CONCENTRATION OF BIOPETROL SYNTHESIZED FROM STEARIC ACID THROUGH CATALYTIC CRACKING USING ZEOLITE AS CATALYST

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JUDUL: **CONCENTRATION OF BIOPETROL SYNTHESIZED FROM STEARIC ACID THROUGH CATALYTIC CRACKING USING ZEOLITE AS CATALYST**

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NUR AINI LIYANA BINTI ZAKARIA

A thesis submitted in fulfillment of the
requirements for the award of the degree of
Bachelor of Chemical Engineering

Faculty of Chemical and Natural Resources Engineering
Universiti Malaysia Pahang

APRIL 2009

DECLARATION

I declare that this thesis entitled *"Concentration of Biopetrol synthesized from Stearic Acid through catalytic cracking using Zeolite as catalyst"* is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ………………………………
Name : Nur Aini Liyana binti Zakaria
Date : APRIL 2009

DEDICATION

To my beloved parents Abah, Ma, and siblings,

ACKNOWLEDGEMENT

At first I want thanked to Ilahi for all those good blessings as I tend to finish this research. This research, did gave me big impact in my life as I able to managed and hold the responsible to strive to the fullest effort. I do believe in phrase "Practice makes Perfect" as during the first trial of the experiment the result is quite handful and disappointing. But when it comes to the second and third trials, everything is much easier and I tend to solve the problems smoothly. I want to thank to all people around who do care and help me out when it comes to difficulties.

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ABSTRACT

Biopetrol is an alternative fuel that have same characteristic with the petrol in term of its molecular formula, or to be specifically the octane number of molecules. In this research, the objective is to study the effect of catalyst which is Zeolite to enhance the production of Isooctane from Stearic Acid. The experiment starts with heating the Stearic Acid until it melts at 69.6^{o}C, and then 20 grams of zeolite being added and the heating process proceed until the temperature reached 98^{o}C as the isooctane produce will evaporates. The next experiment is repeated by with different mass of zeolite which is 10 grams, 5 grams and 1 gram. This reaction is called as Catalytic Cracking, where the zeolite will enhance the production of Isooctane. From the result obtained from the Gas Chromatogram result, the percentage of actual concentration of isooctane obtained for 20 g Zeolite is 3.95%, 10g zeolite is 2.769%, 5g zeolite is 3.387% and 1g Zeolite is 2.946%. The highest percentage of Isooctane produced is Stearic Acid distillate with 20g Zeolite, which is 3.95%. This experiment could be more effective by applying dynamic state concept to the process.

ABSTRAK

Biopetrol adalah salah satu alternatif bahan bakar yang mempunyai karakter yang sama dengan petrol biasa iaitu dari segi formula molekulnya. Untuk lebih tepat lagi, ia mempunyai bilangan oktana dalam struktur molekul. Dalam penyelidikan ini, objektif utama adalah untuk mengkaji kesan pemangkin Zeolite dalam meningkatkan lagi penghasilan Isooktana (biopetrol) daripada Asid Stearik. Experimen di mulakan dengan memanaskan Asid Stearik sehingga menjadi cecair pada suhu 69.6^{o}C dan seterusnya ditambah dengan zeolite dan penasan diteruskan sehingga suhu mencecah 98^{o}C di mana isooktana yang sudah terbentuk akan meruap. Experimen diulang dengan menggunakan berat Zeolite yang berbeza iaitu 10 gram, 5 gram and 1 gram. Kaedah in dikenali sebagai kaedah penguraian pemangkin, di mana Zeolte akan membantu meningkatkan penghasilan isooktana. Daripada keputusan Gas Chromatogram yang diperolehi, peratusan kepekatan untuk 20 g Zeolite adalah 3.95%, 10g zeolite adalah 2.769 %, 5g zeolite 3.387% dan 1g zeolite adalah 2.946%. Kepekatan isooktana yang diperolehi dari didihan Asid Stearik dengan 20 g zeolite iaitu 3.95%. Experimen ini dapat diperbaiki dengan mengaplikasi konsep keadaan dinamik ke atas prosess ini.

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LIST OF SYMBOLS

P - Pressure
M - Mass
ΔH - Enthalpy change of reaction
ΔS - Entropy change of reaction
ΔG - Energy change of reaction
T - Temperature
P - Density
M - Viscosity of liquid (Pa.s)
H - Heat transfer coefficient
^{o}C - Degree Celsius
kg - Kilogram
K - Degree Kelvin
M - Meter
N - Number of moles
L - Liter

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CHAPTER 1

INTRODUCTION

1.0 Introduction

Based on world crisis today, demanding the fuel price risen up, it is really burden people. Since then, people getting on researches to search new alternative fuel to substitute petrol and bio-diesel. As a brief introduction, bio-petrol is one of bio-fuel that could replace petrol and diesel. Bio-fuel is much cheaper and bio-economical such as bio-diesel and bio-petrol. Bio-fuel produced from acid can be used as fuel. Advantages of biofuels are the following:

(a) biofuels are easily available from common biomass sources,
(b) They are represents a carbon dioxide-cycle in combustion,
(c) biofuels have a considerable environmentally friendly potential,
(d) There are many benefits the environment, economy and consumers in using biofuels, and
(e) They are biodegradable and contribute to sustainability [1].

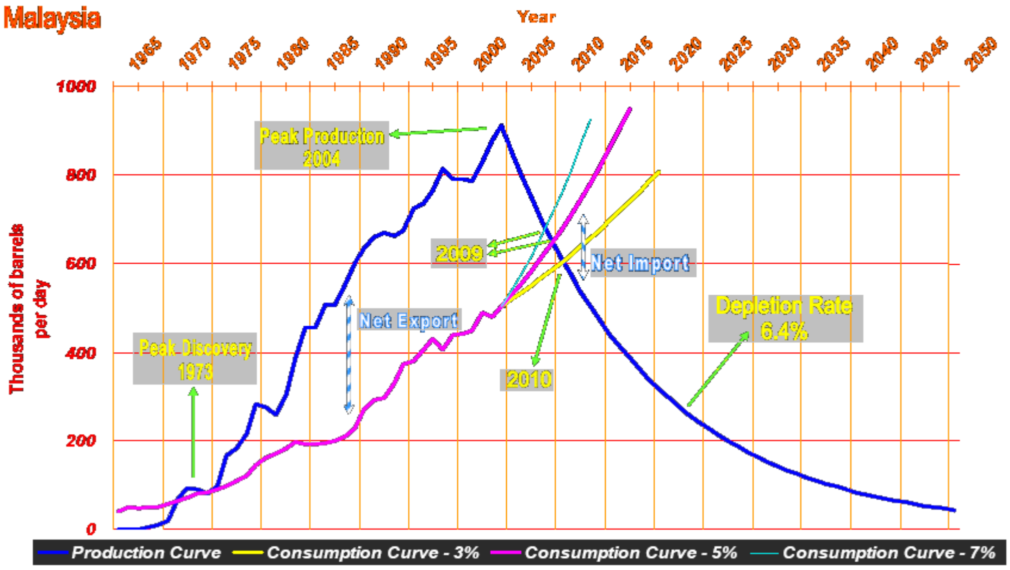
This research is about production of bio-petrol from catalytic cracking of stearic acid using zeolite as catalyst. The former research produced bio-petrol without using any catalyst. So, this study is to find out the effectiveness of catalytic cracking in producing biopetrol.

1.1 Problem Statement

The idea of this research is to solve out the fuel crises that really need to have other alternative. Currently, the world is depending to the only major source of energy as known as the petroleum fossil fuel. It is a nonrenewable energy and the next few decades the source to the crude oil will be finished. Since then, various studies are done to specify the global time in oil production between 1996 to 2035.

As we knew, Malaysia owns some petroleum sources and well known as one of the main fuel exporter to the world. Now, a few plants are using biomass energy technologies use waste or plant matter to produce energy with a lower level of greenhouse gas emissions than fossil fuel sources. Most of countries aware of this issue and they tend to develop research and modern technology to produce biofuels.

Figure 1.1 : Malaysia's Looming Energy Crisis (M. Noor, 2008)



Malaysia is the one of the major oil exporters in the world. Malaysia also has the crisis of the declining of these mineral sources. Malaysia's oil production is decreases in 2004 and would then decline by 6.4 percent annually. Figure 1.1 shows the declining Malaysia oil's production by 2004. Forecast, by 2009 to 2010 Malaysia will become a net importer because out of mineral sources (petroleum) and the demand of oil increasing. Bio-petrol is one of bio-fuels which can be fuel alternatives in substituting petrols and diesel.

At an average, about 0.1 tonne of Palm Oil Mill Effluent (POME) is generated for every tone of fresh fruit bunch processed. POME consists of water soluble components of palm fruits, saturated fatty acids as palmatic acid, stearic acid and oleic acid and also suspended materials as palm fiber and oil. Despite of its biodegradability, the POME cannot be discharged without treating it. This is because POME is very acidity and could pollute environment. By thinking of this, producing biopetrol from the POME can be the alternative solutions for treating the POME. At the same time, contribute to the production of biopetrol from the fatty acids. Producing petrol from the waste of palm oil will give an alternative choice to the users, especially for petrol-engine vehicles' owners. In addition, this biopetrol, which is graded 100 for its octane number, burns very smoothly so biopetrol can reduce emissions of some pollutants (Omar, 2005).

Stearic acid is one of the dominative components in palm oil waste. Its disposal into water supply sources causes serious water pollution. Besides that the loss of stearic acid as a useful industrial component also occurs so that it is not utilized much and always eliminated to improve and upgrade the quality of crude palm oil. Thus, it is disposed as palm oil waste and then pollutes water resources by its spillage.

At the fuel peak time, this research could be the very important as it could be commercialize. In this research, the concentration of isooctane that is produced from Stearic Acid and also the conversion of fatty acids form desired isooctane in biopetrol will be the objectives as well as studying the effect of catalyst to the production of Isooctane.

1.2 Objectives

- To analyze isooctane from Stearic Acid.
- To improve the production of biopetrol octane from catalytic cracking of Stearic Acid.

1.3 Scopes

There are some scopes that have been identified in this research. The scopes are listed as below:-

- To apply the catalytic cracking process.
- To determine the amount catalytic cracking process.
- To describe and understand the molecular arrangement in cracking process.
- To identify the presence of isooctane in the product solution by using Gas Chromatography
- To determine the amount of isooctane in sample obtained using Gas Chromatography method as well.
- To study the effectiveness of catalyst to the cracking process based on the amount of the catalyst.

CHAPTER 2

LITERATURE REVIEW

2.0 Introduction

Petroleum or fossil fuel has been the limited source. Since then, various studies are done to specify the global time in oil production between 1996 to 2035. Biomass energy technology use waste or plant matter to produce energy with a lower level of greenhouse gas emissions than the fossil fuel sources [3].Nowadays, in developed countries, there are researches and technologies to employ and applied efficient bioenergy conversion by using biofuels whereas getting competitively cheap compared with petrol.

Definition of fuel is any material that is burned or altered in order to obtain energy [4]. Fuel does releases energy when it is consumed by combustion or nuclear fusion. Fuel could neither store the energy nor released the energy when needed. The energy released can be transform into work.

All carbon-based life from microorganisms to animals and humans depend on and use fuels as their source of energy. Their cells engage in an enzyme-mediated chemical process called metabolism that converts energy from food or solar power into a form that can be used to sustain life [5]. In addition, there are some techniques where energy can be converted into another. As application of this conversion energy, human uses heat to cook and generating electricity daily application.

2.1 Fossil Fuel

Petroleum crude oil also known as fossil source fuels which only can be obtained from the earth's crust. Fossil fuel range from the highest boiling crude oil to the most volatile material with low carbon: hydrogen ratio such as methane. It is generally accepted that they formed from the fossilized remains of dead plants and animals [6] by exposure to heat and pressure for millions years. It was estimated by the Energy Information Administration that in 2006 primary sources of energy consisted of petroleum 36.8%, coal 26.6%, natural gas 22.9%, amounting to an 86% share for fossil fuels in primary energy production in the world. Non-fossil sources included hydroelectric 6.3%, nuclear 6.0%, and (geothermal, solar, wind, wood waste) amounting 0.9 percent [7].

As known, the fossil fuels are non-renewable resources as the take millions year to form and keeps depleting much faster than it forms. Currently fossil fuel is the major energy source; in the future insufficient fossil fuel will gives big impact to human. This contributes toward the generation of renewable energy to get any kind of energy and fuel alternative other than fossil fuel.

2.2 Biofuel

Biofuel is referred as liquid or gaseous fuels that are produced recent dead biological mass and the fossil fuel also derived from long term dead biological material or predominantly called as biomass. As theoretically, biofuels can be produced form any biological carbon source. A variety of biofuels can be produced from biomass resources including liquid fuels, for example ethanol, methanol, biodiesel, Fisher-Tropsch diesel, carboxylic acid and gaseous fuel such as methane and hydrogen.

Biofuels is an environmental-friendly fuel so, due to its environmental merits, it is not possible the biofuel will replace the fossil fuels in the automotive fuel market. Advabtages of biofuels are the following: (a) biofuels are easily available from common biomass sources, (b) they are representing a carbon doxide- cyle in combustion, (c) biofuels have a considerable environmentally friendly potential, (d) beneficial in environment, economic and consumer by using biofuel, (e) they are biodegradable and contribute to sustainability [8]. Liquid and gaseous fuels can be produce from biomass through thermochemical and biological reactions. It is proven that biofuels produced from biomass is non-polluting, have reliable resources and sustainable.

Most of the biofuel is burned to release its stored chemical energy. The largest advantage of biofuel in comparison of other fuel is that the energy within the biomass can be stored for an indefinite time-period without any danger.

2.2.1 Types of Biofuel

Biofuels stand for liquid or gaseous fuel for transport produced from biomass. They may be pure (100%) biofuels for dedicated vehicles or blend fuels in such a proportion that they can substitute conventional motor fuels without affecting car performance [8].

2.2.2 Bioalcohol

The alcohol fuels are usually of biological rather than petroleum sources. When obtained from biological sources, they are sometimes known as bioalcohols. Bioalcohol is one kind of biofuel produced from alcohol such as methanol (CH_3OH), ethanol (C_2H_5OH), propanol (C_3H_7OH), butanol (C_4H_9OH) which can be used for motor fuels. But, still the bioalcohol in development and research stages.

2.2.3 Bioethanol

Bioethanol is a fuel derived from plants such as wheat, sugar beet, corn and wood. Ethanol or ethyl alcohol produced by hydrolysis and fermentation process where the carbohydrates in the plant material can be converted into sugar by hydrolysis process. The fermentation process is to convert sugar to alcohols by microorganism, usually yeast. The resulting alcohol from the process is ethanol

2.2.4 Biomethanol

Currently, methanol made from the natural gas, but it also can be produced via partial oxidation reactions [9]. In figure 2.1 shows the production of biomethanol from carbohydrates by gasification and partial oxidation with O_2 and H_2O.

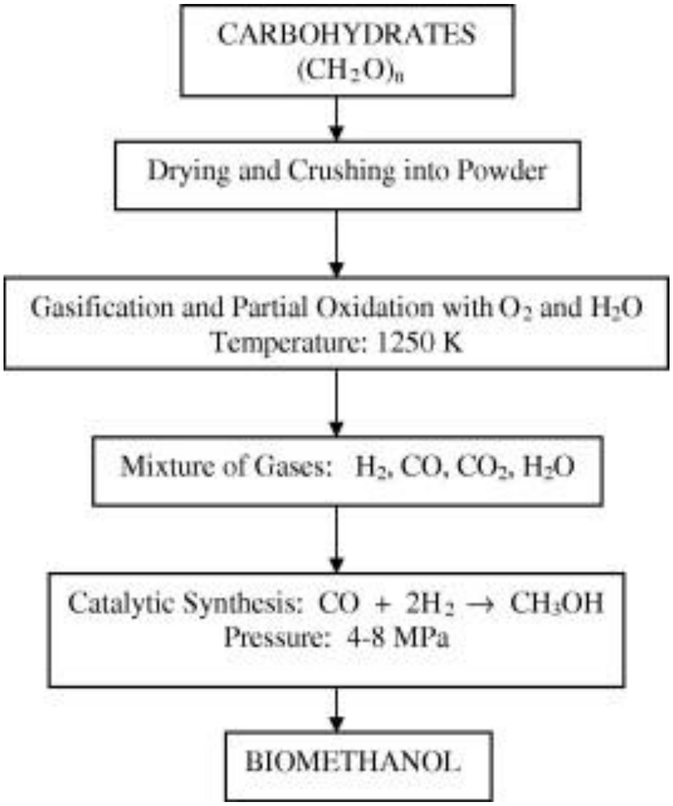


Figure 2.1: Biomethanol from carbohydrates by gasification and partial oxidation with O_2 and H_2O.

Adding sufficient hydrogen to the synthesis gas to convert all of the biomass into methanol carbon than double the methanol produced from the same biomass base [10].

As a renewable resource, biomass represents a potentially inexhaustible supply of feedstock for methanol production.

2.2.5 Vegetable Oils or Biodiesel

Vegetable oils are chemically triglycerides molecules where three fatty acids groups are attached to one glycerol molecule. It can be converted into maximum liquid and gaseous hydrocarbons by pyrolysis, decarboxylation, deoxygenation and catalytic cracking process [11, 12]. The liquid produced from pyrolysis process has similar chemical components to the conventional diesel fuel and can be used as alternative engine fuel. The process transesterification converts the vegetables oils into methyl, ethyl, 2-propyl and butyl esters by presence of potassium and/ or sodium alkoxides as catalyst. This is process is purposely done to lower down the viscosity of the oil by transforming the large-branched molecular structure into smaller-straight chain bio-oil molecules [8]. Biodiesel is technically proved that it is much better than the convectional petroleum diesel fuel.

2.2.6 Fischer-Tropsch Diesel or Bio-oil

The Fischer-Tropsch process is one of the advanced conversion technology that contribute the gasification of biomass feedstock, cleaning and conditioning the produced synthesis gas and liquid biofuel. Any type of biomass can be used as a feedstock, including woody and grassy materials and agricultural and forestry residues. The biomass is gasified to produce synthesis gas, which is a mixture of carbon monoxide (CO) and hydrogen (H_2). Prior to synthesis, this gas can be conditioned using the water gas shift to achieve the required H_2/CO ratio for the synthesis. The liquids produced from the syngas, which comprise various hydrocarbon fractions, are very clean which is sulphur free, straight-chain hydrocarbons, and can be converted further to automotive fuels. Figure 2.2 below, shows the process steps in production of green diesel and green gasoline facilities from biomass via Fisher–Tropsch synthesis.

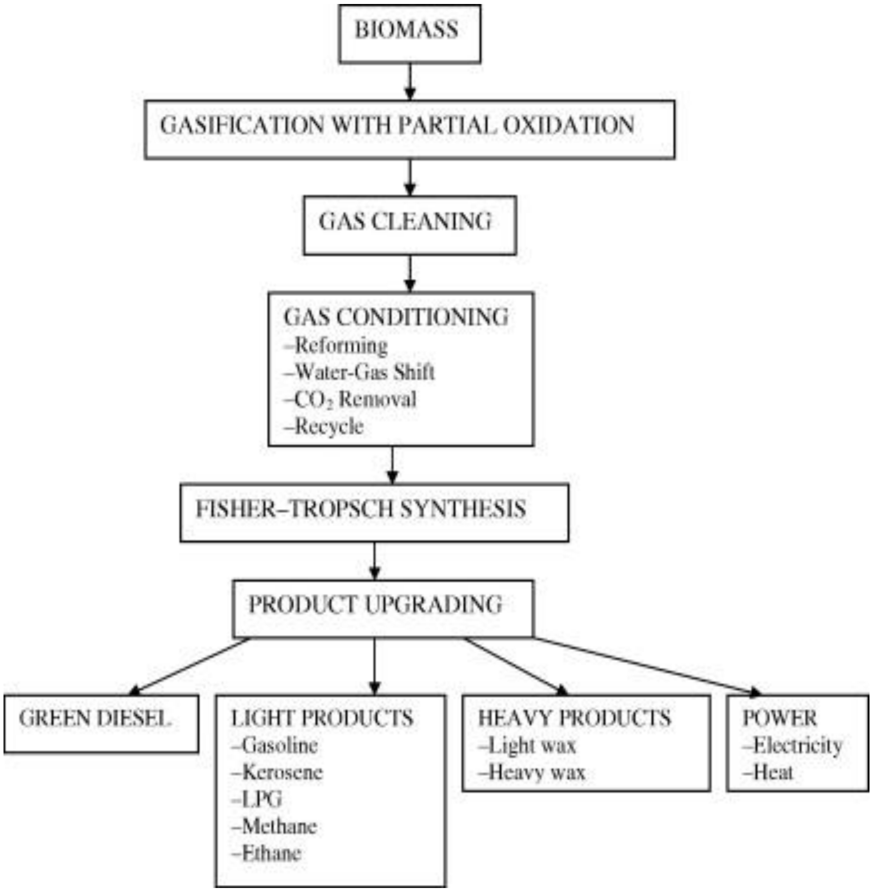


Figure 2.2: Green diesel and green gasoline facilities from biomass via Fisher–Tropsch synthesis

2.2.7 Bio-gas

Term "biogas" is usually defined as methane and carbon dioxide mixture produced from anaerobic digestion of biomass sludge such as animal wastes, sewage sludge and industrial effluents. This biogas produced in digester filled with feedstock like dung or sewage [8]. Basically, the process includes the anaerobic digestion of organic material by anaerobes. The solid byproduct, digestive, can be used as a biofuel or a fertilizer.

This kind of conversion do contribute energy and at the same time is beneficial effect to environment as well as human health, this is because during the digestion process bacteria in the sludge killed.

2.2.8 Biopetrol

Gasoline or petrol is a complex mixture of hydrocarbons which consists a mixture of C_4 to C_{10} alkanes. However isooctane ($C_{18}H_{18}$) as dominative component in petrol is assigned an octane number of 100. Isooctane or 2, 2, 4-trimethylpentane ($CH_3C(CH_3)_2CH_2CH(CH_3)CH_3$) is burns smoothly with a little knock in petrol engine. It is the highest quality of petrol (Mansur, 2005).

The other alternative to produce petrol is by using biopetrol. Biopetrol is defined as fuel which has the same characteristics with the commercial petrol in terms of its molecular formula.

2.3 Petroleum Refining

Petroleum refining is the process of separating the many compounds present in crude petroleum. This process is called fractional distillation where the crude oil is heated; where several of the compounds in crude oil will boil at different temperatures and change to gases; and are later condensed back into liquids. This is based on principle which the longer the hydrocarbon, the higher the boiling points [16].

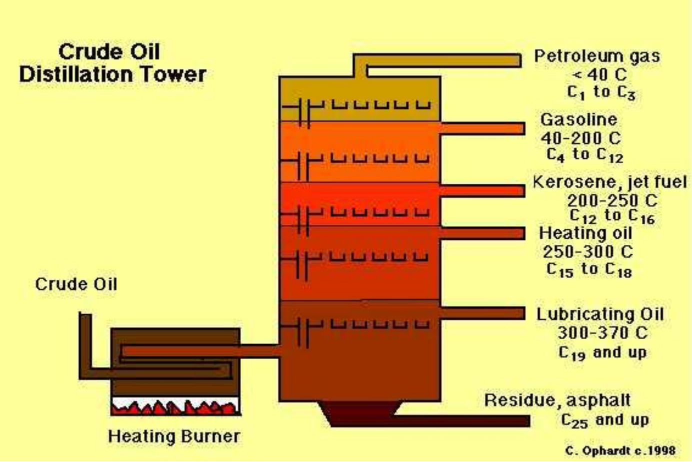


Figure 2.3: Distillation column in refining petroleum

Basically the crude petroleum is heated and changed into a gas. The gases are passed through a distillation column which becomes cooler as the height increases. When a compound in the gaseous state cools below its boiling point, it condenses into a liquid. The liquids may be drawn off the distilling column at various heights. Petroleum refining is industrial process plant where pure crude oil is processed and refined into various useful petroleum products such as gasoline, diesel fuel, heating oil, kerosene and liquefied petroleum gas [15] as shown in figure 2.3. It is very complex plant completed with different equipments and process. Table 2.1 shows the boiling points of each compound during the refining process.

Table 2.1: Boiling point for each compound in hydrocarbon cracking (Solomons, 1997)

Boiling Range of Fraction	**Number of Carbon Atoms per Molecule**	**Use**
Below 20	C1 – C4	Natural Gas, bottled gas, petrochemicals
20 – 60	C5 – C6	Petroleum ether, solvents
60 – 100	C6 – C7	Ligroin, solvents
40 – 200	C5 – C10	Gasoline (straight run gasoline)
175 – 325	C12 – C18	Kerosene and jet fuel
250 – 400	C12 and higher	Gas oil, fuel oil, and diesel oil

In refining petroleum, there are few processes in converting crude oil into smaller useful components such as Catalytic Cracking, Hydrocracking, Alkylation and Catalytic Reforming. Catalytic cracking also known as Fluid Catalytic Cracking (FCC) is one of the most important conversion processes in petroleum refineries. It is used to convert high boiling hydrocarbons of petroleum crude oil to become more valuable gasoline, olefinic gaseous and other products. Originally, cracking process of hydrocarbon in refining petroleum starts with thermal cracking. Then, the technology upgrades and completely replaced by catalytic cracking which is much better than thermal cracking. By catalytic cracking, the gasoline produced is higher octane rating and produce more byproducts with more olefinic which means more valuable.

At high temperature, the FCC process vaporizes and breaks the long chain hydrocarbon liquid into much shorter molecules with the presence of fluidized powdered catalyst with a moderate pressure. These technologies are based on an acidic zeolite as the catalyst. Currently, synthetic zeolites are widely used as catalysts in the petrochemical industry, for instance in fluid catalytic cracking and hydrocracking process. The hydrogen forms of zeolites are prepared by the ion-exchange makes zeolite to be a powerful solid-state acid, and can facilitate a host of acid-catalyzed reactions, such as isomerisation, alkylation, and cracking.

Zeolite is being widely used in petroleum refining industries. It can be a strong evidence that zeolite has good physical and chemical properties in enhance cracking process. In production of isooctane from stearic acid can be improved by applying zeolite in catalytic cracking of Stearic Acid.

2.3.1 Gasoline

Gasoline or petrol is a petroleum-derived liquid mixture, primarily used as fuel in internal combustion engines. It is one of the minor products produced by the fractional distillation of crude oil which is separated into fractions accordingly by applying different boiling points of hydrocarbons of varying chain length. It is a complex mixture of over 500 hydrocarbons that may have between 5 to 12carbons where smaller hydrocarbon rating C_5 to C_{12}. Each barrel of crude oil distilled in the fractional distillation, will approximately produce 25% gasoline. In table 2.1 below showed the typical composition of gasoline.

Table 2.2: Typical composition of gasoline

Typical Composition of Gasoline		
General Name	**Examples**	**Percentage (%)**
Aliphatic - straight chain	Heptane	30-50
Aliphatic - branched	Isooctane	
Aliphatic - cyclic	Cyclopentane	20-30
Aromatic	Ethyl benzene	20-30

It consists mostly of aliphatic hydrocarbons, enhanced with isooctane or the aromatic hydrocarbons toluene and benzene to increase its octane rating. Normally some additives are being added in order to increase performance and reducing emissions. Some mixtures also contain significant quantities of ethanol as a partial alternative fuel.

2.3.2 Octane number

The octane number is a measurement of fuel resistance to knock. The octane number is determined by comparing the characteristics of a gasoline to isooctane (2,2,4-trimethylpentane). Isooctane is assigned an octane number of 100. It is a highly branched compound that burns smoothly, with little knock. In comparison, heptane with a straight chain, unbranched molecule is given an octane rating of 0 because of its bad knocking properties.

Conventional gasoline which directly produced from the refinery distillation column has an octane number of about 70. In other words, conventional gasoline has the same knocking properties as a mixture of 70% isooctane and 30% heptane. Many of these compounds are straight chain alkanes. Cracking, isomerization, and other refining processes can be used to increase the octane rating of gasoline to about 90 [16].

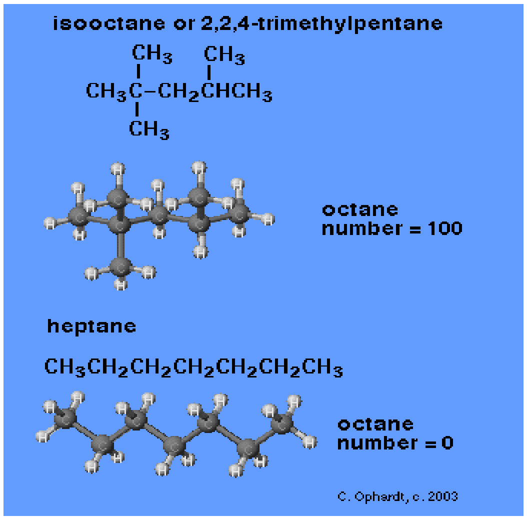


Figure 2.4: Comparison of octane number between isooctane and heptane

2.3.3 Biopetrol from Stearic Acid

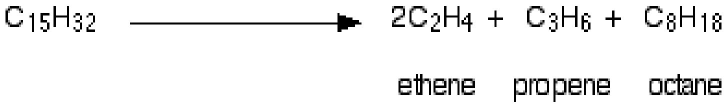
Stearic acid is prepared by treating animal fat with water at a high pressure and temperature, leading to the hydrolysis of triglycerides. It can also be obtained from the hydrogenation of some unsaturated vegetable oils. Stearic Acid or also known as Octadecanoic acid $C_{18}H_{36}O_2$ is a fatty acid which is normally found in animal and plants.

In producing biopetrol from Stearic Acid, catalytic cracking process is applied in order to break the hydrocarbon bonding of Stearic acid to become smaller molecules of hydrocarbon such as ethylene, methane, isooctane and smaller molecules of carboxylic acid. At first, melt the Stearic Acid which its boiling point is at 68^0C. The heating proceeds until 98^0C with additional of zeolite catalyst to enhance the cracking process and anti bumping granules to sustain uniform temperature for the whole solution. The solution produced, contain various components with smaller carbon arrangement. This is caused by random isomerization. The desired component is octane which is major component of gasoline.

The catalytic cracking of the saturated stearic acid yields much higher gasoline and gas (H2, C1-C4) fractions than rapeseed oil and oleic acid (fatty acid with a single unsaturated bond). In addition, the concentration of aromatics in the obtained gasoline from the stearic acid is significantly lower than that of the rapeseed oil and oleic acid [13].

2.4 Cracking Process

Definition of cracking is the name given to breaking up large hydrocarbon molecules into smaller ones. This is achieved by using high pressures and temperatures without a catalyst, or lower temperatures and pressures in the presence of a catalyst. The source of the large hydrocarbon molecules is often the naphtha fraction or the gas oil fraction from the fractional distillation of crude oil (petroleum). These fractions are obtained from the distillation process as liquids, but are re-vaporized before cracking.
The hydrocarbon molecules are broken up in a fairly random way to produce mixtures of smaller hydrocarbons, some of which have carbon-carbon double bonds. Figure 2.1 shows the hydrocarbons broken into smaller hydrocarbons.



$$C_{15}H_{32} \longrightarrow 2C_2H_4 + C_3H_6 + C_8H_{18}$$

Figure 2.5: Cracking process of $C_{15}H_{32}$

One possible reaction involving the hydrocarbon $C_{15}H_{32}$ might produce ethene, propene and octane in figure 2.2. This is only one way in which this particular molecule might break up. The ethene and propene are important materials for making plastics or producing other organic chemicals. The octane is one of the molecules found in petrol (gasoline).

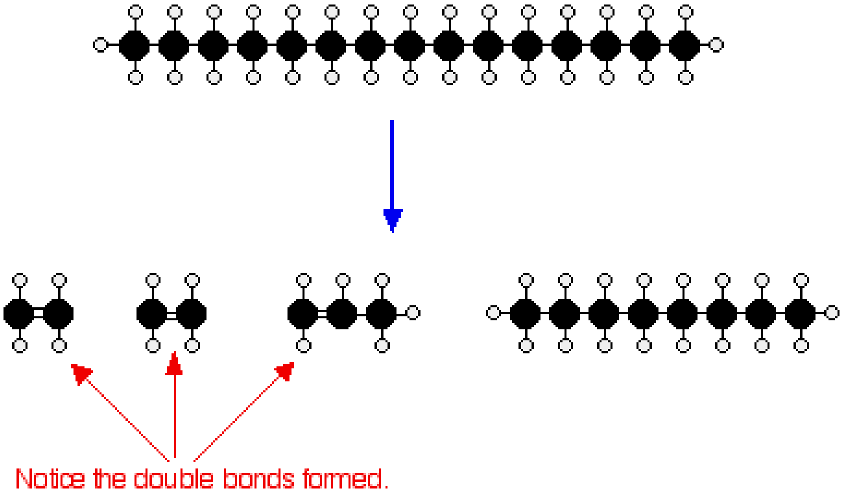


Figure 2.6: Molecules are broken into smaller hydrocarbon

2.4.1 Thermal Cracking

Modern high-pressure thermal cracking operates at absolute pressures of about 7,000 kPa. An overall process of disproportionation can be observed, where "light", hydrogen-rich products are formed at the expense of heavier molecules which condense and are depleted of hydrogen. The actual reaction is known as homolytic fission and produces alkenes, which are the basis for the economically important production of polymers [14].

Thermal cracking does not go via ionic intermediates like catalytic cracking. Instead, carbon-carbon bonds are broken so that each carbon atom ends up with a single electron as shown in figure 2.3. In other words, free radicals are formed. Reactions of the free radicals lead to the various products.

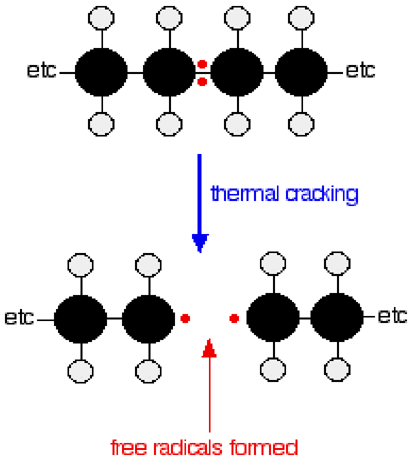


Figure 2.7: Overall view of thermal cracking process (Clark, 2003)

The main reactions of thermal cracking include:

1. Initiation reactions, where a single molecule breaks apart into two free radicals. Only a small fraction of the feed molecules actually undergo initiation, but these reactions are necessary to produce the free radicals that drive the rest of the reactions. In steam cracking, initiation usually involves breaking a chemical bond between two carbon atoms, rather than the bond between a carbon and a hydrogen atom.

$$\mathbf{CH_3CH_3 \rightarrow 2\ CH_3\bullet}$$

2. Hydrogen abstraction, where a free radical removes a hydrogen atom from another molecule, turning the second molecule into a free radical.

$$\mathbf{CH_3\bullet + CH_3CH_3 \rightarrow CH_4 + CH_3CH_2\bullet}$$

3. Radical decomposition, where a free radical breaks apart into two molecules, one an alkene, the other a free radical. This is the process that results in the alkene products of steam cracking.

$$\mathbf{CH3CH2\bullet \rightarrow CH2{=}CH2 + H\bullet}$$

4. Radical addition, the reverse of radical decomposition, in which a radical reacts with an alkene to form a single, larger free radical. These processes are involved in forming the aromatic products that result when heavier feedstocks are used.

$$\mathbf{CH_3CH_2\bullet + CH_2{=}CH_2 \rightarrow CH_3CH_2CH_2CH_2\bullet}$$

5. Termination reactions, which happen when two free radicals react with each other to produce products that are not free radicals. Two common forms of termination are recombination, where the two radicals combine to form one larger molecule, and disproportionation, where one radical transfers a hydrogen atom to the other, giving an alkene and an alkane.

$$\mathbf{CH_3\bullet + CH_3CH_2\bullet \rightarrow CH_3CH_2CH_3}$$
$$\mathbf{CH_3CH_2\bullet + CH_3CH_2\bullet \rightarrow CH_2{=}CH_2 + CH_3CH_3}$$

2.4.2 Catalytic Cracking

Modern cracking uses zeolites as the catalyst. These zeolites are complex of alkaline metal aluminosilicates, and they have large lattices of aluminium, silicon and oxygen atoms carrying a negative charge. They are, of course, associated with positive ions such as sodium ions.

The zeolites used in catalytic cracking are chosen to give high percentages of hydrocarbons with between 5 and 10 carbon atoms - particularly useful for petrol (gasoline). It also produces high proportions of branched alkanes and aromatic hydrocarbons like benzene.

The zeolite catalyst has sites which can remove hydrogen from an alkane together with the two electrons which bound it to the carbon. That leaves the carbon atom with a positive charge. Ions like this are called carbonium ions (or carbocations).

So figure 2.4 shows the basic mechanism of catalytic cracking. Reorganisation of these leads to the various products of the reaction.

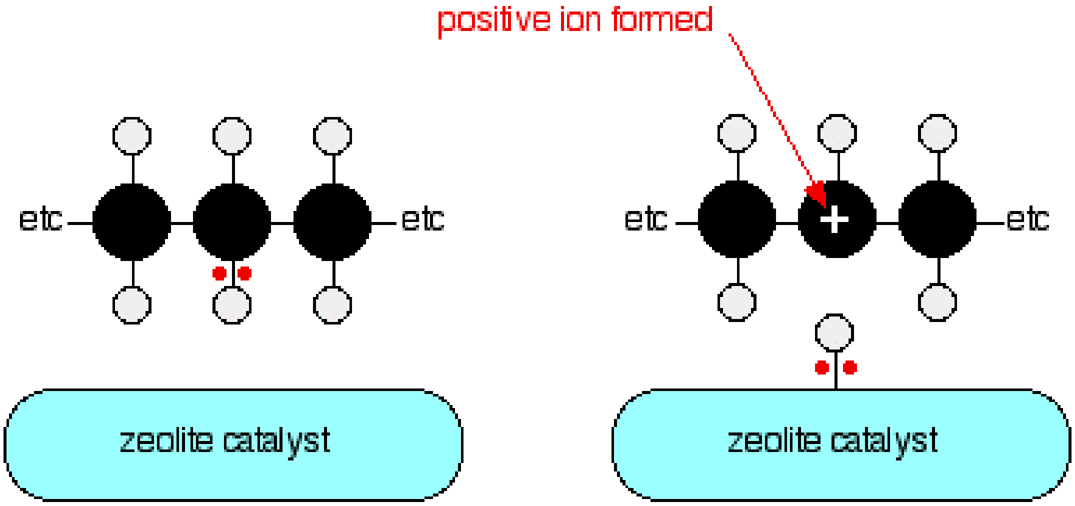


Figure 2.8 : Mechanism of Catalytic cracking

2.4.3 Thermal Cracking versus Catalytic Cracking

Thermal cracking is a simpler technique compared to catalytic cracking. In industries nowadays, thermal cracking operates in high-pressure about 7,000 kPa. It is also consume heat to breaks the carbon bonding of the reactants. This is thermal cracking is higher cost than catalytic cracking.

For the catalytic cracking, the process does not consume much heat, because the usage of zeolite will lower down the activation of the process and react as thermal cracking. But the advantage is the heat being consumed is lower than thermal cracking and the process is reduced in less time.

Other than that, catalytic cracking is better because of the usage of zeolites as catalyst can contribute a higher conversion of isooctane. This indicates that the rate of conversion of fatty acid is temperature dependent and increases with temperature.

2.5 Zeolite Overview

Zeolite are crystalline aluminosilicates with fully cross-linked open framework structures made up by SiO4 and AlO4 tetrahedral. It also a catalyst with three-dimensional molecular sieve whereas a material with selective adsorption properties. So is capable in separating components of mixture based on the molecular size and shape. In addition to Si^{4+} and Al^{3+}, other elements can also be present in the zeolitic framework. They need not be isoelectronic with Si^{4+} or Al^{3+}, but must be able to occupy framework sites. Aluminosilicate zeolites display a net negative framework charge, but other molecular sieve frameworks may be electrically neutral. Zeolite confined with molecules in small spaces which causing changes in structure and reactivity. The hydrogen ions form of zeolite be the acidic properties as it is the powerful sold-state acid which can facilitate a host of acid-catalyzed reactions such as isomerization, and cracking process.

Figure 2.9: The real picture of Zeolite catalyst

2.5.1 Zeolite Structures

The framework of a molecular sieve is based on an extensive three-dimensional network in which the polyhedral sites, usually tetrahedral, are linked by oxygen atoms. The crystalline framework contains cages and channels of discrete size and 3-30 Å in diameter. The primary building unit of a molecular sieve is the individual tetrahedral unit.

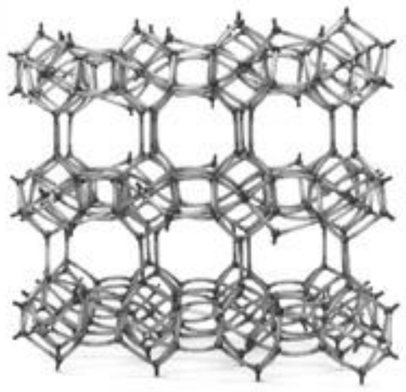


Figure 2.10: The structure of Zeolite

2.5.2 Zeolite Properties

Molecular sieves are selective, high-capacity adsorbents because of their high intracrystalline surface area and strong interactions with adsorbents. Molecules of different size generally have different diffusion properties in the same molecular sieve. Molecules are separated on the basis of size and structure relative to the size and geometry of the apertures of the sieve. Molecular sieves adsorb molecules, in particular those with a permanent dipole moments, and exhibit other interactions not found in other sorbents. Different polar molecules have a different interaction with the molecular sieve framework, and may thus be separated by a particular molecular sieve. This is one of the major uses of zeolites.

Zeolites with low Si/Al ratios have strongly polar anionic frameworks. The exchangeable cations create strong local electrostatic fields and interact with highly polar molecules such as water. The cation-exchange behaviour of zeolites depends on (1) the nature of the cation species, the cation size (both anhydrous and hydrated) and cation charge, (2) the temperature, (3) the concentration of the cationic species in the solution, (4) the anion associated with the cation in solution, (5) the solvent (most exchange has been carried out in aqueous solutions, although some work has been done in organics), and (6) the structural characteristics of the particular zeolite.

Cation exchange in a zeolite is accompanied by an alteration of stability, adsorption behavior and selectivity, catalytic activity and other properties. In some cases, the introduction of a larger or smaller cation will decrease or enlarge the pore opening.

Shape selectivity, including reactant shape selectivity, product shape selectivity or transition-state shape selectivity, plays a very important role in molecular sieve catalysis. The channels and cages in a molecular sieve are similar in size to medium-sized molecules. Different sizes of channels promote the diffusion of different reactants, products or transition-state species. High crystallinity and the regular channel structure are the principal features of molecular sieve catalysts.

Reactant shape selectivity results from the limited diffusivity of some of the reactants, which cannot effectively enter and diffuse inside the crystal. Product shape selectivity occurs when slowly diffusing product molecules cannot rapidly escape from the crystal, and undergo secondary reactions. Restricted transition-state shape selectivity is a kinetic effect arising from the local environment around the active site: the rate constant for a certain reaction mechanism is reduced if the necessary transition state is too bulky to form readily.

The catalytic sites in the zeolite are strong acid as equivalent to 90% sulphuric acid and provide most of the catalytic activity. The acidic sites are provided by the alumina tetrahedral.

2.6 Chemicals

2.6.1 Stearic Acid

Stearic acid (IUPAC systematic name: octadecanoic acid) is a saturated fatty acid that occurs in many animal, vegetable fats and oils. It is a waxy solid, and its chemical formula is $CH_3(CH_2)_{16}COOH$. Stearic acid is prepared by treating animal fat with water at a high pressure and temperature, leading to the hydrolysis of triglycerides with removal of glycerol. It can also be obtained from the hydrogenation of some unsaturated vegetable oils. Common stearic acid is actually a mix of stearic acid and palmitic acid, although purified stearic acid is available separately. Stearic acid is in fatty acid group, which is a carboxylic acid with long carbon chain.

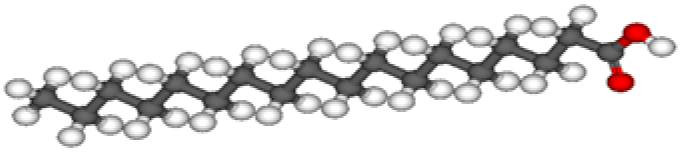


Figure 2.11: 3-D molecular structure of Stearic Acid

2.6.1.1 Properties of Stearic Acid

Stearic acid is a saturated fatty acid that occurs in many animal and vegetable fats and oils. Stearic acid also can be characterized as fatty acid which in group carboxylic acid with a long unbranched aliphatic chain. Fatty acids derived from natural fats and oils may be assumed to have at least 8 carbon atoms.

Saturated fatty acids do not contain any double bonds or other functional groups along the chain. The term "saturated" refers to hydrogen, in that all carbons which is apart from the carboxylic acid group (–COOH) contain as many hydrogen as possible. Saturated fatty acids form straight chains and, as a result, can be packed together very tightly, allowing living organisms to store chemical energy very densely. The fatty tissues of animals contain large amounts of long-chain saturated fatty acids. Below,

Table 2.3 roughly shows the physical and chemical properties of Stearic acid and figure 2.11 shows molecular structure of Stearic acid.

Table 2.3: Physical and Chemical properties of Stearic Acid

Synonyms	Octadecanoic acid; 1-heptadecanecarboxylic acid
Appearance	White or yellowish-white powder.
Molecular formula	$CH_3\ (CH_2)_{16}COOH$
Molecular weight	284.48 g/mol
Melting point ^0C	69 - 70°C
Boiling point ^0C	383°C
Specific gravity	0.94
Solubility in water	Insoluble in water.

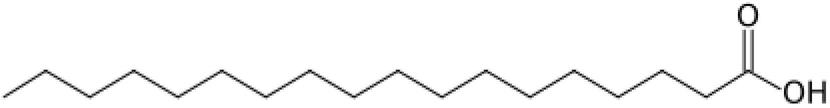


Figure 2.12: molecular structure of stearic acid

2.6.2 Isooctane

Isooctane is also known as 2,2,4-Trmethylpentane which is an octane isomer which defines the 100 point of the octane number as the zero point is heptane. Isooctane is the major component of gasoline. In this experiment, isooctane is the major product which to produce it from Stearic Acid through catalytic cracking using zeolite as catalyst.

Isooctane is produced on a massive scale in the petroleum industry, usually as a mixture with related hydrocarbons. The alkylation process alkylates isobutane with isobutylene using a strong acid catalyst. In the NExOCTANE process, isobutylene is dimerized into isooctene and then hydrogenated to isooctane.

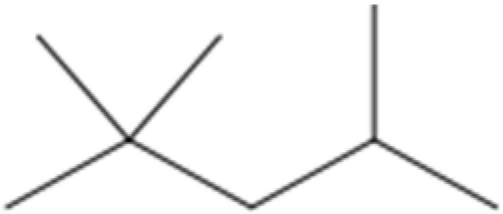


Figure 2.13: Skeletal structure of isooctane

2.6.2.1 Properties of isooctane

Each chemical substance has different physical and chemical properties, which make it varies with each other. Physical and chemical properties of isooctane shows in table 2.4.

Table 2.4: Physical and Chemical properties of Isooctane (Safety data for 2,2,4-trimethylpentane, 2005)

Synonyms	Isobutyltrimethylpentane, 2,2,4-Trimethylpentane
Appearance	colourless liquid
Molecular formula	C_8H_{18} or $CH_3C(CH_3)_2CH_2CH(CH_3)CH_3$
Molecular weight	114.22 g/mol
Melting point ^{0}C	-107.38°C
Boiling point ^{0}C	99.3°C
Density	0.688 g/ml
Specific gravity	0.692
Solubility in water	Immiscible
Auto ignition temperature:	396°C

Isooctane 100 is a mixture of C_8 isoparaffins virtually does not contain any aromatic or sulphur compounds. Isooctane has -48kJ/g of enthalphy of combustion.

Isooctane 100 has a range of properties including low toxicity, lack of color, high color stability, low odor and rapid evaporation or drying which makes it an excellent solvent for a variety of surface applications [14]. Isooctane is particularly useful in the formulation of gasolines containing ethanol in direct blending, for example in those produced in the United States, thanks to its extremely low volatility that counterbalances the very high volatility of ethanol.

Isooctane is a flammable liquid and vapor even it physically a clear liquid with odor of gasoline. It is harmful to skin, eyes, respiratory tract, liver, kidneys and central nervous system. Where liquid contact is possible impervious coveralls are recommended. To minimize the possibility in other handling and storage operations, we should wear appropriate PPE to include chemical resistant gloves, boots and apron. Other than that, we should wear protective eye glasses and do the experiment inside fume hood to avoid the isooctane vaporized to the open air. Isooctane also must be kept in a good ventilation area and keep away from any source of ignition.

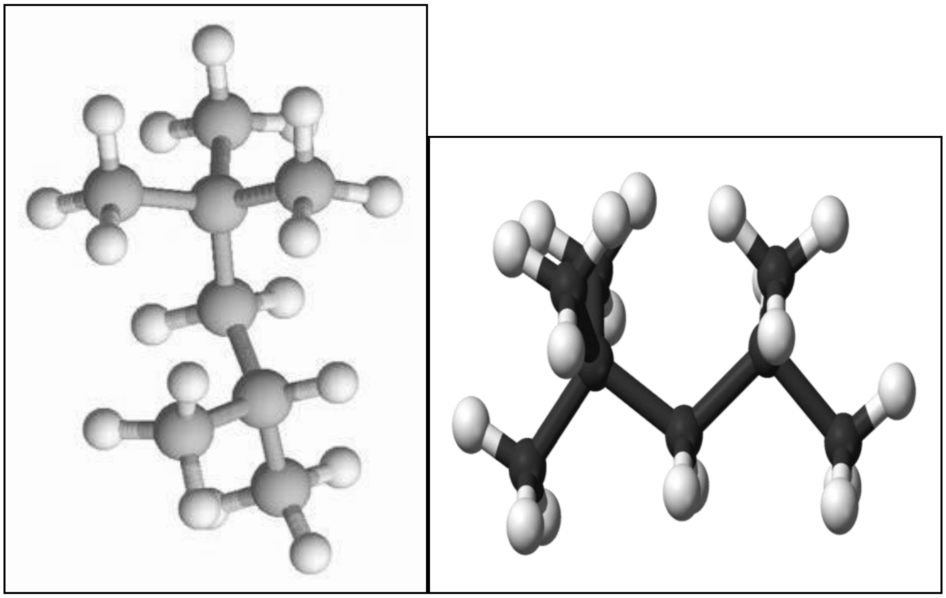


Figure 2.14: 3-dimensional molecular structure of isooctane

CHAPTER 3

METHODOLOGY

3.1 Chemical Substances

In this research, the raw materials are stearic acid, zeolite catalyst, hexane as dilution agent and solvent for standard, anti bumping granules (solid) functioning as an agent to uniform the temperature of the whole solution while solution is being heated, and isooctane as standard solution for Gas Chromatography calibration.

3.2 Apparatus

Apparatus used in this research is the heating mantel 250mL, condenser, thermometer 360^{o}C, round-bottom flask 50mL and heating plate. Figure 3.1 indicated set up of the apparatus.

Figure 3.1 : Set Up of the Apparatus

3.3 Experimental Works

There are 4 major scopes of experimental works that need to be achieving in order to study the production of isooctane from the samples. They are:

i. Preparation of calibration curve for standard pure isooctane
ii. Sample preparation (stearic acid distillate)
iii. Analysis the sample using Gas Chromatography Method
iv. Determination of isooctane concentration obtained from palmitic acid distillate

3.4 Preparation of Calibration Curve for Isooctane

Six standard calibration solution of 40 mL isooctane-hexane mixtures with different compositions are prepared, injected into vials, labeled and analyzed using gas chromatography method to find the peak area of hexane and isooctane for each calibration isooctane-hexane mixture.

Table 3.1 : Sample of Isooctane-Hexane Mixture

Vial	Composition (%)	
	Isooctane (mL)	**Hexane(mL)**
1	50 % (20mL)	50% (20mL)
2	40% (16mL)	60% (24mL)
3	30% (12mL)	70% (28mL)
4	20% (8mL)	80% (32mL)
5	10% (4mL)	90% (36mL)
6	0% (0mL)	100% (40mL)

These standard calibration solutions need to be injected into Gas Chromatogram for every test. This because, when we leave the isooctane-hexane in the G.C, there is some possibilities that the solution will react and produce new peak in the calibration curve. This will lead to different calibration curve for isooctane and hexane.

3.4 Sample Preparation

In this experiment, there will be 4 sample need to be prepared and analyzed. Table 3.2 shows the components.

Table 3.2 : Samples using 4 different mass of catalyst

Mass of Stearic acid (grams)	Mass of catalyst (grams)
100g	20g
100g	10g
100g	5g
100g	1g

3.4.1 Experiment 1: Heating Stearic Acid with 20g of Zeolite

For your information, the stearic acid used in this experiment is in solid phase. So before proceeding with the production of iso-octane, the stearic acid is heated until it melts at 69.4 ^{o}C. At the same time, the stearic acid is heated with 10 g anti-bumping granules and **20 g zeolite catalyst** to enhance the reaction. Anti- bumping granules able to uniform the separation of heat to the whole solution in order to make sure the reaction of the process is consistent.

As the stearic acid melts, the heating process continues until isooctane were produced. This reaction is called thermal cracking; but in addition of catalyst it is called catalytic cracking. At this moment, the heat will breaks the hydrocarbons bonding and rearrange it to isooctane and other compounds.

The heating process should not exceed 98^{o}C which is isooctane boiling point or else all of isooctane will vaporize. Cool down the apparatus and a portion of distillate at this stage is withdrawn using a syringe, which then injected into a vial and ready to be analyzed by G.C. Figure 3.2 below indicates the flow diagram of the preparation method used.

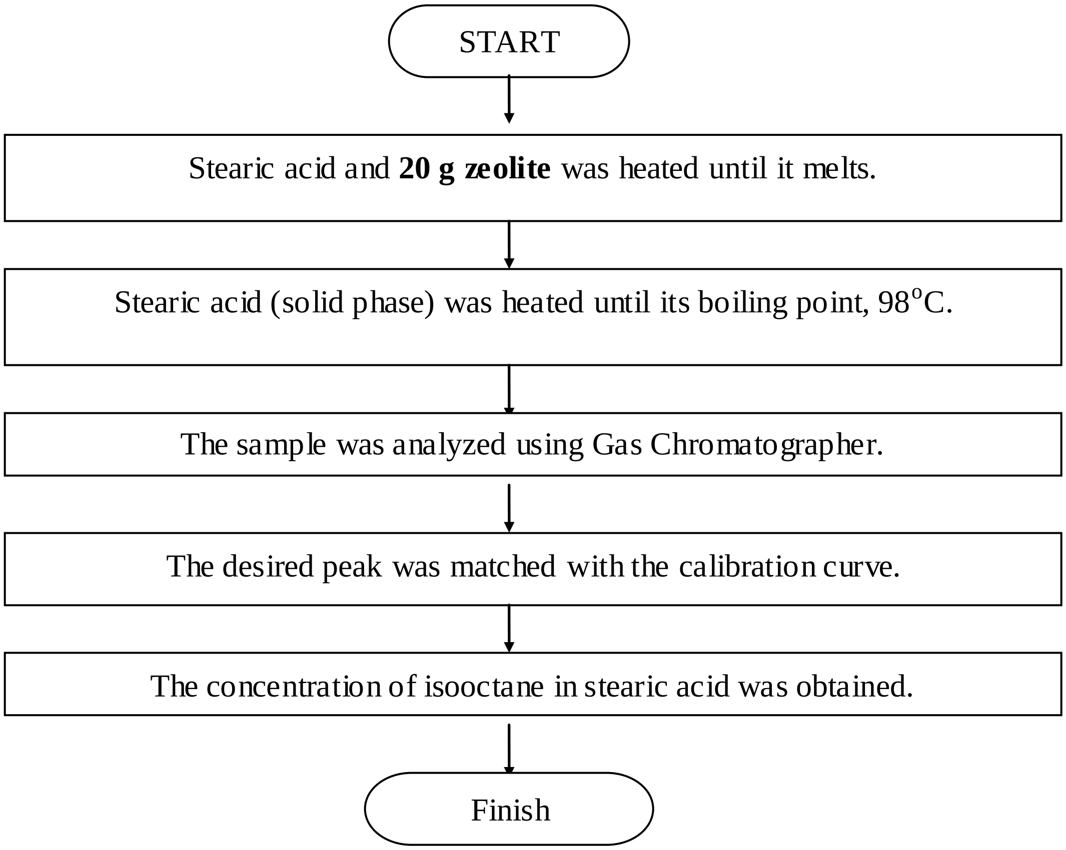


Figure 3.2 : Flow Diagram of Experiment 1

3.4.2 Experiment 2: Heating Stearic Acid with 10g of Zeolite

The process is still same as experiment 1. Stearic acid is heated until it melts at 69.4 ^{o}C. At the same time, the stearic acid is heated with 10 g anti-bumping granules and **10 g zeolite catalyst**. The mass of catalyst is being reduced. As the stearic acid melts, the heating process continues until isooctane were produced. This reaction is called thermal cracking; but in addition of catalyst it is called catalytic cracking. At this moment, the heat will breaks the hydrocarbons bonding and rearrange it to isooctane and other compounds.

The heating process should not exceed 98^{o}C which is isooctane boiling point or else all of isooctane will vaporize. Cool down the apparatus and a portion of distillate at this stage is withdrawn using a syringe, which then injected into a vial and ready to be analyzed by G.C.

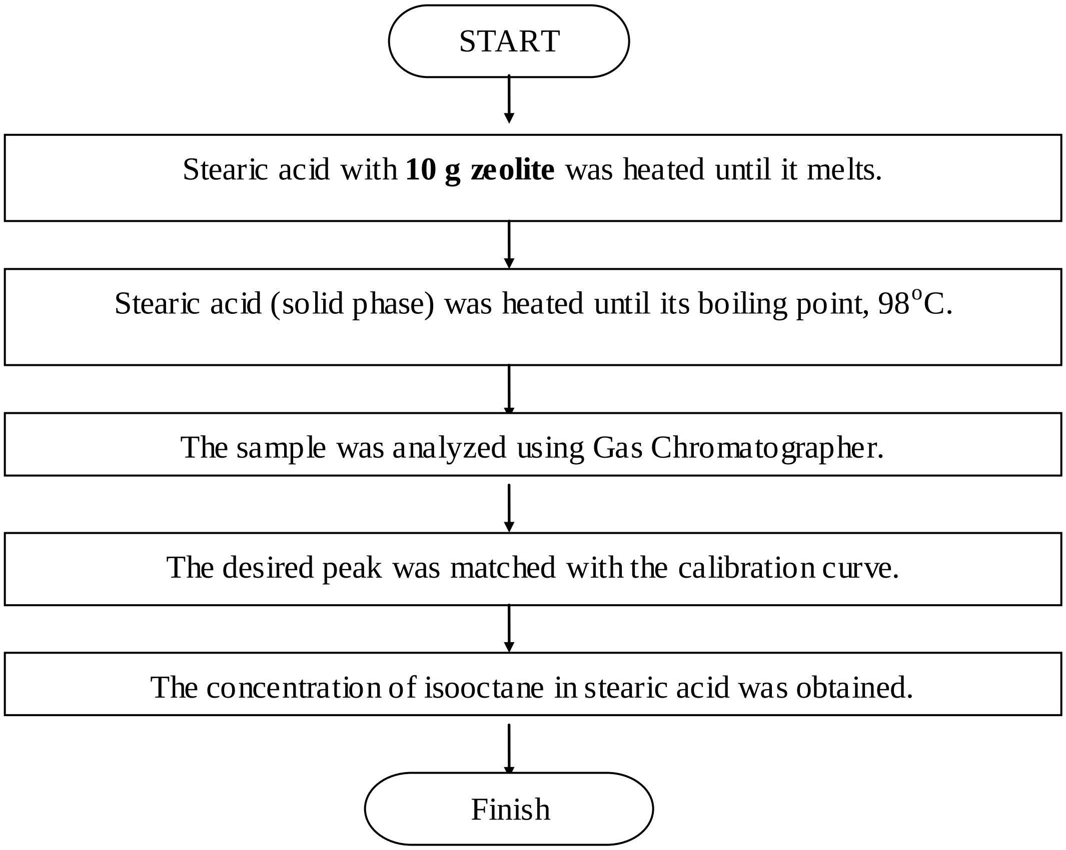


Figure 3.3: Flow Diagram of Experiment 2

3.4.3 Experiment 3: Heating Stearic Acid with 5 g of Zeolite

The process is still same as experiment 1. Stearic acid is heated until it melts at 69.4 °C. At the same time, the stearic acid is heated with 10 g anti-bumping granules and **5 g zeolite catalyst**. The mass of catalyst is being reduced. As the stearic acid melts, the heating process continues until isooctane were produced. This reaction is called thermal cracking; but in addition of catalyst it is called catalytic cracking. At this moment, the heat will breaks the hydrocarbons bonding and rearrange it to isooctane and other compounds.

The heating process should not exceed 98°C which is isooctane boiling point or else all of isooctane will vaporize. Cool down the apparatus and a portion of distillate at this stage is withdrawn using a syringe, which then injected into a vial and ready to be analyzed by Gas Chromatography method. Figure 3.4 below indicates the flow diagram of the method to use.

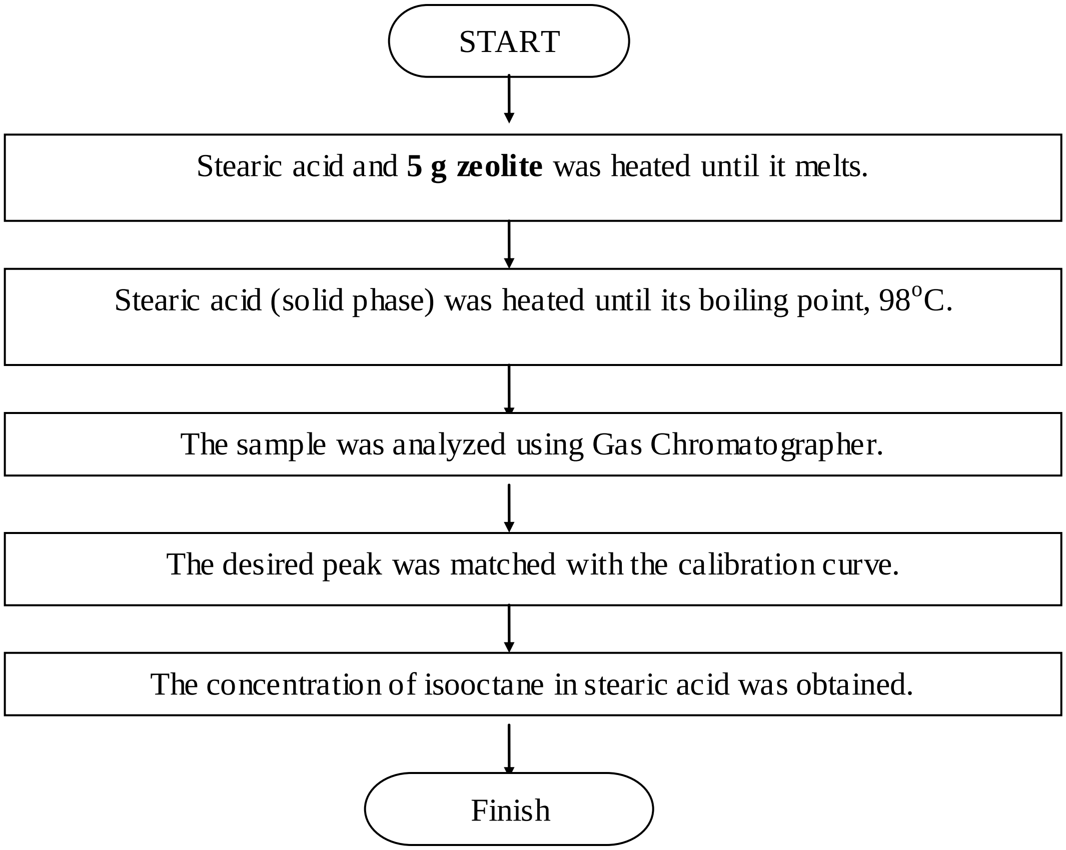


Figure 3.4: Flow Diagram of Experiment 3

3.4.4 Experiment 4: Heating Stearic Acid with 1 g of Zeolite

The process is still same as experiment 1. Stearic acid is heated until it melts at 69.4 ^oC. At the same time, the stearic acid is heated with 10 g anti-bumping granules and **1 g zeolite catalyst**. There is no presence of catalyst. As the stearic acid melts, the heating process continues until isooctane were produced. This reaction is called thermal cracking; but in addition of catalyst it is called catalytic cracking. At this moment, the heat will breaks the hydrocarbons bonding and rearrange it to isooctane and other compounds.

The heating process should not exceed 98^oC which is isooctane boiling point or else all of isooctane will vaporize. Cool down the apparatus and a portion of distillate at this stage is withdrawn using a syringe, which then injected into a vial and ready to be analyzed by Gas Chromatography method. Figure 3.5 below indicates the flow diagram of the method to use.

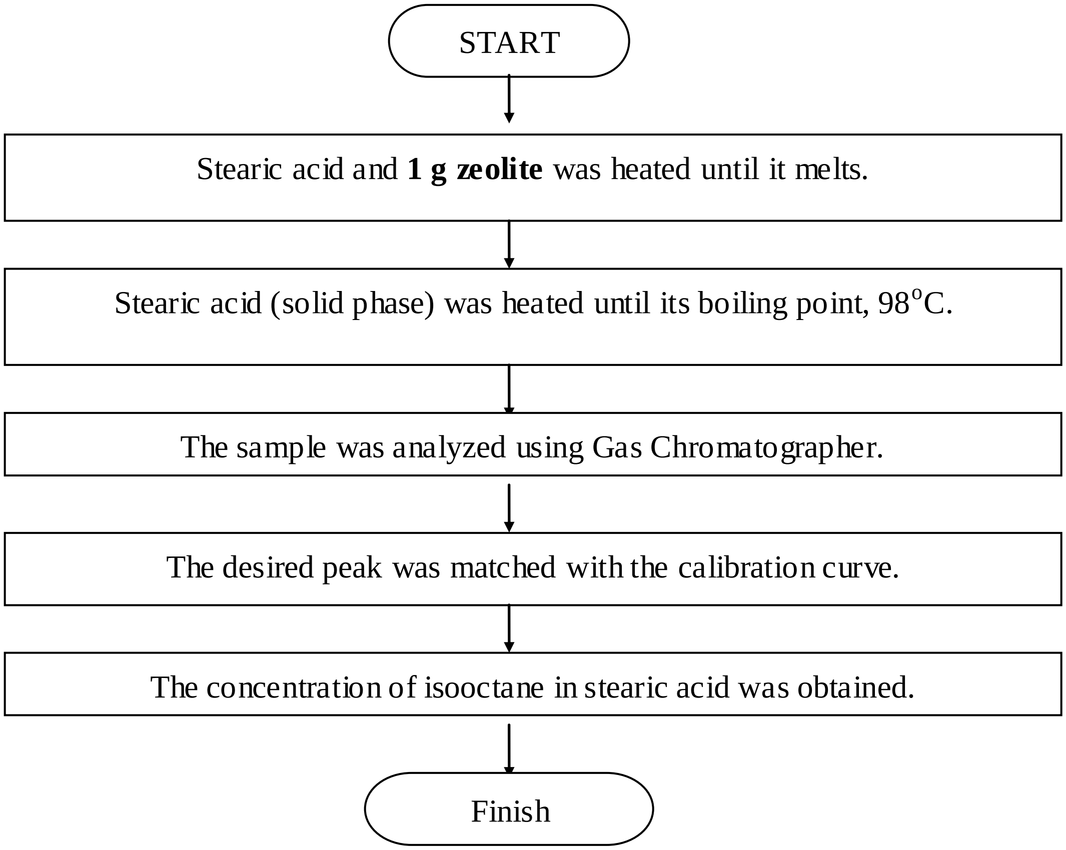


Figure 3.5: Flow Diagram of Experiment 4

Figure 3.6 below, showed the stearic acid distillate with 20 g of zeolite is ready for the next analysis samples. The samples need to be diluted immediately diluted with hexane into labeled Schott bottles and keep the samples warm inside 69.4 °C water bath to avoid the sample solidifies.

Figure 3.6: Stearic Acid distillate with 20g zeolite

3.5 Preparation of Analysis samples

For each major sample, the samples were diluted with different percentage of dilution agent which shown below. The sample will be diluted accordingly with hexane to get more samples to be analyzed. Table 3.3 showed the preparation analysis sample for each samples. Figure 3.7 showed the analysis samples of stearic acid distillate 20g zeolite, with concentration 20%, 10%, 5 % and 1 % respectively.

Table 3.3: The percentage of concentration in each analysis sample

Samples	Hexane
1%	99%
5%	95%
10%	90%
20%	80%

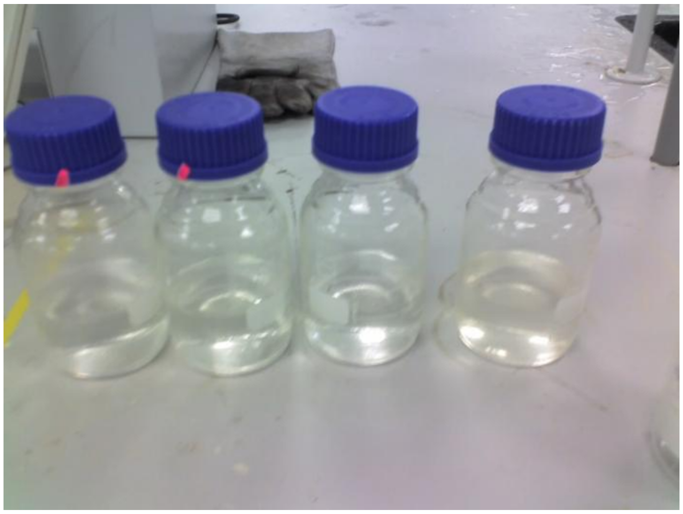


Figure 3.7: Analysis samples of Stearic Acid distillate

After being calculated, the total volume of the sample obtained is approximately 118 ml. So, each sample for 20g, 10 g, 5g and 1 g zeolites will be diluted according as stated in the table 3.4 below.

Table 3.4: Volume of samples and hexane needed to get the exact concentration

Samples	Volume of Sample (ml)	Volume of Hexane (ml)
1%	1.18	116.82
5%	5.9	112. 1
10%	11.8	106.2
20%	23.6	94.4

Each major sample will be diluted with four different concentration 20%, 10%, 5% and 1 % as stated before. So each sample will have 4 sample analyses. Overall samples will be 16 samples that will be injected into Gas Chromatographer, which are shown below in table 3.5:

Table 3.5: Analysis Samples

Stearic Acid with 20 g Zeolite	
1	1 % sample / 99 % hexane
2	5% sample / 95 % hexane
3	10% sample / 90 % hexane
4	20% sample / 80 % hexane
Stearic Acid with 10 g Zeolite	
5	1 % sample / 99 % hexane
6	5% sample / 95 % hexane
7	10% sample / 90 % hexane
8	20% sample / 80 % hexane
Stearic Acid with 5 g Zeolite	
9	1 % sample / 99 % hexane
10	5% sample / 95 % hexane
11	10% sample / 90 % hexane
12	20% sample / 80 % hexane
Stearic Acid with 1 g Zeolite	
13	1 % sample / 99 % hexane
14	5% sample / 95 % hexane
15	10% sample / 90 % hexane
16	20% sample / 80 % hexane

3.6 Analysis with Gas Chromatographer (GC)

3.6.1 Application of GC

Gas Chromatograph is a chemical analysis instrument functions to separate chemicals in complex sample and at same time it is a volatile material. A gas chromatograph technically using a narrow flow-through tube which is called as column, where each different chemical constituents of samples pass through in a carrier gas stream at different rates depending on their various chemical and physical properties. The stationary phase in the column is to separate those different components which causing it to exit the column at differ time. This contributes to the differential in retention time for each component. Though, Gas Chromatography does effected by carrier gas flow rate, pressure and temperature.

Figure 3.8: Gas Chromatographer

Theoretically, the process starts with injections of standard solutions of isooctane-hexane into Gas Chromatography collect data for reference data of composition of isooctane and also hexane as dilution agent. After that, all the 16 samples will be injected into Gas Chromatography one by one. It will burn the sample with different temperature to detect each component existing in each sample. The Chromatogram will appear to show every peak obtained for each component, the highest and the sharpest peak showed the highest concentration component in the sample. The peak appears first usually the lowest number carbon as the retention time do affect by the molecules size.

Table 3.6: Gas Chromatographer FID Data Condition (Omar, 2006)

Temperature Column	Initial 50^{o}C, hold 3 minutes, program at 8^{o}C/min to 185^{o}C, hold 5 minutes
Injector Temperature	225^{o}C
Detector Temperature	25^{o}C
Injection size	1.0µL (10:1 split)
Hydrogen flow	35mL/min
Air flow	450mL/min
Nitrogen make up	35mL/min
Carrier gas	Helium, Compress Air, H_2, N_2

3.6.2 Analysis Method

For analysis method, there will be 6 standard solutions of isooctane-hexane and 16 samples will be injected and analyzed by Gas Chromatographer. At first, the 6 standard solutions will be placed and followed by samples starts at vial 7 until vial 22. Table 3.7 and table 3.8, 3.9, 4.0 and table 4.1 below showed the samples contained in vial 1 until vial 22.

Table 3.7: Vials for standard solutions hexane- isooctane

Vial	**Standard Solutions**
1	50% isooctane, 50% hexane
2	40% isooctane, 60% hexane
3	30% isooctane, 70% hexane
4	20% isooctane, 80% hexane
5	10% isooctane, 90% hexane
6	0% isooctane, 100% hexane

Table 3.8: Vials for Stearic Acid with 20 g Zeolite

Vial	**Stearic Acid with 20 g Zeolite**
7	1 % sample / 99 % hexane
8	5% sample / 95 % hexane
9	10% sample / 90 % hexane
10	20% sample / 80 % hexane

Table 3.9: Vials for Stearic Acid with 10 g Zeolite

Vial	**Stearic Acid with 10 g Zeolite**
11	1 % sample / 99 % hexane
12	5% sample / 95 % hexane
13	10% sample / 90 % hexane
14	20% sample / 80 % hexane

Table 3.10: Vials for Stearic Acid with 5 g Zeolite

Vial	**Stearic Acid with 5 g Zeolite**
15	1 % sample / 99 % hexane
16	5% sample / 95 % hexane
17	10% sample / 90 % hexane
18	20% sample / 80 % hexane

Table 3.11: Vials for Stearic Acid with 1 g Zeolite

Vial	**Stearic Acid with 1 g Zeolite**
19	1 % sample / 99 % hexane
20	5% sample / 95 % hexane
21	10% sample / 90 % hexane
22	20% sample / 80 % hexane

These samples are placed accordingly in the gas chromatography's rack. GC was set up according to the GC condition in Table 3.2. All samples are then being analyzed to measure the peak area. Each of the sample takes about 20 minutes to be analyzed including 5 minutes time to cool it down before another samples can be analyze.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Results for Standard Isooctane

In Gas Chromatogram method, the sample should be injected with standard solution in order to have a specific range in detecting the compound. So, the standard isooctane mixtures are being prepared for each concentration. There are 50% isooctane, 40 % isooctane, 30% isooctane, 20% isooctane, 10 % isooctane and 0% isooctane. The standard mixtures are contained solvent hexane and pure isooctane.

4.1.1 Standard solution 50% isooctane

For the standard solution 50%, the composition of the mixture is 50% isooctane, 50% hexane. After being analyzed by Gas Chromatography method, the result shown as stated below in Table 4.1. It is indicated that, * as hexane and ** as isooctane. As stated below, the retention time of hexane is at 3.634 min, while isooctane is 4.332 min.

Table 4.1: Retention Time and Area for 50% Standard solution

Retention Time [min]	**Area [pA*s]**	**% Area**
3.446	1150.2	0.087
3.52	3745.2	0.437
***3.634**	448372.9	34.091
****4.332**	546779.3	41.574

4.1.2 Standard solution 40% isooctane

For the standard solution 40%, the composition of the mixture is 40% isooctane, 60% hexane. After being analyzed by Gas Chromatography method, the result shown as stated below in Table 4.2. As stated below, the retention time of hexane is at 3.61 min, while isooctane is 4.295 min.

Table 4.2: Retention Time and Area for 40% standard solution

Retention Time [min]	**Area [pA*s]**	**% Area**
3.408	1430.2	0.104
3.482	6911	0.505
***3.61**	699771.5	51.114
3.758	21784.4	1.541
****4.295**	490375.6	35.819

4.1.3 Standard solution 30% isooctane

For the standard solution 30%, the composition of the mixture is 30% isooctane, 70% hexane. After being analyzed by Gas Chromatography method, the result shown as stated below in Table 4.3. As stated below, the retention time of hexane is at 3.611 min, while isooctane is 4.259 min.

Table 4.3: Retention Time and Area for 30% standard solution

Retention Time [min]	**Area [pA*s]**	**% Area**
3.04	1594.4	0.141
3.474	7784.2	0.688
***3.611**	731657.3	64.633
3.754	22597.9	1.966
****4.259**	291829.5	25.779

4.1.4 Standard solution 20% isooctane

For the standard solution 20%, the composition of the mixture is 20% isooctane, 80% hexane. After being analyzed by Gas Chromatography method, the result shown as stated below in Table 4.4. As stated below, the retention time of hexane is at 3.412 min, while isooctane is 3.981 min.

Table 4.4: Retention Time and Area for 20% standard solution

Retention Time [min]	**Area [pA*s]**	**% Area**
3.236	1733.2	0.166
3.289	8663.1	0.797
***3.412**	806417.6	77.741
3.54	24594.1	2.371
****3.981**	178277	17.186

4.1.5 Standard solution 10% isooctane

For the standard solution 10%, the composition of the mixture is 10% isooctane, 90% hexane. After being analyzed by Gas Chromatography method, the result shown as stated below in Table 4.5. As stated below, the retention time of hexane is at 3.649 min, while isooctane is 4.229 min.

Table 4.5: Retention Time and Area for 10% standard solution

Retention Time [min]	Area [pA*s]	% Area
3.423	2044.7	0.209
3.49	5888.4	0.601
3.51	3755.9	0.384
***3.649**	857454.1	87.588
3.789	26256.3	2.682
****4.229**	83559.2	8.536

4.1.6 Standard solution 0% isooctane

For the standard solution 0%, the composition of the mixture is 0% isooctane, 100% hexane. After being analyzed by Gas Chromatography method, the result shown as stated below in Table 4.6. As stated below, the retention time of hexane is at 3.57 min, while isooctane in no detectable as 0 concentration in the standard isooctane-hexane mixture.

Table 4.6: Retention Time and Area for 0% standard solution

Retention Time [min]	Area [pA*s]	% Area
3.354	2143.5	0.209
3.416	10101.9	0.986
***3.57**	978981.5	95.544
3.697	29716.8	2.90
4.081	3691.1	0.360

From the data above, the peak area and retention time for Isooctane is been recorded into the table 4.7 below to get the calibration curve as the reference to compare the retention time of Isooctane in samples.

Table 4.7: Retention Time and Area for Standard Isooctane

Concentration of Isooctane (%)	Retention Time [min]	Peak Area [pA*s]
0%	0	0
10%	4.295	26256.3
20%	4.259	178277
30%	3.981	291829.5
40%	3.789	490375.6
50%	4.332	546779.3

From the data of Standard Isooctane, the calibration curve can be plotted. This is because the calibration curve can be used in order to determine the exact concentration for each sample by comparing the retention time with standard calibration curve.

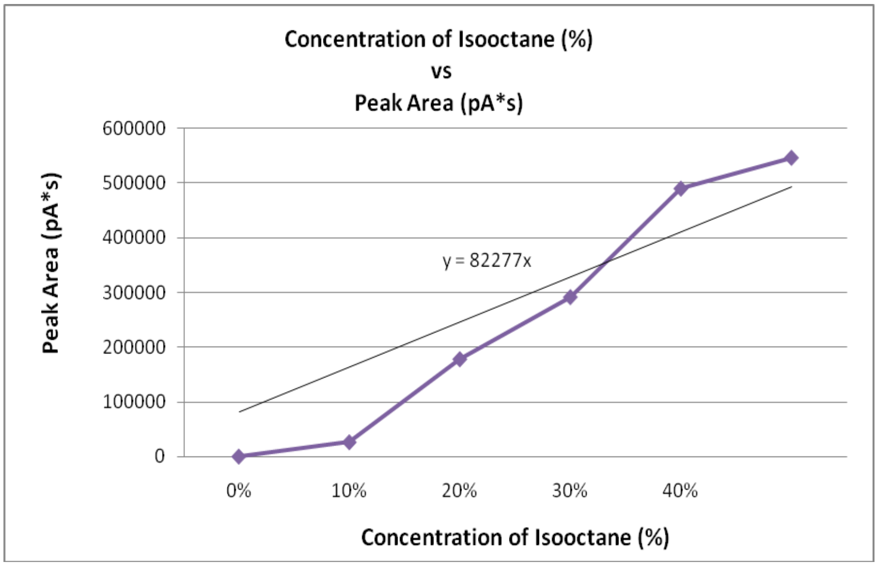


Figure 4.1: Calibration Curve for Standard Isooctane

4.2 Results for Samples (Stearic Acid distillate)

4.2.1 Stearic Acid with 20 g Zeolite

For Stearic Acid distillate with 20 g zeolite, the retention time for desired isooctane in range 3.884 min to 4.679 min, where the peak shown in the chromatogram is 5051.3 pA*s, 5289.2 pA*s, 5352.7 pA*s and 5277.3 pA*s for respectively for sample 20%, 10%, 5% and 1%. In all tables below, the * symbol indicate the desired isooctane. The overall data shown in the table 4.8 below;

Table 4.8: Retention Time and Area 20g Zeolite

Sample	Retention Time [min]	Area [pA*s]	%Area
20%	3.711 3.860 *4.679	1013017.5 75335.6 5051.3	83.573 6.215 0.417
10%	3.564 3.694 *3.921 4.064	956706 41647.7 5289.2 6.0023E-1	82.302 6.483 0.455 0.00
5%	3.710 3.859 *4.078	994597.4 77434.3 5352.7	81.972 6.382 0.441
1%	3.530 3.658 *3.884 4.064	974413.9 75799.3 5277.3 5.6E-1	81.612 6.349 0.442 0.00450

*shows the desired isooctane

4.2.2 Stearic Acid with 20 g Zeolite 10 g Zeolite

For Stearic Acid distillate with 10 g zeolite, the retention time for desired isooctane in range 3.759 min to 4.082 min, where the peak shown in the chromatogram is 4347.1 pA*s, 4393.9 pA*s, 4314.5 pA*s and 6157.5 pA*s for respectively for sample 20%, 10%, 5% and 1%. In all tables below, the * symbol indicate the desired isooctane. The overall data shown in the table 4.9 below;

Table 4.9: Retention Time and Area 10g Zeolite

Sample	Retention Time [min]	Area [pA*s)	%Area
20%	3.413 3.539 *3.759	953671.1 66544.4 4347.1	84.299 5.882 0.384
10%	3.441 3.567 *3.789 4.015	973286 67666.9 4393.9 3.1	84.277 5.859 0.380 0.00678
5%	3.718 3.864 *4.082	1101736.8 71751.6 4314.5	85.408 5. 562 0.334
1%	3.715 3.864 *4.082	1058629.8 86659.1 6157.5	80.950 6.627 0.471

4.2.3 Stearic Acid with 5 g Zeolite

For Stearic Acid distillate with 5 g zeolite, the retention time for desired isooctane in range 3.726 min to 4.083 min, where the peak shown in the chromatogram is 5670.8 pA*s, 3895 pA*s, 5570.8 pA*s and 2616.6 pA*s for respectively for sample 20%, 10%, 5% and 1%. In all tables below, the * symbol indicate the desired isooctane. The overall data shown in the table 4.10 below;

Table 4.10: Retention Time and Area 5g Zeolite

Sample	Retention Time [min]	Area [pA*s)	%Area
20%	3.704	852345.9	79.132
	3.858	78244.2	7.264
	*4.079	5670.8	0.441
10%	3.436	978482.9	85.266
	3.562	63702.6	5.551
	*3.784	3895	0.339
	4.009	3.1	0.0
5%	3.717	1071620.3	83.762
	3.865	81746.8	6.390
	*4.083	5570.8	0.435
1%	3.387	1035131.3	86.594
	3.508	54648.6	4.572
	*3.726	2616.6	0.219
	3.945	3.1	0.00054

4.2.4 Stearic Acid with 1 g Zeolite

For Stearic Acid distillate with 1 g zeolite, the retention time for desired isooctane in range 3.718 min to 4.079 min, where the peak shown in the chromatogram is 5198.7 pA*s, 5098.7 pA*s, 5108.4 pA*s and 3159.1 pA*s for respectively for sample 20%, 10%, 5% and 1%. In all tables below, the * symbol indicate the desired isooctane. The overall data shown in the table 4.11 below;

Table 4.11: Retention Time and Area 1g Zeolite

Sample	Retention Time [min]	Area [pA*s)	%Area
20%	3.705	867824	82.378
	3.757	75568.7	6.317
	3.859	4.1	0.00035
	*4.079	5198.7	0.486
10%	3.708	956623.3	82.378
	3.858	73358	6.317
	*4.077	5098.7	0.439
5%	3.375	946470.1	83.503
	3.502	83717	7.386
	*3.718	5108.4	0.451
1%	3.613	968372.2	84.647
	3.687	18.2	0.002
	3.747	57252.5	5.005
	*3.971	3159.1	0.276

4.3 Result of analysis Samples

4.3.1 Sample Analysis 20%

For the analysis samples 20%, the sample of 20% of stearic acid distillate is diluted with 80% of hexane. Based on the figure 4.2 below, the mass of zeolite do effect the production of isooctane from stearic acid. At 10g zeolite, the concentration is decreased.

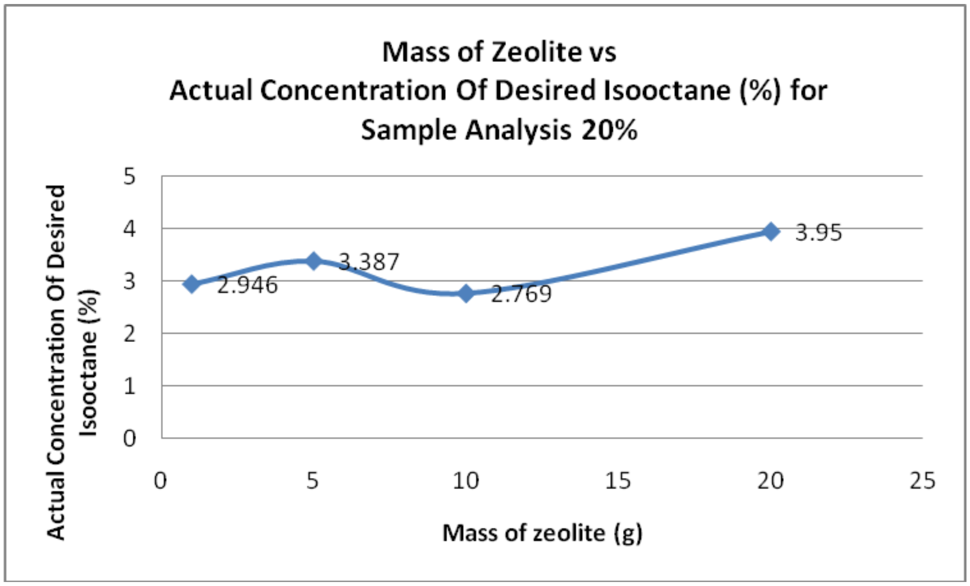


Figure 4.2: Graph of Mass of Zeolite versus Actual Isooctane obtained

4.3.2 Sample Analysis 10%

For the analysis samples 10%, the sample of 10% of stearic acid distillate is diluted with 90% of hexane. Based on the figure 4.3 below, the mass of zeolite do effect the production of isooctane from stearic acid. But the result is opposite to the theory as the more catalyst being used, the less the isooctane produced.

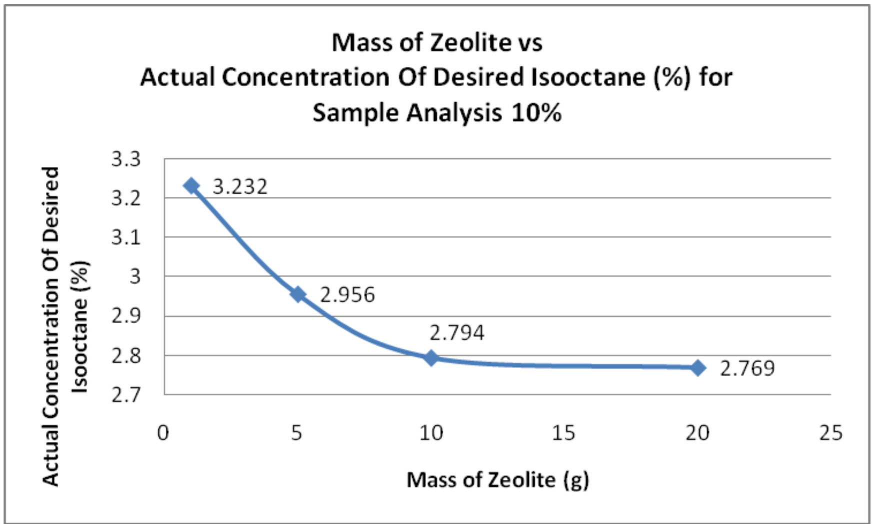


Figure 4.3: Graph of Mass of Zeolite versus Actual Isooctane obtained

4.3.3 Sample Analysis 5%

For the analysis samples 5%, the sample of 5% of stearic acid distillate is diluted with 95% of hexane. Based on the figure 4.4 below, the mass of zeolite do effect the production of isooctane from stearic acid. The result do followed the theory as the more catalyst being used, the more isooctane produced. But for 5g zeolite, the isooctane produced is highest than others.

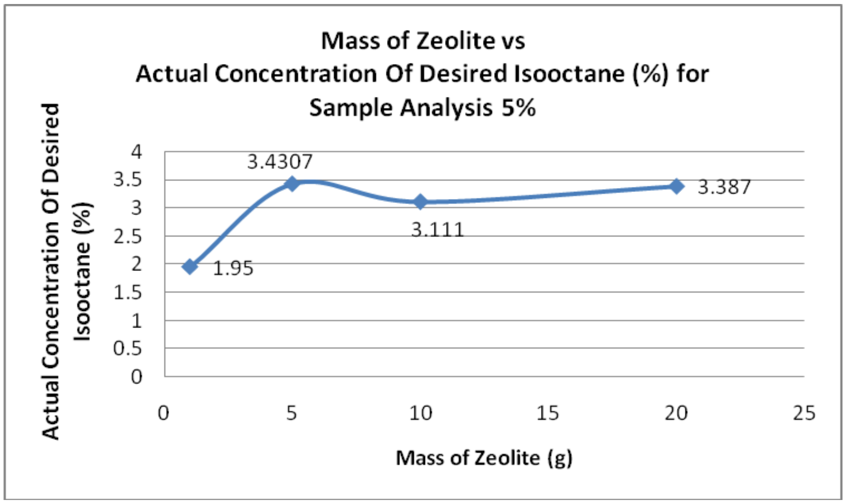


Figure 4.4: Graph of Mass of Zeolite versus Actual Isooctane obtained

4.3.4 Analysis Sample 1%

For the analysis samples 1%, the sample of 1% of stearic acid distillate is diluted with 99% of hexane. Based on the figure 4.5 below, the mass of zeolite do effect the production of isooctane from stearic acid. The result do followed the theory as the more catalyst being used, the more isooctane produced. But for 5g zeolite, the isooctane produced is highest than others.

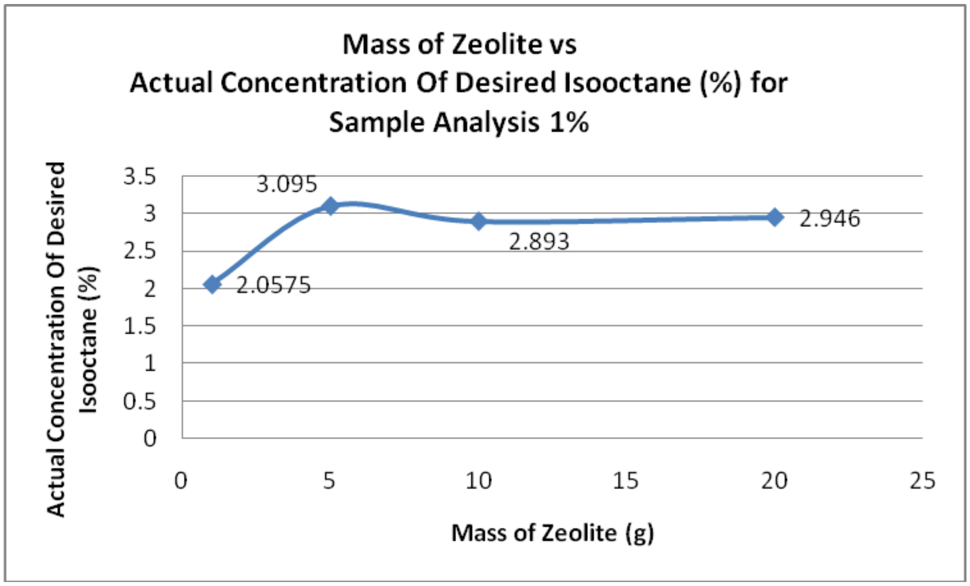


Figure 4.5: Graph of Mass of Zeolite versus Actual Isooctane obtained

4.4 Discussion

In this experiment, the catalytic cracking is used in order to breaks the carbon bonding of the long hydrocarbon molecules and re-order the structure into smaller hydrocarbon molecules. Basic mechanism in catalytic cracking is the long chains of the hydrocarbons are broken into various smaller hydrocarbon radicals by random. Then, the radicals restructure in different arrangement of molecules and become various kinds of compounds through isomerization process. But there are no further research been done to examine the other compounds.

From the Gas Chromatogram result, the percentage of actual concentration of isooctane obtained for 20 g of Zeolite is 3.95%, 10g zeolite is 2.769%, 5g zeolite is 3.387% and 1g Zeolite is 2.946%. The theory is, the more catalyst being used, the more products are obtained. The result shown that, 20 grams of catalyst obtained the highest percentage isooctane of all which is approved the theory made. But, for the next experiment, the percentage is fluctuating. The percentage of isooctane obtained should be decreasing. But though, this shows that the factor of catalyst do effect the efficiency of catalytic cracking.

4.4.1 Dilution by Solvent

In order to use the Gas Chromatogram method, the sample should be diluted with the suitable solvent to be injected. For this case, the sample with isooctane double diluted with Hexane which is the dilution agent which makes the concentration of the samples is so small. The sample also need to be colorless and non-particle liquid as the solid particle can trap inside the Gas Chromatogram column.

In determining the actual concentration of the Isooctane produced, the backward calculation is used. The dilution agent is eliminated as the main assumption. Then, for the next samples, assume that the samples are directly injected into Gas Chromatogram without any dilution process. All the other samples is calculated using the backward calculations, which shown in below. All the data are recorded in Table 4.12.

- **Example of Calculations**

Stearic Acid with 20g Zeolite (20% sample 80% solvent)

Actual peak area isooctane (%) = [peak area isooctane (%)/ [100-peak area hexane (%)]]*100

= [0.417/ (100-85.73)]* 100

= 2.922%

Actual peak area (pA*s) = [peak area isooctane (pA*s)/peak area isooctane (%)]*actual peak area of isooctane (%)

= [5651.3/0.417]*2.922

= 35599.757 pA*s

From the equation shown in standard calibration curve (Figure 4.2):

$$Y=82277X$$

Actual concentration of Isooctane (%) = actual peak area (pA*s)/ 82277

= 35599.757 /82277

= 48.13%

The others calculations are using same method and recorded in table 4.12 below.

Table 4.12: Actual Concentration of all samples

Analysis Samples	**Actual Peak Area (%)**	**Actual Peak Area [pA*s]**	**Actual Concentration Of Desired Isooctane (%)**
Stearic acid with 20 grams Zeolite			
20% Sample	***2.922**	**35599.757**	**3.95**
10% Sample	2.57	29875.26	2.9875
5% Sample	2.446	29688.67	2.968
1% Sample	2.4037	28699.199	2.8699
Stearic Acid with 10 grams Zeolite			
20% Sample	2.446	27690.1214	2.769
10% Sample	2.4168	27945.204	2.794
5% Sample	2.2889	29567.24	2.956
1% Sample	2.4724	32322.2993	3.232
Stearic Acid with 5 grams Zeolite			
20% Sample	2.634	33870.49	3.387
10% Sample	2.7069	31101.402	3.111
5% Sample	2.6789	34307.163	3.4307
1% Sample	1.6335	19516.968	1.95
Stearic Acid with 1 grams Zeolite			
20% Sample	2.7544	29463.578	2.946
10% Sample	2.4912	28933.6707	2.893
5% Sample	2.733	30956.224	3.095
1% Sample	1.7976	20575.356	2.0575

*showed the highest isooctane obtained

4.4.2 Retention Time

In Chromatography term, it is the most commonly used procedure in contemporary chemical analysis and the first configuration of chromatography equipment to be produced in a single composite unit and made commercially available was the gas chromatograph. Retention time is the time between injection and the elution of the solute molecules and those of the mobile phase.

From the analysis sample, the retention time of the isooctane is not constant. As in the table 3 the Isooctane standard shows the retention time for the Isooctane is in average 3.789 min to 4.332 min. So, any peak with large area can be considered as Isooctane. For an instance, there is 1 sample have out range of isooctane. For example, Isooctane of sample of 10 grams Zeolite 20% peak out at 3.759 min. The flow rate of carrier gas would be factors that effecting the retention time of the compounds detected in Gas Chromatogram. This is because the flow rate does not constant, and it is impossible to control the flow rate. It does affected by surroundings, which always changing. So, this gives interference to the gas chromatogram and effect the retention time.

4.3.3 Catalyst Factor

In this research, zeolite is used as catalyst to enhance the production of isooctane from stearic acid. Zeolite is a crystalline aluminosilicates with fully cross-linked open framework structures made up by SiO4 and AlO4 tetrahedral. Zeolite also known as the greatest catalyst of all because of it is highly porosity and has pores and channels so the surface area of the catalyst is high. There will be lots of area to serve the adsorption process. The surface area for Zeolite is unlimited. By using zeolite catalyst, the more product we can get if compared to granular metal catalyst.

In addition, the surface area of the catalyst also factors in enhancing the reaction process. As shown in figure 4.6 below, the size and surface area of zeolite catalyst being used in this experiment is different as the shape itself is different. In a heterogeneous catalyst, many reactions proceed on the surface of catalyst. To increase the catalyst efficiency and enhance the process, it is essential to make the surface area larger. When precious metals like platinum, palladium, and so on are used as catalysts, they are pulverized from 1 to 100nm to be distributed and carried on the porous supports with a pore structure and a large surface area. This enlarges the surface area for reaction, and increases the catalyst activity per unit weight.

Figure 4.6 :Zeolite Catalyst

Therefore, the surface area of zeolite will affect the production of isooctane obtained from the catalytic process. Theoretically, as the more catalyst being added to the process, the more products will be obtained as the catalyst enhancing the reaction. This explains why the production of the isooctane is not constant and do not follow the theory. Besides that, this crystalline structure is characterized by three dimensional pore systems with well defined diameter of pores also contributing to the effectiveness of zeolite. The crystalline structure will give higher surface area to the zeolite, so there is more surface area for the catalytic cracking process.

4.3.4 Contamination Factor

While preparation of samples and standard solutions of hexane-isooctane, there might be contamination occur and contaminate the samples. This is because of by using contaminated glassware and apparatus. So, in order to get constant result, all of the apparatus should be clean with appropriate cleaning detergent, rinse excessively and dried. Even this factor is quite simple, but it does affect the experiment. Especially any experiment that includes Gas Chromatography as a method, need to highlight this situation. This is because, when the contaminated samples being injected into Gas Chromatographer, the contaminated compounds in samples will gives the enormous unknown peak to gas chromatogram.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.0 Conclusions

The basic objectives in this research is to analyze the existence and determine the concentration of the isooctane produced from catalytic cracking of Stearic Acid by using Zeolite as catalyst. From the result obtained, this research could approve that the presence of the catalyst does improve the production of biopetrol octane from catalytic cracking.

Result Obtained from the Gas Chromatogram result, the percentage of actual concentration of isooctane obtained for 20 g Zeolite is 3.95 %, 10g zeolite is 3.323 %, 5g zeolite is 3.4307 % and 1g Zeolite is 3.232 %. The theory is, the more catalyst being used, the more products are obtained. The result shown that, 20 grams of catalyst obtained the highest percentage isooctane of all which is approved the theory made. But, for the next experiment, the percentage is fluctuating. The percentage of isooctane obtained should be decreasing. But though, this shows that the factor of catalyst do effect the efficiency of catalytic cracking.

Based from this research, biopetrol from carboxylic acid does have the potential to be future biofuel with further additional research. This research could altered by applying high temperature would increase the production of isooctane.

5.1 Recommendation

This research is basically to study the effectiveness of catalyst in fundamental cracking hydrocarbons. It is important to improve the analytical and technical method of the experimental work in order to get more isooctane (biopetrol) from stearic acid distillate. The development of real-time techniques such as spectroscopy is useful in confirmation of identification of producing biopetrol from stearic acid and could be alternative fuel in future.

It is recommended to increase the temperature in range 300- 400^{o}C of the catalytic cracking in order to enhance more production of isooctane. This is because most of hydrocracking and fluid catalytic cracking uses high temperature in the processs. It follows from the preliminary experiments of cracking of rapseed oil to obtain hydrocarbon fuel which is sufficiently need high reaction temperature which is above about 310 °C.

In addition, In order to increase the concentration of biopetrol (isooctane) in stearic acid, it is recommended to apply dynamic catalytic cracking method, which means the catalyst should be in dynamic state instead of static state. This is because the dynamic catalytic cracking using certain heterogeneous solid catalysts is more effective and it is already practiced in all petroleum refineries. Additionally, this might help to produce more isooctane rather than using static process.

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APPENDIX A

Material Safety Data Sheet

Safety Data for Stearic Acid

General

Synonyms: 1-heptadecanecarboxylic acid, octadecanoic acid, n-octadecanoic acid,

Molecular formula: $C_{18}H_{36}O_2$

CAS No: 57-11-4

EINECS No: 200-313-4

Physical data

Appearance: shiny white flakes or powder

Melting point: 67 - 69 C

Boiling point: 361 C [183 - 184 C at 1 mm Hg]

Vapour density:

Vapour pressure:

Density (g cm^{-3}): 0.87

Stability

Stable. Combustible. Incompatible with bases, reducing agents, oxidizing agents.

Toxicology

Eye, skin and respiratory irritant

Toxicity data

(Refer appendix C)

IVN-RAT LD50 22 mg kg^{-1}

IVN-MUS LD50 23 mg kg^{-1}

SKN-RBT LD50 > 5000 mg kg^{-1}
ORL-RAT LDLO 4640 mg kg^{-1}

Risk phrases

(The meaning of any risk phrases which appear in this section is given in appendix D)

R36 R37 R38.

Transport information

Non-hazardous for air, sea and road freight.

Personal protection

Safety glasses.

Safety phrases

(The meaning of any safety phrases which appear in this section is given in appendix E)

R36 R37 R38.

Material Safety Data Sheet

Safety Data For Isooctane / 2,2,4-Trimethylpentane

General

Synonyms: isobutyltrimethylpentane, isooctane

Molecular formula: $(CH_3)_3CCH_2CH(CH_3)_2$

CAS No: 540-84-1

EC No: 208-759-1

Annex I Index No: 601-009-00-8

Physical data

Appearance: colourless liquid

Melting point: -107 C

Boiling point: 98 C

Vapour density: 3.9

Vapour pressure: 41 mm Hg at 21 C

Specific gravity: 0.692

Flash point: -7 C (closed cup)

Explosion limits: 1 - 6%

Autoignition temperature: 396 C

Stability

Stable. Highly flammable. Incompatible with oxidizing agents, reducing agents.

Toxicology

Harmful if inhaled or ingested. May be harmful through skin contact. Eye, respiratory and skin irritant.

Risk phrases

(The meaning of any risk phrases which appear in this section is given in appendix D)

R11 R20 R22 R36 R37 R38 R50 R53 R65 R67.

Environmental information

Very toxic to aquatic organisms - may cause long-term damage to the environment.

Transport information

(The meaning of any UN hazard codes which appear in this section is given appendix F)

UN No 1262. Hazard class 3.0. Packing group II.

Personal protection

Safety glasses, good ventilation, keep away from sources of ignition.

Safety phrases

(The meaning of any safety phrases which appear in this section is given appendix F)

S16 S26 S33 S36 S37 S39.

APPENDIX B

Abbreviations used in Toxicity data

dom	domestic animal (goat, sheep)
dpo	Drcsophila pseudo-obscura
emb	embryo
esc	Escherichia cold
eug	Euglena gracilis
eye	administration into eye (irritant)
ice	intracerebral
icv	intracervical
idr	intradermal
idu	intraduodenal
ihl	inhalation
isp	intraspinal
itr	intratracheal
itt	intratesticular
iu	international unit
iut	intrauterine
ivg	intravaginal
ivn	**intravenous**
kdy	kidney
LC50	lethal concentration 50 percent kill
LCLo	lowest published lethal concentration
LD50	**lethal dose 50 percent kill**
LDlo	lowest published lethal dose
leu	leukocyte
Liq	liquid
lng	lung
lvr	liver
MOD	moderate irritation effects
mol	mole
mppcf	million particles per cubic foot
mrc	gene conversion and mitotic recombination
msc	mutation in mammalian somatic cells
mul	multiple routes
mus	**mouse**

orl	**oral**
ORM	Other Regulated Material (DoT)
oth	other cell types
otr	oncogenic transformation
ovr	ovary
par	parenteral
pg	picogram
pgn	pigeon
pic	phage inhibition capacity
pig	pig
Pk	peak concentration
pmol	picomole
post	after birth
ppb	parts per billion (v/v)
pph	parts per hundred (v/v) (percent)
ppm	parts per million (v/v)
ppt	parts per trillion (v/v)
preg	pregnant
qal	quail
rat	**rat**
STEL	short term exposure limit
TC	toxic concentration (other than lowest concentration)
TCLo	lowest published toxic concentration
TD	toxic dose (other than lowest toxic dose)
TDLo	lowest published toxic dose
Y	year

APPENDIX C

Risk Phrases

- R1 Explosive when dry.
- R2 Risk of explosion by shock, friction, fire or other source of ignition.
- R3 Extreme risk of explosion by shock, friction, fire or other sources of ignition.
- R4 Forms very sensitive explosive metallic compounds.
- R5 Heating may cause an explosion.
- R6 Explosive with or without contact with air.
- R7 May cause fire.
- R8 Contact with combustible material may cause fire.
- R9 Explosive when mixed with combustible material.
- R10 Flammable.
- **R11 Highly flammable.**
- R12 Extremely flammable.
- R13 Extremely flammable liquefied gas
- R14 Reacts violently with water.
- R15 Contact with water liberates extremely flammable gases.
- R16 Explosive when mixed with oxidizing substances.
- R17 Spontaneously flammable in air.
- R18 In use, may form inflammable/explosive vapour-air mixture.
- R19 May form explosive peroxides.
- **R20 Harmful by inhalation**.
- R21 Harmful in contact with skin.
- **R22 Harmful if swallowed.**
- R23 Toxic by inhalation.
- R24 Toxic in contact with skin.
- R25 Toxic if swallowed.
- R26 Very toxic by inhalation.
- R27 Very toxic in contact with skin.
- R28 Very toxic if swallowed.
- R29 Contact with water liberates toxic gas.
- R30 Can become highly flammable in use.
- R31 Contact with acids liberates toxic gas.
- R32 Contact with acid liberates very toxic gas.
- R33 Danger of cumulative effects.
- R34 Causes burns.
- R35 Causes severe burns.
- **R36 Irritating to eyes.**
- **R37 Irritating to respiratory system.**
- **R38 Irritating to skin.**
- R39 Danger of very serious irreversible effects.
- R40 Limited evidence of a carcinogenic effect.
- R41 Risk of serious damage to the eyes.

- R42 May cause sensitization by inhalation.
- R43 May cause sensitization by skin contact.
- R44 Risk of explosion if heated under confinement.
- R45 May cause cancer.
- R46 May cause heritable genetic damage.
- R47 May cause birth defects
- R48 Danger of serious damage to health by prolonged exposure.
- R49 May cause cancer by inhalation.
- **R50 Very toxic to aquatic organisms.**
- R51 Toxic to aquatic organisms.
- R52 Harmful to aquatic organisms.
- **R53 May cause long-term adverse effects in the aquatic environment.**
- R54 Toxic to flora.
- R55 Toxic to fauna.
- R56 Toxic to soil organisms.
- R57 Toxic to bees.
- R58 May cause long-term adverse effects in the environment.
- R59 Dangerous to the ozone layer.
- R60 May impair fertility.
- R61 May cause harm to the unborn child.
- R62 Risk of impaired fertility.
- R63 Possible risk of harm to the unborn child.
- R64 May cause harm to breastfed babies.
- **R65 Harmful: may cause lung damage if swallowed.**
- R66 Repeated exposure may cause skin dryness or cracking.
- **R67 Vapours may cause drowsiness and dizziness**.
- R68 Possible risk of irreversible effects.

APPENDIX D

EC Safety Phrases

- S1 Keep locked up.
- S2 Keep out of the reach of children.
- S3 Keep in a cool place.
- S4 Keep away from living quarters.
- S5 Keep contents under ... (there follows the name of a liquid).
- S6 Keep under ... (there follows the name of an inert gas).
- S7 Keep container tightly closed.
- S8 Keep container dry.
- S9 Keep container in a well-ventilated place.
- S12 Do not keep the container sealed.
- S13 Keep away from food, drink and animal foodstuffs.
- S14 Keep away from ... (a list of incompatible materials will follow).
- S15 Keep away from heat.
- **S16 Keep away from sources of ignition.**
- S17 Keep away from combustible material.
- S18 Handle and open container with care.
- S20 When using, do not eat or drink.
- S21 When using do not smoke.
- S22 Do not breathes dust.
- S23 Do not breathes vapor.
- S24 Avoid contact with skin.
- S25 Avoid contact with eyes.
- **S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.**
- S27 Take off immediately all contaminated clothing.
- S28 After contact with skin, wash immediately with plenty of soap-suds.
- S29 Do not empty into drains.
- S30 Never add water to this product.
- **S33 Take precautionary measures against static discharges.**
- S35 This material and its container must be disposed of in a safe way.
- **S36 Wear suitable protective clothing.**
- **S37 Wear suitable gloves**.
- S38 In case of insufficient ventilation, wear suitable respiratory equipment.
- **S39 Wear eye / face protection.**
- S40 To clean the floor and all objects contaminated by this material, use .... (there follows suitable cleaning material).

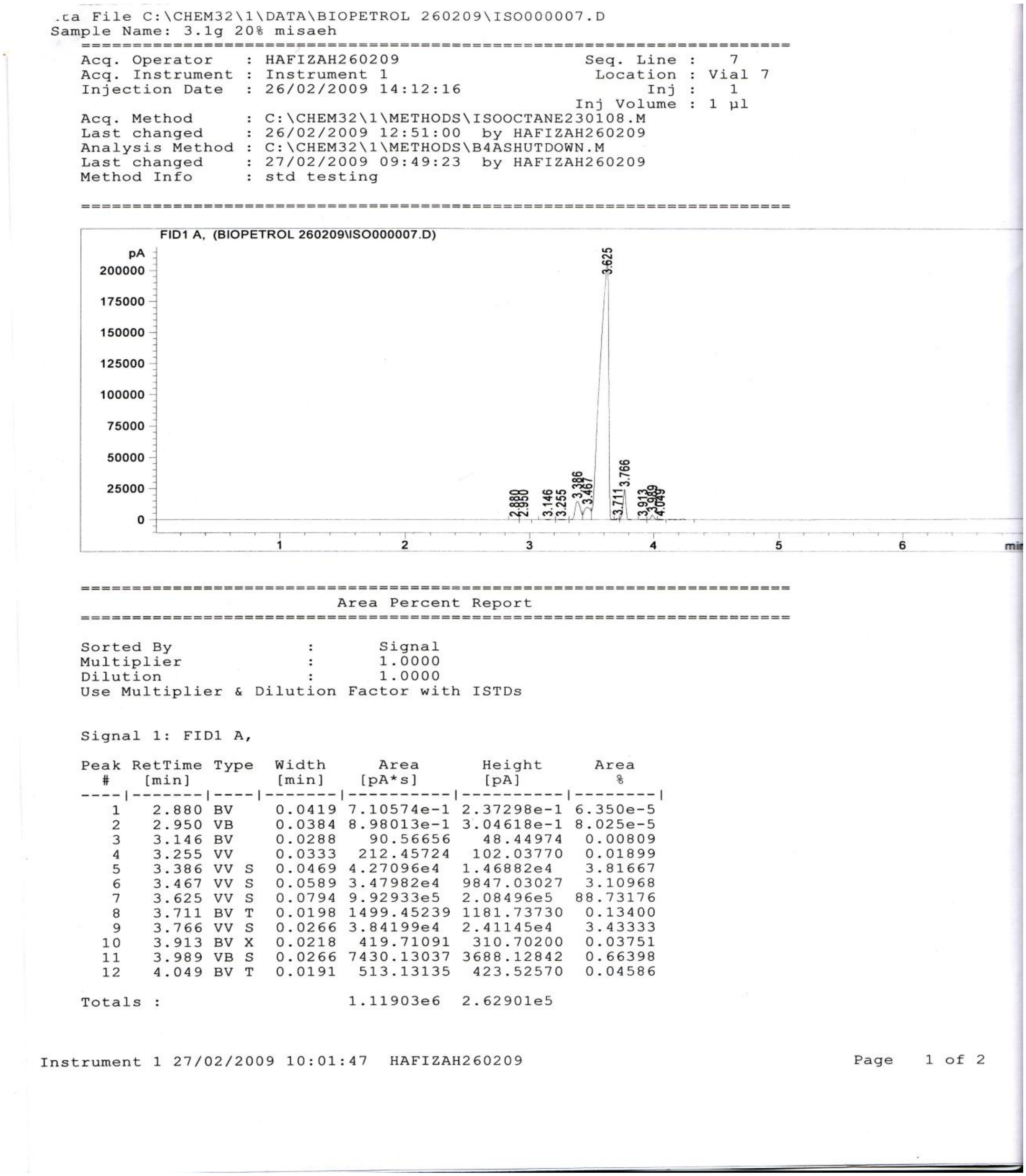
APPENDIX E

UN Hazard Codes

- Class 1 Explosive
    - 1.1 Substances with a mass explosion hazard
    - 1.2 Substances which present a projection hazard but no mass explosion hazard
    - 1.3 Substances which present both a fire hazard and a minor blast or projection hazard (or both) but not a mass explosion hazard
    - 1.4 No significant hazard
    - 1.5 Very insensitive substances with a mass explosion hazard
    - 1.6 Very insensitive articles with no mass explosion hazard
- Class 2 Gases
    - 2.1 Flammable gases
    - 2.2 Non-flammable, non-toxic gases
    - 2.3 Toxic gases
- **Class 3 Flammable liquids**
- Class 4 Flammable solids
    - 4.1 Flammable solids, self-reactive substances and solid desensitized explosives
    - 4.2 Materials liable to spontaneous combustion
    - 4.3 Substances which, in contact with water, release flammable gases
- Class 5. Oxidizing substances and organic peroxides
    - 5.1 Oxidizing agents
    - 5.2 Organic peroxides
- Class 6 Toxic and infectious substances
    - 6.1 Toxic substances
    - 6.2 Infectious substances
- Class 7 Radioactive substances and articles
- Class 8 Corrosive substances
- Class 9 Miscellaneous dangerous substances

APPENDIX F

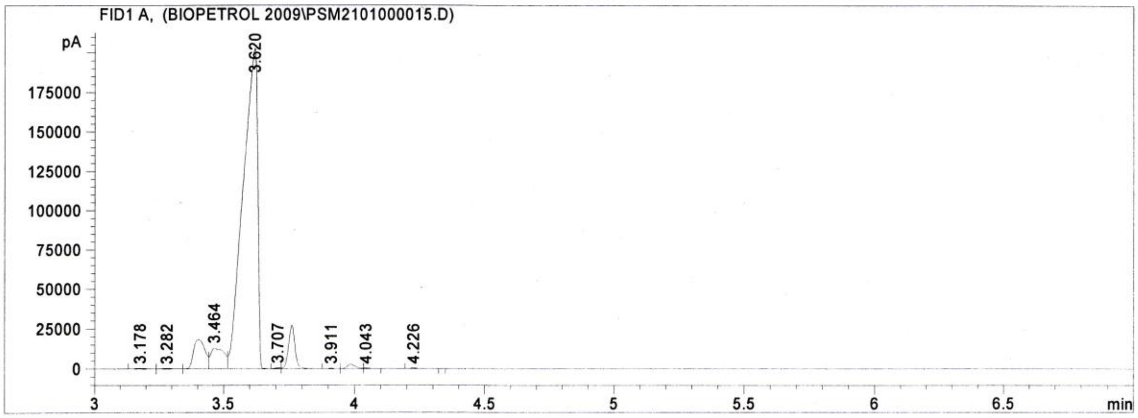
Result Chromatogram



```
.ta File C:\CHEM32\1\DATA\BIOPETROL 260209\ISO000007.D
Sample Name: 3.1g 20% misaeh
   =====================================================================
   Acq. Operator   : HAFIZAH260209                     Seq. Line :    7
   Acq. Instrument : Instrument 1                       Location : Vial 7
   Injection Date  : 26/02/2009 14:12:16                     Inj :    1
                                                      Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 26/02/2009 12:51:00  by HAFIZAH260209
   Analysis Method : C:\CHEM32\1\METHODS\B4ASHUTDOWN.M
   Last changed    : 27/02/2009 09:49:23  by HAFIZAH260209
   Method Info     : std testing

   =====================================================================
```

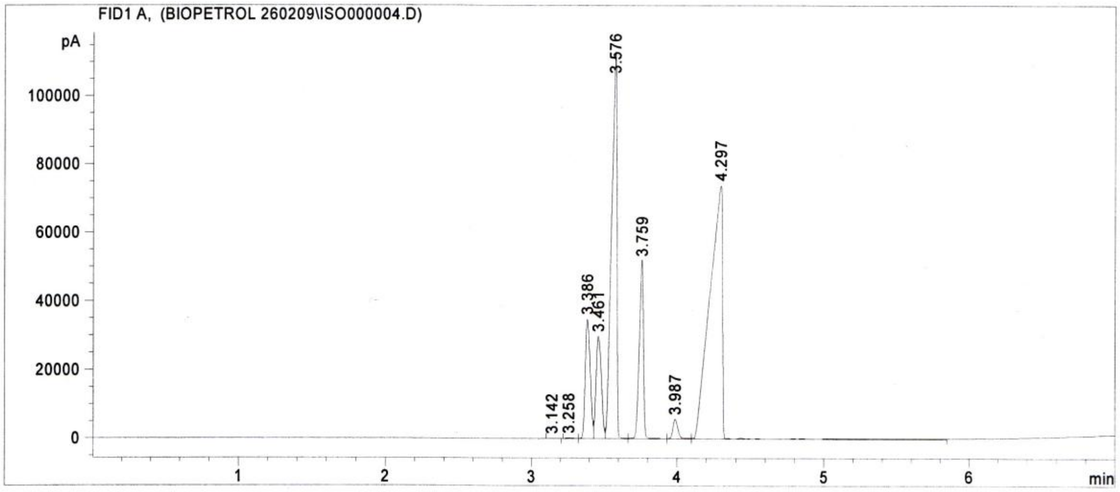
```
   =====================================================================
                         Area Percent Report
   =====================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]        %
   ----|-------|----|-------|----------|----------|--------|
      1   2.880 BV    0.0419 7.10574e-1 2.37298e-1 6.350e-5
      2   2.950 VB    0.0384 8.98013e-1 3.04618e-1 8.025e-5
      3   3.146 BV    0.0288   90.56656   48.44974  0.00809
      4   3.255 VV    0.0333  212.45724  102.03770  0.01899
      5   3.386 VV S  0.0469 4.27096e4  1.46882e4   3.81667
      6   3.467 VV S  0.0589 3.47982e4  9847.03027  3.10968
      7   3.625 VV S  0.0794 9.92933e5  2.08496e5  88.73176
      8   3.711 BV T  0.0198 1499.45239 1181.73730  0.13400
      9   3.766 VV S  0.0266 3.84199e4  2.41145e4   3.43333
     10   3.913 BV X  0.0218  419.71091  310.70200  0.03751
     11   3.989 VB S  0.0266 7430.13037 3688.12842  0.66398
     12   4.049 BV T  0.0191  513.13135  423.52570  0.04586

   Totals :                  1.11903e6  2.62901e5


Instrument 1 27/02/2009 10:01:47  HAFIZAH260209                      Page    1 of 2
```

```
Data File C:\CHEM32\1\DATA\BIOPETROL 2009\PSM2101000015.D
Sample Name: 3 5g 20% misaeh
    =====================================================================
    Acq. Operator   : hafizah 210109                    Seq. Line :   3
    Acq. Instrument : Instrument 1                       Location : Vial 15
    Injection Date  : 21/01/2009 14:59:39                     Inj :   1
                                                       Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 21/01/2009 14:24:03  by hafizah 210109
    Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 23/01/2009 16:11:51  by hafizah 230109
                      (modified after loading)
    Method Info     : isooctane/psm

    =====================================================================
```



```
    =====================================================================
                             Area Percent Report
    =====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [pA*s]      [pA]        %
    ----|-------|----|-------|----------|----------|--------|
       1   3.178 VV    0.0366  606.07294  260.82706  0.06412
       2   3.282 VV    0.0405  362.11026  140.63789  0.03831
       3   3.402 VV S  0.0493 5.58135e4  1.81728e4   5.90505
       4   3.464 VV S  0.0500 4.68888e4  1.30273e4   4.96081
       5   3.620 VV S  0.0649 7.88762e5  2.02494e5  83.45066
       6   3.707 BV T  0.0162  328.29959  341.12045  0.03473
       7   3.761 VB S  0.0263 4.60992e4  2.72076e4   4.87728
       8   3.911 BV X  0.0268  181.24968  104.14397  0.01918
       9   3.986 VV X  0.0302 5606.31348 2763.32349  0.59315
      10   4.043 VV X  0.0243  266.03668  161.07217  0.02815
      11   4.226 VB X  0.0260  270.05179  153.74348  0.02857

    Totals :                  9.45183e5  2.64826e5


    =====================================================================
                             Summed Peaks Report
    =====================================================================
```

```
:\CHEM32\1\DATA\BIOPETROL 260209\ISO000004.D
ame: iso 30%
=====================================================================
Acq. Operator   : HAFIZAH260209                     Seq. Line :   4
Acq. Instrument : Instrument 1                       Location : Vial 4
Injection Date  : 26/02/2009 13:32:16                     Inj :   1
                                                   Inj Volume : 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed    : 26/02/2009 12:51:00  by HAFIZAH260209
Analysis Method : C:\CHEM32\1\METHODS\B4ASHUTDOWN.M
Last changed    : 27/02/2009 09:49:23  by HAFIZAH260209
Method Info     : std testing

=====================================================================
```



```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   3.142 BB    0.0337    3.89724    1.83981  0.00039
   2   3.258 BV    0.0327  263.08887  129.44440  0.02637
   3   3.386 VV S  0.0388 8.28349e4  3.47470e4   8.30173
   4   3.461 VV S  0.0428 7.61389e4  2.96787e4   7.63066
   5   3.576 VV S  0.0421 2.81371e5  1.11511e5  28.19909✓ iso
   6   3.759 VV S  0.0310 1.01719e5  5.16230e4  10.19429✓ hex
   7   3.987 VV S  0.0353 1.27704e4  5663.29785  1.27985
   8   4.297 VBAS  0.0768 4.42701e5  7.39039e4  44.36762

Totals :                  9.97802e5  3.07258e5


=====================================================================
                          *** End of Report ***
```