VALUE ADDED OF CRUDE GLYCEROL FROM BIODIESEL PRODUCTION BY THIN FILM DISTILLATION

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A SPECIAL PROJECT SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIRMENTS FOR THE DEGREE OF BACHELOR OF SCIENCE
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KING MONGKUT'S INSTITUTEOF TECHNOLOGY LADKRABANG
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ABSTRACT

Dealing with the excessive glycerol from biodiesel production is now becoming a dilemma to the industry. The crude glycerol contains too many contaminants to find a useful application while a high purity has a numerous applications in industry.

The purpose of this study is to increase value-added utilization of crude glycerol from biodiesel production:purification by thin film distillation. The goal was achieved through a several objectives; to design and construct the thin film distillation system for glycerol purification and to study the appropriate condition and parameters for the constructed system. Thin film distillation system consist of 5 parts which are thin film evaporator unit , fractionating column, condenser, product and trap drums and vacuum pump. Thin film evaporator unit was designed to be a rotatable cylindrical tube with 18 cm. diameter and 25cm. length in a 30 liters stainless steel bath. HYSYS simulation program was used for designed a fractionating column unit which contain 7 stainless steel trays in a 18.4 cm. diameter and 118 cm height stainless steel column. After connected all of 5 parts together, thin film distillation system was operated with 4 liters of crude glycerol from Patum Vegetable Oil .,Co.Ltd. The performance of the thin film distillation system was tested. It was found that the thin film distillation system can be operated at a condition of 20 mbar and 180°c and can be used to distil crude glycerol.

Acknowledgement

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Chapter1

Introduction

1.1 Motivation

Our economy and lifestyle rely on the use of fossil fuel.Today the expanding economics of developing nations such as China and India are reflecting a dramatically increasing need for energy. Nearly 90% of our energy consumption come from oil, natural gas and coal. Approximately 10% came from nuclear power and renewable energy with questions of global energy availability and security looming large in our future, this unbalanced consumption is clearly unsustainable and a threat to this an future generation. This has led to a search for technologies that generate fuels and materials from renewable carbon sources, such as plant biomass. Depending on the component of the biomass used as feedstock and the technology employed to transform this component into the desired product, at least three general platforms have been envisioned: the sugar, synthesis gas, and oil platforms. The sugar and oil platforms are the most well-established today, with bioethanol and biodiesel being examples of their commercial products, respectively. Biodiesel is produced by the transesterification of vegetable oils or animal fats with an alcohol to produce esters.

Biodiesel is a fuel that has much the same characteristics as normal diesel oil; on the contrary, it is derived from vegetable oils or animal fat not petroleum. To produce biodiesel from these oils, they are subjected to a chemical reaction, which is called transesterification. A residue forms due to transesterification, called glycerol

Glycerol is generated about 10% of total weight of synthesis biodiesel. The rapid increase in biodiesel production is resulting in a worldwide surplus of glycerol. For every 10 kg of oil used to manufacture biodiesel, about 1 kg glycerol will be produced. Once considered a valuable co-product in the biodiesel process, crude glycerol is rapidly becoming a waste product with an attached disposal cost.

The current annual amount of glycerol arising from this biodiesel production will continue to rise proportionally compare to the existing world market for pure glycerol for high quality industrial applications still stable. This means that either new applications for glycerol need to be developed and or the existing pathways need to be expanded. This should be possible as there are more than a thousand potential applications for glycerol can be identified

Dealing with the excessive glycerol from biodiesel production is now becoming a dilemma to the industry. The crude glycerol possesses very low value due to the impurities present. There is an urgent demand that alternative yet value-added applications have to be found to ensure biodiesel industry's stronger and healthier growth.

Purified glycerol has hundreds of commercial uses from food to pharmaceuticals. Besides added value to glycerol, purification give many advantages include minimize waste from biodiesel production. This research will be conducted on crude glycerol for value-added by purification.

1.2 Objectives of this project

1. Design and construct the thin film distillation system for glycerol purification.

2. Study and find the appropriate condition and parameters for the constructed system.

1.3 Scope of study

1. Design and construct the thin film distillation system.

2. Study the performance of the thin film distillation system; distillation of crude glycerol from Patum Vegetable Oil Co.,Ltd.

3. Characterize the purified glycerol.

1.4 Expected results

1. To obtain an appropriate thin film distillation system which can purified crude glycerol.

2. To obtain purified glycerol.

3. To minimize waste from biodiesel production and to add value to crude glycerol.

Chapter 2

Literature Review

2.1 Biodiesel

Biodiesel is the name of a clean burning alternative fuel, produced from domestic, renewable resources. Biodiesel is not petroleum, but it can be blended at any level with petroleum diesel to create a biodiesel blend. It can be used in compression-ignition (diesel) engines with little or no modifications. Biodiesel is simple to use, biodegradable, nontoxic, and essentially free of sulfur and aromatics.

2.1.1 Biodiesel Production

Biodiesel is made through a chemical process called transesterification whereby the glycerol is separated from the fat or vegetable oil. The process leaves behind two products which are methyl esters (the chemical name for biodiesel) and glycerol (a valuable byproduct usually sold to be used in soaps and other products).

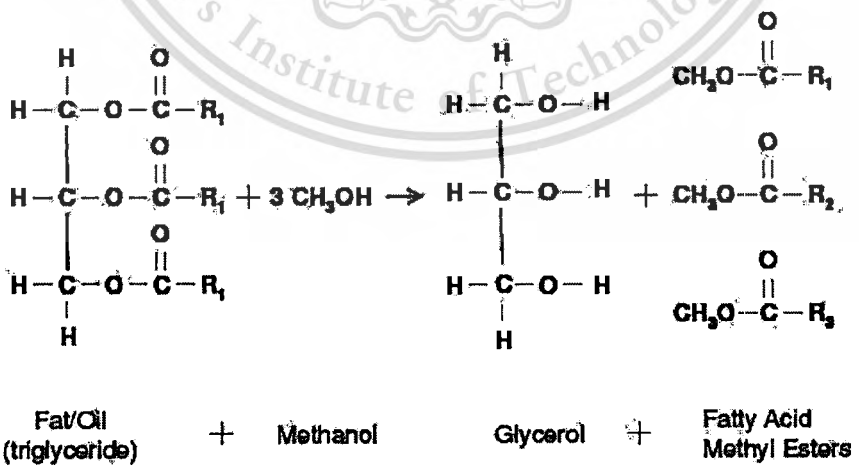


Figure 2.1 Tranesterification. [1]

There are three basic routes to ester production from oils and fats include;

1.) Base catalyzed transesterification of the oil with alcohol.
2.) Direct acid catalyzed esterification of the oil with methanol.
3.) Conversion of the oil to fatty acids, and then to Alkyl esters with acid catalysis.

The majority of the alkyl esters produced today are done with the base catalyzed reaction because it is the most economic for several reasons such as

1.) Low temperature (150 °F) and pressure (20 psi) processing.
2.) High conversion (98%) with minimal side reactions and reaction time.
3.) Direct conversion to methyl ester with no intermediate steps.
4.) Exotic materials of construction are not necessary.

A fat or oil is reacted with an alcohol, like methanol, in the presence of a catalyst to produce glycerol and methyl esters or biodiesel. The methanol is charged in excess to assist in quick conversion and recovered for reuse. The catalyst is usually sodium or potassium hydroxide which has already been mixed with the methanol. The most widely used vegetable oil feedstocks for biodiesel are soybean oil in the United States and rapeseed oil in the EU. However, both these feedstocks yield impressively low volumes of biodiesel per hectare when compared to the more tropical crops of Jatropha and Palm Oil found in Southeast Asia, Africa, and Latin America. [2]

Table 2.1 Yields of typical biodiesel feed stocks [3]

Crop	Kg oil/ha	Litres oil/ha	US gal/acre
Soybean	375	446	48
Rapeseed	1,000	1,190	127
Jatropha	1,590	1,892	202
Oil Palm	5,000	5,950	635

2.1.2 Biodiesel Production Capacity

Biodiesel has a dramatically increase in the production because of these main motives:

- An alternative product from agriculture

- Creating jobs in rural regions and boosting the agricultural sector

- Securing domestic energy supply and reducing dependence on fossil fuel imports

- Reducing man-made CO_2 emissions

- Reducing traffic emissions like CO, SO_2 and NO_x

World capacity, production and consumption of biodiesel grew on average by 32% per year during 2000–2005, and the industry looks set for even faster growth rates—115% per year for capacity, and 101% per year for demand—in the years to 2008 and beyond. [4]

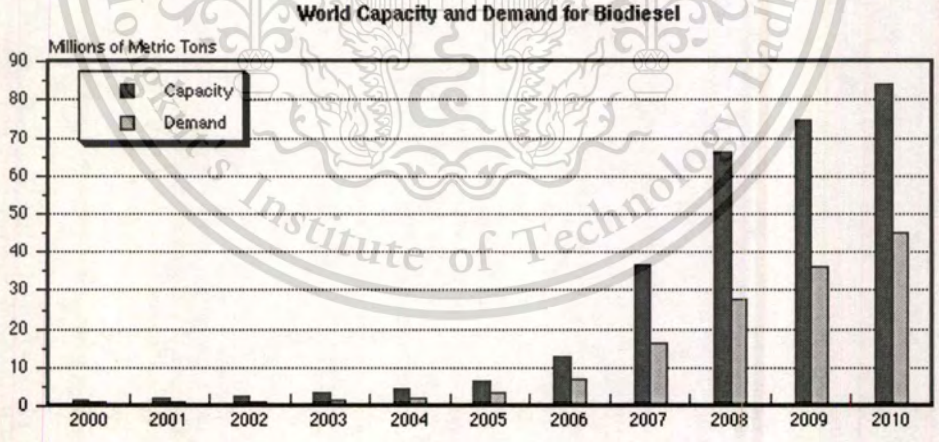


Figure 2.2 Diagram of world capacity and demand for biodiesel. [5]

Biodiesel production in the United States has been increasing dramatically recently, from 500,000 gallons in 1999 to 30 million gallons in 2004. The federal biodiesel tax incentive that went into effect January 2005 is causing biodiesel demand to climb even more and encourages entrepreneurs to invest in more biodiesel production facilities. The immediate production capacity of biodiesel is estimated to be 150 million gallon per year; this capacity can be doubled or tripled in a time frame of 12 months. [6]

The following pie charts show world production/consumption of biodiesel in 2005 and 2010 by region.

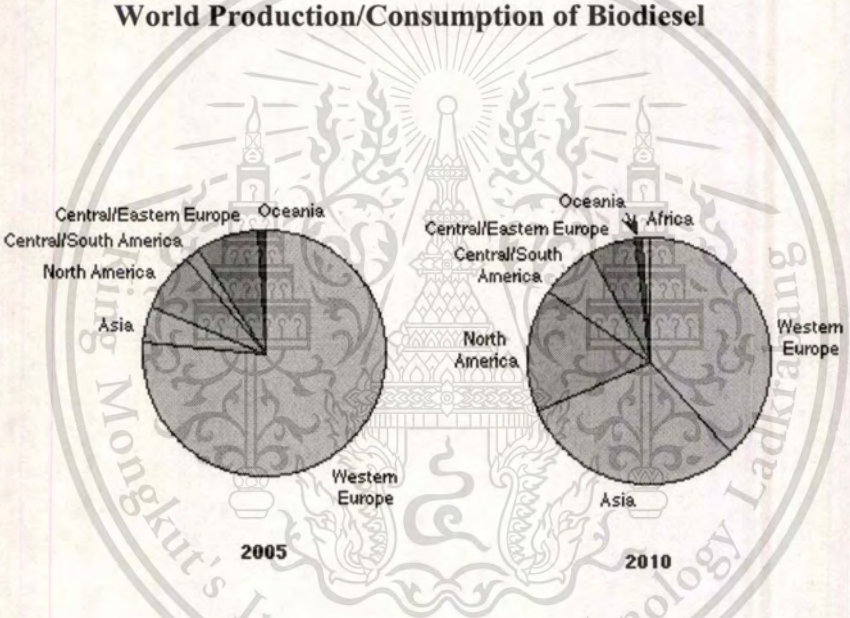


Figure 2.3 Pie charts show world production/consumption of biodiesel in 2005 and 2010 by region [7]

The French and the German biodiesel production capacity continued to grow fastest in Europe. At this moment the installed German capacity for producing biodiesel from rapeseed oil and other vegetable and animal oils and fats, including waste frying oil, has reached some 4.8 million tons per year (December 2007)[8]

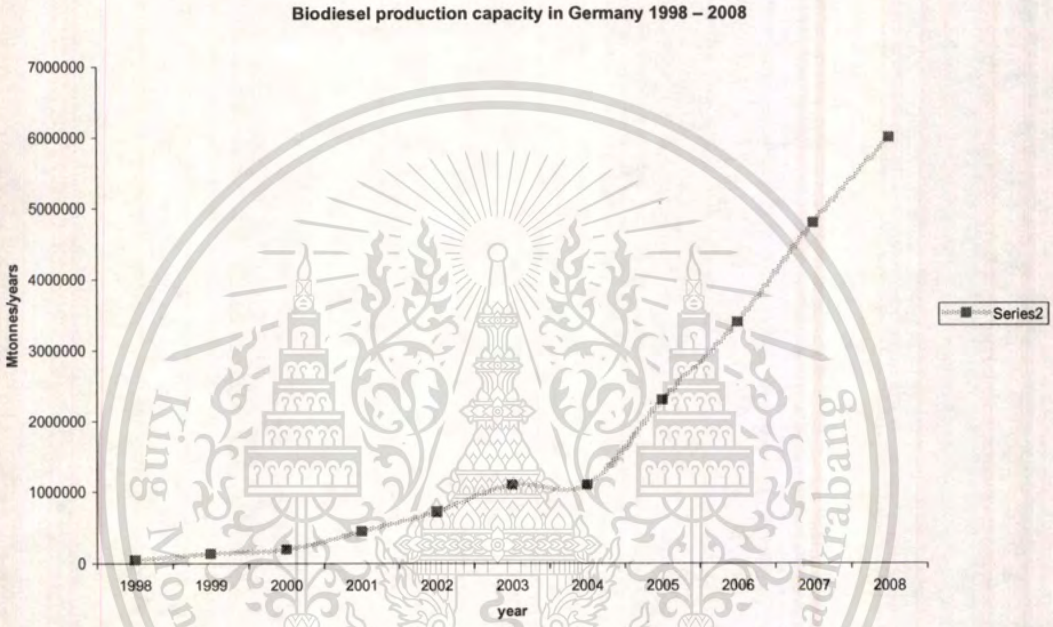
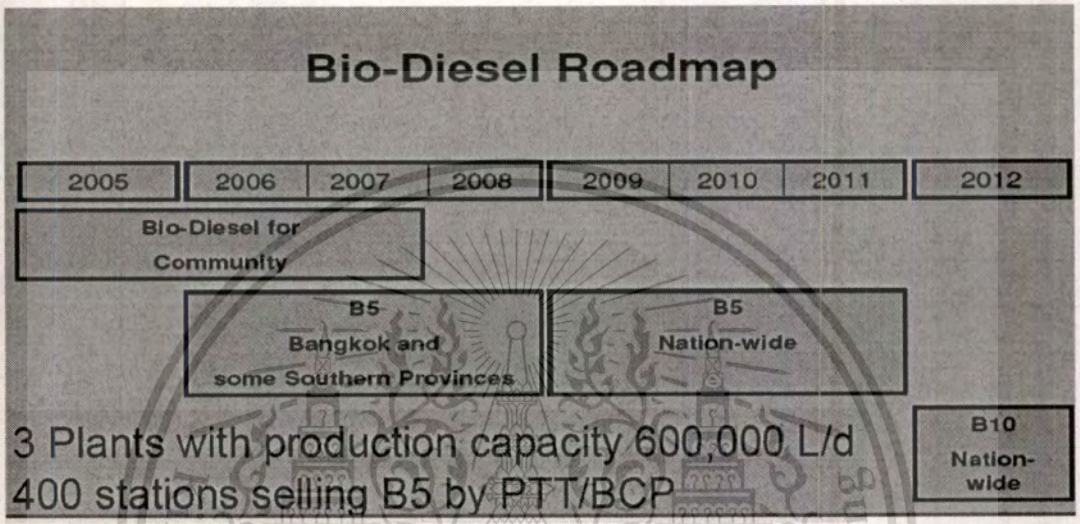


Figure2.4 Biodiesel production capacity in Germany 1998 – 2006. [9]

Biodiesel Production in Thailand

Because of Thailand is the agricultural country, a raw material of biodiesel production are abundant. The main feedstock for biodiesel production is oil palm. Because oil palm is a plant with high competitive potential due to its lower costs in production and marketing than other plants. Besides, palm oil can be utilized diversity in consumption goods. A present production capacity of biodiesel is at 500,000 liter/day. In 2012, the biodiesel production capacity will be at 8.5 million liter/day due to the cultivation area of oil palm will extend to 10 million rai in 2011. Furthermore, the palm stearin, which is a by-product from palm oil

extraction, can also be used as a raw material to produce biodiesel. Another raw materials from agricultural in Thailand that have high potential to produce biodiesel are coconut oil, soy bean oil, ground nut oil, sunflower oil and jatropha oil.



	2005	2006	2007	2008	2009	2010	2011	2012
Plantation (M.rai/year)	0.26	0.60	0.67	1.07	1.40			
Esters (M.litres/day)	0.03	0.06	0.36	0.46	0.76	1.76	3.96	8.50
Bio-Diesel (M.litres/day)	0.60	1.20	7.00	9.00	15.00	35.00	79.00	85.00
	Bio-Diesel for Community		B5					B10

Figure 2.5 Thailand's Biodiesel Roadmap. [10]

The advantages of using biodiesel are as follow;

-Reduction in air pollution because biodiesel have high oxygynate.

-Non toxic

-Reduction of oil spill in the transport oil at the oil pipe.

-The lubricating effects of the biodiesel may extend the lifetime of engines

The disadvantage of using biodiesel

-It takes energy to produce biodiesel from oil palm,including the energy of fertilizing and harvesting.

-Biodiesel cleans the dirt from engines , the dirt then collects in the fuel filter.

-Biodiesel has not yet distributed widely as petroleum diesel.

2.2 Glycerol

Glycerol is the common name of the organic compound whose chemical structure is $HOCH_2$-CHOH- CH_2OH. Propane-1,2,3-triol or glycerin (USP), as it is also called, consists of a chain of three carbon atoms with each of the end carbon atoms bonded to two hydrogen atoms (C-H) and a hydroxyl group (-OH) and the central carbon atom is bonded to a hydrogen atom (C-H) and a hydroxyl group (-OH). [11]

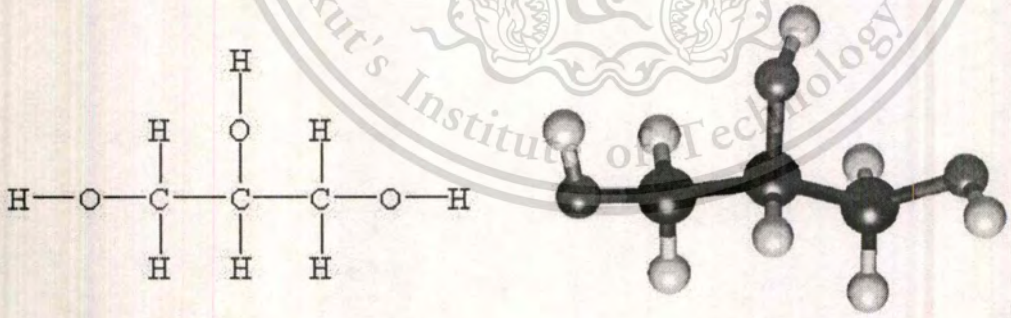


Figure 2.6 The structure of glycerol. [12]

Glycerol is a trihydric alcohol because it contains three hydroxyl or alcohol groups. Glycerol is a thick liquid with a sweet taste that is found in fats and oils.

2.2.1 History of Glycerol

Glycerol was discovered in 1779; when the Swedish chemist "Carl Wilhelm Scheele" (1742-1786) washed glycerol out of a heated a mixture of lead oxide (PbO) and olive oil. Up until 1889, people didn't know how to recover glycerol from the soap making process, so commercially produced glycerin mostly came from the candle making industry.In 1889, a viable way to separate the glycerin out of the soap was finally implemented. Since the number one use of glycerin was to make nitroglycerin, which was used to make dynamite, making soap suddenly became a lot more profitable. [13]

- Crude Glycerol

Crude glycerol is an impure form of glycerol and is primarily made as by product from the manufacture of biodiesel. It is not pass the purification process and has a contaminants which are depend up on raw materials and the production process. The typical composition of crude glycerol is given in Table 2.2

Table 2.2 Typical composition of crude glycerol[14]

Property	Value	Unit
Genetically modified origin	Possible	
Glycerol content	77 – 90%	wt% A.R.
Ash content	3,5 – 7%	wt% A.R.
Moisture content	0,1 – 13,5%	wt% A.R.
Lower calorific value	14,9 – 17,5	MJ/kg A.R.
Kinematic viscosity	120	mm2/s
3-monopropylenediol	200 – 13.500	ppm
Methanol	0,01 – 3,0%	wt%
MONG*	1,6 – 7,5%	wt%
pH	4,5 – 7,4	
Sulphate	0,01 – 1,04	wt%
Phosphate	0,02 – 1,45	wt%
Acetate	0,01 – 6,0	wt%

- Purified Glycerol

Purified Glycerol means a glycerol that pass the purification process and can use in the industry. The purified glycerol can be dividing into 4 quality grade those are:

- Chemical grade

- Dynamite grade

- Technical grade

- Pharmaceutical grade

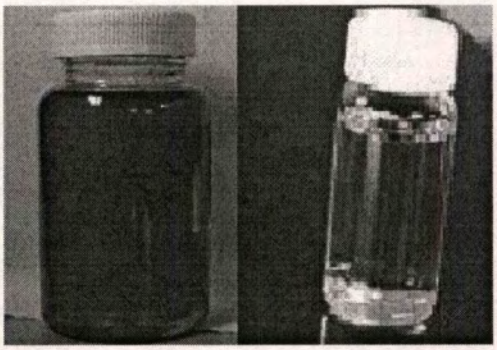


Figure 2.7 Crude glycerol and purified glycerol. [15]

2.2.2 Glycerol properties

- Physical properties

Glycerin is a neutral, sweet-tasting, colorless, thick liquid which freezes to a gummy paste and which has a high boiling point. Glycerin can be dissolved into water or alcohol, but not oils and all of hydrocarbons. Glycerin is also highly "hygroscopic" which means that it absorbs water from the air. [16]

Table2.3 Physical properties of glycerol [17]

Property	
Molecular Weight	92.09 g
Boiling Point (101.3 kPa)	290.0 °C
Melting Point	18.0 °C
Density (20 ° C)	1.261 g/cm^2
Refractive Index,$n^{20}{}_D$	1.4740
Dynamic Viscisoty (20 ° C)	1.410 Pa.s
Compressibility (28.5 ° C)	2.1 x 10^{-4} MPa^{-1}

Gravity coefficient of thermal expansion (15-20°C)	0.000615 K^{-1}
Surface Tension (20 ° C)	63.4 mN/m
Heat of formation	669 kJ/mol
Heat of combustion	1662 kJ/mol
Heat of vaporization (55 ° C)	88.2 kJ/mol
(195 ° C)	76.1 kJ/mol
Heat of fusion (18 ° C)	1662kJ/mol
Heat of solution (infinite dilution)	88.2kJ/mol
Heat Capacity (26 ° C)	2.41kJ/kg K^{-1}
(-80 ° C)	1.91kJ/kg K^{-1}
(-108 ° C)	0.91kJ/kg K^{-1}
Thermal conductivity (0 ° C)	0.29$Wm^{-1}K^{-1}$
Diffusion coefficient of water into glycerol (20 ° C)	1.336 x 10^{-11} m^2/s
Specific electrical conductivity (20 ° C)	0.1 µS/cm
Relative dielectric constant (25 ° C)	42.48
Flash point	177 ° C
Fire point	204° C
Autoignition temperature	429° C

Glycerol has a normal boiling point at 290 ° C at the atmosphere (101.3 kPa) and the relation between boiling point and vapor pressure of glycerol is shown in table 2.4

Table 2.4 The relation between boiling point and vapor pressure of glycerol. [18]

Temperature(° C)	Vapor pressure(kPa)
290	101.3
266	53.3
222	13.3
204	6.67
175	2.00
152	0.67
130	0.18
100	0.03
20	Less than 0.0001

- Chemical Properties

Glycerol can have a reaction similar to general alcohol.When heated up to 160°c in a presence of a little acid , glycerol decomposed forming an acrolein,a toxic , corrosive gas but in a situation of neutral , glycerol can stand with the thermal up to 275 ° C without any decomposition .[19]

2.2.3 Glycerol Production

The Glycerol production can be classified as natural glycerol production and synthesis glycerol production.

2.2.3.1 Natural Glycerol Production

A natural glycerol can be produced by a several method like saponification, transesterification and high pressure splitting.

- Saponification

Saponification is the hydrolysis of an ester under basic conditions to form an alcohol and the salt of a carboxylic acid which is soap. Chemically, soap is a salt of a fatty acid. This reaction use fats or oil and base as starting materials. The fats and the bases are hydrolyzed by water; the free glycerol heads bond with the free hydroxyl groups to form glycerol and the free sodium molecules bond with the fatty acid tails to form soap.

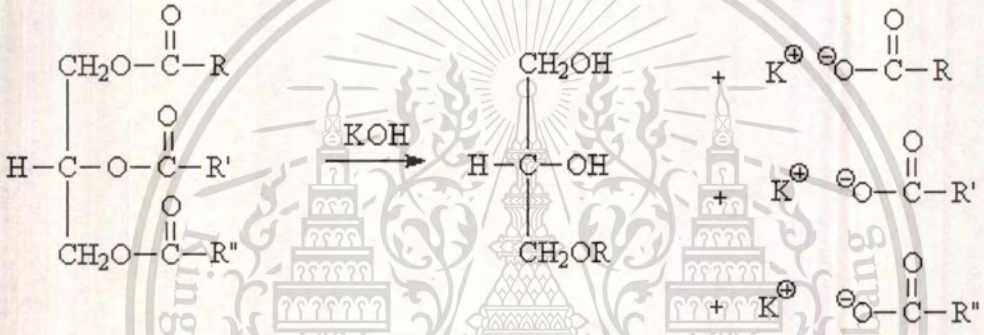


Figure 2.8 Saponification Reaction. [20]

- Transesterification

The Transesterification process is the reaction of a triglyceride (fat/oil) with an alcohol to form esters and glycerol. A triglyceride has a glycerine molecule as its base with three long chain fatty acids attached. The characteristics of the fat are determined by the nature of the fatty acids attached to the glycerine. The nature of the fatty acids can in turn affect the characteristics of the biodiesel. During the esterification process, the triglyceride is reacted with alcohol in the presence of an acid or basic catalyst.

1. Acid Catalyst Tranesterification

Acid-catalyzed Traneserification use sulfonic, sulfuric acids, phosphoric acid and hydrochloric acid as catalyst. These catalysts give high yields in alkyl ester, but the reactions are slow. This process suitable for oil which many free fatty acid and high water content.

2. Basic Catalyst Transesterification

Basic-catalyzed Traneserification use sodium hydroxide, potassium hydroxide and potassium carbonate as catalyst. This process is faster than acid-catalyzed reaction. And it is cheaper so, it is a popularly process to produce biodiesel.

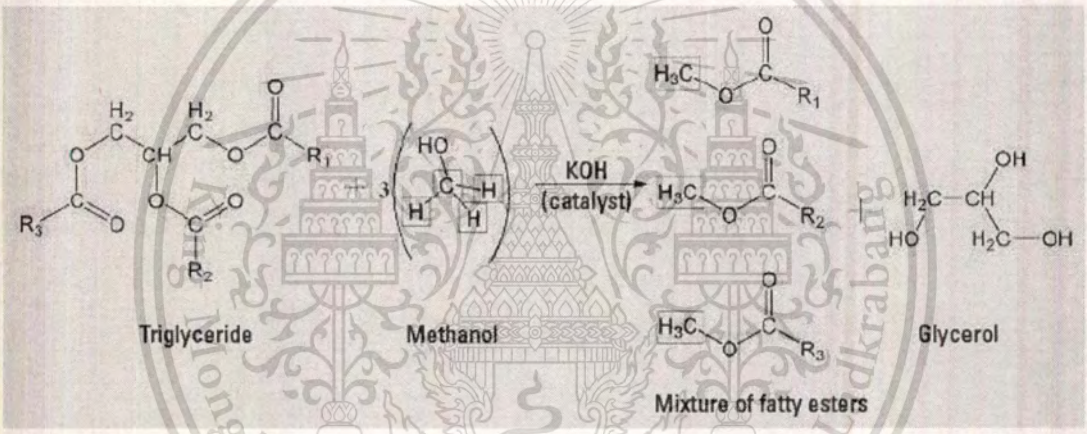


Figure 2.9 Basic catalyst transesterification reaction. [21]

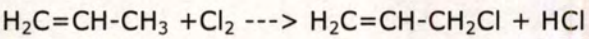
2.2.3.2 Synthesis Glycerol Production

- Production from Allyl Chloride

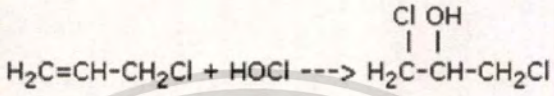
The first synthetic glycerol was produced in 1943 by I.G. Farben in Oppau and Heydebreck and in 1948 by Shell in Houston, Texas. This method became available once the high-temperature chlorination of propene to allyl chloride could be controlled properly. The allyl chloride produced is oxidized with hypochlorite to dichlorohydrin, which is converted without

isolation to epichlorohydrin by ring closure with calcium or sodium hydroxide. Hydrolysis to glycerol is carried out with sodium hydroxide or sodium carbonate. [22]

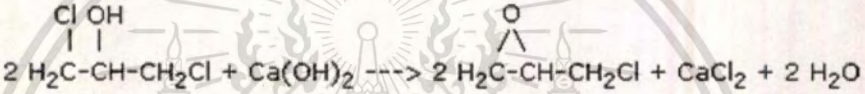
Chlorination of propene to allyl chloride:



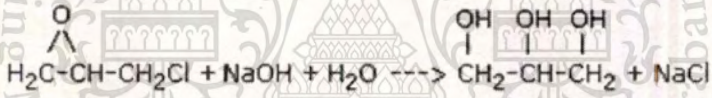
Hypochlorination:



Dehydrochlorination:



Hydrolysis of epichlorohydrin to glycerol:



2.2.4 Applications of glycerol

Glycerol has found many different applications, both as a material in its own right, for example as a food ingredient or a plasticiser, and as a chemical building block for other materials.

Major applications include:

- Food and drink

Glycerol can be use in manufacture of mono- and diglycerides for use as emulsifiers.It is used as filler and sweetener in low-fat food products such as cookies and used as a humectant

and sweetener in a wide range of products – keeps cakes moist.When used as a food additive, glycerol has the Code: E422.[23]

- Pharmaceuticals

It can be used as a solvent, moistener, humectant, and bodying agent in tinctures, elixirs, ointments, and syrups; plasticizer for medicine capsules; other uses include suppositories, ear infection remedies, anesthetics, cough remedies, lozenges, gargles, and carrier for antibiotics and antiseptics. In livestock medicine, as a postmilking teat dip alone, glycerin had a small beneficial effect on reducing colonization by Staphylococcus aureus.

- Agricultural sprays

It can be used of as an adjuvant with humectant properties increasing herbicide activity in low humidity conditions. Used as a spreader or wetting agent–a material that increases the area that a droplet of a given volume of spray mixture will cover on a target. Used as an antifoaming agent in micronised wettable sulfur. This enables the product to be mixed in a tank effectively and sprayed on evenly.

- Cosmetics and toiletries

It can be used as an emollient in toothpaste, skin creams and lotions, shaving preparations, deodorants and makeup.

- Tobacco

It can keep tobacco moist and soft to prevent breaking and crumbling during processing; ensures freshness in packaged cigarettes and other tobacco products.

- Surface coatings

It can be used in the manufacture of alkyd resins; this is an important component of surface coatings.

- Paper and printing

It can be used as a plasticizer and lubricant in the manufacture of paper; used with other ingredients in specialty treatments such as grease-proofing; alkyd resins also an important constituent of many printing inks.

- Lubricants

Due to its non-toxic character, it can be used in lubricants for food and other machinery where product purity is essential.

- Textiles

It can be used as a conditioning agent used widely in lubricating, sizing, and softening yarn and fabric; lubricates many kinds of fibers in spinning, twist setting, knitting, and weaving operations.

- Rubber and plastics

It can be used as a lubricant and plasticizer for plastic.

- Urethane polymers

It can be a fundamental chemical component of polyethers for urethane foams.

- Electrical and electronics

It can be widely employed in manufacturing electrolytes for electrolytic condensers, which are used in radios and neon lights, and in processes for electrodeposition and treatment of metals

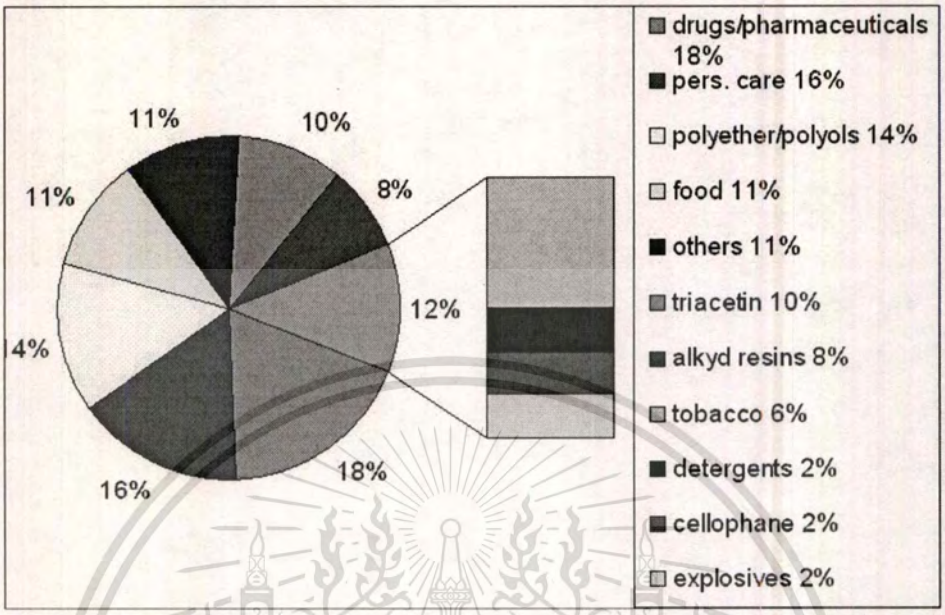


Figure2.10 Pie chart of Glycerol applications. [24]

2.2.5 Glycerol Situation

The rapid increase in biodiesel production is resulting in a worldwide production of glycerol as a by-product with enormous trend upwards. For every 10 kg of oil used to manufacture biodiesel, about 1 kg glycerol will be produced. This raw glycerol become as biodiesel's waste. Glycerol prices and demand drop drastically by over production. Pricing for glycerol is at historic lows and has recently been in the freefall mode.

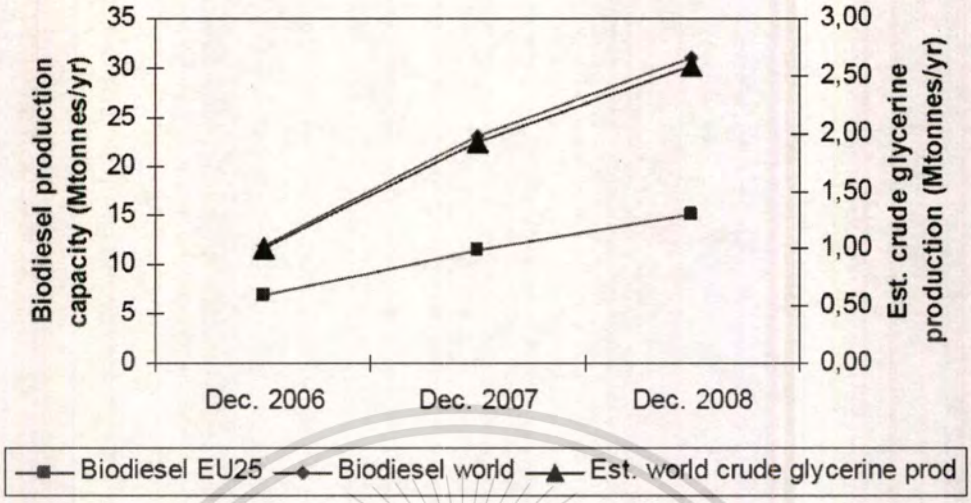


Figure2.11 Development of biodiesel production capacity and estimated glycerol production. [25]

- **Glycerol's Price**

From the 1970s until the last few years, high purity natural glycerol had a fairly stable price from about $1200 per tons to $1800 per tons. [26] This was based on stable markets and production. Prices often surged outside these ranges, but sustained high prices made it worthwhile for users to reformulate with alternative materials such as orbital and synthetic glycerol, whereas sustained low prices encouraged its use in other applications, pushing out petrochemical materials.

Although biodiesel would happen, and would be big, in the mid 1990s, they were unable to take any effective action. Increasing quantities of glycerol began to be dumped into a relatively inelastic market, and by 2005 prices were in free fall. The volumes of new glycerol are huge and increasing. Announced plans will add 2.2 million tons of glycerol to current production in the USA and EU by 2010. In 2005 the total world demand for glycerol was estimated at 900 kT. The production of glycerol is becoming dominated by new players, as recently as 2003

glycerol prices were around $1200 per tons. Today prices are around $600 per tons and falling. [27].

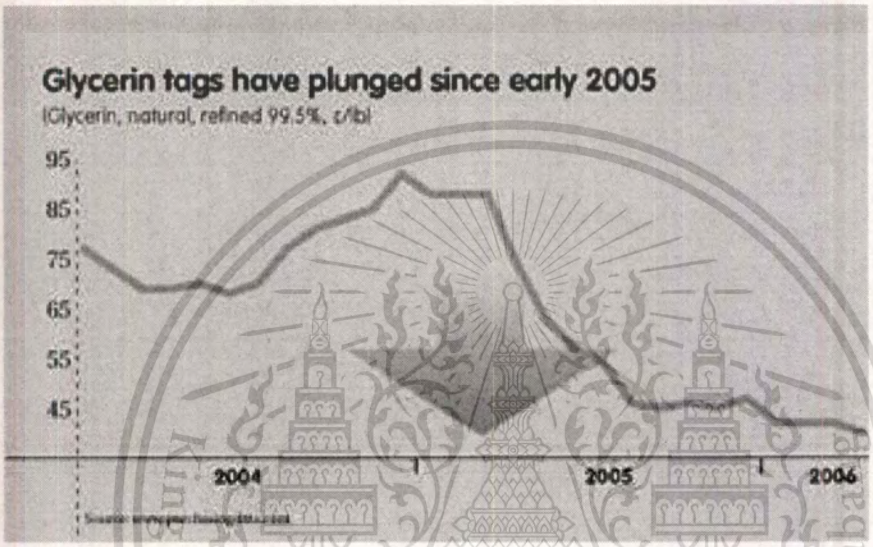


Figure 2.12 Impact of biodiesel glycerol on the glycerol market prices (99.5%, $/pound). [28]

The value of crude glycerol has also dropped. Both the year 2006 and 2007 are characterised by low glycerol prices. Current prices in the US are quoted at $0 - $70 per tons [29]. Most biodiesel producers attach zero value to the crude glycerol, and at least some producers have to pay for transport to a purification unit. Crude glycerol can be assumed to have a negative value on the future. The crude glycerol depending on the purity. The refined glycerol market is described as being strong. (with new feed and chemical applications) while the crude glycerol market is described as weak.

Prices, are under severe pressure, is determining factor in the majority of end user sectors, however quality is fundamental in the main sectors such as pharmaceuticals, personal care and food.

As more and more crude glycerol is continuously generated from the biodiesel industry, it is very important that economical ways of the low-grade glycerol utilization be explored to further defray the cost of biodiesel production in the growing global market.

2.3 Distillation

Distillation is a process used to separate or partially separate components in a mixture by boiling (vaporization) followed by condensation. Distillation separates chemicals by the difference in how easily they vaporize. The two major types of classical distillation include continuous distillation and batch distillation. Continuous distillation, as the name says, continuously takes a feed and separates it into two or more products. Batch distillation takes on lot at a time of feed and splits it into products by selectively removing the more volatile fractions over time. [29]

2.3.1 Distillation Categories [30]

Distillation services can be sorted out into many different categories. Here are some basic definitions:

- System composition

System refers to the chemical components present in the mixture being distilled.

- Binary distillation is a separation of only two chemicals. A good example is separating ethyl alcohol (ethanol) form water.

- Multicomponent distillation is the separation of a mixture of chemicals. A good example is petroleum refining. Crude oil is a very complex mixture of hydrocarbons with literally thousands of different molecules.

- Processing Mode

Processing mode refers to the way in which feed and product are introduced and withdrawn from the process

- Continuous distillation is feed is sent to the still all the time and product is drawn out at the same time.

- Batch distillation is when the amount going into the still and the amount going out of the still is not supposed to be the same all the time. The distiller fills a container at the start, then heats it, as time goes by the vapors are condensed to make the overhead. When the proper quantity of overhead is made, the distiller stops the still and empties it out ready for a new batch.

- Equipment Type

Distillation equipment includes two major categories, trays and packing.

- Trays force a rising vapor to bubble through a pool of descending liquid.

- Packing creates a surface for liquid to spread on. The thin liquid film has a high surface area for mass-transfer between the liquid and vapor

2.3.2 Distillation Theory [31]

Binary mixtures are those having two components. Three-component mixtures could be called ternary mixtures. There can be Vapor-Liquid Equilibrium data for mixtures with even more components, but such data becomes copious and is often hard to show graphically. Vapor-Liquid Equilibrium data is often shown at a certain overall pressure, such as 1 atm or whatever pressure a process of interest is conducted at. When at a certain temperature, the total of partial

pressures of all the components becomes equal to the overall pressure of the system such that vapors generated from the liquid displace any air or other gas which maintained the overall pressure, the mixture is said to boil and the corresponding temperature is the boiling point

The boiling point of a liquid is the temperature at which the vapor pressure of the liquid equals the environmental pressure surrounding the liquid.The normal boiling of a liquid ia the temperature at which the vapor pressure of the liquid equals the defined atmospheric pressure at sea level, 1 atmosphere.

- Vapor-Liquid Equilibrium Diagram

For each component in a binary mixture, one could make a vapor-liquid equilibrium diagram. Such a diagram would graph liquid mole fraction on a horizontal axis and vapor mole fraction on a vertical axis. In such VLE diagrams, liquid mole fractions for components 1 and 2 can be represented as x_1 and x_2 respectively, and vapor mole fractions of the corresponding components are commonly represented as y_1 and y_2. Similarly for binary mixtures in these VLE diagrams:

$$x_1 + x_2 = 1 \text{ and } y_1 + y_2 = 1$$

These types of VLE diagrams are used in the McCabe-Thiele method to determine the number of equilibrium stages needed to distill a given composition binary feed mixture into one distillate fraction and one bottoms fraction.Corrections can also be made to take into account the incomplete efficiency of each tray in a distillation column when compared to a theoretical plate.

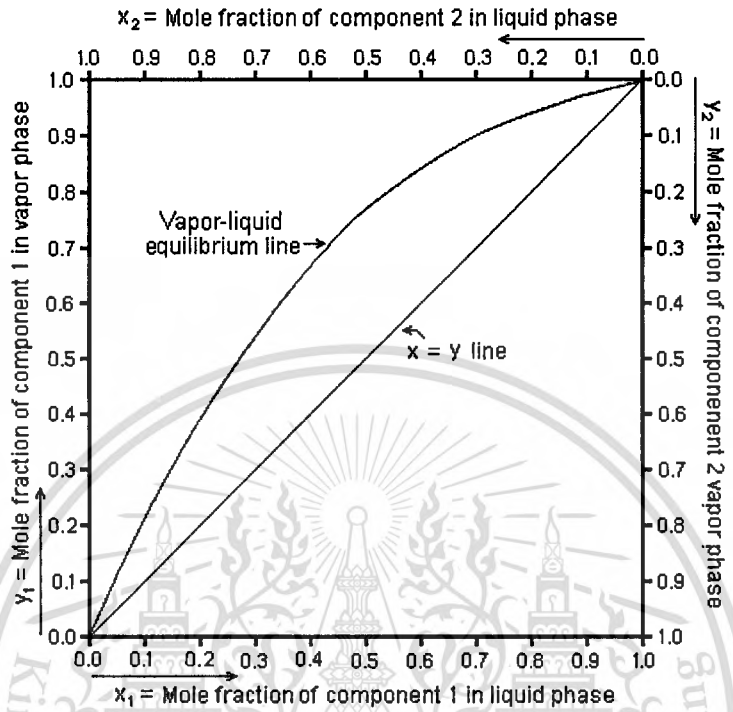


Figure 2.13 Vapor-Liquid Equilibrium Diagram. [32]

- Clausius-Clapeyron relation[33]

The Clausius-Clapeyron relation is a way of characterizing the phase transition between two states of matter. The Clausius-Clapeyron equation for the liquid – vapor boundary may be use in the following form;

These types of VLE diagrams are used in the McCabe-Thiele method to determine the number of equilibrium stages needed to distill a given composition binary feed mixture into one distillate fraction and one bottoms fraction.Corrections can also be made to take into account the incomplete efficiency of each tray in a distillation column when compared to a theoretical plate.

$$\ln\left(\frac{P_2}{P_1}\right) = \frac{\Delta H_{vap}}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right)$$

Where T_1 and P_1 are a corresponding temperature and vapor pressure

T_2 and P_2 are temperature and pressure at another point

ΔH_{vap} is the molar enthalpy of the vaporization

R is the gas constant (8.314 J mol^{-1} K^{-1})

The Clausius-Clapeyron equation allows us to estimate the vapor pressure at another temperature, if the vapor pressure is known at some temperature, and if the enthalpy of vaporization is known.

- Henry's Law[34]

Henry's law is one of the gas laws, formulated by William Henry. It states that "At a constant temperature, the amount of a given gas dissolved in a given type and volume of liquid is directly proportional to the partial pressure of that gas in equilibrium with that liquid"

A formula for Henry's Law is

$$e^p = e^{kc}$$

Where e is approximately 2.7182818, the base of the natural logarithm (also

called Euler's number)

p is the partial pressure of the solute above the solution

c is the concentration of the solute in the solution

k is the Henry's Law constant, which has units such as L·atm/mol,

atm/(mol fraction) or $Pa \cdot m^3/mol$.

Taking the natural logarithm of the formula , give us the more commonly used formula

$$P = kc$$

- Raoult's law[36]

Established by François-Marie Raoult, Raoult's law states: the vapor pressure of an ideal solution is dependent on the vapor pressure of each chemical component and the mole fraction of the component present in the solution. Once the components in the solution have reached chemical equilibrium, the total vapor pressure of the solution is

$$P_{solution} = (P_1)_{pure} x_1 + (P_2)_{pure} x_2 + \cdots$$

And the individual vapor pressure for each component is

$$P_i = (P_i)_{pure} x_i$$

Where $(Pi)_{pure}$ is the vapor pressure of the pure component

X_i is the mole fraction of the component in solution

Consequently, as the number of components in a solution increases, the individual vapor pressures decrease, since the mole fraction of each component decreases with each additional component. If a pure solute which has zero vapor pressure (it will not evaporate) is dissolved in a solvent, the vapor pressure of the final solution will be lower than that of the pure solvent. Raoult's law is similar in that it assumes that the physical properties of the components are identical. The more similar the components are, the more their behavior approaches that described by Raoult's law. For example, if the two components differ only in isotopic content, then the vapor pressure of each component will be equal to the vapor pressure of the pure substance P_0 times the mole fraction in the solution.

Today distillation is the most important industrial separation technology. It is particularly well suited for high purity separations since any degree of separation can be obtained with a fixed energy consumption by increasing the number of equilibrium stages. It is relatively straightforward to derive models of distillation columns based on almost any degree of detail, and also to use such models to simulate the behavior on a computer. However, such simulations may be time consuming and often provide limited insight.

- Thin film distillation

Thin film distillation, the method that is used to eliminate the influence of the static height of liquid and consume less energy.Thin film is a thermal separation process for thermal sensitive products. Short residence time and low evaporation temperature will cause a minimum thermal stress to the distilled product. Typical applications are high molecular organic compounds particularly from the fields of chemistry, pharmaceutical and food industry

In a thin film evaporator, raw material will be heated up on the internal surface of a heated tube until its temperature reach to the lower evaporate temperature. Condensation takes place in a condenser located outside, which is connected to a vacuum system.

Typical pressure range is the "fine vacuum range", e.g. between 1 and 0.001 mbar. At this pressure the required evaporation temperature will decrease .[37]

2.3.3 Design of distillation columns

Design and operation of a distillation column depends on the feed and desired products.Given a simple, binary component feed, analytical methods such as the McCabe-Thiele method or the Fenske equation[38] can be used. For a multi-component feed, simulation models are used both for design and operation.

Bubble-cap "trays" or "plates" are one of the types of physical devices which are used to provide good contact between the up flowing vapor and the down flowing liquid inside an industrial fractionating column.

The efficiency of a tray or plate is typically lower than that of a theoretical 100% efficient equilibrium stage.[39] Hence, a fractionating column almost always needs more actual, physical plates than the required number of theoretical vapor-liquid equilibrium stages from designed model.

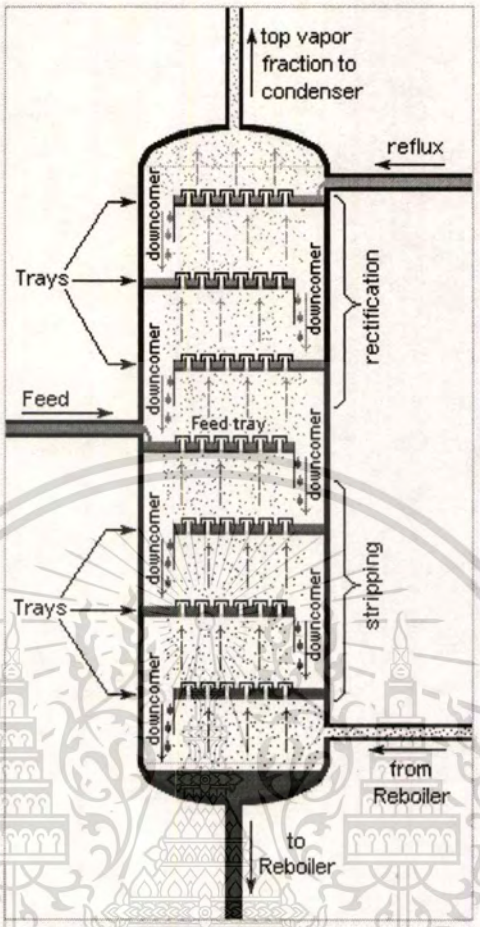


Figure 2.14 Typical bubble-cap trays in a fractionating column [40]

- Computer Simulation

HYSYS is powerful software for simulation of chemical plants and oil refineries developed by Hyprotech. It includes tools for estimation of physical properties and liquid-vapor phase equilibria, heat and material balances, and simulation of many types of chemical engineering equipment.

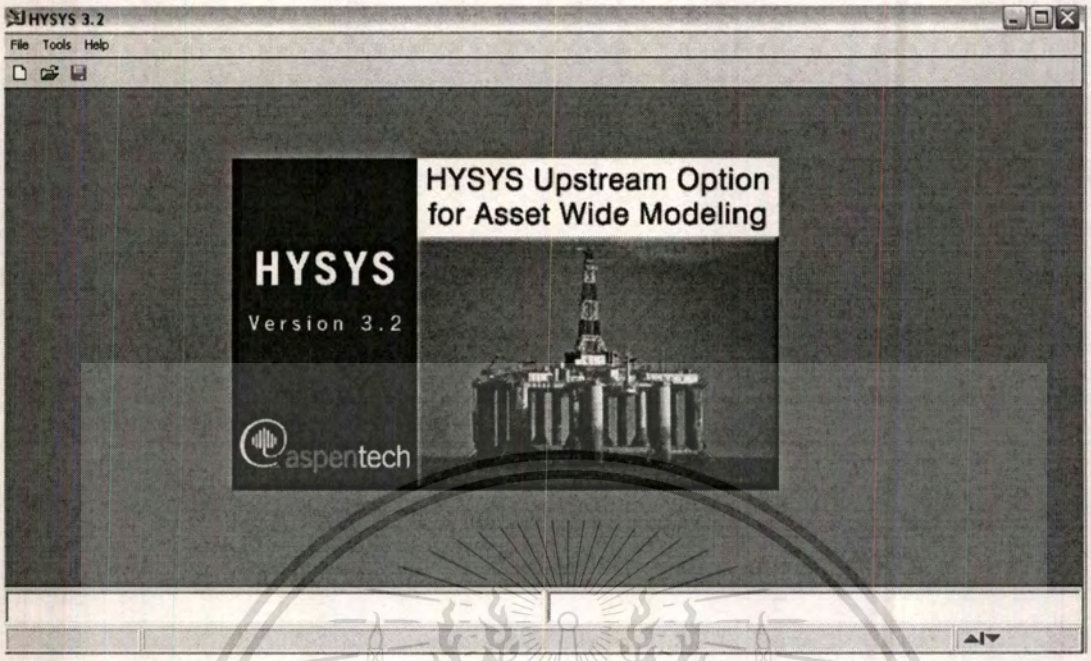


Figure 2.15 HYSYS Simulation Program.

-Fixed Variables:

Existing distillation columns have variables that are fixed that we cannot manipulate to alter the output. These variables are for instance: the height of the column , the number of trays, the plate efficiency, the tray holdups, the material of the tower, the thickness of the walls and so on.

-The independent variables:

There are a number of variables we will have control over so we can vary the column output depending on our project goals:

- Flow rates (feed, distillate, bottoms)
- Reflux ratio
- Steam flow rate

• Feed F (that is feed temperature and pressure as well as feed concentrations and mass flowrate)

Consider effects of changes of these variables on key distillation performance indicators. Note that there are limits to the control of these variables with the equipment available and not all of these variables will affect performance in a measurable way.

With the fixed and independent variables defined for HYSYS, we can now ask HYSYS to solve for not only the exit concentrations at the top and the bottom of the column but also all variables in the column. HYSYS will allow us to adjust any of these independent variables to either maximize or minimize the product component concentrations.

Design mode is also possible. HYSYS will allow us to input the desired concentrations and then determine the necessary conditions to be applied.

Chapter 3

Experimental

3.1 Chemicals and Instruments

3.1.1 Chemicals

1.) Crude glycerol from Patum Vegetable Oil Co.,Ltd..

3.1.2 Instruments

1.) Hyprotect HYSYS simulation program

2.) Vacuum pump model JD60, JAVAC PTY Co.,Ltd

3.) Water pump model A-3S, Rikakikai Co.,Ltd

4.) Manometer

5.) Digital Thermocouple model Autotherm 2 plus,Gallen Kamp Co.,Ltd

6.) Fourier Transform Infrared Spectroscopy (FTIR) model Spectrum GX Perkin ElmerCo.,Ltd

3.2 Procedure

3.2.1 Collected and studied in glycerol properties and related information

3.2.2 Designed a Thin film distillation system

1.) Thin film evaporator unit

The main function of the evaporator unit is to convert crude glycerol from a liquid to a gaseous state. In a thin film evaporator unit, the crude glycerol forms a thin film on the surface of a rotatable cylindrical tube and heat up until the lower boiling component starts to evaporate. Evaporation from a thin film will eliminate the influence of the static height of liquid and consume less energy. The vapors are then evacuated and liquefied in a cold tube of condenser. In this project, the thin film evaporator unit was designed, It consists of a rotatable cylindrical tube, crude glycerol bath, valves, manifolds and a temperature controller. Stainless steel was used for producing the evaporator because of its thermal resistance. In our process, we operated at 180° c and stainless steel can be used at temperature up to 870 ° C. [42]

2.) Fractionating Column

A fractionating column is an essential item used in the distillation of liquid mixture as to separate the mixture into its component parts based on the differences in their volatilities. A fractionating column always have a theoretical plates or tray inside. The function of a theoretical plate or tray is a zone in which two phases, such as liquid and vapor phases of a substance, establish an equilibrium with each other. The performance of a separation process depends on having a series of equilibrium stages. In other words, having more theoretical plates increases the efficiency of the separation process.

In this project, HYSYS Simulation program was used to help us designed the fractionating column. We added the estimate concentration of product that we expected and the condition we planned to operate then a numbers of theoretical plates were calculated by HYSYS.

- Procedure

- Created a new simulation

Selected File/New/Case .In HYSYS, your simulation is referred to as case.This will open up the Simulation Basis Manager which is where all of components and their properties can be specified.

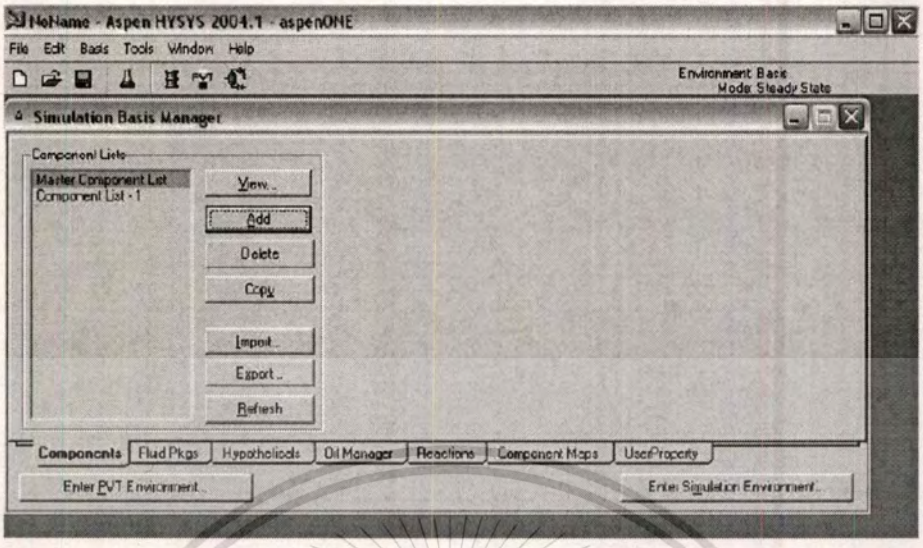


Figure 3.1 Created a new simulation

- Added a components to the simulation

Added all of the components in raw material. In our crude glycerol consist of fatty acid methyl ester, glycerol, water and methanol.

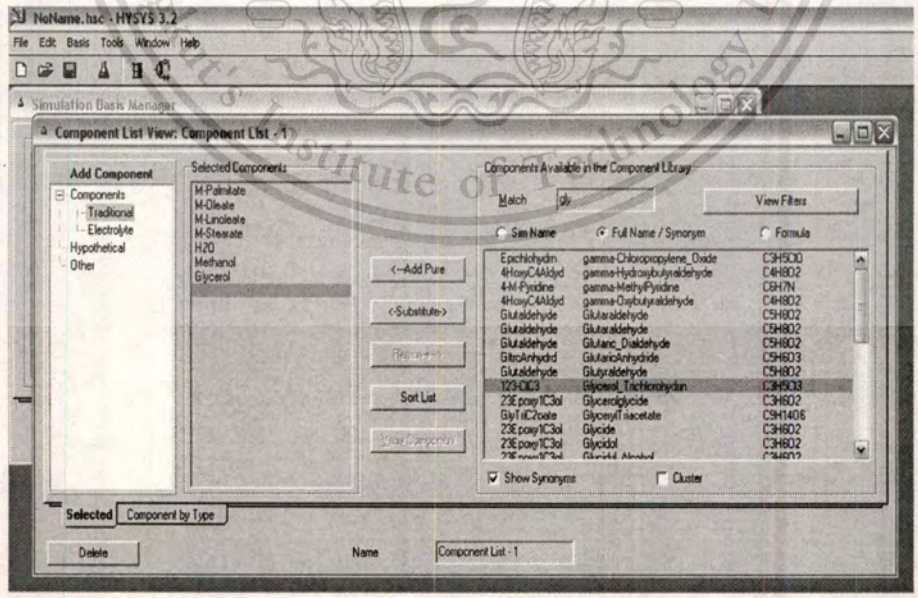


Figure 3.2 Added the components to the simulation.

- Added a fluids package

The fluids package is use to calculate the fluids/thermodynamic properties of the components and mixtures. Therefore, it is important to select a correct fluids package since this forms the basis results returned by simulation. For glycerol distillation, it generally uses the UNIQUAC as fluid package to calculate the fluids/thermodynamic properties. [43]

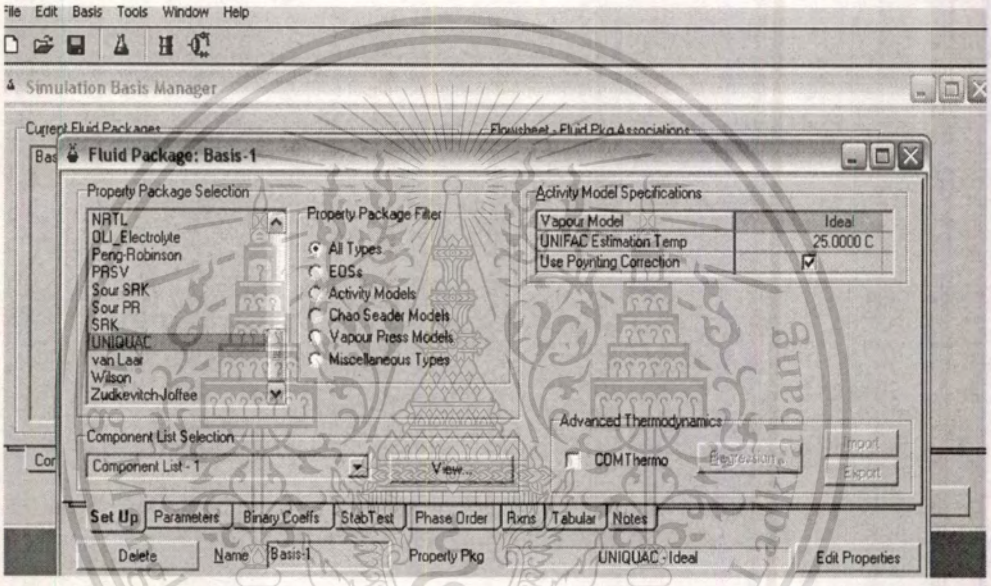


Figure 3.3 Added a fluids package.

- Entered simulation environment

After completed all necessary input to begin a simulation, clicked on Enter Simulation Environment buttons as show in figure 3.4;

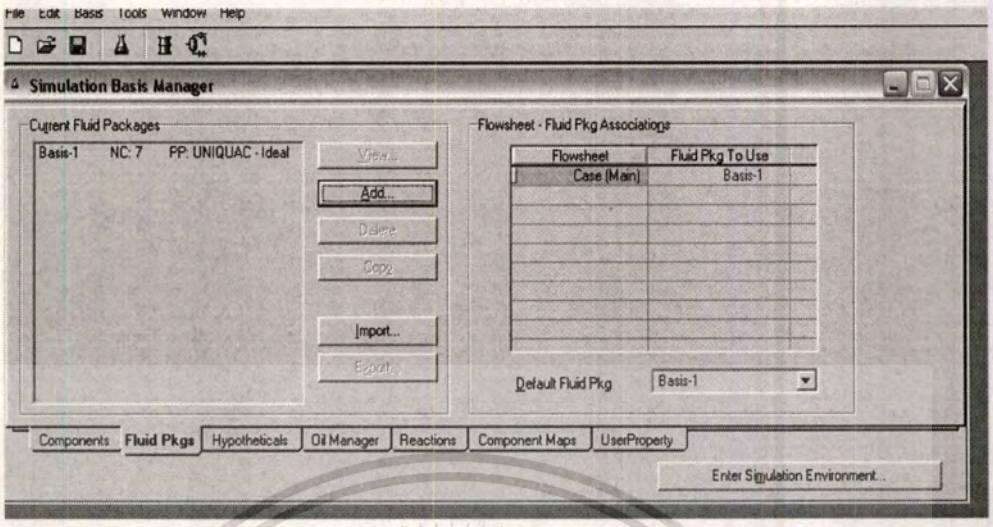


Figure 3.4 Entered simulation environment.

- Added condition to material stream

For material stream that will be used as input, we needed to supply four variables these are composition, flow rate, and two from temperature, pressure or vapor/ phase fraction .

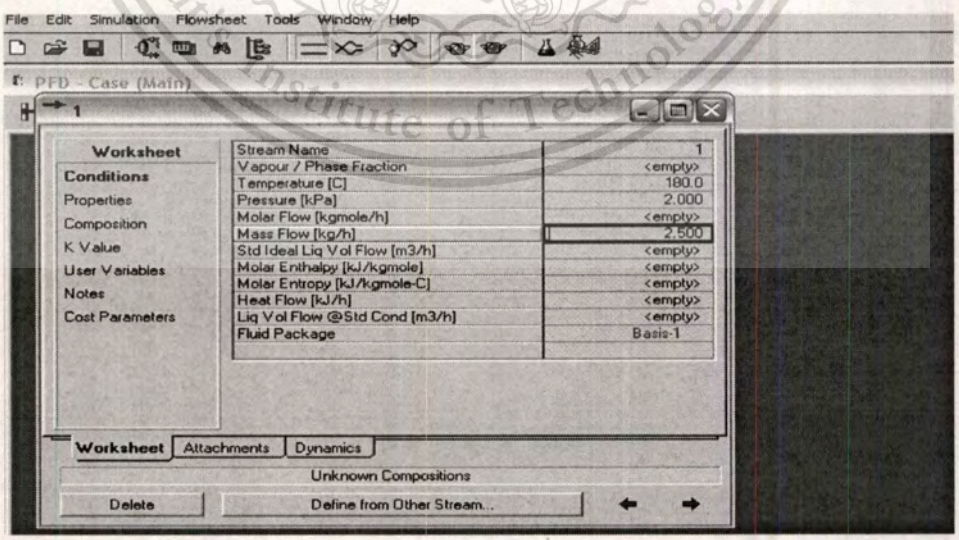


Figure 3.5 Added conditions to material stream

- Added composition to material stream

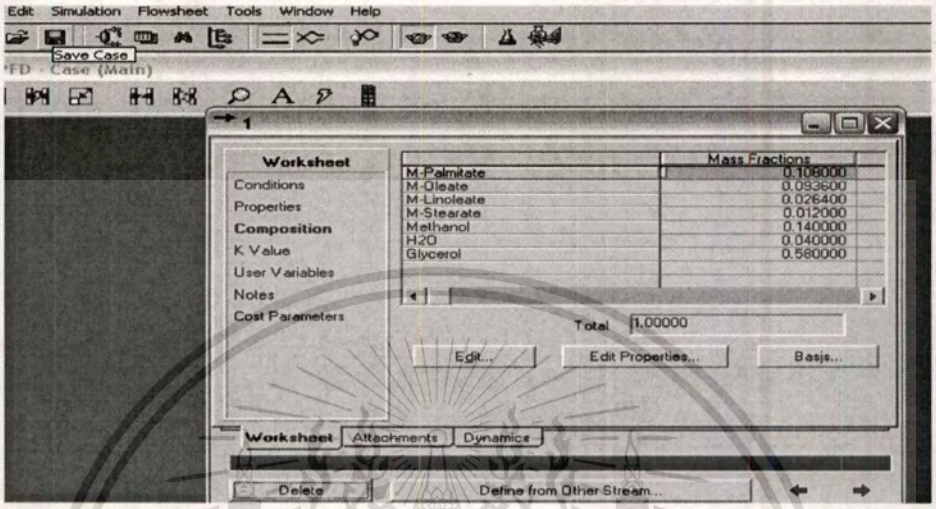


Figure 3.6 Added compositions to material stream.

- Selected a short cut distillation column and added all required streams.

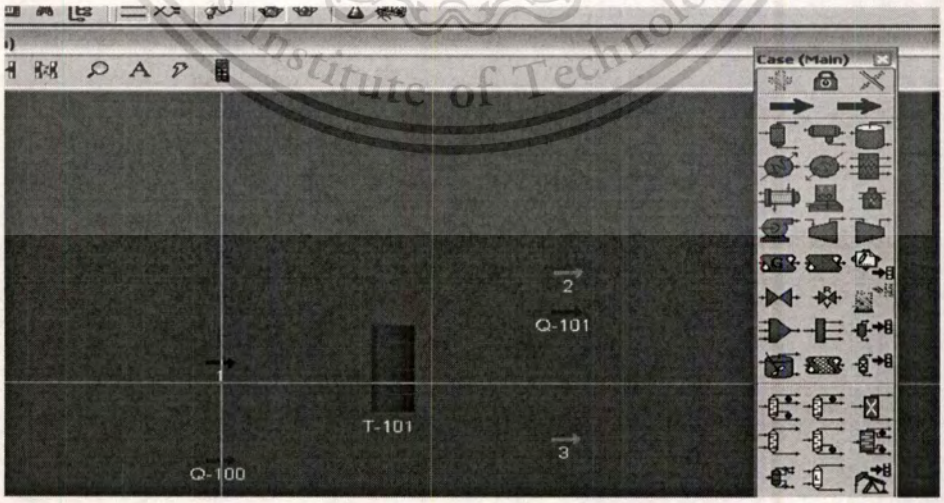


Figure 3.7 Selected a short cut distillation column and added all required streams.

Stream 1 was referred to raw material stream. Stream 2 and Stream 3 were referred to distillate and residue. Steam Q-100 and Stream Q-101 were energy stream that referred to energy supplied to the system and energy released by condenser.

- Connected all stream to distillation column by clicked on distillation column/connection.

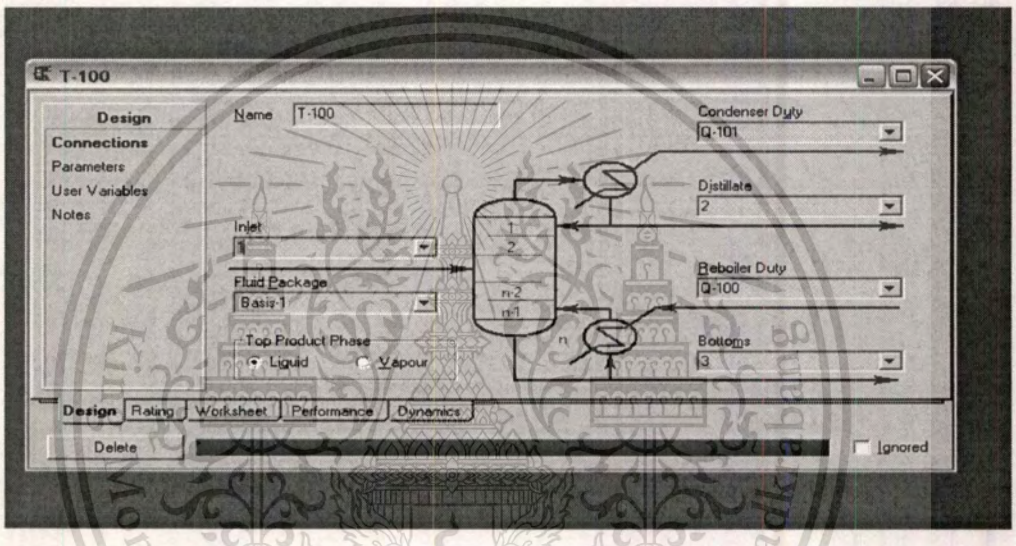


Figure 3.8 Connected all stream to distillation column.

- Added all necessary parameter

The parameters we needed to input are the expected amount of light key in bottom and heavy key in distillate, condenser and reboiler pressure. After inputted all of these, a minimum reflux ratio was shown then multiply by 1.5 to get an external reflux ratio.

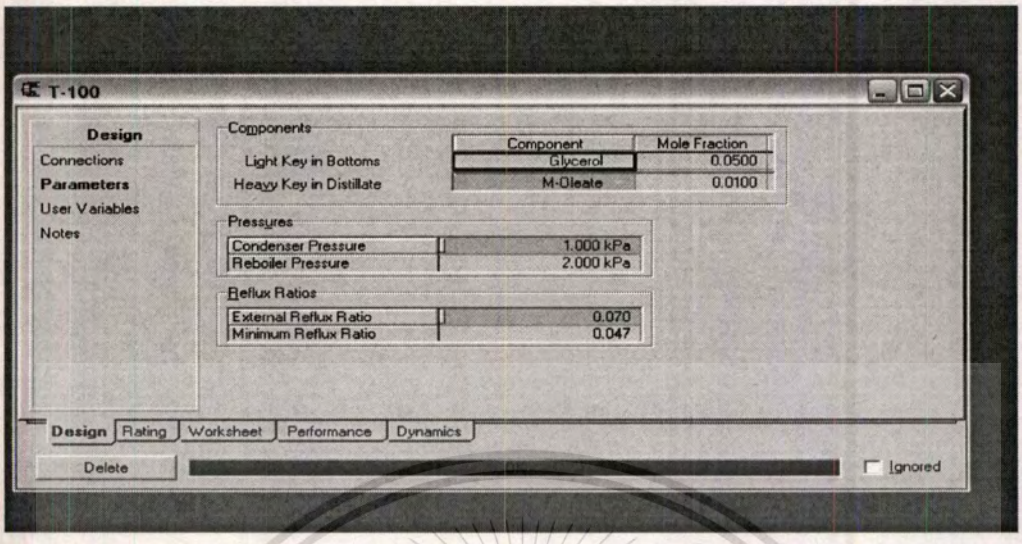


Figure 3.9 Added all necessary parameter.

- After completed the step above, clicked on a Performance button, a predicted number of tray will show.

Figure 3.10 Conditions predicted by HYSYS simulation program.

3.2.3 The construction of a thin film distillation system

The thin film distillation system composes of 5 parts as follow;

1.) Thin film evaporator unit

- A rotatable cylindrical tube was made from stainless steel. It has 18 cm. diameter and 25 cm. length.

- Placed a ceramic heater on the internal surface of a rotatable cylindrical tube .

- Connected a cylindrical tube with motor for rotating.

- Put a rotatable cylindrical tube into a stainless steel bath.

2.) Fractionating column unit

- A fractionating column was made from a stainless steel column with 18.4 cm diameter and 118 cm height.

- Punched a hole for jointing a fractionating column with a condenser.

- Placed 7 stainless steel trays inside a fractionating column. Each tray is 13.8 cm. far from each other.

- Wrapped a fractionating column by fiber glass insulator.

3).Condenser

- Condenser made from a stainless steel pipe diameter 0.5 inch and 25 cm. long. Cooling by a cold water from a water pump model A-3S, Rikakikai Co.,Ltd

4.) Product collector

- This part composes of product drum and trap drum.

- Connected one side of a product drum to a condenser and another side to a trap drum.

5.) Vacuum unit

- This part composes of manometer and vacuum pump.

- Connected the trap drum to a manometer and vacuum pump model JD60, JAVAC PTY Co.,Ltd.

Assembled all of 5 parts together. Seal every joint by rubber sheet and silicone glue.

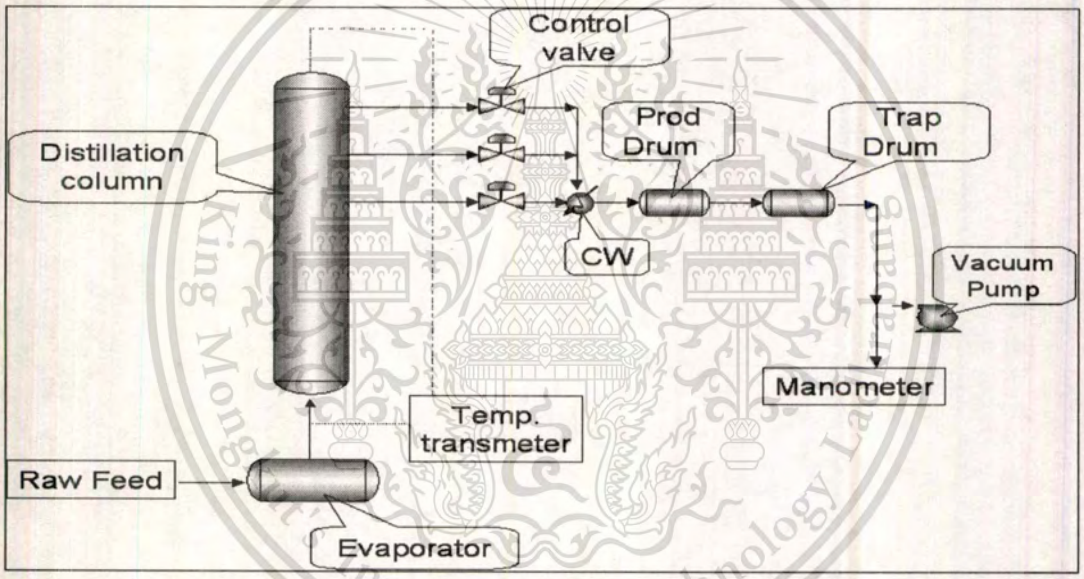


Figure 3.11 Schematic of a designed of thin film distillation system

3.2.4 Tested run a Thin film distillation system with crude glycerol

1). Poured a 4 liters of prepared crude glycerol into a thin film evaporator unit.

2). Run a Thin film distillation system under a conditions of 20 mbar and 180°c for 2 hour.

3.) Collected a distilled glycerol.

3.2.5 Product treatment

1.) Added an activated carbon bleaching agent to a distilled glycerol. The weight ratio of activated carbon : distilled glycerol was approximately 1:5.

2.) Stir and leave for 2 days.

3.) Seperated an activated carbon by a vacuum filter.

3.2.6 Product Characterization

1). Fourier Transform Infrared (FTIR) Spectroscopy

- Added a small drop of product into a cell.
- Took a cell to a Fourier Transform Infrared (FTIR) Spectrometer
- Scanned for 5 times
- Analyzed the result.

Chapter 4

Results and Discussion

4.1 Collected and studied in crude glycerol properties and related information

1.) Crude glycerol was obtained from Patum Vegetable Oil Co., Ltd. After leaving it for a while, two phases of liquid were observed. The upper phase was biodiesel and the lower phase was crude glycerol as shown in figure 4.1. The specification of crude glycerol that tested by Patum Vegetable Oil Co., Ltd was shown in table 4.1

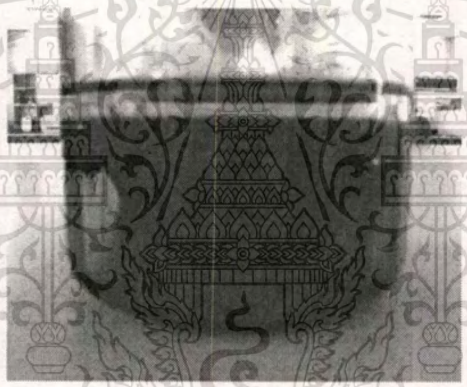


Figure 4.1 Crude glycerol.

Table 4.1 Crude glycerol specification from Patum Vegetable Oil Co.,Ltd.[41]

Parameter	Value	Unit	Method
Glycerol Purity	56 min.	% wt	ACOS Ea 6-51
Salt (as K)	4.0 max	% wt	AOCS Cc 17-79 (in-house)
Density at 25 °C	1.08 0 -1.150	g/ml	Vol / Mass
Methanol content	14.0 max	% wt	Evaporation (in-house)
Acid Oil	14.0 max	% wt	Acidulation (in-house)
%Moisture	4.0 max	g / 100 g	AOCS Ca 2e-84

4.2 Design and Construction of a Thin film distillation system

1.) Thin film evaporator unit

Thin film evaporator unit consists of a 30 liters stainless steel crude glycerol bath, a stainless steel rotatable cylindrical tube with 18 cm. diameter and 25 cm. length, a ceramic heater was placed on the internal surface. The cylindrical tube was connected with motor for rotating. The design and construction of the thin film evaporator unit were shown in figure 4.2-4.6.

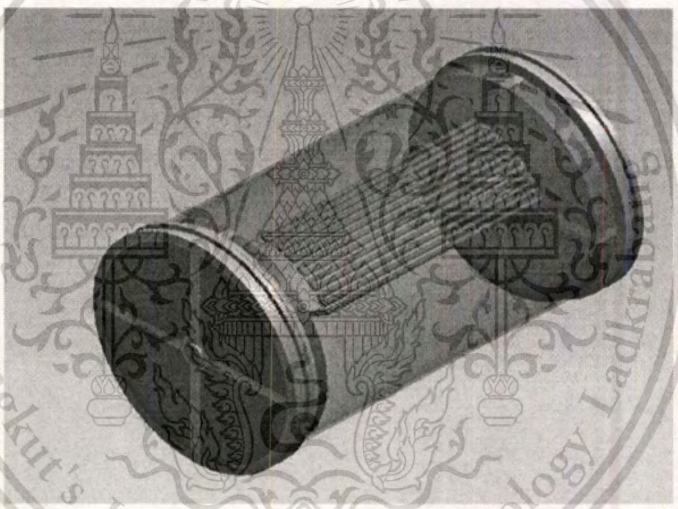


Figure 4.2 Schematic of the designed of thin film evaporator

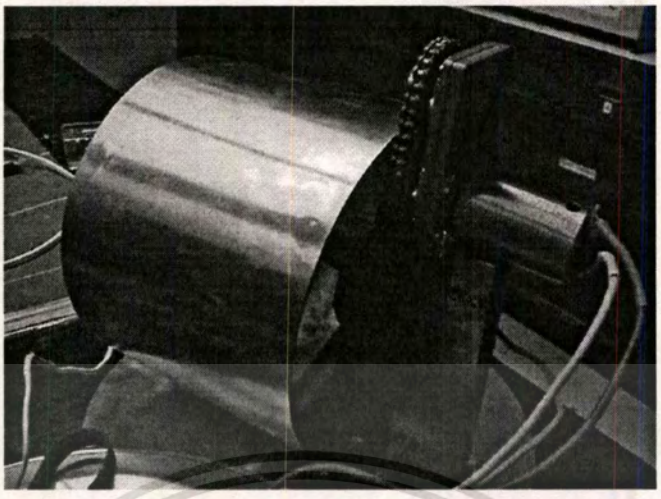


Figure 4.3 The constructed thin film evaporator.

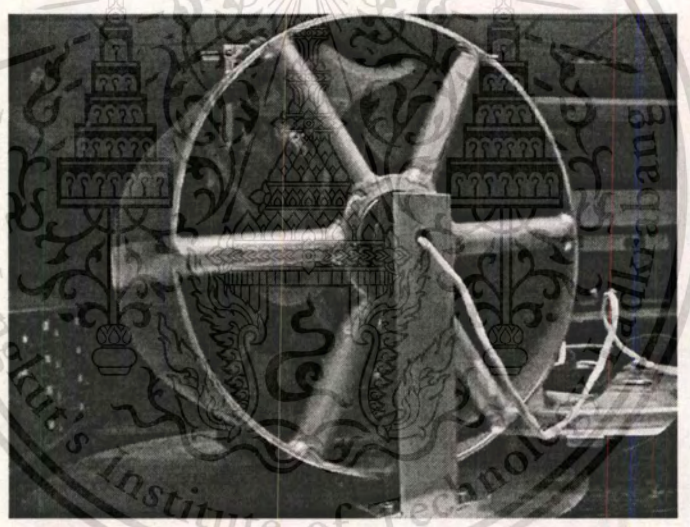


Figure 4.4 A ceramic heater on the internal surface of the rotatable cylindrical tube.

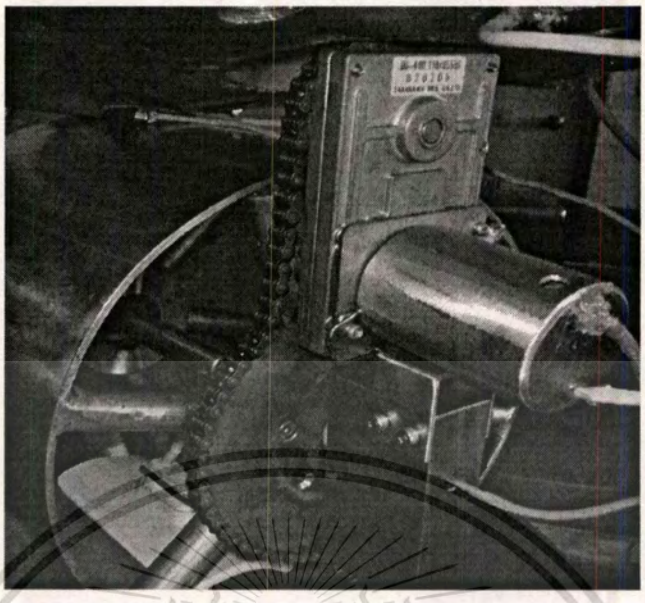


Figure 4.5 The cylindrical tube connected with a motor.

Figure 4.6 The thin film evaporator unit

2.) Fractionating column unit

Fractionating column unit composed of trays inside a stainless steel column. From the simulation by using Hypotect HYSYS simulation program , a stainless steel column with

diameter 14.8cm, height 118 cm. was contained with a 7 stainless steel trays as shown in figure 4.7 and 4.8 respectively.

Trays

Minimum Number of Trays	2.577
Actual Number of Trays	7.389
Optimal Feed Stage	3.025

Temperatures

Condenser [C]	-1.310
Reboiler [C]	208.3

Flows

Rectify Vapour [kgmole/h]	0.036
Rectify Liquid [kgmole/h]	0.002
Stripping Vapour [kgmole/h]	0.002
Stripping Liquid [kgmole/h]	0.002
Condenser Duty [kJ/h]	-3025.864
Reboiler Duty [kJ/h]	43.666

Figure 4.7 The result from Hypotect HYSYS simulation program.

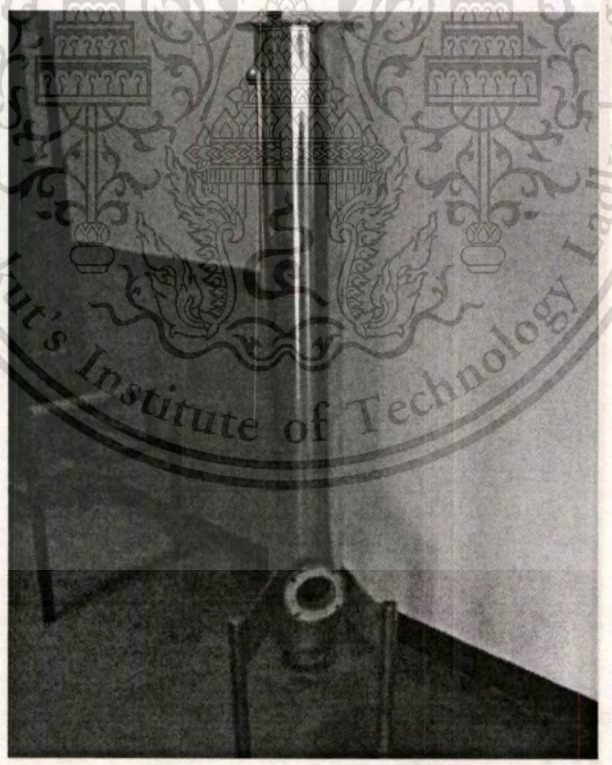


Figure 4.8 The fractionating column

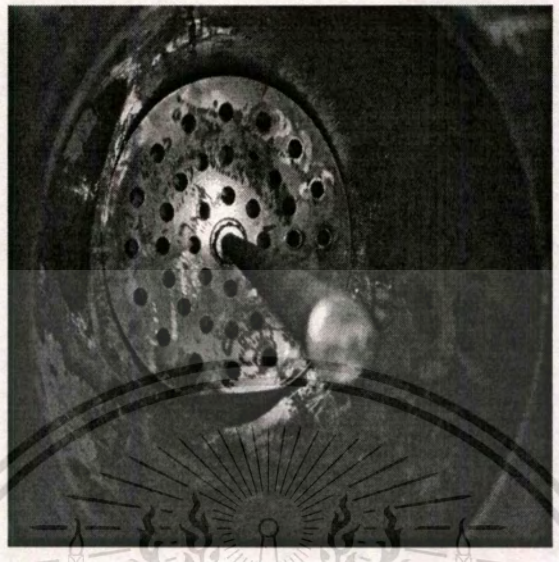


Figure 4.9 A trays placed in fractionating column.

A fractionating column was wrapped by fiber glass insulator which shown in figure 4.10

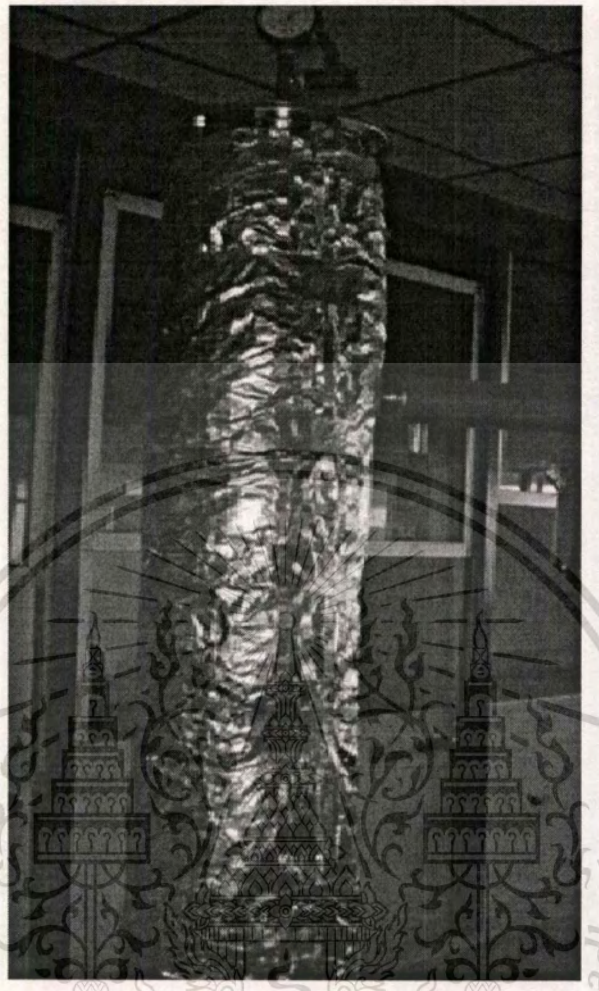


Figure 4.10 The fractionating Column wrapped by fiber glass insulator.

3.) Condenser

Condenser made from a stainless steel pipe with diameter 0.5 inch and 25 cm.length. A condenser was jointed with a fractionating column which was shown in figure 4.11.

Figure 4.11 The condenser jointed with a fractionating column.

4.) Product drum and Trap drum

Product drum and Trap drum are stainless steel pots. A product drum was connected with a condenser and the another side of product drum was connected with a trap drum.

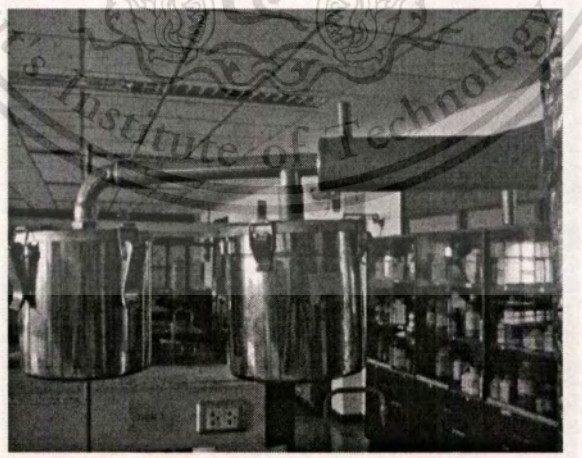


Figure 4.12 Product and trap drums.

5.) Vacuum unit

Vacuum unit composes of manometer and vacuum pump. A 3 ways tube was connected from trap drum to manometer and vacuum pump.

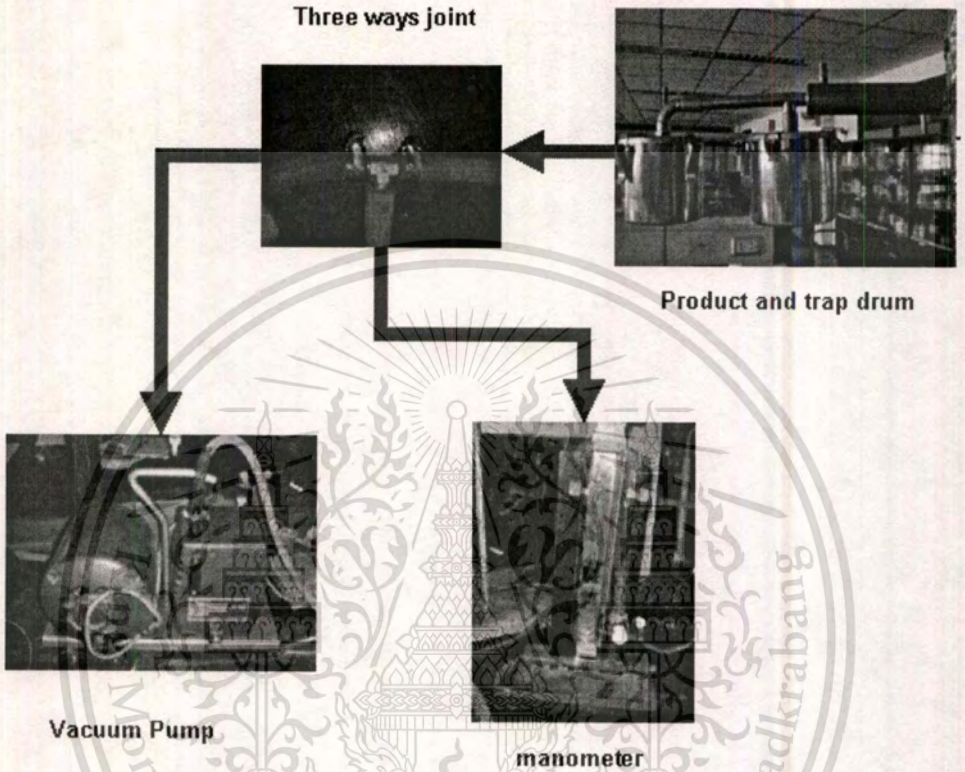


Figure 4.13 A connection of trap drum, manometer and vacuumpump.

Connected all of 5 parts together. Seal every joint by rubber sheet and silicone glue.

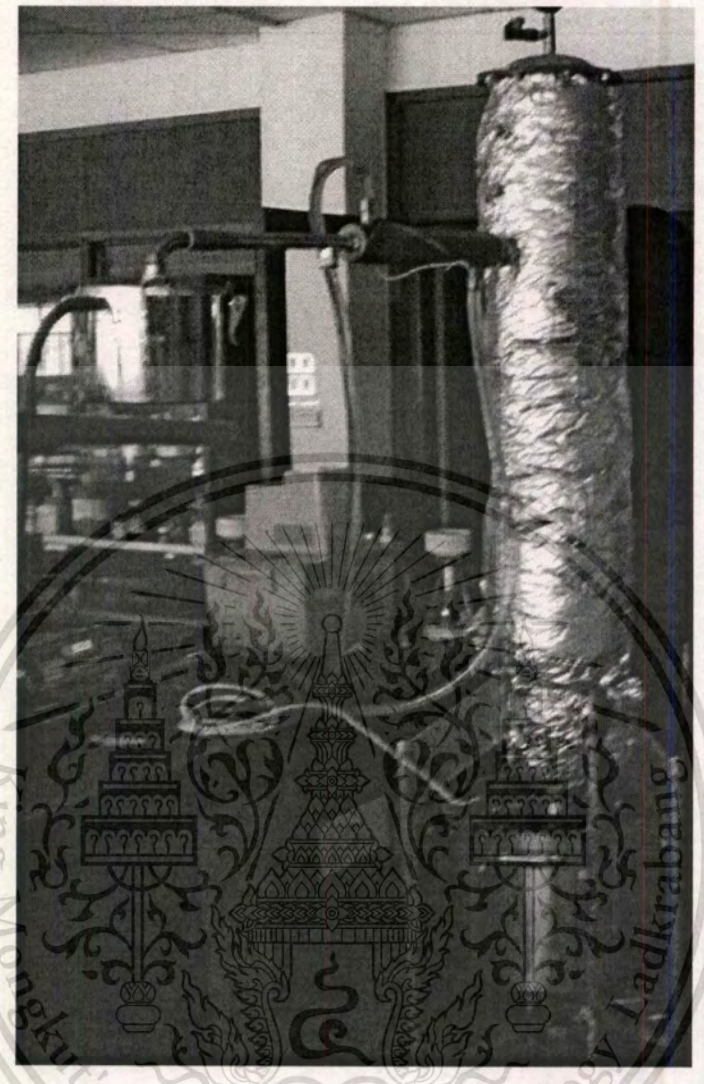


Figure 4.14 The thin film distillation system

4.3 Distillation of crude glycerol by the constructed thin film distillation system

1.) Prepared 4 liters of crude glycerol from Patum Vegtable Oil.Co., Ltd as shown in figure 4.15

Figure 4.15 A Prepared crude glycerol.

2.) Poured a 4 liters of prepared crude glycerol into the thin film evaporator unit.

Figure 4.16 Glycerol in thin film evaporator unit.

3). Run a Thin film distillation system under a conditions of 20 mbar and 180°c for 2 hour.

- When the temperature reached at 90° to 100°C, A water and methanol was withdraw from product and trap drum as shown in figure 4.17.

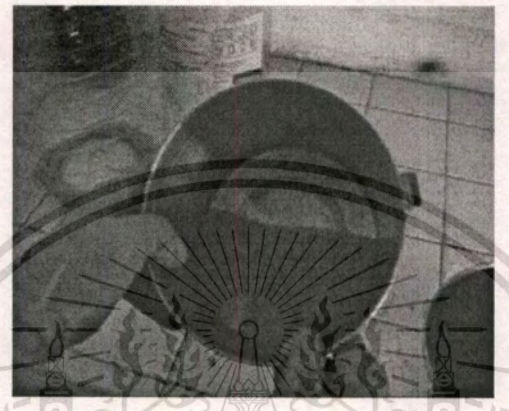


Figure 4.17 Water and methanol which collected when the temperature reach at 100°c.

- Continued to run the thin film distillation system under a pressure of 20 mmHg and 180°C for 2 hours and collect a product .

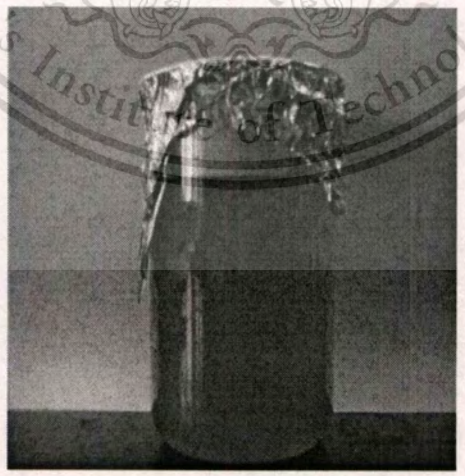


Figure 4.18 A distilled glycerol from thin film distillation.

4.4 Product treatment

1.) Added an activated carbon bleaching agent to a distilled glycerol The weight ratio between activated carbon : glycerol was approximately 1:5.

2.) Stir and leaved for 2 days.

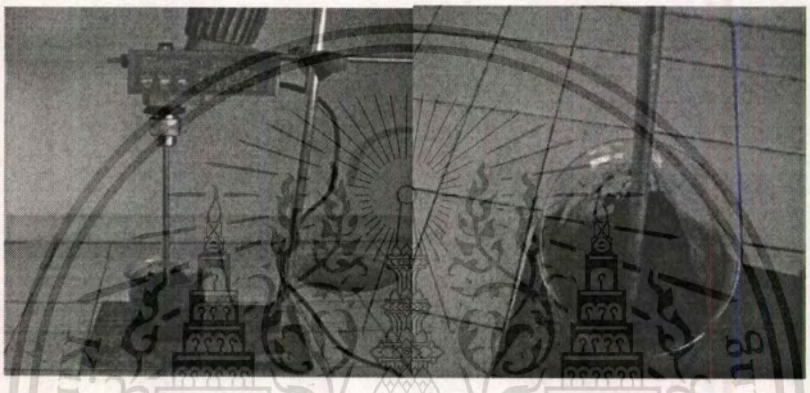


Figure 4.19 Product treatment by activated carbon

3.) Seperated an activated carbon by vacuum filtered. After that a viscous,colorless liquid would be obtained.

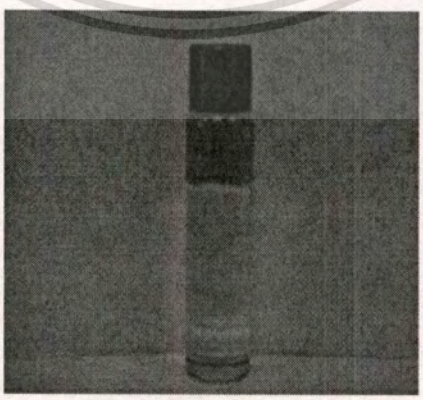


Figure 4.20 A distilled glycerol after treatment by activated carbon

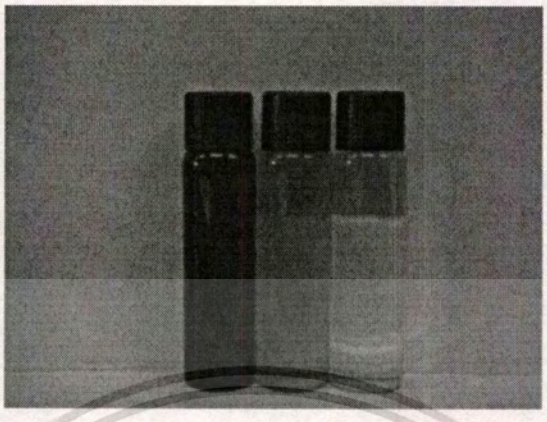


Figure 4.21 The color of glycerol at different purities.

The left bottle was a crude glycerol, a middle was a product from thin film distillation and a right bottle was a product from thin film distillation after treatment by an activated carbon.

4.5 Product characterization

A distilled glycerol after treated by an activated carbon and 99.5% purified glycerol (reference) were characterized by using FTIR (Fourier Transform Infrared Spectroscopy). Their spectrograms were shown in figure 4.22

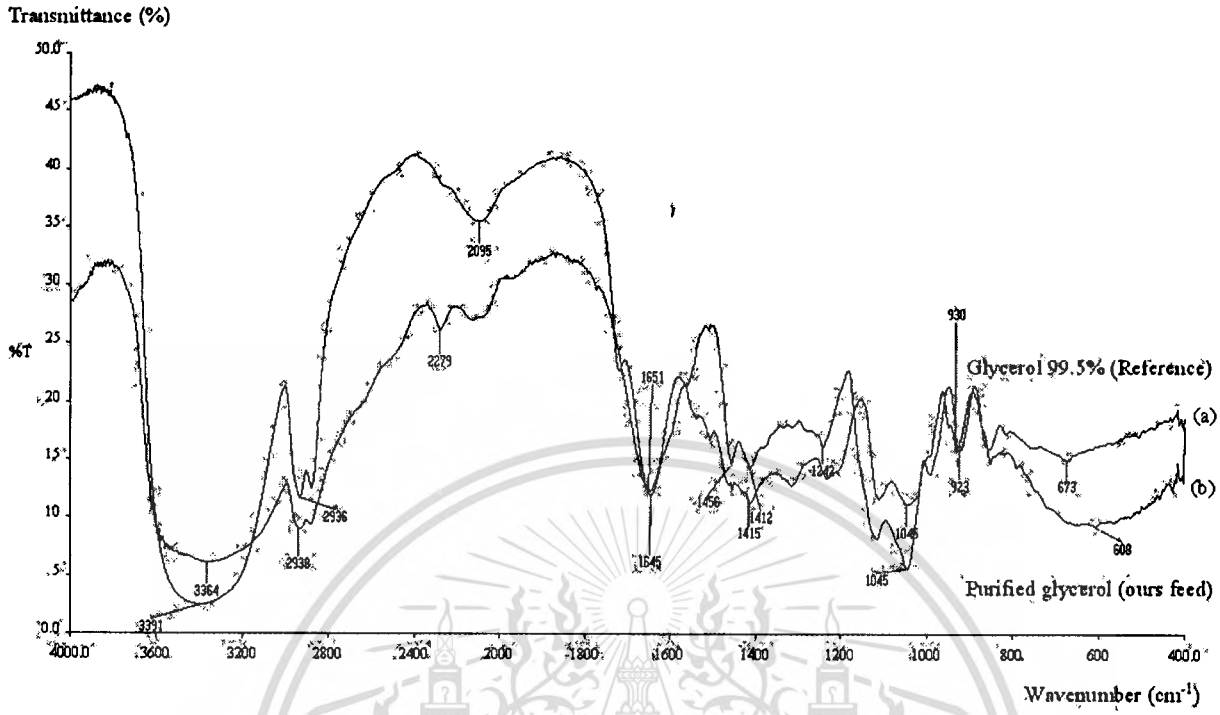


Figure 4.22 Spectrogram of distillated glycerol after treated by activated carbon (a) and 99.5% purified glycerol (b)

Spectrum analysis

1.) Result analysis of purified glycerol following to the standard pharmaceutical grade of APS Finechem was shown in table 4.2.

Table 4.2 Result analysis of purified glycerol following to the standard pharmaceutical grade.

Range	Structural	99.5% purified glycerol (reference)	A distilled glycerol after treated by activated carbon
3500-3480 cm^{-1}	O-H stretching (Alcohol)	3361	3391
3000-2800 cm^{-1}	C-H stretching	2938	2936
1075-1010 cm^{-1}	C-OH stretching (primary alcohol)	1045	1045
1120-1100 cm^{-1}	Secondary alcohol	1120	1116
1150-1140 cm^{-1}	Tertiary alcohol	non	non
1950-1600 cm^{-1}	C=O stretching	1651	1645
1610-1550 cm^{-1}	Ionic carboxyl (COO)	non	non

Chapter 5

Conclusion and Recommendation

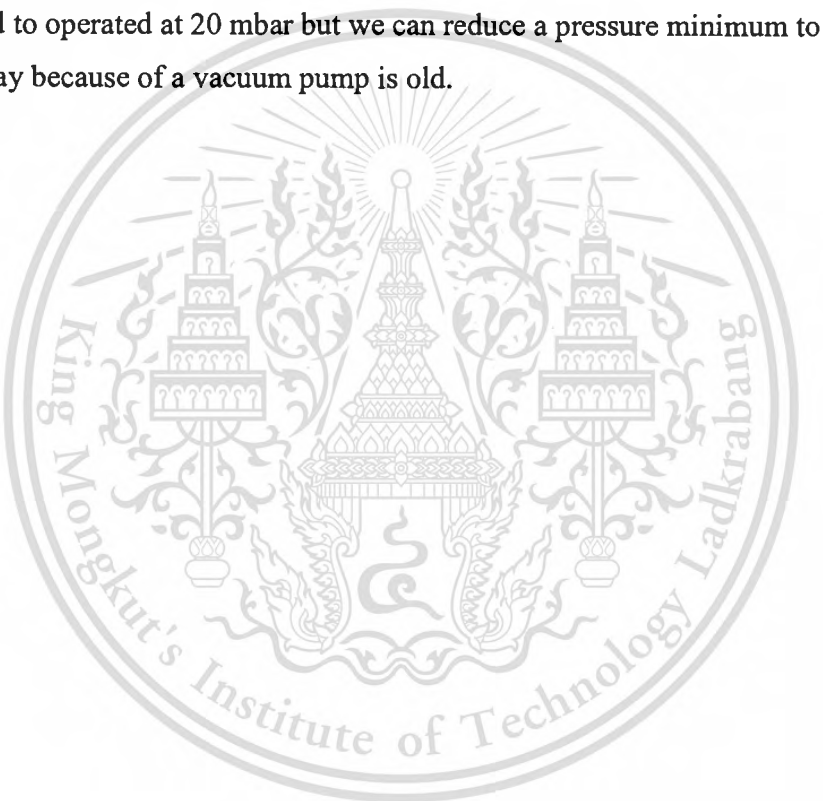
5.1Conclusion

Glycerol is a by-product from biodiesel production. High purity glycerol is a very important industrial feedstock. Its applications are found in food, drug, cosmetic and tobacco industries. However, crude glycerol derived from biodiesel production contains too many contaminants to find a useful application. As more and more crude glycerol is continuously generated from the biodiesel industry, purification process is very important

This special project is a value-added to crude glycerol from biodiesel production: purification by thin film distillation. Due to a glycerol is a high boiling point chemical, 290°c at 1 atm ,a vacuum distillation technique was introduced to this project to reduce a boiling temperature. Thin film distillation technique, was used to eliminate the static height of liquid and consume less energy. Thin film distillation system consist of 5 parts which are thin film evaporator unit , fractionating column, condenser, product and trap drums and vacuum pump. Thin film evaporator unit was designed to be a raotatable cylindrical tube with 18 cm. diameter and 25cm. length in a 30 liters stainless steel bath. HYSYS simulation program was used for designed a fractionating column unit which contain 7 stainless steel trays in a 18.4 cm. diameter and 118 cm height stainless steel column. After connected all of 5 parts together, thin film distillation system was operated with 4 liter of crude glycerol from Patum Vegetable Oil .,Co.Ltd. under the condition of 20 mbar and 180°c for 2 hours. This crude glycerol was obtain from biodiesel production via the reaction of palm oil and methanol by using a potassium hydroxide as catalyst . The performance of the thin film distillation system was tested. It was found that the thin film distillation system can be operated at a conditions of 20 mbar and 180°c and can be used to distil crude glycerol.

5.2 Recommendation

1. Because of founding a small amount of glycerol in a trap drum, a condenser pipe should be longer or use a higher flow rate of cooling water for better condensation of glycerol.
2. A number of a sockets for electrical wire around a thin film evaporator unit should be lesser in order to avoid leaking of air that lead to a failure to control pressure.
3. This thin film distillation system has a limitation on controlling pressure, we have planned to operated at 20 mbar but we can reduce a pressure minimum to 40 mbar. This may because of a vacuum pump is old.



Appendix A

1.) Fourier Transform Infrared (FT-IR)

FT-IR is the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Like a fingerprint no two unique molecular structures produce the same infrared spectrum. This makes infrared spectroscopy useful for several types of analysis. FT-IR can identify unknown materials and determine the quality or consistency of a sample. The amount of components in a mixture also determine by FT-IR Infrared spectroscopy has been a workhorse technique for materials analysis in the laboratory for over seventy years. An infrared spectrum represents a fingerprint of a sample with absorption peaks which correspond to the frequencies of vibrations between the bonds of the atoms making up the material. Because each different material is a unique combination of atoms, no two compounds produce the exact same infrared spectrum. Therefore, infrared spectroscopy can result in a positive identification (qualitative analysis) of every different kind of material. In addition, the size of the peaks in the spectrum is a direct indication of the amount of material present. With modern software algorithms, infrared is an excellent tool for quantitative analysis. The original infrared instruments were of the dispersive type. These instruments separated the individual frequencies of energy emitted from the infrared source. This was accomplished by the use of a prism or grating. An infrared prism works exactly the same as a visible prism which separates visible light into its colors (frequencies). A grating is a more modern dispersive element which better separates the frequencies of infrared energy. The detector measures the amount of energy at each frequency which has passed through the sample. This results in a spectrum which is a plot of intensity vs. frequency. Fourier transform infrared spectroscopy is preferred over dispersive or filter methods of infrared spectral analysis for several reasons:

- It is a non-destructive technique
- It provides a precise measurement method which requires no external calibration

- It can increase speed, collecting a scan every second
- It can increase sensitivity – one second scans can be co-added together to ratio out random noise
- It has greater optical throughput
- It is mechanically simple with only one moving part

Fourier Transform Infrared (FT-IR) spectrometry was developed in order to overcome the limitations encountered with dispersive instruments. The main difficulty was the slow scanning process. A method for measuring all of the infrared frequencies simultaneously, rather than individually, was needed. A solution was developed which employed a very simple optical device called an interferometer. The interferometer produces a unique type of signal which has all of the infrared frequencies "encoded" into it. The signal can be measured very quickly, usually on the order of one second or so. Thus, the time element per sample is reduced to a matter of a few seconds rather than several minutes. Fourier Transform Infrared (FT-IR) spectrometry was shown in Figure A1

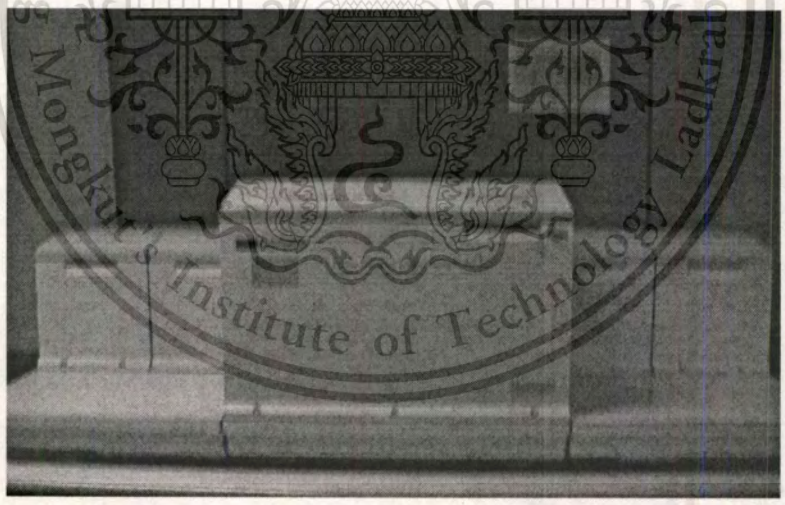


Figure A1 Fourier Transform Infrared (FT-IR) machine.

Appendix B

Glycerol standard quality: analytical reagent

Description: clear, odorless, viscous liquid

Table B1 show physical and chemical properties of 99.5 % of purified glycerol.

	Min
Assay	99.5 %v/v
Colour(APHA)	10
Density at room temperature (25°C)	1.2570

Maximum limits of impurities (%):

R.A.I	0.005
Neutrality	Passed test
Cl cpds. (as Cl)	0.003
SO_4	0.001
H.M. (as Pb)	0.0002
Acrolein, glucose & ammonium cpds.	Passed test
Ag reducing subs.	Passed test
Fatty acid ester	Passed test
Subs. Darkened by H_2SO_4	Passed test
Water	0.5

Appendix C

Activated carbon

Figure C1 Activated carbon

- Standard of Granular Activated carbon
- Product: Granular activated Coconut Shell Based Carbon
- Grade: YAO 6×12
- Test method: ASTM, unless otherwise stated
- Application: High recommended for Air purification and Volatile Organic compounds removal

Table C1 Physical properties of activated carbon from Carbokarn Co.,Ltd.

Physical properties	**Specification**
Particle size distribution : +6 (3.35 mm.) 6 × 12 (3.35 – 1.70 mm.) -12(1.70 mm.)	Max. 5% Min. 90% Max.5%
Apparent Density (g/cc)	Min. 0.47
Moister(%w/w)(As packed)	Max. 8
ASH(%w/w)(As packed)	Max. 3.5
pH	9-11
Surface area (m^2/g)(BET)	Min.1200
Iodine number (mg/g) (AWWA B 604)	Min.1150
Carbon tetrachloride adsorption (%W/W) (ASTM 3467-88)	Min.60
Hardness number (%) (ASTM 3802-79)	Min.97
Methylene blue (cc/g) (JIS K 1474-1991)	Min210

Appendix D

1.) Vacuum pump model JD60, JAVAC PTY Co.,Ltd: The result of Vacuum pump can perform approximately under a pressure of 20 mmHg.

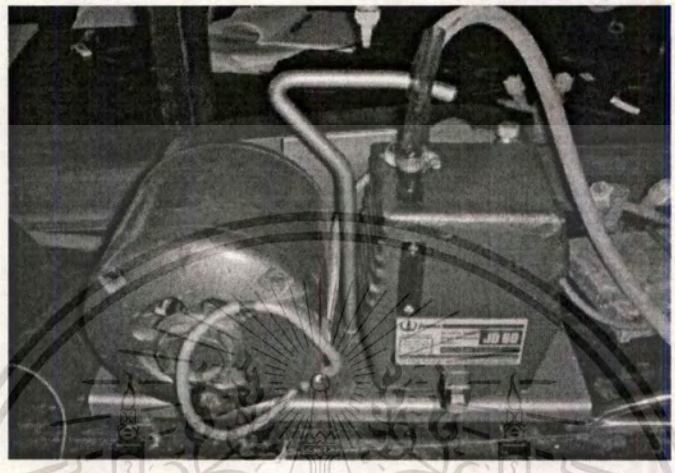


Figure D1 Vacuum pump model JD60, JAVAC PTY Co.,Ltd

2.) Manometer.

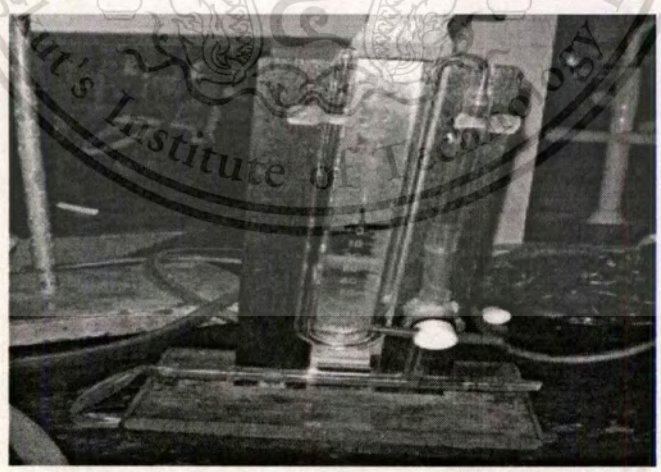


Figure D2 Manometer

3.) Digital Thermocouple model Autotherm 2 plus, Gallen Kamp Co.,Ltd

Figure D3 Digital Thermocouple model Autotherm 2 plus,Gallen Kamp Co.,Ltd

4.) Voltmeter Thaipattana Machtronic CO.,Ltd.: Generate the electric current to drive Thin film evaporator. The thin film evaporator was driven by 5 ampere of Voltmeter.

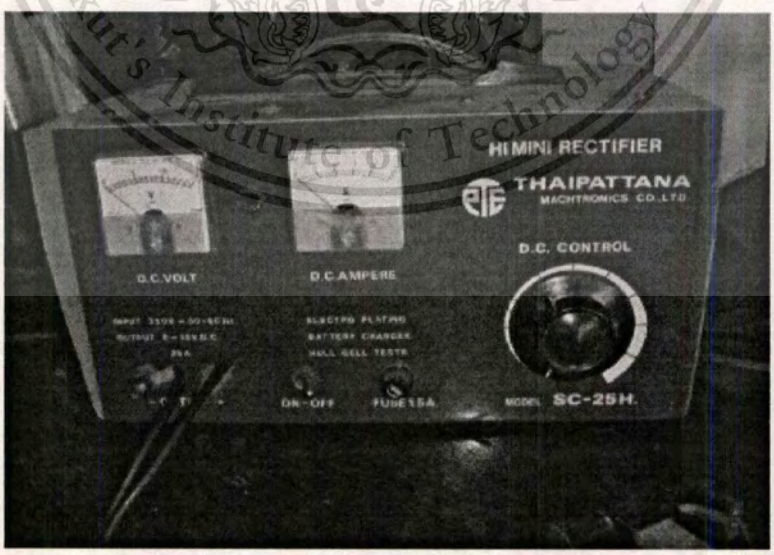


Figure D4 Voltmeter Thaipattana Machtronic CO.,Ltd.

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