MICROWAVE SYNTHESIS OF TRIACETYLGLYCEROL FROM GLYCEROL BY USING WATER FILTER RESINS AND AMBERLYST-15 AS CATALYST

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Title	Microwave synthesis of triacetylglycerol from glycerol by using water filter resins and Amberlyst-15 as catalyst.	
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ABSTRACT

The objective of this study is to synthesis triacetylglyceral from glycerol by product using microwave irradiation to improve conversion, selectivity and reduce time consumption. The reaction will carried out under microwave irradiation with 100 watt to 300 watt of microwave power and 60 to 90 seconds for 100w and 360 to 540 seconds for 300w over 5% weight of amberlyst-15 or water filter resin as catalyst with molecular ratio of triacetylglycerol and glycerol of 9: 1. After that, product obtained are characterized by Fourier transform infrared spectroscopy to confirm that triacetylglycerol are formed before tested with Gas chromatography-mass spectrometry for the quantity. Water filter resins gives equivalently equal conversion and selectivity under the combined microwave condition as amberlyst-15 (1.111% and 1.346% respectively). The reaction under microwave irradiation give a low selectivity toward triacetylglycerol ,for instance, it's have a very high selectivity toward monoacetylglycerol.

Keywords : triacetylglycerol, water filter resins, Amberlysts-15, glycerol, value-added

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Chapter 1

Introduction

1.1 Motivation

The petroleum, limited and non-regenerative energy, is the one of most important feedstock for many applications in both as energy and as reactant for many chemicals production. But due to its limited amount in the nature which being consumed largely each year cause the oil price to rapidly increase as the reservoirs nearly depleted with this rate of consumption. Thus, the alternative sources of energy based on renewable source such as biodiesel are being researched.

Biodiesel is the ester of fatty acid combines with alcohol which largely being used as renewable energy source in place of petroleum. By transesterification of triglyceride from natural fat and oil with alcohol, usually methanol or ethanol, in present of catalyst, the biodiesel is produced with glycerol as by product. His Majesty the king also encourages people to produce biodiesel from used cooking oil to replace petroleum fuel1]. With this, the popularity and expansion increase each year, the amounts of glycerol being produced as by product are also increase, cause the price of glycerol to decrease rapidly. Thus, many studies have siege this opportunities and focusing on principal to increase the value of these by product to the other effective, cheap, non toxic and biodegradable compound's feed stock[2]. One of these applications is tri-acetyl glycerol, which can be used as gasoline's anti-knock additive and properties improve additive for biodiesel or as reactant feedstock for other substance[3].

Ritwan Masae and his teammate have studied the synthesis of tri-acetyl glycerol via glycerol and acetic acid using Amberlyst-15[4] or acidic water filter resin as catalyst under each difference conditions in order to find optimum condition. They found that acidic water filter resin can be used as substitute of Amberlyst-15 with almost the same yield and conversions which can greatly reduce cost of operation in the greater scale[5].

Also, microwave irradiation has became an increasingly interesting method to heat materials in chemical laboratories, offering a clean, cheap, convenient, selective and instantaneous method of heating especially conventional household microwave oven which has received increasing interest in organic synthesis due to remarkable rate enhancement of some reactions over conventional reactions. This is one of the powerful techniques of non-contact heating. Microwave energy can be directly and uniformly absorbed throughout the entire volume of an object, causing it to heat up evenly and rapidly.

Due to these advantages, the microwave irradiation has been selected in this thesis and applied to the production of tri-acetyl glycerol which produce from relatively non-toxic reagent and have many useful applications as described before.

This research shall studies the condition which appropriate for producing tri-acetyl glycerol by using conventional microwave to irradiate as a heat source for the reaction and catalyze the reaction by water filter resins as compare to commercial amberlyst-15.

1.2 Objectives

1. To synthesis triacetylglycerol with high conversion rate and selectivity.

2. To optimize the microwave reaction conditions by using water filter resin as catalyst compare to conventional Amberlyst catalyst.

1.3 Scope to study

1. To study microwave synthesis of acetylated glycerol preparation in laboratory scale.
2. To compare triacetylglycerol production in terms of selectivity and conversion rate under conventional heater and microwave irradiation with irradiation power of 100 and 300W and reaction time between 60 second to 540 second.

1.4 Expected result

1. The results of triacetylglycerol production from microwave synthesis could be higher than previous research which done over conventional hotplate..
2. Water filter resin would give more or equally percent conversion of glycerol to triacetylglycerol as well as in term of selectivity compare to commercial amberlyst-15.
3. Water filter resin could be used instead of Amberlyst-15 which will greatly reduce the operation cost.

Chapter 2

THEORY AND LITERATURE REVIEW

2.1 Glycerol

Properties

Glycerol is a chemical with the formula as stated in Figure 2.1 which many common name such as glycerin 1, 2, 3-propanetriol or 1, 2, 3-trihydroxypropane. Glycerol has no odor, colorless, vicious and can completely soluble in water due to its tri hydric alcohol. Glycerol has a very high boiling point that is 290 °C, melting point is 17.8 °C, density is 1.26 g/cm^3 and viscosity is 1.2 Pa·s .

Glycerol is nontoxic chemical with a very sweet taste and mainly produces as byproduct of biodiesel manufacture and byproduct of refining of cooking and salad oils.

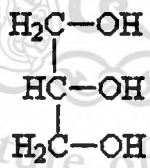

Figure 2.1 : Structure of Glycerol [6]

Application

Glycerol is a chemical that has literally thousands of uses from feed stock in chemical plants to cosmetic and personal care product. Some dietary advocates accept glycerin as a sweetener compatible with low carbohydrate diets since glycerol has a very sweet taste but contain lesser glycemic index and has difference metabolic pathway within the body.

As a raw material, glycerol is used to produce many chemical with polyol-based foam, mono- and di-glycerides or even so called tri-nitroglycerin (TNT) essential ingredient to produce dynamite.

In pharmaceutical and personal application use, glycerol is mainly use as humectants and lubricant. Glycerol can be found in many products such as lotions, shampoos, soup, toothpastes, elixirs and water based personal lubricants.

2.2 Triacetylglycerol

Properties

Triacetylglycerol, which also known as triacetin, 1,2,3-propanetriyl triacetate or glycerin triacetate , is a chemical compound with the formula as stated in the figure 2.2. It is derivation of glycerol with strong acetic acid. Triacetylglycerol is a clear, combustible and oily liquid with a bitter taste and a fatty odor. It is slightly soluble in water but soluble in alcohol and ether. Triacetin melts at -78 °C, boils at 258-260 °C and stable under ordinary condition. The way to produce triacetylgylcerol is esterification reaction and produces water as by-product.

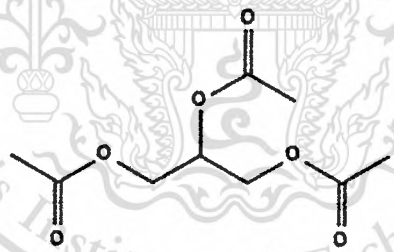

Figure 2.2 : Structure of Triacetylglycerol[5]

Application

Triacetin is found in some food like butter as it is used as a food additive for the solvency of flavorings for the function of humectants and also used in perfumery and cosmetics for these applications. It is used as an antifungal agent in external medicine for topical treatment of superficial fungal infections of the skin. Triacetin is applied to cigarette filter as a plasticizer. It is used as a gelatinizing agent in explosives.

Triacetin is also known as fuel additive which served as antiknock agent that can reduce engine knocking in gasoline and alos can improvr cold flow along with viscosity properties of biodiesel[5].

2.3. Acetic acid

Properties

Acetic acid or ethanoic acid is one of the simplest carboxylic acid with the structure as stated in figure 2.3. It is a colourless liquid that when undiluted is also called *glacial acetic acid* which comes from the ice-like crystals that form slightly below room temperature at 16.6 °C (61.9 °F). Acetic acid has a distinctive sour taste and pungent smell like vinegar. Acetic acid is completely soluble in water. Although it is classified as a weak acid, concentrated acetic acid is corrosive, and attacks the skin. Acetic acid has a boiling point at 119 °C, melting point is 17 °C, density is 1.049g/cm^3 and viscosity is 1.22 Pa·s.

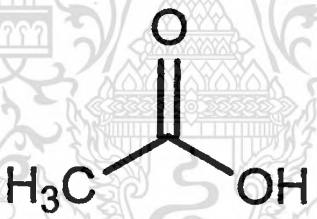

Figure 2.3 : Structure of acetic acid[7]

Application

Acetic acid is an important chemical reagent and industrial chemical, mainly used in the production of cellulose acetate mainly for photographic film and polyvinyl acetate for wood glue, as well as synthetic fibres and fabrics. In households, diluted acetic acid is often used in descaling agents. In the food industry, acetic acid is used under the food additive code E260 as an acidity regulator and as a condiment.

2.4 Ion Exchange Resins [5]

Ion exchange resins consist of a polymeric matrix and a functional group with a mobile ion which can be exchanged with other ions presenting in the solution to be treated. Ion exchange resins are classified as **cation exchangers,** which have **positively charged mobile ions** available for exchange, and **anion exchangers,** whose exchangeable ions are **negatively charged.** Both anion and cation resins are produced from the **same basic organic polymers** but **differ** in the **ionizable group** attached to the **hydrocarbon network.** It is this **functional group** that determines the chemical behavior of the resin. Resins can be broadly classified as strong or weak acid cation exchangers or strong or weak base anion exchangers. In an ion exchange process, cations or anions in a liquid solution (usually aqueous) replace dissimilar and displaceable ions of the same charge contained in the ion exchange resin. The most common synthetic structures are :

- Cross-linked polystyrene (as shown in figure 2.4)
- Cross-linked polymethacrylate (as shown in figure 2.5)
- Phenol-formaldehyde

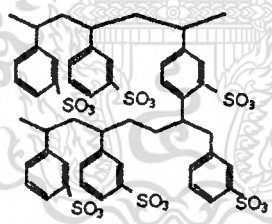

Figure 2.4 : Strong acid cationic resin polystyrenic type

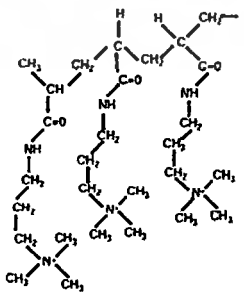

Figure 2.5 : Strong base anionic resin polyacrylic type

The polystyrene type is the most commonly used. The functional group determines whether the ion exchange is a cationic type or an anionic type.

2.3.1 Types of ion exchange resins

2.3.1.1 Strong acid cation resins

Strong acid resins are so named because their chemical behavior is similar to that of a strong acid. The resins are **highly ionized** in both the **acid ($R\text{-}SO_3H$)** and **salt ($R\text{-}SO_3Na$)** form of the **sulfonic acid group.** They can convert a metal salt to the corresponding acid by the reaction :

$$2(R\text{-}SO_3H) + NiCl_2 \longrightarrow (R\text{-}SO_4)Ni + 2HCl$$

The hydrogen and sodium forms of strong acid resins are highly dissociated and the exchangeable Na^+ and H^+ are readily available for exchange **over the entire pH range.** Consequently, the exchange capacity of strong acid resins is **independent of solution pH.** These resins would be used in the **hydrogen form** for **complete deionization;** they are used in the **sodium form** for **water softening** (calcium and magnesium removal). After exhaustion, the resins are converted back to the hydrogen form (regenerated) by contact with a strong acid solution, or the resins can be convened to the sodium form with a sodium chloride solution. For the above reaction, hydrochloric acid (HCl) regeneration would result in a concentrated nickel chloride ($NiCl_2$) solution.

2.3.1.2 Weak acid cation resins

In the weak acid resins, the ionizable group is a **carboxylic acid (COOH)** as opposed to the sulfonic acid group (SO_3H) used in strong acid resins [5]. These resins behave similarly to weak organic acids that are weakly dissociated. The **degrees of dissociation of the weak acid resins are strongly influenced by the solution pH.** Consequently, resins capacity depends in part on solution pH. The typical weak acid resins have limited capacity below a pH of 6.0, making them unsuitable for deionizing acidic metal finishing wastewater.

2.3.1.3 Strong base anion resins

Like strong acid resins, strong base resins are **highly ionized** and can be used **over the entire pH range**. These resins are used in the **hydroxide (OH)** form for water deionization. They will react with anions in solution and can convert an acid solution to pure water:

$$R\text{-}NH_3OH + HCl \longrightarrow R\text{-}NH_3Cl + HOH$$

Regeneration with concentrated sodium hydroxide (NaOH) converts the exhausted resins to the hydroxide form.

2.3.1.4 Weak base anion resins

Weak base resins are like weak acid resins in that the **degree of ionization** is **strongly influenced by pH.** Consequently, weak base resins exhibit minimum exchange capacity above a pH of 7.0. The weak base resins do not have a hydroxide ion form as does the strong base resin. These resins **merely absorb strong acids**, they cannot split salts.

$$R\text{-}NH_2 + HCl \longrightarrow R\text{-}NH_3Cl$$

Consequently regeneration needs only to neutralize the absorbed acid, it need not provide hydroxide ions. Less expensive weakly basic reagents such as ammonia (NH_3) or sodium carbonate can be employed.

2.3.1.5 Heavy-metal-selective chelating resins.

Chelating resins behave similarly to weak acid cation resins but exhibit a high degree of selectivity for heavy metal cations. Chelating resins are analogous to chelating compounds found in metal finishing wastewater; that is, they tend to form stable complexes with the heavy metals. In fact, the functional group used in these resins is an EDTA compound. The resins structure in the sodium form is expressed as R-EDTA-Na.

The high degree of selectivity for heavy metals permits separation of these ionic compounds from solutions containing high background levels of calcium, magnesium, and sodium ions. The chelating resins exhibit greater selectivity for heavy metals in its sodium form than in its hydrogen form. Regeneration properties are similar to those of the weak acid resins. Their high cost is the disadvantage of using the heavy-metal-selective chelating resins.

2.3.2 Properties of ion exchange resins

2.3.2.1 Crosslinkage

The amount of crosslinking depends on the proportions of different monomers used in the polymerization step. Practical ranges are 4 % to 16 % [5]. Resins with very low crosslinking tend to be watery and change dimensions markedly depending on which ions are bound. Properties that are interrelated with crosslinking are:

- Moisture Content

A physical property of the ion exchange resins that changes with changes in crosslinkage is the moisture content of the resins. For example sulfonic acid groups attract water, and this water is tenaciously held inside each resins particle. The quaternary ammonium groups of the anion resins behave in a similar manner. The figure 2.5 shows a plot of moisture content change with changes in crosslinkage.

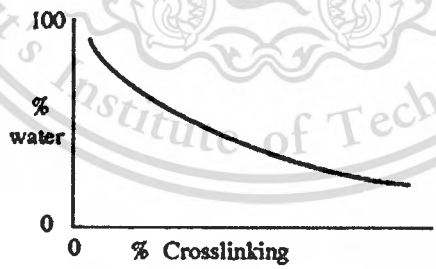

Figure 2.6 : Moisture content versus crosslinkage

- Equilibration Rate

Ion exchange reactions are reversible reactions with equilibrium conditions being different for different ions. Crosslinkage has a definite influence on the time required for an ion to reach equilibrium.

The ion exchange resins that are highly crosslinked quite resist to the diffusion of various ions through them and hence, the time required to reach equilibrium is much longer

- Capacity

The total capacity of the ion exchange resins is defined as the total number of chemical equivalents available for exchange per some unit weight or unit volume of resins. The capacity may be expressed in the terms of milliequivalents per dry gram of resins or in the terms of milliequivalents per dry gram of resins or in the terms of millequivalents per milliliter of wet resins. The more highly crosslinked the resins, the more difficult it becomes to introduce additional funcitonal groups. Sulfonation is carried out after the crosslinking has been completed and the sulfonic acid groups are introduced inside the resins particle as well as over its surface. Likewise, the quaternary ammonium groups are introduced after the polymerization has been completed and they too are introduced both inside the particle as well as on its surface. Fewer functional groups can be introduced inside the particles when they are highly crosslinked and hence the total capacity on a dry basis drops slightly.

This situation is reversed when a wet volume basis is used to measure the capacity on resins. Although fewer functional groups are introduced into the highly crosslinked resins, these groups are spaced closer together on a volume basis because the volume of water is reduced by the additional crosslinking. Thus the capacity on a wet volume basis increases as cross-linking increases. The figure 2.6 describes the changes in capacity as crosslinking is changed.

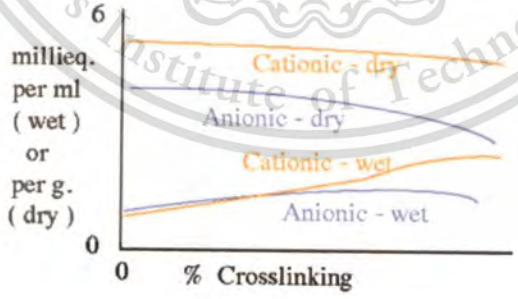

Figure 2.7 : Typical resins capacities

2.3.2.2 Particle size

The physical size of the resins particle is controlled during the polymerization step. Screens are used to sieve resins to get a fairly uniform range of sizes. Mesh sizes in the following table refer to U.S. Standard screens. A higher mesh number means more and finer wires per unit area and thus a smaller opening. Properties that are interrelated with Particle size are equlibration rate and flow rate.

2.3.2.3 Equilibration Rate

The particle size of the ion exchange resins influence the time required to establish equilibrium conditions. There are two types of diffusion that must be considered in ion exchange equilibrium. The first is called film diffusion or the movement of ions from a surrounding solution to the surface of an ion exchange particle. The second is called internal diffusion and is the movement of ions from the surface to the interior of an ion exchange particle. Film diffusion is usually the controlling reaction in dilute solutions whereas internal diffusion is controlling in more concentrated solutions. The particle sizes of the ion exchange resins affect both film diffusion and internal diffusion. A fine mesh particle presents more surface area for film diffusion and also contains less internal volume through which an ion must diffuse. A decrease in particle size thus shortens the time required for equilibration.

2.3.2.4 Flow Rate

Ion exchange processes are usually carried out in columns with the resins resting on a suitable support. Liquids may be processed either up-flow or down-flow through such columns. The spherical particles of ion exchange resins resist the flowing of a liquid through or around them. The smaller the particle size, the greater will be this resistance against which a liquid must flow. This resistance goes up very rapidly when particles smaller than 100 mesh are employed. Figure 2.7 illustrates the decrease in flow rates with decreasing particle sizes.

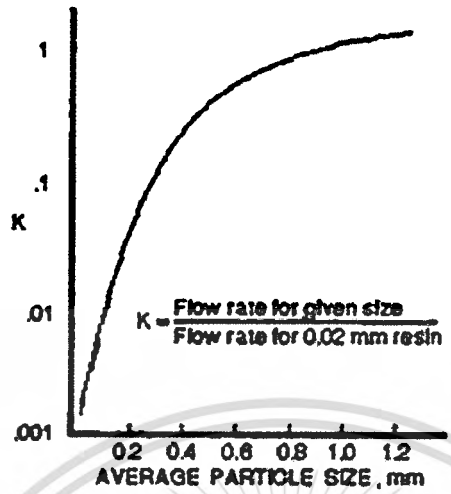

Figure 2.8 : Flow rate in relation to particle sizes

(Illustrates the decrease in flow rates with decreasing particle sizes)

2.4 Amberlysts-15 [5]

2.4.1 Amberlysts-15 wet

Amberlysts-15 wet are the strongly acidic, sulfonic acid, macroreticular polymeric resins based on crosslinked styrene divinylbenzene copolymers. Their continuous open pore structure and excellent physical, thermal and chemical stability makes them the resins of choice in many applications. They also possess a greater resistance to oxidants such as chlorine, oxygen and chromates than most other polymeric resins. Amberlysts-15can be used directly in aqueous systems or in organic media after conditioning with a water miscible solvent .

Amberlysts-15 wet are used in a wide variety of organic reactions. They have the optimal balance of surface area, acid capacity, activity and pore diameter to make them the catalysts of choice for etherification (MTBE, ETBE, TAME), esterification and hydration reactions. Their optimized pore size distribution makes them an excellent catalyst when fouling is anticipated. The typical properties were shown in table 2.1 and table 2.2.

Table 2.1 Typical properties (These properties are typical but do not constitute specifications).

Properties	Value/Form
Physical form	Opaque beads
Ionic form as shipped	Hydrogen
Concentration of active sites [1]	≥ 1.7 eq/L ≤ 4.7 eq/kg
Moisture holding capacity [1]	52 to 57% (H^+ form)
Shipping weight	770 g/L (48 lbs/ft^3)
Particle size	
Uniformity coefficient	≤ 1.70
Harmonic mean size	0.60 to 0.85 mm
Fines content [1]	< 0.36 mm : 1.0% max
Coarse beads	> 1.18 mm : 5.0% max
Nitrogen BET	
Surface area	53 m^2/g
Average pore diameter	300 Å
Total pore volume	0.40 ml/g
Shrinkage	Water to methanol: 5%
	Water to MTBE: 9%
	Water to hexane: 22%
	Water to dry: 37%

*[1] Contractual value

Test methods are available on request.

Table 2.2 Suggested operating conditions.

Maximum operating temperature	120°C (250°F)
Minimum bed depth	1000 mm (39 inches)
Operating flow rate	1 to 5 BV/h (LHSV)
Pressure drop limitation	1 bar (15 psig) across the bed

Amberlysts-15 can be used for processes where ionic or organic impurities have to be removed or recovered from process liquor. Both cationic and anionic compounds can be removed through either ionic or adsorptive interactions of the polymer and their acidic groups with the impurity. Their excellent resistance against oxidation makes them the superior resins in many applications. The table 2.3 shows the suggested operating conditions.

Table 2.3 Suggested operating conditions.

Operating Conditions	Value	
pH rang	0-14	
Maximum operating temperature	120°C (250°F) in non aqueous media	
Minimum bed depth	1000 mm (39 inches)	
Service flow rate	1 to 40 BV/h (0.125 to 5 gpm/ft ^{3})	
Regenerants	**HCl**	**H_2SO_4**
Flowrate (BV/h)	4 to 8	4 to 8
Flowrate (gpm/ft 3)	0.5 to 1.0	0.5 to 1.0
Concentration (%)	4 to 10	1 to 5
Level (g/L)	40 to 100	40 to 200
Level (lbs/ft 3)	2.5 to 6	2.5 to 12
Minimum contact time	30 minutes	
Slow rinse	2 BV (15 gal/ft ^{3}) at regeneration flow rate	
Fast rinse	2 to 4 BV (15 to 30 gal/ft ^{3}) at service flow rate	

2.4.2 Amberlysts-15 dry

Amberlysts-15 dry are the bead form, strongly acidic ion exchange resins developed particularly for heterogeneous acid catalysis of a wide variety of organic reactions. They are also useful in non aqueous ion- exchange systems for the removal of cationic impurities.

The macroreticular pore structure of amberlysts-15 dry permits ready access of liquid or gaseous reactants to the hydrogen ion sites located throughout the bead, thus ensuring successful performance even in non swelling organic media. The main applications are alkylation, esterification, etherification, condensation hydrolysis. The typical properties were shown in table 2.4 and table 2.5 indicated the suggested operation conditions.

Table 2.4 Typical properties (These properties are typical but do not constitute specifications).

Properties	Value/Form
Physical form	Opaque beads
Ionic form as shipped	Hydrogen
Concentration of active sites [1]	4.7 eq/kg
Water content [1]	1.6% (H^+ form)
Shipping weight	38 g/L (610 lbs/ft^3)
Fines content [1]	< 0.36 mm : 1.0% max
Nitrogen BET	
Surface area	53 m^2/g
Average pore diameter	300 Å
Total pore volume	0.40 cc/g
Swelling dry to phenol	38%

*[1] Contractual value

Test methods are available on request.

Table 2. 5 Suggested operating conditions.

Maximum operating temperature	120°C (250°F)
Minimum bed depth	600 mm (24 inches)
Operating flow rate	1 to 5 BV/h (LHSV)
Pressure drop limitation	1 bar (15 psig) across the bed

2.5 Water Filter Resins [5]

Water filter resins are devices that can improve the overall taste, smell and appearance of drinking water and can remove some chemical substances. Used mainly for drinking and cooking purposes, filters are the most inexpensive and most easily available method of water purification. However, purification using filters is not 100%. In general, water filter resins remove only specific types of substances and are labeled for what they will remove, such as chlorine or lead. Water filters do not remove microorganisms; and, are intended for use with water that is known to be microbiologically safe. No single water filter resins can be used to remove all types of substances from water. There are many types of water filter resins models on the market. Different drinking water treatment systems have their own advantages and disadvantages and must be investigated individually to identify the unit or combination of units best suited for your household .

Types of water filter resins:

2.5.1 Particle Filters

These filters operate on one basic principle: they use a membrane to screen out or trap particles based on their size. These filters are rated according to the pore size of the membrane, which is measured in microns, the lower the number of microns (i.e. the smaller the pore size) the more effective the filter.

For example, while a filter that removes particles down to 5 microns in size produces clean water, one that stops particles 0.5 microns in size, produces cleaner water.

There are two common particle filters are fiber and ceramic:

Fiber filters are made out of cellulose, rayon, or another type of fiber. These filters have larger pores that prevent large particles such as dirt from crossing their tight fiber layer, but will allow dissolved contaminants such as lead and mercury to pass through.

Ceramic filters, like fiber filters, trap asbestos fibers, some bacteria and parasites (though not enough to disinfect the water) and other particles in its small ceramic pores. They will not remove dissolved chemical contaminants such as sodium.

2.5.2 Activated Carbon (AC) Filters

Activated carbon filtration as shown in figure 2.8 is most effective in removing organic contaminants from water. Because organic chemicals are often responsible for taste, odor and color problems, activated carbon filtration can generally be used to improve aesthetically objectionable water that is treated to attract certain contaminants. When water passes through this type of filter, the carbon particles attract and remove contaminants (including dissolved substances such as hydrogen sulphide, heavy metals such as lead, mercury and copper and chlorine).

There are two types of activated carbon filters are granular and solid block:

Granular activated carbon (GAC) filters use a cartridge packed with granules of activated carbon. When water passes through the filter, the filter's numerous carbon granules trap particles and remove substances dissolved in the water (such as chlorine, heavy metals and harmful organic compounds).

A Solid-Block Activated Carbon filter have activated carbon not in the form of small granules, but instead is composed of activated carbon particles that have been compressed into a dense material through which water travels. With very small pores and a larger surface area for the carbon to absorb particles, there is a better chance of trapping contaminants such as pesticides, chlorine, lead and asbestos than there is with other filters.

Figure 2.9 : Solid-Block Activated Carbon filters

2.5.3 Resins Filters

Resins filters as shown in figure 2.9 consist of a module that contains resins that can remove contaminants such as lead and other heavy metals, as well as minerals that cause deposits in kettles and coffee makers. These contaminants have an electrical charge and are removed by attaching to an opposite charge found on the resins. Resin filters can be combined with activated carbon filters to remove a wide range of particles and dissolved substances.

Figure 2.10 : Resins Filters

2.6 Characterization Techniques

2.6.1 Feed and product characterizations

2.6.1.1 Gas Chromatography-Mass Spectrometry (GC-MS) [5]:

Gas chromatography-mass spectrometry is a method that combines the features of gas-liquid chromatography and mass spectrometry to identify different substances within a test sample. Applications of GC-MS include drug detection, fire investigation, environmental analysis, explosives investigation, and identification of unknown samples. GC-MS can also be used in airport security to detect substances in luggage or on human beings. Additionally, it can identify trace elements in materials that were previously thought to have disintegrated beyond identification.

The GC-MS is composed of two major building blocks: the gas chromatograph (as shown in figure 2.11) and the mass spectrometer (as shown in figure 2.12). The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties (e.g. 5% phenyl polysiloxane). The difference in the chemical properties between different molecules in a mixture will separate the molecules as the sample travels the length of the column. The molecules take different amounts of time (called the retention time) to come out of (elute from) the gas chromatograph, and this allows the mass spectrometer downstream to capture, ionize, accelerate, deflect, and detect the ionized molecules separately. The mass spectrometer does this by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio.

These two components, used together, allow a much finer degree of substance identification than either unit used separately. It is not possible to make an accurate identification of a particular molecule by gas chromatography or mass spectrometry alone. The mass spectrometry process normally requires a very pure sample while gas chromatography using a traditional detector (e.g. Flame Ionization Detector) detects multiple molecules that happen to take the same amount of time to travel through the column (*i.e.* have the same retention time) which results in two or more molecules to co-elute. Sometimes two different molecules can also have a similar pattern of ionized fragments in a mass spectrometer (mass

spectrum). Combining the two processes reduces the possibility of error, as it is extremely unlikely that two different molecules will behave in the same way in both a gas chromatograph and a mass spectrometer. Therefore, when an identifying mass spectrum appears at a characteristic retention time in a GC-MS analysis, it typically lends to increased certainty that the analyte of interest is in the sample.

The equipment used for gas chromatography generally consists of an injection port at one end of a metal column packed with substrate material and a detector at the other end of the column. A carrier gas propels the sample down the column. The technician uses flow meters and pressure gauges to maintain a constant gas flow. A gas that does not react with the sample or column is essential for reliable results. For this reason, carrier gases are usually argon, helium, hydrogen, nitrogen, or hydrogen. Many analysts use helium because it does not react. Hydrogen usually is a good carrier gas but it may react and convert the sample into another substance. The ultimate choice for a carrier gas may depend on the type of detector used.

To ensure proper separation, the sample must enter the column in a discreet, compact packet. Normally the sample is injected into the injection port with a hypodermic needle and syringe capable of measuring the specimen amount. The needle is stuck into a replaceable neoprene or silicone rubber septum that covers the injection port. The injection port is maintained at a temperature at which the sample vaporizes immediately. Ideally, the sample spreads evenly along the cross section of the column, forming a plug.

The column is a metal tube, often packed with a sand-like material to promote maximum separation. Columns are commonly obtained pre-packed by vendors. As the sample moves through the column, the different molecular characteristics determine how each substance in the sample interacts with the column surface and packing. The column allows the various substances to partition themselves.

Substances that do not like to stick to the column or packing will move through the column rapidly. Substances that do not like to stick to the column or packing are impeded but eventually elute from the column. Ideally, the various components in the sample separate before eluting from the column end.

The GC instrument uses a detector to measure the different compounds as they emerge from the column. Among the available detectors are the argon ionization detector, flame ionization detector, flame

emission detector, cross section detector, thermal conductivity detector, and the electron capture detector. Choosing the proper detector depends upon the use. Some considerations are that the flame detectors destroy the sample, the thermal conductivity detector is universally sensitive, and the argon ionization detector requires argon as a carrier gas. The spectral output is usually stored electronically and displayed on a monitor. The technician can produce a hard copy record.

The argon ionization detector does not detect water, carbon tetrachloride, nitrogen, oxygen, carbon dioxide, carbon monoxide, ethane, or compounds containing fluorine. The flame ionization detector does not respond to water, nitrogen, oxygen, carbon dioxide, carbon monoxide, helium, or argon. If a specimen contains water, a flame ionization detector should be used. The electron capture detector cannot detect simple hydrocarbons but does detect compounds containing halides, nitrogen, or phosphorus.

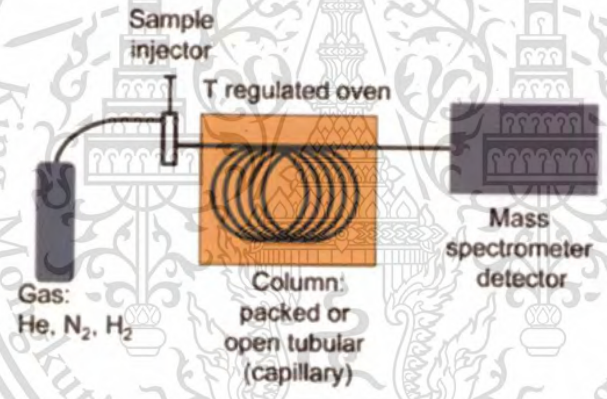

Figure 2.11 : GC-MS schematic

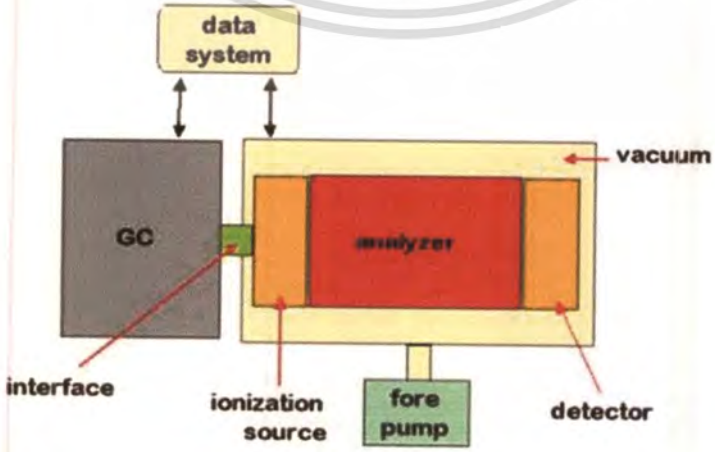

Figure 2.12 : Mass spectrometer components

2.6.1.2 Fourier Transform Infrared Spectroscopy (FTIR) [5]:

Fourier transform infrared spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time. FTIR technique has

made dispersive infrared spectrometers all but obsolete (except sometimes in the near infrared) and opened up new applications of infrared spectroscopy.

The term Fourier transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical algorithm) is required to convert the raw data into the actual spectrum.

The goal of any absorption spectroscopy (FTIR, ultraviolet-visible ("UV-Vis") spectroscopy, etc.) is to measure how well a sample absorbs light at each wavelength. The most straightforward way to do this, the "dispersive spectroscopy" technique, is to shine a monochromatic light beam at a sample, measure how much of the light is absorbed, and repeat for each different wavelength. Fourier transform spectroscopy is a less intuitive way to obtain the same information. Rather than shining a monochromatic beam of light at the sample, this technique shines a beam containing many different frequencies of light at once, and measures how much of that beam is absorbed by the sample. Next, the beam is modified to contain a different combination of frequencies, giving a second data point. This process is repeated many times. Afterwards, a computer takes all this data and works backwards to infer what the absorption is at each wavelength. The beam described above is generated by starting with a broadband light source one containing the full spectrum of wavelengths to be measured. The light shines into a certain configuration of mirrors, called an interferometer, that allows some wavelengths to pass through but blocks. The beam is modified for each new data point by moving one of the mirrors; this changes the set of wavelengths that pass through. As mentioned, computer processing is required to turn the raw data into the desired result (light absorption for each wavelength). The processing required turns out to be a common algorithm called the Fourier transform as shown in figure 2.12 (hence the name, "Fourier transform spectroscopy"). The raw data is sometimes called an "interferogram".

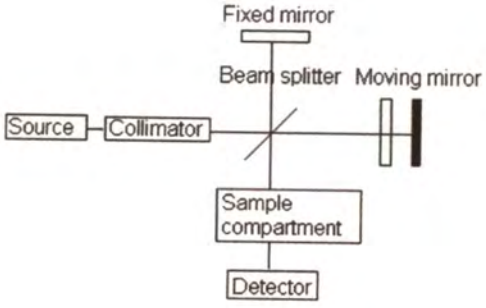

Figure 2.13 : Schematic diagram of a Michelson interferometer, configured for FTIR

2.7 Microwave

Microwaves are electromagnetic waves which have frequencies between 300MHz to 300GHz, so it means that they have wavelength about 1 millimeter to 1 meter. The boundary of microwave is between radio and infrared (in wavelength) as shown in figure 2.14:

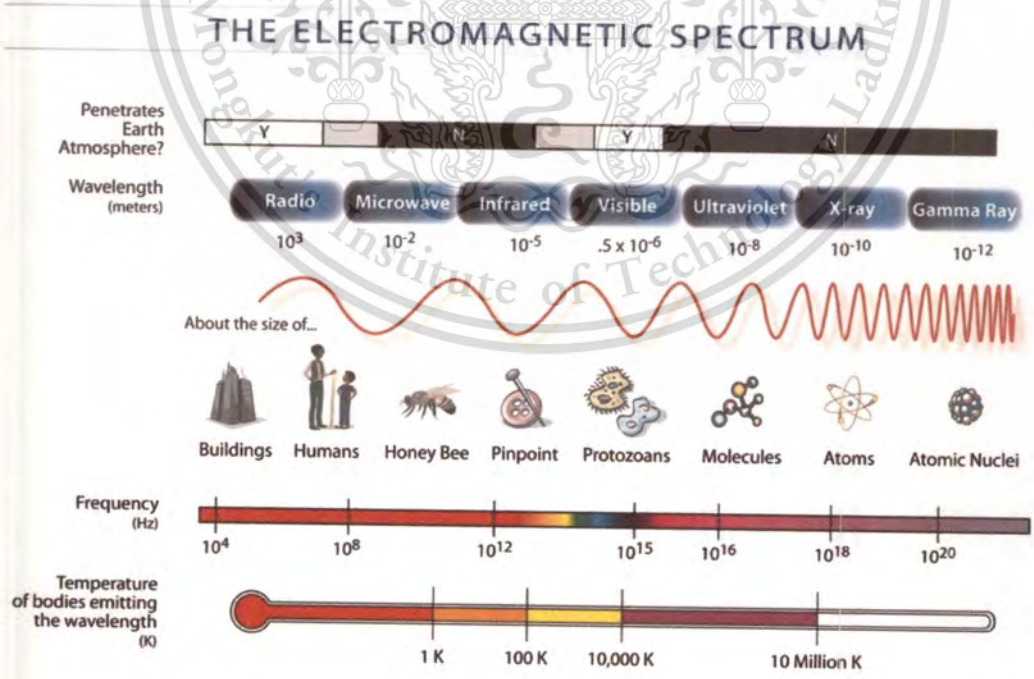

Figure 2.14 radio and infrared (in wavelength)

A machine which uses this technique well known as "Microwave", it can generate wavelengths of signals roughly the same as the dimensions equipment. Microwaves can also be used as distributer circuit elements and transmission-line theory are more useful methods for design and analysis. . Open-wire and coaxial transmission lines give way to waveguides and stripline , and lumped-element tuned circuits are replaced by cavity resonators or resonant lines. Effects of reflection, polarization, scattering, diffraction and atmospheric absorption usually associated with visible light are of practical significance in the study of microwave propagation. The same equations of electromagnetic theory apply at all frequencies.

Microwave oven uses microwave radiation (non-ionizing at about 2.45 GHz) passing through food; causing dielectric heating then fats, water or high polar compound absorb the energy. Water in the liquid state possesses many molecular interactions which broaden the absorption peak. In the vapor phase, isolated water molecules absorb at around 22 GHz, almost ten times the frequency of the microwave oven. The oven became famous in Western countries as a food heating more than 30 years now.

In the recent decade microwave irradiation has been becoming famous instead of the conventional heating in many chemical reactions. In many cases, accelerated chemical reaction by using microwave-assisted have higher yield under milder condition. The microwave heating has been recently applied to the biodiesel production. It was proven that transesterification with base homogeneous catalysts could be significantly accelerated under microwave heating. Microwave heating is used in industrial processes for drying and curing products. Many semiconductor processing techniques use microwaves to generate plasma for such purposes as reactive ion etching and plasma-enhanced chemical vapor deposition (PECVD). It also frequencies typically ranging from 110 – 140 GHz are used in stellarators and more notably in tokamak experimental fusion reactors to help heat the fuel into a plasma state. The upcoming ITER Thermonuclear Reactor is expected to range from 110–170 GHz and will employ Electron Cyclotron Resonance Heating (ECRH).

Microwaves do not contain sufficient energy to chemically change substances by ionization, and so is an example of nonionizing radiation. The word "radiation" refers to the fact that energy can radiate. The term in this context is not to be confused with radioactivity. It has not been shown conclusively that microwaves (or other nonionizing electromagnetic radiation) have significant adverse biological effects at low levels. Some, but not all, studies suggest that long-term exposure may have a carcinogenic effect. This is separate from the risks associated with very high intensity exposure, which can cause heating and burns like any heat source, and not a unique property of microwaves specifically.

When injury from exposure to microwaves occurs, it usually results from dielectric heating induced in the body. Exposure to microwave radiation can produce cataracts by this mechanism, because the microwave heating denatures proteins in the crystalline lens of the eye (in the same way that heat turns egg whites white and opaque) faster than the lens can be cooled by surrounding structures. The lens and cornea of the eye are especially vulnerable because they contain no blood vessels that can carry away heat. Exposure to heavy doses of microwave radiation (as from an oven that has been tampered with to allow operation even with the door open) can produce heat damage in other tissues as well, up to and including serious burns which may not be immediately evident because of the tendency for microwaves to heat deeper tissues with higher moisture content.

Literature Review

Ritwan Masae and his team have interested in large surplus of glycerol that is formed as a byproduct from biodiesel production and finding the alternative use of this byproduct.

They have studied the esterification of glycerol with glacial acetic acid over acidic ion exchange resin as a function of several reaction parameter, such as, type of catalyst, amount of catalyst, reaction temperature and mole ratio. The study was to prove that water filter resin with sulfonic acid type based upon a styrene-divinylbenzene copolymer can be used as substitute for Amberlyst-15 catalyst after it were converted into acid form. The product produced is triaccetyl glycerol which used as additive for biodiesel and many other used. The optimal condition is temperature of 95°C and an acetic acid to glycerol mole ratio is 9:1 over 5% weight of water filter resins as catalysts. Higher the temperature than 95°C will result in lower the conversion received. The result show that percent conversion of glycerol reached to 99.7 while the percent conversion to triacetylglycerol was 16.05% compare to reaction using Amberlyst-15 as catalyst at it optimum condition, using temperature of 105°C, acetic acid to glycerol mole ratio of 9:1 with 5% weight of Amberlysts-15, give conversion to triacetyl glycerol equal to 22.05% . This result indicated that water filter resin can be used as substitute of Amberlyst-15 in this reaction[5].

Xiaoyuan Liao had studied a two-step method to obtain high selectivity and high conversion rate triacetylglycerol from glycerol. The esterification of glycerol with acetic acid was carried out over many type of resins and zeolites. Amberlysts-35 was found to be an excellent catalyst. The reaction conditions were optimized by testing catalysts, temperatures, feedstock ratios as well as loads of catalysts. The optimal conditions an acetic acid to glycerol molar ratio of 9:1 with 5% weight of catalysts at reaction temperature for 4 hour. After the reaction of the optimal condition, the selectivity of triacetylglycerol reaches almost 100% in 15 min by adding into acid anhydride. Recycling experiments indicate that no significant deactivation of Amerlysts-35 occurred during the reaction[8].

B. Toukoniitty had studied the esterification of propionic acid with ethyl alcohol over an ion-exchange resin catalyst under both microwave dielectric heating and convective/conductive heating. Series of kinetic experiments were carried out in a single-mode microwave loop reactor, equipped with a heating band as well to directly compare the efficiency of both heating. The reaction were carried out at reaction temperature of 105 8C and total pressure of 7 bar. Different initial molar ratios of propionic acid-to-ethanol (from 1:2 to 2:1) were investigated. The result shown that the highest product yield was observed with equimolar initial ratio but the kinetics and equilibrium of this reaction were unaffected by the method of heating[9].

Chapter 3

Experimental Details

3.1 Instrument and Apparatus

3.1.1 Fluke 561 IR- Thermometer with stainless thermocouple.

3.1.2 Vacuum filter

3.1.3 Gas Chromatography equipped with Mass spectrometer

3.1.4 Fourier Transform Infrared Spectroscopy

3.1.5 pH indicator

3.1.6 Glass apparatus

3.1.7 Samsung's commercial microwave oven model MW73CD-E

3.2 Chemicals

3.2.1) Commercial grade Glycerol ($CH_2OHCHOHCH_2OH$)

3.2.2) Commercial grade Glacial Acetic acid (CH_3COOH)

3.2.3) Amberlysts-15 (A-15)

3.2.4) 1.0M Commercial grade Nitric acid (HNO_3)

3.2.5) Water filter resins

3.2.6) Distilled water

3.3 Experimental preparations

3.3.1 Convert water filter resins to hydrogen form

3.3.1.1) Place approximately 5 g of water filter resins in the funnel placed over a 1.5 L beaker.

3.3.1.2) Rinse 1 L of 1 M nitric acid through water filter resins and left for 1 night.

3.3.1.3) Rinse excess acid off by distilled water then filter it by vacuum filter.

3.3.1.4) Dry the resin at 100°C in the oven for 8 hours.

3.3.2 Finding the temperature of microwave irradiation for each power and time.

3.3.1.2) Weight 9.2g of glycerol and mix with 54g of acetic acid for the mole ratio of acetic acid : glycerol at 9:1.

3.3.1.2) Place the mixture into the microwave then measure the temperature in different Microwave power and time then record the results.

3.4 Experimental procedures

3.4.1 Test for optimal microwave condition.

3.4.1.1) Weight 9.2g of glycerol and mix with 54g of acetic acid for the mole ratio of acetic acid : glycerol at 9:1.

3.4.1.2) Place mixture into the microwaveable apparatus along with or without 0.48 g of catalyst (amberlyst-15 or activated water filter resins) and put into microwave.

3.4.1.3) Set the condition of microwave as table 3.1 then start.

3.4.1.4) Test the product with FT-IR and GC-MS.

3.4.1.5) Compare the result for the optimal condition.

3.4.2 Test the combine microwave condition.

3.4.2.1) Weight 9.2g of glycerol and mix with 54g of acetic acid for the mole ratio of acetic acid : glycerol at 9:1.

3.4.2.2) Place mixture into the microwaveable apparatus along with 0.48 g of catalyst (amberlyst-15 or activated water filter resins) and put into microwave.

3.4.2.3) Set the condition of microwave at 300w for 120 second then start

3.4.2.4) After first condition finish, set the condition of microwave at 100w for 180 second then start.

3.4.2.5) Test the product with FT-IR and GC-MS.

Table 3.1 : The experimental by varying the type of catalysts, microwave power and time

Molar ratio of acetic acid : glycerol	Catalyst (5% weight)	Microwave power (Watt)	Time (sec)
9:1	No catalyst	100	360
			540
		300	90
			120
	Amberlyst-15	100	360
			540
		300	60
			90
			120
	Water filter resins	100	360
			540
		300	60
			90
			120

Chapter 4

Results and Discussion

4.1 Relation of Microwave power, time and temperature

From the research [5], the experiments were carried out with various temperature and were found that the best condition for production of Triacetylglycerol is 9:1 of acetic acid to glycerol, 5% weight of water filter resins at 95°C and 6:1 of acetic acid to glycerol, 5% weight of Amberlysts-15 at 105°C. But conventional microwave oven does not has the temperature control function, to resemble those condition the final temperature of each microwave power and time were acquired.

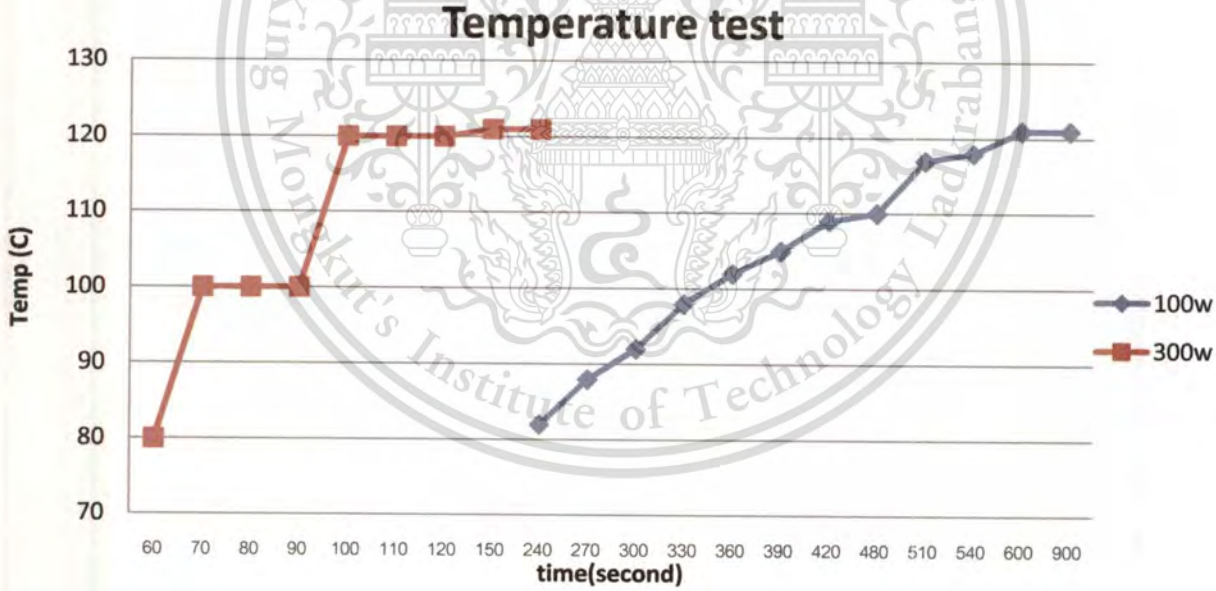

Figure 4.1 : The temperature from each Microwave power related to time

Figure 4.1 show the temperature from each Microwave power with time. The desired temperatures are 95°C and 105°C. At 300w, 60 and 90 second were selected and 360 and 540 second were selected for 100w since they give the nearest value while 120 second at 300w are selected to study that at temperature more than 100°C still give lower conversion under Microwave irradiation. Note that the temperature selected cannot be exceeding 120°C because the catalyst's structure will be collapse.

Also, combined condition was created with initial high microwave power at 300w for 120 second to accelerate the temperature to the point before maintain the temperature for longer period with low powered 100w for 180 second to study the effect of accelerated heated compare to normally heated.

4.2 Fourier Transform Infrared (FTIR) Spectroscopy Analysis

The FTIR Spectra of liquid films placed between the KBr windows were registered within the range of 4000-400 cm^{-1} and were used with Perkin-Elmer System 2000R spectrometer.

Figure 4.2 is the example of product from esterification reaction under microwave irradiation at microwave power of 100w for 540 second using Amberlyst-15 as catalyst

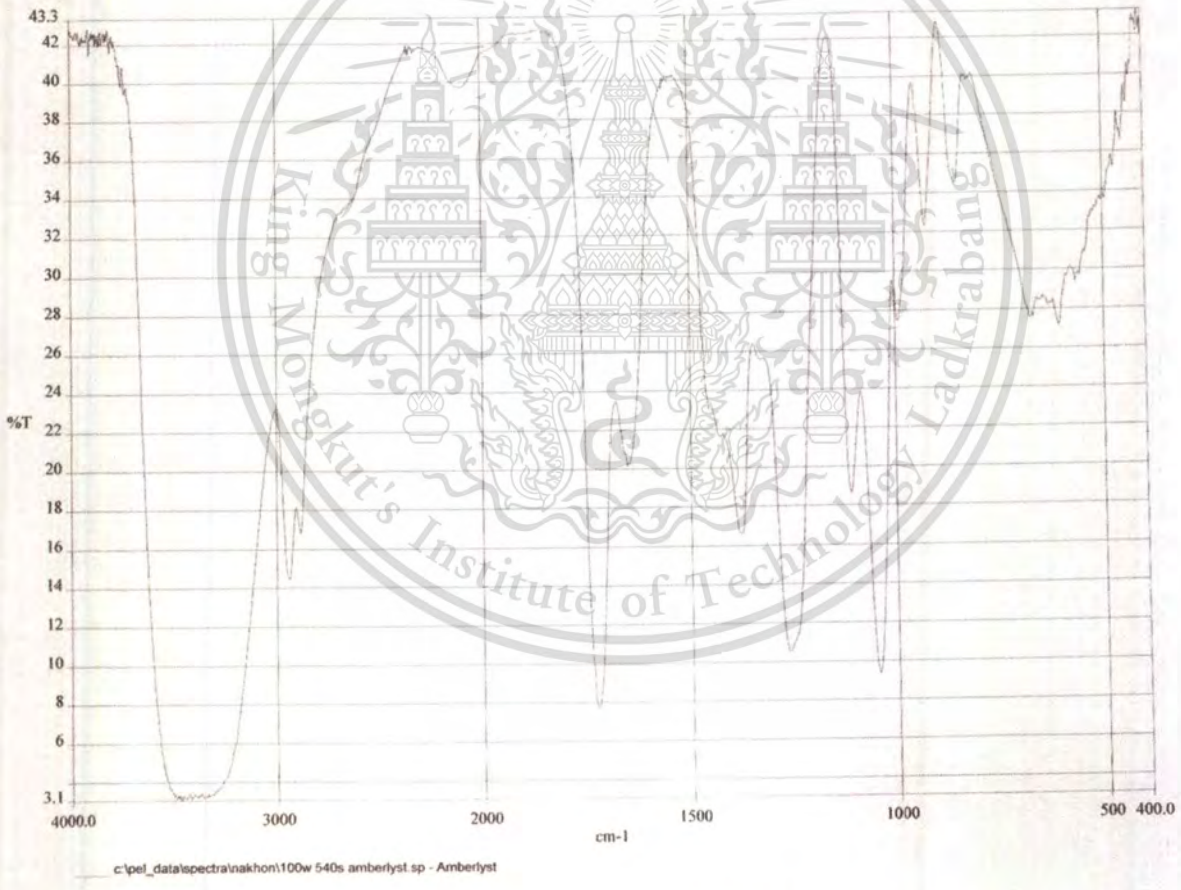

Figure 4.2 : The FTIR spectrum of the esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 100w and 540 second.

The first intense stretching band at 3462 cm^{-1} indicates to the υ(OH) stretching band that come from OH-groups of glycerol, acetic acid left from reactant, mono-, di-, triacetylglycerol product along

with water as byproduct mix together which lead to a very intense stretching band. Next intense band is at 1727 cm^{-1} which represent ν(C=O) from carbonyl ester. The acetate νCC(=O)-O stretching and the δ (C-H) bending of CH_3 band ,which is characteristic signal of triacetylglycerol, can be found at 1264 cm^{-1} and 1378 cm^{-1} respectively. The secondary ν(C-O) stretching band at 1046cm^{-1} and the primary ν(C-O) stretching band at 1111 cm^{-1} indicate C-O bond that can be found in both glycerol and triacetylglycerol structure.

4.3 Gas Chromatography- Mass Spectrometry (GC-MS) Analysis.

The products were measured in both qualitative and quantitative aspect by using Gas Chromatrography-Mass spectrometer (GC-MS). The figure shows an example of typical gas chromatogram of the product and can be further analyse for quantity by using integrated area for percentage of products (mono-, di-, triacetylglycerol).

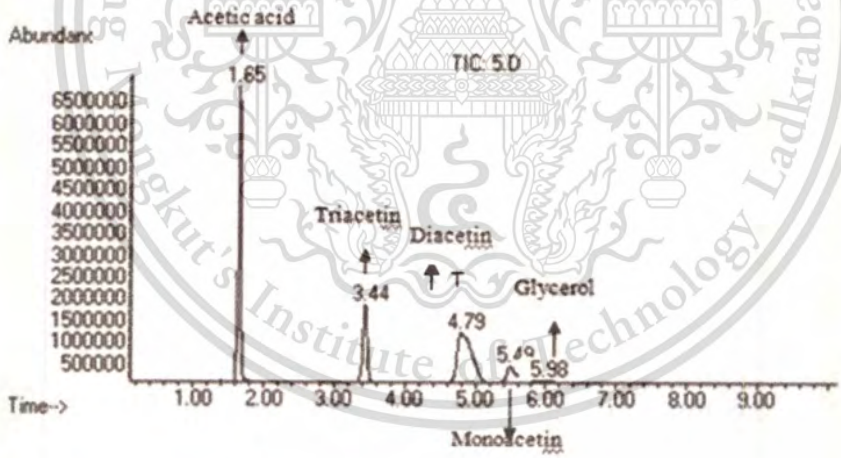

Figure 4.3 : Gas Chromatogram of the esterification reaction products (between acetic acid and glycerol)

4.4 Effect of reaction condition

The effect of catalyst types, microwave power and special condition were determined and the results are discussed in the following sections.

4.4.1 Effect of catalyst types

From the Gas Chromatography Spectrometry (GC-MS), the product mixture of esterification reaction were carried out resemble to the research [5]. The best condition of the research is 9:1 of acetic acid to glycerol, 5% weight of water filter resins at 95°C and 6:1 of acetic acid to glycerol, 5% weight of Amberlysts-15 at 105°C which give the conversion to triacetylglycerol equal to 16.05% and 22.05% respectively. For this experiment, the best condition for water filter resin is 300w for 120 second then 100w for 180 second with 1.11% and for Amberlyst-15 is 300w for 120 second then 100w for 180 second with 1.34%. This shows almost the same conversion for both Amberlyst-15 and water filter resin which indicates that water filter resin can be used as substitute of Amberlyst-15

4.4.2 Effect of Microwave power and time

The result is also being observed that the effect of Microwave irradiation is affect the reaction or not. The observation shall be categories in each catalyst.

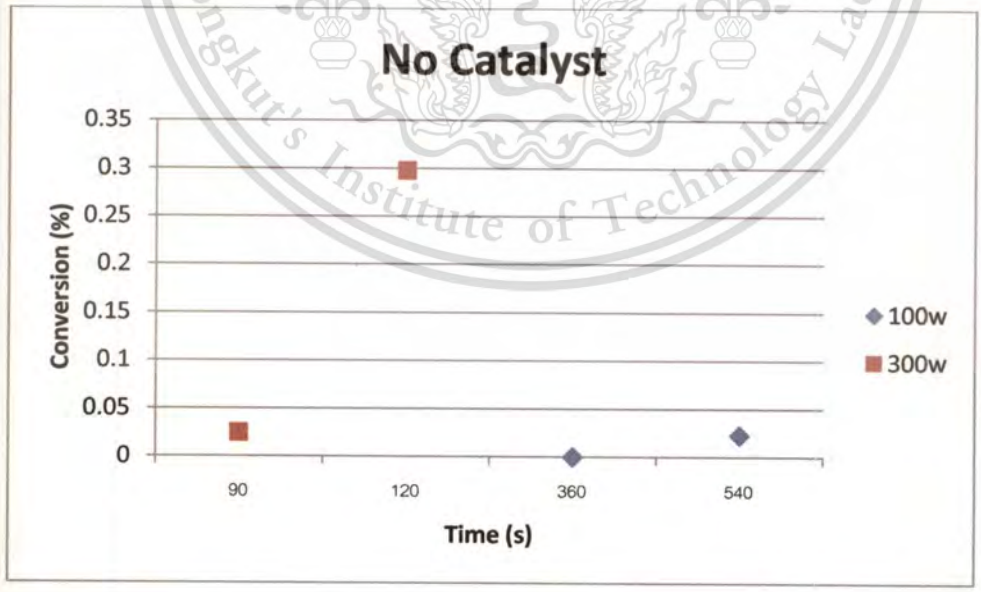

Figure 4.4 : The effect of Microwave power on the percentage of triacetylglycerol conversion. All reaction were performed at 9:1 acetic acid per glycerol mole ratio with no catalyst.

Figure 4.4 show the percentage of triacetylglycerol conversion with varies microwave power and time. As it can be seen, the conversion is very low or no reaction at all since there is no present of any catalyst. The highest conversion here is at 300w for 120 second with 0.298% while others show almost no sign of reaction even with 100w for 540 second that reach the same temperature with much longer time. But according to temperature data shows that 300w 120 second has reach 120°C since 100 second passed. This shows that microwave high acceleration of temperature rising play a significant role in this reaction.

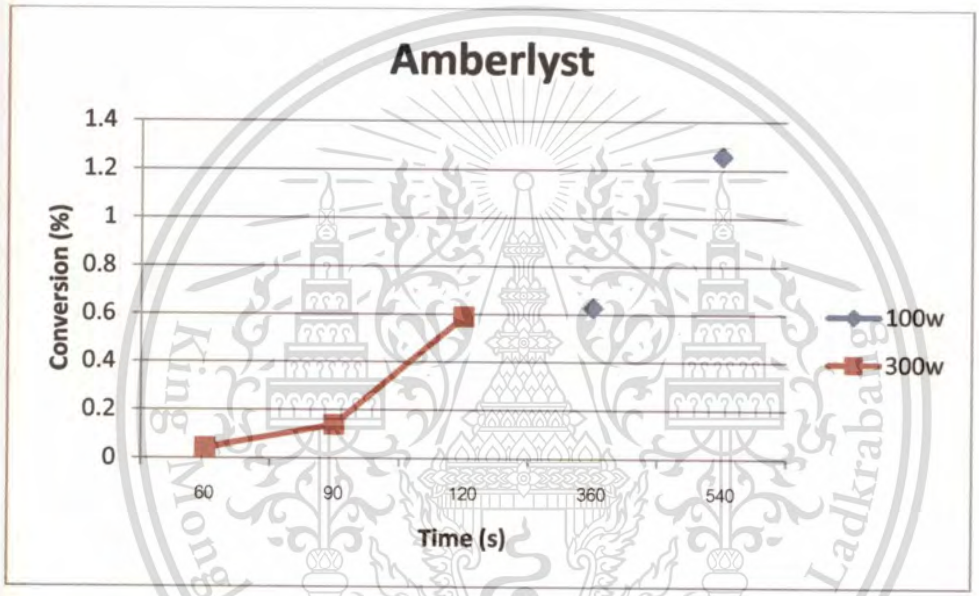

Figure 4.5 : The effect of Microwave power on the percentage of triacetylglycerol conversion. All reaction was performed at 9:1 acetic acid per glycerol mole ratio with Amberlyst-15 as catalyst.

Figure 4.5 show the percentage of triacetylglycerol conversion with varies microwave power and time of esterification reaction using Amberlyst-15 as catalyst. The best conversion here is at 100w for 540 second with 1.256%. In this case the microwave power does not much affect the reaction as 300w 360 second conditions give lower conversion compare to 100w 60second conditions even they have approximately same temperature according to temperature test, this show that the important part here is time used for reaction.

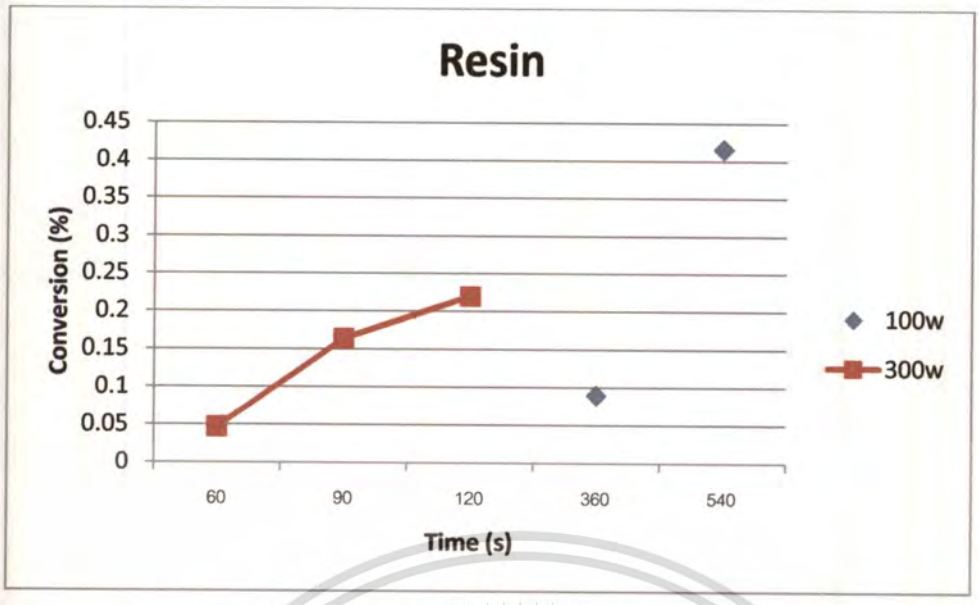

Figure 4.6 : The effect of Microwave power on the percentage of triacetylglycerol conversion. All reaction was performed at 9:1 acetic acid per glycerol mole ratio with Water filter resin as catalyst.

Figure 4.6 show the percentage of triacetylglycerol conversion with varies microwave power and time of esterification reaction using Water filter resin as catalyst. The highest conversion in this case is at 100w for 540 second with 0.416%. the interesting point here is at 300w for 120 second that gives 0.22% conversion which is lower than using no catalyst at all. The reason is water filter resin cannot tolerate to the temperature over 120°C and became deactivated.

4.4.2 Effect of combined microwave condition.

The idea of combined condition is to accelerate the temperature rise to the desire point with high microwave power before trying to maintain it with low microwave power. The result is sastifactoty with 1.34% conversion to triacetylglycerol using Amberlyst-15 catalyst and 1.11% using Water filter resin. These conditions gives a higher conversion due to the longer time it reach reaction temperature since 300w period has heat it up with higher speed that the result is exceed its 540 second condition.

Chapter 5

Conclusion and Recommendations

5.1 Conclusion

The esterification of glycerol with acetic acid under microwave irradiation using water filter resin and Amberlyst-15 as catalyst with various conditions was studied. Gas Chromatography-Mass Spectrometry (GC-MS) and Fourier Transform Infared spectroscopy (FTIR) were used to characterize the product mixture (Mono-, di-, triacetylglycerol) in both quantitative and qualitative. The product was focused on triacetylglycerol which can be used as a good additive for biodiesel to improve many properties ,for example, cold flow and viscosity.

To optimized the reaction, the experiments were carried out under varies microwave power and time. The result show that the major impact to the result is time, the longer time with low microwave power give better conversion compare to higher power with shorter time. The combined microwave condition also show a good combination which give a higher conversion to triacetylglycerol with lower time. The optimal condition were found to be 300w for 120 second then 100w for 180 second with 1.11% conversion and for Amberlyst-15 is 300w for 120 second then 100w for 180 second with 1.34% conversion to triacetylglycerol. But even with optimal condition, it's still far lower than the previous research (with 22.05% conversion at a temperature of 105°C and an acetic acid to glycerol conversion is 9:1 over 5% weight of Amberlyst-15 as catalysts) but for instance, reaction under microwave irradiation processed a very high selectivity toward monoacetylglycerol. The higher temperature (>120°C) can be resulted in deactivation of Water filter resin.

5.2 Recommendations

The recommendations for further study are the influence of microwave irradiation wavelength over the reaction and the difference of using pulse microwave from household microwave oven. One should also design the reaction to have better seal along with modification to the microwave itself for preventing the reactant lost during experiment and a better mixing. Ones also should study with the technique for effectively separate a mixture of esterification reaction products.

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Appendices

Appendix A : Experimental data

Table A.1.1 : The experimental results of temperature testing at 100w (°C)

Time(sec)	240	270	300	330	360	390	420	480	510	540	600
1st time	82.0	88.0	90.6	98.3	102.0	104.7	109.0	110.0	116.8	118.0	121.0
2nd time	81.8	88.4	93.3	99.0	102.0	105.8	109.2	109.7	117.5	118.3	121.1
3rd time	83.0	88.0	93.3	98.2	101.0	104.7	109.3	109.6	116.9	118.3	121.0
mean	82.27	88.13	92.4	98.5	101.67	105.07	109.17	109.77	117.06	118.2	121.03

Table A.1.2 : The experimental results of temperature testing at 100w (°C)

Time(sec)	60	70	80	90	100	110	120	150	240
1st time	82.0	100.0	100.1	100.0	120.0	120.1	120.0	121.0	121.0
2nd time	81.8	100.0	100.0	100.0	120.0	120.0	120.1	121.0	121.0.0
3rd time	83.0	100.0	99.9	100.0	120.0	120.0	119.9	121.1	121.0
mean	80	100.0	100.0	100.0	120.0	120.1	120.0	121.03	121

Table A.2 : The experimental results by varying type of catalyst, microwave power(w) and time(s).

Molar ratio of acetic acid : glycerol	Catalyst (5%weight)	Microwave power (Watt)	Time(sec)	Conversion of glycerol (%)	Conversion to triacetylglycerol (%)	Conversion to diacetylglycerol (%)	Conversion to monoacetylglycerol (%)
9:1	No catalyst	100	360	75.158	0	0.457	8.687
			540	78.442	0.023	1.627	12.186
		300	90	74.561	0.025	0.618	7.904
			120	73.281	0.298	2.950	14.963
	Amberlyst-15	100	360	79.333	0.628	4.685	16.443
			540	77.273	1.256	8.973	24.365
		300	60	82.493	0.041	1.757	11.205
			90	79.964	0.138	1.496	10.425
			120	83.039	0.590	4.883	16.630
		300 then 100	120 then 180	74.659	1.346	8.425	22.641
	Water filter resins	100	360	76.701	0.090	1.376	9.754
			540	85.63	0.416	2.382	8.706
		300	60	77.500	0.047	0.749	7.491
			90	78.094	0.164	1.508	8.711
			120	81.121	0.220	1.562	10.053
		300 then 100	120 then 180	87.824	1.111	5.892	17.350

The percentage of conversion of glycerol and the percentage of Conversion of triacetylglycerol can be calculated by the following:

Conversion of glycerol (%)

Conversion of glycerol (%) = 100% - %Glycerol Remain

Conversion to triacetylglycerol(%)

Conversion to triacetylglycerol(%) = the summation of triacetylglycerol from GC-MS

Weight of catalyst used(%)

Amount of catalyst used is determined by amount of glycerol since acetic acid is added accessively.

5% weight of Glycerol = 9.2g x 5% = 0.48g

Appendix B : Percentage conversion to triacetylglycerol in each catalyst

Table B.1 : The experimental results by varying microwave power(w) and time(s) with no catalyst.

Molar ratio of acetic acid : glycerol	Microwave power (Watt)	Time(sec)	Conversion to triacetylglycerol (%)
9:1	100	360	0
		540	0.023
	300	90	0.025
		120	0.298

Table B.2 : The experimental results by varying microwave power(w) and time(s) with Amberlyst-15 as catalyst (5% weight).

Molar ratio of acetic acid : glycerol	Microwave power (Watt)	Time(sec)	Conversion to triacetylglycerol (%)
9:1	100	360	0.628
		540	1.256
	300	60	0.041
		90	0.138
		120	0.59

Table B.2 : The experimental results by varying microwave power(w) and time(s) with Water filter resin as catalyst (5% weight).

Molar ratio of acetic acid : glycerol	Microwave power (Watt)	Time(sec)	Conversion to triacetylglycerol (%)
9:1	100	360	0.09
		540	0.416
	300	60	0.047
		90	0.164
		120	0.22

Appendix C : The Fourier Transform Infrared (FTIR) Spectra of each experiments

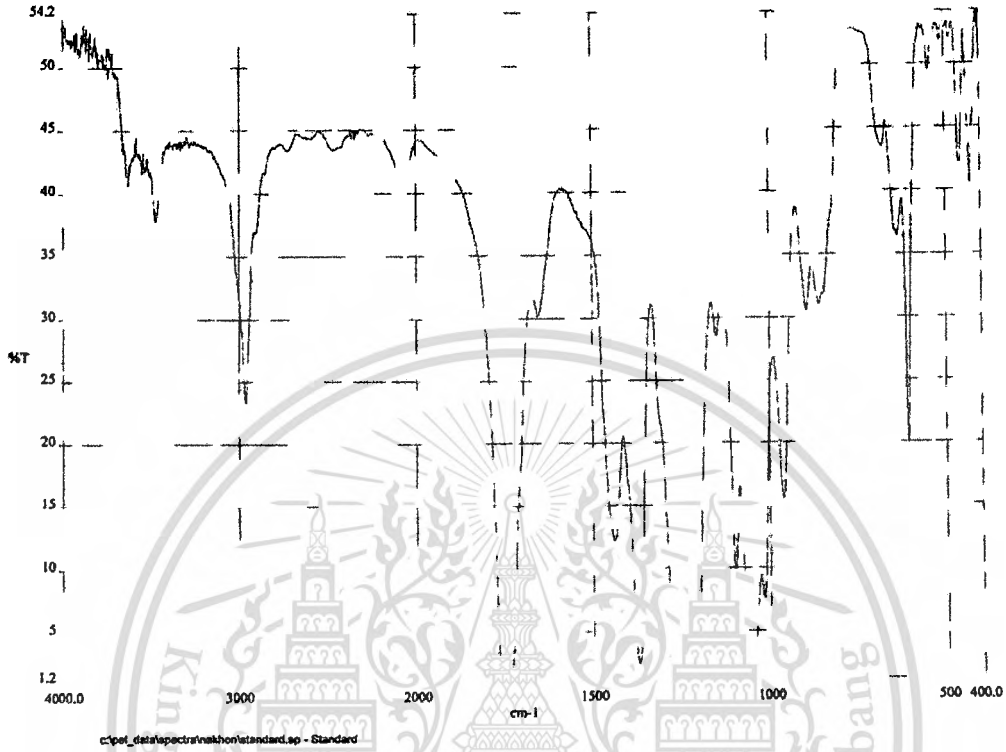


Figure C.1 : The FTIR spectrum of pure triacetylglycerol

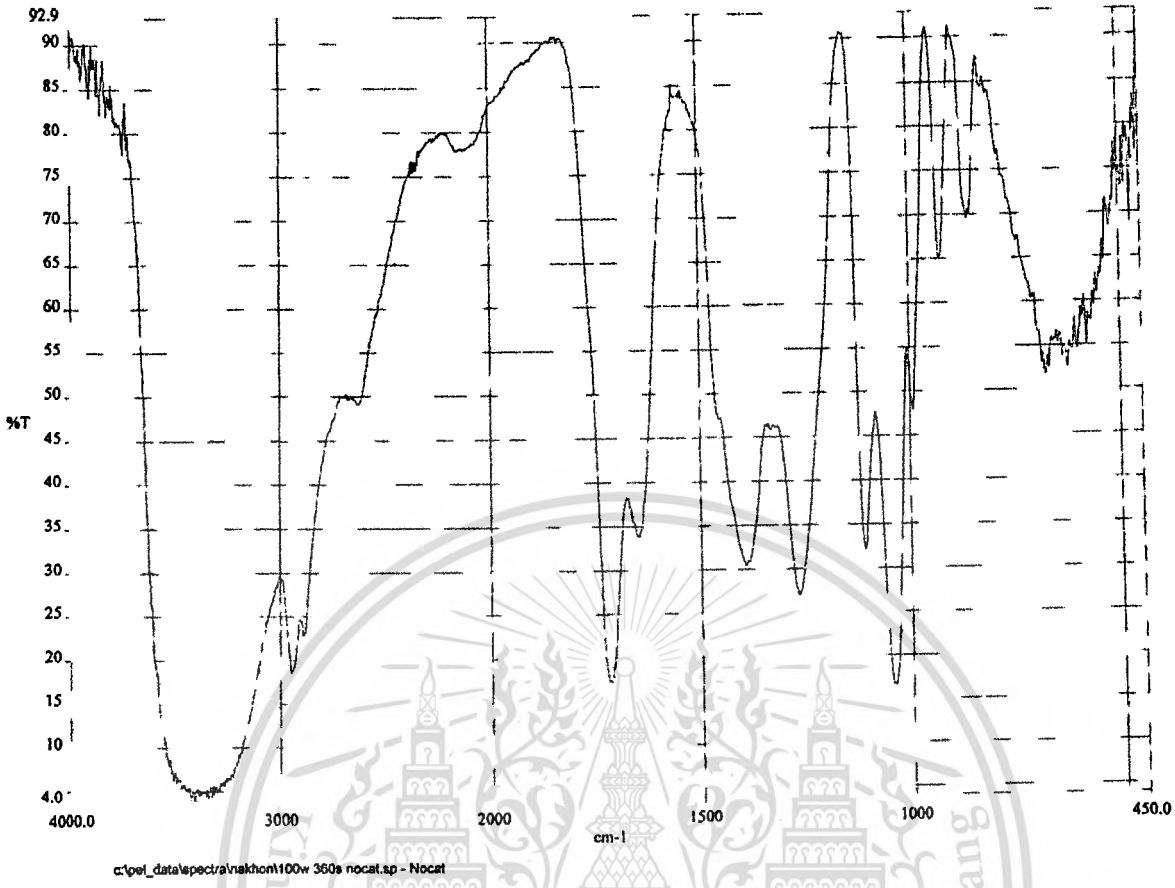


Figure C.2 : The FTIR spectrum of the esterification between glycerol and acetic acid with no catalyst at 100w and 360 second.

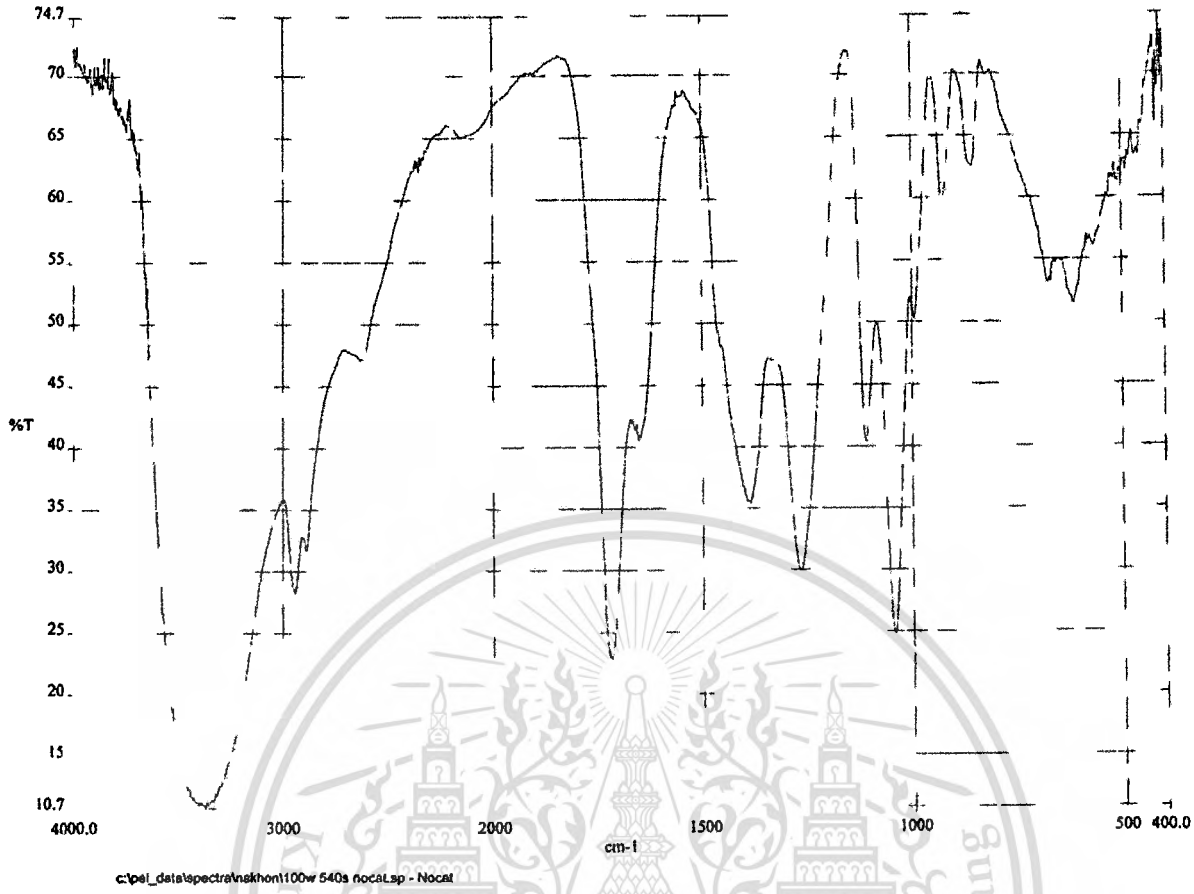


Figure C.3 : The FTIR spectrum of the esterification between glycerol and acetic acid with no catalyst at 100w and 540 second.

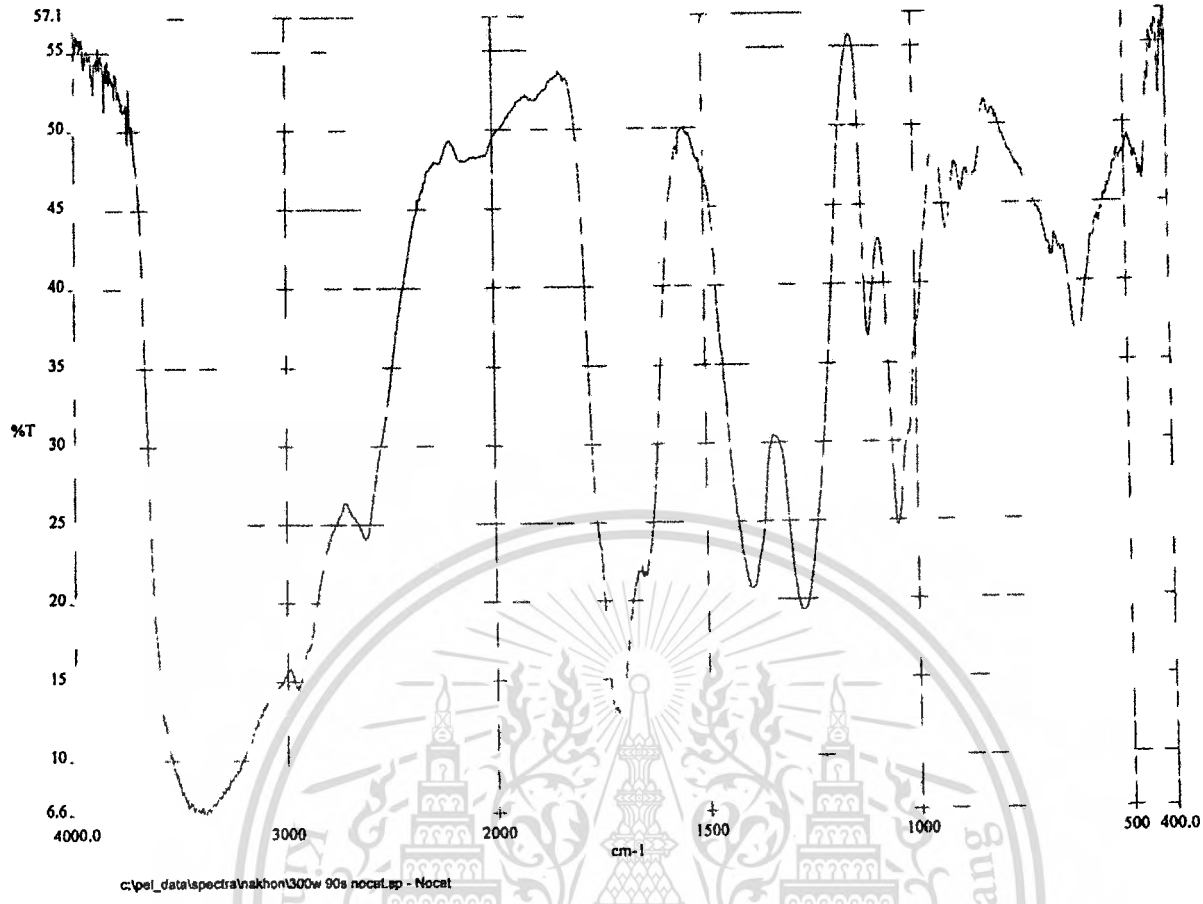


Figure C.4 : The FTIR spectrum of the esterification between glycerol and acetic acid with no catalyst at 300w and 90 second.

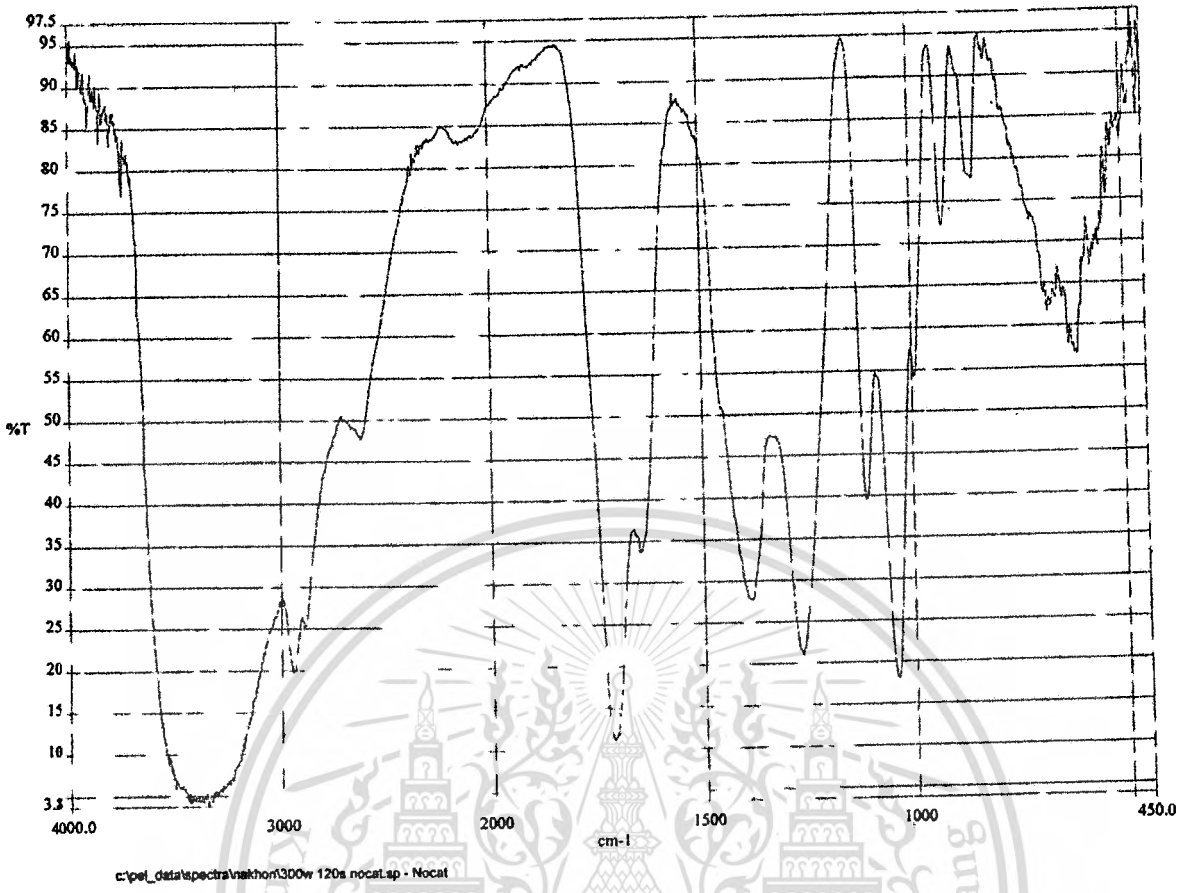


Figure C.5 : The FTIR spectrum of the esterification between glycerol and acetic acid with no catalyst at 300w and 120 second.

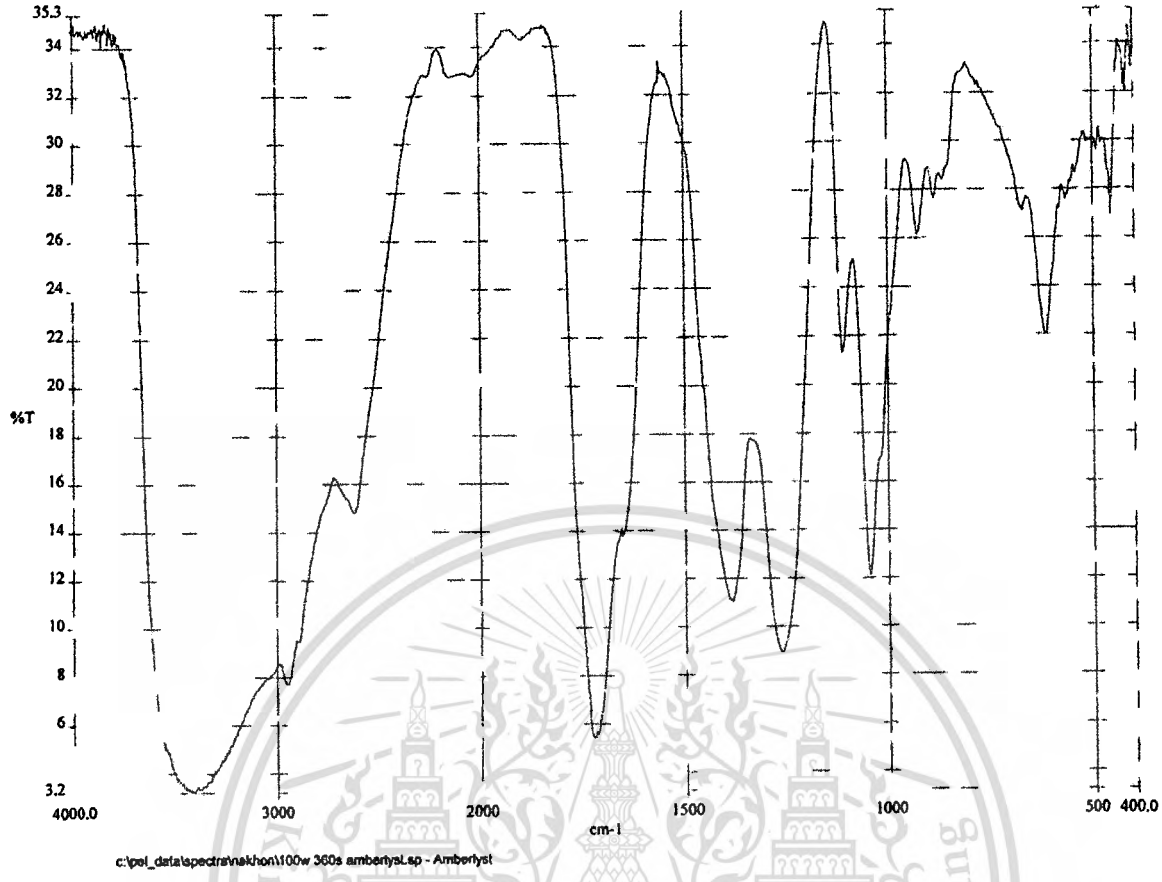


Figure C.6 : The FTIR spectrum of the esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 100w and 360 second.

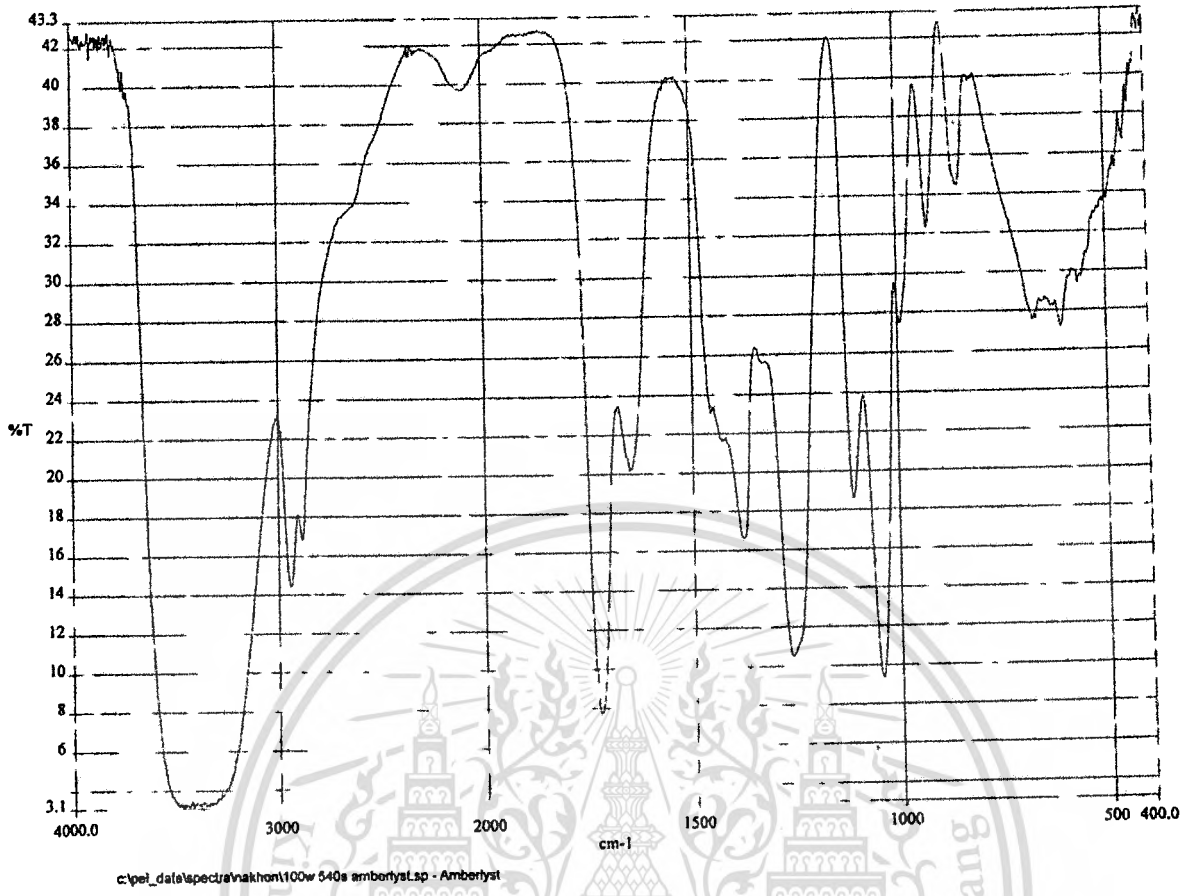


Figure C.7 : The FTIR spectrum of the esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 100w and 540 second.

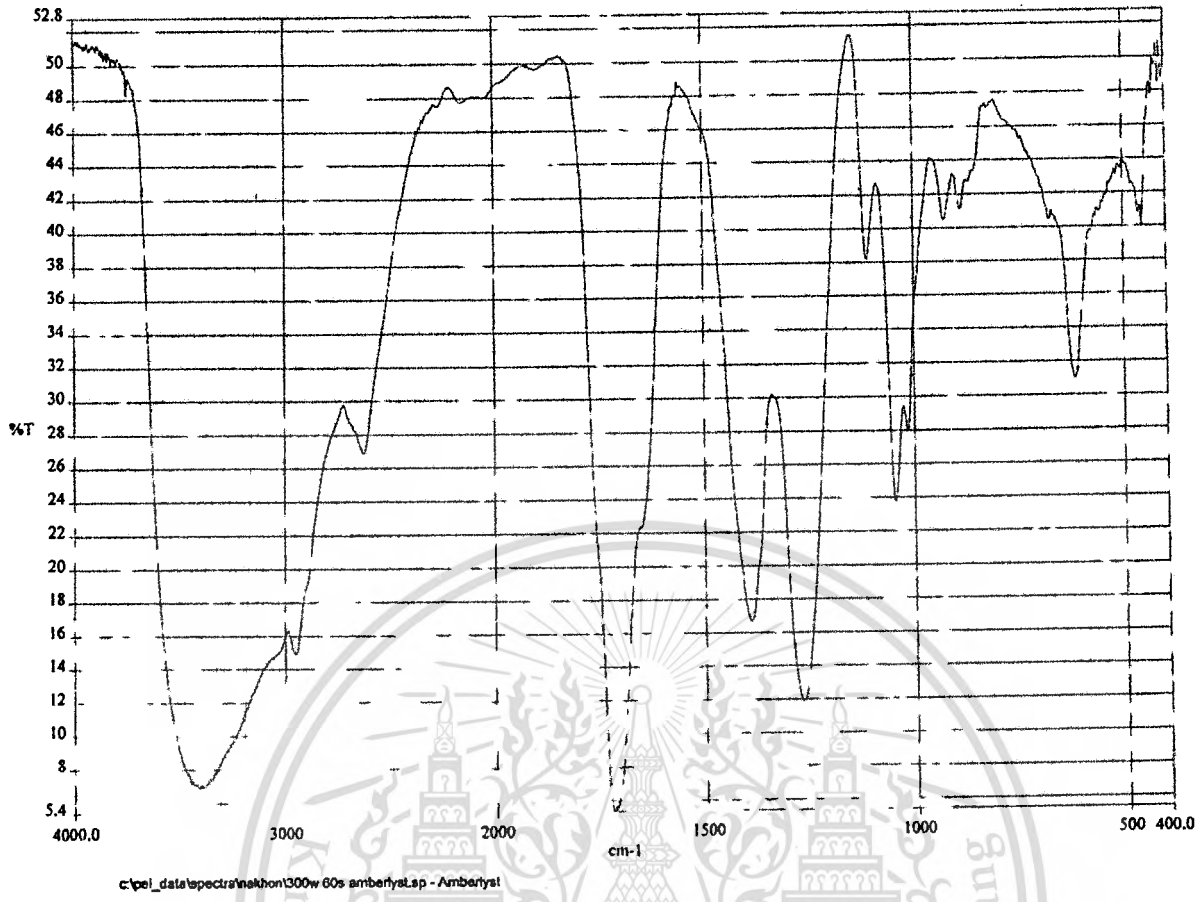


Figure C.8 : The FTIR spectrum of the esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 300w and 60 second.

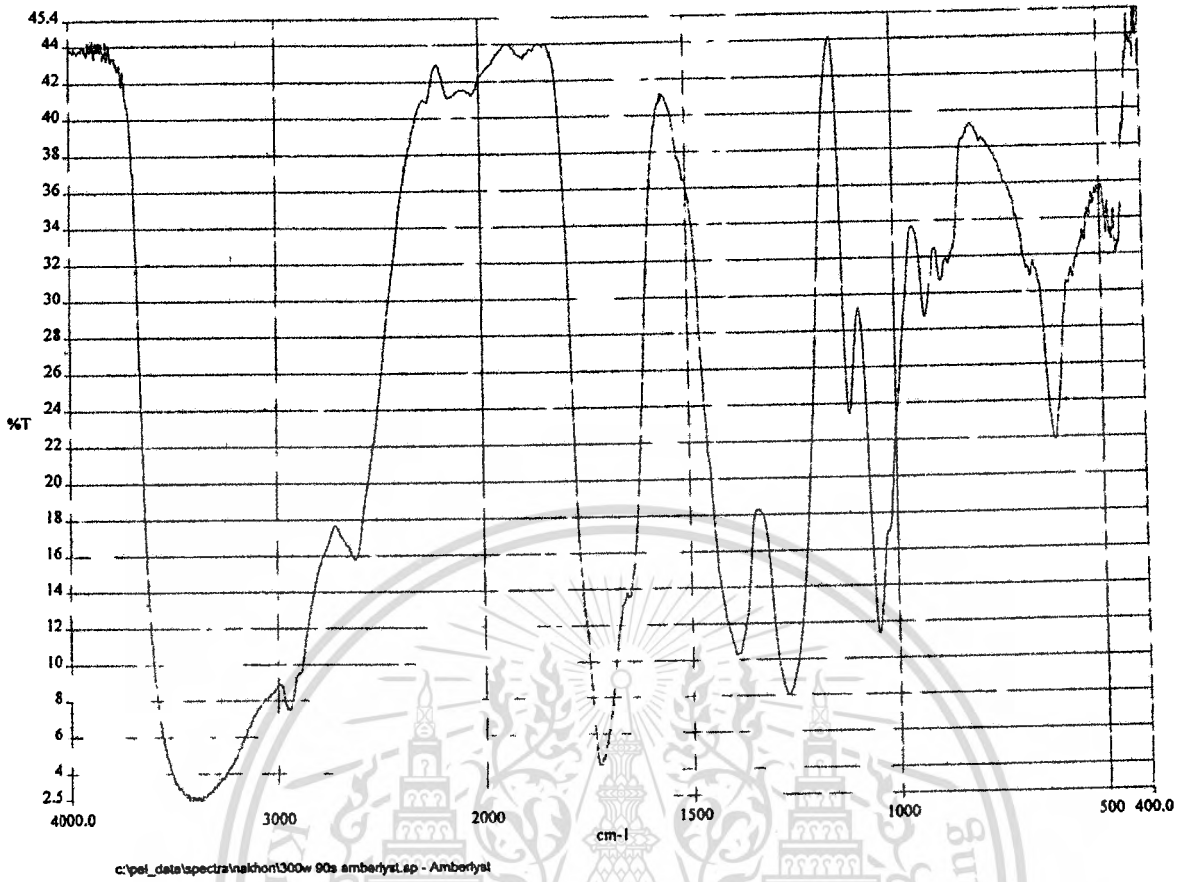


Figure C.9 : The FTIR spectrum of the esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 300w and 90 second.

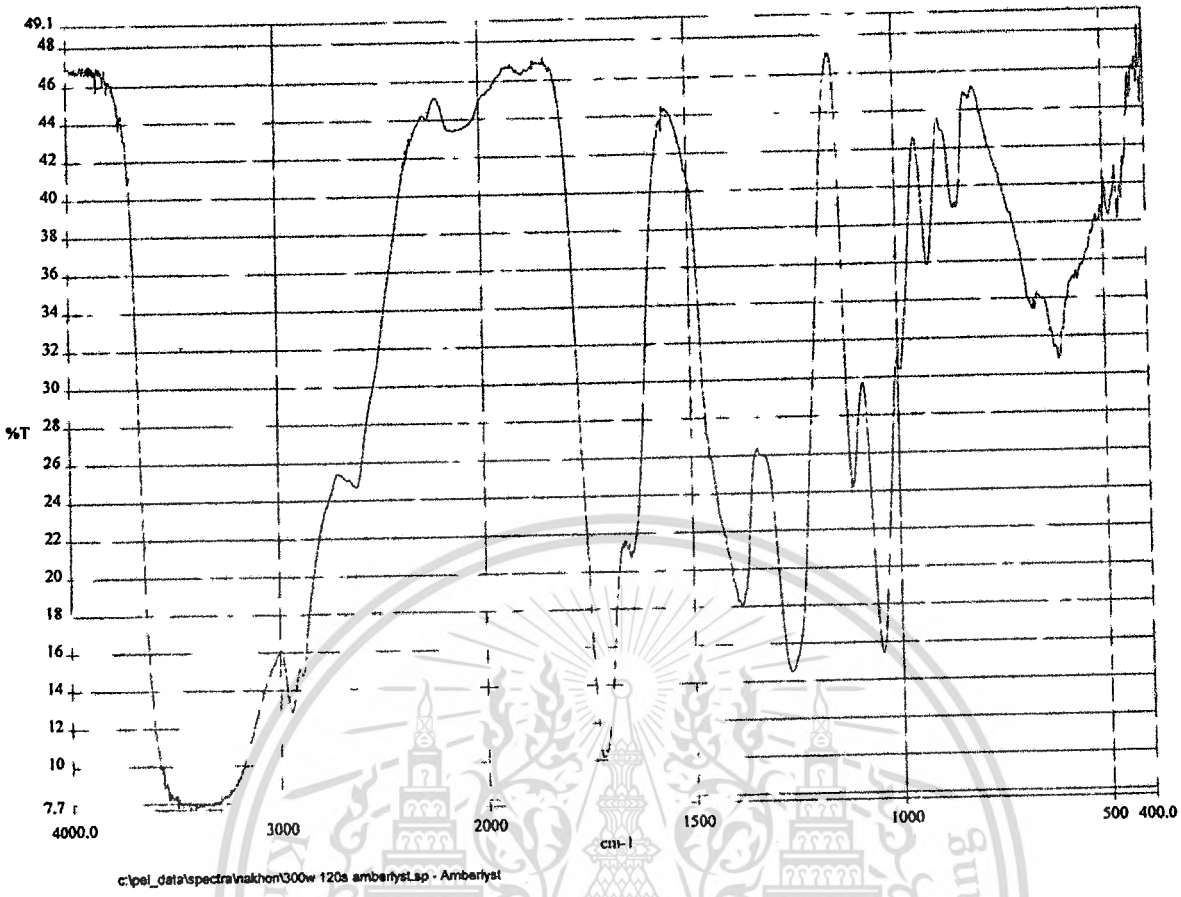


Figure C.10 : The FTIR spectrum of the esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 300w and 120 second.

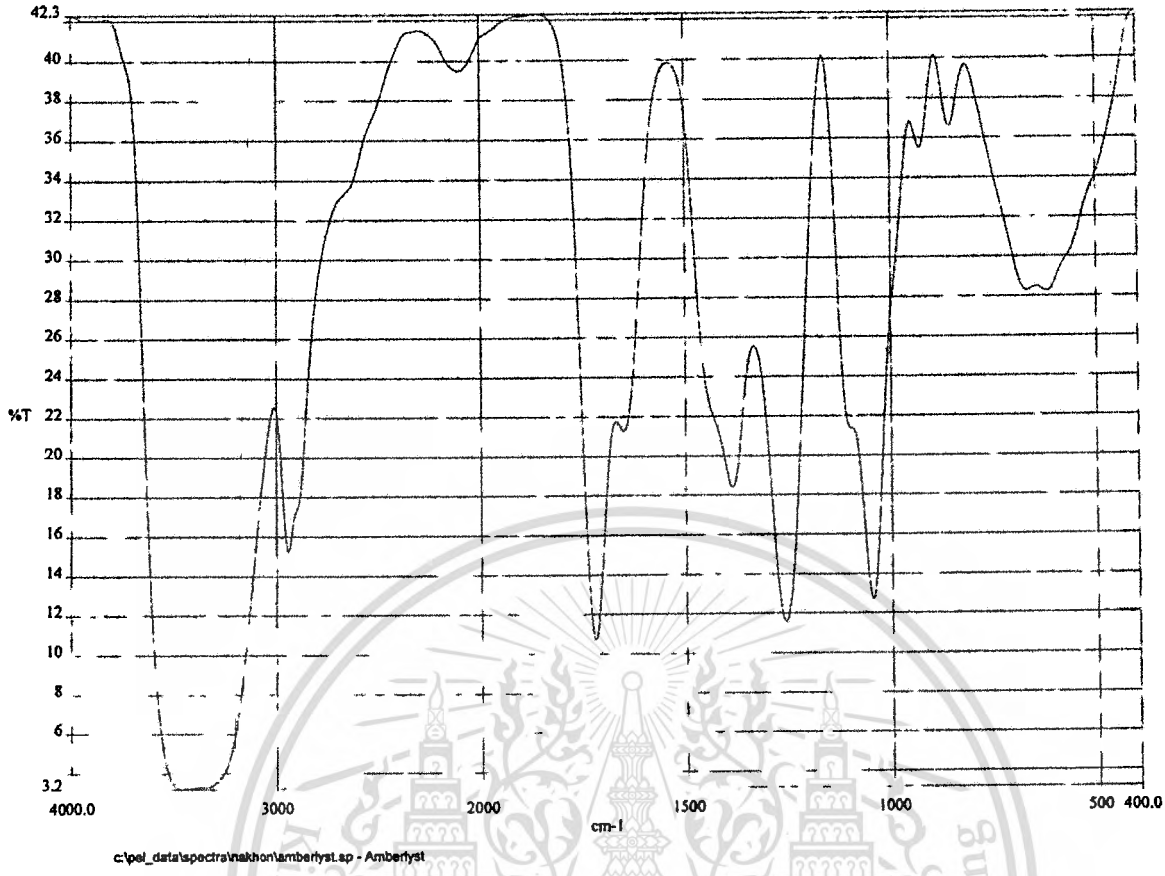


Figure C.11 : The FTIR spectrum of esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 300w for 120 second then 100w for 180 second.

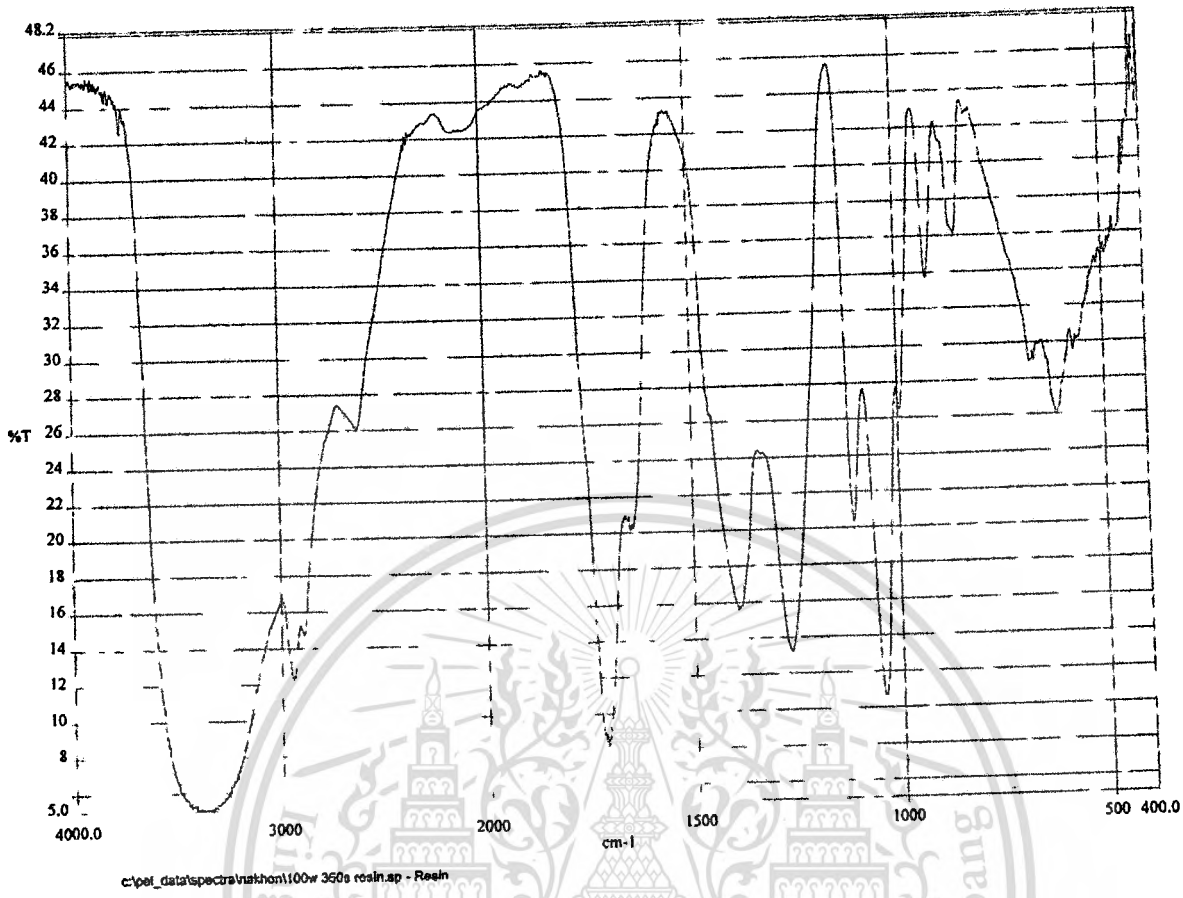


Figure C.12 : The FTIR spectrum of esterification between glycerol and acetic acid with Water filter resin as catalyst at 100w and 360 second.

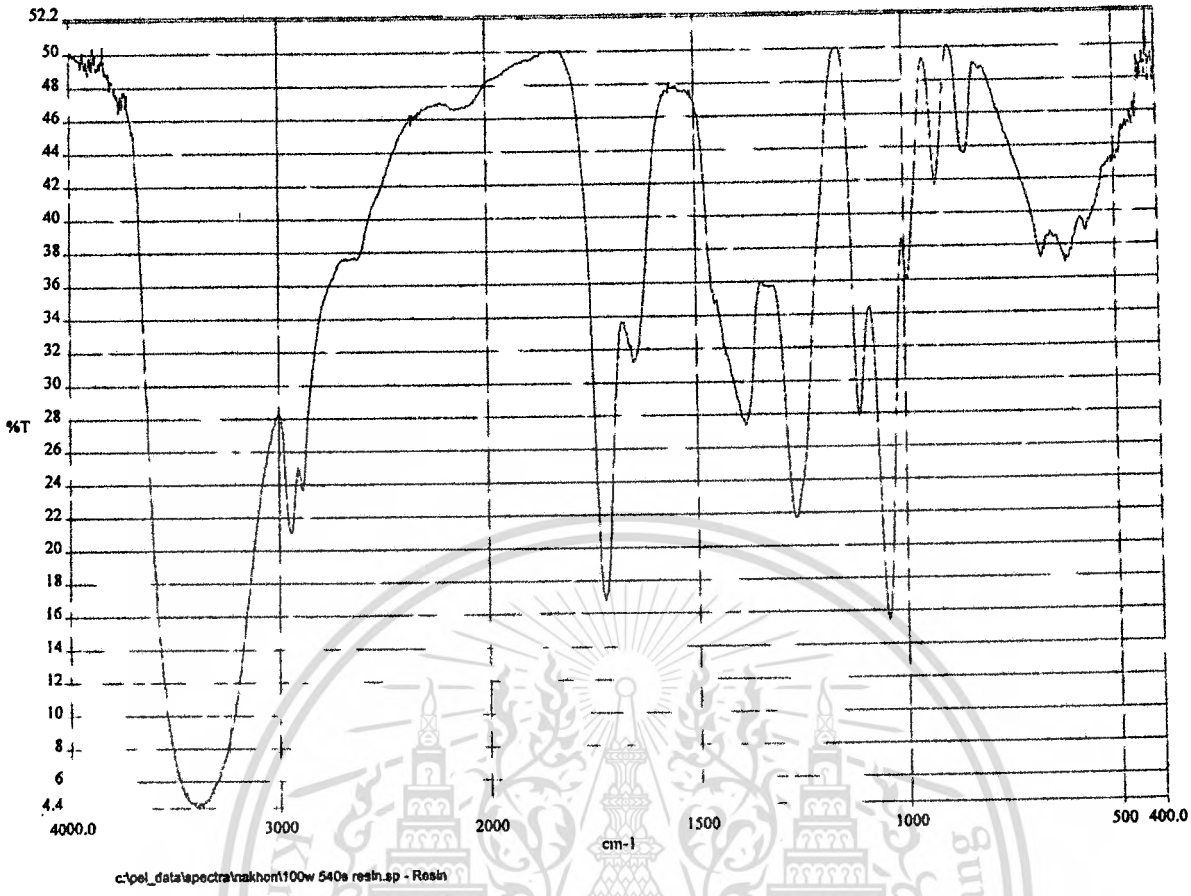


Figure C.13 : The FTIR spectrum of esterification between glycerol and acetic acid with Water filter resin as catalyst at 100w and 540 second.

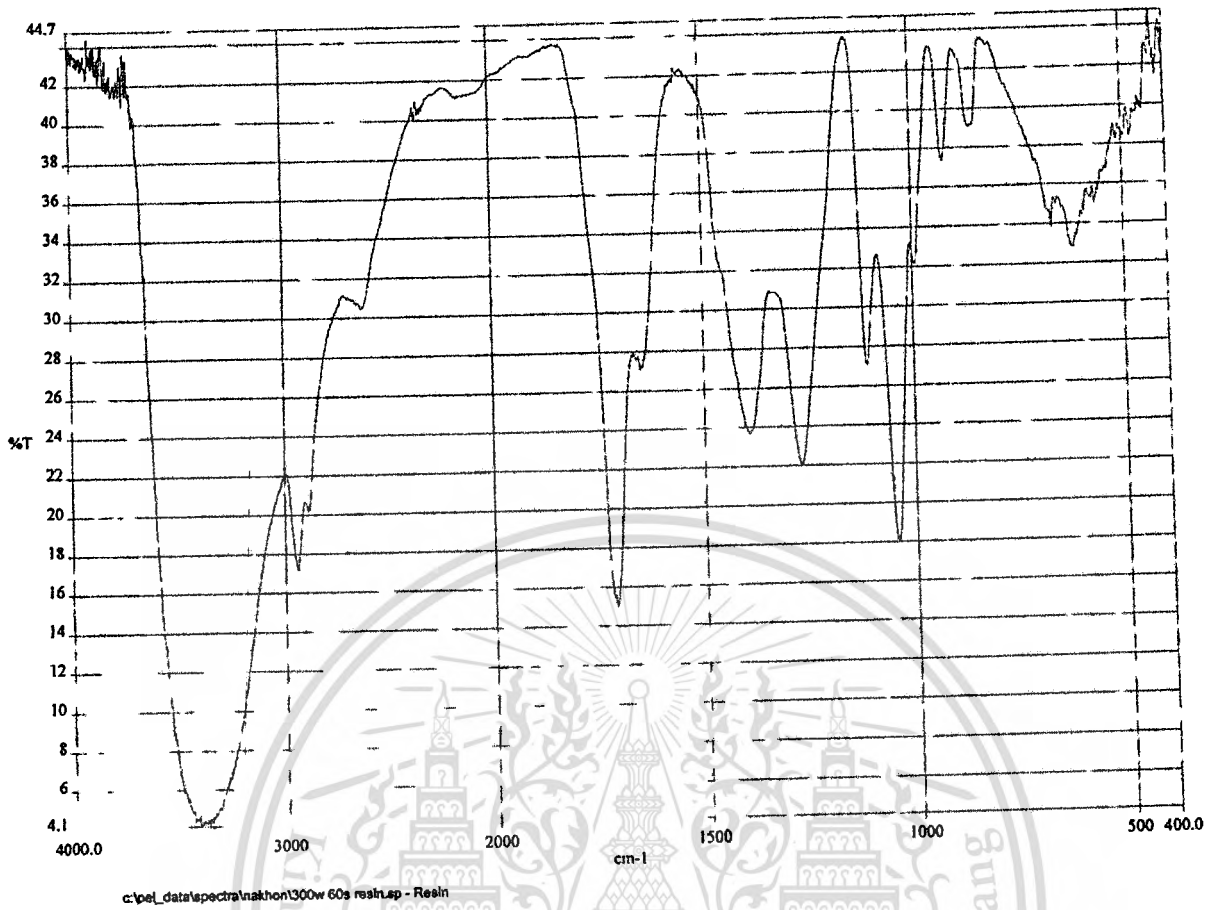


Figure C.14 : The FTIR spectrum of esterification between glycerol and acetic acid with Water filter resin as catalyst at 300w and 60 second.

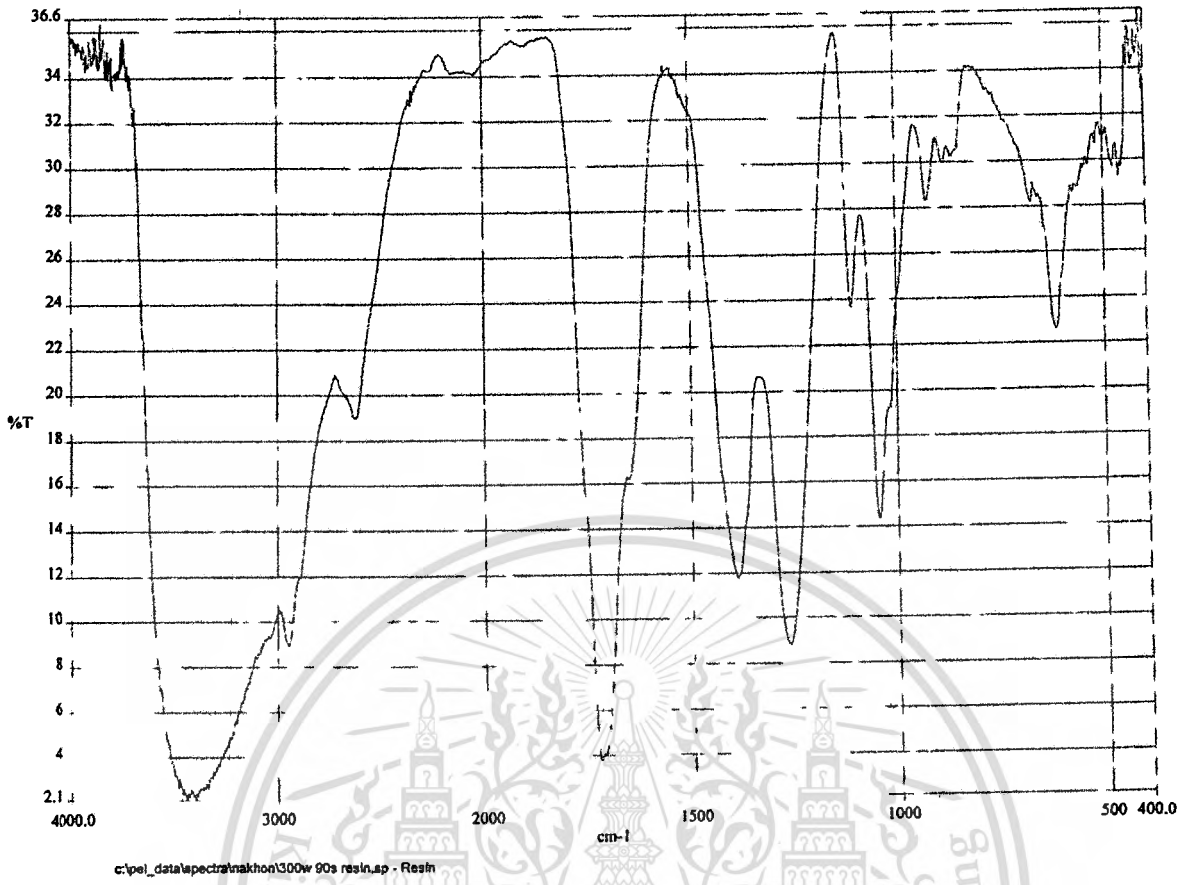


Figure C.15 : The FTIR spectrum of esterification between glycerol and acetic acid with Water filter resin as catalyst at 300w and 90 second.

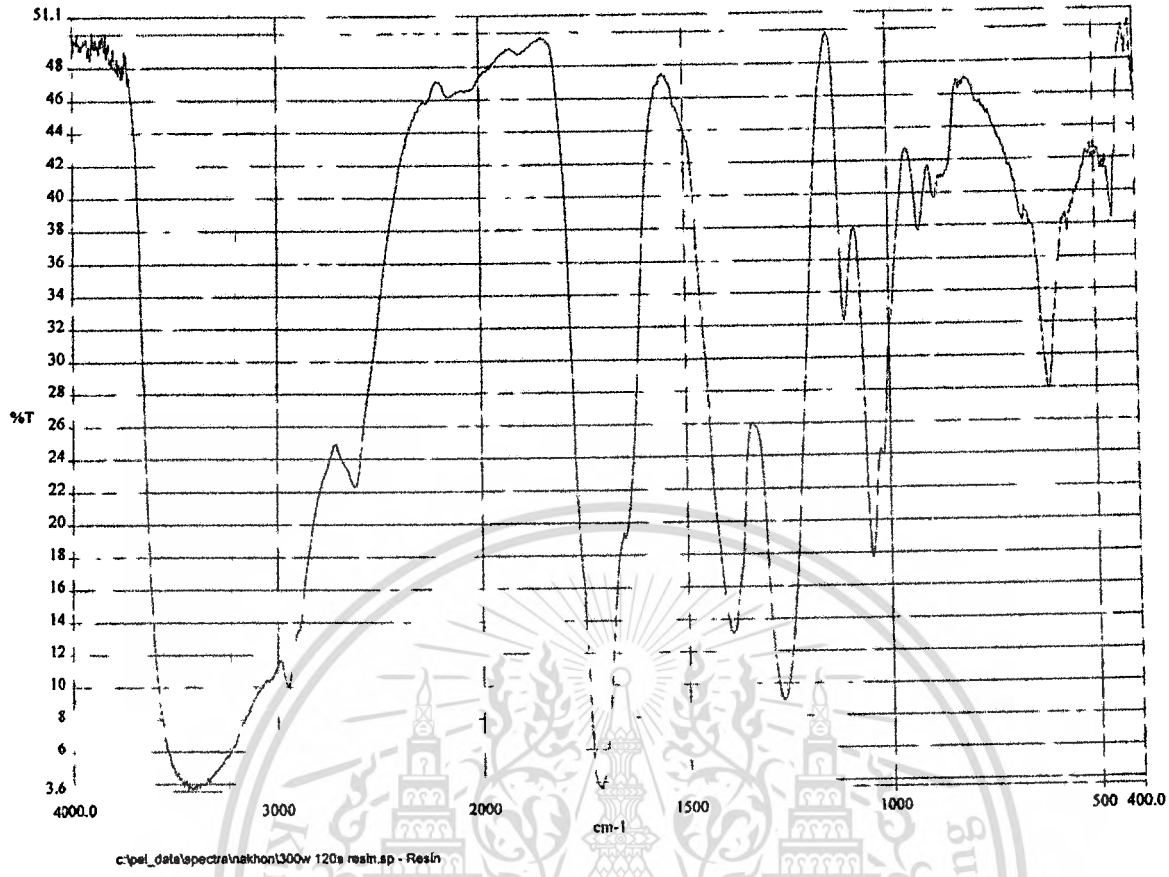


Figure C.16 : The FTIR spectrum of esterification between glycerol and acetic acid with Water filter resin as catalyst at 300w and 120 second.

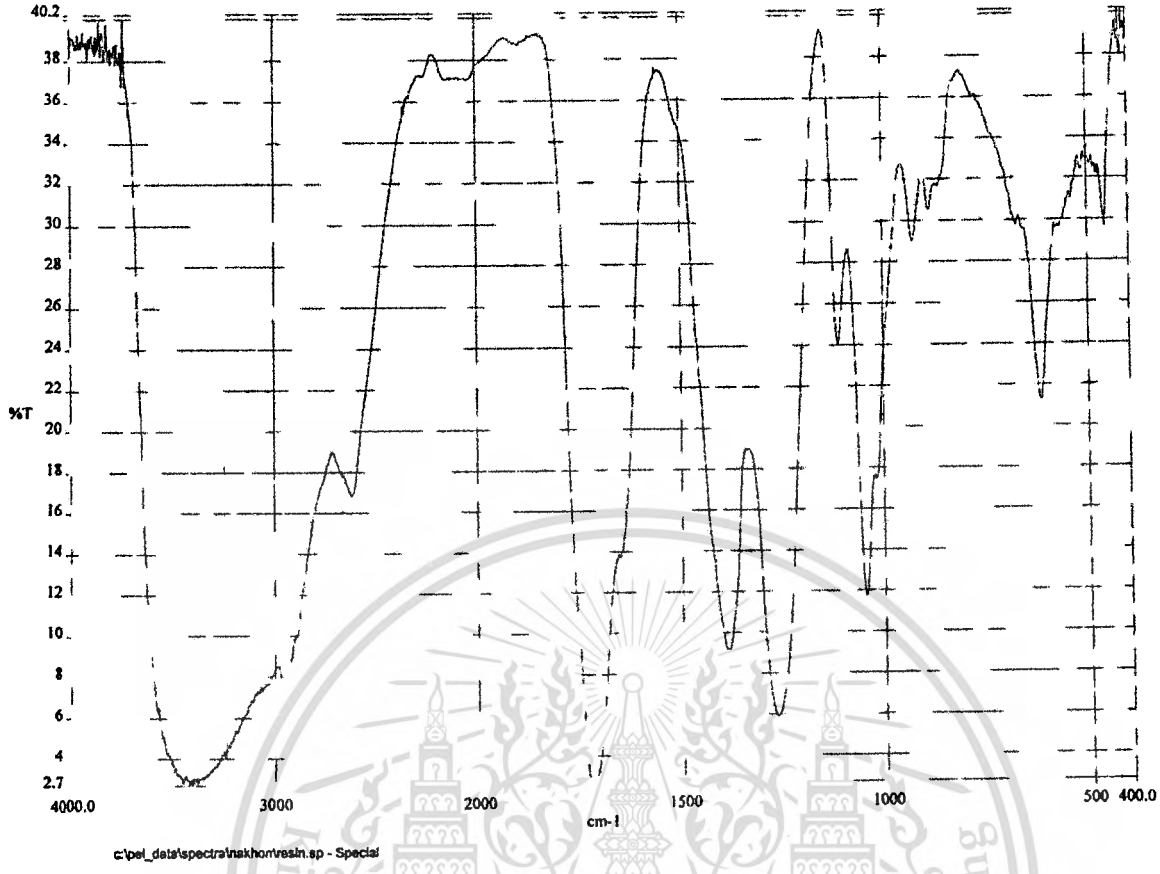
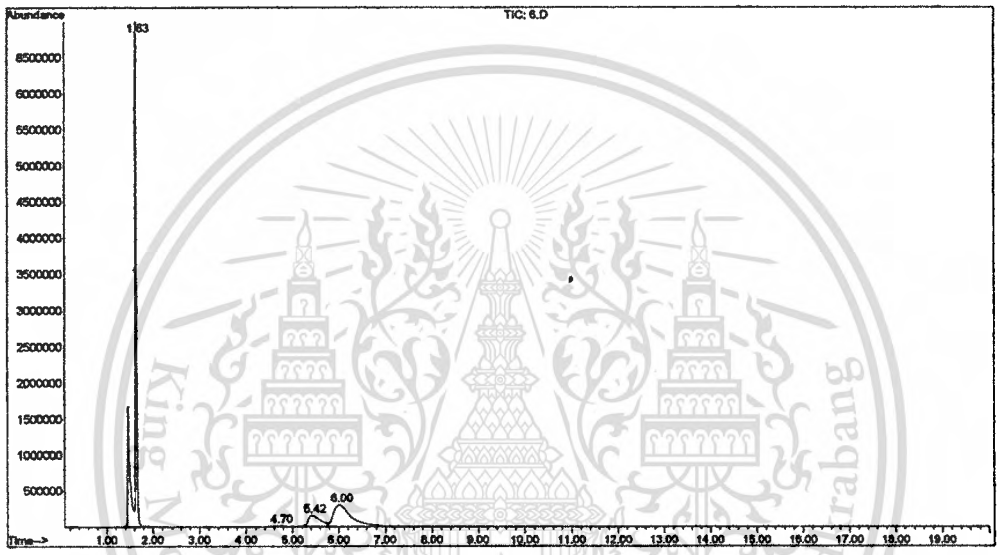


Figure C.17 : The FTIR spectrum of esterification between glycerol and acetic acid with Water filter resin as catalyst at 300w for 120 second then 100w for 180 second.

Appendix D : Gas Chromatography – Mass Spectroscopy (GC - MS) Chromatogram for each experiments

```
File       : D:\55_0023\6.D
Operator   :
Acquired   : 16 May 2012   9:57     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 6
Misc Info  :
Vial Number: 1
```



Area Percent Report

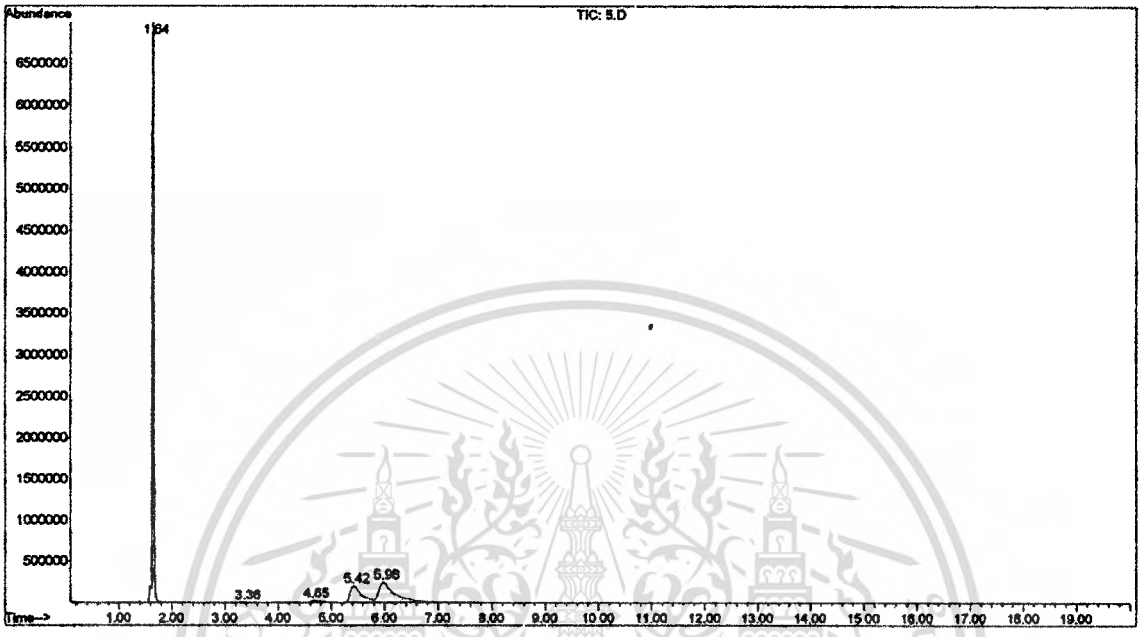
```
Data File : D:\55_0023\6.D                          Vial: 1
Acq On    : 16 May 2012   9:57                   Operator:
Sample    : sample 6                             Inst    : Instrumen
Misc      :                                      Multiplr: 1.00
                                                 Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.629	252	287	324	rM	7868277	21572763	100.00%	66.014%
2	4.705	832	857	900	rM 2	10682	149387	0.69%	0.457%
3	5.422	933	990	1052	rM 3	154684	2838682	13.16%	8.687%
4	6.000	1053	1097	1312	rM 2	303343	8118257	37.63%	24.842%

Sum of corrected areas: 32679089

Figure D.1 : GC-MS Chromatogram of esterification between glycerol and acetic acid with no catalyst at 100w and 360 second.

```
File       : D:\55_0023\5.D
Operator   :
Acquired   : 16 May 2012   9:20     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 5
Misc Info  :
Vial Number: 1
```



Area Percent Report

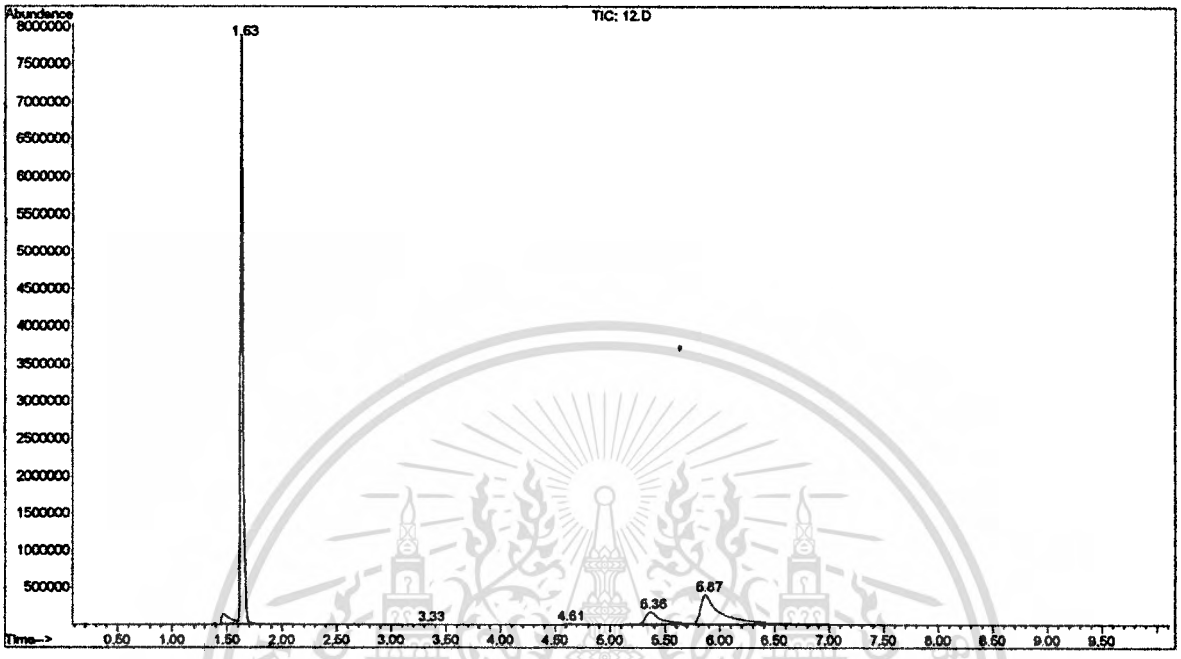
```
Data File : D:\55_0023\5.D                          Vial: 1
Acq On    : 16 May 2012   9:20                   Operator:
Sample    : sample 5                             Inst    : Instrumen
Misc      :                                      Multiplr: 1.00
                                                 Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.640	275	289	325	rM	7435620	15111118	100.00%	64.607%
2	3.361	603	608	614	rM 3	2797	5308	0.04%	0.023%
3	4.651	834	847	916	rM 2	24694	380492	2.52%	1.627%
4	5.417	965	989	1059	rM 3	193918	2850318	18.86%	12.186%
5	5.978	1060	1093	1283	rM 2	237432	5042206	33.37%	21.558%

Sum of corrected areas: 23389442

Figure D.2 : GC-MS Chromatogram of esterification between glycerol and acetic acid with no catalyst at 100w and 540 second.

```
File       : D:\55_0023\12.D
Operator   :
Acquired   : 16 May 2012  13:01     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 12
Misc Info  :
Vial Number: 1
```



Area Percent Report

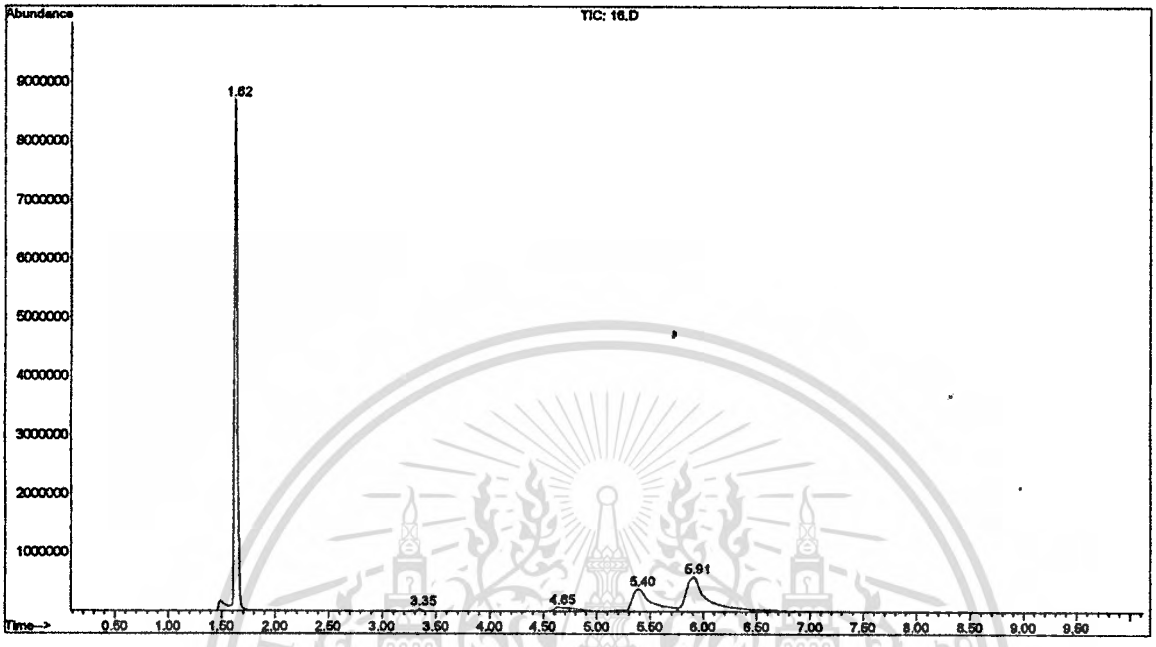
```
Data File : D:\55_0023\12.D                          Vial: 1
Acq On    : 16 May 2012  13:01                    Operator:
Sample    : sample 12                             Inst    : Instrumen
Misc      :                                       Multiplr: 1.00
                                          Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.629	252	287	307	rM	7861231	13693182	100.00%	66.013%
2	3.334	598	603	609	rM 3	2576	5160	0.04%	0.025%
3	4.613	832	840	894	rM 2	12985	128243	0.94%	0.618%
4	5.363	961	979	1044	rM 2	165711	1639598	11.97%	7.904%
5	5.870	1046	1073	1246	rM 3	394105	5276934	38.54%	25.439%

Sum of corrected areas: 20743117

Figure D.3 : GC-MS Chromatogram of esterification between glycerol and acetic acid with no catalyst at 300w and 90 second.

```
File       : D:\55_0023\16.D
Operator   :
Acquired   : 16 May 2012  15:24     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 16
Misc Info  :
Vial Number: 1
```



Area Percent Report

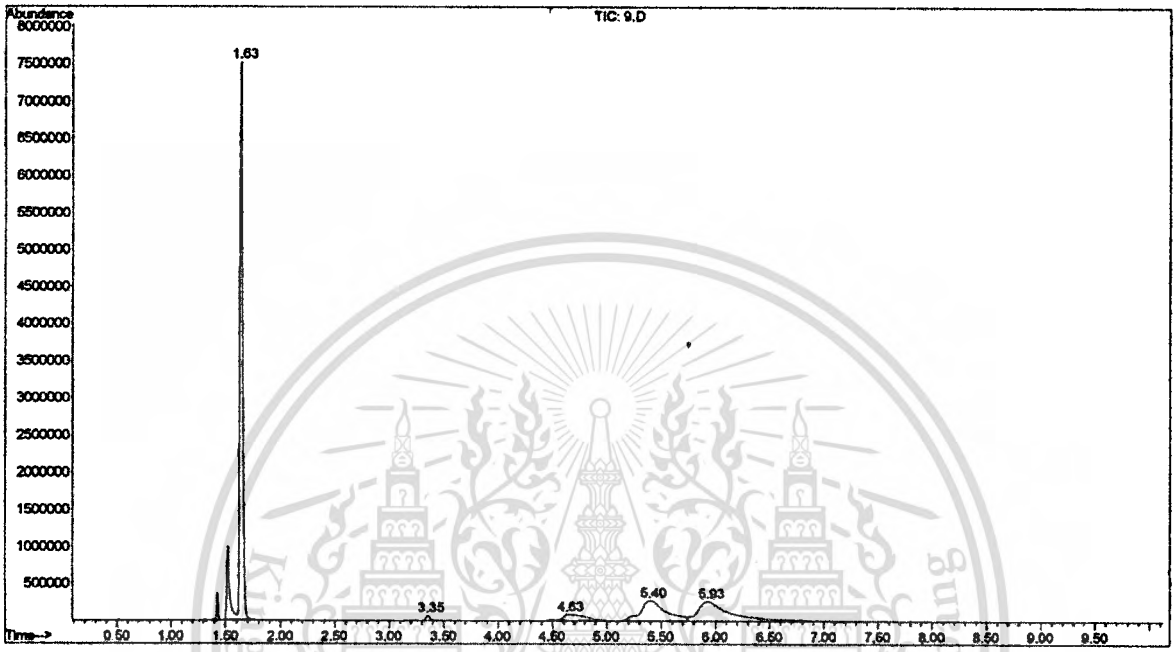
```
Data File : D:\55_0023\16.D                          Vial: 1
Acq On    : 16 May 2012  15:24                    Operator:
Sample    : sample 16                              Inst    : Instrumen
Misc      :                                        Multiplr: 1.00
                                                Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.624	256	286	338	rM	8700406	18019792	100.00%	55.069%
2	3.351	598	606	616	rM	41840	97624	0.54%	0.298%
3	4.645	828	846	913	rM 2	70805	965376	5.36%	2.950%
4	5.395	931	985	1052	rM 3	381933	4896318	27.17%	14.963%
5	5.913	1053	1081	1343	rM	597358	8743109	48.52%	26.719%

Sum of corrected areas: 32722219

Figure D.4 : GC-MS Chromatogram of esterification between glycerol and acetic acid with no catalyst at 300w and 120 second.

```
File       : D:\55_0023\9.D
Operator   :
Acquired   : 16 May 2012  11:28     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 9
Misc Info  :
Vial Number: 1
```



Area Percent Report

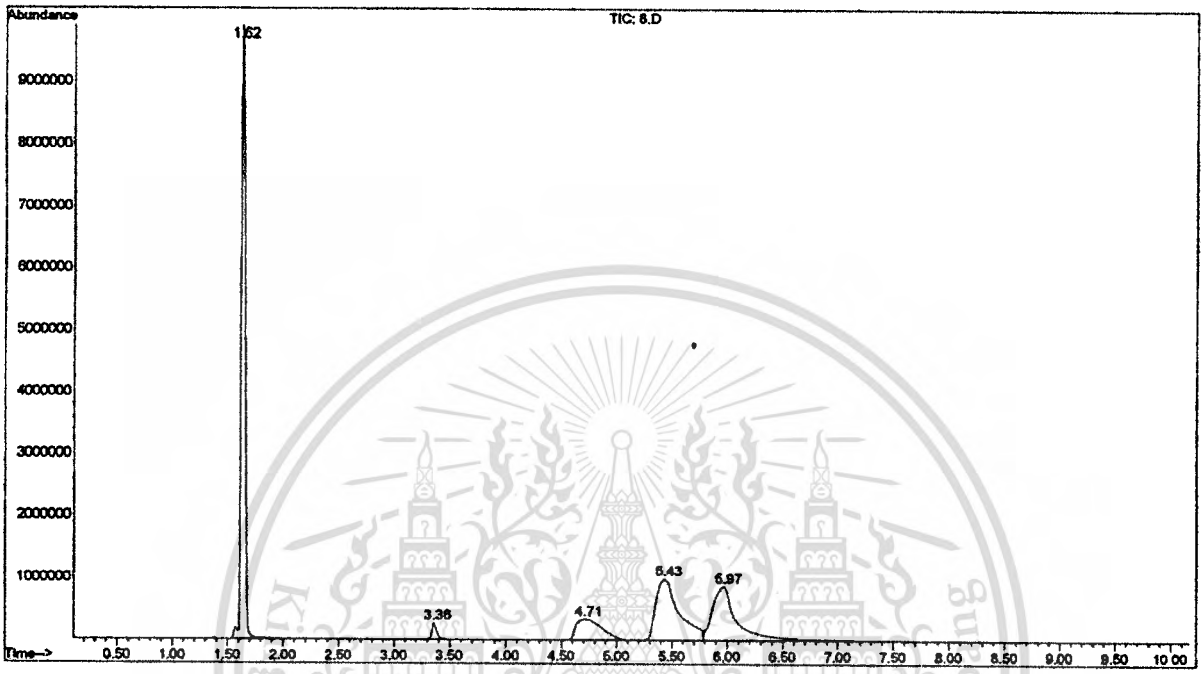
```
Data File : D:\55_0023\9.D                          Vial: 1
Acq On    : 16 May 2012  11:28                   Operator:
Sample    : sample 9                             Inst    : Instrumen
Misc      :                                      Multiplr: 1.00
                                                 Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.635	263	288	304	rM	7512930	15343320	100.00%	57.578%
2	3.351	599	606	623	rM	67613	167230	1.09%	0.628%
3	4.635	813	844	910	rM 2	80514	1248398	8.14%	4.685%
4	5.396	939	985	1048	rM 3	276327	4381775	28.56%	16.443%
5	5.930	1050	1084	1245	rM 3	264347	5507295	35.89%	20.667%

Sum of corrected areas: 26648018

Figure D.5 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 100w and 360 second.

```
File       : D:\55_0023\8.D
Operator   :
Acquired   : 16 May 2012  11:02     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 8
Misc Info  :
Vial Number: 1
```



Area Percent Report

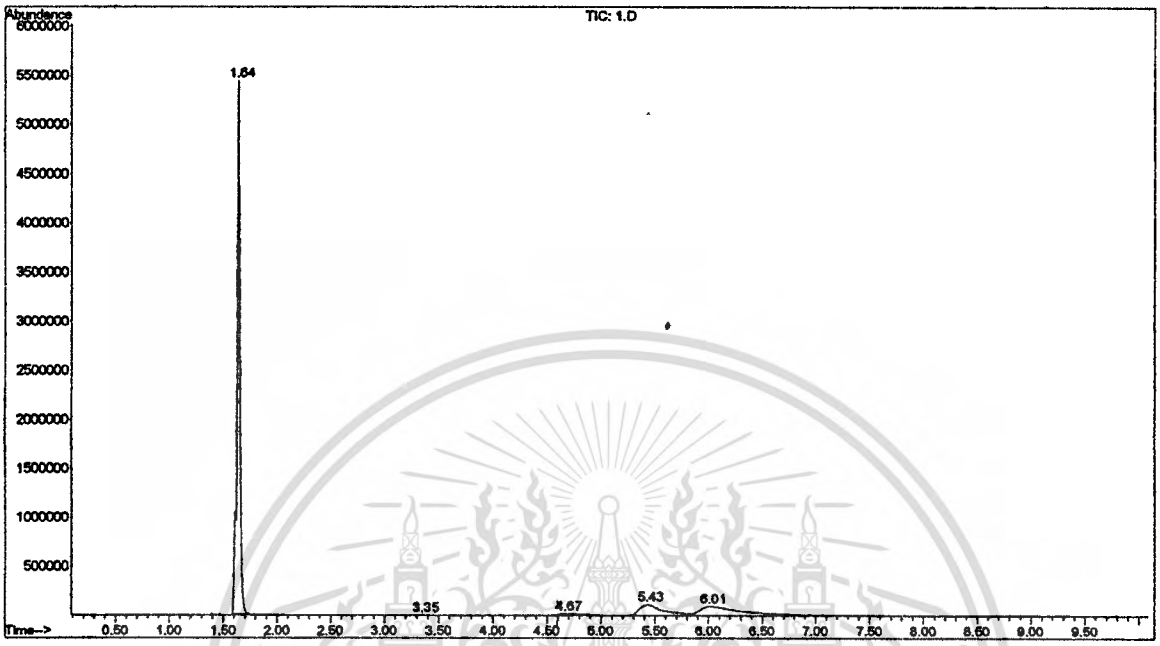
```
Data File : D:\55_0023\8.D                              Vial: 1
Acq On    : 16 May 2012  11:02                      Operator:
Sample    : sample 8                                Inst    : Instrumen
Misc      :                                         Multiplr: 1.00
                                                Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.619	272	285	341	rM	9916317	25036403	100.00%	42.678%
2	3.361	596	608	630	rM	259730	736979	2.94%	1.256%
3	4.710	830	858	928	rM 2	330674	5263731	21.02%	8.973%
4	5.433	952	992	1056	rM 2	987371	14293252	57.09%	24.365%
5	5.973	1058	1092	1300	rM 2	871301	13332447	53.25%	22.727%

Sum of corrected areas: 58662812

Figure D.6 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 100w and 540 second.

```
File       : D:\55_0023\1.D
Operator   :
Acquired   : 15 May 2012  14:47     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 1
Misc Info  :
Vial Number: 1
```



Area Percent Report

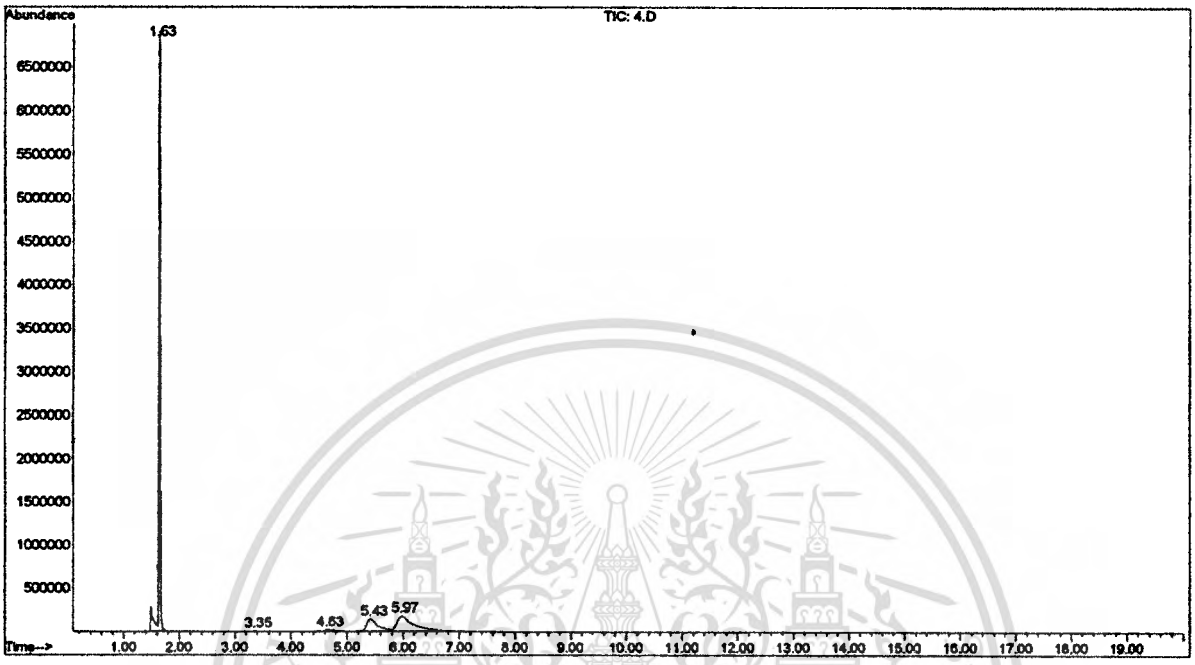
```
Data File : D:\55_0023\1.D                          Vial: 1
Acq On    : 15 May 2012  14:47                   Operator:
Sample    : sample 1                             Inst    : Instrumen
Misc      :                                      Multiplr: 1.00
                                                 Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.640	279	289	306	rM	5435058	10288418	100.00%	69.490%
2	3.351	602	606	615	rM 3	2406	6130	0.06%	0.041%
3	4.667	829	850	911	rM 2	18396	260096	2.53%	1.757%
4	5.428	961	991	1062	rM 2	112305	1658910	16.12%	11.205%
5	6.011	1062	1099	1285	rM 4	94114	2592007	25.19%	17.507%

Sum of corrected areas: 14805561

Figure D.7 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 300w and 60 second.

```
File       : D:\55_0023\4.D
Operator   :
Acquired   : 15 May 2012  16:30     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 4
Misc Info  :
Vial Number: 1
```



Area Percent Report

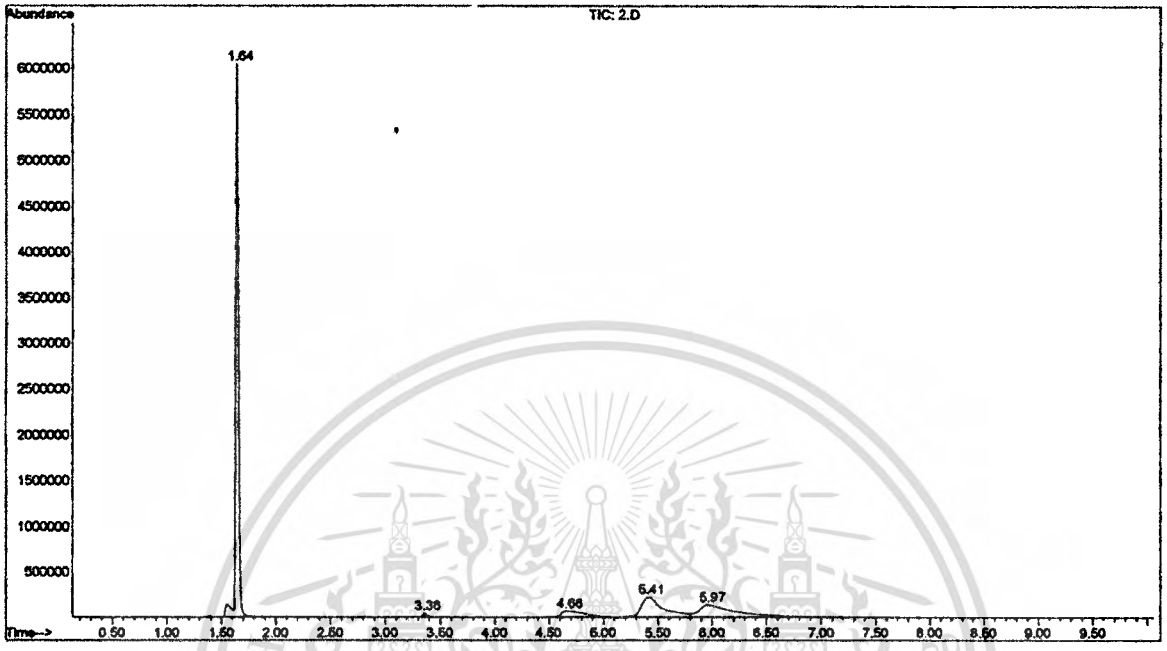
```
Data File : D:\55_0023\4.D                          Vial: 1
Acq On    : 15 May 2012  16:30                   Operator:
Sample    : sample 4                             Inst    : Instrumen
Misc      :                                      Multiplr: 1.00
                                                 Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.635	258	288	313	rM	6903779	12726338	100.00%	67.905%
2	3.351	600	606	616	rM 2	12227	25810	0.20%	0.138%
3	4.635	835	844	897	rM	22752	280395	2.20%	1.496%
4	5.428	962	991	1053	rM 4	141130	1953718	15.35%	10.425%
5	5.973	1054	1092	1262	rM 4	176204	3755089	29.51%	20.036%

Sum of corrected areas: 18741350

Figure D.8 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 300w and 90 second.

```
File       : D:\55_0023\2.D
Operator   :
Acquired   : 15 May 2012  15:19     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 2
Misc Info  :
Vial Number: 1
```



Area Percent Report

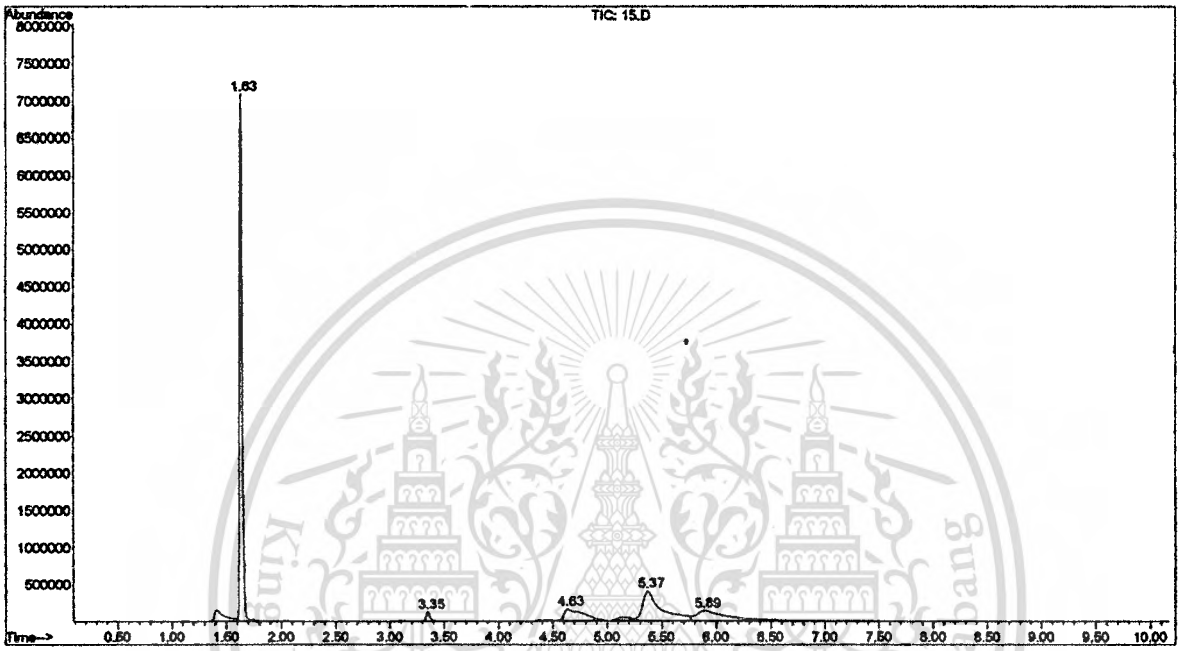
```
Data File : D:\55_0023\2.D                          Vial: 1
Acq On    : 15 May 2012  15:19                   Operator:
Sample    : sample 2                             Inst    : Instrumen
Misc      :                                      Multiplr: 1.00
                                                 Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.640	268	289	314	rM	6045509	11353950	100.00%	60.936%
2	3.356	599	607	621	rM	41940	109924	0.97%	0.590%
3	4.662	833	849	916	rM 2	63575	909778	8.01%	4.883%
4	5.406	947	987	1059	rM 3	221445	3098631	27.29%	16.630%
5	5.967	1060	1091	1251	rM 3	135606	3160249	27.83%	16.961%

Sum of corrected areas: 18632532

Figure D.9 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 300w and 120 second.

```
File       : D:\55_0023\15.D
Operator   :
Acquired   : 16 May 2012  14:39     using AcqMethod ACETIC
Instrument :  Instrumen
Sample Name: sample 15
Misc Info  :
Vial Number: 1
```



Area Percent Report

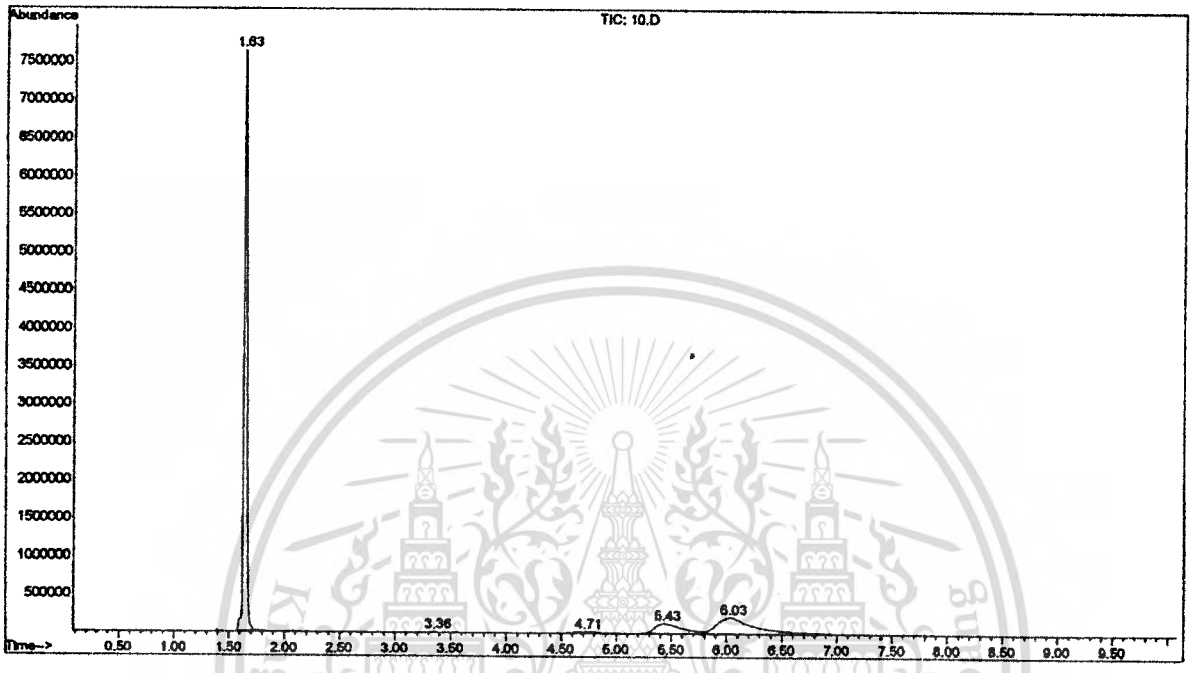
```
Data File : D:\55_0023\15.D                          Vial: 1
Acq On    : 16 May 2012  14:39                    Operator:
Sample    : sample 15                             Inst    : Instrumen
Misc      :                                       Multiplr: 1.00
                                                  Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.629	239	287	315	rM	7093043	12493193	100.00%	55.412%
2	3.350	598	606	615	rM	126482	303472	2.43%	1.346%
3	4.635	829	844	906	rM	158465	1899472	15.20%	8.425%
4	5.368	920	980	1052	rM	405339	5104697	40.86%	22.641%
5	5.886	1054	1076	1214	rM 3	135066	2745270	21.97%	12.176%

Sum of corrected areas: 22546104

Figure D.10 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Amberlyst-15 as catalyst at 300w for 120 second then 100w for 180 second.

```
File       : D:\55_0023\10.D
Operator   :
Acquired   : 16 May 2012  11:56     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 10
Misc Info  :
Vial Number: 1
```



Area Percent Report

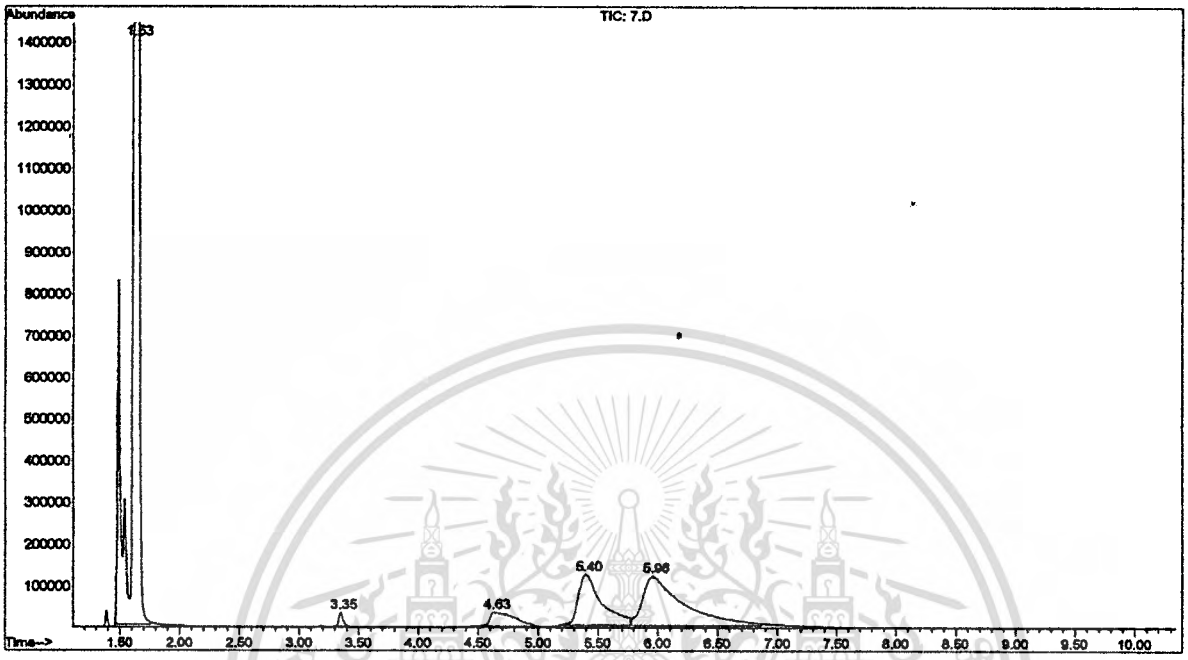
```
Data File : D:\55_0023\10.D                          Vial: 1
Acq On    : 16 May 2012  11:56                    Operator:
Sample    : sample 10                             Inst    : Instrumen
Misc      :                                       Multiplr: 1.00
                                                  Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.635	277	288	315	rM	7640874	14563118	100.00%	65.481%
2	3.356	600	607	615	rM 2	8647	19959	0.14%	0.090%
3	4.710	824	858	921	rM 2	18740	306034	2.10%	1.376%
4	5.433	957	992	1057	rM 3	127813	2169351	14.90%	9.754%
5	6.032	1058	1103	1284	rM 2	211287	5181842	35.58%	23.299%

Sum of corrected areas: 22240304

Figure D.11 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Water filter resin as catalyst at 100w and 360 second.

```
File       : D:\55_0023\7.D
Operator   :
Acquired   : 16 May 2012  10:33     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 7
Misc Info  :
Vial Number: 1
```



Area Percent Report

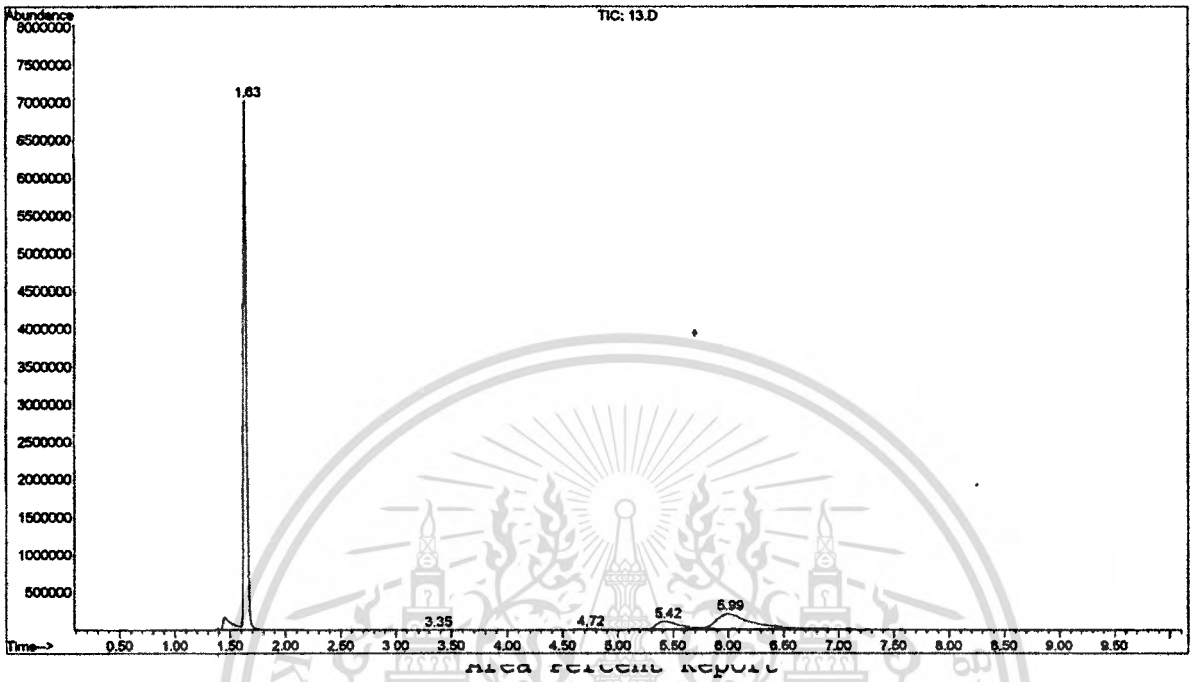
```
Data File : D:\55_0023\7.D                          Vial: 1
Acq On    : 16 May 2012  10:33                   Operator:
Sample    : sample 7                             Inst    : Instrumen
Misc      :                                      Multiplr: 1.00
                                                 Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.635	256	288	311	rM	6371391	14343758	100.00%	74.126%
2	3.345	597	605	616	rM	33874	80442	0.56%	0.416%
3	4.629	827	843	905	rM 2	33714	460975	3.21%	2.382%
4	5.395	947	985	1055	rM 3	122547	1684636	11.74%	8.706%
5	5.957	1056	1089	1257	rM 2	118530	2780763	19.39%	14.370%

```
Sum of corrected areas:    19350574
```

Figure D.12 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Water filter resin as catalyst at 100w and 540 second.

```
File       : D:\55_0023\13.D
Operator   :
Acquired   : 16 May 2012  13:46     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 13
Misc Info  :
Vial Number: 1
```



Area Percent Report

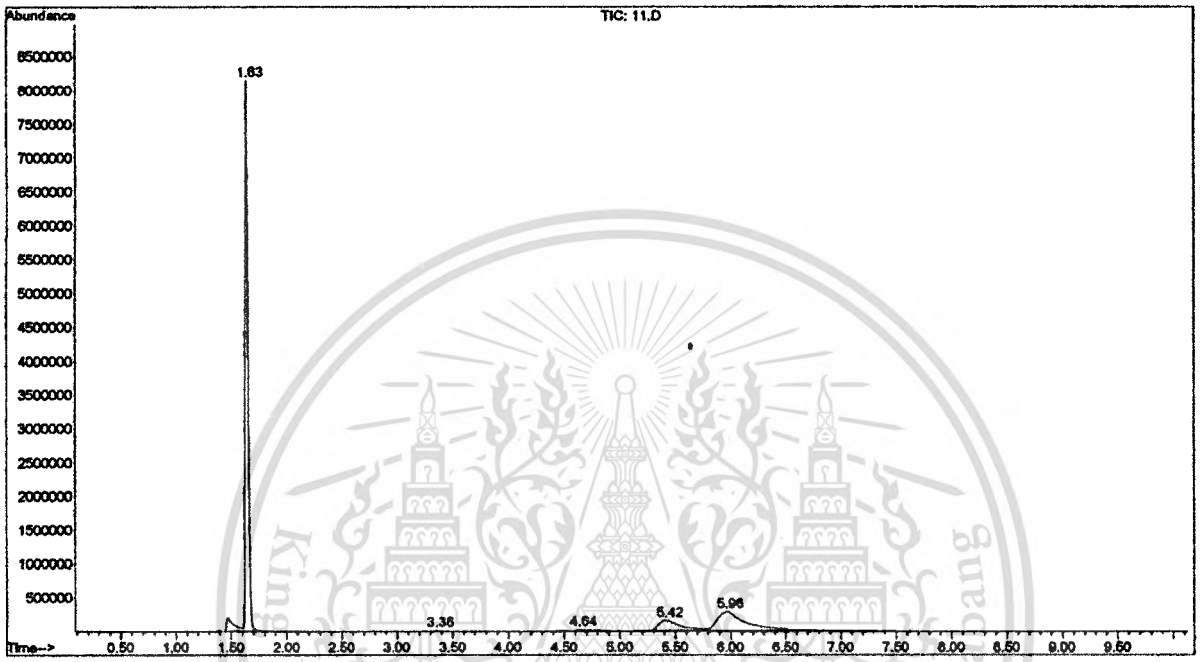
```
Data File : D:\55_0023\13.D                              Vial: 1
Acq On    : 16 May 2012  13:46                        Operator:
Sample    : sample 13                                  Inst   : Instrumen
Misc      :                                           Multiplr: 1.00
                                                  Sample Amount: 0.00

Signal    : TIC
```

Peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.629	249	287	324	rM	7028329	14645427	100.00%	69.212%
2	3.351	599	606	615	rM 4	4170	10050	0.07%	0.047%
3	4.721	832	860	909	rM 2	11270	158581	1.08%	0.749%
4	5.422	958	990	1052	rM 3	104420	1585141	10.82%	7.491%
5	5.989	1053	1095	1265	rM 3	207769	4761128	32.51%	22.500%

```
Sum of corrected areas:    21160327
```

Figure D.13 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Water filter resin as catalyst at 300w and 60 second.

```
File       : D:\55_0023\11.D
Operator   :
Acquired   : 16 May 2012  12:36     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 11
Misc Info  :
Vial Number: 1
```



Area Percent Report

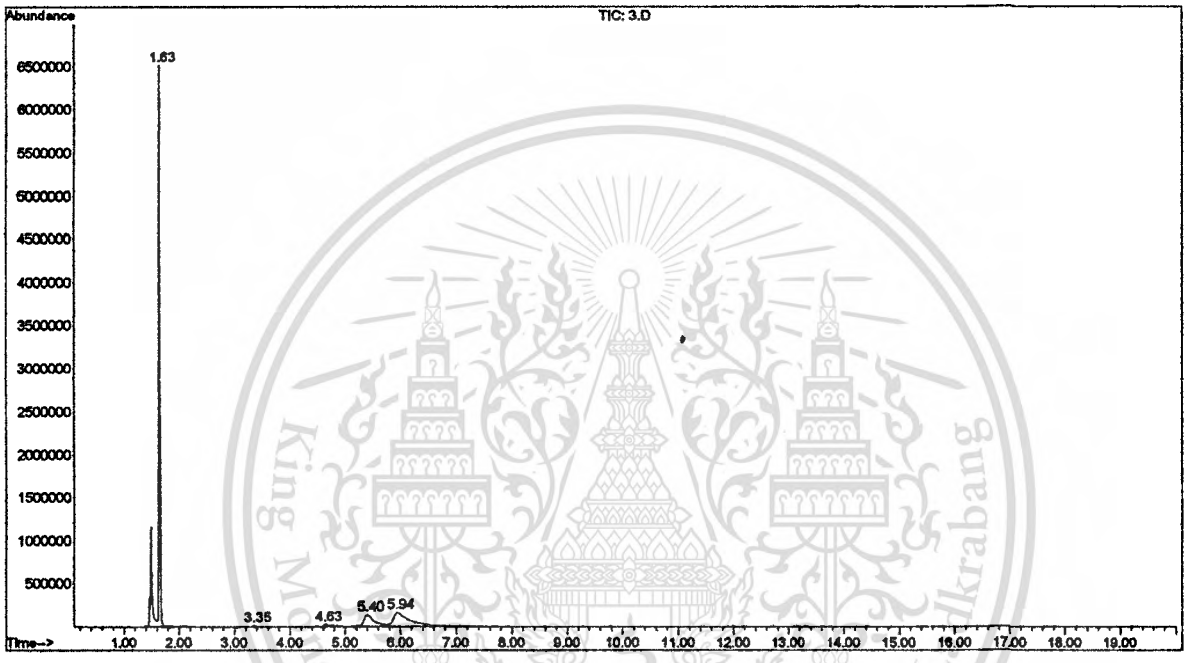
```
Data File : D:\55_0023\11.D                          Vial: 1
Acq On    : 16 May 2012  12:36                    Operator:
Sample    : sample 11                             Inst    : Instrumen
Misc      :                                       Multiplr: 1.00
                                                  Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.635	253	288	314	rM	8145063	16269859	100.00%	67.666%
2	3.356	601	607	615	rM	19041	39475	0.24%	0.164%
3	4.640	832	845	908	rM 2	27303	362672	2.23%	1.508%
4	5.417	963	989	1053	rM 3	153505	2094432	12.87%	8.711%
5	5.962	1054	1090	1241	rM 2	284233	5277974	32.44%	21.951%

Sum of corrected areas: 24044412

Figure D.14 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Water filter resin as catalyst at 300w and 90 second.

File : D:\55_0023\3.D
Operator :
Acquired : 15 May 2012 15:50 using AcqMethod ACETIC
Instrument : Instrumen
Sample Name: sample 3
Misc Info :
Vial Number: 1



Area Percent Report

Data File : D:\55_0023\3.D
Acq On : 15 May 2012 15:50
Sample : sample 3
Misc :

Vial: 1
Operator:
Inst : Instrumen
Multiplr: 1.00
Sample Amount: 0.00

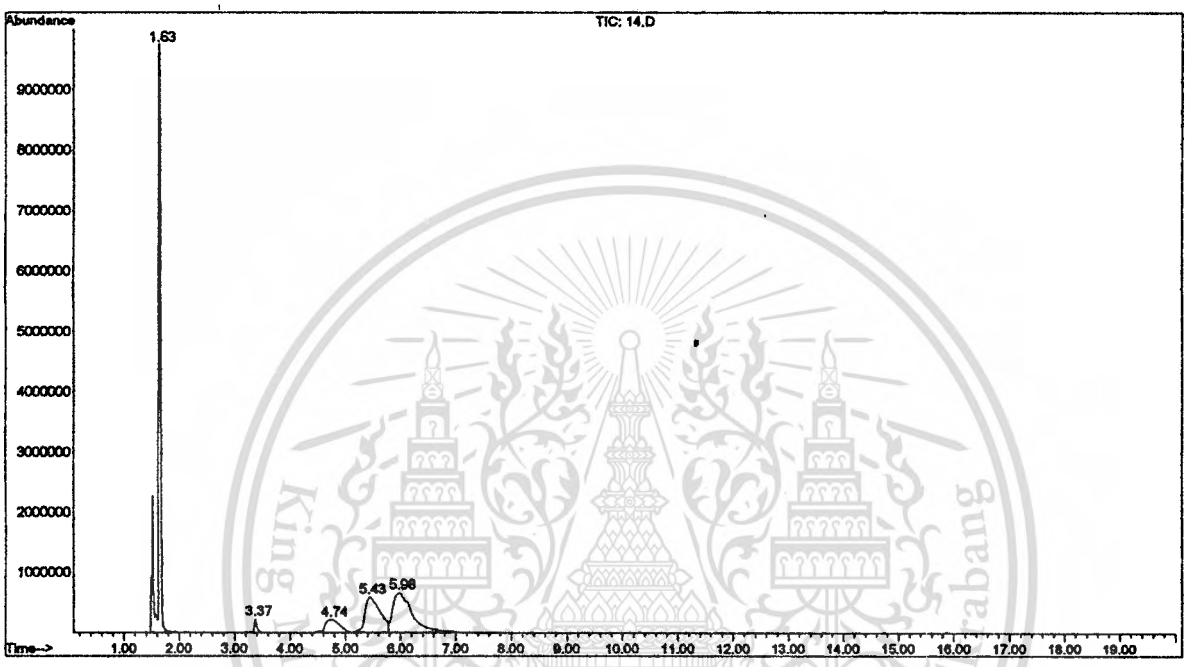
Signal : TIC

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.635	253	288	322	rM	6518290	12956945	100.00%	69.285%
2	3.351	600	606	615	rM 2	17819	41169	0.32%	0.220%
3	4.635	834	844	900	rM	25379	292118	2.25%	1.562%
4	5.401	937	986	1052	rM 2	136572	1880042	14.51%	10.053%
5	5.940	1053	1086	1261	rM 2	165109	3530631	27.25%	18.879%

Sum of corrected areas: 18700905

Figure D.15 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Water filter resin as catalyst at 300w and 120 second.

```
File       : D:\55_0023\14.D
Operator   :
Acquired   : 16 May 2012  14:16     using AcqMethod ACETIC
Instrument :   Instrumen
Sample Name: sample 14
Misc Info  :
Vial Number: 1
```



Area Percent Report

```
Data File : D:\55_0023\14.D                          Vial: 1
Acq On    : 16 May 2012  14:16                    Operator:
Sample    : sample 14                             Inst    : Instrumen
Misc      :                                       Multiplr: 1.00
                                                  Sample Amount: 0.00

Signal    : TIC
```

peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.629	258	287	323	rM	9747486	31404023	100.00%	50.306%
2	3.367	600	609	631	rM	224468	693240	2.21%	1.111%
3	4.737	812	863	927	rM 2	210255	3678384	11.71%	5.892%
4	5.433	942	992	1055	rM 2	582428	10830722	34.49%	17.350%
5	5.978	1056	1093	1286	rM 2	655270	15819113	50.37%	25.341%

Sum of corrected areas: 62425482

Figure D.16 : GC-MS Chromatogram of esterification between glycerol and acetic acid with Water filter resin as catalyst at 300w for 120 second then 100w for 180 second.