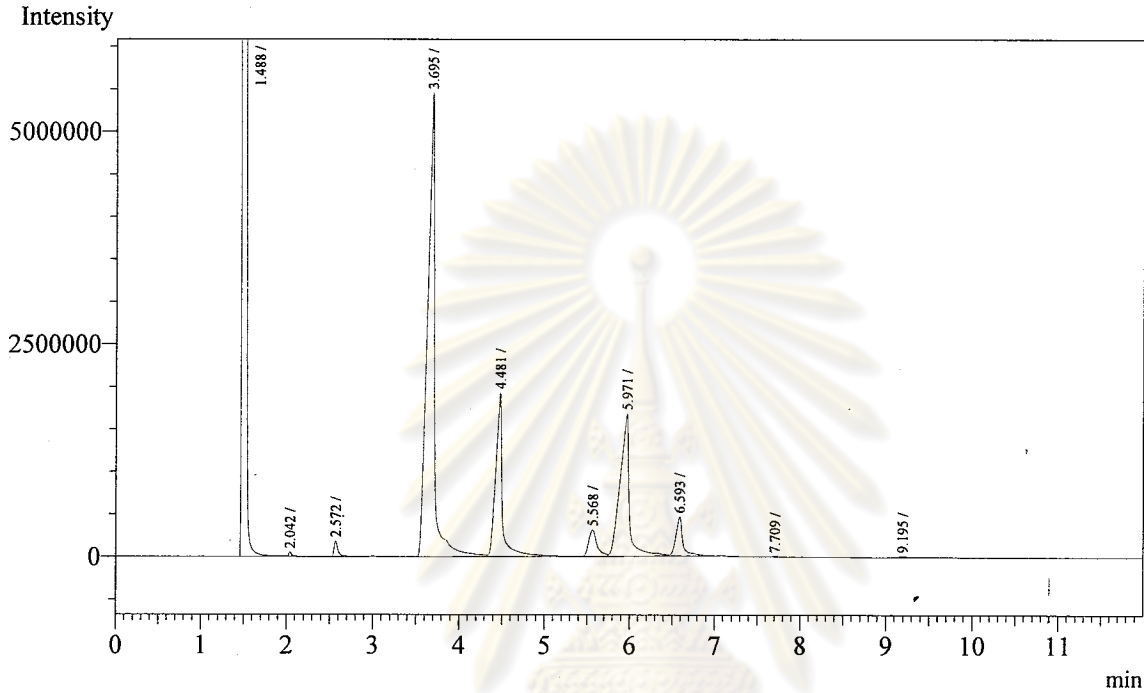


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.488	834409209	78747981	0.000	S	
2	2.043	116191	50587	0.000		
3	2.573	572558	177822	0.000		
4	3.693	31969107	5311503	0.000		
5	4.481	10803333	1913555	0.000	V	
6	5.567	2083034	306977	0.000		
7	5.965	12308477	1601172	0.000	SV	
8	6.590	2554422	428868	0.000	V	
9	9.198	109532	13633	0.000		
Total		894925863	88552098			

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 2/9/2552 14:35:50
 User Name : Admin
 Vial# : 24
 Sample Name : S30
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA113.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

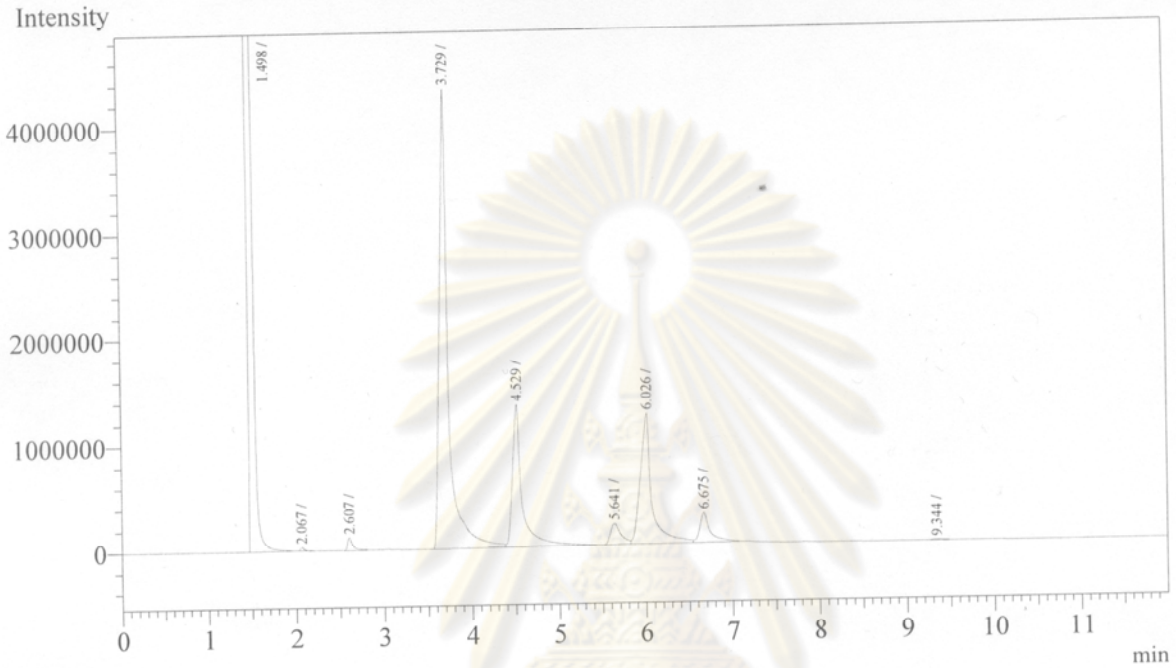


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.488	825801937	74556342	0.000	S	
2	2.042	116512	51533	0.000		
3	2.572	592348	184069	0.000		
4	3.695	32697168	5404359	0.000		
5	4.481	10746058	1921619	0.000	V	
6	5.568	2117537	310208	0.000		
7	5.971	13080052	1677557	0.000	SV	
8	6.593	2708443	459846	0.000	V	
9	7.709	38211	6995	0.000		
10	9.195	112790	14160	0.000		
Total		888011056	84586688			

จุฬาลงกรณ์มหาวิทยาลัย
 วิทยาลัยพยาบาล

Analysis Date & Time : 23/9/2552 12:20:56
 User Name : Admin
 Vial# : 1
 Sample Name : S31
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA120.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm

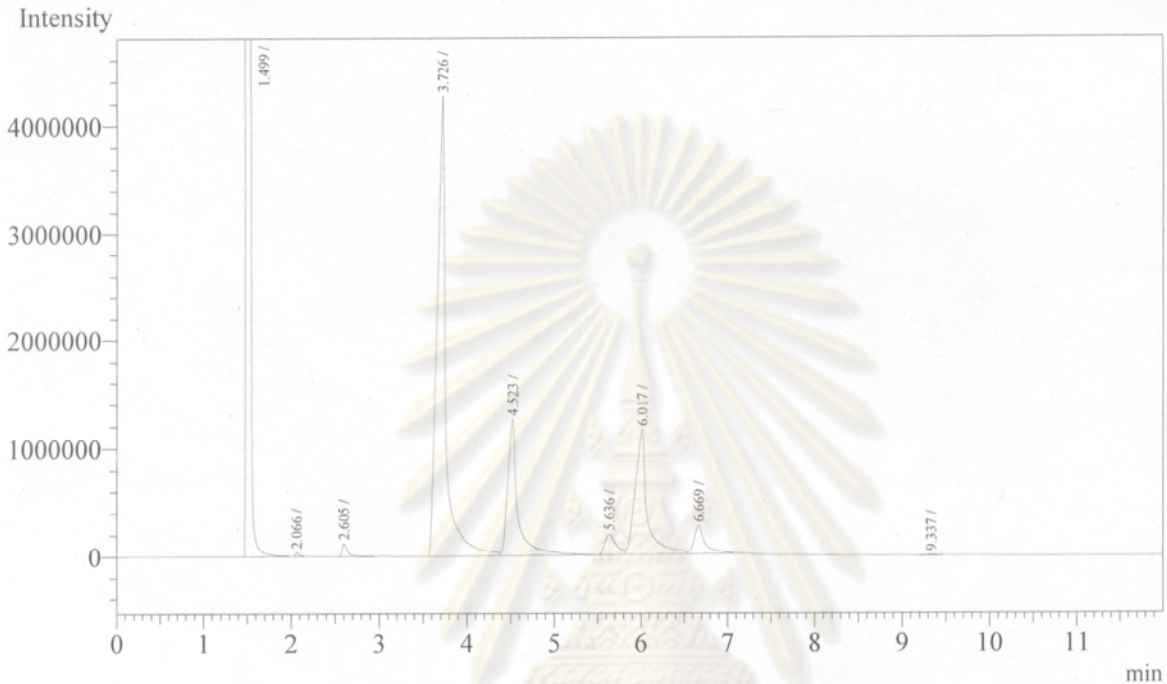


Peak#	Ret.Time	Area	Height	Conc.	Unit Mark ID#	Cmpd Name
1	1.498	841365412	59912699	0.000		
2	2.067	102835	35344	0.000		
3	2.607	516430	121743	0.000		
4	3.729	29917281	4307670	0.000		
5	4.529	10324640	1337273	0.000	V	
6	5.641	1703747	201958	0.000		
7	6.026	11254661	1226554	0.000	V	
8	6.675	2321611	281735	0.000	V	
9	9.344	81147	7949	0.000		
Total		897587764	67432925			

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

Analysis Date & Time : 23/9/2552 12:34:35
 User Name : Admin
 Vial# : 2
 Sample Name : S32
 Sample ID : UNK-0001
 Sample Type : Unknown
 Injection Volume : 1.00
 ISTD Amount :

Data Name : D:\ACID\FA121.gcd
 Method Name : D:\ACID\FATTY ACID-2.gcm



Peak#	Ret.Time	Area	Height	Conc.	Unit Mark	ID#	Cmpd Name
1	1.499	861831613	80169979	0.000			
2	2.066	107481	37301	0.000			
3	2.605	538943	123109	0.000			
4	3.726	30440893	4261588	0.000			
5	4.523	10266877	1254664	0.000	V		
6	5.636	1711736	191889	0.000			
7	6.017	11144708	1153326	0.000	V		
8	6.669	2289799	261481	0.000	V		
9	9.337	82961	7505	0.000			
Total		918415011	87460842				

ศูนย์วิทยาศาสตร์
 จุฬาลงกรณ์มหาวิทยาลัย

BIOGRAPHY

Mr. Chatri Kriengyakul was born in Kamphaengpetch province on April 25, 1967. He graduated from Chulalongkorn University and received the Bachelor degree in Mechanical Engineering in 1989. Since graduated from the university he worked for engineering and construction in petrochemical and oil & gas industries. In 2006 he entered the Graduate School of Chulalongkorn University to continue his study of Chemical Engineering.



ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย