

BIOPETROL SYNTHESIZED FROM RUBBER SEED OIL USING  
ZEOLITE AS CATALYST : EFFECT OF HEXANE IN SOLVENT  
EXTRACTION OF RUBBER SEED.

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**UNIVERSITI MALAYSIA PAHANG**  
**BORANG PENGESAHAN STATUS TESIS**

**JUDUL : BIOPETROL SYNTHESIZED FROM RUBBER SEED OIL USING ZEOLITE AS CATALYST : EFFECT OF HEXANE IN SOLVENT EXTRACTION OF RUBBER SEED**

**SESI PENGAJIAN : 2011/2012**

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**BIOPETROL SYNTHESIZED FROM RUBBER SEED OIL USING ZEOLITE  
AS CATALYST : EFFECT OF HEXANE IN SOLVENT EXTRACTION OF  
RUBBER SEED**

**SHANKER A/L SHAMUGAM**

**A thesis submitted in fulfillment  
of the requirements for the award of the Degree of  
Bachelor of Chemical Engineering (Pure)**

**Faculty of Chemical & Natural Resources Engineering  
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**JANUARY 2012**

### **STUDENT'S DECLARATION**

I hereby declare that this thesis entitled “Biopetrol synthesized from rubber seed oil using zeolite as catalyst : effect of hexane in solvent extraction of rubber seed” is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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## DEDICATION

*To my beloved parents and siblings*

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## ABSTRACT

Petrol is the most demanding fuel nowadays. Petrol is used especially as a fuel for cars, aircraft and other vehicles. The declining petroleum source that we face today causes the increasing of the petroleum prices all over the world. This problem can be solved by using an alternative source to replace the usual commercial petrol we are using now. This can be done by producing biopetrol or biofuel. Currently, most of the bio-petrol is produced from oils such as the utilization of palm oil for the production of more environmental friendly bio-fuels. . The objective of this study is to synthesize biopetrol from fatty acid in rubber seed oil via catalytic cracking using zeolite as catalyst. Extracting rubber seed oil from the rubber seeds, is a favorable method as rubber seed oil contains more fatty acid to be produced into bio-petrol. The rubber seeds are readily available in our country for ages, cheap and help to improve the socioeconomic issues. The rubber seed oil is extracted using the Soxhlet Extraction method. The cleaned, shelled and milled rubber seeds are placed into a thimble in the main chamber of the extractor. The solvent, hexane in the receiving flask is left to boil until it vaporizes and condenses filling up the main chamber, extracting the rubber seed oil from the rubber seeds. The catalytic cracking of the mixture of 0.1L of rubber seed oil, 20g of catalyst which is in this case zeolite and anti-bumping granule( to assist well distributed boiling) at 300<sup>0</sup>C for 45 minutes is to boost up the rate of reaction so that more successful reactions between the reactant particles can occur. The presence of Isooctane in a sample detected using Gas Chromatogram indicating that bio-petrol can be produced. Standards of different ratio mixtures of hexane and Isooctane were used to obtain chromatograms for Isooctane until a calibration curve is plotted from which the Isooctane produced can be determined. The results show that the actual concentration of Isooctane is very high. This could be explained using the cause of interlayer spacing of catalyst structure, larger surface area for reactions to occur, various types of fatty acid mixture present in rubber seed oil, the incorrect chromatogram modifications and also the contamination in rubber seed oil. The volume of solvent used will affect the percentage of concentration of Isooctane in samples obtained. As a conclusion, bio-petrol can be produced from rubber seed oil using zeolite catalyst in the catalytic cracking process.



## ABSTRAK

Petrol adalah bahan api yang paling banyak diperlukan pada masa kini. Petrol digunakan sebagai bahan api kenderaan. Kemerosotan sumber petrol pada masa kini menyebabkan harga petrol terus meningkat di seluruh dunia. Masalah ini dapat diselesaikan dengan adanya satu sumber alternatif untuk menggantikan petrol komersial biasa. Ini boleh dilakukan dengan menghasilkan biopetrol. Pada masa kini kebanyakan biopetrol dihasilkan daripada minyak sawit untuk menghasilkan biopetrol yang mesra alam. Objektif kajian ini adalah untuk menghasilkan biopetrol daripada asid lemak daripada biji getah melalui kaedah pemecahan asid lemak berpemangkin dengan zeolite sebagai pemangkin. Pengekstrakan minyak getah daripada biji getah adalah cara yang efektif disebabkan biji getah mengandungi lebih banyak asid lemak yang dapat ditukarkan kepada biopetrol. Biji getah boleh didapati dengan senang di Negara kita sejak dulu, murah, dan bantu memperbaiki isu sosio ekonomi. Minyak biji getah diekstrak menggunakan pengeksrak soklet. Biji getah dibersihkan, dikuliti, dan dikisar. Kemudian diisikan ke dalam bidal dan disimpan kedalam pengeksrak soxhlet. Pelarut iaitu heksana dibenarkan mendidih di bekas menerima di bawah sekali sehingga ianya menyejat dan seterusnya mengkondensasi di bahagian utama di atas dengan seterusnya mengekstrak minyak biji getah daripada biji getah. Seterusnya pemecahan berpemangkin 0.1 liter minyak biji getah dengan 20 gram pemangkin iaitu zeolite dilakukan pada suhu  $300^{\circ}\text{C}$  selama 45 minit untuk meningkatkan lagitindak balas antara bahan tindak balas. Kehadiran isooctan dalam dalam bahan sample ditentukan dengan menggunakan kromatogram gas untuk membuktikan kehadiran biopetrol. Sample dengan beza kepekatan campuran heksana dan isooctane digunakan untuk mendapatkan kromatogram sehingga penuntukan garis tepat diperolehi. Keputusan akhir menunjukkan kepekatan akhir isooctane sebenarnya agak tinggi. Ini boleh dijelaskan menggunakan teori bentok permukaan pemangkin dan luas permukaan yang didapati untuk tindak balas, pelbagai jenis asid lemak yang dapat diperolehi dalam biji getah, ralat pada kromatogram gas, dan benda asing dalam minyak biji getah. Akhirnya didapati jumlah pelarut yang digunakan mempengaruhi jumlah isooctan yang diperolehi. Kesimpulannya biopetrol dapat dihasilkan menggunakan zeolite sebagai pemangkin dalam proses pemecahan berpemangkin dengan berjayanya.

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**LIST OF SYMBOLS**

P	-	Pressure
m	-	Mass
$\Delta H$	-	Enthalpy change of reaction
$\Delta S$	-	Entropy change of reaction
$\Delta G$	-	Energy change of reaction
T	-	Temperature
$\rho$	-	Density
$\mu$	-	Viscosity of liquid (Pa.s)
h	-	Heat transfer coefficient
$^{\circ}\text{C}$	-	Degree Celsius
kg	-	Kilogram
K	-	Degree Kelvin
m	-	Meter
n	-	Number of moles
L	-	Liter

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## **CHAPTER 1**

### **1.0 INTRODUCTION.**

#### **1.1 History Of Biofuel**

Bio-fuels seem to be hot topic in today's energy markets, so what's the scoop? First of all, biofuels come in many varieties, but in general, there are two main products, those that replace (or blend with) gasoline, and those that replace (or are blended with) middle distillate fuels, such as diesel fuel, or home heating oil. For gasoline replacement and blending, the predominant fuel is ethanol, which is a water-clear liquid that is distilled from a variety of plants, but most commonly corn in the US. Distillate fuel substitutes can also be derived from a variety of plants such as soybeans, palm seeds, and peanuts, or even animal by-products such as used cooking oil grease. Resulting fuel is usually referred to as Biodiesel or B100 (100% Biofuel, no petroleum). When used as a heating oil blend, the product is sometimes referred to as Bioheat. When oils derived from these sources start out, they are not suitable for combustion, or use in a diesel engine, they need to be processed first. The process for converting raw fats and oils into "Biodiesel" separates the oil from the glycerin (one of the main components of soap). Once the glycerin is removed, the resulting Biodiesel can be used virtually interchangeably with diesel fuel or home heating oil. One important benefit of these fuels is that they have similar energy value per gallon as conventional heating oil, with the added benefits of the clean burning Biofuel.

Biofuels in the solid form has been in use ever since man discovered fire. Wood was the first form of biofuel that was used even by the ancient people for cooking and heating. With the discovery of electricity, man discovered another way of utilizing the biofuel. Biofuel had been used since a very long time for the production of electricity. This form of fuel was discovered even before the discovery of the fossil fuels, but with the exploration of the fossil fuel like gas, coal, and oil the production and use of biofuel suffered a severe impact. With the advantages placed by the fossil fuels they gained a lot of popularity especially in the developed countries. Liquid biofuel have been used in the automotive industry since its inception.

One of the first inventors to convince the people of the use of ethanol was a German named Nikolaus August Otto. Rudolf Diesel is the German inventor of the diesel engine. He designed his diesel engine to run in peanut oil and later Henry Ford designed the Model T car which was produced from 1903 to 1926. This car was completely designed to use hemp derived biofuel as fuel. However, with the exploration of huge supplies of crude oil some of the parts of Texas and Pennsylvania petroleum became very cheap and thus lead to the reduction of the use of biofuels. Most of the vehicles like trucks and cars began using this form of fuel which was much cheaper and efficient.

In the period of World War II, the high demand of biofuels was due to the increased use as an alternative for imported fuel. In this period, Germany was one of the countries that underwent a serious shortage of fuel. It was during this period that various other inventions took place like the use of gasoline along with alcohol that was derived from potatoes. Britain was the second country which came up with the concept of grain alcohol mixed with petrol. The wars frames were the periods when the various major technological changes took place but, during the period of peace, cheap oil from the gulf countries as well as the Middle East again eased off the pressure.

With the increased supply the geopolitical and economic interest in biofuel faded away. A serious fuel crisis again hit the various countries during the period of 1973 and 1979, because of the geopolitical conflict. Thus (OPEC), organization of the petroleum

Exporting countries made a heavy cut in exports especially to the non OPEC nations. The constant shortage of fuel attracted the attention of the various academics and governments to the issues of energy crisis and the use of biofuels. The twentieth century came with the attention of the people towards the use of biofuels. Some of the main reasons for the people shifting their interest to biofuels were the rising prices of oil, emission of the greenhouse gases and interest like rural development. ( [biofuel.org.uk](http://biofuel.org.uk))

Bio-fuels are better for the environment mainly because they contain no sulfur. When a fuel containing sulfur is burned, sulfur dioxide SO<sub>2</sub> is produced, which is a harmful gas that has been known to cause acid rain. Although the petroleum portion of the fuel still contains sulfur, the bio portion does not. Biofuels also reduce NOX emissions, which are greenhouse gasses. There are also many indirect environmental benefits of bio-fuels. For instance, the crops grown to produce bio-fuels such as soybeans consume a great deal of CO<sub>2</sub>, which is a greenhouse gas. Biodiesel (B100) is also non-toxic, biodegradable, and environmentally benign if spilled.

## **1.2 Research Background.**

Biofuel production as a renewable source are renewable, efficient, and clean. As petroleum becomes depleted, we grow ever more dependant on foreign sources of oil, often located in unstable parts of the world, and while biofuels make up only a small portion of the energy market, their role is becoming increasingly more vital every day. t Soaring prices of fossil fuels, geo-political issues and environmental pollution associated with fossil fuel use has led to worldwide interest in the production and use of bio-fuels. Both the developed and developing countries have developed a range of policies to encourage production of combustible fuels from plants that triggered public and private investments in bio-fuel crop research and development, and bio-fuels production. In this article, we discuss the potential benefits of bio-fuels in increasing the farmers' incomes, reducing environment pollution, the crop options and research and development interventions required to generate feed stocks to produce bio-fuels to meet projected demand without compromising food/fodder security in developing countries. Producing petrol from the waste of palm oil

(palmitic acid) will give an alternative choice to the users, especially for petrol engine vehicles' owners. In addition, this bio petrol, which is graded 100 for its octane number, burns very smoothly so bio petrol can reduce emissions of some pollutants. Research is currently being developed by teams of the of Chemical Engineering in Universiti Teknologi Petronas, (UTP) on the synthesise of biofuel from rubber seeds in Malaysia. It states that rubber-seed oil has high content of free fatty acids (FFAs). Besides, there was between 30% and 40% of oil in rubber seeds and 1kg of rubber seeds could produce between 300ml and 400ml of biodiesel fuel (M.T. Azizan, 2008). The natural form of rubber seed oil is highly acidic functional groups such as carbonyl, olefinic unsaturation, ester, glyceryl, methylene and terminal methyl are present in rubber seed oil. While, Chemical engineering lecturer Mohammad Tazli Azizan said that using rubber seeds is a better option because the oil in those seeds was suitable for use in cold countries, unlike palm oil. The rubber seed oil is not viscous and can be used in cold climates without much modification. There are between 30% and 40% of oil in rubber seeds. Rubber seeds were not edible but could be found in abundance in the country, adding that there were 1.2 million ha of rubber plantation in Malaysia. Using oil derived from rubber seeds instead of oil palms could easily avert the debate of a conflict between food and fuel.

### **1.3 Objectives**

- a) To synthesise Isooctane from rubber seeds
- b) To analyze the concentration of Isooctane by heterogeneous catalytic cracking of fatty acid using Zeolite as catalyst.

## **1.4 Problem Statement**

### **1.4.1 Peak Oil.**

Peak oil is the point in time when the maximum rate of global petroleum extraction is reached, after which the rate of production enters terminal decline. This concept is based on the observed production rates of individual oil wells, and the combined production rate of a field of related oil wells. The aggregate production rate from an oil field over time usually grows exponentially until the rate peaks and then declines, sometimes rapidly until the field is depleted. This concept is derived from the Hubbert curve, and has been shown to be applicable to the sum of a nation's domestic production rate, and is similarly applied to the global rate of petroleum production. Peak oil is often confused with oil depletion; peak oil is the point of maximum production while depletion refers to a period of falling reserves and supply.

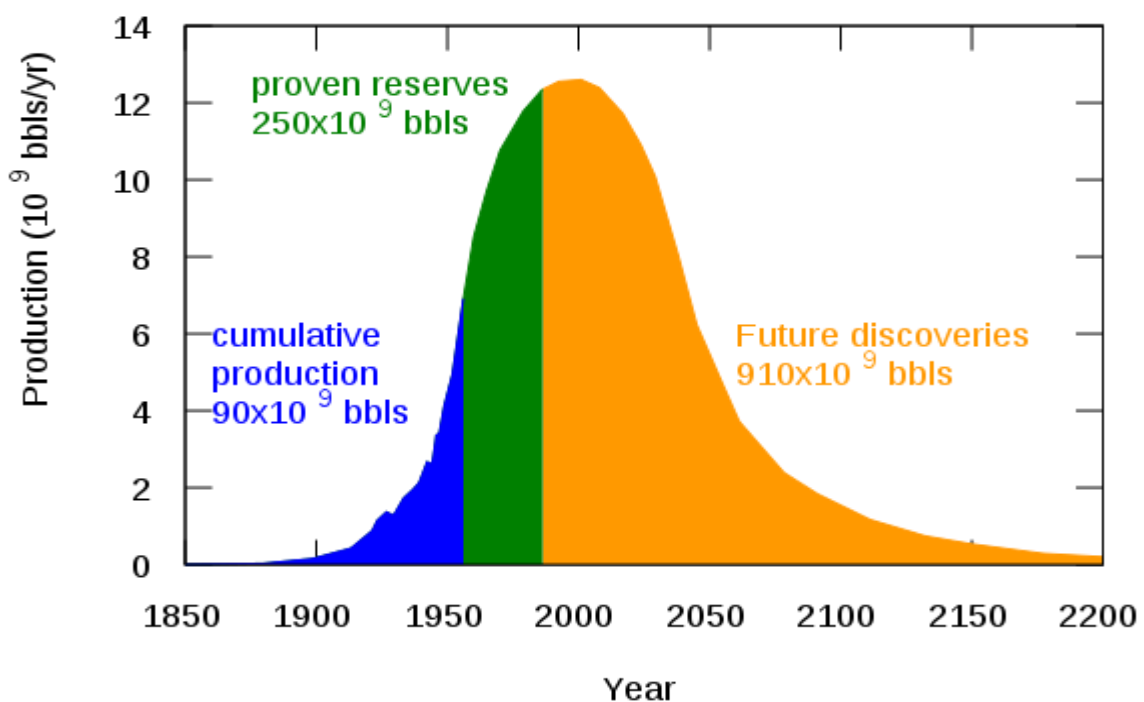
M. King Hubbert created and first used the models behind peak oil in 1956 to accurately predict that United States oil production would peak between 1965 and 1970. His logistic model, now called Hubbert peak theory, and its variants have described with reasonable accuracy the peak and decline of production from oil wells, fields, regions, and countries, and has also proved useful in other limited-resource production-domains. According to the Hubbert model, the production rate of a limited resource will follow a roughly symmetrical logistic distribution curve (sometimes incorrectly compared to a bell-shaped curve) based on the limits of exploitability and market pressures.

Some observers, such as petroleum industry experts Kenneth S. Deffeyes and Matthew Simmons, believe the high dependence of most modern industrial transport, agricultural, and industrial systems on the relative low cost and high availability of oil will cause the post-peak production decline and possible severe increases in the price of oil to have negative implications for the global economy. Predictions vary greatly as to what exactly these negative effects would be. If political and economic



changes only occur in reaction to high prices and shortages rather than in reaction to the threat of a peak, then the degree of economic damage to importing countries will largely depend on how rapidly oil imports decline post-peak.

Optimistic estimations of peak production forecast the global decline will begin by 2020 or later, and assume major investments in alternatives will occur before a crisis, without requiring major changes in the lifestyle of heavily oil-consuming nations. These models show the price of oil at first escalating and then retreating as other types of fuel and energy sources are used. Pessimistic predictions of future oil production operate on the thesis that either the peak has already occurred, that oil production is on the cusp of the peak, or that it will occur shortly. The International Energy Agency (IEA) says production of conventional crude oil peaked in 2006. Throughout the first two quarters of 2008, there were signs that a global recession was being made worse by a series of record oil prices.



**Figure 1.1:** A logistic distribution shaped production curve, as originally suggested by M. King Hubbert in 1956.

### 1.4.2 Demand For Oil

The demand side of peak oil is concerned with the consumption over time, and the growth of this demand. World crude oil demand grew an average of 1.76% per year from 1994 to 2006, with a high of 3.4% in 2003-2004. World demand for oil is projected to increase 37% over 2006 levels by 2030 (118 million barrels per day ( $18.8 \times 10^6 \text{ m}^3/\text{d}$ ) from 86 million barrels ( $13.7 \times 10^6 \text{ m}^3$ )), due in large part to increases in demand from the transportation sector. A study published in the journal Energy Policy predicted demand would surpass supply by 2015 (unless constrained by strong recession pressures caused by reduced supply).

Energy demand is distributed amongst four broad sectors: transportation, residential, commercial, and industrial. In terms of oil use, transportation is the largest sector and the one that has seen the largest growth in demand in recent decades. This growth has largely come from new demand for personal-use vehicles powered by internal combustion engines. This sector also has the highest consumption rates, accounting for approximately 68.9% of the oil used in the United States in 2006, and 55% of oil use worldwide as documented in the Hirsch report. Transportation is therefore of particular interest to those seeking to mitigate the effects of peak oil. Although demand growth is highest in the developing world, the United States is the world's largest consumer of petroleum. Between 1995 and 2005, U.S. consumption grew from 17,700,000 barrels per day ( $2,810,000 \text{ m}^3/\text{d}$ ) to 20,700,000 barrels per day ( $3,290,000 \text{ m}^3/\text{d}$ ), a 3,000,000 barrels per day ( $480,000 \text{ m}^3/\text{d}$ ) increase. China, by comparison, increased consumption from 3,400,000 barrels per day ( $541,000 \text{ m}^3/\text{d}$ ) to 7,000,000 barrels per day ( $1,100,000 \text{ m}^3/\text{d}$ ), an increase of 3,600,000 barrels per day ( $572,000 \text{ m}^3/\text{d}$ ), in the same time frame.

As countries develop, industry and higher living standards drive up energy use, most often of oil. Thriving economies such as China and India are quickly becoming large

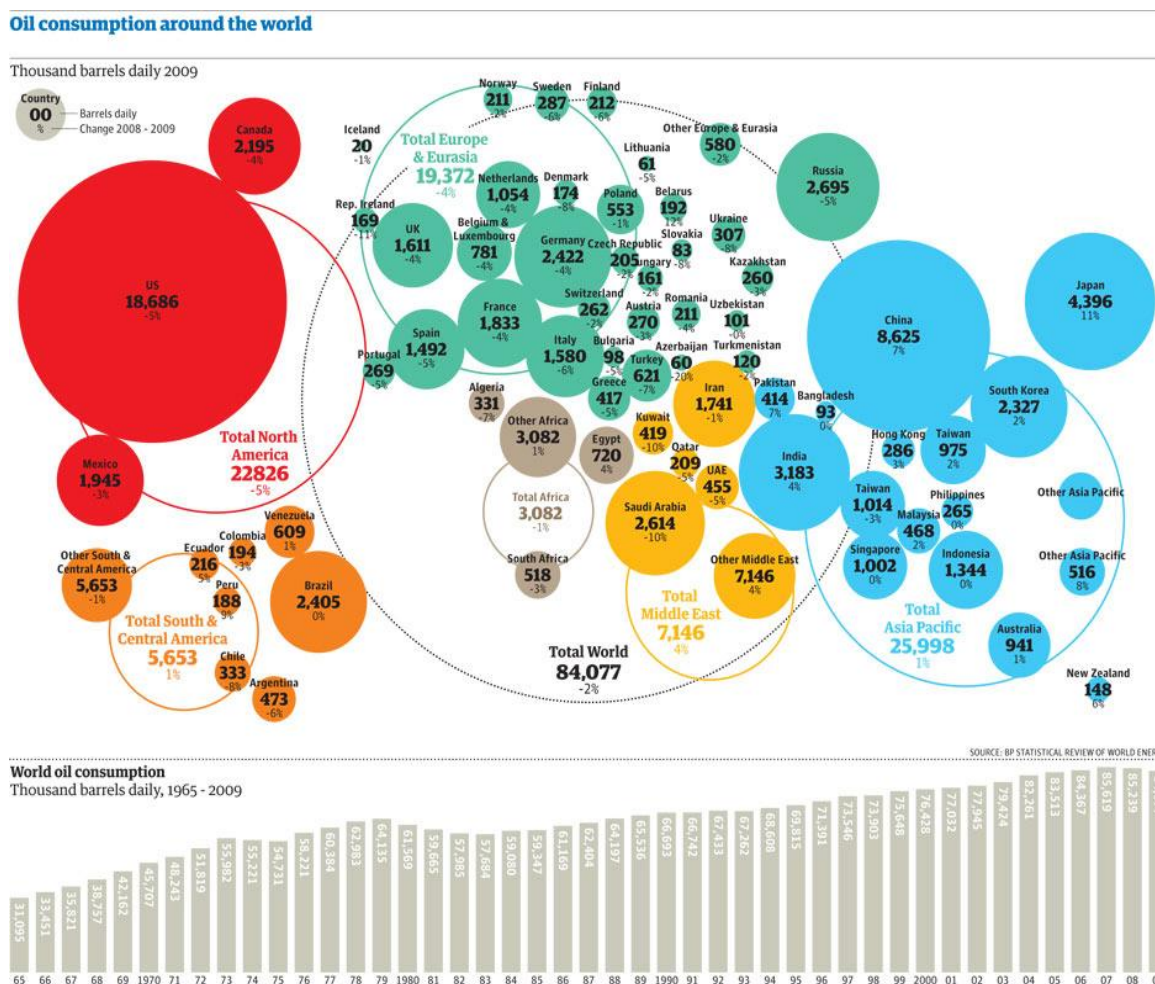
oil consumers. China has seen oil consumption grow by 8% yearly since 2002, doubling from 1996-2006. In 2008, auto sales in China were expected to grow by as much as 15-20%, resulting in part from economic growth rates of over 10% for 5 years in a row.

Although swift continued growth in China is often predicted, others predict that China's export dominated economy will not continue such growth trends due to wage and price inflation and reduced demand from the United States. India's oil imports are expected to more than triple from 2005 levels by 2020, rising to 5 million barrels per day ( $790 \times 10^3 \text{ m}^3/\text{d}$ ).

The International Energy Agency estimated in January 2009 that oil demand fell in 2008 by 0.3%, and that it would fall by 0.6% in 2009. Oil consumption had not fallen for two years in a row since 1982-1983.

The Energy Information Administration (EIA) estimated that the United States' demand for petroleum-based transportation fuels fell 7.1% in 2008, which is "the steepest one-year decline since at least 1950." The agency stated that gasoline usage in the United States may have peaked in 2007, in part due to increasing interest in and mandates for use of biofuels and energy efficiency.

The EIA now expects global oil demand to increase by about 1,600,000 barrels per day ( $254,000 \text{ m}^3/\text{d}$ ) in 2010. Asian economies, in particular China, will lead the increase. China's oil demand may rise more than 5% compared with a 3.7% gain in 2009, the CNPC said.



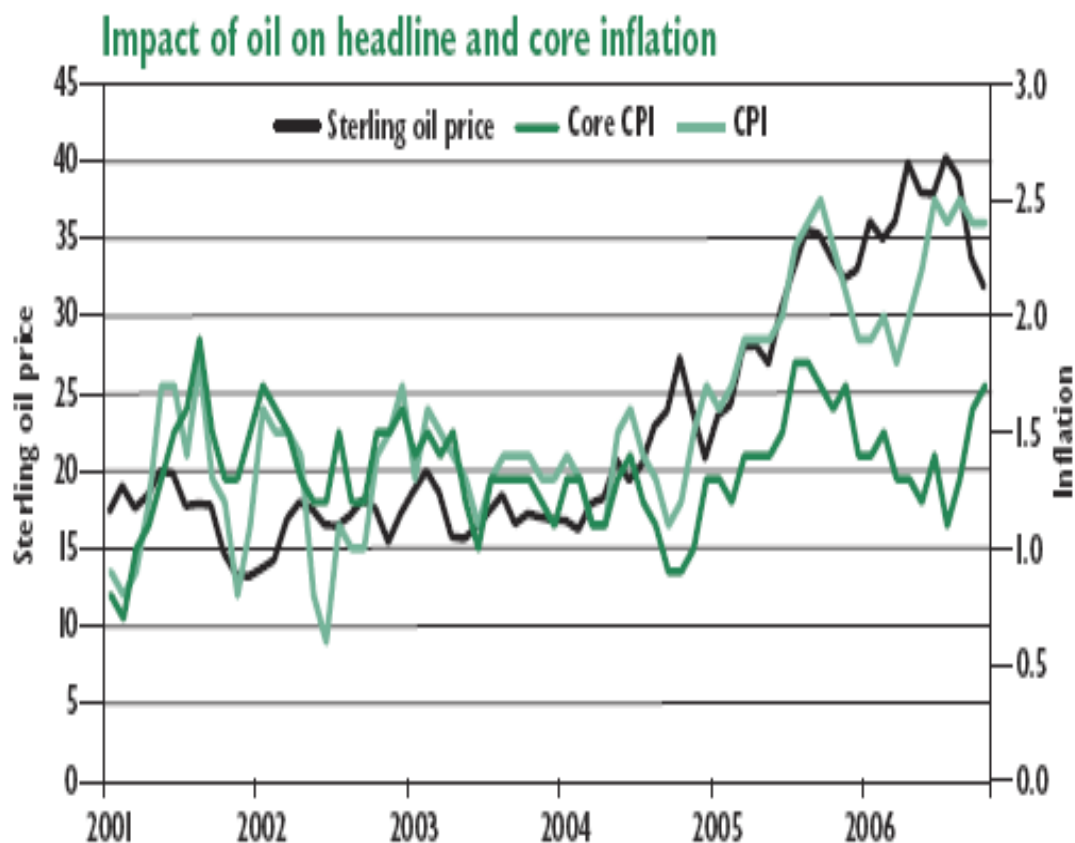
**Figure 1.2:** BP energy statistics: oil consumption around the world.

### 1.4.3 Oil Price In Malaysia And The Consequences.

The fossil fuels prices are very unstable and unpredictable. Furthermore, this price hike is estimated to be increasing for the next few more years. These changes of the prices of the fossil fuels have given a difficult time to the consumers to allocate a large sum of money to pay only for the petrol itself.

With the increasing prices of the fossil fuels, everything else indirectly will be more and more expensive. The manufacturing and transportation company will take this

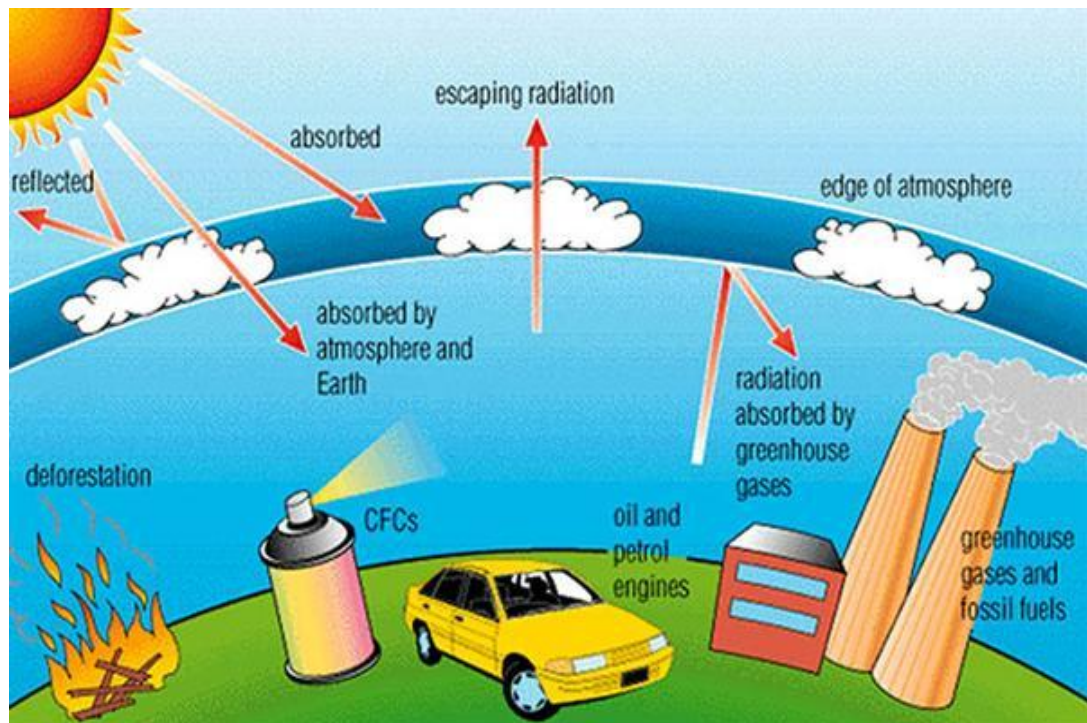
advantage to increase the costs. It will cause many domino effects in terms of goods and services cost as well as will affect the global economic growth and stability. Unfortunately to the consumers, the boss will never raise an increase the workers' salary. People with higher salary can still manage to pay for the price hike but for the lower income group, they will definitely feel the pinch. Bio-petrol is better than fuel from under the ground or drilled and refined fuel which is more expensive because of the cost of production. In addition, the bio-petrol is much cheaper than the fossil fuels because it is derived from biomass such as natural plants, vegetable crops like palm oil and rubber seeds as well as the agricultural or forestry waste. The process to produce bio-petrol is not as expensive as the fossil fuels. Therefore, it is believed that the bio-petrol will help to fulfill the demand of the world in the future.



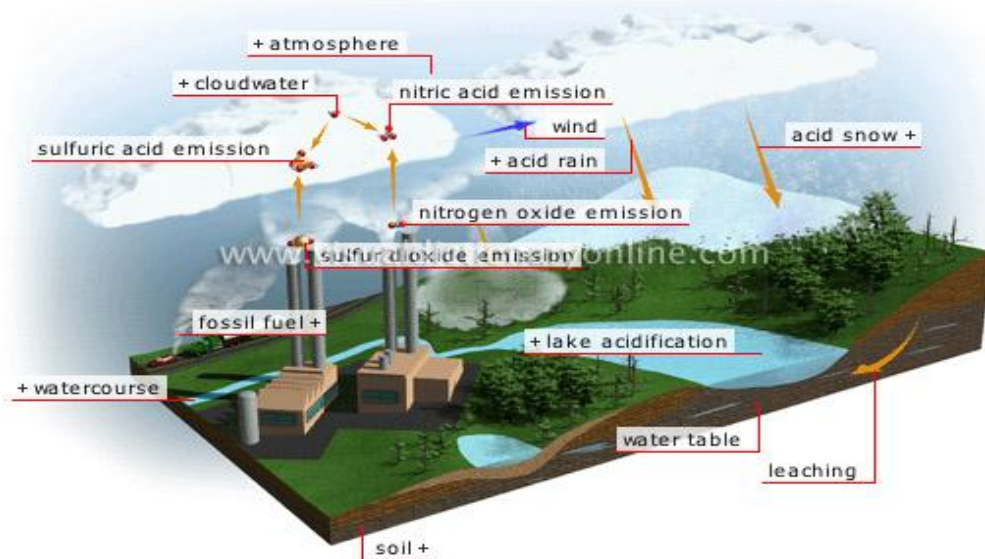
**Figure 1.3:** Impact of oil on headline and core inflation

#### **1.4.4 Disadvantages Of Fossil Fuel**

- Pollution is a major disadvantage of fossil fuels. This is because they give off carbon dioxide when burned thereby causing a greenhouse effect. This is also the main contributory factor to the global warming experienced by the earth today.
- Coal also produces carbon dioxide when burned compared to burning oil or gas. Additionally, it gives off sulphur dioxide, a kind of gas that creates acid rain.
- Environmentally, the mining of coal results in the destruction of wide areas of land. Mining this fossil fuel is also difficult and may endanger the lives of miners. Coal mining is considered one of the most dangerous jobs in the world.
- Power stations that utilize coal need large amounts of fuel. In other words, they not only need truckloads but trainloads of coal on a regular basis to continue operating and generating electricity. This only means that coal-fired power plants should have reserves of coal in a large area near the plant's location.
- Use of natural gas can cause unpleasant odors and some problems especially with transportation.
- Use of crude oil causes pollution and poses environmental hazards such as oil spills when oil tankers, for instance, experience leaks or drown deep under the sea. Crude oil contains toxic chemicals which cause air pollutants when combusted.



**Figure 1.4:** Greenhouse effect



**Figure 1.5:** Acid rain formation.



**Figure 1.6:** Oil spillage and harm to environment and other organisms.

#### **1.4.5 Advantages Of Biofuel.**

- Doesn't require any radical changes to switch to the use of biofuels- unlike the difficulties in switching to other renewable energy sources such as solar and wind power.
- Are cheaper than fossil fuels. Many governments are now offering tax incentives to buy greener cars that run on biofuels (ethanol being one example).
- Are considered 'carbon neutral' by some people. This is because the carbon dioxide they release when burnt is equal to the amount that the plants absorbed out of the atmosphere. Therefore, they don't contribute to global warming. However, it does require some fuel to power the machinery on the farms where biofuels are produced. Still, they are better than fossil fuels! Research suggests that they reduce carbon emissions by 50-60%.



- Reduce dependence on foreign oils. Oil fluctuates in price rapidly, so changing to biofuels will help buffer against the change.
- Emit less particulate pollution than other fuels, especially diesel.
- Are renewable sources of energy as you can just keep producing more.
- Ethanol is very inexpensive to produce.
- Can help prevent engine knocking.

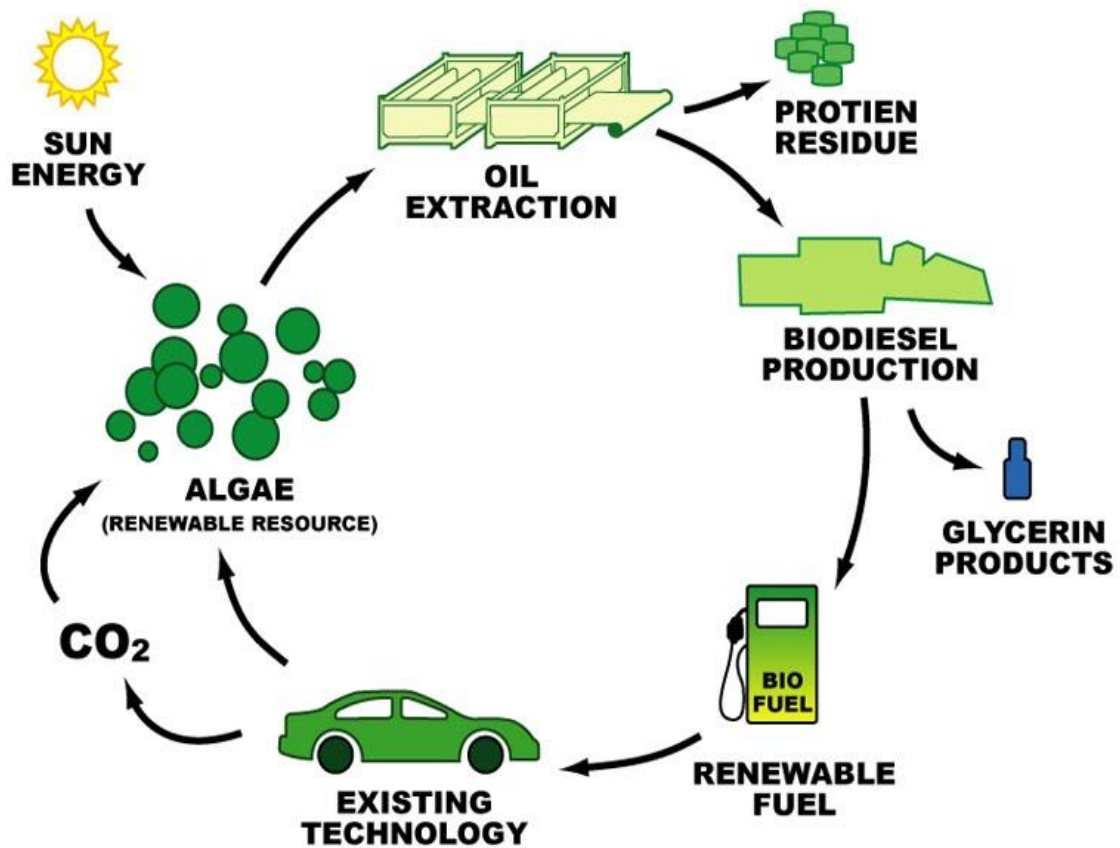


Figure 1.7: Biofuel cycle



**Figure 1.8:** Advantages of biofuel and its nature friendliness.

## 1.5 Research Scope

In order to accomplish the objectives, the scope of this research is focusing on the criteria that are stated as below:-

- i. The extraction of rubber seed oil from rubber seeds using Soxhlet Extraction
- ii. The application of catalytic cracking process to crack the fatty acid complex into smaller hydrocarbon molecules
- iii. The determination of the concentration of Isooctane through an analysis using the Gas Chromatography method

## 1.6 Rational And Significance

The Rationale and Significance statements are as below:-

- a) Bio-petrol is a renewable energy source and is biodegradable of which can be used as a substitute for the fossil fuels
- b) Bio-petrol is environment friendly as it brings about the reduction of greenhouse gases emission, sulphur free and non-aromatic
- c) Bio-petrol has oxygen levels from 10% to 45% while petroleum has essentially none making the chemical properties of bio-petrol very much useful than the petroleum
- d) Isooctane or 2,2,4-trimethylpentane is used to prevent knocking in the engines as it helps to improve the efficiency of combustion and increase the engine's performance
- e) Easy availability of fatty acid in most of the natural plants especially in rubber seeds
- f) Rubber seeds are easily available in Malaysia as Malaysia is one of the leading producers of natural rubber in the world (Indonesia, Malaysia and Thailand together account for around 72% of all natural rubber production.)

- g) Use of non-edible feedstock such as rubber seeds in bio-petrol production would reduce the feedstock cost
- h) Catalytic cracking provide higher conversion of hydrocarbon than thermal cracking does by lowering the activation energy of the reaction.

## **CHAPTER 2**

### **2.0 LITERATURE REVIEW**

#### **2.1 Introduction**

##### **2.1.1 Fuel**

Fuel is any material that stores energy that can later be extracted to perform mechanical work in a controlled manner. A fuel contains energy, mostly heat, that can be released and then manipulated. Most fuels used by humans undergo combustion, a redox reaction in which a combustible substance releases energy after it ignites and reacts with the oxygen in the air. Other processes used to convert fuel into energy include various other exothermic chemical reactions and nuclear reactions, such as nuclear fission or nuclear fusion. Fuels are also used in the cells of organisms in a process known as cellular respiration, where organic molecules are oxidized to release usable energy. Hydrocarbons are by far the most common source of fuel used by humans, but many other substances, such as radioactive metals, are currently used as well.

### 2.1.2 Fossil Fuel

Fossil fuels are fuels formed by natural resources such as anaerobic decomposition of buried dead organisms. The age of the organisms and their resulting fossil fuels is typically millions of years, and sometimes exceeds 650 million years. The fossil fuels, which contain high percentages of carbon, include coal, petroleum, and natural gas. Fossil fuels range from volatile materials with low carbon:hydrogen ratios like methane, to liquid petroleum to nonvolatile materials composed of almost pure carbon, like anthracite coal. Methane can be found in hydrocarbon fields, alone, associated with oil, or in the form of methane clathrates. It is generally accepted that they formed from the fossilized remains of dead plants and animals by exposure to heat and pressure in the Earth's crust over millions of years. This biogenic theory was first introduced by Georg Agricola in 1556 and later by Mikhail Lomonosov in the 18th century.

It was estimated by the Energy Information Administration that in 2007 primary sources of energy consisted of petroleum 36.0%, coal 27.4%, natural gas 23.0%, amounting to an 86.4% share for fossil fuels in primary energy consumption in the world. Non-fossil sources in 2006 included hydroelectric 6.3%, nuclear 8.5%, and others (geothermal, solar, tide, wind, wood, waste) amounting to 0.9 percent. World energy consumption was growing about 2.3% per year.

Fossil fuels are non-renewable resources because they take millions of years to form, and reserves are being depleted much faster than new ones are being made. The production and use of fossil fuels raise environmental concerns. A global movement toward the generation of renewable energy is therefore under way to help meet increased energy needs.

The burning of fossil fuels produces around 21.3 billion tonnes (21.3 gigatonnes) of carbon dioxide (CO<sub>2</sub>) per year, but it is estimated that natural processes can only absorb about half of that amount, so there is a net increase of 10.65 billion tonnes of atmospheric carbon dioxide per year (one tonne of atmospheric carbon is equivalent to 44/12 or 3.7 tonnes of carbon dioxide). Carbon dioxide is one of the greenhouse gases that enhances radiative forcing and contributes to global warming, causing the average surface

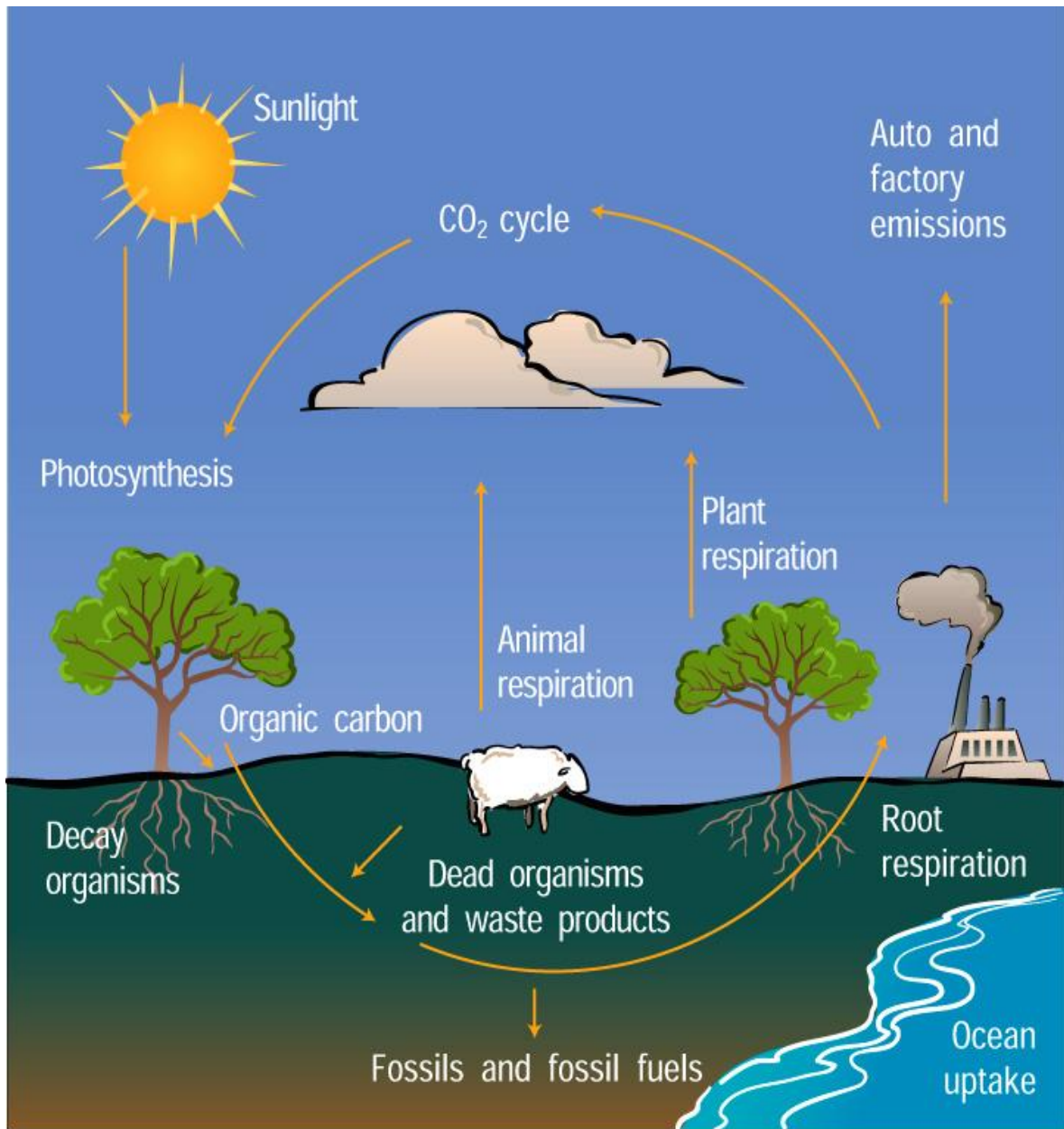
temperature of the Earth to rise in response, which most climate scientists agree will cause major adverse effects.

### **2.1.3 Origin Of Fossil Fuel.**

Petroleum and natural gas are formed by the anaerobic decomposition of remains of organisms including phytoplankton and zooplankton that settled to the sea (or lake) bottom in large quantities under anoxic conditions, millions of years ago. Over geological time, this organic matter, mixed with mud, got buried under heavy layers of sediment. The resulting high levels of heat and pressure caused the organic matter to chemically alter, first into a waxy material known as kerogen which is found in oil shales, and then with more heat into liquid and gaseous hydrocarbons in a process known as catagenesis.

There is a wide range of organic, or hydrocarbon, compounds in any given fuel mixture. The specific mixture of hydrocarbons gives a fuel its characteristic properties, such as boiling point, melting point, density, viscosity, etc. Some fuels like natural gas, for instance, contain only very low boiling, gaseous components. Others such as gasoline or diesel contain much higher boiling components.

Terrestrial plants, on the other hand, tend to form coal and methane. Many of the coal fields date to the Carboniferous period of Earth's history. Terrestrial plants also form type III kerogen, a source of natural gas.



**Figure 2.1:** Fossil fuel cycle



#### **2.1.4 Biofuel**

Biofuels are a wide range of fuels which are in some way derived from biomass. The term covers solid biomass, liquid fuels and various biogases. Biofuels are gaining increased public and scientific attention, driven by factors such as oil price spikes, the need for increased energy security, and concern over greenhouse gas emissions from fossil fuels.

Bioethanol is an alcohol made by fermenting the sugar components of plant materials and it is made mostly from sugar and starch crops. With advanced technology being developed, cellulosic biomass, such as trees and grasses, are also used as feedstocks for ethanol production. Ethanol can be used as a fuel for vehicles in its pure form, but it is usually used as a gasoline additive to increase octane and improve vehicle emissions. Bioethanol is widely used in the USA and in Brazil.

Biodiesel is made from vegetable oils, animal fats or recycled greases. Biodiesel can be used as a fuel for vehicles in its pure form, but it is usually used as a diesel additive to reduce levels of particulates, carbon monoxide, and hydrocarbons from diesel-powered vehicles. Biodiesel is produced from oils or fats using transesterification and is the most common biofuel in Europe.

Biofuels provided 1.8% of the world's transport fuel in 2008. Investment into biofuels production capacity exceeded \$4 billion worldwide in 2007 and is growing.

#### **2.1.5 Petrol.**

Petrol or also known as gasoline is a petroleum-derived liquid mixture which is primarily used as a fuel in internal combustion engines. It is also used as a solvent, mainly known for its ability to dilute paints. It consists mostly of aliphatic hydrocarbons obtained by the fractional distillation of petroleum, enhanced with iso-octane or the aromatic hydrocarbons toluene and benzene to increase its octane rating. Small quantities of various additives are common, for purposes such as tuning engine performance or reducing harmful exhaust emissions. Some mixtures also contain significant quantities of ethanol as a partial alternative fuel. Petrol is a complex mixture of hydrocarbons which consists a mixture of C4 to C10 alkanes. However isooctane (C<sub>8</sub>H<sub>18</sub>) as dominative component in

petrol is assigned an octane number of 100. Isooctane or 2, 2, 4-trimethylpentane ( $\text{CH}_3\text{C}(\text{CH}_3)_2\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_3$ ) 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.

### **2.1.6 Biopetrol In The Future**

Biopetrol is an alternative fuel that is a renewable energy with a lower cost rather than petrol itself. Biopetrol from palm oil is biodegradable and non-toxic. From the environmental point view, all the fuel from vegetable sources is environmentally friendly. From the technical point of view, biofuel (biopetrol) is technically compatible with current internal combustion engine. Slight modification might be required to enhance the power. Biopetrol can be the solution for the future air emission control. The application of existing biodiesel from palm oil in motor vehicle (diesel engine) has been proven successful. However its commercial viability requires further in depth study (Mansur, 2005).

There are four primary reasons for encouraging the development of biopetrol in Malaysia:

- Provides market for excess production of palm oil.
- The prices of petroleum are increasing.
- Decreases the total import of petroleum.
- Fuel (biopetrol) from vegetable oil is renewable and environmental friendly.

### **2.1.7 Biopetrol From Palmitic Acid**

Palmitic acid or hexadecanoic acid ( $\text{CH}_3(\text{CH}_2)_{14}\text{COOH}$ ) is a fatty acid which is found in animal fats and vegetable oils. To produce isooctane from palmitic acid, thermal cracking process is needed. Heat is supplied at palmitic acid's melting point within a range of  $63^\circ\text{C} - 64^\circ\text{C}$  to melt the solid palmitic acid. After it turns to the liquid, the heating must be continuous until the isooctane's boiling point of  $98^\circ\text{C}$  by using heating mantle to form new arrangements of carbon compounds including isooctane. The sample produced will have lots of hydrocarbon chains because of the heat that breaks the carbon chain randomly. Alkanes from C5 until C9 are categorized as gasoline.

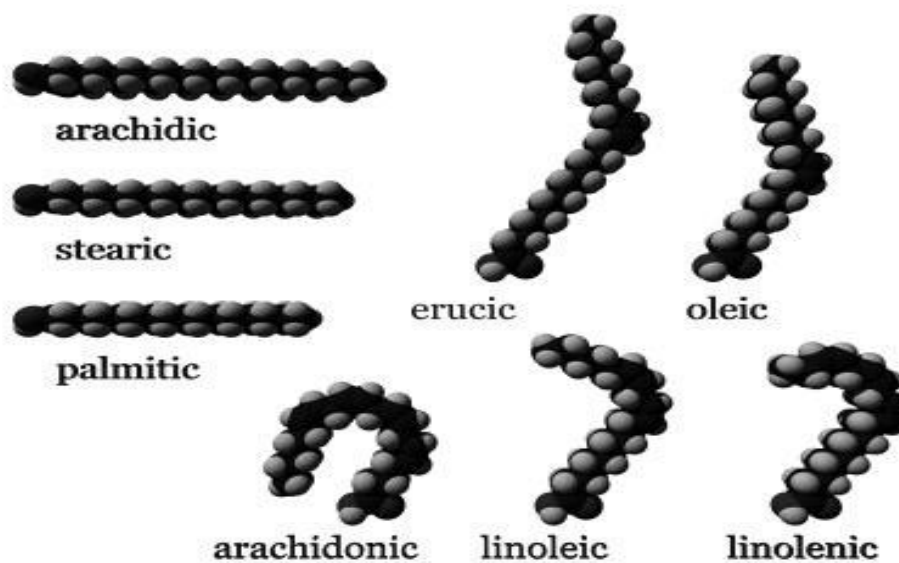
### **2.1.8 Bio-petrol From Fatty Acids In Vegetable Oil**

Vegetable oils are becoming a promising alternative to biofuel because they are renewable in nature and can be produced locally and environmental friendly as well. They have practically no sulfur content, offer no storage difficulty, and they have excellent lubrication properties. Moreover, vegetable oils yielding trees absorb more carbon dioxide from the atmosphere during their photosynthesis than they add to the atmosphere on burning. Hence, they essentially help to alleviate the increasing carbon dioxide content in the atmosphere. The substitution of diesel oil by renewable fuels produced within the country generates higher foreign exchange savings, even for the major oil exporting countries. Therefore, developing countries can use this kind of project not only to solve their ecological problems but also to improve their economy. In view of the several advantages vegetable oils has potential to replace petroleum-based fuels in the long run.

In the recent years, systematic efforts have been made by several researchers and to use the various vegetable oils as fuel in compression ignition engines. The calorific value of vegetable oil is comparable to that of diesel. However, their use in direct injection on engines is restricted by some unfavorable physical properties, particularly their viscosity. The viscosity of vegetable oil is about ten times higher than that of petrol. Therefore, the

vegetable oil cause poor fuel atomization, incomplete combustion and carbon deposition on the injector and valve seats resulting in serious engine fouling. This necessitates the reduction in viscosity of the vegetable oils for use as fuel in CI engines. The commonly employed methods to reduce the viscosity of vegetable oils are blending with petrol, emulsification, pyrolysis, cracking and transesterification. Among these, transesterification of vegetable oils appears to be more suitable because the byproduct (glycerol) has commercial value.

Transesterification (alcoholysis) is the chemical reaction between triglycerides and alcohol in the presence of catalyst to produce mono-esters. The long and branched chain triglyceride molecules are transformed to mono-esters and glycerin. Transesterification process consists of a sequence of three consecutive reversible reactions. That is, conversion of triglycerides to diglycerides, followed by the conversion of diglycerides to monoglycerides. The glycerides are converted into glycerol and yielding one ester molecule in each step. The properties of these esters are comparable to that of petrol.



**Figure 2.2:** Fatty acids

### 2.1.9 Characterization Of Rubber Seed Oil

The annual rubber seed production potential in India is about 150 kg per hectare. Rubber seed kernels (50–60% of seed) contain 40–50% of brown color oil. The estimated availability of rubber seeds in India is about 30,000 tons per annum, which can yield rubber seed oil to the tune of about 5000 tons. Rubber trees yield 3-seeded ellipsoidal capsule, each carpel with one seed. Rubber seeds are ellipsoidal, variable in size, 2.5–3 cm long, mottled brown, lustrous, weighing 2–4 g each. Capsules are spread over the ground. These are collected and kernels are separated by breaking the capsules. These kernels are dried to remove the moisture. The kernels are crushed in the crushers and the oil is filtered. At present rubber seed oil does not find any major applications and hence even the natural production of seeds itself remain underutilized. The filtered oil is used as feedstock for the biodiesel production in this study.

The fatty acid composition and the important properties of rubber seed oil in comparison with other oils is given in Table 1. It consists of 18.9% saturation comprising of palmitic and stearic acids and 80.5% unsaturation comprising mainly of oleic, linoleic and linolenic acids. Saturation fatty acid methyl esters increase the cloud point, cetane number and improve stability whereas more polyunsaturates reduce the cloud point and cetane number and stability. The type and percentage of fatty acids contained in vegetable oil depends on the plant species and on the growth conditions of the plant. Though vegetable oils are of very low volatility in nature, it quickly produces volatile combustible compounds upon heating.

**Table 2.1:** Properties of rubber seed oil in comparison with the other oils

Property	Rubber seed oil	Sunflower oil	Rapeseed oil	Cotton seed oil	Soybean oil
Fatty acid composition (%)					
(i) Palmitic acid C <sub>16:0</sub>	10.2	6.8	3.49	11.67	11.75
(ii) Stearic acid C <sub>18:0</sub>	8.7	3.26	0.85	0.89	3.15
(iii) Oleic acid C <sub>18:1</sub>	24.6	16.93	64.4	13.27	23.26
(iv) Linoleic acid C <sub>18:2</sub>	39.6	73.73	22.3	57.51	55.53
(v) Linolenic acid C <sub>18:3</sub>	16.3	0	8.23	0	6.31
Specific gravity	0.91	0.918	0.914	0.912	0.92
Viscosity (mm <sup>2</sup> /s) at 40 °C	66.2	58	39.5	50	65
Flash point (°C)	198	220	280	210	230
Calorific value (MJ/kg)	37.5	39.5	37.6	39.6	39.6
Acid value	34	0.15	1.14	0.11	0.2

**Figure 2.3** Rubber seed

The free fatty acid content of unrefined rubber seed oil was about 17%, i.e. acid value of 34. The yield of esterification process decreases considerably if FFA value is greater than 2%. Canakci and Van Gerpan and found that transesterification would not occur if FFA content in the oil were about 3%. It has been found that the alkaline catalyzed transesterification process is not suitable to produce esters from unrefined oils. In order to reduce the acid value the oil is to be refined. Refining of vegetable oils increases the overall production cost of the biofuel. Acid esterification is a typical method of producing biodiesel from high FFA oil . But it requires more methanol and is time consuming also. Development of any method to produce the biodiesel from high FFA oils is significant. Hence, the efforts are made to esterify a typical high FFA type of oil, i.e. rubber seed oil in this study.

**Table 2.2:.** Physicochemical properties of crude and refined (bleached) rubber seed oil

Analysis	Crude RSO	Refined RSO
Physical state at 30°C	Liquid	Liquid
Colour	Golden yellow	Golden yellow
Specific gravity at 30°C	0.922	0.918
Refractive index at 40°C	1.4654	1.4650
Viscosity at 30°C	41.24	37.85
Smoke point(°C)	245	244
Flash point (°C)	294	290
Fire point (°C)	345	345
Acid value	4.0	1.0
Free fatty acid	2.0	0.5
Peroxide value (meg/kg)	2.5	1.0
Iodine value	142.6	142.6
Saponification value	194.0	185.8
Unsaponification matter	1.18	0.16

The proximate composition of rubber seed kernel indicates that the seed contains 45.63% oil, 2.71% ash, 3.71% moisture, 22.17% protein, and 24.21% carbohydrate. The result shows that rubber seed is a potential source of oil and hence justifies the study on possible industrial uses. However the moisture content is moderately high and may cause a serious degradation of the fatty materials by hydrolysis, thereby rendering the material poor in stability. Drying the kernel at 60°C for 24 h and storing in almost air-tight container have been found to improve storage (Njoku, 1993).

The physical and chemical properties of the crude and bleached oils are shown in Table 1. From the result, bleaching of the oil decreased the acid value, peroxide value, unsaponifiable matter, and viscosity. This could be explained by the fact that bleaching removes impurities and contaminants present in the oil which include pigments, soaps, non-hydratable lecithins, oxidized fats and many other non-lipid materials (Hoffmann, 1986).

## **2.2 Catalytic Cracking(CC)**

Catalytic cracking is the most important conversion process used in petroleum refineries. It is widely used to convert the high-boiling, high-molecular weight hydrocarbon fractions of petroleum crude oils to more valuable gasoline, olefinic gases and other products. Cracking of petroleum hydrocarbons was originally done by thermal cracking which has been almost completely replaced by catalytic cracking because it produces more gasoline with a higher octane rating. It also produces byproduct gases that are more olefinic, and hence more valuable, than those produced by thermal cracking. The feedstock to a CC is usually that portion of the crude oil that has an initial boiling point of 340 °C or higher at atmospheric pressure and an average molecular weight ranging from about 200 to 600 or higher. This portion of crude oil is often referred to as heavy gas oil. The CC process vaporizes and breaks the long-chain molecules of the high-boiling hydrocarbon liquids into much shorter molecules by contacting the feedstock, at high temperature and moderate pressure, with a fluidized powdered catalyst. In effect, refineries use fluid catalytic cracking to correct the imbalance between the market demand



for gasoline and the excess of heavy, high boiling range products resulting from the distillation of crude oil.

In the production of the bio-petrol using rubber seeds, the main component to be made into bio-petrol is actually the fatty acid. Like what have been discussed earlier, fatty acid is a long unbranched aliphatic chain carboxylic acid. The complex structure of the fatty acid is very hard to react with any other substances. Therefore, for any reactions to occur, the fatty acid has to be broken into simpler chain molecules.

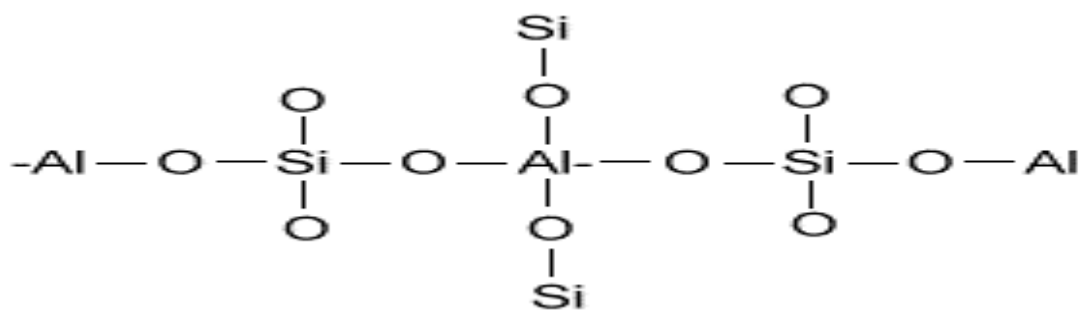
For this study, heterogeneous catalytic cracking is applied. Catalytic cracking is a route to produce liquid fuels that contain linear and cyclic paraffins, olefins, aldehydes, ketones, and carboxylic acid. Catalytic cracking process has been widely used in oil industry. It is widely used to convert the high-boiling, high-molecular weight hydrocarbon fractions of petroleum crude oils to more valuable gasoline, olefinic gases and other products [James H. Gary and Glenn E. Handwerk, 2001][ James. G. Speight, 2006][ Reza Sadeghbeigi, 2000]. The petroleum refinery process in which heavy oil is passed through metal chambers (called catalytic crackers or cat crackers) under pressure and high temperature in the presence of catalysts. This boiling breaks up heavy, large and more complex long-chain oil molecules into lighter, smaller, and simpler short-chain molecules by the breaking of carbon-carbon bonds in the precursors. The rate of cracking is dependent on the temperature and pressure applied as well as the presence of the catalyst. Temperature, heating rate, residence time and type of catalyst choice are important process control parameters (Ni et al., 2006).

Zeolite is used as an acid catalyst because it is very well known for its adsorbent qualities and has been used successfully in scientific trials . This characteristic of Zeolite is very important because as it helps to boost up the rate of reaction for the production of bio-petrol and it provides more surfaces for reactions to occur.

## 2.3 Zeolites As Heterogenous Catalyst

### 2.3.1 Introduction Of Zeolite

Zeolites are microporous, aluminosilicate minerals commonly used as commercial adsorbents. The term zeolite was originally coined in 1756 by Swedish mineralogist Axel Fredrik Cronstedt, who observed that upon rapidly heating the material stilbite, it produced large amounts of steam from water that had been adsorbed by the material. Based on this, he called the material zeolite, from the Greek ζέω (zeō), meaning "boil" and λίθος (lithos), meaning "stone". As of November 2010, 194 unique zeolite frameworks have been identified, and over 40 naturally occurring zeolite frameworks are known. Zeolites are widely used in industry for water purification, as catalysts, and in nuclear reprocessing. Their biggest use is in the production of laundry detergents. They are also used in medicine and in agriculture.



**Figure 2.4:** Basic zeolite structure

### 2.3.2 Properties Of Zeolite

Zeolites have a porous structure that can accommodate a wide variety of cations, such as  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and others. These positive ions are rather loosely held and can readily be exchanged for others in a contact solution. Some of the more common mineral zeolites are analcime, chabazite, clinoptilolite, heulandite, natrolite, phillipsite, and stilbite. An example mineral formula is:  $\text{Na}_2\text{Al}_2\text{Si}_3\text{O}_{10}\cdot 2\text{H}_2\text{O}$ , the formula for natrolite.

Natural zeolites form where volcanic rocks and ash layers react with alkaline ground water. Zeolites also crystallize in post-depositional environments over periods ranging from thousands to millions of years in shallow marine basins. Naturally occurring zeolites are rarely pure and are contaminated to varying degrees by other minerals, metals, quartz, or other zeolites. For this reason, naturally occurring zeolites are excluded from many important commercial applications where uniformity and purity are essential.

Zeolites are the aluminosilicate members of the family of microporous solids known as "molecular sieves." The term molecular sieve refers to a particular property of these materials, i.e., the ability to selectively sort molecules based primarily on a size exclusion process. This is due to a very regular pore structure of molecular dimensions. The maximum size of the molecular or ionic species that can enter the pores of a zeolite is controlled by the dimensions of the channels. These are conventionally defined by the ring size of the aperture, where, for example, the term "8-ring" refers to a closed loop that is built from 8 tetrahedrally coordinated silicon (or aluminum) atoms and 8 oxygen atoms. These rings are not always perfectly symmetrical due to a variety of effects, including strain induced by the bonding between units that are needed to produce the overall structure, or coordination of some of the oxygen atoms of the rings to cations within the structure. Therefore, the pores in many zeolites are not cylindrical.

### 2.3.3 Availability Of Zeolite

Conventional open pit mining techniques are used to mine natural zeolites. The overburden is removed to allow access to the ore. The ore may be blasted or stripped for processing by using tractors equipped with ripper blades and front-end loaders. In processing, the ore is crushed, dried, and milled. The milled ore may be air-classified as to particle size and shipped in bags or bulk. The crushed product may be screened to remove fine material when a granular product is required, and some pelletized products are produced from fine material.

Currently, the world's annual production of natural zeolite is about 4 million tons. Of this quantity, 2.6 million tons are shipped to Chinese markets to be used in the concrete industry. Eastern Europe, Western Europe, Australia, and Asia are world leaders in supplying the world's demand for natural zeolite. By comparison, only 57,400 metric tons of zeolite (only 1% of the world's current production) is produced in North America; only recently has North America realized the potential for current and future markets.

There are several types of synthetic zeolites that form by a process of slow crystallization of a silica-alumina gel in the presence of alkalis and organic templates. One of the important processes used to carry out zeolite synthesis is sol-gel processing. The product properties depend on reaction mixture composition, pH of the system, operating temperature, pre-reaction 'seeding' time, reaction time as well as the templates used. In sol-gel process, other elements (metals, metal oxides) can be easily incorporated. The silicalite sol formed by the hydrothermal method is very stable. Also the ease of scaling up this process makes it a favorite route for zeolite synthesis.

Synthetic zeolites hold some key advantages over their natural analogs. The synthetics can, of course, be manufactured in a uniform, phase-pure state. It is also possible to manufacture desirable zeolite structures which do not appear in nature. Zeolite A is a well-known example. Since the principal raw materials used to manufacture zeolites are silica and alumina, which are among the most abundant mineral components on earth, the potential to supply zeolites is virtually unlimited. Finally, zeolite manufacturing processes

engineered by man require significantly less time than the 50 to 50,000 years prescribed by nature. Disadvantages include the inability to create crystals with dimensions of a comparable size to their natural counterparts.



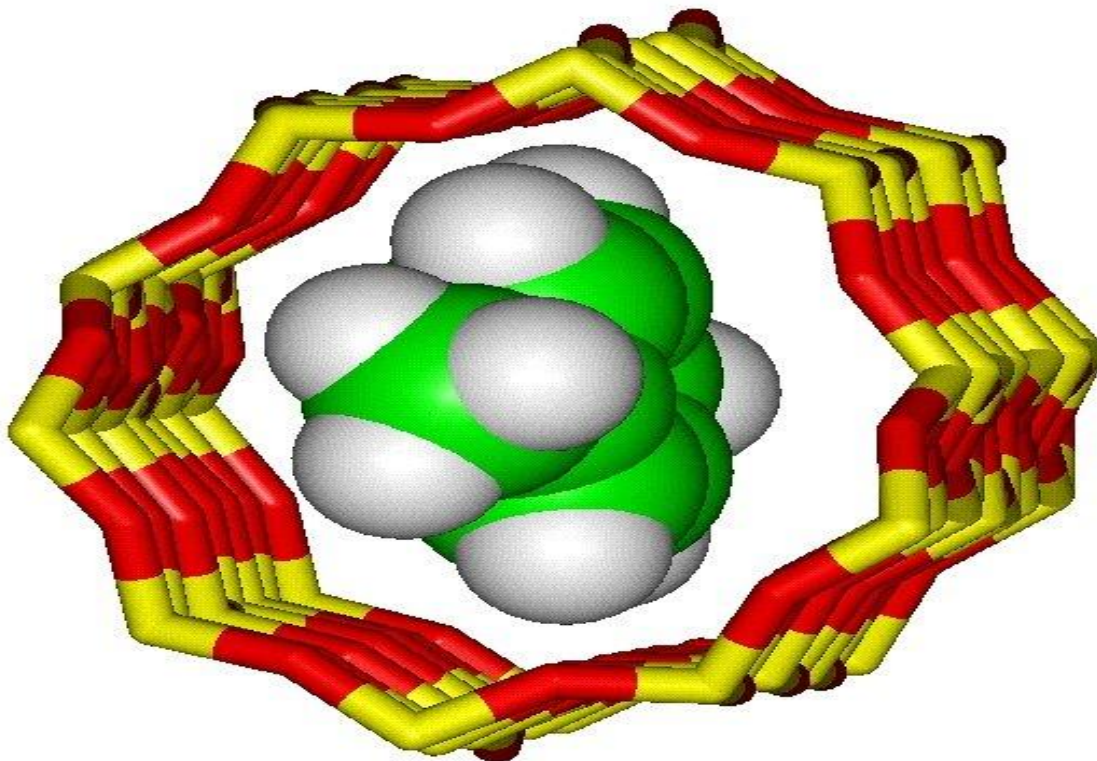
**Figure 2.5:** Zeolites in granule bulks

### **2.3.4 Usage And Speciality Of Zeolite**

#### **2.3.4.1 Adsorption And Separation**

The shape-selective properties of zeolites are also the basis for their use in molecular adsorption. The ability preferentially to adsorb certain molecules, while excluding others, has opened up a wide range of molecular sieving applications. Sometimes it is simply a matter of the size and shape of pores controlling access into the zeolite. In other cases different types of molecule enter the zeolite, but some diffuse through the channels more quickly, leaving others stuck behind, as in the purification of para-xylene by silicalite. Cation-containing zeolites are extensively used as desiccants due to their high affinity for water, and also find application in gas separation, where molecules are differentiated on the basis of their electrostatic interactions with the metal ions. Conversely,

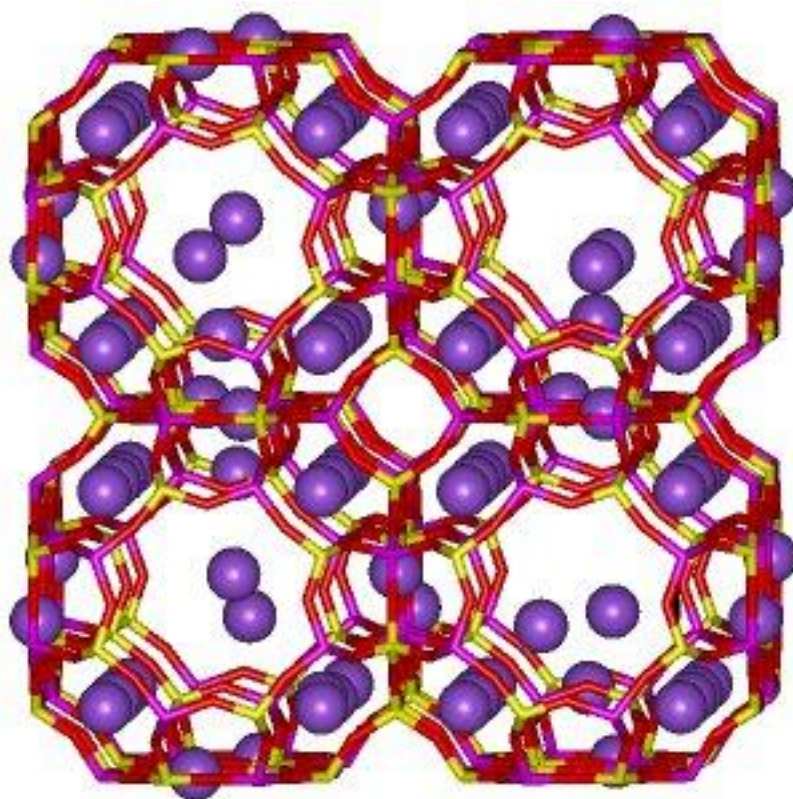
hydrophobic silica zeolites preferentially absorb organic solvents. Zeolites can thus separate molecules based on differences of size, shape and polarity.



**Figure 2.6:** The shape of para-xylene means that it can diffuse freely in the channels of silicalite

#### 2.3.4.2 Ion Exchange

The loosely-bound nature of extra-framework metal ions (such as in zeolite NaA, right) means that they are often readily exchanged for other types of metal when in aqueous solution. This is exploited in a major way in water softening, where alkali metals such as sodium or potassium prefer to exchange out of the zeolite, being replaced by the "hard" calcium and magnesium ions from the water. Many commercial washing powders thus contain substantial amounts of zeolite. Commercial waste water containing heavy metals, and nuclear effluents containing radioactive isotopes can also be cleaned up using such zeolites.



**Figure 2.7:** Sodium Zeolite A, used as a water softener in detergent powder

### 2.3.4.3 Zeolites And The Environment

Zeolites contribute to a cleaner, safer environment in a great number of ways. In fact nearly every application of zeolites has been driven by environmental concerns, or plays a significant role in reducing toxic waste and energy consumption. In powder detergents, zeolites replaced harmful phosphate builders, now banned in many parts of the world because of water pollution risks. Catalysts, by definition, make a chemical process more efficient, thus saving energy and indirectly reducing pollution. Moreover, processes can be carried out in fewer steps, minimising unnecessary waste and by-products. As solid acids, zeolites reduce the need for corrosive liquid acids, and as redox catalysts and sorbents, they can remove atmospheric pollutants, such as engine exhaust gases and ozone-

depleting CFCs. Zeolites can also be used to separate harmful organics from water, and in removing heavy metal ions, including those produced by nuclear fission, from water.

## 2.4 Extraction

Liquid-liquid extraction, also known as solvent extraction and partitioning, is a method to separate compounds based on their relative solubilities in two different immiscible liquids, usually water and an organic solvent. It is an extraction of a substance from one liquid phase into another liquid phase. Liquid-liquid extraction is a basic technique in chemical laboratories, where it is performed using a separatory funnel. This type of process is commonly performed after a chemical reaction as part of the work-up.

The term partitioning is commonly used to refer to the underlying chemical and physical processes involved in liquid-liquid extraction but may be fully synonymous. The term solvent extraction can also refer to the separation of a substance from a mixture by preferentially dissolving that substance in a suitable solvent. In that case, a soluble compound is separated from an insoluble compound or a complex matrix.

Solvent extraction is used in nuclear reprocessing, ore processing, the production of fine organic compounds, the processing of perfumes, the production of vegetable oils and biodiesel, and other industries.

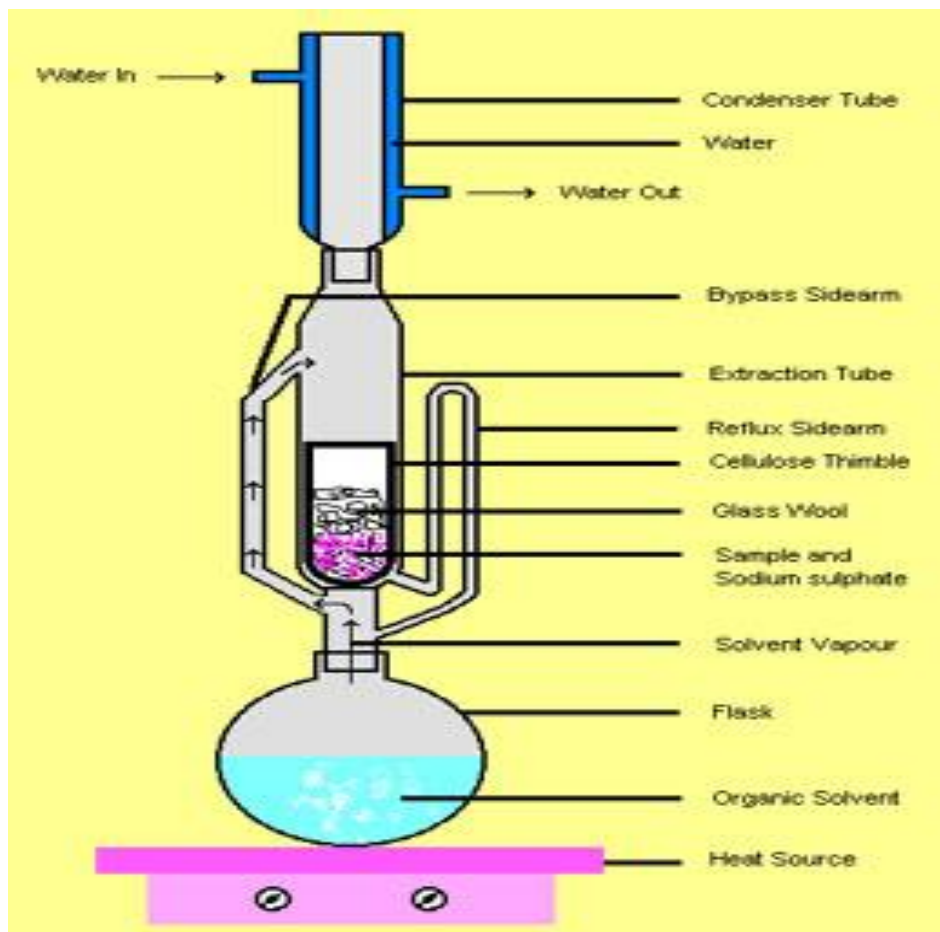
Liquid-liquid extraction is possible in non-aqueous systems: In a system consisting of a molten metal in contact with molten salt, metals can be extracted from one phase to the other. This is related to a mercury electrode where a metal can be reduced, the metal will often then dissolve in the mercury to form an amalgam that modifies its electrochemistry greatly. For example, it is possible for sodium cations to be reduced at a mercury cathode to form sodium amalgam, while at an inert electrode (such as platinum) the sodium cations are not reduced. Instead, water is reduced to hydrogen. A detergent or fine solid can be used to stabilize an emulsion, or third phase.



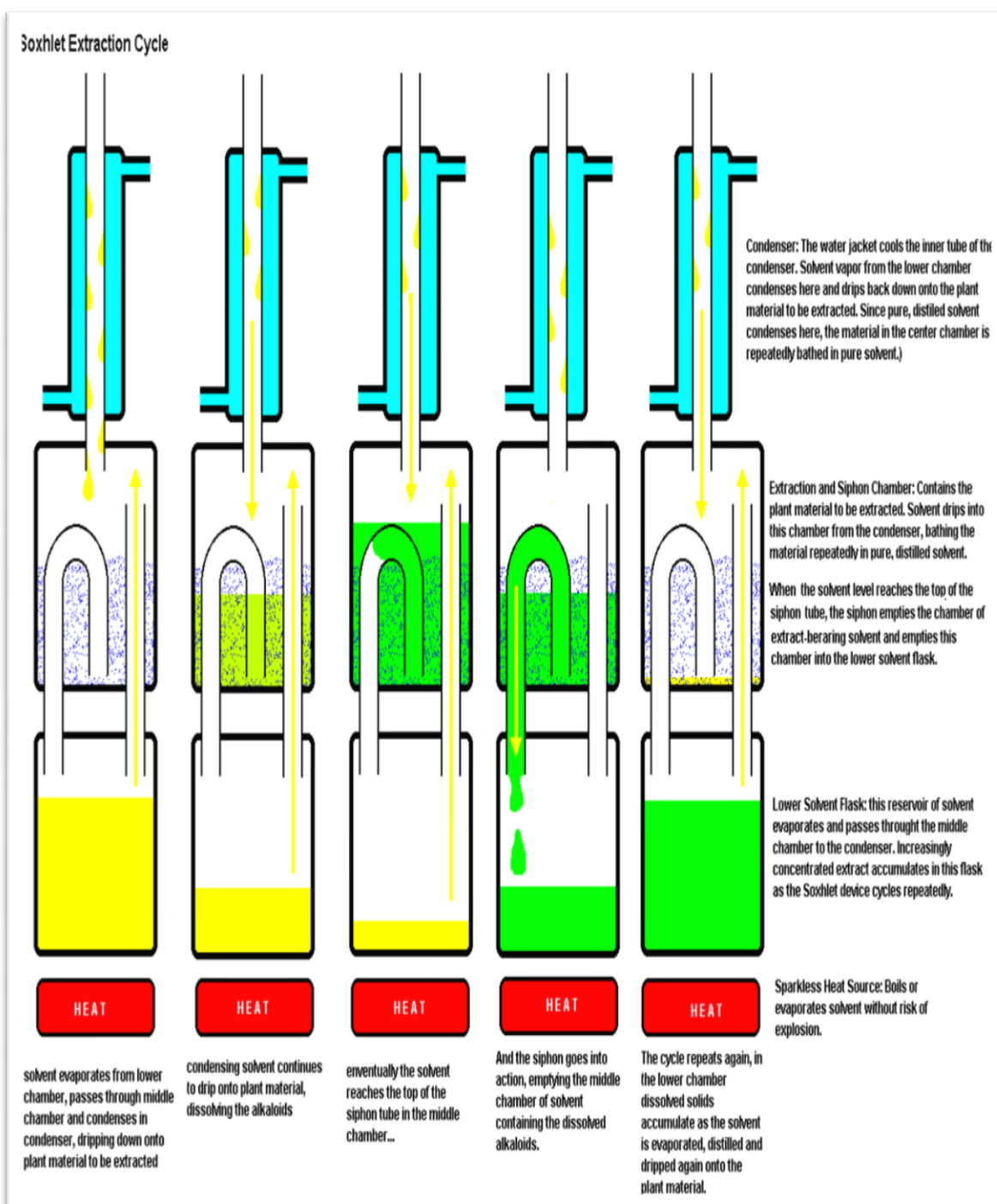
## 2.5 Soxhlet Extraction

A Soxhlet extractor is a piece of laboratory apparatus invented in 1879 by Franz von Soxhlet. It was originally designed for the extraction of a lipid from a solid material. However, a Soxhlet extractor is not limited to the extraction of lipids. Typically, a Soxhlet extraction is only required where the desired compound has a limited solubility in a solvent, and the impurity is insoluble in that solvent. If the desired compound has a significant solubility in a solvent then a simple filtration can be used to separate the compound from the insoluble substance. Fruit extraction in progress. The sample is placed in the thimble. Normally a solid material containing some of the desired compound is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the Soxhlet extractor. The Soxhlet extractor is placed onto a flask containing the extraction solvent. The Soxhlet is then equipped with a condenser. The solvent is heated to reflux. The solvent vapour travels up a distillation arm, and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapour cools, and drips back down into the chamber housing the solid material. The chamber containing the solid material slowly fills with warm solvent. Some of the desired compound will then dissolve in the warm solvent. When the Soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle may be allowed to repeat many times, over hours or days. During each cycle, a portion of the non-volatile compound dissolves in the solvent. After many cycles the desired compound is concentrated in the distillation flask. The advantage of this system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled.

After extraction the solvent is removed, typically by means of a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble, and is usually discarded.



**Figure 2.8:** Soxhlet extractor



**Figure 2.9:** Soxhlet extraction cycle.

## 2.6 Evaporation Process

Evaporation is a thermal separation process, widely used for concentration of liquids in the form of solutions, suspensions, and emulsions. Concentration is accomplished by boiling out a solvent, normally water, from the liquid. In most cases, concentrate resulting from the evaporation process is the final product. Sometimes, however, the evaporated, volatile component is also a main product, as, for example, during solvent recovery.

Evaporation processes fall into two general categories:

- Film type evaporation.
- Suppressed boiling type evaporation.

Film type evaporation involves the generation of a thin product film over a heat transfer surface to promote minimal resistance to heat transfer. Parameters are set such that optimum film boiling occurs within the evaporator body. Caution must be used to ensure a continuous film hydraulic condition (wetting rate) and to prevent nucleate boiling; otherwise, the rate of heat transfer will fall off dramatically, while the rate of scaling on the heat transfer surface will dramatically increase. Suppressed boiling type evaporation involves superheating a product above its boiling point, while maintaining a backpressure within the system to prevent boiling in the evaporator body. This is commonly referred to as a "heat and flash" principle.

A major requirement of the evaporation process is to maintain the quality of the liquid during evaporation and to avoid heat damage to the concentrate. This normally necessitates exposing the liquid to the lowest possible boiling temperature for the shortest period of time. Protecting concentrate quality and other physical requirements of the finished product have resulted in development of many different evaporator types. Additional demands for energy efficiency and minimal impact on the environment have driven development toward innovative evaporation process designs and plant configurations. GEA Evaporation Technologies companies have made major contributions to these developments, making the

company a world leader in evaporation, distillation, and crystallization technologies. The company is supported by its test and development facilities, where its core technologies are continually improved and applied to new products.

## **2.7 Rotary Evaporator**

The rotary evaporator is a device for gently and efficiently evaporating solvents from a mixture. It consists of a heated rotating vessel (usually a large flask) which is maintained under a vacuum through a tube connecting it to a condenser. The rotating flask is heated by partial immersion in a hot water bath. The flask's rotation provides improved heat transfer to the contained liquid; the rotation also strongly reduces the occurrence of 'bumps' caused by superheating of the liquid. The solvent vapors leave the flask by the connecting tube and are condensed in the condenser section. The condenser section is arranged so that the condensed vapors drain into another flask where they are collected. It is a very efficient way of rapidly removing large quantities of solvent. The major use in chromatography is the recovery of non-volatile solutes in preparative chromatography and the recovery of solvents for recycling. The device is also used for preparing coated supports for gas chromatography. For this application a weighed amount of support is placed in the flask, the required amount of stationary phase dissolved in excess of solvent is added to the solid and mixture tumble dried. This procedure has two advantages; as well as drying the support it ensures a very even distribution of the stationary phase throughout the support.

Design of a rotary evaporator

The main components of a rotary evaporator are:

1. A motor unit that rotates the evaporation flask or vial containing the user's sample.
2. A vapor duct that is the axis for sample rotation, and is a vacuum-tight conduit for the vapor being drawn off of the sample.
3. A vacuum system, to substantially reduce the pressure within the evaporator system.
4. A heated fluid bath (generally water) to heat the sample.

5. A condenser with either a coil passing coolant, or a "cold finger" into which coolant mixtures such as dry ice and acetone are placed.
6. A condensate-collecting flask at the bottom of the condenser, to catch the distilling solvent after it re-condenses.
7. A mechanical or motorized mechanism to quickly lift the evaporation flask from the heating bath.

The vacuum system used with rotary evaporators can be as simple as a water aspirator with a trap immersed in a cold bath (for non-toxic solvents), or as complex as a regulated mechanical vacuum pump with refrigerated trap. Glassware used in the vapor stream and condenser can be simple or complex, depending upon the goals of the evaporation, and any propensities the dissolved compounds might give to the mixture (e.g., to foam or "bump"). Commercial instruments are available that include the basic features, and various traps are manufactured to insert between the evaporation flask and the vapor duct. Modern equipment often adds features such as digital control of vacuum, digital display of temperature and rotational speed, and vapor temperature sensing.



**Figure 2.9.1:** Rotary evaporator.

Vacuum evaporators as a class function because lowering the pressure above a bulk liquid lowers the boiling points of the component liquids in it. Generally, the component liquids of interest in applications of rotary evaporation are research solvents that one desires to remove from a sample after an extraction, for instance, following a natural product isolation or a step in an organic synthesis. Use of a "rotavap" therefore allows liquid solvents to be removed without excessive heating of what are often complex and sensitive solvent-solute combinations.

Rotary evaporation is most often and conveniently applied to separate "low boiling" solvents such as n-hexane or ethyl acetate from compounds which are solid at room temperature and pressure. However, careful application also allows removal of a solvent from a sample containing a liquid compound if there is minimal co-evaporation

(azeotropic behavior), and a sufficient difference in boiling points at the chosen temperature and reduced pressure.

Solvents with higher boiling points such as water (100 °C at standard atmospheric pressure, 760 torr), dimethylformamide (DMF, 153 °C at the same), or dimethyl sulfoxide (DMSO, 189 °C at the same), can also be evaporated if the unit's vacuum system is capable of sufficiently low pressure. (For instance, both DMF and DMSO will boil below 50 °C if the vacuum is reduced from 760 torr to 5 torr.) However, more recent developments are often applied in these cases (e.g., evaporation while centrifuging or vortexing at high speeds). Rotary evaporation for high boiling hydrogen bond-forming solvents such as water is often a last recourse, as other evaporation methods or freeze-drying (lyophilization) are available. This is partly due to the fact that in such solvents, the tendency to "bump" is accentuated (see below). The modern centrifugal evaporation technologies are particularly useful when one has many samples to do in parallel, as in medium- to high-throughput synthesis now expanding in industry and academia.

Evaporation under vacuum can also, in principle, be performed using standard organic distillation glassware without rotation of the sample. The key advantages in use of a rotary evaporator are

1. That the centrifugal force and the frictional force between the wall of the rotating flask and the liquid sample result in the formation of a thin film of warm solvent being spread over a large surface.
2. The forces created by the rotation suppress bumping. The combination of these characteristics and the conveniences built into modern rotary evaporators allow for quick, gentle evaporation of solvents from most samples, even in the hands of relatively inexperienced users. Solvent remaining after rotary evaporation can be removed by exposing the sample to even deeper vacuum, on a more tightly sealed vacuum system, at ambient or higher temperature (e.g., on a Schlenk line or in a vacuum oven).

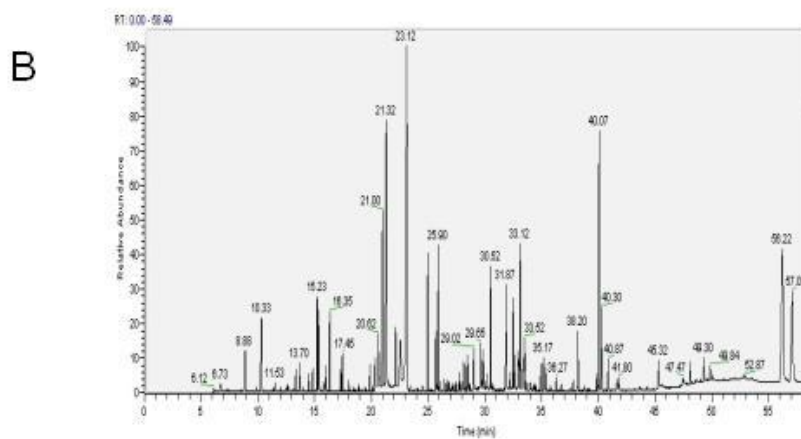
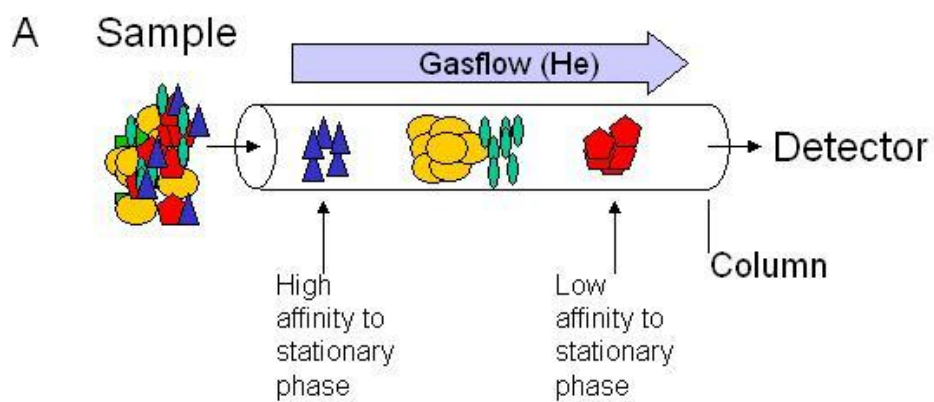


A key disadvantage in rotary evaporations, besides its single sample nature, is the potential of some sample types to bump, e.g. ethanol and water, which can result in loss of a portion of the material intended to be retained. Even professionals experience periodic mishaps during evaporation, especially bumping, though experienced users become aware of the propensity of some mixtures to bump or foam, and apply precautions that help to avoid most such events. In particular, bumping can often be prevented by taking homogeneous phases into the evaporation, by carefully regulating the strength of the vacuum (or the bath temperature) to provide for an even rate of evaporation, or, in rare cases, through use of added agents such as boiling chips (to make the nucleation step of evaporation more uniform). Rotary evaporators can also be equipped with further special traps and condenser arrays that are best suited to particular difficult sample types, including those with the tendency to foam or bump.

## **2.8 Gas Chromatography**

Gas chromatography - specifically gas-liquid chromatography - involves a sample being vapourised and injected onto the head of the chromatographic column. The sample is transported through the column by the flow of inert, gaseous mobile phase. The column itself contains a liquid stationary phase which is adsorbed onto the surface of an inert solid. The process of gas chromatography is carried out in a specially designed instrument. A very small amount of liquid mixture is injected into the instrument and is volatilized in a hot injection chamber. Then, it is swept by a stream of inert carrier gas through a heated column which contains the stationary, high-boiling liquid. As the mixture travels through this column, its components go back and forth at different rates between the gas phase and dissolution in the high-boiling liquid, and thus separate into pure components. Just before each compound exits the instrument, it passes through a detector. When the detector "sees" a compound, it sends an electronic message to the recorder, which responds by printing a peak on a piece of paper. The GC consists of an injection block, a column, and a detector. An inert gas flows through the system. The injection chamber is a heated cavity which serves to volatilize the compounds. The sample is injected by syringe into this chamber

through a port which is covered by a rubber septum. Once inside, the sample becomes vaporized and is carried out of the chamber and onto the column by the carrier gas.



**FIGURE 2.9.2** Gas chromatography.

### 2.8.1 Factors That Affect GC Separations

Efficient separation of compounds in GC is dependent on the compounds traveling through the column at different rates. The rate at which a compound travels through a particular GC system depends on the factors listed below:

- Volatility of compound: Low boiling (volatile) components will travel faster through the column than will high boiling components
- Polarity of compounds: Polar compounds will move more slowly, especially if the column is polar.
- Column temperature: Raising the column temperature speeds up all the compounds in a mixture.
- Column packing polarity: Usually, all compounds will move slower on polar columns, but polar compounds will show a larger effect.
- Flow rate of the gas through the column: Speeding up the carrier gas flow increases the speed with which all compounds move through the column.
- Length of the column: The longer the column, the longer it will take all compounds to elute. Longer columns are employed to obtain better separation.

Generally the number one factor to consider in separation of compounds on the GCs in the teaching labs is the boiling points of the different components. Differences in polarity of the compounds is only important if you are separating a mixture of compounds which have widely different polarities. Column temperature, the polarity of the column, flow rate, and length of a column are constant in GC runs in the Organic Chemistry Teaching Labs. For each planned GC experiment, these factors have been optimized to separate your compounds and the instrument set up by the staff.

## 2.8.2 Detectors

There are many detectors which can be used in gas chromatography. Different detectors will give different types of selectivity. A non-selective detector responds to all compounds except the carrier gas, a selective detector responds to a range of compounds with a common physical or chemical property and a specific detector responds to a single chemical compound. Detectors can also be grouped into concentration dependant detectors and mass flow dependant detectors. The signal from a concentration dependant detector is related to the concentration of solute in the detector, and does not usually destroy the sample Dilution of with make-up gas will lower the detectors response. Mass flow dependant detectors usually destroy the sample, and the signal is related to the rate at which solute molecules enter the detector. The response of a mass flow dependant detector is unaffected by make-up gas. Have a look at this tabular summary of common GC detectors:

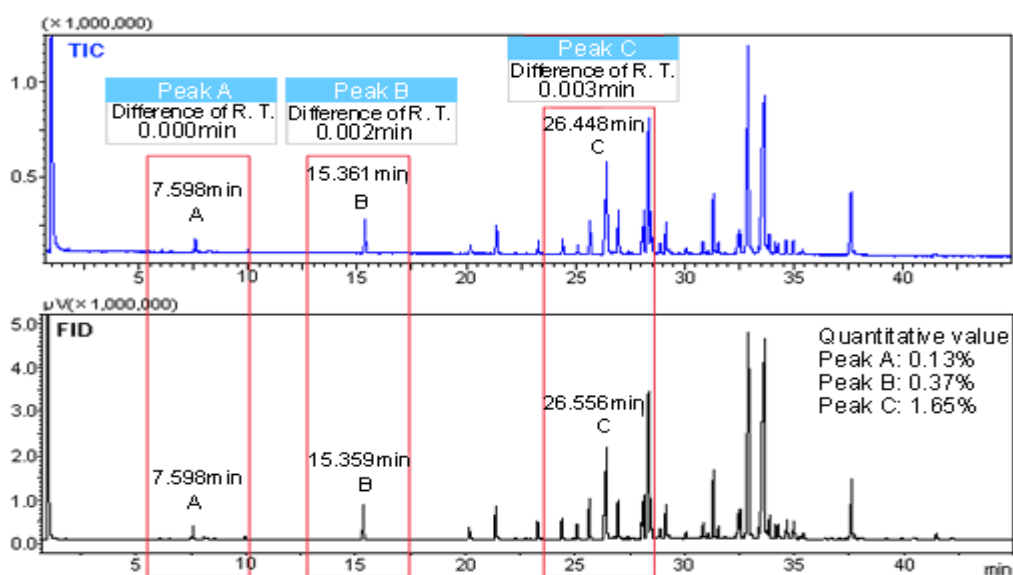


Fig. 1

**FIGURE 2.9.2 :** Gas chromatography detecting system.

Table 2.3 Detectors used in gas chromatography.

Detector	Type	Support gases	Selectivity	Detectability	Dynamic range
Flame ionization (FID)	Mass flow	Hydrogen and air	Most organic cpds.	100 pg	10 <sup>7</sup>
Thermal conductivity (TCD)	Concentration	Reference	Universal	1 ng	10 <sup>7</sup>
Electron capture (ECD)	Concentration	Make-up	Halides, nitrates, nitriles, peroxides, anhydrides, organometallics	50 fg	10 <sup>5</sup>
Nitrogen-phosphorus	Mass flow	Hydrogen and air	Nitrogen, phosphorus	10 pg	10 <sup>6</sup>
Flame photometric (FPD)	Mass flow	Hydrogen and air possibly oxygen	Sulphur, phosphorus, tin, boron, arsenic, germanium, selenium, chromium	100 pg	10 <sup>3</sup>
Photo-ionization (PID)	Concentration	Make-up	Aliphatics, aromatics, ketones, esters, aldehydes, amines, heterocyclics, organosulphurs, some organometallics	2 pg	10 <sup>7</sup>
Hall electrolytic conductivity	Mass flow	Hydrogen, oxygen	Halide, nitrogen, nitrosamine, sulphur		

### 2.8.3 Pyrolysis

**Pyrolysis** is a thermochemical decomposition of organic material at elevated temperatures without the participation of oxygen. It involves the simultaneous change of chemical composition and physical phase, and is irreversible. The word is coined from the Greek-derived elements pyr "fire" and lysis "separating".

Pyrolysis is a special case of thermolysis, and is most commonly used for organic materials, being, therefore, one of the processes involved in charring. The pyrolysis of wood, which starts at 200–300 °C (390–570 °F),<sup>[1]</sup> occurs for example in fires where solid fuels are burning or when vegetation comes into contact with lava in volcanic eruptions. In general, pyrolysis of organic substances produces gas and liquid products and leaves a solid residue richer in carbon content, char. Extreme pyrolysis, which leaves mostly carbon as the residue, is called carbonization.

The process is used heavily in the chemical industry, for example, to produce charcoal, activated carbon, methanol, and other chemicals from wood, to convert ethylene dichloride into vinyl chloride to make PVC, to produce coke from coal, to convert biomass into syngas and biochar, to turn waste into safely disposable substances, and for transforming medium-weight hydrocarbons from oil into lighter ones like gasoline. These specialized uses of pyrolysis may be called various names, such as dry distillation, destructive distillation, or cracking.

Pyrolysis also plays an important role in several cooking procedures, such as baking, frying, grilling, and caramelizing. In addition, it is a tool of chemical analysis, for example, in mass spectrometry and in carbon-14 dating. Indeed, many important chemical substances, such as phosphorus and sulfuric acid, were first obtained by this process. Pyrolysis has been assumed to take place during catagenesis, the conversion of buried organic matter to fossil fuels. It is also the basis of pyrography. In their embalming process, the ancient Egyptians used a mixture of substances, including methanol, which they obtained from the pyrolysis of wood.

Pyrolysis differs from other high-temperature processes like combustion and hydrolysis in that it does not involve reactions with oxygen, water, or any other reagents. In practice, it is

not possible to achieve a completely oxygen-free atmosphere. Because some oxygen is present in any pyrolysis system, a small amount of oxidation occurs.

The term has also been applied to the decomposition of organic material in the presence of superheated water or steam (hydrous pyrolysis), for example, in the steam cracking of oil.

#### **2.8.4 Bioethanol**

Ethanol fuel is ethanol (ethyl alcohol), the same type of alcohol found in alcoholic beverages. It is most often used as a motor fuel, mainly as a biofuel additive for gasoline. World ethanol production for transport fuel tripled between 2000 and 2007 from 17 billion to more than 52 billion litres. From 2007 to 2008, the share of ethanol in global gasoline type fuel use increased from 3.7% to 5.4%. In 2010 worldwide ethanol fuel production reached 22.95 billion U.S. liquid gallons(bg) (86.9 billion liters), with the United States as the top producer with 13.2 bg (50 billion liters), accounting for 57.5 percent of global production. Ethanol fuel has a "gasoline gallon equivalency" (GGE) value of 1.5 US gallons (5.7 L).

Ethanol fuel is widely used in Brazil and in the United States, and together both countries were responsible for 88 percent of the world's ethanol fuel production in 2010. Most cars on the road today in the U.S. can run on blends of up to 10% ethanol, and the use of 10% ethanol gasoline is mandated in some U.S. states and cities. Since 1976 the Brazilian government has made it mandatory to blend ethanol with gasoline, and since 2007 the legal blend is around 25% ethanol and 75% gasoline . By December 2010 Brazil had a fleet of 12 million flex-fuel automobiles and light trucks and over 500 thousand flex-fuel motorcycles regularly using neat ethanol fuel (known as E100).

Bioethanol is a form of renewable energy that can be produced from agricultural feedstocks. It can be made from very common crops such as sugar cane, potato, manioc and corn. There has been considerable debate about how useful bioethanol will be in replacing gasoline.

## **CHAPTER 3**

### **3.0 METHODOLOGY**

#### **3.1 Materials**

Blended rubber seeds kernel is used as the sample for the extraction and hexane is used as non-polar solvent in this research. Other than that, zeolite is used as acid catalyst in catalytic cracking process and anti-bumping granular also is used to make the liquid boil smoothly.

#### **3.2 Apparatus And Equipments**

In doing this research experiment we need several apparatus and equipments. They are such as Soxhlet Extractor , Rotary Evaporator, Gas Chromatography, Analytical Balance, Heating Mantel, Measuring Cylinder 100 mL, Flask 250 mL, Beaker 100 mL, Filter Funnel, Filter Papers, Thermometer 110°C, vials, 0.2µm syringe filter and Syringe 5mL and Thimble Filter



### 3.3 Chemical Substances

The chemical substances that are needed for the experimental procedures are raw material – 500gram of Rubber Seeds, additives – Boiling Chips, solvent – Standard Hexane (99% purity)Standard and Isooctane (99% purity)

### 3.4 Experimental Works

The extraction of fatty acid (rubber seed oil) is carried out using soxhlet extraction method. This research *will be conducted in 6 major steps. First is* sample preparation. Next is extraction of fatty acid( rubber seed oil). It is followed by evaporation of hexane. Then it is continued with catalytic cracking. And next step is preparation of standard calibration curve. And the last step is to analyse using gas chromatography method

#### 3.4.1 Sample Preparation

Firstly, rubber seeds in good condition is cleaned and shelled. Then the shelled seeds are grinded into smaller particles using a grinder. After that, the grinded rubber seeds kernels are dried at 60°C in desiccators for 1 day to remove all of the moisture inside the rubber seed particles.

#### 3.4.2 Extraction Of Rubber Seed Oil Using Soxhlet Extractor

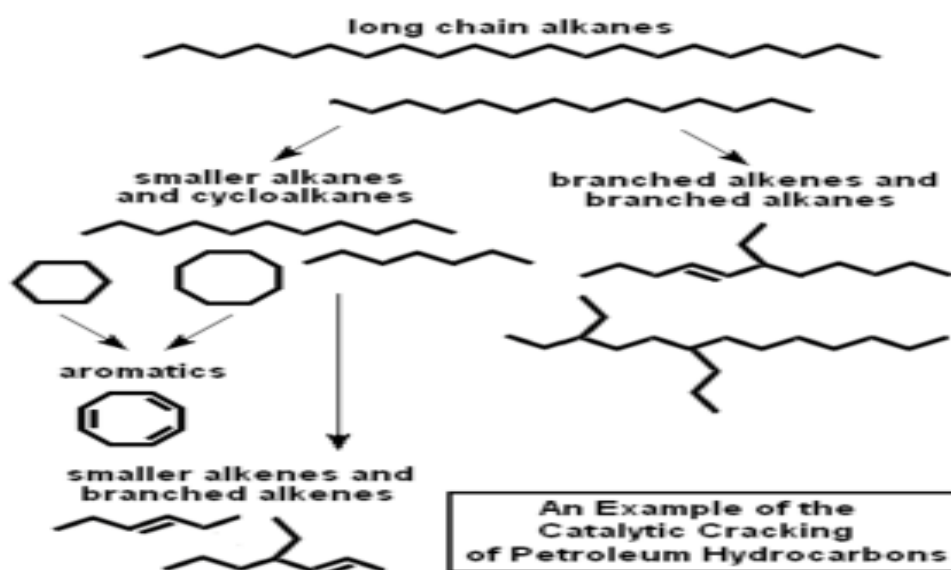
First 500 grams of rubber seeds are cleaned shelled and blended using a blender. The blended rubber seeds are inserted into a thimble filter until two third portion of the thimble filter and then inserted into the siphon exit of the soxhlet extractor. The receiving flask is filled with the hexane solvent. Then switch on the heater until the temperature of the solvent reaches about 75<sup>0</sup>C or until the solvent is boiling. The experiment is continued for approximately six hours. Switched off the heater and let the mixture solution of hexane and oil to cool down. The mass of rubber seed is maintained at 73.7 grams while the amount of solvent used is changed to difren ratios from 1:2, 1:3 and 1:4.

### 3.4.3 Evaporation Of Solvent By Rotary Evaporator

First the mixture is then brought to the rotary evaporator to evaporate the solvent at a temperature of around  $75^{\circ}\text{C}$  (slightly higher than the boiling point of the hexane). Then the liquid remaining in the flask is the pure rubber seed oil.

### 3.4.4 Heterogenous Catalytic Cracking Using Zeolite As Catalyst

The rubber seed oil is transferred into a flask. Then 5 grams of zeolite catalyst is added into the flask containing 25mL of rubber seed oil... The mixture is then heated until  $300^{\circ}\text{C}$  and maintained it for 45 minutes After that, the samples are filtered to remove the solid catalyst to obtain the distilled product oil.



**Figure 3.0 :** Catalytic cracking of petroleum hydrocarbon

### 3.4.5 Preparation Of Standards Using Isooctane

Isooctane and hexane are prepared according to the portions in table 3.1. Then the mixtures of the standards are injected into each vial through a 0.2 $\mu$ m syringe filter of about 1.5mL.

**Table 3.1** Composition of the Isooctane-Hexane mixture

Vials	Composition	
	Isooctane	Hexane
1	0%	100%
2	20%	80%
3	40%	60%
4	50%	50%
5	60%	40%
6	80%	20%

### 3.4.6 Gas Chromatogram Analysis

#### 3.4.6.1 Gas Chromatogram Standard Analysis

The vials are labeled and must be arranged in sequence on the auto-injectors VS auto samplers at the gas chromatography's vial rack. The standards are analyzed using the Gas Chromatography. The conditions of the Gas Chromatography are set as in Table 3.2. The chromatogram (peak area versus retention time) of each standard are then obtained. Then, a calibration curve (peak area versus concentration) is plotted.

**Table 3.2** : Gas Chromatographer (GC) condition

Temperature Column	Initial 50°C, hold 3 minutes, program at 8°C/min to 120°C, hold 5 minutes
Detector Type	Flame Ionization Detector (FID)
Injector Temperature	250°C
Detector Temperature	250°C
Injection Size	1.0µL (10:1 split)
Hydrogen Flow	35mL/min
Air Flow	400mL/min
Helium Make Up	35mL/min
Carrier Gas	Helium, Compress Air, H <sub>2</sub> , N <sub>2</sub>
Carrier Gas Pressure	5 bar for every carrier gas

The distilled product oil for each mass of catalyst used will be diluted with hexane as a solvent to give the four different mixture solutions – 1%, 5%, 10%, and 20% of distilled products. The samples are injected into the 1.5mL vials using the 0.2µm syringe filter of about 1.5mL. The vials are labeled and must be arranged in sequence on the auto-injectors VS auto samplers at the gas chromatography's vial rack after the standard is placed. Samples obtained (for every diluted mixture solutions) are analyzed using Gas Chromatography. The conditions of the Gas Chromatography are set as in Table 3.2. The chromatogram (peak area versus retention time) of each standard are then obtained. Then, a calibration curve (peak area versus concentration) is plotted.

## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### **4.1 OBSERVATION**

General observations are more to physical properties which are based on their appearance, odor or changes in physical condition. However for further analysis on sample concentration, mathematical and graphical method are applied and discussed in detail through this chapter.

In this research, hexane which has chemical formula  $C_6H_{14}$  is used as solvent to extract rubber seed oil from rubber seeds kernel since it is non-polar solvent. Rubber seed oil is soluble in non-polar hexane solvent in the form of homogeneous solution following the principle of "like dissolves like". A popular aphorism used for predicting solubility is "like dissolves like". This statement indicates that a solute will dissolve best in a solvent that has a similar chemical structure to itself. This view is simplistic, but it is a useful rule of thumb. The overall solvation capacity of a solvent depends primarily on its polarity. Mainly at room temperature, hexane is in liquid form due to its boiling point ( $69^{\circ}C$ ) but it easily evaporated if there is some heat is supplied. It is colourless liquid, with gasoline-like odour. This hexane is handled with carefully since it is very hazardous for human. The observation inside the evaporation tube for the first cycle of extraction of rubber seed oil, the yellowish colour of rubber seed oil is dissolves in the hexane solvent.

When the liquid reaches the overflow level, a siphon aspirates the solute from the thimble-holder and unloads it back into the distillation flask, thus carrying the extracted

rubber seed oil into the bulk liquid. This operation is repeated until extraction is complete. After 6 hours, a yellowish liquid formed. This solution consists of the mixture of hexane and rubber seed oil. The yellowish solution then is let's to be cooled at the room temperature to avoid the evaporation of hexane during transferring the solution into anti-UV light glass bottle. The function of anti-UV light glass bottle is to prevent the samples from damaging by UV or sun light. Then the solution is stored at room temperature before go to the next step which is evaporation of hexane.

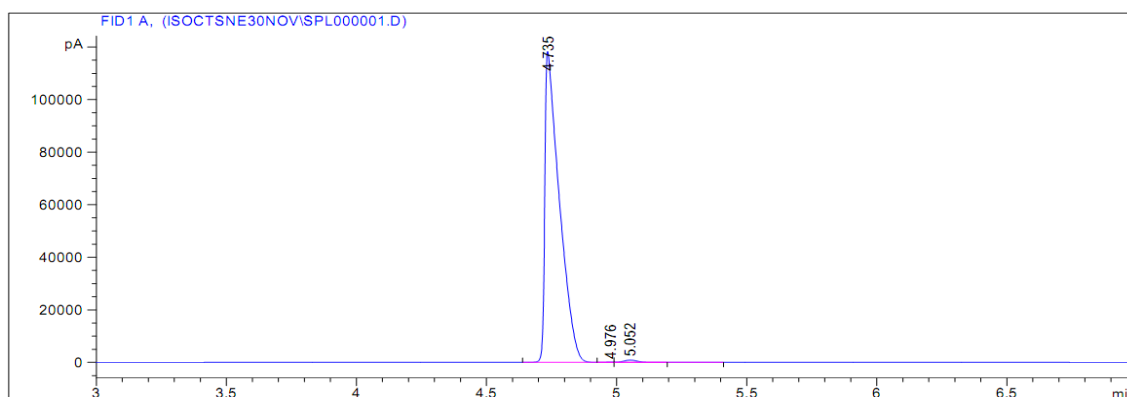
Then a rotary evaporator is used to evaporate the hexane-rubber seed oil and separate them. After the hexane is evaporated using rotary evaporator, a viscous brownish liquid is obtained. Rubber seed oil consists of saturated and unsaturated fatty acid depending on double bonds and most of the fatty acids have a chain of four to 28 carbons with differ in length. It is in form of liquid at the room temperature due to its high boiling point ( $286^{\circ}\text{C}$  for oleic acid) where they will not easy to broken into light hydrocarbon except heat is supplied and normally its odor is known as peculiar lard-like odor. However, when heating process is started and temperature reached  $300^{\circ}\text{C}$  after almost 45 minutes of heating the color and its odor changes into quite darken than the color before heating while the smell a bit rancid.

At this temperature sample is known as isooctane. The changes happen due to oxidation process where the "double bond" of monounsaturated fatty acid bond of fatty acid is broken by heating process and isomerizes into saturated hydrocarbon and other derivatives with the existent of air and oxygen. Mean, the heating process itself is called as oxidization process.

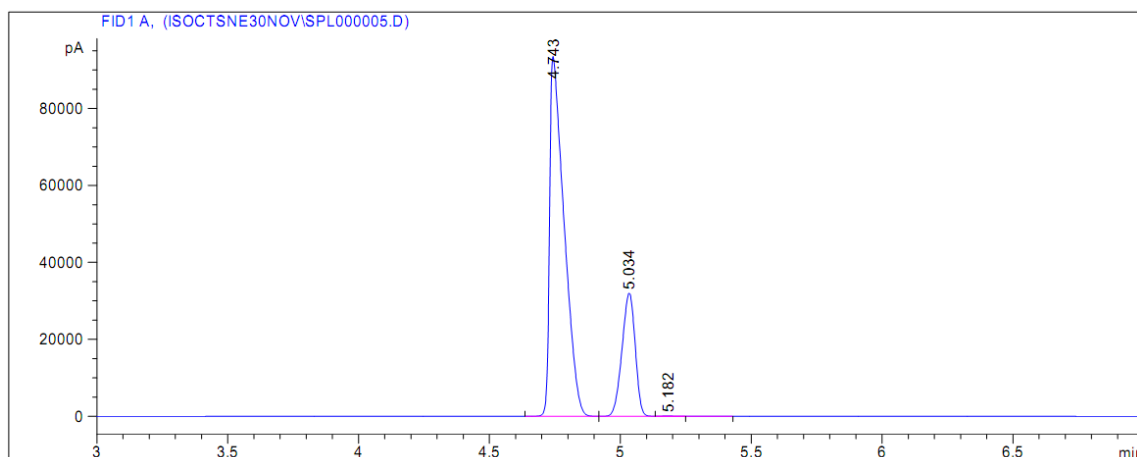
Zeolite catalyst which also acts as the booster for this reaction changes its color from white to brown. However, the chemicals properties of catalyst is still constant since it does not involved in reaction but the function is to reduce the activation energy and make the reaction go faster. Filtration of solid catalyst and cracked rubber seed oil using filter paper is a process where the brown color oil is dropping through the filter funnel. Its takes almost 10 minutes for each drop of oil to drop into the small conical flask and one day is needed to achieve around 10ml of oil because the oil is saturated with high amount of catalyst.

## 4.2 Qualitative Analysis for Standard Isooctane Calibration Curve

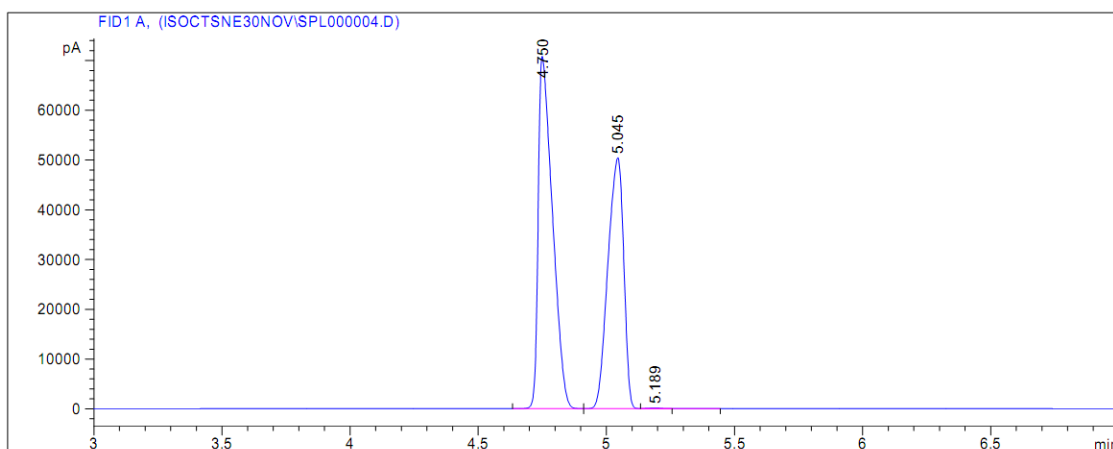
Gas chromatography is used to test the purity of the particular substance. According to the theory, gas chromatograms provide an excellent means of confirming the presence or absence of a suspected compound in a mixture. A broad variety of samples can be analyzed as long as the compounds are sufficiently thermal stable and volatile enough. In this case, isooctane is the suspected compound need to recover from the sample. Supposedly no new peak should be appeared on addition of the known compound such as in standard isooctane. If the standards are no contaminant from instrument factor, the chromatogram should be obviously show only two narrow and height peaks where the hexane peak ( $C_6H_{14}$ ) comes out first then isooctane ( $C_8H_{18}$ ) due to its high volatility factor and low boiling point factor. The retention time of the highest peak of blank (100% hexane) and second highest peak in 40% isooctane 60% hexane became as the references for the retention time of other concentration percentages



**Figure 4.1** Chromatogram of 0% Standard Isooctane

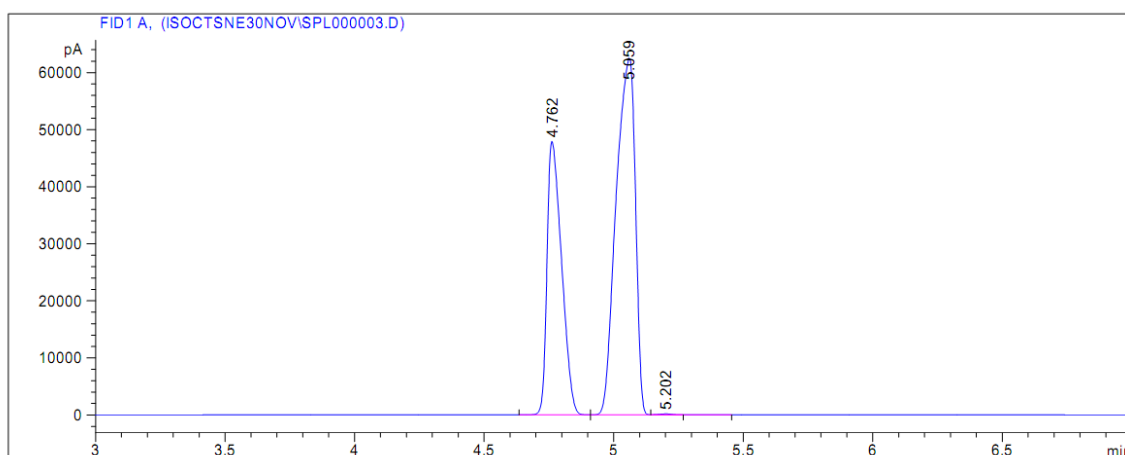


**Figure 4.2** Chromatogram of 20% Standard Isooctane

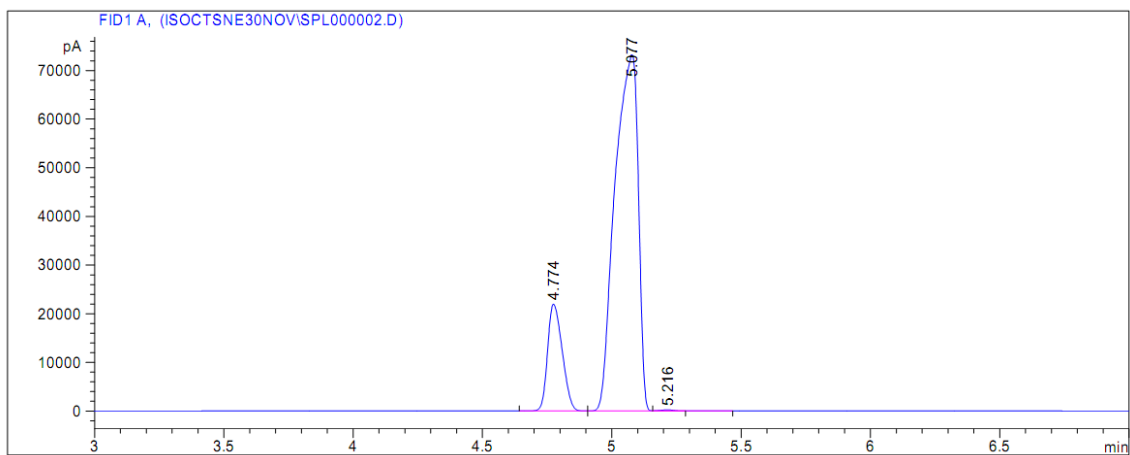


**Figure 4.3** Chromatogram of 40% Standard Isooctane





**Figure 4.4** Chromatogram of 60% Standard Isooctane



**Figure 4.5** Chromatogram of 80% Standard Isooctane

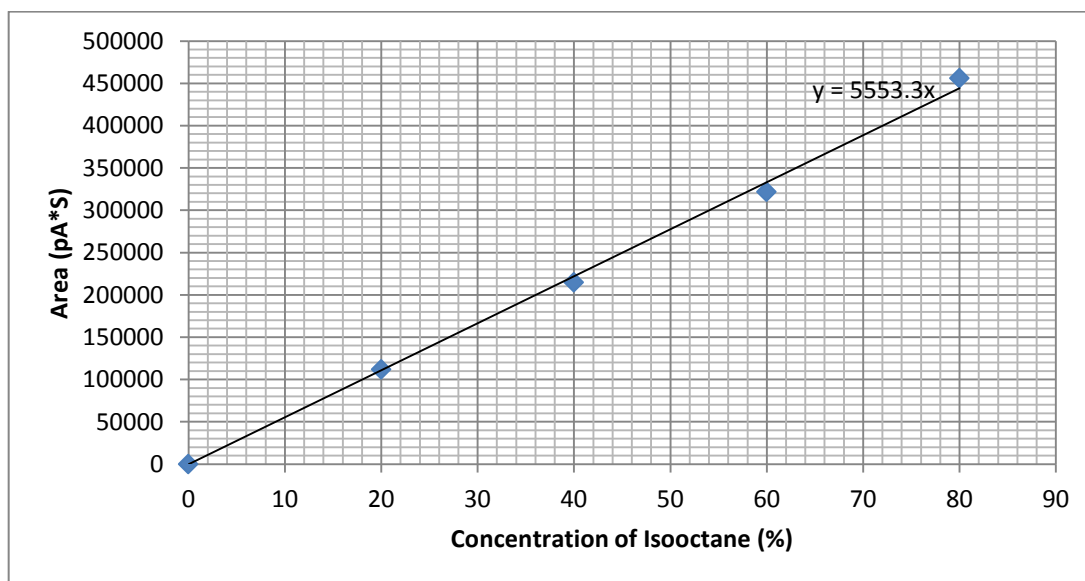
**Table 4.1** Chromatogram Analysis for Standard Isooctane

<b>CONCENTRATION OF ISOCTANE (%)</b>		<b>HEXANE</b>	<b>ISOCTANE</b>
<b>0%</b>	<i>Retention time(min)</i>	4.735	0
	<i>Peak Area ( pA*s)</i>	4.56634E+05	0
	<i>Peak Area (%)</i>	99.96915	0
<b>20</b>	<i>Retention time(min)</i>	4.743	5.034
	<i>Peak Area ( pA*s)</i>	3.57789E+05	1.12664E+04
	<i>Peak Area (%)</i>	76.05197	23.94803
<b>40</b>	<i>Retention time(min)</i>	4.750	5.045
	<i>Peak Area ( pA*s)</i>	2.72851E+05	2.15759E+05
	<i>Peak Area (%)</i>	55.84228	44.15772
<b>60</b>	<i>Retention time(min)</i>	4.762	5.059
	<i>Peak Area ( pA*s)</i>	1.86902E+05	3.22600E+05
	<i>Peak Area (%)</i>	36.68334	63.31666
<b>80</b>	<i>Retention time(min)</i>	4.774	5.077
	<i>Peak Area ( pA*s)</i>	8.61127E+04	4.56298E+05
	<i>Peak Area (%)</i>	15.85654	84.02133

In order to obtain the standard calibration curve for the determination of the actual percentage of isooctane present in each of the samples, the peak area of each samples are obtained from each chromatogram. A graph of peak area (pA\*s) versus concentration of the isooctane (%) are plotted:

**Table 4.2** Data of Concentration of Isooctane and the Area

Concentration of Isooctane (%)	Area (pA*s)
0	0
20	1.12E+05
40	2.16E+05
60	3.23E+05
80	4.56E+05



**Figure 4.6** Standard Calibration Curve

From Figure 4.9, the increasing straight line indicates that, as the concentration of Isooctane increase, the peak area increases as well. In other words, the peak area of Isooctane is directly proportional to its concentration. This calibration curve is used to determine the actual concentration of Isooctane of each sample by calibrating the Isooctane's peak area of each sample in the calibration curve to get the concentration.

By using the mathematical approach, the straight line is taken from the best symmetrical degree among the plotted data and the standard calibration curve equation for isooctane is:

$$y = 5553x \quad (\text{equation 1})$$

From this equation, the actual concentration of Isooctane present in each sample can be determined by manual calculation.

### 4.3 Concentration of Actual Isooctane in Sample by Backward Calculation

Since concentration of isooctane inside the unknown sample is studied, thus backward calculation is used to estimate the actual concentration of isooctane produce from the cracking process. Knowing that, sample must be diluted with hexane before entering the gas chromatography. So, the concentration (peak area) directly read from the GC computer software are actually the concentration of dilution of hexane and isooctane. In order to quantify the exact amount of desired isooctane, the experimental results are recalculated by eliminated the hexane dilution. We assumed that samples are directly injected for analysis without dilution process. The entire calculations techniques are drafted as below while the example of calculation is shown in Appendix A. Step of backward calculation are as follow:

1. Actual peak area isooctane

$\frac{\text{Actual peak Area isooctane (\%)}}{[100 - \text{Peak area hexane (\%)}]} = \frac{\text{Peak area isooctane (\%)}}{100} \times 100(\%)$
--

2. Actual peak area

$\frac{\text{Actual peak Area isooctane (pA*s)}}{(\text{pA*s})} = \frac{\text{Peak area isooctane (pA*s)}}{\text{Peak area isooctane (\%)}} \times \frac{\text{actual peak area of isooctane (\%)}}{(\%)}$
--

$$y = 5553x \quad (\text{equation 1})$$

#### 4. Actual concentration of Isooctane

$\text{Actual concentration of Isooctane (\%)(x)} = \frac{\text{Actual peak area of isooctane (pA*s) (y)}}{5553}$
---

The chromatogram analysis of the samples is obtained and the peak areas are identified of which chemical compound based on the retention time of Hexane and Isooctane based on the Chromatogram of the standard analysis. It is decided that the retention time of Isooctane and Hexane is around 5.05 and 4.75 minute respectively. The peak areas of Isooctane and Hexane area identified and the table below shows the details from the data of the analysis:

**Table 4.3** Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio1:2

Dilution Samples (%)	Isooctane			Hexane		
	Retention Time	Peak Area (pA*s)	Area (%)	Retention Time	Peak Area (pA*s)	Area (%)
<b>1</b>	5.055	2862.46582	0.67768	4.741	4.19502e5	99.31554
<b>5</b>	5.057	1674.54102	0.67741	4.751	2.45523e5	99.32259
<b>10</b>	5.059	2399.13892	0.73998	4.753	3.21769e5	99.24532
<b>15</b>	5.060	1848.14673	0.65239	4.758	2.81442e5	99.34761
<b>20</b>	5.059	2086.08130	0.74645	4.759	2.77333e5	99.23643

**Table 4.4** Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio 1:3

Dilution Samples (%)	Isooctane			Hexane		
	Retention Time	Peak Area (pA*s)	Area (%)	Retention Time	Peak Area (pA*s)	Area (%)
1	5.054	2637.00122	0.73564	4.746	3.55719e5	99.23460
5	5.058	2515.25610	0.77513	4.751	3.21910e5	99.20385
10	5.060	2599.04980	0.69351	4.747	3.72128e5	99.29646
15	5.059	2443.49072	0.74110	4.752	3.27199e5	99.23833
20	5.060	2016.82568	0.66938	4.756	2.99246e5	99.31882

**Table 4.5** Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio 1:4

Dilution Samples (%)	Isooctane			Hexane		
	Retention Time	Peak Area (pA*s)	Area (%)	Retention Time	Peak Area (pA*s)	Area (%)
1	5.055	2582.51392	0.72215	4.747	3.54925e5	99.24744
5	5.061	2274.26147	0.68481	4.753	3.29791e5	99.30392
10	5.061	2694.59937	0.68948	4.746	3.88049e5	99.29227
15	5.060	2300.12207	0.66223	4.751	3.44960e5	99.31828
20	5.061	1950.56885	0.63130	4.757	3.06961e5	99.34767

In order to calculate the actual concentration of Isooctane present in each of the samples, a backward calculation is carried out manually knowing that the samples must be diluted with the Hexane solvent before entering into the Gas Chromatogram. This is to make it easier for the samples to be injected into the column for analysis. Below are the equations used for the determination of Isooctane concentration in each sample;

✚ Actual Peak area of Isooctane (%)

*Actual Peak Area of Isooctane (%)*

$$= \frac{\text{Peak Area of Isooctane (\%)}}{[(100 - \text{Peak Area of Hexane})\%]} \times 100\%$$

✚ Actual Peak Area of Isooctane (pA\*s)

*Actual Peak Area of Isooctane (pA \* s)*

$$= \frac{\text{Peak Area of Isooctane (pA * s)}}{\text{Peak Area of Isooctane (\%)}} \times [\text{Actual Peak Area of Isooctane (\%)}]$$

✚ Actual Concentration of Isooctane (%)

- Based on equation 1 where the;

$y = \text{Actual Peak Area of Isooctane (pA * s)}$  and

$x = \text{Actual Concentration of Isooctane (\%)}$

*Actual Concentration of Isooctane (%)*

$$= \frac{\text{Actual Peak Area of Isooctane (pA * s)}}{5553}$$

The results of the calculations are shown in the Table below:

**Table 4.6** :Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio 1:2

<i>Dilution (%)</i>	<i>Area Isooctane (pA*s)</i>	<i>Isooctane Area (%)</i>	<i>Hexane Area (%)</i>	<i>Actual Peak Area Isooctane (%)</i>	<i>Actual Peak Area (pA*s)</i>	<i>Actual Isooctane Concentration (%)</i>	<i>Experimental Isooctane concentration (%)</i>
1	2862.46582	0.67768	99.23460	88.53933	373983.0095	67.3479	0.51548
5	1674.54102	0.67741	99.20385	85.08573	210329.8521	37.8768	0.30156
10	2399.13892	0.73998	99.29646	105.17952	341009.5972	61.4010	0.43204
15	1848.14673	0.65239	99.23833	95.77351	271315.4717	48.8593	0.33282
20	2086.08130	0.74645	99.31882	109.5819	306245.2362	55.1495	0.37567

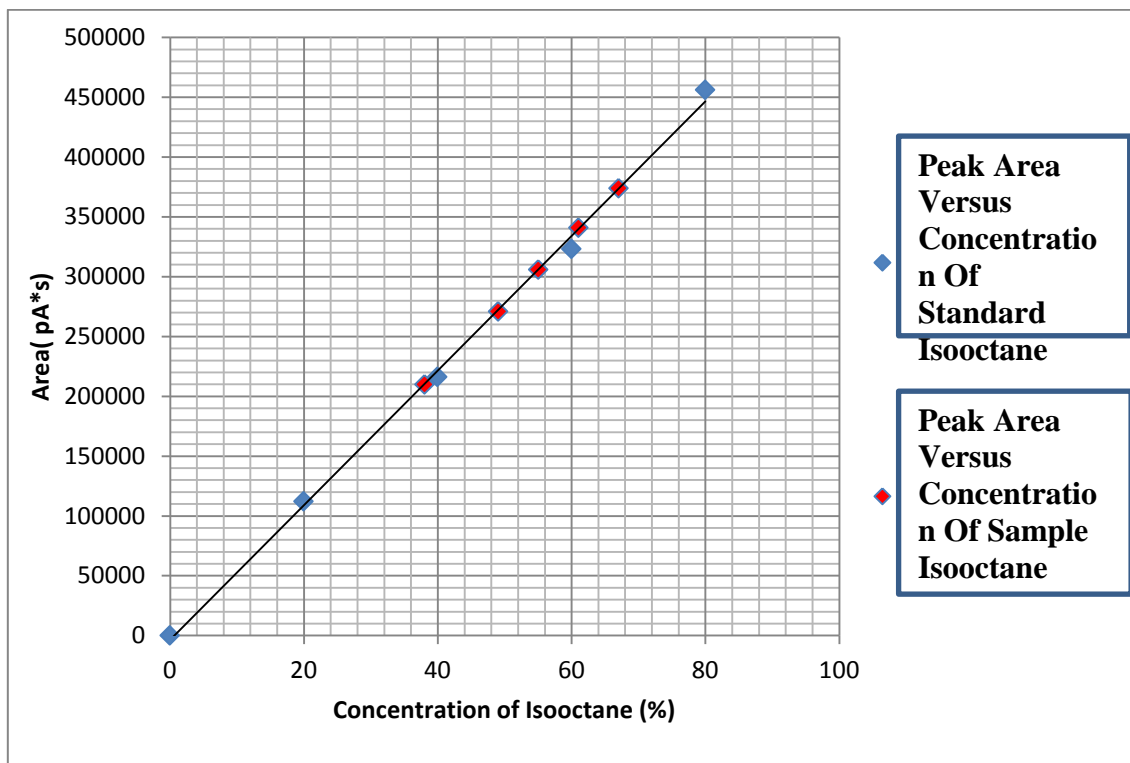


**Table 4.7** Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio 1:3

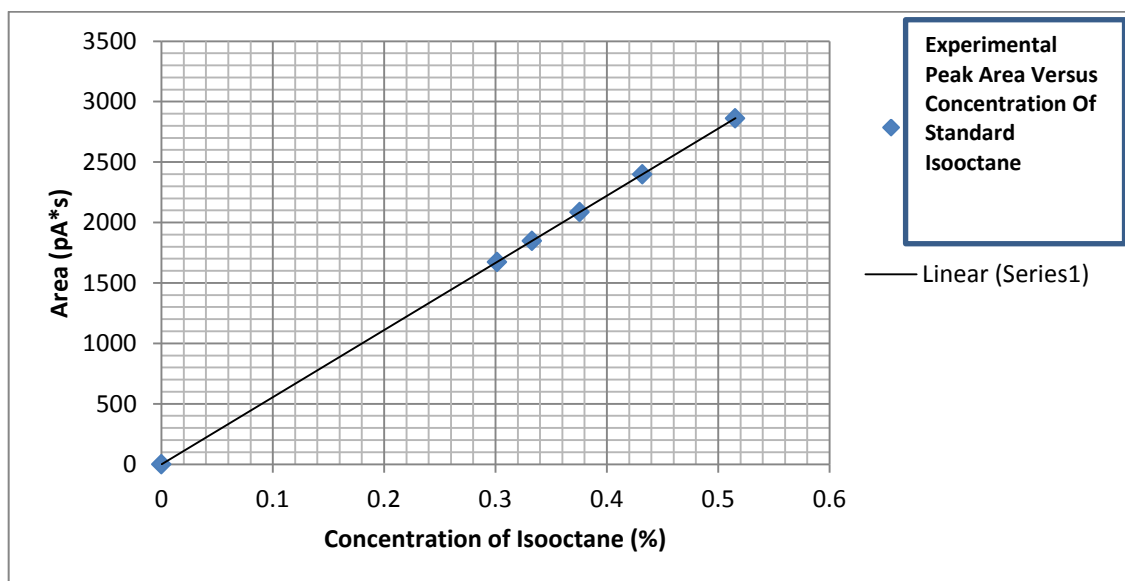
<i>Dilution (%)</i>	<i>Area Isooctane (pA*s)</i>	<i>Isooctane Area (%)</i>	<i>Hexane Area (%)</i>	<i>Actual Peak Area Isooctane (%)</i>	<i>Actual Peak Area (pA*s)</i>	<i>Actual Isooctane Concentration (%)</i>	<i>Experimental Isooctane concentration (%)</i>
1	2637.00122	0.73564	99.23460	96.11185	253447.0313	45.6415	0.47488
5	2515.25610	0.77513	99.20385	97.35979	315927.4132	56.8931	0.45295
10	2599.04980	0.69351	99.29646	98.57435	369424.5956	66.5270	0.46804
15	2443.49072	0.74110	99.23833	97.29940	320807.0057	57.7718	0.44003
20	2016.82568	0.66938	99.31882	98.26771	296078.2290	53.3186	0.36320

**Table 4.8:** Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio 1:4

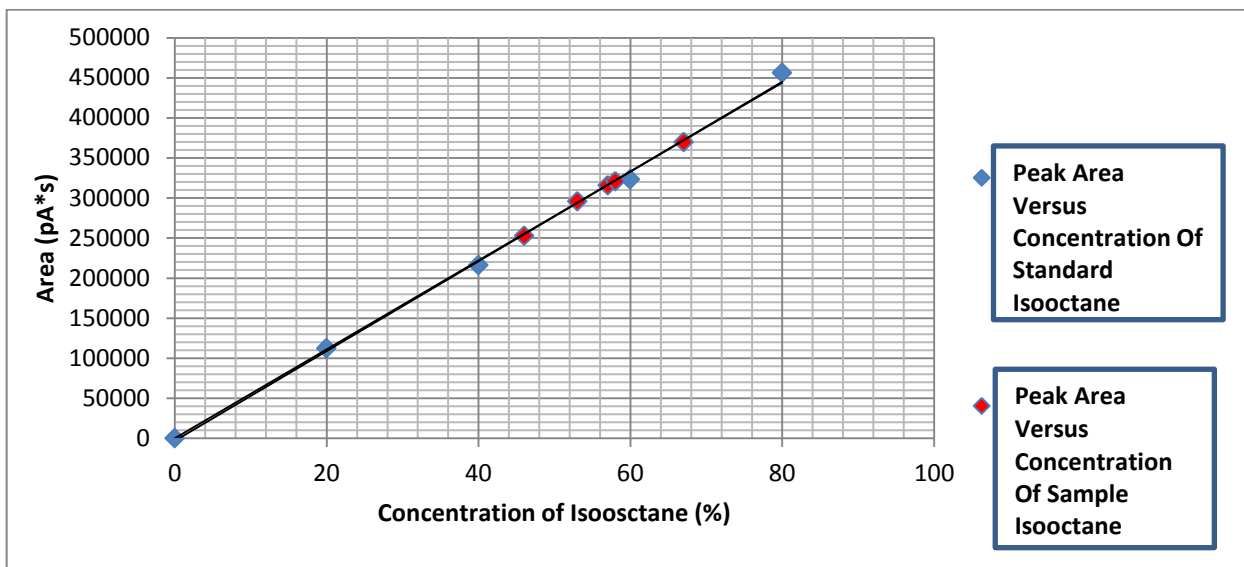
<i>Dilution (%)</i>	<i>Area Isooctane (pA*s)</i>	<i>Isooctane Area (%)</i>	<i>Hexane Area (%)</i>	<i>Actual Peak Area Isooctane (%)</i>	<i>Actual Peak Area (pA*s)</i>	<i>Actual Isooctane Concentration (%)</i>	<i>Experimental Isooctane concentration (%)</i>
1	2582.513 92	0.72215	99.2474 4	95.9591 2	343163.85 67	61.7979	0.46507
5	2274.261 47	0.68481	99.3039 2	98.3809 3	326724.15 10	58.8374	0.40956
10	2694.599 37	0.68948	99.2922 7	97.4213	380738.32 82	68.5644	0.48525
15	2300.122 07	0.66223	99.3182 8	97.1410 5	337399.82 25	60.7599	0.41421
20	1950.568 85	0.63130	99.3476 7	96.7761 7	299015.65 93	53.8476	0.35126



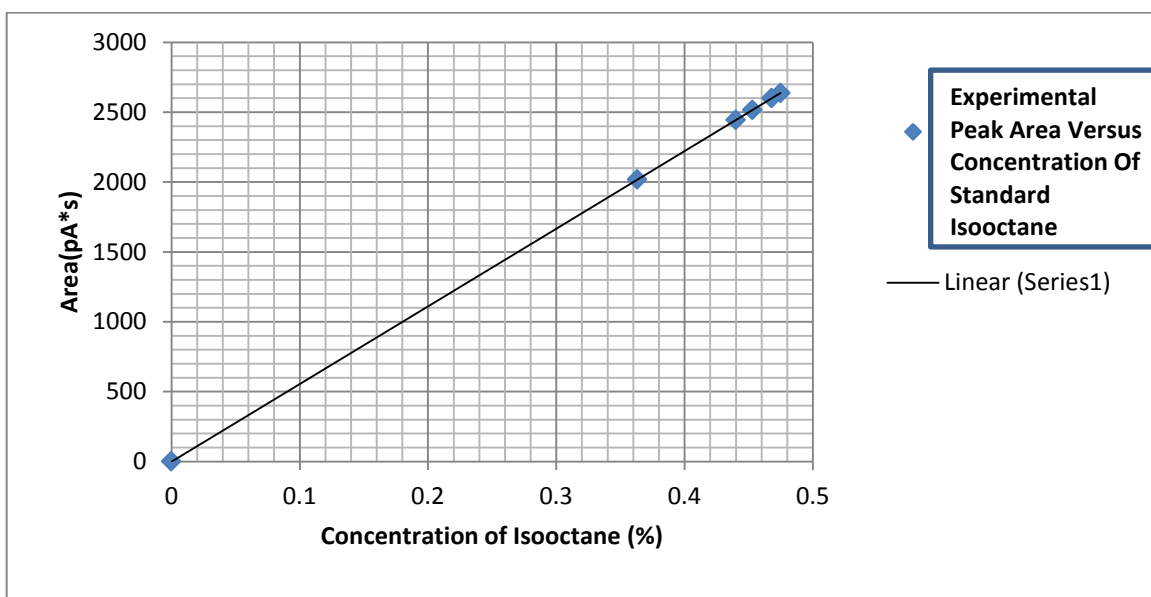
**Figure 4.7:** Concentration of Actual Isooctane Present In Samples 1:2



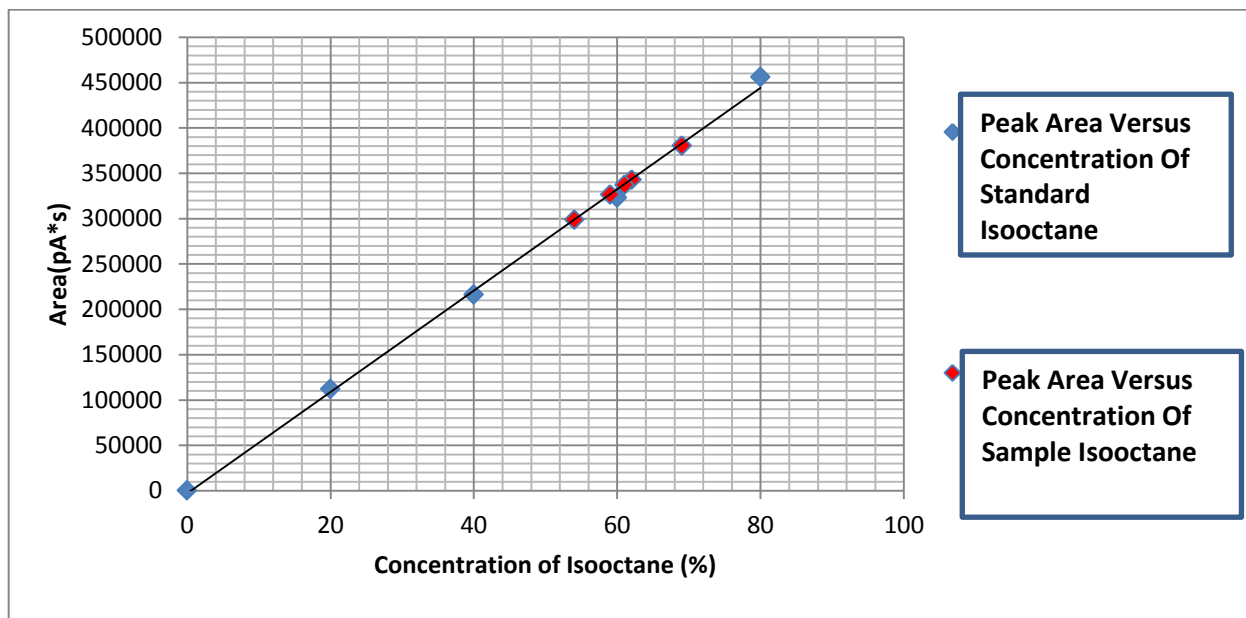
**Figure 4.8 :** Concentration of Experimental Isooctane Present In Samples 1:2



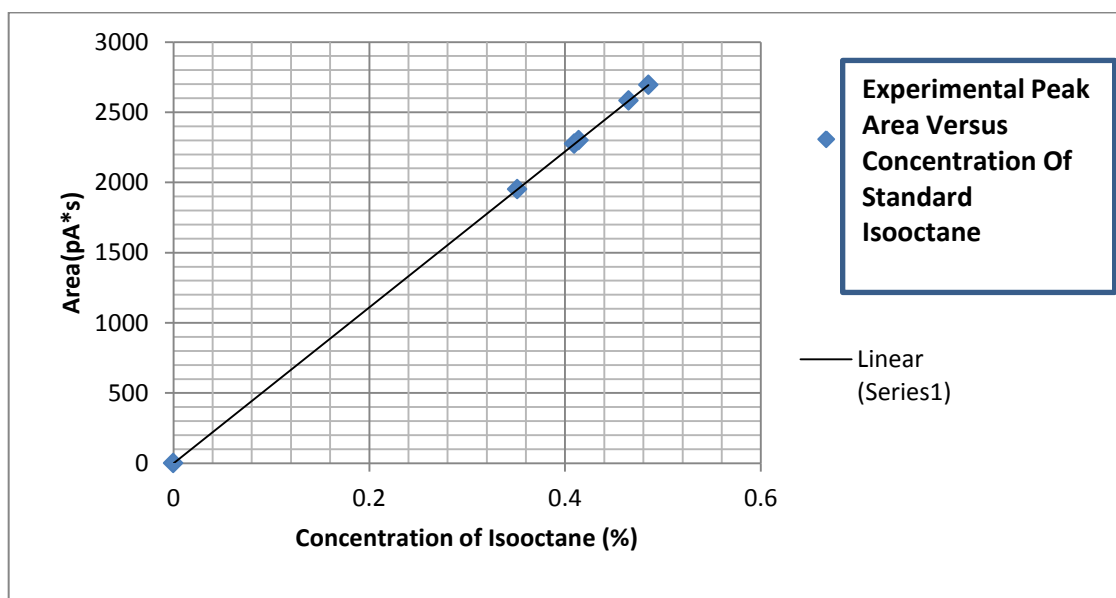
**Figure 4.9** :Concentration of Actual Isooctane Present In Samples 1:3



**Figure 4.10** Concentration of Experimental Isooctane Present In Samples 1:3



**Figure 4.11:**Concentration of Actual Isooctane Present In Samples 1:4



**Figure 4.12:**Concentration of Experimental Isooctane Present In Samples 1:4

#### **4.4 DISCUSSION**

Catalytic cracking method is used in order to break the long hydrocarbon chain into smaller, simpler and more useful bits of hydrocarbon compounds. The long-chain hydrocarbon molecules are first broken in a fairly random way to produce mixtures of various smaller hydrocarbon radicals, and then these free radicals will recombine in different arrangements through isomerization process. The desired Isooctane molecules are formed through this isomerization process. The presence of Isooctane produced in the rubber seed oil indicating that bio-petrol can be produced in this process.

#### **4.5-1 Various Types of Fatty Acids present in the RSO**

Rubber seed oil is a mixture or combination of various fatty acids such as oleic acid and stearic acid. Due to the various types of fatty acids present in the rubber seed oil, the percentage yield of Isooctane concentration in the samples will be very high as there will be multiple choices for the formation of Isooctane during the catalytic cracking process. It is because, during the catalytic cracking process, the Zeolite catalyst can react with any one of the various types of fatty acids in the rubber seed oil to form Isooctane. Rubber seed oil consists of mainly saturated fatty acids that have long chains with no double bonds. In addition, certain longer chain fatty acids that are present in the rubber seed oil could not be detected as they tend to have physical properties of oil than that of the fatty acid properties. Therefore, it is possible for the catalyst particle to react with each and every type of the fatty acids present in the rubber seed oil for the formation of Isooctane in the sample. In addition to that, each and any of the C-C bonds can be broken up to form Isooctane. This is logical as the Zeolite catalyst can swell to several times of its original volume as discussed earlier and hence, more percentage of Isooctane concentration can be formed.

#### **4.5-2 Contamination Factor**

During the experiment, there might be impurities present in the apparatus and glassware used that will affect the determination of Isooctane especially during the usage of Gas Chromatogram. Besides that, contamination factor can occurred during the solidification of the distillate product after the distilled product is collected and removed from the source of heat. Isooctane is a liquid at room temperature but the unreacted fatty acid will affect the Isooctane formation as the temperature falls below the melting point of the fatty acids. The more the sample is dominated by the distilled product, the higher chances for the sample to be solidified.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATIONS

#### 5.1 CONCLUSION

The purpose of this research is to extract rubber seed oil from rubber seeds to be produced into Isooctane (Bio-petrol) using Zeolite catalyst for the catalytic cracking process. This is very important especially for the application of Bio-petrol produced from agricultural material (rubber seeds) in the transportation sector to substitute the diminishing supply of fossil fuels. Besides that, the bio-petrol produced from agricultural material can help to minimize the pollution of our mother earth with its capability of reducing the emission of green house gases to the environment by promoting better efficiency of complete burning inside the car engines.

From the data obtained, the result shows that the concentration of Isooctane present in samples affected by amount of solvent used in extraction process. It is because in this research, only 25ml of RSO is added with 5grams of catalyst which will produces a thick mixture. When there is more catalyst than sample oil in the catalytic cracking, it can be assumed that all the long chains of fatty acid in RSO are broken to form Isooctane.

The higher ratio of samples diluted does not mean that more yield of Isooctane concentration can be produced. The high and decreasing percentage of concentration of



Isooctane in the samples can be explained based on the cause of interlayer spacing of catalyst structure, larger surface area for reactions to occur, various types of fatty acid mixture present in the rubber seed oil, and the contamination factor.

To conclude, Isooctane can be synthesized using Zeolite catalyst in the catalytic cracking of rubber seed oil extracted from rubber seeds based on the Gas Chromatogram analysis data. In other words, catalytic cracking of fatty acids present in the rubber seed oil is very beneficial in producing a renewable source of energy to replace the diminishing supply of the polluting fossil fuels in the future. However, this alternative method should be constantly updated with further modern technology in terms of techniques, economics, environment and the feasibility for large scale or industrial usage.

## **5.2 RECOMMENDATIONS**

In order to obtain more precise results, it is best recommended to modify the vertical axis peak area of the chromatogram to eliminate or to minimize all the other smaller concentration of impurities present in the diluted samples. Through this modification, the elimination of other impurities will help to show a more accurate percentage of concentration of Isooctane in the samples.

Besides that, the presence of other impurities in the samples can be eliminated by using cleaner glassware and apparatus. For example, the glassware should be disinfected and dry cleaned first before being used in the experiment. It is because the presence of impurities in the samples will affect the percentage yield of Isooctane.

In addition, human errors such as parallax reading should be minimized to the lowest extent to achieve better results. Human errors can be minimized by taking the average values of each measurement or data reading taken.

Lastly, in order to obtain more rubber seed oil for the formation of Isooctane through the catalytic cracking process, it is better to use fresh rubber seeds plucked from trees immediately to prevent any loss of fatty acid contents in the rubber seeds for a higher conversion of rubber seeds to produce rubber seed oil.

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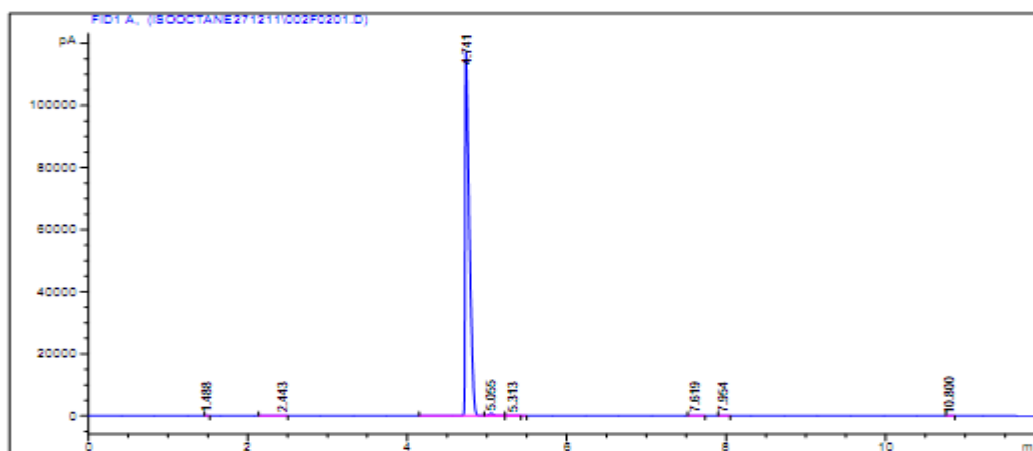
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## APPENDIX A

GC FID Analysis: 1% isooctane dilution



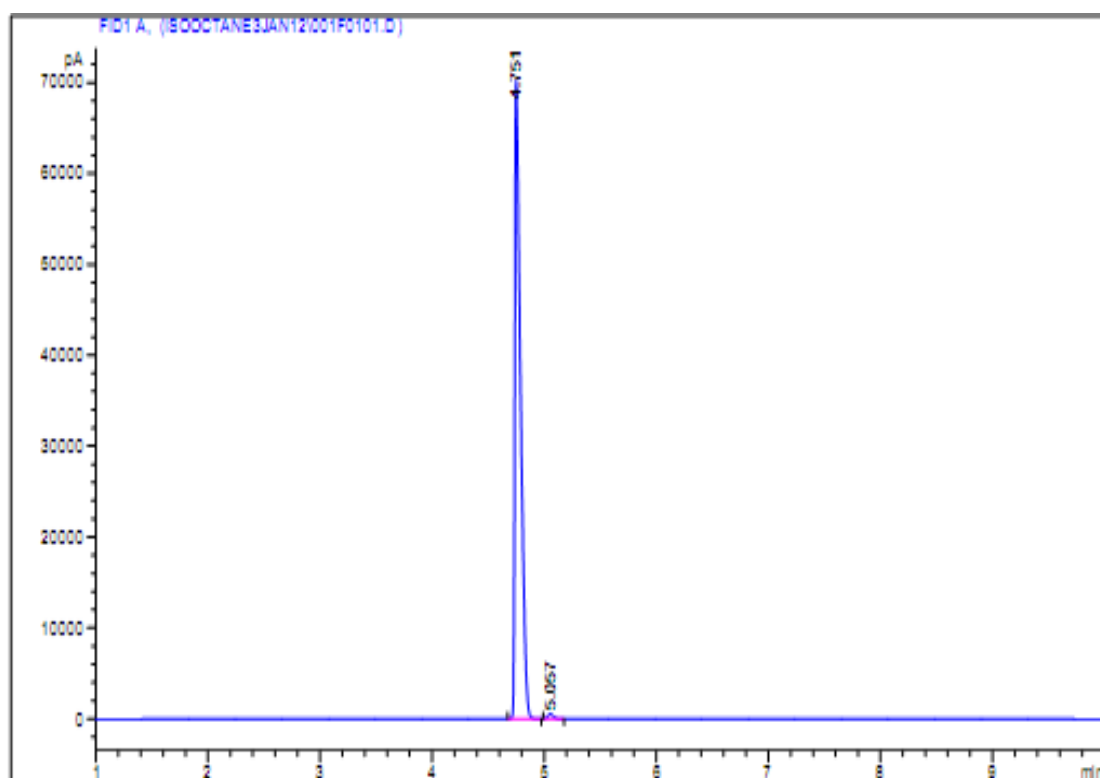
```
=====
                          Area Percent Report
=====
```

```
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	1.488	SV	0.0311	1.016466	4.54415	0.00024
2	2.443	SB	0.0363	1.01566	4.118825	0.00024
3	4.741	VB S	0.0533	4.195025	11.10025	99.315524
4	5.055	SV X	0.0532	20.465582	85.110335	0.677668
5	5.313	VB X	0.0546	20.965582	86.013385	0.04968
6	7.619	VB	0.0644	1.11046	2.5	0.00026
7	7.954	SB	0.0436	2.07164	6.41741	0.00049
8	10.800	SB	0.059	1.39436	6.039224	0.00033
9	12.730	VB	0.0427	1.06686	3.2201	0.00025

## GC FID Analysis: 5% isooctane dilution

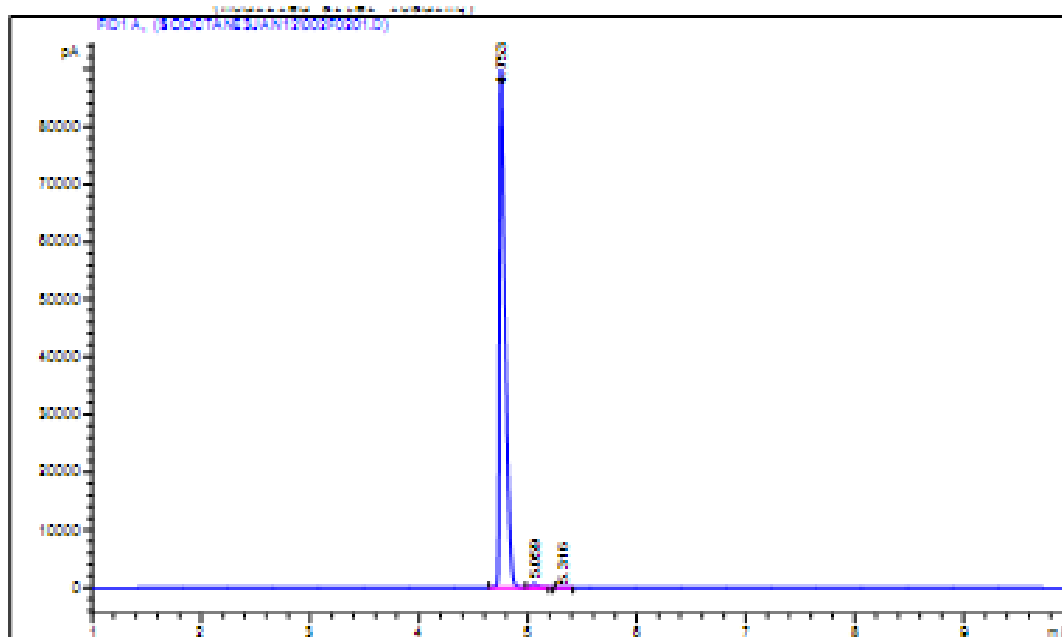
=====  
Area Percent Report  
=====

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	4.751	BB S	0.0499	2.45523e5	6.99695e4	99.32259
2	5.057	BB	0.0512	1674.54102	511.33301	0.67741

## GC FID Analysis: 10% isooctane dilution



```

-----
                          Area Percent Report
-----

```

```

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

```

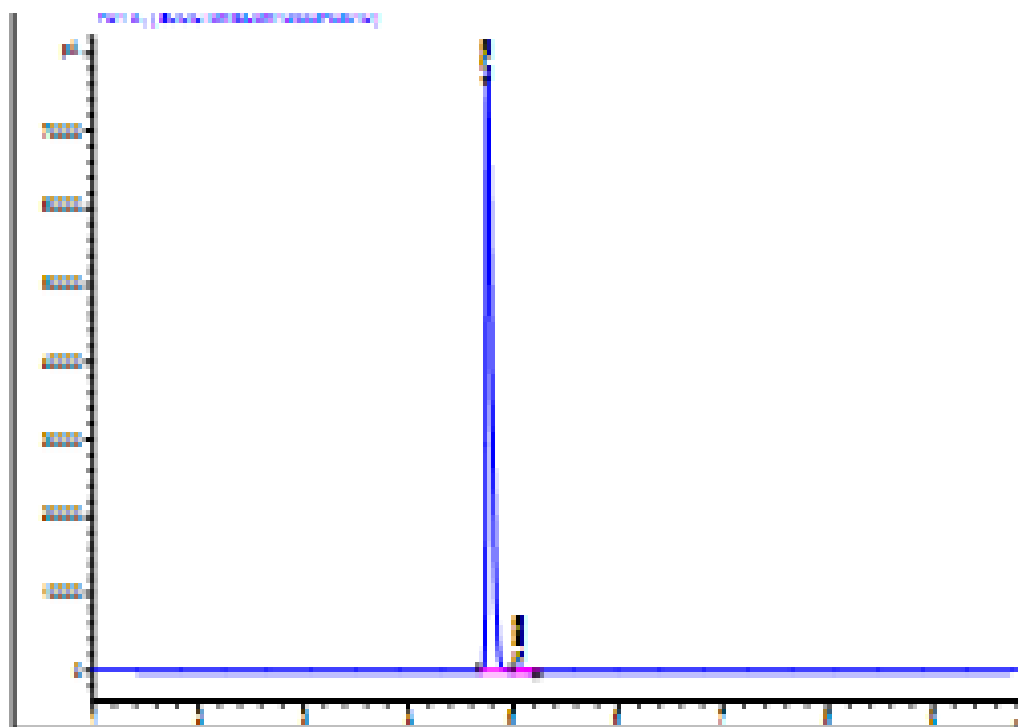
```

Signal 1: FID1 A,

```

Peak #	RetTime (min)	Type	Width (min)	Area (pA*min)	Height (pA)	Area %
1	4.753	SS S	0.0518	3.21769e5	8.98113e4	99.24532
2	5.059	SS T	0.0525	2399.13892	709.12183	0.73998
3	5.318	SS	0.0548	47.84848	13.82182	0.01470

## GC FID Analysis: 15% isooctane dilution



Area Percent Report

## Area Percent Report

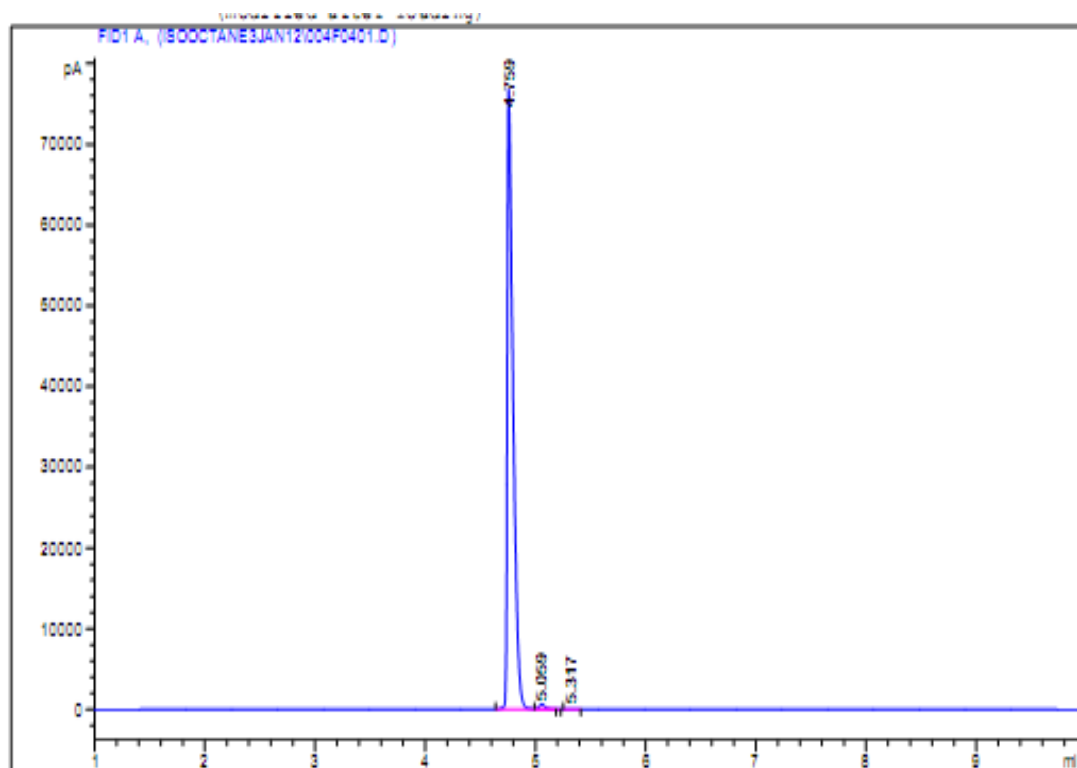
Area Percent Report

Method By: Signal  
 Multiplier: 1.0000  
 Dilution: 1.0000  
 Use Multiplier & Dilution Factors with IRTCs

Signal: 1: 15% isooctane dilution

Peak	Retention Time [min]	Type	Width [min]	Area [pA*min]	Height [pA]	Area %
1	4.788	IRTC	0.0020	3.82442e8	7.78187e4	99.9974
2	8.042	IRTC	0.0017	1868.14473	843.42271	0.00259

## GC FID Analysis: 15% isooctane dilution



```
=====
                          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	4.759	BB S	0.0522	2.77333e5	7.66497e4	99.23643
2	5.059	BB T	0.0524	2086.08130	617.67920	0.74645
3	5.317	BB	0.0559	47.83094	13.33977	0.01712

**APPENDIX B****1. Calculation for Sample and Solvent (Hexane)**

a)  $1\% \text{ of Sample} \times 10 \text{ mL} = 0.01 \text{ mL}$

$99\% \text{ of Hexane} \times 10 \text{ mL} = 9.99 \text{ mL}$

b)  $5\% \text{ of Sample} \times 10 \text{ mL} = 0.05 \text{ mL}$

$95\% \text{ of Hexane} \times 10 \text{ mL} = 9.50 \text{ mL}$

c)  $10\% \text{ of Sample} \times 10 \text{ mL} = 0.10 \text{ mL}$

$90\% \text{ of Hexane} \times 10 \text{ mL} = 9.00 \text{ mL}$

d)  $20\% \text{ of Sample} \times 10 \text{ mL} = 0.20 \text{ mL}$

## 2. Back Calculation 1% Sample + 99% Hexane

$$\begin{aligned}
 \text{Actual peak area isooctane (\%)} &= [\text{peak area isooctane (\%)} / [100 - \text{peak area hexane} \\
 &\quad (\%)]] * 100 \\
 &= [9.36010 / (100 - 90.53465)] * 100 \\
 &= 98.888\%
 \end{aligned}$$

$$\begin{aligned}
 \text{Actual peak area (pA*s)} &= [\text{peak area isooctane (pA*s)} / \text{peak area isooctane (\%)}] * \text{actual} \\
 &\quad \text{peak area of isooctane (\%)} \\
 &= [2.62409 * 10^4 / 90.53465] * 98.888\% \\
 &= 277231.1642 \text{ pA*s}
 \end{aligned}$$

From the equation shown in standard calibration curve

$$Y = 5553X$$

Where,

y = Actual Peak area of isooctane (pa\*s)

x = Actual concentration of isooctane (%)

$$\begin{aligned}
 \text{Actual concentration of Isooctane (\%)} &= (\text{actual peak area (pA*s)} / 5553) \\
 &= ((277231.1642 / 5553)) \\
 &= 50\%
 \end{aligned}$$

## APPENDIX C

## Material Safety Data Sheet (MSDS)

## Safety data for hexane



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**General**

Synonyms: n-hexane, normal hexane, hexyl hydride

Molecular formula:  $C_6H_{14}$

CAS No: 110-54-3

EC No: 203-777-6

EC Index No: 601-037-00-0



**Physical data**

Appearance: colourless liquid

Melting point: -95 C

Boiling point: 69 C

Vapour density: 3 (air = 1)

Vapour pressure: 132 mm Hg at 20 C

Specific gravity: 0.659

Flash point: -10 F

Explosion limits: 1.2% - 7.7%

Autoignition temperature: 453 F

**Stability**

Stable. Incompatible with oxidizing agents, chlorine, fluorine, magnesium perchlorate. Highly flammable. Readily forms explosive mixtures with air. Note low flash point.

**Toxicology**

May cause impaired fertility. Harmful by inhalation. Irritant. May cause CNS depression. Prolonged exposure may cause serious health damage.

**Environmental information**

Harmful in the environment - may cause long-term adverse effects.

**Personal protection**

Safety glasses. Effective ventilation. Remove sources of ignition from the working area.