

**BIOPETROL SYNTHESIZED FROM STEARICACID – HETEROGENOUS
CATALYTIC CRACKING BY ZEOLITE**

MOHAMAD NAQUIB FITRI BIN MOHD NADZRI

UNIVERSITI MALAYSIA PAHANG

UNIVERSITI MALAYSIA PAHANG

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JUDUL : **BIOPETROL SYNTHESIZED FROM STEARIC ACID -
HETEROGENEOUS CATALYTIC CRACKING BY
ZEOLITE**

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Signature :
Supervisor : Syaiful Nizam b. Hassan
Date :

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CATALYTIC CRACKING BY ZEOLITE**

MOHAMAD NAQUIB FITRI BIN MOHD NADZRI

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

**Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang**

May 2010

I declare that this thesis entitled “Biopetrol Synthesized from Stearic Acid – Heterogenous Catalytic Cracking By Zeolite” is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.”

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Name : Mohamad Naquib Fitri Bin Mohd Nadzri

Date : 30 April 2010

*Special Dedication to my parents& family members,
my friends, my fellow colleague
and all faculty members*

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ABSTRACT

Biopetrol is the alternative way to reduce the application of using petroleum where it is a new technology that more environmental friendly and furthermore it is the latest finding in research after biodiesel, bio-ethanol and bio-butanol. Biofuel is defined as fuel produced from derivation of vegetable oils and specifically, biopetrol is defined as fuel which has the same characteristic with the petrol, but is produced from stearic acid that is contained in palm oil where the conversion of stearic acid is done to get the molecular formula and structure of isooctane. Due to reduction of fossil fuel, environmental issues, and rising of petrol price, biopetrol can be the alternative fuel to the fossil fuel. In this research, stearic acid are used as a raw material, catalytic cracking are method that used together with zeolite as a catalyst. The experiment starts with heating the Stearic Acid until it melts at 69.6°C , and then 10 grams of zeolite being added and the heating process proceed until the temperature reached 98°C as the isooctane produced at level 1. The next experiment is repeated with different level of speed at level 2, 3 and 4. All the product samples are analyzed with Gas Chromatographer (GC). The isooctane concentrations are increased when the speed of stirring are increased, but not obviously. From the result obtained from the Gas Chromatogram result, the percentage of actual concentration of isooctane obtained for level 1 is 5.873%, level 2 is 5.921%, level 3 is 5.886% and level 4 is 5.873%. These yields are much higher than the yields produced from normal catalytic cracking method which is from 3% to 6 %. It showed that the heterogeneous catalytic cracking method will produce much higher yields for biopetrol synthesized compared to normal catalytic cracking method. This experiment should be conducted in more proper dynamic method to optimize the yields.

ABSTRAK

Biopetrol adalah salah satu langkah alternatif bagi mengurangkan penggunaan minyak petroleum dari pelbagai aspek dan ia juga merupakan teknologi yang mesra alam selepas menemukan bio-diesel, bio-ethanol dan bio-butanol. Biofuel didefinisikan sebagai bahan api yang dihasilkan daripada terbitan oleh minyak sayuran. Secara spesifiknya biopetrol didefinisikan sebagai bahan api yang mempunyai sifat yang sama dengan petrol tetapi ia dihasilkan daripada asid palmitik di mana komposisinya banyak terdapat pada minyak sawit. Oleh kerana sumber bahan api fosil yang semakin kurang, isu alam sekitar, kenaikan harga petrol, biopetrol boleh menjadi bahan api alternatif kepada bahan api fosil. Dalam kajian ini, asid oleic digunakan sebagai bahan mentah, teknik penguraian dengan pemangkin digunakan serta ketulan kuprum sebagai pemangkin. Experimen di mulakan dengan memanaskan Asid Stearik sehingga menjadi cecair pada suhu 69.6°C dan seterusnya ditambah dengan zeolite dan penasan diteruskan sehingga suhu mencecah 98°C di mana isooktana yang sudah terbentuk sambil dikacau pada kelajuan pertama. Experimen diulang dengan menggunakan kelajuan putaran pada tahap 2, 3 dan 4. Semua sampel produk dianalisis dengan menggunakan *Gas Chromatographer*. Kepekatan isooktana yang dikehendaki akan bertambah apabila kelajuan putaran bertambah, tetapi tidak terlalu jelas. Daripada keputusan Gas Chromatogram yang diperolehi, peratusan kepekatan isooctana untuk kelajuan pertama adalah 5.873%, kelajuan kedua adalah 5.921%, ketiga 5.886% dan kelajuan keempat adalah 5.872%. Penghasilan ini adalah lebih tinggi daripada hasil yang diperolehi daripada penghuraian haba berkatalis biasa iaitu dari 3% kepada 6%. Ini menunjukkan kaedah penghuraian berkatalis dinamik adalah lebih baik. Eksperimen ini patut dijalankan dalam keadaan dinamik dengan cara yang lebih baik bagi mendapatkan hasil yang lebih optimum.

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CHAPTER I

INTRODUCTION

1.1 Introduction

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. Biopetrol is one kind of biofuel defined as fuel which has the same characteristics with commercial petrol in terms of its molecular formula.

Biofuel is suitable for vehicle fuel and had already been tested. Biofuel produced from natural vegetable oil or fats can be used as transportation fuel or fuel additive in the vehicles to reduce their emissions. Plant oils are attracting increased attention in this respect (Bhatia *et al.*, 2003).

Biopetrol is one of biofuel and suitable for petrol vehicle consumer. During the future century, demand of petrol will rises due to increasing of vehicle consumer. Thus, something new need to be done to make sure fuel problem will not rise.

1.2 Identification of Problem

1.2.1 Petrol Oil Prices

Nowadays, the fuel price in Malaysia still high and keep increasing from day to day, but actually the global oil price is cheaper than the price in our country. This situation happens because of the crisis of world economy today. Refer to figure 1, although the crude oil price now decreasing but the trend kept increase from year to year.

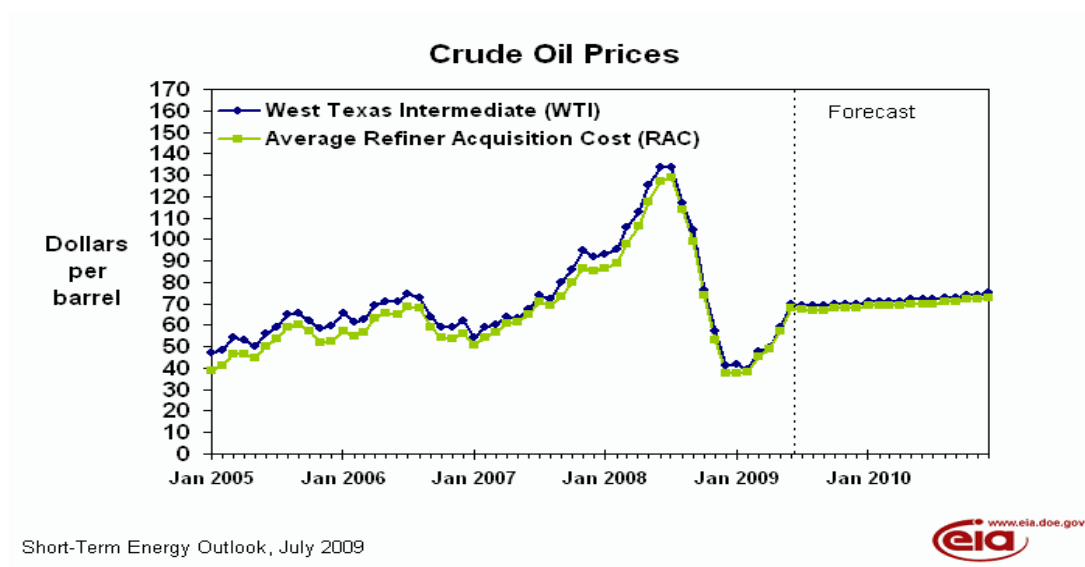


Figure 1.1: Price of World Crude Oil from January 1995 until now.

1.2.2 Air Pollution and Health

Driving a car is the most polluting act an average citizen commits. Combustion engines contribute to greenhouse gas accumulation in the atmosphere and are responsible for climate changes. Thus, long term solutions require that vehicles use less polluting energy sources such as biofuels. Biofuels burned in diesel engines have a better environmental profile. Negative environmental consequences of fossil fuels and

concerns about petroleum supplies have spurred the search for renewable transportation biofuels (Hill et al).

1.3 Objectives

Objective can be defined as guidance to make sure that the problem is solve with an appropriate solution. The objectives for this proposal are:

- i) To improve the concentration of isooctane produced from stearic acid by using modified catalytic cracking method.
- ii) To compare the yields of isooctane produced using the modified catalytic cracking method.

1.4 Scopes of Study

To make sure the objectives succeed, this research is focusing on the criteria that are stated as below:-

- i) To describe the molecular arrangements of the substances in cracking
- ii) To understand the catalytic cracking and distillation process.
- iii) To apply the dynamism of catalytic cracking process.
- iv) To determine the yield of desired isooctane component in the product through Gas Chromatography analysis.

1.5 Rationale and Significance

There are many advantages of biofuel which are:

- i) Create a new development technology in biopetrol where it can apply for petrol-used vehicle engines.
- ii) Biopetrol is an environmentally friendly alternative liquid fuel for current petrol.
- iii) The CO₂ reduction potential of biofuels is enormous in comparison with fossil fuels.
- iv) Biofuels are biodegradable

CHAPTER II

LITERATURE REVIEW

2.0 Definition of fuel

Fuel (from Old French *feuaille*, the combination phrase from *feu* (fire; ultimately from Latin focus fireplace, hearth) is a substance that may be burned in air (or any other oxidant-containing substance), i.e. that so quickly reacts with oxygen that heat and light is emitted in the form of a sustained flame. Usually 'fuel' and only refer to easily flammable substances in air (the air is the oxidiser needed by a fuel to burn, and it is needed in larger quantities than fuels, so, a first glance on it seems appropriate). . Fuels are used as convenient energy stores because of their high specific energy release when burnt with omnipresent ambient air.

By referring the World Encyclopedia (2008), the term fuel refers to substance that is burned or otherwise modified to produce energy, usually in the form of heat. Apart from fossil hydrocarbons (coal, oil and gas) and firewood and charcoal, the term also applies to radioactive materials used in nuclear power stations.

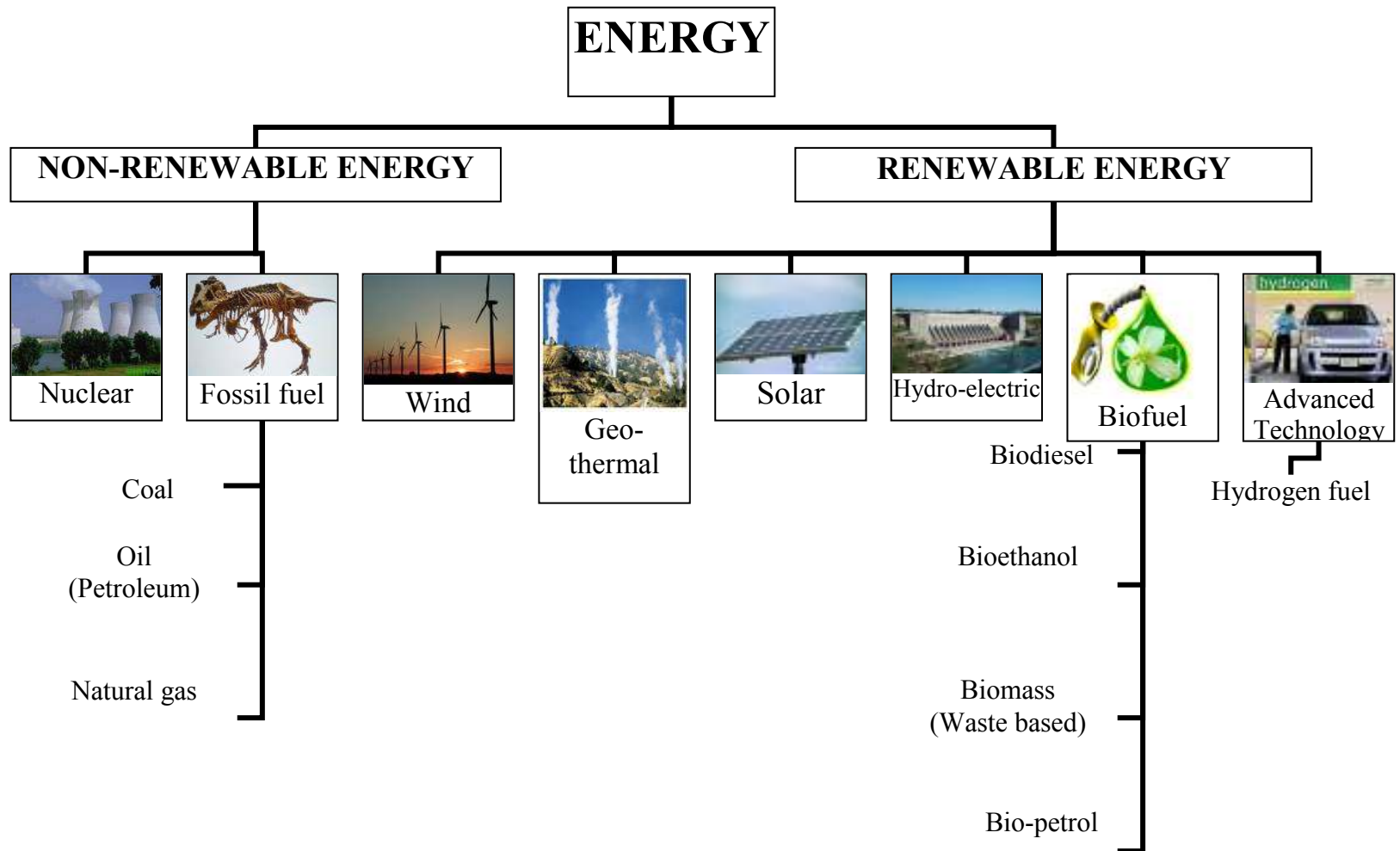


Figure 2.1 Energy's Classification

2.1 Biofuel

Biofuel is defined as liquid or gaseous fuel that can be produced from the utilization of biomass substrates and can be serving as a (partial) substitute for fossil fuels or can be broadly defined as solid, liquid, or gas fuel derived from recently dead material. 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 and biopetrol.

2.1.1 Types of Biofuel

2.1.1.1 Biodiesel

Biodiesel is a natural and renewable domestic fuel alternative for diesel engines made from vegetable oils, mostly soy and corn. It contains no petroleum, is nontoxic and biodegradable. It is made using an alcohol like methanol and a chemical process that separates glycerine and methyl esters (biodiesel) from fats or vegetable oils.

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

2.1.1.2 Bioethanol

Bioethanol fuel is mainly produced by the sugar fermentation process, although it can also be manufactured by the chemical process of reacting ethylene with steam. Bioethanol which is also known as cellulosic ethanol or lignocellulosic or chee is not

made from oils and fats but from lignocellulose, a structure material that consist mostly plant feedstock. Production of bioethanol used sources of sugar that come from fuel or energy crops.

Ethanol or ethyl alcohol is a clear colourless liquid, it is biodegradable, low in toxicity and cause little environmental pollution if split. Ethanol burns to produce carbon dioxide and water. It has high octane fuel and replaced lead as an octane enhancer in petrol. The most common blend is 10% ethanol and 90% petrol (E10). Blending ethanol with gasoline would oxygenated the fuel mixture and the result it would burns more completely and reducing pollution of emissions.

2.1.1.3 Biomass

The term "biomass" refers to any form of plant or animal tissue. In the energy industry, biomass refers to wood, straw, biological waste products such as manure, and other natural materials that contain stored energy. The energy stored in biomass can be released by burning the material directly, or by feeding it to micro-organisms that use it to make biogas, a form of natural gas. Energy from biomass is still used around the world, for everything from cooking and heating to generating electricity.

Using biomass can help reduce global warming compared to a fossil fuel-powered plant. Plants use and store carbon dioxide (CO₂) when they grow. CO₂ stored in the plant is released when the plant material is burned or decays. By replanting the crops, the new plants can use the CO₂ produced by the burned plants. So using biomass and replanting helps close the carbon dioxide cycle. However, if the crops are not replanted, then biomass can emit carbon dioxide that will contribute toward global warming.

2.1.1.4 Biopetrol

Biopetrol is defined as fuel which has the same characteristics with commercial petrol in terms of its molecular formula. 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 ($CH_3C(CH_3)_2CH_2CH(CH_3)CH_3$) burns smoothly with a little knock in petrol engine. It is the highest quality of petrol (Mansur, 2005:1).

Biopetrol from palm oil is biodegradable and non-toxic since from the environmental point of view, fuel from vegetable sources is environmentally friendly. It is able to suppress certain pollutants that come up from the exhaust, with the exception of NO_x in certain cases, where unpredictable results occurred. From the technical point of view it can be said that biofuel (biopetrol) is technically compatible with the current internal combustion engine. Slight modification might be required to enhance the power. Biopetrol could be an answer to the future air emission control. The application of existing biodiesel from palm oil in motor vehicle has been proven to be successful (Prateepchaikul and Apichato, 2003).

2.2 Refining of Petroleum

Petroleum is a complex mixture of organic liquids called crude oil and natural gas, which occurs naturally in the ground and was formed millions of years ago. Crude oil varies from oilfield to oilfield in colour and composition, from a pale yellow low viscosity liquid to heavy black 'treacle' consistencies.

An oil refinery is an organised and coordinated arrangement of manufacturing processes designed to produce physical and chemical changes in crude oil to convert it into everyday products like petrol, diesel, lubricating oil, fuel oil and bitumen.

2.2.1 The refining process

The refinery begins with the separation of crude oil into different fractions by distillation. The fractions are further treated to convert them into mixtures of more useful saleable products by various methods such as cracking, reforming, alkylation, polymerisation and isomerisation. These mixtures of new compounds are then separated using methods such as fractionation and solvent extraction. Impurities are removed by various methods, e.g. dehydration, desalting, sulphur removal and hydrotreating.

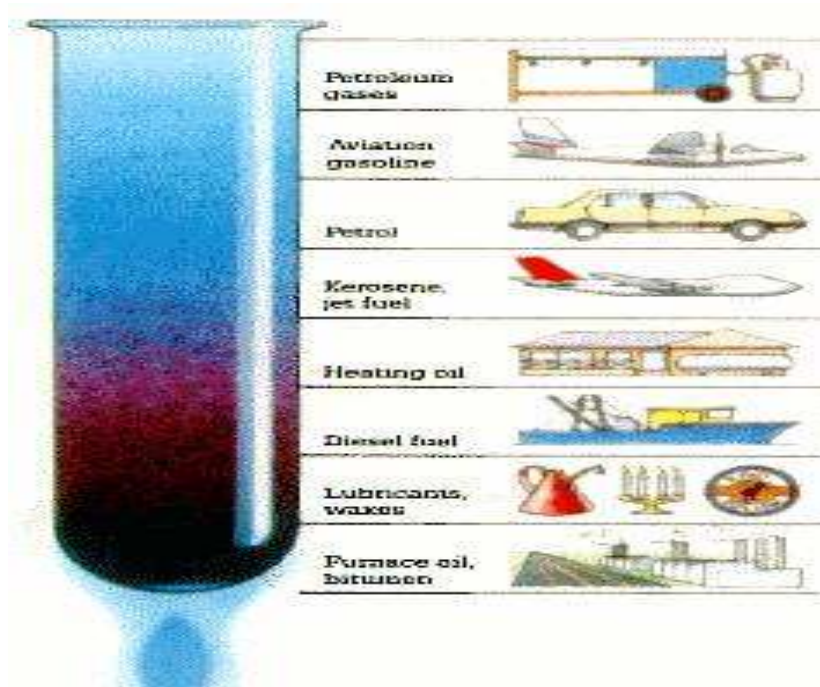


Figure 2.2. Cracking of Crude Oil

2.2.2 Types of Cracking

Cracking processes break down heavier hydrocarbon molecules (high boiling point oils) into lighter products such as petrol and diesel. These processes include catalytic cracking, thermal cracking and hydrocracking.

2.2.2.1 Catalytic Cracking

Catalytic cracking is used to convert heavy hydrocarbon fractions obtained by vacuum distillation into a mixture of more useful products such as petrol and light fuel oil. In this process, the feedstock undergoes a chemical breakdown, under controlled heat (450 - 500°C) and pressure, in the presence of a catalyst - a substance which promotes the reaction without itself being chemically changed. Small pellets of silica - alumina or silica - magnesia have proved to be the most effective catalysts.

The cracking reaction yields petrol, LPG, unsaturated olefin compounds, cracked gas oils, a liquid residue called cycle oil, light gases and a solid coke residue. Cycle oil is recycled to cause further breakdown and the coke, which forms a layer on the catalyst, is removed by burning. The other products are passed through a fractionator to be separated and separately processed.

2.2.2.2 Fluid Catalytic Cracking

Fluid catalytic cracking uses a catalyst in the form of a very fine powder which flows like a liquid when agitated by steam, air or vapour. Feedstock entering the process immediately meets a stream of very hot catalyst and vaporises. The resulting vapours keep the catalyst fluidised as it passes into the reactor, where the cracking takes place and where it is fluidised by the hydrocarbon vapour.

The catalyst next passes to a steam stripping section where most of the volatile hydrocarbons are removed. It then passes to a regenerator vessel where it is fluidised by a mixture of air and the products of combustion which are produced as the coke on the catalyst is burnt off. The catalyst then flows back to the reactor. The catalyst thus undergoes a continuous circulation between the reactor, stripper and regenerator sections.

The catalyst is usually a mixture of aluminium oxide and silica. Most recently, the introduction of synthetic zeolite catalysts has allowed much shorter reaction times and improved yields and octane numbers of the cracked gasolines.

2.2.2.3 Thermal Cracking

Thermal cracking uses heat to break down the residue from vacuum distillation. The lighter elements produced from this process can be made into distillate fuels and petrol. Cracked gases are converted to petrol blending components by alkylation or polymerisation. Naphtha is upgraded to high quality petrol by reforming. Gas oil can be used as diesel fuel or can be converted to petrol by hydrocracking. The heavy residue is converted into residual oil or coke which is used in the manufacture of electrodes, graphite and carbides.

2.2.2.4 Hydrocracking

Hydrocracking can increase the yield of petrol components, as well as being used to produce light distillates. It produces no residues, only light oils. Hydrocracking is catalytic cracking in the presence of hydrogen. The extra hydrogen saturates, or hydrogenates, the chemical bonds of the cracked hydrocarbons and creates isomers with the desired characteristics. Hydrocracking is also a treating process, because the hydrogen combines with contaminants such as sulphur and nitrogen, allowing them to be removed.

Gas oil feed is mixed with hydrogen, heated, and sent to a reactor vessel with a fixed bed catalyst, where cracking and hydrogenation take place. Products are sent to a fractionator to be separated. The hydrogen is recycled. Residue from this reaction is mixed again with hydrogen, reheated, and sent to a second reactor for further cracking

under higher temperatures and pressures. In addition to cracked naphtha for making petrol, hydrocracking yields light gases useful for refinery fuel, or alkylation as well as components for high quality fuel oils, lube oils and petrochemical feedstocks.

Following the cracking processes it is necessary to build or rearrange some of the lighter hydrocarbon molecules into high quality petrol or jet fuel blending components or into petrochemicals. The former can be achieved by several chemical process such as alkylation and isomerisation.

2.3 Zeolite as Catalyst

Zeolites occur in nature and have been known for almost 250 years as aluminosilicate minerals. Examples are faujasite, mordenite, offretite, ferrierite, erionite and chabazite. Today, these and other zeolite structures are of great interest in catalysis, yet their naturally occurring forms are of limited value, because (i) they almost always contain undesired impurity phases, (ii) their chemical composition varies from one deposit to another and even from one stratum to another in the same deposit, and (iii) nature did not optimize their properties for catalytic applications.

2.3.1 Structure of Zeolite

The elementary building units of zeolites are SiO_4 and AlO_4 tetrahedra. Adjacent tetrahedra are linked at their corners via the joints among oxygen atoms in each tetrahedron, and these linkages result within an inorganic macromolecule with a structurally distinct three-dimensional framework. The evidence from this building principle that the net formulae of the tetrahedra are SiO_2 and AlO_2^- , i.e. one negative charge resides at each tetrahedron in the framework which has an aluminum atom in the center of the entire tetrahedron. The framework of a zeolite contains channels, channel

intersections and/or cages with dimensions from 0.2 to 1 nm of the channel's diameter. Inside these voids are water molecules and small cations which compensate the negative framework charge.

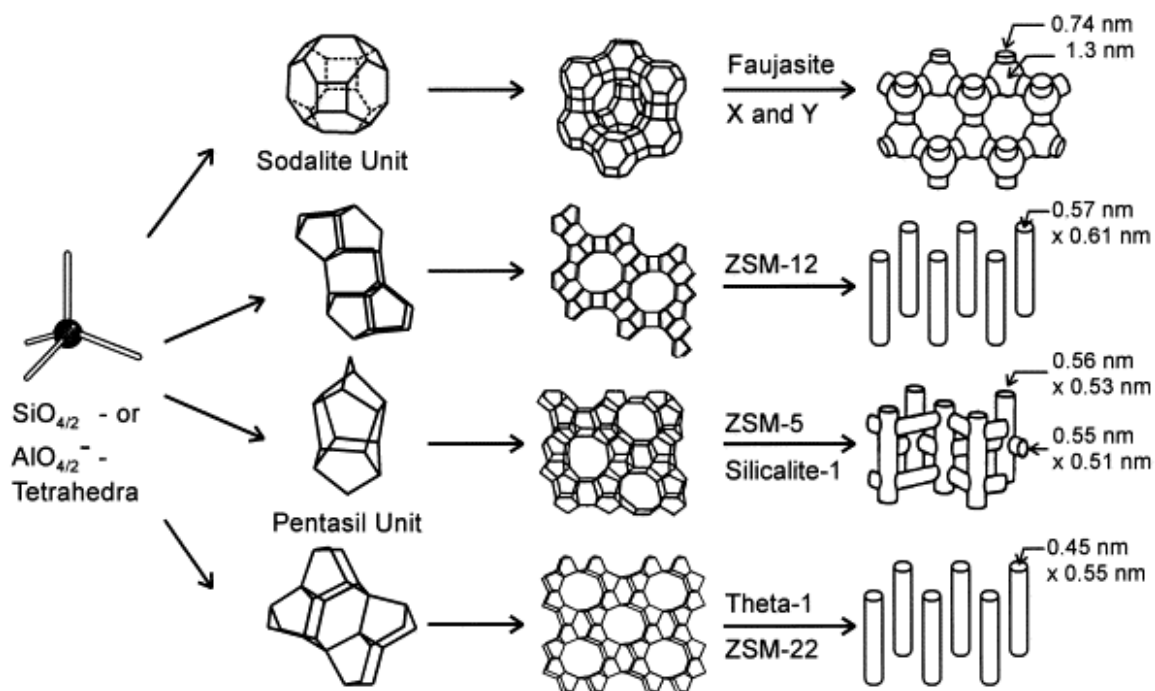
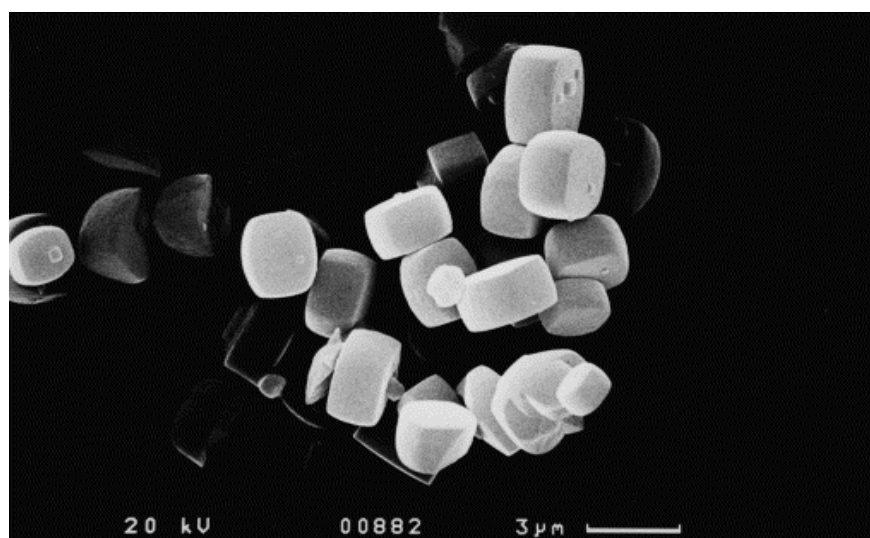


Figure 2.3. Structures of four selected zeolites (from top to bottom: faujasite or zeolites X, Y; zeolite ZSM-12; zeolite ZSM-5 or silicalite-1; zeolite Theta-1 or ZSM-22) and their micropore systems and dimensions.

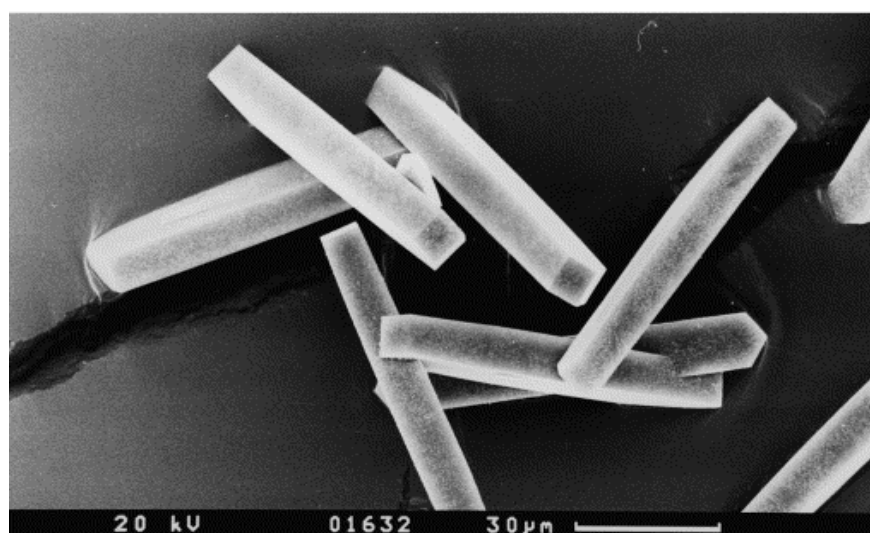
2.3.2 Crystal Size

The size of zeolite crystals is often in the order of one to several micrometers. A typical example is depicted in Fig. 4a which shows tablets of zeolite ZSM-5 with dimensions of 1 to 3 μm . Some zeolites which are relevant to catalysis can, however, be synthesized in very small crystals with a size down to ca. 5 nm (such small crystals are X-ray-amorphous (P.A. Jacobs, E.G. Derouane, J. Weitkamp, J. Chem. Soc. Chem. Comm. (1981) 591) or in very large crystals up to ca. 100 μm or even 1 mm (Springer

Verlag, Berlin, Heidelberg, New York (1998)). As an example, large crystals of zeolite ZSM-5 are shown in Fig. 4b. For catalytic applications, both a decreased and an increased crystal size can be desirable: Upon decreasing the crystal size, the diffusional paths of the reactant and product molecules inside the pores become shorter, and this can result in a reduction or elimination of undesired diffusional limitations of the reaction rate.



(a)



(b)

Figure 2.4 : Scanning electron micrographs showing crystals of zeolite ZSM-5. (a, top): Tablets of ca. $2 \times 2 \times 1 \mu\text{m}$; (b, bottom): Bars of ca. $80 \times 10 \times 10 \mu\text{m}$.

2.4 Chemicals

2.4.1 Isooctane

2,2,4-Trimethylpentane, also known as isooctane, is an octane isomer which defines the 100 point on the octane rating scale. It is an important component of gasoline. Isooctane is targeted as product of biopetrol because petrol itself is dominated by isooctane, with small amount of heptane and a little presence of benzene. Isooctane is derived through isomerization of octane with certain conditions and the presence of catalyst, as performed in petroleum industries. The octane number used as petrol production's parameter to measure the tendency of petrol to auto-ignite and knock in petrol-used engines.

Table 2.1: Physical and chemical properties of isooctane

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 $^{\circ}C$	-107.38 $^{\circ}C$
Boiling point $^{\circ}C$	99.3 $^{\circ}C$
Density	0.688 g/ml
Specific gravity	0.692
Solubility in water	Immiscible

Since the petrol consists mixture of isooctane and heptane, so the octane number is graded based on composition of both alkanes in petrol. The higher octane number determined represents the higher composition of isooctane in petrol, which gives low tendency to auto-ignite, little knock and smooth burning, which is assigned as petrol with high quality.

2.4.2 Stearic Acid



Figure 2.5: 3-D molecular structure 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.

Stearic acid is in fatty acid group, which is a carboxylic acid with long carbon chain. 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 $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$. 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.



Figure 2.6: molecular structure of stearic acid

The fatty tissues of animals contain large amounts of long-chain saturated fatty acids, such as eicosanoic acid ($\text{CH}_3(\text{CH}_2)_{18}\text{COOH}$), octadecanedioic acid ($(\text{CH}_2)_{16}(\text{COOH})_2$), myristic acid etc.

Table 2.2 : The physical and chemical properties of Stearic acid.

Synonyms	Octadecanoic acid; 1-heptadecanecarboxylic acid
Appearance	White or yellowish-white powder.
Molecular formula	$\text{CH}_3 (\text{CH}_2)_{16} \text{COOH}$
Molecular weight	284.48 g/mol
Melting point $^{\circ}\text{C}$	69 - 70 $^{\circ}\text{C}$
Boiling point $^{\circ}\text{C}$	383 $^{\circ}\text{C}$
Specific gravity	0.94
Solubility in water	Insoluble in water.

CHAPTER III

METHODOLOGY

3.1 Chemical Substances

- i. Stearic acid as the main reagent
- ii. Hexane as the solvent compound/dilution agent
- iii. Isooctane as the standard substance for analysis
- iv. Anti bumping granules (solid) as an agent to uniform the temperature.

3.2 Equipment

- i. Heating mantle 250 ml
- ii. Round bottom flask 250 ml, 3 necks
- iii. Thermometer 200⁰C
- iv. Heating plate
- v. Measuring cylinder 50 ml
- vi. Analytical balance
- vii. Fume hood
- viii. Syringe
- ix. Gas Chromatographer (GC)

3.3 Experimental Works

Generally, the methodology in production of isooctane from the samples (stearic acid) can be achieved by 4 major scopes of experimental works:

- i. Preparation of calibration curve for pure isooctane
- ii. Sample preparation (isooctane from stearic acid)
- iii. Analysis sample using Gas Chromatography
- iv. Determine the concentration of isooctane obtained from stearic acid

3.4 Preparation of Calibration Curve For Pure Isooctane

There six calibration isooctane-hexane mixtures (Table 3.1) with 40mL volume are prepared, injected into several vials, labeled and analyzed using gas chromatography method to find the peak area of isooctane and hexane for each calibration isooctane-hexane mixture.

Table 3.1: Sample of Isooctane-Hexane Mixture

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

3.5 Experiment 1: Heating and Mixing Stearic and Zeolite at 100 rpm

The stearic acid was in form of solid form while in room temperature and its melting point at 69.4°C. The stearic acid is heated with 10 g anti-bumping granules and 20 g zeolite catalyst to enhance the reaction. This is because in previous research when using 20 g of zeolite the concentration of the product is higher compare to other amount of zeolite used. 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.

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 the sample taken using a syringe, which then injected into a vial and ready to be analyzed by G.C.

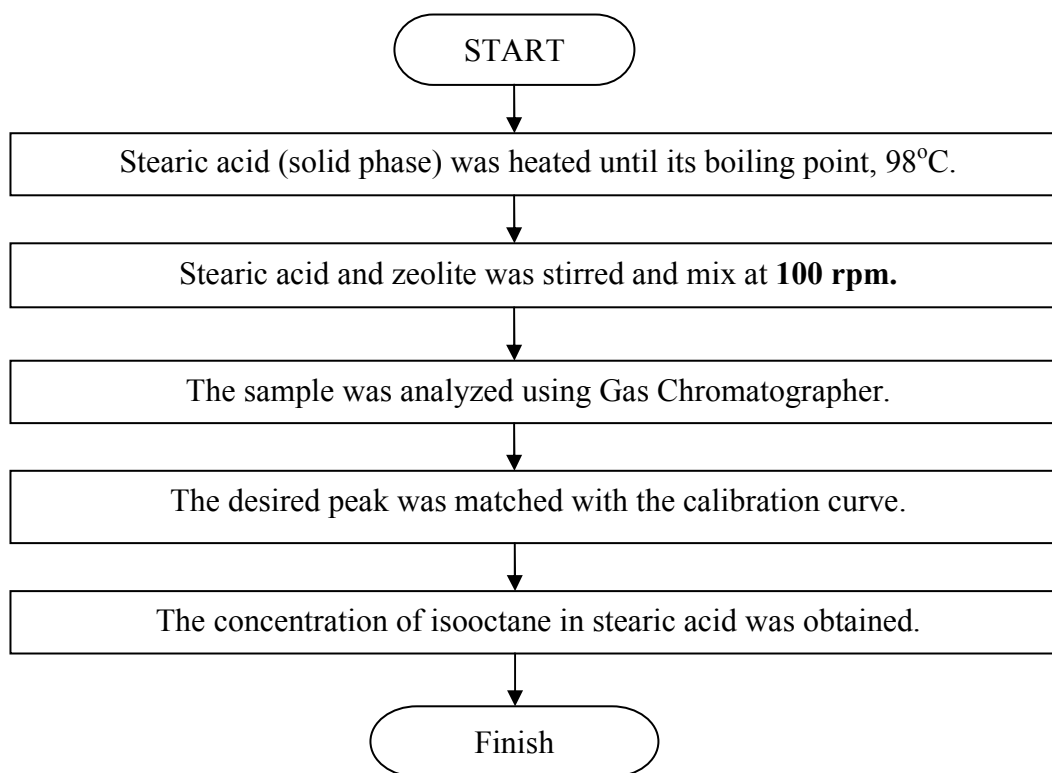


Figure 3.1 : Flow Diagram of Experiment 1

3.6 Analysis with Gas Chromatographer (GC)

Each mixture of melt stearic acid and zeolite melted at different stirrer speeds is added with hexane to be diluted and analyzed. The mixture for each sample added with hexane as in the Table 3.2.

Table 3.2: The percentage of concentration in each analysis sample

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

Condition of Gas Chromatographer use to analyze the composition of isooctane in the sample:-

Table 3.3: Gas Chromatographer Data Condition by referring to (PerkinElmer, Inc., 2004 & Omar, 2006)

Temperature Column	Initial 50°C, hold 3 minutes, program at 8°C/min to 185°C, hold 5 minutes
Injector Temperature	225°C
Detector Temperature	25°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

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Result standard calibration curve for pure isooctane

Standard calibration curve is needed to find the concentration of actual desired isooctane in the samples. The standard isooctane mixture contained of pure isooctane and hexane with composition mixture as recorded in Table 3.1 were analyzed with Gas Chromatography method. Isooctane chemical was required reference amount to make the standard calibration curve. Figure below show the chromatograms of the isooctane standards those consisting 0% isooctane (100% hexane), 20% isooctane, 30% isooctane, 40% isooctane, and 50% isooctane that are analyzed by using Gas Chromatographer. Then, the next of each chromatograms which are the table retention time and area for every components exist in the mixture of standard isooctane.

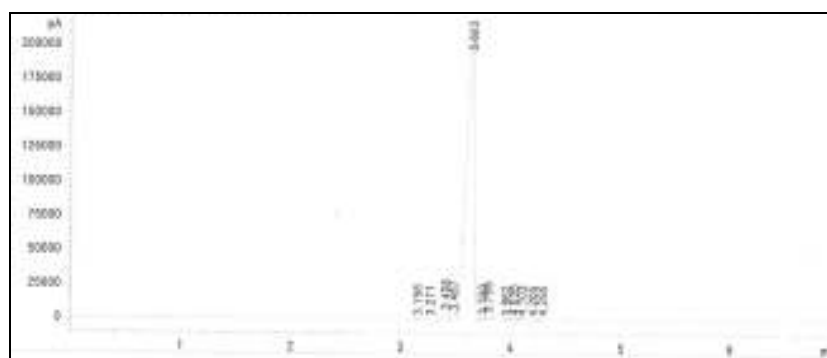
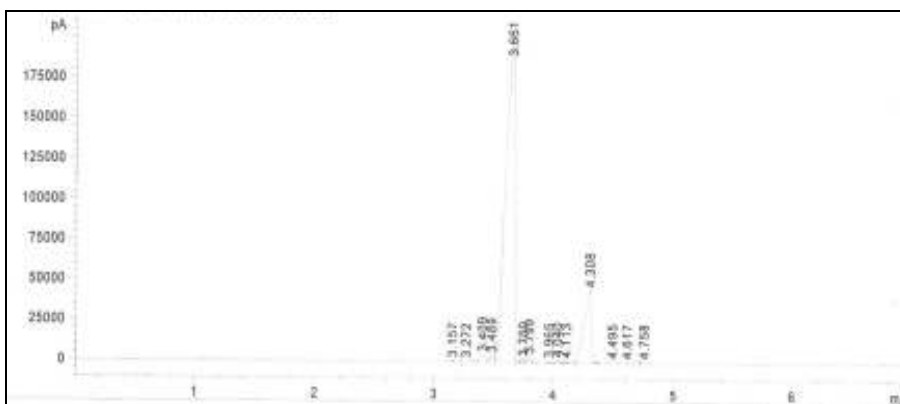


Figure 4.1: Chromatogram of Isooctane Standard for 0% Isooctane (100% Hexane)

Table 4.1: Retention Time and Area of Mixture Components For 0% Isooctane Standard

Percent Composition (%)	Item	Hexane	Isooctane
0	Retention time(min)	3.663	0
	Peak Area (pA*s)	981826	0
	Peak Area (%)	96.58262	0

**Figure 4.2 :** Chromatogram of Isooctane Standard for 20% Isooctane**Table 4.2:** Retention Time and Area of Mixture Components For 20% Isooctane Standard

%Composition	Item	Hexane	Isooctane
0	Retention time(min)	3.661	4.308
	Peak Area (pA*s)	902865	427.3564
	Peak Area (%)	93.79448	0.0444

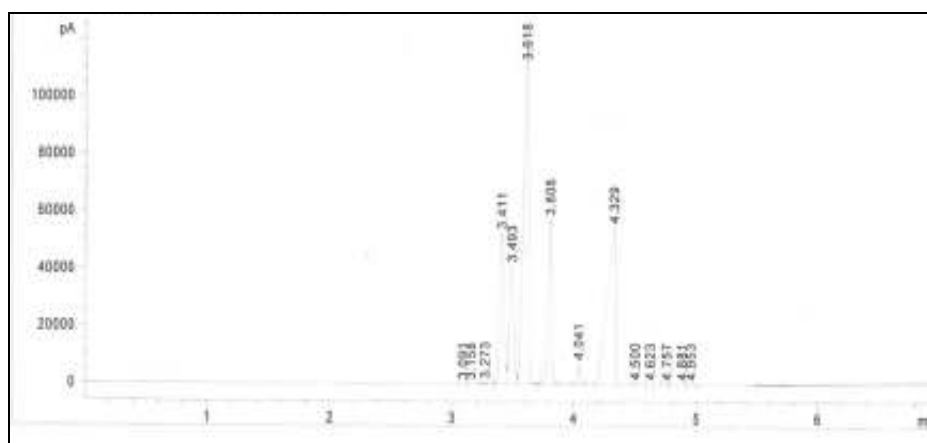
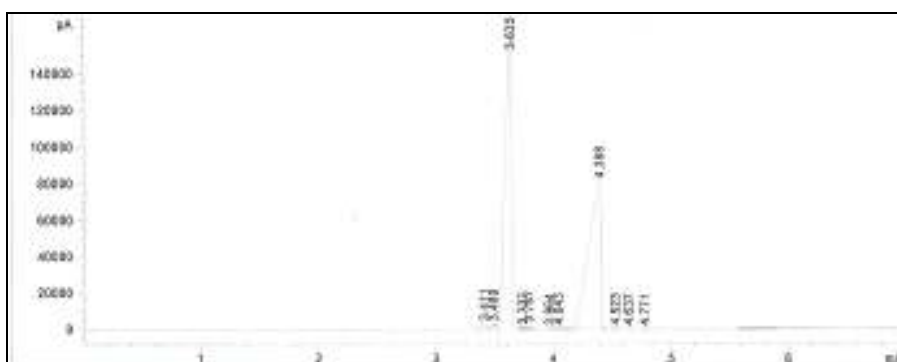
**Figure 4.3:** Chromatogram of Isooctane Standard for 30% Isooctane (70% Hexane)

Table 4.3: Retention Time and Area of Mixture Components For 30% Isooctane Standard

Percent Composition	Item	Hexane	Isooctane
0	Retention time(min)	3.618	4.329
	Peak Area (pA*s)	867473	123960
	Peak Area (%)	84.9769	12.14297

**Figure 4.4:** Chromatogram of Isooctane Standard for 40% Isooctane (60% Hexane)**Table 4.4:** Retention Time and Area of Mixture Components For 40% Isooctane Standard

Percent Composition	Item	Hexane	Isooctane
0	Retention time(min)	3.635	4.388
	Peak Area (pA*s)	706064	313349
	Peak Area (%)	67.70538	30.04738

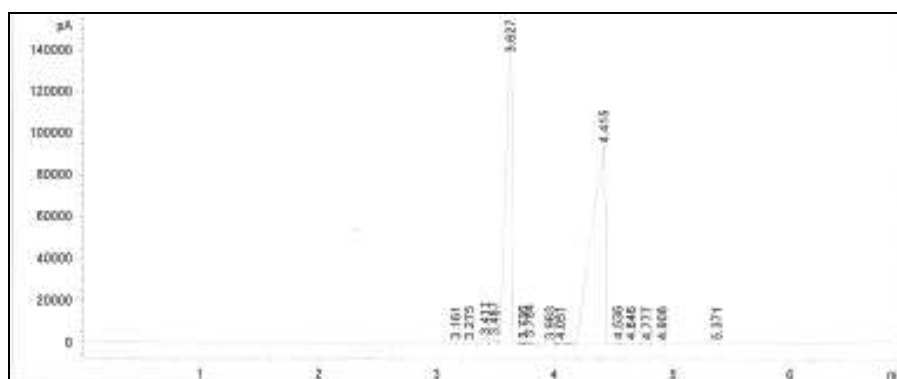
**Figure 4.5:** Chromatogram of Isooctane Standard for 50% Isooctane (50% Hexane)

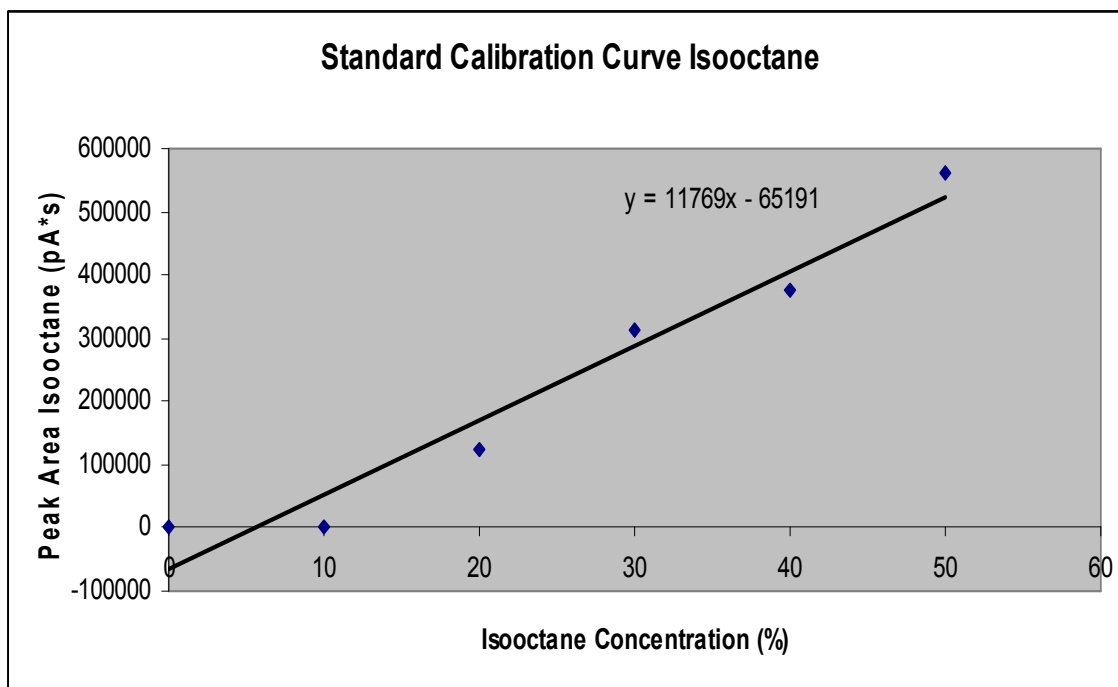
Table 4.5: Retention Time and Area of Mixture Components For 50% Isooctane Standard

% Composition	Item	Hexane	Isooctane
0	Retention time(min)	3.627	4.415
	Peak Area (pA*s)	464268	560814
	Peak Area (%)	44.62297	53.90247

From Table 4.1 until 4.5, the tables describe the time where the compound exist and also its peak area. The retention time of each percent of Isooctane standard put in the table below and the graph were plotted.

Table 4.6: Retention Time For Isooctane Standard

Standard Isooctane	Retention Time difference
0	0.00
10	0.616
20	0.602
30	0.658
40	0.678
50	0.717
average	0.5452

**Figure 4.6:** Standard Calibration Curve

4.2 Observation

4.2.1 Stearic Acid at level 1 (600rpm)

Stearic acid was in white solid form at room temperature. When the heat was supplied, the stearic acid started melting and at 64°C which is the stearic acid's melting point, all of the solid stearic acid melts and becomes colorless liquid. The temperature was maintained at 98°C to make sure no isooctane escaped to the air. The stirrer started after all the solid acid become liquid. The movement while heating and stirring at this level was smooth but it looks like the stirring speed still not enough. After two hours cracking, the solutions become cloudy.

4.2.2 Stearic acid at level 2 (800rpm)

Stearic acid was in white solid form at room temperature. When the heat was supplied, the stearic acid started melting and at 64°C which is the stearic acid's melting point, all of the solid stearic acid melts and becomes colorless liquid. The temperature was maintained at 98°C to make sure no isooctane escaped to the air. The stirrer started after all the solid acid become liquid. The movement while heating and stirring at second level was smooth and the speeds look energetic. After two hours cracking, the solutions become cloudier.

4.2.3 Stearic acid at level 3 (1000rpm)

Stearic acid was in white solid form at room temperature. When the heat was supplied, the stearic acid started melting and at 64°C which is the stearic acid's melting point, all of the solid stearic acid melts and becomes colorless liquid. The temperature was maintained at 98°C to make sure no isooctane escaped to the air. The stirrer started after all the solid acid become liquid. The movement while heating and stirring at this

level not too smooth and looks vigorous. After two hours cracking, the solutions become very cloudy.

4.2.4 Stearic acid at level 4 (1200rpm)

Stearic acid was in white solid form at room temperature. When the heat was supplied, the stearic acid started melting and at 64°C which is the stearic acid's melting point, all of the solid stearic acid melts and becomes colorless liquid. The temperature was maintained at 98°C to make sure no isooctane escaped to the air. The stirrer started after all the solid acid become liquid. The movement while heating and stirring at this level was too strong and it looks the stirrer not rotated at the same place but sometimes just vibrate. After two hours cracking, the solutions become very cloudy.

4.3 Results for Samples (Stearic Acid)

Data's and values from the raw data's are not described and showed its actual concentration. This is because the samples that are analyzed in Gas Chromatographer (GC) are diluted first twice with hexane as the dilution agent to reduce their concentration. Dilution of the samples before injected to the GC is required because GC is very sensitive equipment especially the column used. Only colorless or more specific is only very dilute and non-particle liquid sample can be injected and run using the GC, to avoid the blocking occurrence in the GC column by the solid particles. From the previous chapter, Chapter 2, the properties of palmitic acid has been described that the stearic acid is solid in room temperature and its melting point is around 60-70 °C. The collecting product from catalytic cracking is tend to be solid again.

So, to get the actual peak area of isooctane and actual concentration of isooctane, the back calculations are needed regarding how much the dilution is done. In this case, twice of dilution are done to achieve the suitable and acceptable liquid dilution. The

calculation method below are used to achieve above description. This calculation included to find the actual peak area of isooctane in percent, actual peak area of isooctane in pA*s, and actual isooctane concentration in percent.

$$\text{i. Actual peak area isooctane (\%)} = [\text{peak area isooctane (\%)} / [100 - \text{peak area hexane (\%)}]] * 100$$

$$\text{ii. Actual peak area (pA*s)} = [\text{peak area isooctane (pA*s)} / \text{peak area isooctane (\%)}] * \text{actual peak area of isooctane (\%)}$$

iii. Actual isooctane concentration is obtained by matching the actual peak area in pA*s on the standard calibration curve shown in figure 4.6 or just using its equation of standard calibration curve that is $y = 11769x - 65191$. 'y' is described as actual peak area of isooctane in pA*s and 'x' is actual isooctane concentration in percent.

All the results of calculated values for catalytic cracking of stearic acid using zeolite catalyst and its respective level of speed are recorded in table below. For more details calculation, see Appendix A.

Table 4.7 : Actual concentration of Isooctane

Level of Speed	Sample	A	B	C	D	E	F	G	AVERAGE
L1	S1	0.05991	827.638	70.72057	29.27943	0.204615	2826.688	5.779394	5.873012
L1	S2	0.05995	828.683	79.366	20.634	0.29054	4016.104	5.880458	
L1	S3	0.06246	818.963	81.483	18.517	0.337312	4422.763	5.915011	
L1	S4	0.06221	809.199	81.809	18.191	0.341982	4448.348	5.917185	
L2	S5	0.06516	811.12	80.943	19.057	0.341922	4256.284	5.900865	5.920934
L2	S6	0.06239	798.874	81.825	18.175	0.343274	4395.455	5.912691	
L2	S7	0.063	850.8	81.717	18.283	0.344582	4653.503	5.934617	
L2	S8	0.0632	852.838	81.717	18.283	0.345676	4664.65	5.935564	

L3	S9	0.06177	816.891	70.7707	29.2293	0.211329	2794.768	5.776682	5.886014
L3	S10	0.06186	817.284	81.8095	18.1905	0.340068	4492.917	5.920972	
L3	S11	0.06194	842.025	81.7995	18.2005	0.34032	4626.384	5.932312	
L3	S12	0.06185	801.821	81.826	18.174	0.340321	4411.913	5.914089	
L4	S13	0.06174	782.516	81.845	18.155	0.340072	4310.196	5.905446	5.872604
L4	S14	0.06202	778.937	81.8089	18.1911	0.340936	4281.968	5.903048	
L4	S15	0.06153	807.478	81.861	18.139	0.339214	4451.613	5.917462	
L4	S16	0.06111	777.771	70.66055	29.33945	0.208286	2650.939	5.764461	
Notes :									
A = Peak area desired isooctane (%) B = Peak area desired isooctane (pA*s) C = Peak area hexane(%)									
D = 100 - peak area hexane (%) E = Actual peak area (%) F = Actual peak area (pA*s)									
G = Actual concentration of isooctane (%)									

4.4 Discussion

Cracking method is used to breaking up large hydrocarbon molecules into smaller and more useful bits of hydrocarbon compounds. The long-chain hydrocarbon molecules are at first broken up in a fairly random way to produce mixtures of various smaller hydrocarbon radicals, and then these radicals recombine in different arrangement through isomerization process. Zeolites catalyst consists complex aluminosilicates, and are large lattices of aluminium, silicon and oxygen atoms carrying a negative charge. They are, associated with positive ions such as sodium ions. The desired isooctane molecules are formed through this synthesized. The isooctane produced is as indicator the biopetrol is synthesized and concentrated in this process. So, the amount of isooctane produced is showed the amount of biopetrol produced. Isooctane is targeted as product of biopetrol because petrol itself is dominated by isooctane.

4.4.1 First Reading of Cracking Stearic Acid

4.4.1.1 Cracking of Stearic Acid At Level 1, 2, 3 and 4.

From the obtained results (see Table 4.7), the percentage concentration of isooctane obtained in catalytic cracking of stearic acid at level 1 which is the speed was around 600 rpm and the product is 5.779394 %. When cracking was done at level 2 (800rpm), level 3 (100rpm) and level 4 (1200rpm) the isooctane obtained are 5.900865%, 5.776685%, and 5.905446% respectively. All the results are plotted in Figure 4.7 which concentration of Isooctane versus stirrer speed level.

Theoretically, when the cracking was stirrer at different speed its actual percent concentrations should not be same between four readings. These are because the more higher the stirrer speed more active catalyst spot will exposed to the reactant. According to the Figure 4.7 below, the concentration of isooctane dose not increase with the stirrer speed. These are happened because of solidify of the product. The graph shows that the concentration was high at level 2 and 4. This means something wrong happened at speed 3 because the value is decreased.

4.4.2 Second Reading of Cracking Stearic Acid

4.4.2.1 Cracking of Stearic Acid At Level 1, 2, 3 and 4.

From the obtained results (see Table 4.7), the percentage concentration of isooctane obtained in catalytic cracking of stearic acid at level 1 which is the speed was around 600 rpm and the product is 5.880458 %. When cracking was done at level 2 (800rpm), level 3 (100rpm) and level 4 (1200rpm) the isooctane obtained are 5.912691%, 5.920972%, and 5.903048% respectively. All the results are plotted in Figure 4.7 which concentration of Isooctane versus stirrer speed level.

Theoretically, when the cracking was stirrer at different speed its actual percent concentrations should not be same between four readings. These are because the more higher the stirrer speed more active catalyst spot will exposed to the reactant. According to the Figure 4.7 below, the concentration of isooctane dose not increase with the stirrer speed. These are happened because of solidify of the product. The graph shows that the concentration was proportional at level 1 until level 3. This means something wrong happened at level 4 because the value is decreased.

4.4.3 Third Reading of Cracking Stearic Acid

4.4.3.1 Cracking of Stearic Acid At Level 1, 2, 3 and 4.

From the obtained results (see Table 4.7), the percentage concentration of isooctane obtained in catalytic cracking of stearic acid at level 1 which is the speed was around 600 rpm and the product is 5.915011%. When cracking was done at level 2 (800rpm), level 3 (100rpm) and level 4 (1200rpm) the isooctane obtained are 5.934616%, 5.932312%, and 5.917462% respectively. All the results are plotted in Figure 4.7 which concentration of Isooctane versus stirrer speed level.

Theoretically, when the cracking was stirrer at different speed its actual percent concentrations should not be same between four readings. These are because the more higher the stirrer speed more active catalyst spot will exposed to the reactant. According to the Figure 4.7 below, the concentration of isooctane dose not increase with the stirrer speed. These are happened because of solidify of the product. The graph shows that the concentration was increased only at level 2. This means something wrong happened at level 3 and 4 because the value is decreased.

Fourth Reading of Cracking Stearic Acid

4.4.3.2 Cracking of Stearic Acid At Level 1, 2, 3 and 4.

From the obtained results (see Table 4.7), the percentage concentration of isooctane obtained in catalytic cracking of stearic acid at level 1 which is the speed was around 600 rpm and the product is 5.917185%. When cracking was done at level 2 (800rpm), level 3 (100rpm) and level 4 (1200rpm) the isooctane obtained are 5.935564%, 5.914089%, and 5.764461% respectively. All the results are plotted in Figure 4.7 which concentration of Isooctane versus stirrer speed level.

Theoretically, when the cracking was stirrer at different speed its actual percent concentrations should not be same between four readings. These are because the more higher the stirrer speed more active catalyst spot will exposed to the reactant. According to the Figure 4.7 below, the concentration of isooctane dose not increase with the stirrer speed. These are happened because of solidify of the product. The graph shows that the concentration was increased only at level 2. This means something wrong happened at level 3 and 4 because the value is decreased.

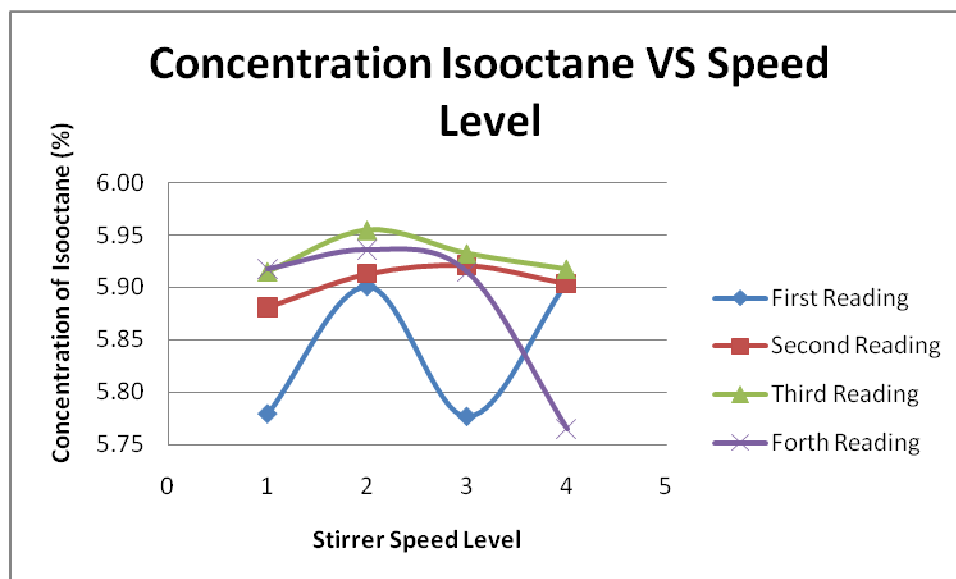


Figure 4.7 : Concentrntion of Isooctane At Differences Reading

4.5 Overall Catalytic Cracking of Stearic Acid

From the results obtained (Table 4.7), cracking of stearic acid using various of stirring speed, its average total percent of isooctane versus its level of speed are plotted in Figure 4.8. From the Figure 4.8 below, percent concentrations of isooctane produced are not increased when increasing of level of speed. These are deviated from the theory and literature. But generally, the results are not too far deviated from the theory.

The values are fluctuated and deviated from the theory (product will increased when the speed of stirrer are increased), because of several caused - role of magnetic stirrer, solidify, and experimental errors.

This experiment was run using conical flask heated by hot plate and the stearic acid is stirred by magnetic stirrer. Supposedly the percentage of isooctane will increased when the speed of stirring increased but the result was deviated. This situation happened because when stirring at high level of speed the magnetic stirrer not rotating perfectly. The stirrer is over speed thus it cannot maintain the initial position and always jumping inside the flask. Thus not all the active size are used optimally during the catalytic reaction.

Product (isooctane) is difficult to be maintained its original amounts and concentrations produced during the experiment tent to changes into solid. So, the amounts and concentrations analyzed are not the actual and original amounts and concentrations produced.

The results values are fluctuated and deviated from the theory also because of experimental errors. Even though this research are not go through the effect of time consumed during the reaction process, but the experimenter are took easy about the time consumed during the reaction process. This catalytic cracking process of all parameter for every reaction should done with same time. Time should be constant variable of the experiment. Different time consumed, different amounts of products produced are different. Maybe during the reading values taken, the parallax error are done. The

apparatus used especially the vials that used for analyzed in GC are not very clean. This is effected the reading values.

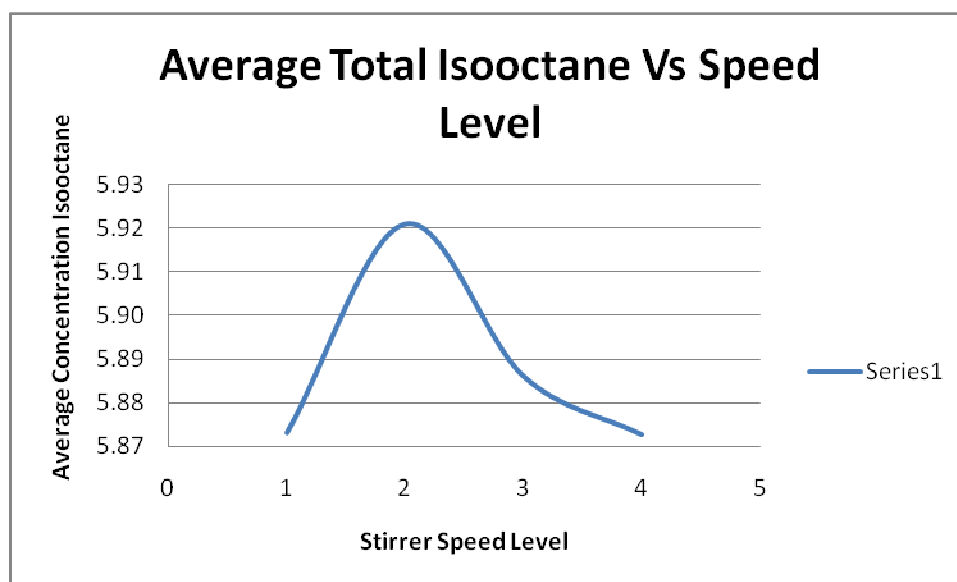


Figure 4.8 : Average Concentrtion of Isooctane At Differences Level

CHAPTER V

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The purposes of this research are to improve the concentration of isooctane produced from stearic acid by using heterogeneous catalytic cracking method and to compare the yields of isooctane produced using this method with the catalytic cracking method.

From the obtained result, the highest percentage concentrations of isooctane obtained are 5.921 % for level 2 of stirrer speed, while the other speed level 1, level 3 and level 4 each obtained 5.873 %, 5.886 % and 5.872 % of concentration of isooctane.

The percentage concentrations of isooctanes produced are increased when the speed of stirring increased. However, the values are fluctuated and deviated from the theory (product will increased when the speed of stirrer are increased), because of several caused - role of magnetic stirrer, solidify, and experimental errors. These yields are much higher than the yields produced from thermal cracking and static catalytic cracking method which is 3 to 5 %. It showed that the heterogeneous catalytic cracking

method will produce much higher yields for biopetrol synthesized compared to thermal cracking method and static catalytic cracking.

5.2 Recommendation

In order to increase the concentration of biopetrol (isooctane) in stearic acid, it is recommended the research is conducted with more proper technologies of dynamic state catalytic cracking process which mean using heating mantel that was equipped with the stirrer also with the digital control beside heating and stirring the stearic acid in conical flask with hot plate. The time of reaction conducted should be constant variable to all the time.

Other research is studied on catalytic cracking of palm oil over various mesoporous catalysts beside microporous zeolite catalysts (Twaiq *et al.*, 2003, 2004; Yean-Sang *et al.*, 2004). Different catalysts will lead to different product distribution of catalytic cracking. Zeolite itself is governed by acidity, pore shape and size. So, use that resulted information to use the very optimum type of zeolite catalyst in biopetrol synthesized. The recommended zeolite is Zeolite Y.

To enhance and to improve the yields of biopetrol synthesized, the best solution and method to minimize the solidify problem after collecting the distilled product should be done. The solidification problem will alter the actual amount of biopetrol yields. The experiment also should minimize the human errors such as parallax reading error and the cleanliness of the apparatus used.

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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: $(\text{CH}_3)_3\text{CCH}_2\text{CH}(\text{CH}_3)_2$

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 in 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 in appendix F)

S16 S26 S33 S36 S37 S39.

APPENDIX B

Abbreviations used in Toxicity data

dom	domestic animal (goat, sheep)
dpo	<i>Drosophila pseudo-obscura</i>
emb	embryo
esc	<i>Escherichia coli</i>
eug	<i>Euglena gracilis</i>
eye	administration into eye (irritant)
ice	intracerebral
icv	intracervical
idr	intradermal
idu	intraduodenal
ihl	inhalation
isp	intrapinal
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 breathe dust.
- S23 Do not breathe 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

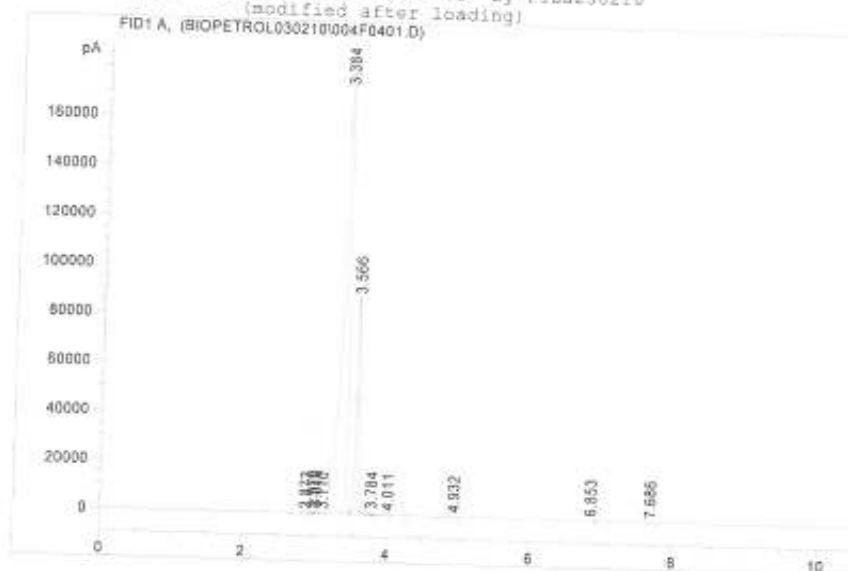
- Class 1 Explosive
 - 1.1 Substances with a mass explosion hazard
 - 1.2 Substances which present a projection hazard but no mass explosion hazard
 - 1.3 Substances which present both a fire hazard and a minor blast or projection hazard (or both) but not a mass explosion hazard
 - 1.4 No significant 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 E

Result Chromatogram

Data File C:\CHEM32\1\DATA\BIOPETROL030210\004FD4D1.D
Sample Name: S4 NQ

```
=====
Acq. Operator   : FIZa030210                      Seq. Line :    4
Acq. Instrument : Instrument 1                      Location  : Vial 4
Injection Date  : 03/02/2010 13:53:37              Inj       :    1
                                                    Inj Volume: 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\ISOOCCTANE230109.M
Last changed    : 03/02/2010 12:42:54 by FIZa030210
Analysis Method : C:\CHEM32\1\METHODS\POME_PSM.M
Last changed    : 24/02/2010 12:43:09 by FIZa230210
                  (modified after loading)
=====
```



Area Percent Report

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

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.873	BV	0.0320	36.81513	17.88972	0.00283
2	2.970	VV	0.0291	659.21735	347.71002	0.05068
3	3.018	VV	0.0360	1517.48267	655.06354	0.11665
4	3.110	VV	0.0412	146.27969	56.43309	0.01124
5	3.384	VV S	0.0877	1.06421e6	1.80599e5	81.80919
6	3.566	VB S	0.0441	2.33049e5	8.79761e4	17.91519
7	3.784	BB X	0.0282	809.19934	444.54868	0.06221
8	4.011	BB X	0.0263	41.23520	24.92029	0.00317
9	4.932	BB	0.0271	5.46984	3.34812	0.00042
10	6.853	BB	0.0409	5.67408	2.14448	0.00044
11	7.686	BB	0.0500	13.75804	4.22451	0.00106
12	11.966	BB	0.2538	350.15909	16.42167	0.02692

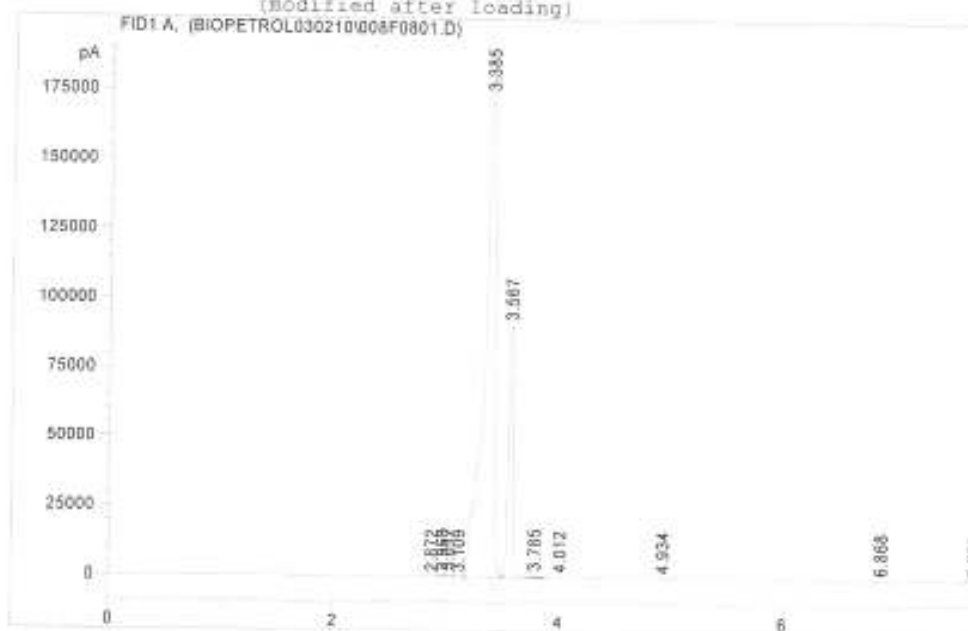
Totals : 1.30084e6 2.70148e5

Instrument 1 24/02/2010 12:47:06 FIZa230210

```

=====
Acq. Operator   : FIZa030210                      Seq. Line :    8
Acq. Instrument : Instrument 1                     Location  : Vial 6
Injection Date  : 03/02/2010 15:26:14              Inj       :    1
                                                    Inj Volume: 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\ISOOCCTANE230108.M
Last changed    : 03/02/2010 12:42:54 by FIZa030210
Analysis Method : C:\CHEM32\1\METHODS\PCME.PSM.M
Last changed    : 24/02/2010 12:56:54 by FIZa230210
                  (modified after loading)
=====

```



Area Percent Report

```

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

```

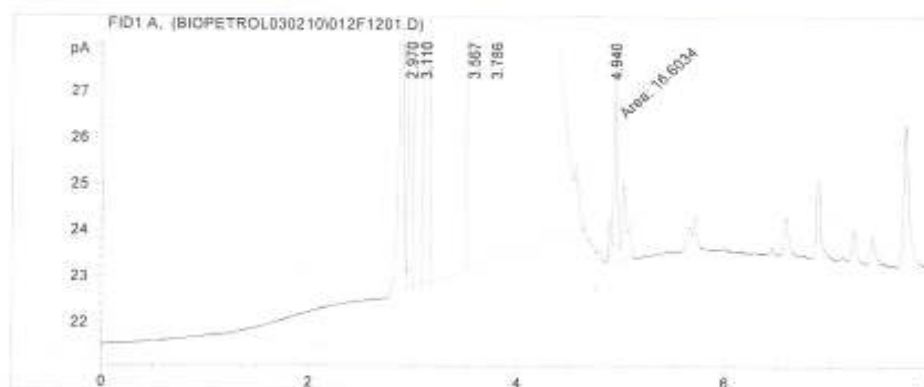
Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.872	BV	0.0316	17.58752	8.67773	0.00130
2	2.968	VV	0.0289	528.91357	281.64243	0.03920
3	3.017	VV	0.0346	1319.24451	579.58484	0.09777
4	3.109	VV	0.0409	140.59496	54.80230	0.01042
5	3.385	VV S	0.0898	1.10269e6	1.81754e5	81.71679
6	3.567	VB S	0.0446	2.43434e5	9.10453e4	18.04016
7	3.785	BB X	0.0280	852.83826	473.53018	0.06320
8	4.012	BB X	0.0249	38.97058	24.08995	0.00289
9	4.934	BV	0.0293	8.27207	4.33732	0.00061
10	6.868	BB	0.0412	5.99981	2.24130	0.00044
11	7.705	BB	0.0500	15.19014	4.54695	0.00113
12	11.526	BB	0.2414	269.55542	13.64902	0.01998
13	12.893	BB	0.0345	52.74550	23.21164	0.00391
14	13.274	BB	0.0804	29.81230	5.01402	0.00221

Instrument 1 24/02/2010 12:59:16 FIZa230210

Data File C:\CHEM32\1\DATA\BIOFETROL030210\012F1201.D
 Sample Name: S12 NQ

```
=====
Acq. Operator   : FIZA030210                      Seq. Line : 12 (6th Level)
Acq. Instrument : Instrument 1                     Location  : Vial 12 (6th Lvl)
Injection Date  : 03/02/2010 16:59:00              Inj       : 1
                                                    Inj Volume: 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\ISOCTANE230108.M
Last changed    : 03/02/2010 12:42:54 by FIZA030210
Analysis Method : C:\CHEM32\1\METHODS\ISOCTANE230109.M
Last changed    : 22/02/2010 12:46:36 by FIZA100210
                  (modified after loading)
Method Info     : isooctane/psm
=====
```



Area Percent Report

```
=====
Sorted By      : Signal
Calib. Data Modified : 27 August 2008 13:05:28
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A,

Peak #	RetTime (min)	Type	Width (min)	Area (pA*s)	Area %	Name
1	2.185		0.0000	0.00000	0.00000	
2	2.253		0.0000	0.00000	0.00000	
3	2.358		0.0000	0.00000	0.00000	
4	2.359		0.0000	0.00000	0.00000	
5	2.762		0.0000	0.00000	0.00000	
6	2.970	VV	0.0292	696.80658	0.05375 ?	
7	3.018	VV	0.0348	1561.94299	0.12048 ?	
8	3.110	VV	0.0417	153.78229	0.01186 ?	
9	3.386	VV S	0.0979	1.06083e6	81.82623 ?	C ₂
10	3.567	VB S	0.0441	2.32381e5	17.92456 ?	
11	3.786	BB X	0.0285	801.82098	0.06185 ?	C ₂
12	4.797		0.0000	0.00000	0.00000	
13	4.940	MM T	0.0601	16.60342	0.00128 ?	

Totals : 1.29644e6

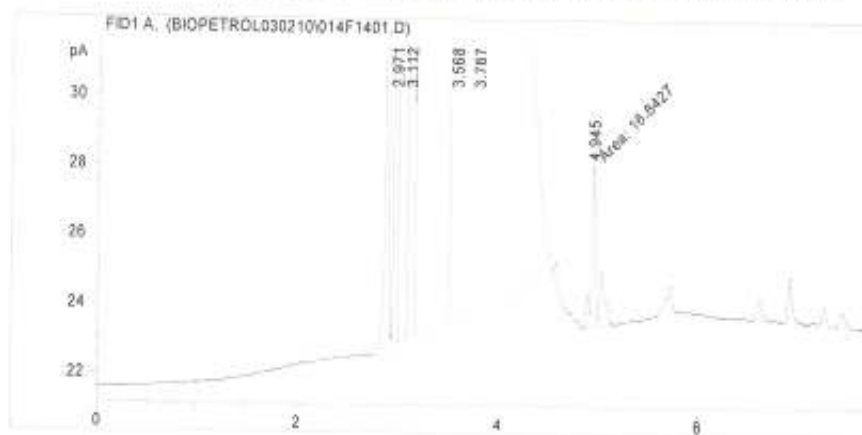
2 Warnings or Errors :

Instrument 1 22/02/2010 12:54:11 FIZA100210

P

Data File C:\CHEM32\1\DATA\BIOPETROL030210\014F1401.D
 Sample Name: S14 NQ

```
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Acq. Operator   : FI2a030210                      Seq. Line : 14 (7th Lo
Acq. Instrument : Instrument 1                     Location  : Vial 14 (7th
Injection Date  : 03/02/2010 17:45:23              Inj       : 1
                                                    Inj Volume: 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\ISOCTANE230108.M
Last changed    : 03/02/2010 12:42:54 by FI2a030210
Analysis Method : C:\CHEM32\1\METHODS\ISOCTANE230108.M
Last changed    : 22/02/2010 12:46:36 by FI2a100210
                  (modified after loading)
Method Info     : isooctane/pem
=====
```



Area Percent Report

```
=====
Sorted By      : Signal
Calib. Data Modified : 27 August 2009 13:05:28
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Area %	Name
1	2.185		0.0000	0.00000	0.00000	
2	2.253		0.0000	0.00000	0.00000	
3	2.308		0.0000	0.00000	0.00000	
4	2.359		0.0000	0.00000	0.00000	
5	2.762		0.0000	0.00000	0.00000	
6	2.971 VV		0.0291	637.12457	0.05073	?
7	3.020 VV		0.0347	1458.26306	0.11611	?
8	3.112 VV		0.0402	145.71402	0.01160	?
9	3.386 VV S		0.0961	1.02748e6	81.80896	? <= C ₆
10	3.568 VB S		0.0435	2.25433e5	17.94924	?
11	3.787 BB X		0.0280	778.93726	0.06202	? <= C ₈
12	4.797		0.0000	0.00000	0.00000	
13	4.945 MM T		0.0606	16.84268	0.00134	?

Totals : 1.25595e6

2 Warnings or Errors :

Instrument 1 22/02/2010 12:56:42 FI2a100210