BIOPETROL SYNTHESIZED FROM RUBBER SEED OIL BY ZEOLITE CATALYST: EFFECT OF ETHANOL IN SOLVENT EXTRACTION OF RUBBER SEEDS

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REGUPATHI RAJA PILLAI A/L GANASEKARAM

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

Faculty of Chemical and Natural Resources Engineering
University Malaysia Pahang

JANUARY 2012

DECLARATION

I declare that this thesis entitled *"Biopetrol Synthesized from Rubber Seed Oil By Zeolite Catalyst: Effect Of Ethanol In Solvent Extraction Of Rubber Seeds"* is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ……

Name : Regupathi Raja Pillai A/LGanasekaram

Date : January 2012

DEDICATION

To my beloved father, mother, sister and my dearest friends

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ABSTRACT

Currently, most of the bio-petrol is produced from the refined or edible type oils such as the utilization of palm oil for the production of more environmental friendly bio-fuels. However, by extracting rubber seed oil (RSO) from the rubber seeds, this method is more favorable as the RSO contains more fatty acid to be produced into bio-petrol. The rubber seeds are readily available, cheap and help to improve the socioeconomic issues. The RSO 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 ethanol in the receiving flask is left to boil until it vaporizes and condensed, filling up the main chamber, extracting the RSO from the rubber seeds. The catalytic cracking of the mixture of 0.1L of RSO, 20g of catalyst and bumping chips at 300^0C for 45 minutes using Zeolite catalyst 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 big. 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 RSO, the incorrect chromatogram modifications and the contamination in RSO. The mass of catalyst and volume of RSO used will affect the percentage of concentration of Isooctane in samples. As a conclusion, Bio-petrol can be produced from RSO using Zeolite catalyst in the catalytic cracking process.

ABSTRAK

Pada masa kini, kebanyakkan bio-petrol dihasilkan daripada minyak tumbuh-tumbuhan yang boleh dimakan contohnya seperti minyak pokok kelapa sawit kerana bio-petrol yang dihasilkan mengunakan mempunyai ciri-ciri mesra alam. Dengan mengekstrak minyak biji getah dari biji getah kita akan mempunyai minyak yang mengandungi kandungan acid lemak yang tinggi yang boleh diproses untuk dijadikan kepada bio-petrol. Biji-biji getah sangat mudah diperolehi dan harganya adalah amat murah. Minyak biji getah diekstrek mengunakan kaedah Soxhlet. Biji getah yang dibersih, dikupas kulitnya dan dikisar dimasukkan ke dalam timble yang kemudian diletak di ruang utama Soxhlet ekstraktor. Pelarut Etanol dalam kelalang penerima dibiarkan mendidih sehinggalah ia menyejat dan mengisi ruang utama dan mengekstrak minyak biji getah dari benih getah. 0.1L minyak biji getah dicampur dengan 20g pemangkin zeolite dan dipanaskan pada suhu 300^0C selama 45 minit. Pemangkin zeolite digunakan adalah untuk meningkatkan kadar tindak balas antara zarah-zarah. Kehadiran Isooctane dalam sampel dikesan menggunakan Kromatogram Gas dan inin menunjukkan bahawa bio-petrol dapat dihasilkan menggunakan minyak benih getah. Standard campura nisbah yang berbeza hexane dan Isooctane telah digunakan untuk mendapatkan kromatogram untuk Isooctane sehingga keluk penentukuran diplotkan hasil daripada kepekatan Isooctane yang dihasilkan. Keputusan menunjukkan bahawa kepekatan sebenar Isooctane adalah sangat besar. Hal ini disebabkan oleh minyak sample yang tidak larut sepenuhnya dalam pelarut hexane, keretakan dan pengisomeran rawak semasa proses keretakan rantai hydrocarbon dengan menggunakan pemangkin dan pencemaran minyak benih getah. Jumlah isipadu pelarut yang digunakan akan mempengaruhi peratus kepekatan Isooctane dalam sampel. Kesimpulannya, bio-petrol dapat dihasilkan daripada minyak benih getah dengan menggunakan zeolite sebagai pemangkin.

TABLE OF CONTENT

2 LITERATURE REVIEW

3 METHODOLOGY

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

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

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Introduction

Environmental pollution is the greatest treat which is faced by the world in these modern times. Environmental pollution is defined as the introduction of contaminants into an environment that causes instability, disorder, harm or discomfort to the ecosystem. There are many factors which contribute to the current pollution level of the world and one of the major factors is combustion of fossil fuels. Fossil fuels are defined as any carbon-containing fuel derived from the decomposed remains of prehistoric plants and animals. Some of the examples of fossil fuels are coal, petroleum and natural gas. Combustion of fossil fuel produces a very high amount of carbon dioxide. The high concentration of carbon dioxide in the atmosphere is the prime factor for today's global warming phenomena.

Today the accessible oil reservoirs which are the major sauce of the fossil fuels are gradually depleting , it is very important to develop an appropriate long-term strategies based on the utilization of the renewable fuel that will gradually substitute the depleting fossil fuel production (Westermann et. 2007). So it has become a top priority for developing or developed countries to find a sustainable fuel which is cheap and easy to

mass produce. One of the major solutions to overcome the problem is by using bio-petrol or the alternative petrol as a renewable and biodegradable type of fuel.

The bio-petrol is majorly used in transportation sector. Bio-petrol or biomass is produced from plants or from agricultural waste. The ethanol production for transport fuel tripled between 2000 and 2007 from 17 billion to more than 52 billion liters, while biodiesel expanded eleven-fold from less than 1 billion to almost 11 billion liters. Altogether bio-fuels provided 1.8% of the worlds transport fuel. Recent estimate indicate a continued high growth. From the time frame of 2007 to 2008 the share of ethanol in global gasoline type fuel use was estimated to increase from 3.78% to 5.46% and the share of biodiesel in global diesel type fuel use from 0.93% to 1.5%.

So from the numbers above it is positive that many countries have agreed that bio-fuel production is the relevant choice to solve the depleting oil reserve problem. Bio-fuels do poses several advantages compare to fossil fuel. Those advantages are renewability, sustainability, common availability, reduction of greenhouse gases emission and its biodegradability.

The saturated fatty acids contain high potential long hydrocarbon chain alkanes which are perfect for making petrol. By using thermal cracking process the fatty acid in the rubber seeds will be broken so that it can be used to produce bio-petrol. The thermal cracking its self doesn’t produce sufficient amount of product so a homogenous catalytic cracking is used to break up the large oil molecules into small molecules which will increase the rate of reaction between the liquid fatty acid and the solid catalyst.

The solid catalyst which will be used in this case is zeolite. The zeolite has been chosen due its quality of being a good absorbent.

1.2 Research Background

Finding a proper substituent for the depleting fossil fuel has become an important research now days. Various researches have been made so that a cheap and sustainable energy source can be found so that we could match up with the increasing demand for fuel. Production of bio-petrol not only matches up with the cheap and sustainable energy criteria but it also able to reduce the pollution towards the environment. Based on the research of production of bio-diesel from rubber seeds done by Department of Mechanical Engineering, National Institute of Technology Calicut, the rubber seed oil has high amount of free fatty acid. The free fatty acid content of unrefined rubber seed oil was about 17%. From the journal of A.S RAMADAS 2004 it has been stated that rubber seed kernels (50% – 60% of seed) contain 40% – 50% of brown color oil. It also has been stated that in its natural form the rubber seed oil has high acidic functional groups such as ester, methylene, terminal methyl and glyceryl.

1.3 Problem Statement

Nowadays fossil fuel price has become a crucial factor which will determine the prices of other goods in the market. It can be said that the price of fuel is proportional to the prices of the goods. So if the price of the fuel increases the prices of other products in the market will also increase. Today the price of the fuel is not at a fixed position and it is keeping on increasing due to the fact of depleting oil reserves. This scenario currently occurs not only in Malaysia, but also involving all other countries in the world. Figure 1.1 proves this scenario occurrence in United States of America.

As an example in Malaysia only, the price of Malaysia petrol has been raise by over 40% in the year 2008. The price for petrol stood at RM 2.70 a liter, an increase of 78 sen while the price of diesel was shot up by RM 1.00 at RM 2.58. Although the prices were reduced to RM 2.15 for petrol and RM 2.50 for diesel by the following month but it was estimate that there will be increase in fuel prices in the following years.

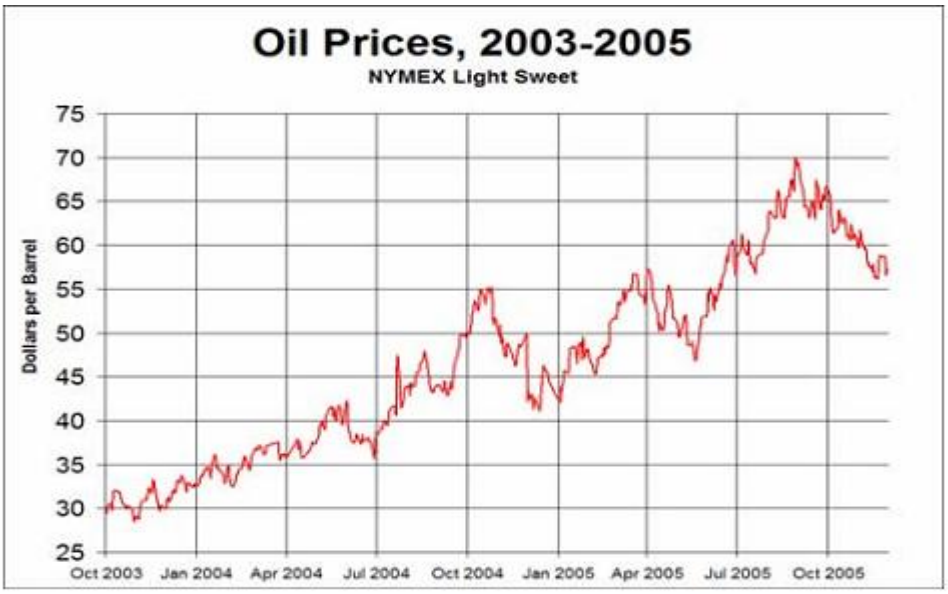

Figure 1.1: New York Mercantile Exchange

Increase in fuel prices would not be a problem if the income of the people does increase according to the fuel prices. But people's salaries will not be increased according to the increase of fuel price. So ordinary people have to bear a higher living cost with the same amount of salary. Production of bio-fuel can prevent this whole scenario from happening. Bio-fuel is better than the fossil fuel from the aspect of production cost because it would not be needed to drill deep into the earth and build factories to refine bio-fuel comparing with fossil fuels those should be refined first. Another factor which makes the bio-fuel to cost less than the fossil fuel is that the source for producing bio-fuel which are plants and agricultural waste are cheap and easy to get whereas on the other hand it is becoming difficult to find new oil reserves. So the rareness of the oil reserves makes the price for fossil fuel go sky high.

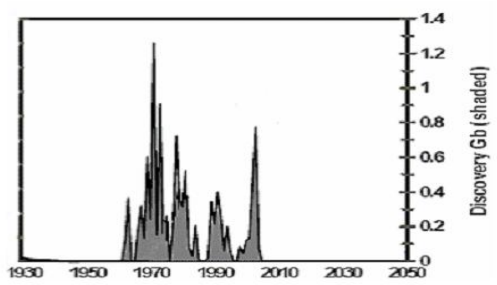

Figure 1.2: Annual Malaysian Discovery of Oil

Based on the Figure 1.2 the most amount of fossil fuel was discovered during the year 1973 and the discoveries of fossil fuel in the years that follows it always seems to be less than the amount in the year 1973. Many reasons can be stated for the short comings of the years after 1973 such as efforts for discovering new oil reserves at minimum level and instruments for discovering fossil fuel were not updated for the modern day standard. Although many reasons can be given for the situation above but the fact that cannot be ignored is the cold hard truth which is Malaysia's oil reserves are running out of fuel. The situation gets even worse when the demand for fuel increases on year by year basis as shown in Figure 1.3 below.

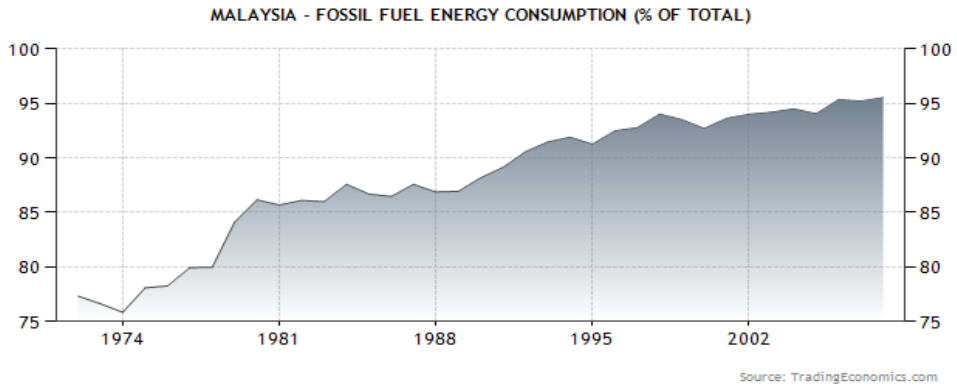

Figure 1.3: Malaysia - Fossil Fuel Energy Consumption

Limited recourses with increasing demand will only spell total chaos. To overcome this problem a lot of fuel sources in a small amount of time is required to be explored. So it is needed to discover more and more fuel reserves to satisfy the market demand. But the problem is even though the effort to find fossil fuel reserves is doubled, only a limited

number of fossil fuel reserves could be found because fossil fuel only can be formed after millions of years. So an alternative way is needed to produce fuel in a large quantity in a minimum amount of time. So the best alternative way is producing bio-fuels.

Bio-fuel is a renewable energy because it is produced from plants which are also renewable bio-organic materials. So it can be simply concluded that the bio-fuel would not diminish as long there are bio-mass. Another advantage of bio-fuel is it is biologically degradable and it will never pollute the environment.

One of the major side effects when it comes to the combustion of fossil fuels is the incomplete combustion between the mixture and the low ranking fuels. When fossil fuels burn efficiently in an excess of air or oxygen the only products are carbon dioxide and water for an example:

Examples of complete combustion burning are:

- Methane + oxygen ==> carbon + water

  $CH_{4\,(g)} + O_{2(g)} ==> C_{(s)} + 2H_2O_{(l)}$

- pentane + oxygen ==> water + carbon dioxide

  $C_5H_{12(g)} + 8O_{2(g)} ==> 5CO_{2(g)} + 6H_2O_{(l)}$

However, if there is not enough oxygen present to completely burn the fuel to carbon dioxide and water other products may form causing pollution and fuel inefficiency. This is referred to as incomplete combustion. The most common partially burned products are likely to be carbon-soot and deadly carbon monoxide (CO), as shown in Figure 1.4. Carbon-soot, a fine black powder-dust is potentially harmful and readily formed in fires and it's classically produced by smoky yellow flames. The soot, like any fine solid 'dust' is harmful when absorbed on the sensitive tissue of the linings of the nose, throat and lungs. Soot deposits cause coughing and sore throat and are ejected from your body through sneezing, coughing, and nose blowing. Coarse particles (10 microns) are inhaled into your

windpipe and settle there, causing irritation and more coughing. Soot is also a 'carrier' of polycyclic aromatic hydrocarbons (PAH's) on it which are carcinogenic. Even very low concentrations of carbon monoxide (CO) can be fatal. Oxygen is carried around the body by a complicated protein molecule in red blood cells called hemoglobin. The bonding between oxygen and hemoglobin is quite weak to allow easy oxygen transfer for cell respiration. Unfortunately, the bonding between carbon monoxide and hemoglobin is stronger, so oxygen is replaced by carbon monoxide and blocks normal cell respiration. The consequences are reduced blood oxygen concentration leading to unconsciousness and eventually death.

Figure 1.4: Percentage of Carbon Monoxide Release Test

The rubber tree (*Hevea brasiliensis*) is a plantation crop which is originated from South America and commercially cultivated around Southeast Asia in the year 1876. Every hector or rubber plantation will yield 100 to 150 kg of rubber seeds. The rubber seeds have

43% oil in composition (Nwokolo et al 1988). Rubber seed oil is a semi-dried substance (Aigbodin & Pillai 2000) that does not contain any unusual fatty acid but it is a rich source of polyunsaturated fatty acids $C_{18:2}$ and $C_{18:3}$ that makes up 52% its total fatty acid composition (Ghandhi et al 1990). Malaysia is an ideal country to start the mass production of bio-petrol from rubber seeds because Malaysia produces almost 20% of the world's natural rubber. A good deal of Malaysia's rubber comes from thousands of privately owned plots of land called small holdings, which are usually about 2 hectares. The rest is grown on big estates owned by various companies; each can cover over a thousand hectares. Altogether, Malaysia has 1.7 million hectares of rubber. So with millions of hectares of rubber plantation it's very to obtain the rubber seeds.

Table 1.1: Properties of Vegetable oil [Knothe G., R.O. Dunn and M.O. Bagby, 1998. Biodiesel: the use of vegetable oils and their derivatives as alternative diesel fuel.]

Property	**Rubber seed oil**	**Sunflower oil**	**Rapeseed oil**	**Cotton seed oil**	**Soybean oil**
Fatty acid composition (%)					
(i) Palmitic acid $C_{16:0}$	10.2	6.8	3.49	11.67	11.75
(ii) Stearic acid $C_{18:0}$	8.7	3.26	0.85	0.89	3.15
(iii) Oleic acid $C_{18:1}$	24.6	16.93	64.4	13.27	23.26
(iv) Linoleic acid $C_{18:2}$	39.6	73.73	22.3	57.51	55.53
(v) Linolenic acid $C_{18:3}$	16.3	0	8.23	0	6.31
Specific gravity	0.91	0.918	0.914	0.912	0.92
Viscosity (mm^2/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

The above Table 1.1 shows the comparison of rubber seed oil to other type of oils. Based on the comparison, it can be observed that rubber seed oil poses the ideal qualities to give higher yield of bio-petrol. Rubber seed also less expensive compared to the plant oils because of its non-edible feature.

1.4 Objective

- To synthesize isooctane from the rubber seeds
- To determine the concentration of isooctane by hereterogeneous catalytic cracking of fatty acid using Zeolite

1.5 Research Scope

In order to accomplish the objectives, following are the criteria which are the scope of this research focusing on:

1) The extraction of rubber seed oil from rubber seeds by using Soxhlet Extraction
2) The usage of catalytic cracking process to crack the fatty acid complex into smaller hydrocarbon molecules.
3) Using the Gas Chromatography method to determine the concentration of Isooctane.

1.6 Rational and Significance

Below are the rationale and significance statements of synthesizing bio-petrol from rubber seed oil:

a) Bio-petrol is an environmental-friendly fuel and can reduce greenhouse gases emission.
b) Bio-petrol is a renewable source of energy and it is biodegradable.
c) The source of the bio-petrol which is rubber seed can be obtained in vast number because in Malaysia there are 1.7 million hector of rubber plantation.
d) Bio-petrol has higher oxygen levels which are from 15% to 45% while fossil fuels have none and this makes bio-petrol more useful than fossil fuel chemically.
e) Catalytic cracking provides higher conversion of hydrocarbon than thermal cracking does by lowering the activation energy of the reaction.

CHAPTER 2

LITERATURE REVIEW

2.0 Introduction

There are many types of energy in this world for an example nuclear energy, hydraulic energy, wind energy, thermal energy and geothermal energy. Every kind of energy has its source of energy and one most important source for thermal energy is fossil fuel. Fossil fuels are one of the most expensive fuel sources in the world and one of the rarest to come by. Due to its rarity and high production cost the fossil fuels price has been increasing on yearly basis.

To prevent this energy crisis alternative energy sources has been looked into so that the increasing demand for energy can be fulfilled. One of the energy sources which is on top of the list is solar energy. Solar energy is being harnessed from the sun. This energy comes in the form of radiation which makes it possible to produce solar electricity from it. The solar energy is important for all life form on earth. Solar rays are the important ingredient in photosynthesis which is an essential process to produce oxygen by plant for all living organism. Furthermore solar energy helps to grow our food, light our days, influence weather patterns and provide heat. Solar energy is harvested by using various techniques such as the use of photovoltaic panels and solar thermal collectors. Photons contained in this solar radiation make the generation of electricity from the sun's rays possible.

Another source of energy which has considered as an ideal alternative energy is geothermal energy. Geothermal energy is the heat energy which is harnessed from within the earth and the energy which has been harnessed will be used to heat water and make steam to turn generator turbines and make electricity. Earth's source of geothermal energy comes from radioactive decay of minerals, from volcanic activity, and from solar energy absorbed at the surface. The term geothermal gradient, is the difference of temperature between the core of the planet and its surface, drives a continuous conduction of thermal energy in the form of heat from the core to the surface. The heat from hot springs which is one form of geothermal energy has been used for bathing since Paleolithic times but nowadays it is better known for generating electricity. Geothermal power has many advantages to it such as cost effective, reliable, sustainable, and environmentally friendly, but it has its share of its disadvantages. Geothermal energy can be found only at tectonic plate boundaries. So for countries such as Malaysia which is situated outside the "Pacific Ring of Fire" it's hard to acquire geothermal energy.

Besides geothermal energy and solar energy there is another type of energy which shows a promising future as an alternative which is Hydropower. Hydropower is generated by using the power of moving water to drive a water turbine and a generator. The amount energy obtained is based on the height of the water level from the water outlet and the volume of water flowing out. The difference in the water level is called the as head. The amount of potential energy contain in water is proportional to the head. To deliver water to a turbine while maintaining pressure arising from the head, a large pipe called a penstock may be used. Malaysia currently has 13 dams to harness the hydro electric so it is safe to say that hydropower has a great prospect for growing in Malaysia.

Although hydropower, solar energy and geothermal have a lot of ideal qualities to become the ideal alternative energy source but the most suitable fuel source is bio-fuel. The term biofuel or biorenewable fuel (refuel) is referred to as solid, liquid or gaseous fuels that are predominantly produced from biomass (Chhetri AB *et al*., 2008). Liquid biofuels being considered world over fall into the following categories: (a) bioalcohols (b) vegetable oils and biodiesels, and (c) biocrude and synthetic oils (Demirbas A *et al*., 2008). Bio-ethanol is made from fermenting the sugar components from the plant and it is made mostly

from sugar and starch crops. With advancement in technology bio-fuels have been extracted from lingo-cellulosic biomass, such stalks of wheat, corn stover, wood and rubber seeds. Compared to the other sources of energy bio-fuel would be the most ideal form of alternative energy because if we use the other sources of energy we need to alter completely the current system of operation for an example if we want to use solar power in our car we need to remove the current engine and have to install specialized solar cell but if we use bio-fuel to power our vehicle we still can use the same engine without any extra modification. So by using bio-fuels we still can use the same system used for fossil fuels and still enjoy all the privileges of a non-polluting energy source. The Figure 2.1 below shows the percentage of use of renewable energy in 2006.

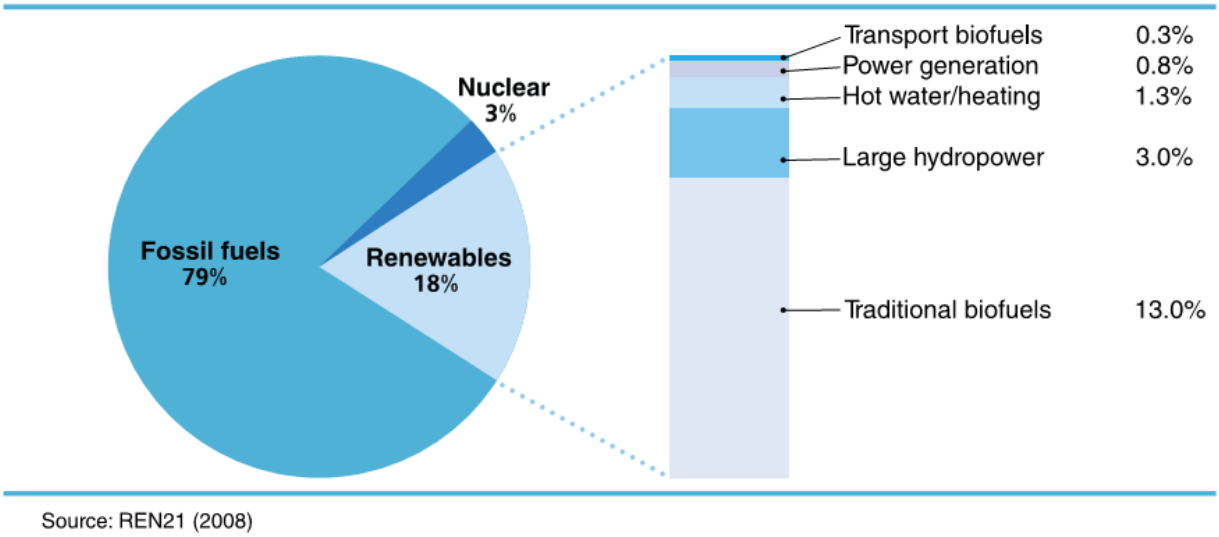

Figure 2.1: Renewable Energy Share of Global Energy Consumption in 2006

2.1 Biofuel

Bio-fuels are the materials that can be combusted which are produced from biomass such as plants, animals and micro-organism. Bio-fuels are liquid or gaseous fuels for the transport sector that are produced from renewable sources [Demirbas, 2007]. The energy acquired for bio-fuels are called as bio-energy. The bio-energy consists of bio-fuel for transportation purposes and processed biomasses which are perfect for heat and electrical

generation. Bio-fuel can be differentiated into three different classes which are first-generation bio-fuels, second generation bio-fuels and third generation bio-fuels.

First generation bio-fuels are produced using conservative methods. The feed for the process would be sugar cane, rapeseed, wheat, sunflower seeds or oil palm. The second generation bio-fuels differ from the first generation fuel sources in the sense that bio-fuel will be produced from different fuel sources. Initially the feed for the first generation feed source from edible materials such as sugar cane and sunflower seeds but in the second generation bio-fuels were produced from non-food sources such as waste biomass and wood. The third generation bio-fuel consists of oilgae which is bio-fuel from algae. Fuels such as bio-propanol and bio-butanol are some of new fuels which are the product of third generation bio-fuel but these fuels are not available in the market yet due to the fact that there is minimum amount of production experiences. The Table 2.1 shows the list of first, second and third generation biofuel.

Table 2.1: Types of Bio-fuel

Table 2.1: Types of biofuels – overview with basic technologies, important feedstocks and examples of co-products

Biofuel	Basic technology	Feedstocks	Co-products
Solid biofuels *	Traditional use of dried biomass for energy	Fuel wood, dried manure	
First generation biofuels			
Plant oils **	1) As transport fuel: Either adaptation of motors to the use of plant oils; or modification of plant oils to be used in conventional motors 2) For generation of electricity and heat in decentralised power resp. CHP stations	1) Rapeseed oil, sunflower, and other oil plants, waste vegetable oil 2) Rapeseed oil, palm oil, jatropha, and other oil plants	Oilcake as animal feed
Biodiesel	Transesterification of oil and fats to provide fatty acid methyl ester (FAME) and use as transport fuel	- Europe: Rapeseed, sunflower, soya - USA: Soya, sunflower; - Canada: Soya, rapeseed (Canola) - South- and Central-America: Soya, palm, jatropha, castor - Africa: Palm, soya, sunflower, jatropha - Asia: Palm, soya, rapeseed, sunflower, jatropha	- Oilcake as animal feed; - Glycerine; - Oilcake in some palm oil mills used for energy recovery
Bioethanol	Fermentation (sugar); hydrolysis and fermentation (starch); use as transport fuel	- USA: Corn - Brazil: Sugar cane - Other South- and Central-America: Sugar cane, cassava - Europe: Cereals, sugar beets - Canada: Maize, cereals; - Asia: Sugar cane, cassava; - Africa: Sugar cane, maize	- Maize and cereals yield animal feed DDGS (Dried Distillers Grains with Solubles). - Sugar cane bagasse is used for energy recovery

Biogas (CH_4, CO_2, H_2)	Fermentation of biomassused either in decentralised systems or via supply into the gas pipeline system (as purified biomethane); 1) For generation of electricity and heat in power resp. CHP stations 2) As transport fuel: either 100% biogas fuel or blending with natural gas used as fuel	Energy crops (e.g. maize, miscanthus, short rotation wood, multiple cropping systems); biodegrable waste materials, including from animal sewage	Residues used as fertiliser (nutrient recycling)
Solid biofuels	1) Densification of biomass by torrefaction or carbonisation (charcoal); 2) Residuals and waste for generation of electricity and heat (e.g. industrial wastes in CHP)	Wood, grass cuttings, switchgrass; grains; charcoal, domestic refuse, and dried manure	
Second generation biofuels			
Bioethanol	Breakdown of cellulosic biomass in several steps incl. hydrolysis and finally fermentation to bioethanol	Ligno-cellulosic biomass like stalks of wheat, corn stover and wood; special-energy-or-biomass crops (e.g. Miscanthus); sugar cane bagasse	
Biodiesel and range of "designer"-biofuels such as biohydrogen, biomethanol, DMF***, Bio-DME****, mixed alcohols	Gasification of low-moisture biomass (<20% water content) provides "syngas" (with CO, H_2, CH_4, hydrocarbons) from which liquid fuels and base chemicals are derived	Ligno-cellulosic biomass like wood, straw, and secondary raw materials like waste plastics	Fischer-Tropsch synthesis can be used to produce various feedstocks for chemical industry (not only for fuel but also e.g. plastics)
Third generation biofuels			
Biodiesel, aviation fuels, bioethanol, biobutanol	Bioreactors for ethanol (production can be linked to sequestering carbon dioxide from power plants); Transesterification and pyrolysis for biodiesel; other technologies under development	Marine macro-algae micro-algae in ponds or bioreactors	High-protein animal feed,biopolymers, agricultural fertilisers

[`] Traditional use of biomass included for complete overview
[``] Also known as straight vegetable oil. Plant oil used as direct fuel in transport is common in German agriculture with about 838,000 tonnes mostly rapeseed oil in 2007, representing 1.4% of total fuel consumption in transport.
[```] 2,5-Dimethylfuran.
[````] Dimethyl ether

Source: own compilation after different sources

The use of bio-fuels decreases the external energy dependence, promotion of regional engineering, increased Research and Development (R&D), decrease in impact of electricity production and transformation, increases the level of services for the rural population, creation of employment, etc [Miguez JL et al., 2006] [Demirbas A et al., 2006][Balat M, 2008]. In recent days bio-fuels such as bio-ethanol has caught the eyes of the public due to the fact that it is more advantageous to use bio-fuel than the conventional fossil fuel. Following are some of the advantages which bio-fuel posses over the traditional fossil fuel:

- Bio-fuel are vastly available from many biomass sources
- The feed product are easy to get
- Environmental friendly
- Cheaper in price compared to fossil fuel
- Biodegradable and sustainable

Many people do wonder how the bio-fuel can adapt to the already existing fuel engine. But the truth of the matter is bio-fuel such as bio-diesel can easily operate the diesel engine without the engine going through major modification. But the engine may need if it has old fuel lines [Randall von Wedel, 1999].

2.2 Bio-petrol from rubber seed oil

The rubber tree originally belongs to Amazon basin (Africa) and later in the 19th century introduced to other countries in the tropical belts of Asia. The Para rubber tree (*Hevea brasiliensis*), belongs to the family Euphrobiaceae and the most economical member of the genus *Hevea*. However, the rubber tree also produces large volumes of seed, which is underutilized. On an average 160 kg of rubber seed is produced per hectare of rubber plantation. The estimated availability of rubber seeds in India is about 30,000 tons per annum, which can yield oil to the tune of about 5000 tons per annum [D.F. Melvin Jose**,** R. Edwin Raj 2010].

The rubber seed oil varies in color from light yellow to brown, depending on the Free Fatty Acids (FFA) content, yellow being on the lower side [D.F. Melvin Jose**,** R. Edwin Raj**,** B. Durga Prasad **,** Z. Robert Kennedy and A. Mohammed Ibrahim 2010**].** The rubber plant which is widely used as a natural source of rubber have been reported tohave oil rich seeds (Njoku et al., 1996). Although there are variations in the oil content of the seed from different countries, the average oil yield have been reported to be 40% (Hilditch and Njoku).The Table 2.2 below shows the properties of rubber seed oil. .

Table 2.2: Physico-chemical properties of rubber seed oil **[**Njoku, O.U., Ononogbu, I.C., Owusu, J.Y., 1996. An investigationof oil of rubber (*He*6*ea bransiliensis*). J. of Rubber Res. Inst. Sri-Lanka 78, 52–59.]

Colour	**Brown**
Specific gravity (at 30 °C)	0.93
Refractive index	1.477
Acid value (mg KOH/g)	19.18
Free fatty acid (% as oleic acid)	9.54
Saponification (mg KOH/g)	181.14
Iodine value (Wijs) (g I_2/100 g)	136.2

Other than production of bio-fuel the rubber seed oil is also has been used for various indurtrial application such as paint, water-reducible alkyds **[**Ikhuoria et al,2005], zinc soaps [Theresa Obuajulu Egbuchunam et al,2007], binders in water-borne coatings [A. I. Aigbodion et al 2002] and in the production of sisal fiber-reinforced polyurethane composites [F.E. Okieimen 2010]. The rubber seed oil possesses many fatty acids. The major saturated fatty acids are palmitic (10.2%) and stearic (8.7%) while the main unsaturated fatty acids are oleic (24.6%), linoleic (39.6%) and linolenic (16.3%).

Fatty acids are the carboxylic acids with long hydrocarbon chains. There are two groups of fatty acids which are saturated and unsaturated. Unsaturated fatty acids resemble saturated fatty acids, except that the chain has one or more double between carbon atoms. The two carbon atoms in the chain that are bound next to either side of the double bond can

occur in a *cis* or *trans* configuration. Saturated fatty acids are long-chain carboxylic acids that usually have between 12 and 24 carbon atoms and have no double bonds. Thus, saturated fatty acids are saturated with hydrogen (since double bonds reduce the number of hydrogens on each carbon). Because saturated fatty acids have only single bonds, each carbon atom within the chain has 2 hydrogen atoms (except for the omega carbon at the end that has 3 hydrogens). The table 2.3 below shows the list of fatty acids that can be found in rubber seed oil.

Table 2.3: Major fatty acid composition of Rubber Seed Oil **[**O.U. Njoku I.C., Ononogbu, O.E. Ikwuagwu , 1999 Production of biodiesel using rubber [Hevea brasiliensis (Kunth. Muell.)] seed oil.]

Fatty acid	Composition (%)
$C_{16:0}$ palmitic acid	10.2
$C_{18:0}$ stearic acid	8.7
Total unsaturated	18.9
$C_{18:1}$ oleic acid	24.6
$C_{18:2}$ linolenic acid	39.6
$C_{18:3}$ linolenic acid	16.3
Total	80.5
Others	0.6

The production of bio-petrol from fatty acids which can be found from rubber seed oil has proven to us that it can provide energy security in the form of supply reliability, minimizing the dependence over fossil fuel, vast availability, renewability and domestic distribution. Furthermore bio-fuel shows its superiority over fossil fuel by displaying characteristics which is shown below.

1) It is biologically degradable and a non-toxic material so it is safe to say that it is a environmental friendly material.
2) It increases the performance, efficiency and life of an engine due to the fact that it contains high amount of isooctane which prevents knocking.

3) The production of bio-petrol from rubber seed oil will not result any food shortage based on the fact that rubber seed are not edible materials.
4) Bio-petrol can make the operation of the engine more smoother and could reduce the discharge of the unburned fuel
5) Any accidental leakage of bio-petrol will not bring any serious effects compared to the leakage of fossil fuel.
6) The molecular structure has no sulfur in it and bio-petrol is produced from biomass thus it will bring about the reduce of greenhouse gas emission.

2.3 Comparison Between Bio-Fuel and Fossil Fuel

Table 2.4 Comparison between Bio-Fuel and Fossil Fuel

ASPECT	BIO-FUEL	FOSSIL FUEL
Greenhouse gas emission and environmental issue	➢ Sustainable and biodegradable ➢ Reduces greenhouse gas emission ➢ Environmental friendly	➢ Not biologically degradable ➢ Contains a large amount of sulfur ad oxygen which causes incomplete combustion which emits greenhouse gases. ➢ Pollutes the environment
Energy security	➢ Renewable energy source which can be produced from biomass	➢ Non-renewable energy source and its it is depleting at a faster rate
Engine performance	➢ Increases engine life ➢ Improves performance and efficiency in the combustion process	➢ Incomplete combustion in the engine
Economy	➢ Creates new job opportunities	➢ Extraction of fossil fuel has high production cost ➢ The reduce in fossil fuel sources

	➢ Increases the agricultural demand for biomass to produce bio-fuel ➢ Improves the rural region	causes the fossil fuel price to raise.

2.4 Catalytic Cracking

The main component in the production of bio-petrol using rubber seed oil is the fatty acids which are found in the rubber seed oil. According to the elaboration earlier fatty acid are carboxylic acids with long hydrocarbon chains. Fatty acids cannot react with other substances in its natural form so for it to undergo any reaction it has to be broken in the form of simple chain molecules.

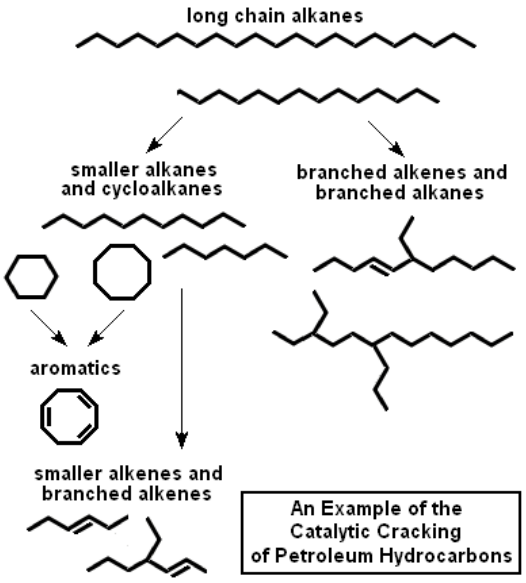

Figure 2.2 Catalytic Cracking Process

For this case the application of heterogeneous catalytic cracking is necessary. The Figure 2.4 above shows the brief description of the catalytic cracking process. The catalytic cracking process involves the presence of acid catalysts (usually solid acids such as silica-alumina and zeolites) which promote a heterolytic (asymmetric) breakage of bonds yielding

pairs of ions of opposite charges, usually a carbocation and the very unstable hydride anion. Carbon-localized free radicals and cations are both highly unstable and undergo processes of chain rearrangement, C-C scission in position beta as in cracking, and intra- andintermolecular hydrogen transfer or hydride transfer. In both types of processes, the corresponding reactive intermediates (radicals, ions) are permanently regenerated, and thus they proceed by a self-propagating chain mechanism. The chain of reactions is eventually terminated by radical or ion recombination [James H. Gary and Glenn E. Handwerk 2001]. Catalytic cracking has been one of the key processes in petroleum refining in the last decades. The fluid catalytic cracking (FCC) process is an important process in refiners for upgrading heavy hydrocarbons to more valuable lighter products mainly gasoline; hence small enhancements in their operation are economically attractive. In order to achieve this objective, it is necessary to implement advanced process control techniques. In a refinery that produces a substantial amount of gasoline, FCC gasoline makes up about 40% of the overall refinery gasoline pool [G. Pandimadevi et al 2009].

The catalytic cracking of hydrocarbons is a chain reaction that is believed to follow the carbonium ion theory developed by Whitmore. This chain mechanism involves three elementary steps: initiation, propagation and termination. The initiation step is represented by the attack of an active site on the reactant molecule to produce the activated complex that, in the gas phase or when using liquid superacids, would correspond to the formation of a carbocation. The chain propagation is represented by the transfer of a hydride ion from a reactant molecule to an adsorbed carbonium ion. Finally, the termination step corresponds to desorption of the adsorbed carbonium ion to give an olefin whilst restoring the initial active site [A. Corma et al 1999].

2.5 Pyrolysis of Mechanisms of Vegetable Oils

Conversion of one substance into another by the means of heat or by heat with the aid of a catalyst is called Pyrolysis (Sonntag, 1979). It involves heating when no air present or oxygen and cleavage of chemical bonds to yield small molecules. Materials that can pyrolyzed are vegetable oil, animal fats, natural fatty acids, and methyl esters of fatty acids.

Pyrolysis liquid products of vegetable oil can be used as alternative engine fuel. Vegetable oils may be converted to liquid product containing gasoline boiling range hydrocarbons. The product compositions are affected by catalyst content and temperature. In pyrolysis, the high molecular materials are heated to high temperatures, so their macromolecular structures are broken down into small molecules and a wide range of hydrocarbons are formed.

The distributions of pyrolysis product depend on the dynamics and kinetic control of different reactions. Thermodynamic calculation shows that the initial decomposition of vegetables oils occurs with the breaking of the C-O bond at lower temperature, and fatty acids are the main products. The pyrolysis temperature should be higher than 675K; at this temperature, the maximum of diesel yield with high content of oxygen can be obtained(Zhenyi et al., 2004). The effect of temperature, the use of catalysts, and the characterization of the products have been investigated (Srivastava and Prasad, 2000). In pyrolysis, the high molecular materials are heated tohigh temperatures, so their macromolecular structures are broken down into smaller molecules, a wide range of hydrocarbons are formed. These pyrolytic products can be divided into a gas fraction, liquid fraction consisting of paraffins, olefins and naphthenes, and solid residue. The cracking process yield a highly unstable low-grade fuel oil, which can be acid-corrosive, tarry, and discolored along with a characteristically foul odor(Demirbas, 2004b).

2.6 Zeolite as a Heterogeneous Catalyst

Catalyst is defined as materials which can accelerate the rate of reaction of a chemical reaction without it undergo any substantial permanent chemical changes. A catalyst plays its part in multiple chemical transformations. Catalysts that increase the reaction rate are called positive catalysts. The catalyst that slows down a catalyst’s reaction rate is called inhibitor or in other words negative catalyst. Substances that increase the activity of catalysts are called promoters, and substances that deactivate catalysts are called catalytic poisons. There are many types of catalysts such as heterogeneous catalyst,

homogenous catalyst, electro catalysts and organocatalysis. But the two most famous of those types are heterogeneous and homogenous catalyst. Heterogeneous catalyst is a catalyst in a different phase from the reactants. Typical examples involve a solid catalyst with the reactants as either liquids or gases. The homogenous catalyst is the catalyst in the same phase as the reactants. The Table 2.5 shows the differences of both of the catalyst.

Table 2.5 Comparison of Homogeneous and Heterogeneous Catalyst

Factors	Homogeneous catalysis	Heterogeneous catalysis
1. Reaction Rate	Fast and high conversion	Moderate conversion
2. After treatment	Catalyst cannot be recovered, must be neutralized leading to waste chemical production	Can be recovered
3. Processing methodology	Limited use of continuous methodology	Continuous fix bed operation possible
4. Presence of water/free fatty acids	Sensitive	Not sensitive
5. Catalyst reuse	Not possible	Possible
6. Cost	Comparatively costly	Potentially cheaper

Zeolites or in other name molecular sieve are microporous crystalline solids with well-defined structures. Zeolites composition in its framework majorly contains aluminum, silicon and oxygen while water, cations and other molecules can be found within its pores. Many zeolites occur naturally as minerals and being mined all around the world. There are also some zeolites which are chemically produced for specific uses. Analcime, chabazite, clinoptilolite,heulandite, natrolite, phillipsite,and stilbite are some of the common zeolite minerals . An example mineral formula is: $Na_2Al_2Si_3O_{10}•2H_2O$, the formula for natrolite Conventional open pit mining techniques are used to mine natural zeolites. Currently, the world's annual production of natural zeolite is about 4 million tons.

Zeolites have the ability to act as catalysts for chemical reactions which take place within the internal cavities. An important class of reactions is that catalysed by hydrogen-exchanged zeolites, whose framework-bound protons give rise to very high acidity. This is exploited in many organic reactions, including crude oil cracking, isomerisation and fuel synthesis. Zeolites can also serve as oxidation or reduction catalysts, often after metals have been introduced into the framework. Examples are the use of titanium ZSM-5 in the production of caprolactam, and copper zeolites in NOx decomposition [*R.G. Bell et al., 2001]*

Zeolites are very environmentally friendly substances. 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.Zeolites have replaced phosphate builders which have been banned in many parts across the globe because it causes water pollution in the production of powder detergents. By acting as catalysts zeolites have made chemical processes to be more efficient and by doing this we are able to save energy and reduce pollution at the same time.

Furthermore by lessening the steps in a reaction amount of waste and by-products have been reduced. 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 exahust 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 [*R.G. Bell et al., 2001]*.

2.7 Soxhlet Extraction

Sample pretreatment is often one of the most time consuming steps of the analytical process, particularly when solid samples are involved. The search for modification of the present devices, the design of new devices and the use of auxiliary sources of energy which

shorten and/or enable automation of sample pretreatment have been the aim of analytical chemists in the last decades.

Solvent extraction of solid samples, which is commonly known as solid–liquid extraction, but which should be referred to, in a more correct use of the physicochemical terminology, as leaching or lixiviation, is one of the oldest ways of solid sample pretreatment. Among the techniques used for implementation of this step, Soxhlet has been the leaching technique mostly used for a long time. This assertion is supported by the fact that Soxhlet has been a standard technique during more than one century and, at present; it is the main reference to which the performance of other leaching methods is compared. In conventional Soxhlet, originally used for the determination of fat in milk , the sample is placed in a thimble-holder, and during operation gradually filled with condensated fresh solvent from a distillation flask. When the liquid reaches the overflow level, a siphon aspirates the solute of the thimble-holder and unloads it back into the distillation flask, carrying the extracted analytes into the bulk liquid. This operation is repeated until complete extraction is achieved. This performance makes Soxhlet a hybrid continuous–discontinuous technique. Inasmuch as the solvent acts stepwise, the assembly can be considered as a batch system; however, since the solvent is recirculated through the sample, the system also bears a continuous character.

The most outstanding advantages of conventional Soxhlet are as follows: the sample is repeatedly brought into contact with the fresh portions of the solvent, thereby helping to displace the transfer equilibrium. The temperature of the system remains relatively high since the heat applied to the distillation flask reaches the extraction cavity to some extent. No filtration is required after the leaching step. Sample throughput can be increased by simultaneous extraction in parallel, since the basic equipment is inexpensive. It is a very simple methodology which needs little specialized training, has the possibility to extract more sample mass than most of the latest methods and is non-matrix dependent.

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.

Normally a solid material containing some of the desired compound is placed inside a thimble made from thickfilter 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.

Figure 2.3: Soxhlet Extractor

The chamber containing the solid material slowly fills with warm solvent. Some of the desired compound will thendissolve in the warm solvent. When the Soxhlet chamber is almost full, the chamber is automatically emptied by asiphon 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.

2.8 Evaporation

Evaporation is a type of vaporization of a liquid that occurs only on the surface of a liquid. The other type of vaporization is boiling, which, instead, occurs on the entire mass of the liquid. Evaporation is also part of the cycle. On average, the molecules in a glass of water do not have enough heat energy to escape from the liquid. With sufficient heat, the liquid would turn into vapor quickly. When the molecules collide, they transfer energy to each other in varying degrees, based on how they collide. Sometimes the transfer is so one-sided for a molecule near the surface that it ends up with enough energy to escape. Liquids that do not evaporate visibly at a given temperature in a given gas have molecules that do not tend to transfer energy to each other in a pattern sufficient to frequently give a molecule the heat energy necessary to turn into vapor. However, these liquids are evaporating. It is just that the process is much slower and thus significantly less visible. Evaporation is an essential part of the water cycle. Solar energy drives evaporation of water from oceans, lakes, moisture in the soil, and other sources of water. In hydrology, evaporation and transpiration are collectively termed evapotranspiration. Evaporation is caused when water is exposed to air and the liquid molecules turn into water vapor, which rises up and forms clouds.

For molecules of a liquid to evaporate, they must be located near the surface, be moving in the proper direction, and have sufficient kinetic energy to overcome liquid-phase intermolecular forces.[1] Only a small proportion of the molecules meet these criteria, so the rate of evaporation is limited. Since the kinetic energy of a molecule is proportional to its temperature, evaporation proceeds more quickly at higher temperatures. As the faster-moving molecules escape, the remaining molecules have lower average kinetic energy, and the temperature of the liquid, thus, decreases. This phenomenon is also called evaporative cooling. This is why evaporating sweat cools the human body. Evaporation also tends to proceed more quickly with higher flow rates between the gaseous and liquid phase and in liquids with higher vapor pressure. For example, laundry on a clothes line will dry (by evaporation) more rapidly on a windy day than on a still day. Three key parts to evaporation are heat, humidity, and air movement. On a molecular level, there is no strict boundary between the liquid state and the vapor state. Instead, there is a Knudsen layer, where the phase is undetermined. Because this layer is only a few molecules thick, at a macroscopic scale a clear phase transition interface can be seen.

A rotary evaporator (or rotavap) is a device used for efficient and gentle removal of solvents from samples by evaporation [Laurence M. Harwood, Christopher J. Moody]. Rotary evaporators are also used in molecular cooking for the preparation of distillates and extracts. 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 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. Modern equipment often adds features such as digital control of vacuum, digital display of temperature and rotational speed, and vapor temperature sensing.

The main parts of a rotary evaporator are:

- A motor unit that rotates the evaporation flask or vial containing the user's sample.
- 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.
- A vacuum system, to substantially reduce the pressure within the evaporator system.

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

Vacuum evaporators as a class function because lowering the pressure above a bulk liquid lowers the boiling points of the component liquids in it. Solvents with higher boiling points such as water (100 °C at standard atmospheric pressure, 760 torr), dimethylformamide (DMF, 153 °C at the same), ordimethyl 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) [Laurence M. Harwood, Christopher J. Moody].

One of the key advantage in using a rotary evaporator is 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. Another advantage that can be gained by using rotary evaporator is that 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. The Figure 2.6 shows one of the latest model of rotary evaporator.

Figure 2.4: Rotary Evaporator

Even though it is a simple operation such as evaporation there are still many hazards associated with it. These hazards include implosions which are resulted from the use of glassware which contains many flaws, such as cracks. There is a possibility for explosions to occur from concentrating unstable impurities during evaporation, as an example when rotavapping an ethereal solution which contains peroxides. There is many other situations where an explosion can take place for an example this can also occur when taking certain unstable compounds, such as organic azides and acetylides, nitro-containing compounds, molecules with strain energy, etc. to dryness. Switching the rotation off during evaporation may also result in an 'explosion' through bumping.

When using the rotary evaporation equipment the user must take precautions to avoid contact with rotating parts, particularly entanglement of loose clothing, hair, or necklaces. Under these circumstances, the winding action of the rotating parts can draw the users into the apparatus resulting in breakage of glassware, burns, and chemical exposure. Extra caution must also be applied to operations with air reactive materials, especially when under vacuum. A leak can draw air into the apparatus and a violent reaction can occur.

Lastly, rotary evaporation equipment is notoriously expensive and easy to break. To minimize risks, laboratories generally restrict the operation of this type of equipment to specifically trained personnel.

2.9 Gas Chromatography

Gas chromatography is used in the separation and analysis of multi component mixtures such as essential oils, hydrocarbons and solvents. Gas chromatography is also known with other names which are vapor-phase chromatography (VPC), or gas-liquid partition chromatography (GLPC). The primary application of GC includes separating the different components of a mixture, testing the purity of a substance or determining the relative amounts of components. In some other situation gas chromatography also helps in the identification process of a compound.in the preparation of pure compounds from a mixture Inpreparative chromatography can be used.

In gas chromatography ther are two phases which are moving phase and stationary phase. In the moving phase or "mobile phase" there will be the presence of a carrier gas, usually an inert gas such as helium or an unreactive gas such as nitrogen. The stationary phase is consist of a microscopic layer of liquid or polymeron an inert solid support, inside a piece of glass or metal tubing called a column which is a homage to the fractionating column used in distillation. The instrument used to perform gas chromatography is called a gas chromatograph.

Gas chromatograph uses a narrow tube which is known as the column to flow-through, in which various chemical elements of a sample pass in a gas stream at different rates based on their various chemical and physical properties and their interaction with a specific column filling, called the stationary phase. Electrical identification will be used to identify the chemicals at the end of the column. Separating different components and causing each one to exit the column at a different time (retention time) is the function of the stationary phase. Other parameters which can be used to change the order or time of retention are the carrier gas flow rate, and the temperature. Figure 2.7 shows one of the most widely used model of gas chromatographer.

Figure 2.5 Gas Chromatographer

There are many detectors which are being used gas chromatography. Figure 2.8 and 2.9 shows Flame Ionization Detector and Thermal Conductivity Detector which are widely available in the market. The selectivity of each detector will differ from one another. The most common detectors which are widely used are the flame ionization detector (FID) and the thermal conductivity detector (TCD). Organic compounds are most effectively detected done with flame ionization. Biochemical compounds can be studied with flame ionization due to the presence of nitrogen, phosphorus, or sulfur atoms. Another reason for this is that biochemical compounds contain high amount of carbon than any other compounds. That means that a biochemical compounds may be more easily detected using flame ionization over the other methods due to the fact that it contains a high amount of carbon and also flame ionization's sensitivity. The thermal conductivity detector (TCD) is a bulk property detector. This detector senses the difference in the thermal conductivity of the column effluent and compares it to a reference flow of carrier gas. Since most compounds have a thermal conductivity much less than that of the common carrier gases of helium or hydrogen, when an analyte elutes from the column, the effluent thermal conductivity is reduced and produces a detectable signal. But both detectors do have some similarities between them. Both are sensitive to a wide range of components, and both work over a wide range of concentrations.

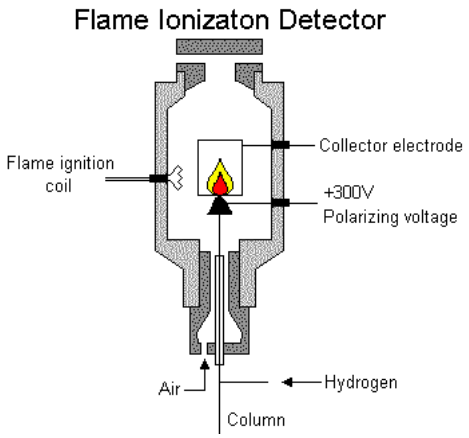

Figure 2.6 Flame Ionization Detector

CHAPTER 3

METHODOLOGY

3.1 Apparatus and Equipments

In this research the apparatus that will be used during experiment progress are Soxhlet Extractor, Rotary Evaporator, Heating Mantle, Gas Chromatography, Flask 250ml, Beaker 100ml, Filter Funnel, Thermometer 110^0C, Vials, 0.2µm syringe filter and Syringe 5ml and Thimble Filter.

3.2 Chemical Substances

The chemical substances that used in this research are rubber seed as the raw material, hexane as the dilution agent for samples and using for the soxhlet extraction process as the dilution agent and as solvent for standard and isooctane as the substance for analysis. The chemicals that will be used during experiment progress are 500 gram of rubber seeds, boiling chips, standard hexane (99% purity), isooctane (100% purity) and standard ethanol (99% purity)

3.3 Experiment Procedures

3.3.1 Extraction of Rubber Seed Oil Using Soxhlet Extractor

90 grams of rubber seeds are cleaned and shelled to extract out the kernels. The seed kernels blended by using a blender. The thimble filter is filled until two third portion with the blended seeds and then inserted into the siphon exit of the soxhlet extractor. The receiving flask is filled with about 180 grams of the ethanol solvent.

Then heater is switched on and operated until the temperature of the solvent reaches about 75 ^{0}C or until the solvent boils. The experiment is left running for approximately six hours. The heater then is switched off and the mixture solution of ethanol and oil is left to cool down. Repeat the procedure with mass ratio rubber seed to solvent of 1:3 and 1:4.The mixture is then transferred into a rotary evaporator.

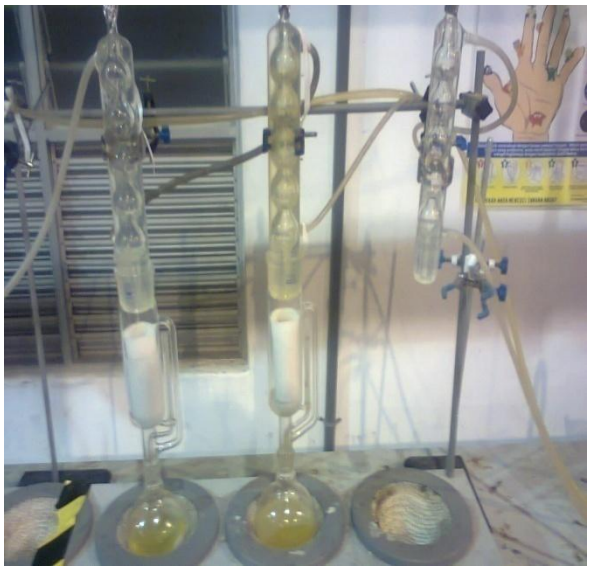

Figure 3.1: Soxhlet Extractor

3.3.2 Evaporation of Solvent by Rotary Evaporator

Inside the rotary evaporator, the mixture of rubber seed oil and solvent transferred from soxhlet extractor is heated to evaporate the solvent at 85 ^{0}C (slightly higher than the boiling point of the ethanol). The liquid remaining in the flask is the pure rubber seed oil,

and the evaporated ethanol is then condensed and gathered inside receiving vessel attached at the evaporator.

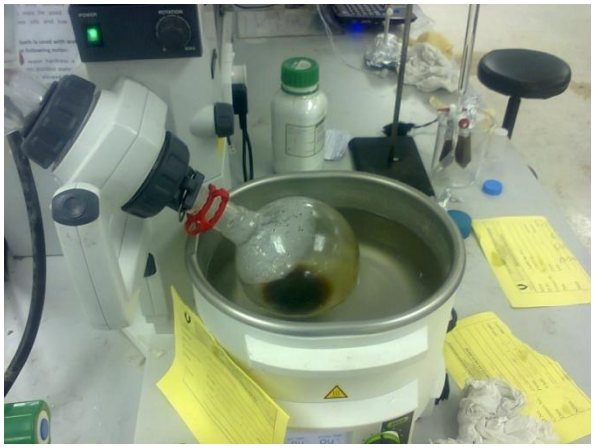

Figure 3.2: Rotary Evaporator

3.3.3 Heterogeneous Catalytic Cracking Using Zeolite as Catalyst

The rubber seed oil is transferred into a flask. 5 grams of zeolite catalyst is added into the flask containing 25 mL of rubber seed oil. The mixture of rubber seed oil and zeolite are placed on a magnetic stirrer. The mixture is then heated using the furnace until 300 ^{0}C and maintained it for forty five minutes. 5 grams anti bumping granules are added into the mixture to ensure the heat energy transfers uniformly. Then, the samples are filtered to remove the solid catalyst to obtain the distilled product oil.

3.3.4 Preparation of Standards Using Pure Isooctane

Isooctane and hexane are prepared according to the portions as shown in Table 3.1. The mixtures of the standards are injected into each vial through a 0.2μm syringe filter of about 1.5 ml.

Table 3.1: Composition of the Isooctane-Hexane mixture

Vials	Composition	
	Isooctane	Hexane
1	0%	100%

2	80%	20%
3	60%	40%
4	40%	60%
5	20%	80%

3.3.5 Gas Chromatography Analysis for Samples

The cracked oil for each mass of catalyst used will be diluted first with hexane as dilution agent to give four different mixture solutions – 1%, 5%, 10%, 15% and 20% of cracked oil samples. These samples are injected into the 1.5mL vials using the 0.2µm syringe filter of about 1.5mL. Vials must be labeled and must be arranged in sequence on the auto-injectors VS auto samplers at the gas chromatographer's vial rack.

Samples obtained (for every diluted mixture solutions) are analyzed using Gas Chromatographer. The conditions of the Gas Chromatographer are set as in Table 3.2. When the chromatograms (peak area versus time) of the standards are obtained, a calibration curve (peak area versus concentration) is plotted using data of standards' chromatograms.

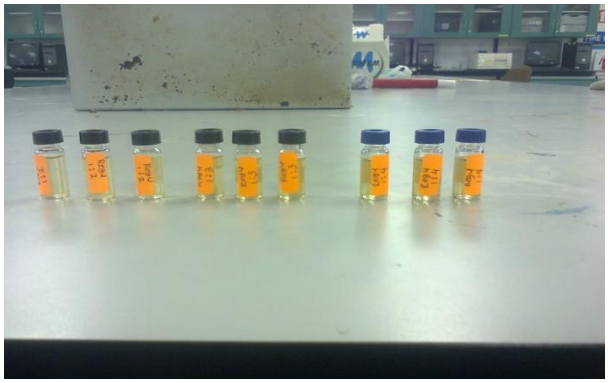

Figure 3.3: Vials containing samples

3.3.6 Gas Chromatogram Standard Analysis

The vials which are labeled must be arranged in sequence on the auto-injectors VS auto samplers at the gas chromatography's vial rack. Then the standards are analyzed using

the gas chromatography method. The conditions of the Gas Chromatograph are set as in Table 3.2. After the chromatogram (peak area versus time) of each standard are then obtained a calibration curve (peak area versus concentration) is plotted.

Table 3.2: Gas Chromatographer (GC) condition

Temperature Column	Initial 50^{o}C, hold 3 minutes, program at 8^{o}C/min to 120^{o}C, hold 5 minutes
Detector Type	Flame Ionization Detector (FID)
Injector Temperature	250^{o}C
Detector Temperature	250^{o}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_2, N_2
Carrier Gas Pressure	5 bar for every carrier gas

CHAPTER 4

RESULTS AND DISCUSSION

4.1 OBSERVATION

Figure 4.1: Mixture of 0.025 liter of rubber seed oil + 5gram of Zeolite + 5gram of boiling chips After Catalytic Cracking

From the Figure 4.1 above, the mixture has changed its color from light brown (before cracking) into dark brown after cracking. The oil can be hardly seen as the mixture became very thick due to the swelling of Zeolite to several times of its original volume. Even though the mixture is filtered, the oil has a very dark colour to it.

Figure 4.2: Filtration of mixture

4.2 ANALYSIS OF STANDARD ISOOCTANE CALIBRATION CURVE

For the analysis of Gas Chromatography Flame Ionization Detector method, the mixtures of different ratio of solvent and the favorable component of the chemical to be detected must be prepared as a standard. These standards are used to determine the presence of the specified chemical compound and its concentration identified through the chromatogram graph of peak area versus retention time. There are six standards prepared according to the ratio of 0% isooctane, 20% isooctane, 40% isooctane, 60% isooctane and 80% isooctane. Below are the Chromatogram Results for the Isooctane Standards:

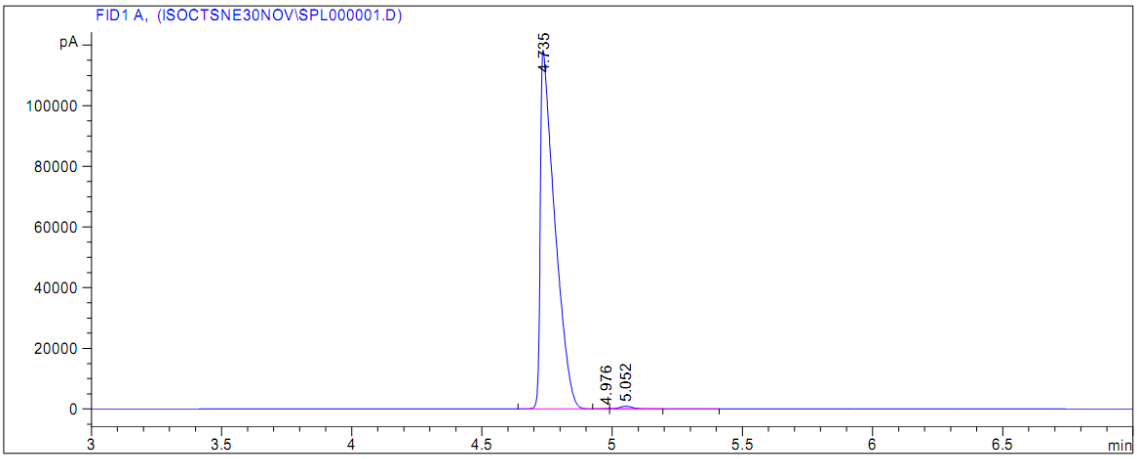

Figure 4.3: Chromatogram of 0% Standard Isooctane

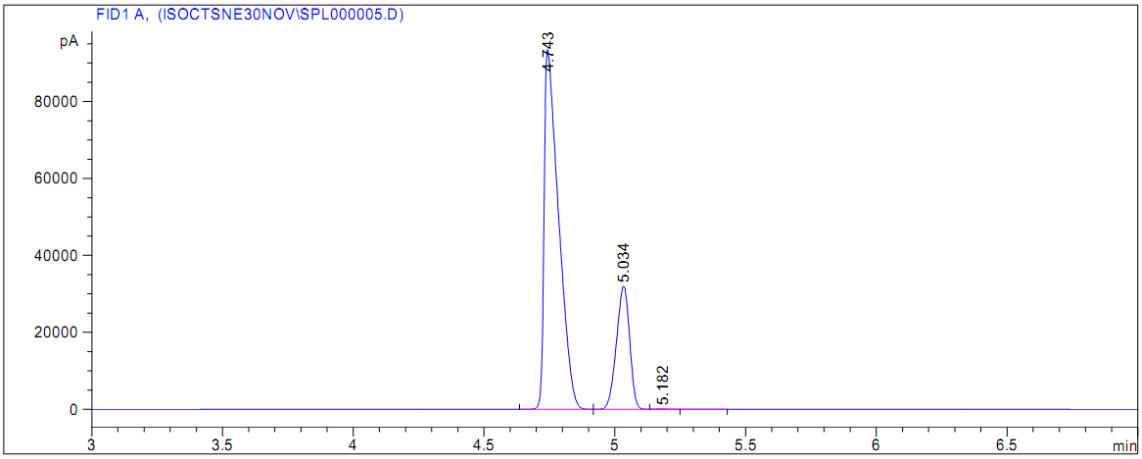

Figure 4.4: Chromatogram of 20% Standard Isooctane

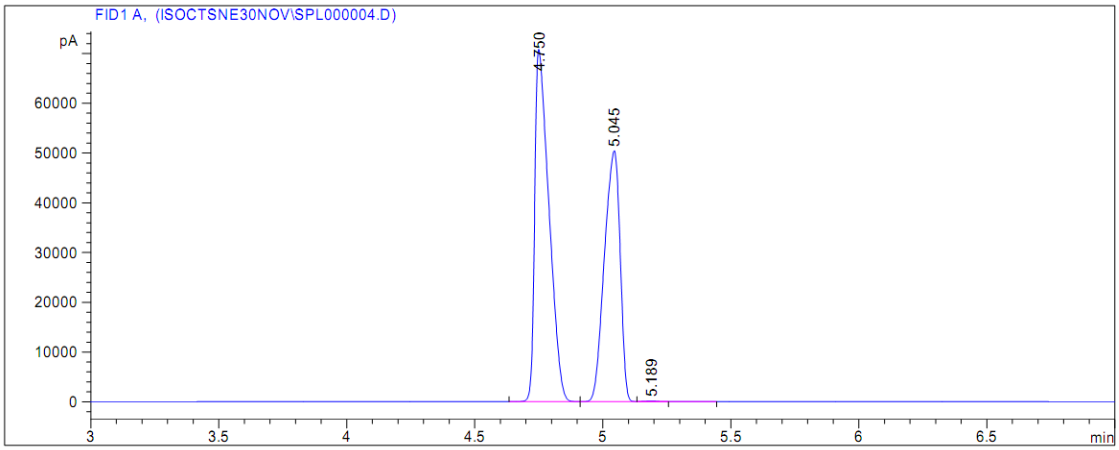

Figure 4.5: Chromatogram of 40% Standard Isooctane

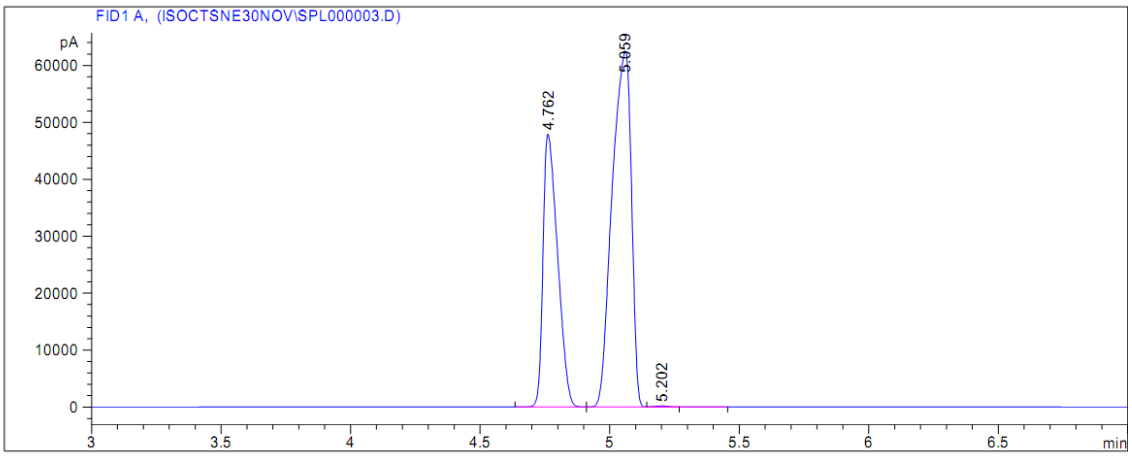

Figure 4.6: Chromatogram of 60% Standard Isooctane

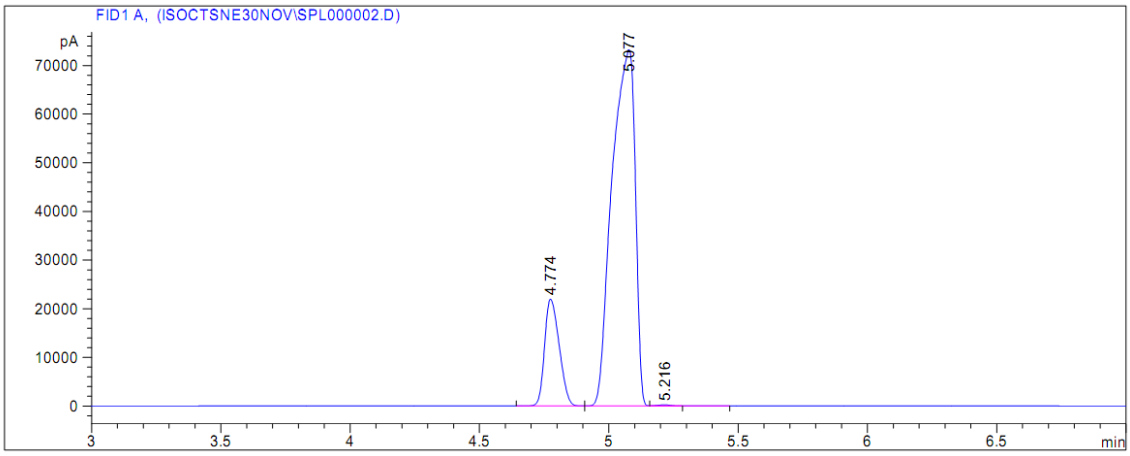

Figure 4.7: Chromatogram of 80% Standard Isooctane

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

Table 4.1: Chromatogram Analysis for Standard Isooctane

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

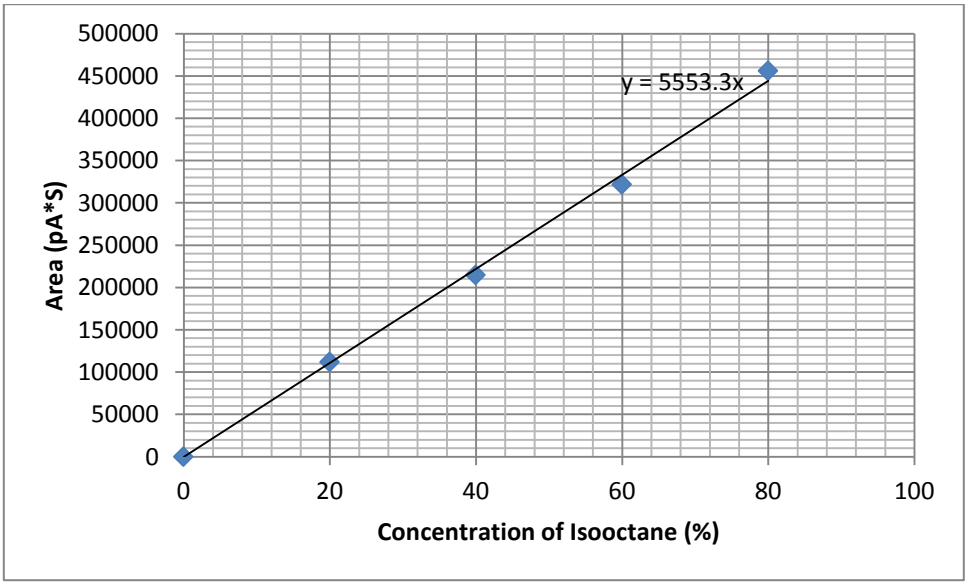

Figure 4.8: Standard Calibration Curve

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:

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

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

4.3 ANALYSIS OF THE ACTUAL CONCENTRATION ISOOCTANE IN SAMPLES

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 Ratio 1:2

Dilution Samples (%)	Isooctane			Hexane		
	Retention Time	**Peak Area (pA*s)**	**Area (%)**	**Retention Time**	**Peak Area (pA*s)**	**Area (%)**
1	5.056	2482.07	0.69183	4.747	3.56232E+05	99.38378
5	5.063	2106.47	0.61622	4.753	3.39732E+05	99.39440
10	5.062	1914.94	0.60560	4.756	3.14293E+05	99.40076
15	5.062	2044.73	0.59924	4.754	3.39177E+05	99.40076
20	5.063	1784.68	0.57828	4.758	3.06836E+05	99.42172

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	5.055	2573.10718	0.70816	4.746	3.60706E+05	99.27282
5	5.064	2121.70825	0.61056	4.753	3.45380E+05	99.38944
10	5.065	1963.85925	0.60848	4.757	3.20769E+05	99.38742
15	5.063	2086.11239	0.58766	4.753	3.52874E+05	99.40551
20	5.065	1757.95520	0.57379	4.760	3.04620E+05	99.42621

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	5.056	3232.73700	0.74911	4.738	4.28310E+05	99.25089
5	5.066	2090.52319	0.60282	4.756	3.44701E+05	99.39718
10	5.064	1981.656662	0.59637	4.756	3.30290E+05	99.3985
15	5.065	2181.23828	0.5890	4.752	3.68097E+05	99.4004
20	5.065	1858.36646	0.57518	4.758	3.21234E+05	99.42482

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 (%)

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

- Actual Peak Area of Isooctane (pA*s)

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

- Actual Concentration of Isooctane (%)
    - Based on equation 1 where the;

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

      x = $Actual\ Concentration\ of\ Isooctane\ (\%)$

$$Actual\ Concentration\ of\ Isooctane\ (\%) = \frac{[Actual\ Peak\ Area\ of\ Isooctane\ (pA * s)]}{5553}$$

Example of Calculation

1. Calculation for Sample and Solvent (Hexane)

a) 1% of Sample x 10 mL = 0.1 mL

99% of Hexane x 10 mL = 9.9 mL

b) 5% of Sample x 10 mL = 0.5 mL

95% of Hexane x 10 mL = 9.5 mL

c) 10% of Sample x 10 mL = 1.0 mL

90% of Hexane x 10 mL = 9.0 mL

d) 20% of Sample x 10 mL = 2.0 mL

80% of Hexane x 10 mL = 8.0 mL

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

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

= [0.69183/ (100-99.38378)]* 100

= **112.2699%**

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

peak area of isooctane (%)

= [2482.07/0.69183]* 112.2699%

= 4.027x10^5**pA*s**

From the equation shown in standard calibration curve (Figure 4.9):

Y=5553X

Where,

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

x = Actual concentration of isooctane (%)

Actual concentration of Isooctane (%) = (actual peak area (pA*s)/5553

= ((4.027x10^5)/5553)*100

= 73%

Table 4.6: Experimental Matrix and Results of Each Sample for Rubber Seed Mass to Solvent Mass Ratio 1:2

Dilution (%)	Area Isooctane (pA*s)	Isooctane Area (%)	Hexane Area (%)	Actual Peak Area Isooctane (%)	Actual Peak Area (pA*s)	Actual Isooctane Concentration (%)
1	2482.07	0.69183	99.38378	112.2699	4.027E+5	73
5	2106.47	0.61622	99.39440	101.7536	3.478E+5	63
10	1914.94	0.60560	99.39440	100.0000	3.162E+5	57
15	2044.73	0.59924	99.40076	100.0000	3.412E+5	61
20	1784.68	0.57828	99.42172	100.0000	3.086E+5	56

Table 4.7: Experimental Matrix and Results of Each Sample for Rubber Seed Mass to Solvent Mass Ratio 1:3

Dilution (%)	Area Isooctane (pA*s)	Isooctane Area (%)	Hexane Area (%)	Actual Peak Area Isooctane (%)	Actual Peak Area (pA*s)	Actual Isooctane Concentration (%)
1	2573.10718	0.70816	99.27282	97.3840	3.538E+5	64
5	2121.70825	0.61056	99.38944	100.0000	3.475E+5	63
10	1963.85925	0.60848	99.38742	99.3306	3.205E+5	58
15	2086.11239	0.58766	99.40551	98.8511	3.509E+5	63
20	1757.95520	0.57379	99.42621	100.0000	3.063E+5	55

Table 4.8: Experimental Matrix and Results of Each Sample for Rubber Seed Mass to Solvent Mass Ratio 1:4

Dilution (%)	Area Isooctane (pA*s)	Isooctane Area (%)	Hexane Area (%)	Actual Peak Area Isooctane (%)	Actual Peak Area (pA*s)	Actual Isooctane Concentration (%)
1	3232.73700	0.74911	99.25089	100.0000	4.315E+5	78
5	2090.52319	0.60282	99.39718	100.0000	3.467E+5	62
10	1981.65662	0.59637	99.3985	99.1471	3.294E+5	59
15	2181.23828	0.5890	99.4004	98.2321	3.637E+5	66
20	1858.36646	0.57518	99.42482	100.0000	3.230E+5	58

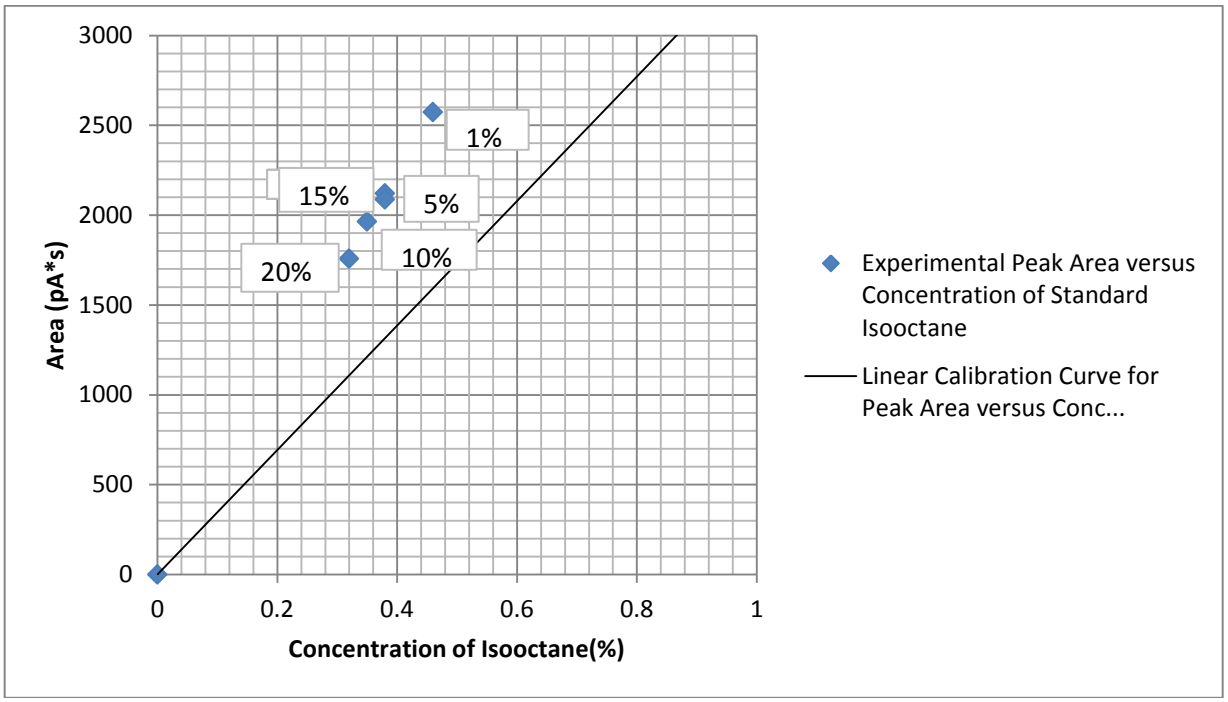

Figure 4.9: Concentration of Experimental Isooctane Present In Samples for Rubber Seed Mass to Solvent Mass Ratio 1:2

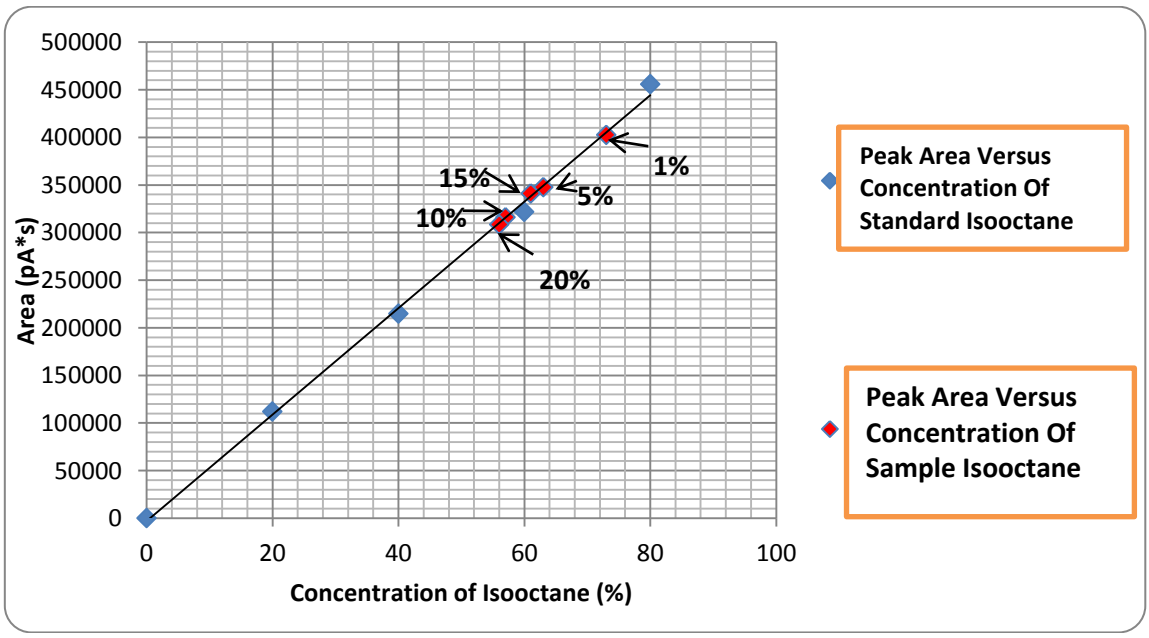

Figure 4.10: Concentration of Actual Isooctane Present In Samples for Rubber Seed Mass to Solvent Mass Ratio 1:2

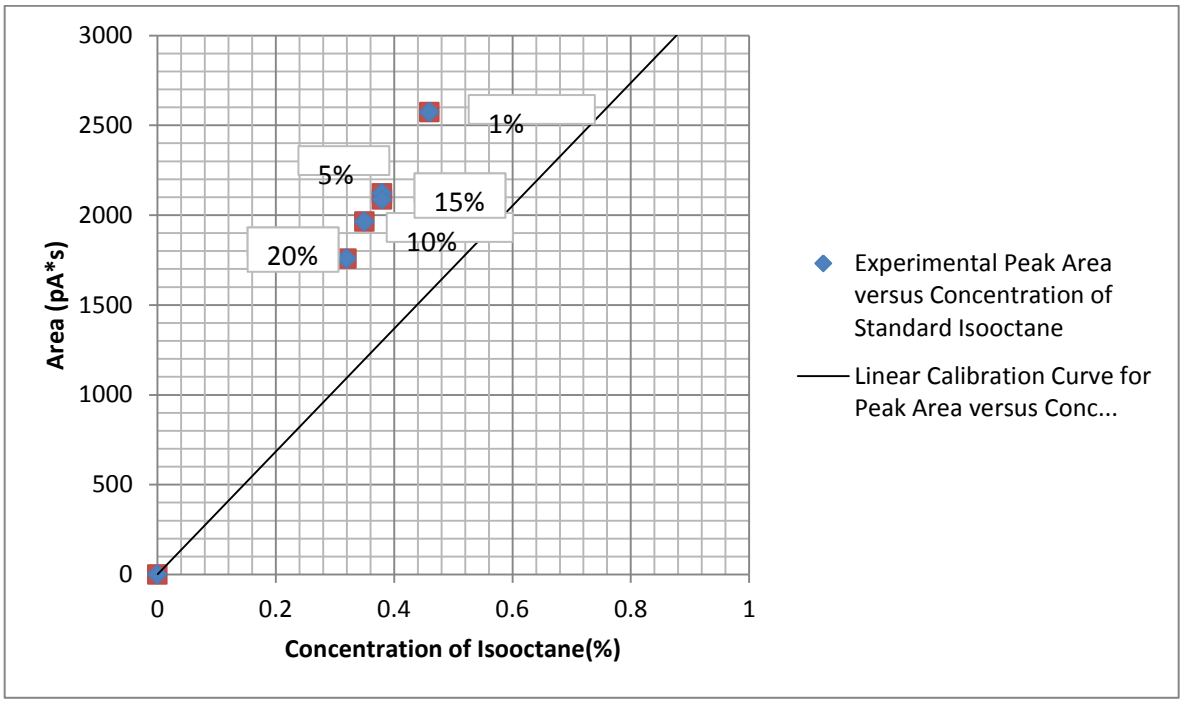

Figure 4.11: Concentration of Experimental Isooctane Present In Samples for Rubber Seed Mass to Solvent Mass Ratio 1:3

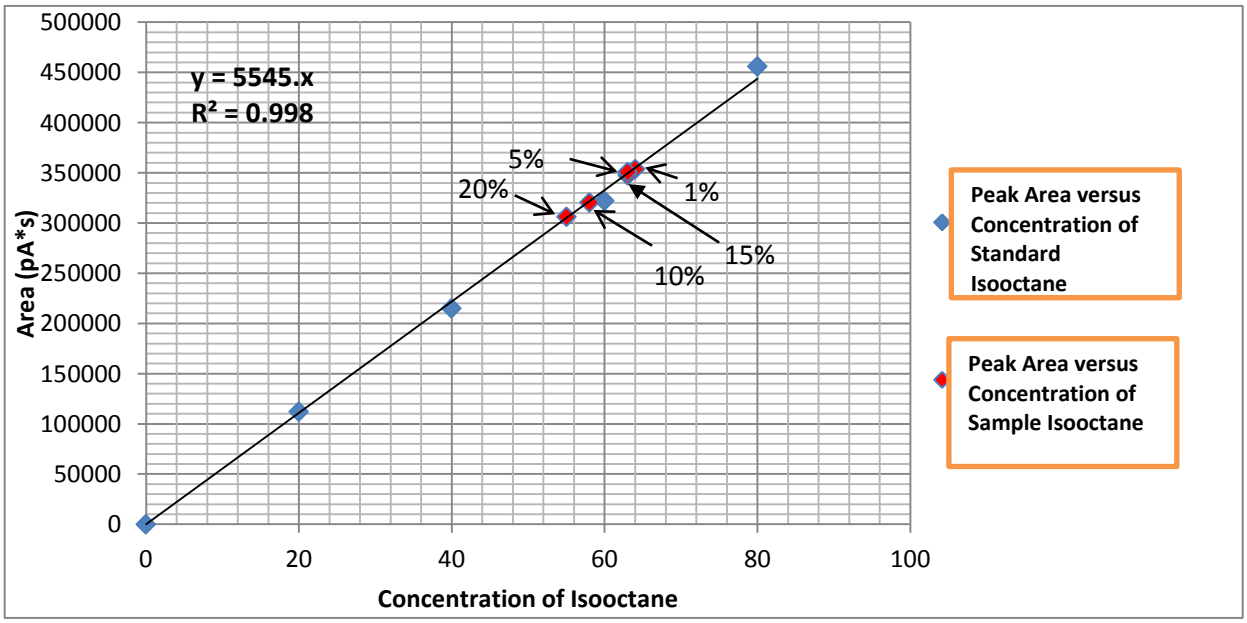

Figure 4.12: Concentration of Actual Isooctane Present In Samples for Rubber Seed Mass to Solvent Mass Ratio 1:3

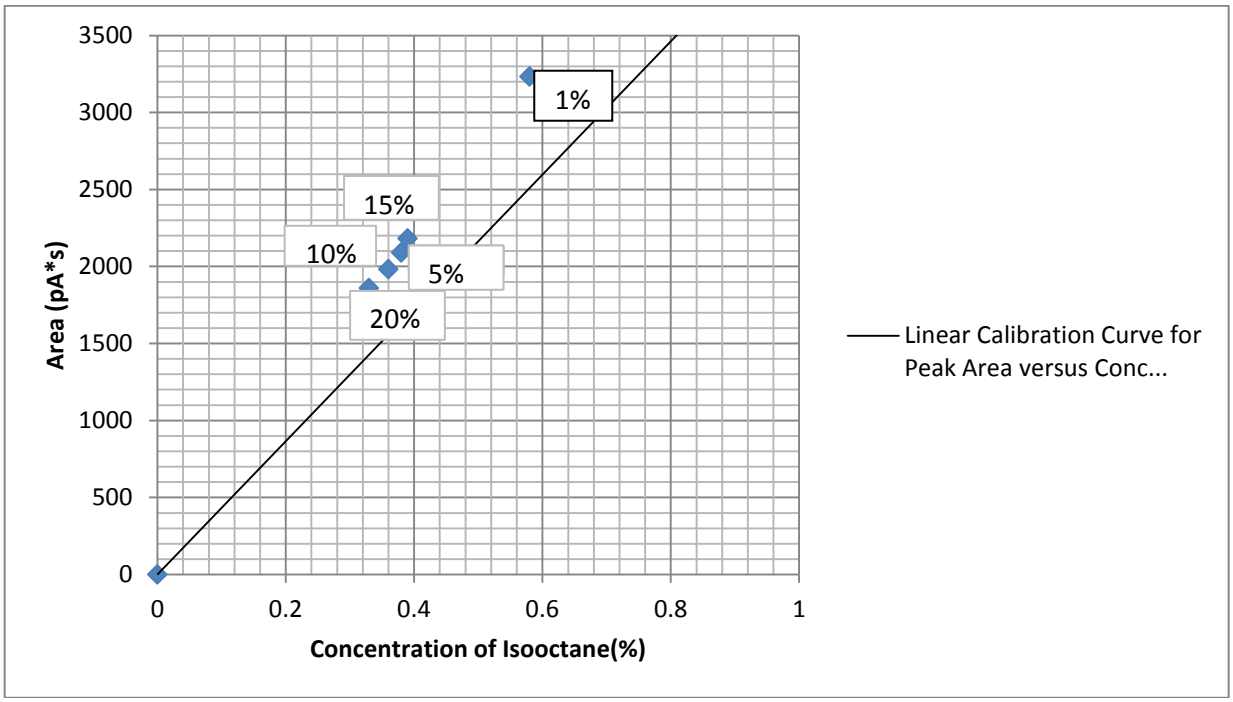

Figure 4.13: Concentration of Experimental Isooctane Present In Samples for Rubber Seed Mass to Solvent Mass Ratio 1:4

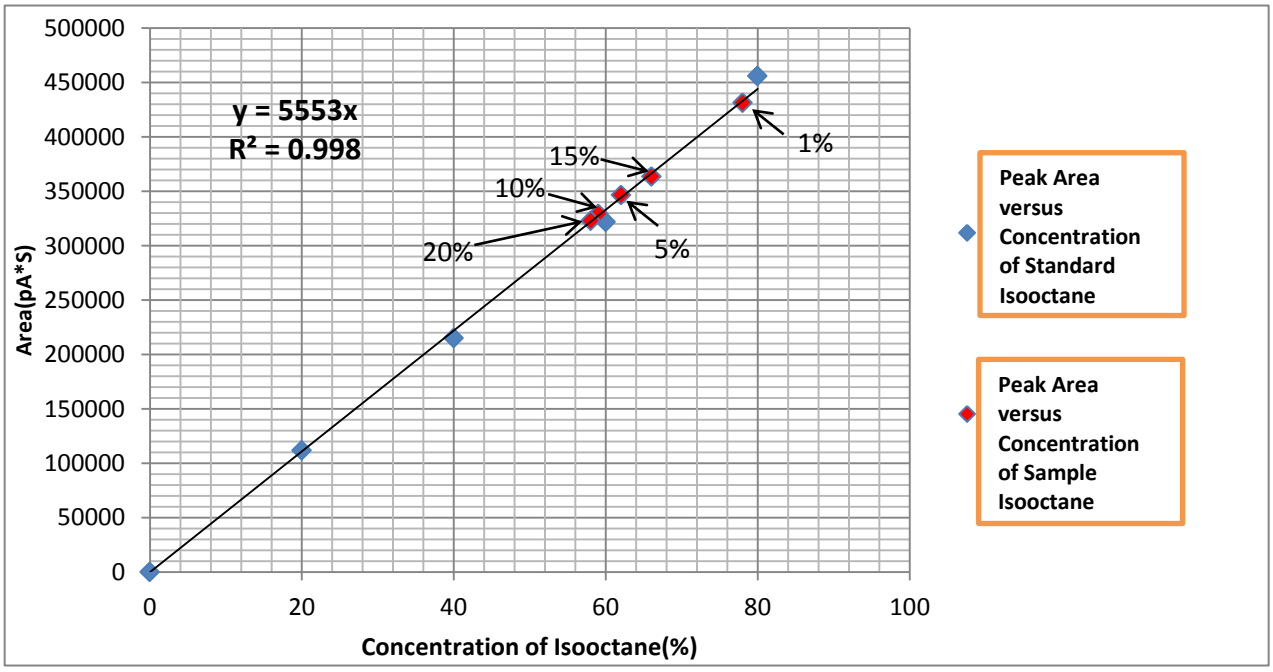

Figure 4.14: Concentration of Actual Isooctane Present In Samples for Rubber Seed Mass to Solvent Mass Ratio 1:4

4.4 DISCUSSION

From the results above we can see different percentage of isooctane have been extracted for different amount of solvent. The solvent which has been used to extract the rubber seed oil is ethanol. The amount of solvent has been has been used in three different mass ratios which are 1:2, 1:3 and 1:4. The rubber seed oil extraction process has been done in these different ratios so that the effect of amount of solvent in extraction of rubber seed oil can be studied. From the tabulated result from tables 4.3, 4.4 and 4.5 we can see the peak area for the isooctane have similar values. The tabulated values are given in table 4.8 .

Table 4.9: Results of Each Mass Ratio for Rubber Seed Mass to Solvent Mass Ratio

GC DILUTION (%)	MASS RATIO	EXPERIMENTAL PEAK AREA (pA*s)	ACTUAL PEAK AREA (pA*s)
1	1:2	2482.07	4.027E+5
1	1:3	2573.10	3.538E+5
1	1:4	3232.73	4.315E+5

The experimental peak area is taken from the Gas Chromatography results and the actual peak area is obtained through backward calculations. The larger peak area indicates higher concentration of isooctane. From the result we can see that the rubber seed to solvent mass ratio 1:2 has almost the same concentration of isooctane when compared to the highest rubber seed to solvent mass ratio of 1:4. So from these results it is clear that the amount of isooctane extracted from the same amount of rubber seed is the same when it is extracted using different amount of solvent. From here we can conclude that the minimum ratio of rubber seed to solvent to extract maximum amount of rubber seed oil is 1:2.

The results in tables 4.3, 4.4 and 4.5 shows decrease in peak area value as the percentage of dilution increases. As the dilution percentage increases the peak area value should have increased. The reduction in value has been caused by few factors. They are:

- **The cracked oil did not completely dissolved in hexane solvent**
- **Random cracking and isomerization during catalytic cracking process**
- **Contamination and Interferences**

The cracked oil did not completely dissolved in hexane solvent

When the oil is diluted in the solvent it needed to be completely dissolved in the solvent so that the sample could give accurate result of concentration of isooctane in the sample. As the amount of oil diluted increases the oil is not completely dissolved in the solvent and form an oil layer at the bottom of the vial. The needle in the gas chromatography machine only takes a small amount of oil and solvent mixture at the top part of the vial and do not reach until the bottom of the vial where the oil layer have formed. Due to this an accurate reading on the exact percentage of oil in the specific sample could not be determined correctly.

Random cracking during catalytical cracking and isomerization during catalytical cracking process

Breaking the long hydrocarbon chain into smaller, simpler and more useful bits of hydrocarbon compounds is the general concept of catalytic cracking. Smaller hydrocarbon radicals are formed by breaking the long chain hydrocarbon molecules in a random way, and through isomerization process these free radicals will recombine in different arrangements. 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.

Hydrocarbon bonds fairly break in the random way because the heat supplied randomly attacks any of the C-C bonds in the fatty acid, which**.** After the hydrocarbon

bonds have been broken in random ways, all the free radicals of the broken fatty acid molecules are attracted towards the catalyst surface and these radicals on its surface are combined in different molecular arrangement. The isomerization occurred through this arrangement. The catalyst makes chemical reaction go faster by lowering the activation energy and increase the conversion. So through this random isomerization not only isooctane is formed but there are also other hydrocarbons formed such as heptane, octane, nonane and decane. Because isooctane is not the only compound that have been produced as a result of catalytic cracking its concentration is not constant and produced in small quantity. When the oil sample is analyzed in gas chromatography due to the fact that other hydrocarbons present together with isooctane the peak area of isooctane in the final result has smaller value than expected value.

Contamination and Interferences

The presence of impurities in the apparatus and the glassware during conducting the experiment will affect the outcome or result of the experiment especially the result for Gas Chromatogram. Other than that the contamination factor can occur during the solidification process of rubber seed oil after it has been separated from ethanol solvent by using rotary evaporator. The isooctane present in a liquid form in room temperature but the unreacted fatty acids in the solution will affect the isooctane formation as the temperature decreases from its melting temperature as it start to solidify.

Figure 4.15: Rubber Seed Oil before solidification process

Figure 4.16: Rubber Seed Oil after solidification process

Low Volume Oil Produced During Catalytic Cracking

During the extraction of rubber seed oil process the rubber seed oil reacts with the solvent ethanol to produce ethylester.This reaction is called esterification. The rubber seed oil extracted by ethanol has different properties compared to rubber seed oil extracted by other solvent such as hexane and acetone. The properties of ethyl ester are shown in the table below.

Table 4.10: Properties of Ethyl and Methyl Ester

Special properties	RME[a]	REE[b]
Specific gravity	0.8802	0.876
Viscosity @ 40^{o}C	5.65	6.11
Cloud point (^{o}C)	0	-2
Pour point (^{o}C)	-15	-10
Flash point (^{o}C)	179	170
Boiling point (^{o}C)	347	273
Cetane number	61.8	59.7
Sulphur (%wt)	0.012	0.012
Gross heat of combustion	40.54	40.51
Net heat of combustion (MJ/kg)	37.77	37.82

[a]ME- represented methyl ester,[b]REE – represented ethyl ester

Source: Adapted from Peterson and Reece (1996)

.

From the table above we can see that the boiling point for the ethyl ester is 273^{0}C. During the catalytic cracking process a temperature of 300^{0}C has been used to crack the hydrocarbon chain in the rubber oil. This temperature is well over 27^{0}C than the actual boiling point of the ethyl ester and this made the ethyl ester to boil vigorously and spill out of the crucible. This is the reason why the volume of the oil is very low after the catalytic cracking process.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

The objective of this research is to extract rubber seed oil from rubber seeds by using different amount of solvent so that it can 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 can produced from a renewable source which is a agricultural material (rubber seeds) in the transportation sector to substitute the rapidly decreasing supply of fossil fuels. Other than that, the bio-petrol produced from a agricultural source can helps to decease the pollution levels in the environment with its capability of reducing the emission of CO_2 to the environment by promoting better efficiency of complete combustion inside the car engines.

From the data obtained, the result shows that the maximum concentration of Isooctane present in the 1:2, 1:3 and 1:4 mass of rubber seed to mass of solvent ratio (for 1% dilution) are 73%, 64% and 78% respectively. The volume of solvent used in the oil extraction process will affect the percentage of concentration of Isooctane in samples. 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 mixture present in the rubber seed oil, the contamination factor and the oil did not completely dissolved in the solvent before going into gas chromatography analysis.

As a conclusion, Bio-petrol is able to be produced using Zeolite catalyst in the catalytic cracking of rubber seed oil extracted from rubber seeds. In other words, the random cracking and isomerization of fatty acids present in the rubber seed oil is very useful in producing a renewable energy source to replace the continuously diminishing fossil fuel. However, this alternative method to produce renewable energy must be constantly updated and researched with further modern technology in the areas of techniques, economics, environment and the feasibility so that it can be scaled up to an industry level.

5.2 RECOMMENDATION

The results of the experiment can be more accurate if GC-MS was used instead of gas chromatography. This is because the GC-MS runs the sample and tell you what the elements in a solution are and what their percentages are. Other than that, the GC-MS will take in the entire sample in the vial so there would not be a problem of forming oil layer at the bottom part of the vial. Furthermore, to obtain more precise results, it is best recommended to modify the vertical axis peak area of the chromatogram which is 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. Another recommendation is using different type of solvent such as dimethyl ether and petroleum ether because it produces much larger amount of Isooctane. Besides that, the samples quality can be assured by using clean glassware and apparatus. As an example, the glassware should be properly cleaned and dryed first before being used in the experiment. In addition, human errors such as parallax reading should be minimized to the lowest level to achieve better results. Human errors can be minimized by taking the average values of each measurement or data reading taken and by having our eye at the correct position so that we can have accurate reading of the scale.

REFERENCES

A.H. Ruffell, S. P. Hesselbo, G. D. Wach, M. I. Simpson & D. S. Wray, 2002 , Fuller's earth (bentonite) in the Lower Cretaceous (Upper Aptian) of Shanklin (Isle of Wight, southern England).

Aigbodion A.I, and C.K.S., Pillai (2000), Preparation, analysts and applications of rubber seed oil and its derivatives in surface coatings, Progress in organic coatings 38, pp, 187-192.

A.S. Ramadhas, S. Jayaraj, C. Muraleedharan , 2004, Biodiesel production from high FFA rubber seed oil.

Ayhan Demirbas, (2008), Energy Conversion and Management; Bio-fuels securing the planet's future energy needs.

Balat M. (2008), Progress in biogas production processes, Energy Edu Sci Technol, 22:15–36.

Bondt, N., Deiman, J.R., van Troostwyk, P. and Lowrenberg, A., 1797. Ann. Chim Phys., 21."48.

Chin, H.F.Enoch, I.C. and Rajahamn, R.M (1977), Seed technology in the topics, Faculty of Agriculture, University Putra Malaysia Publishing, Kuala Lumpur, Malaysia.

Demirbas A. Political, economic and environmental impacts of bio-fuels: A review. Applied Energy 86: 2009; S108-S117.

Demirbas A. (2007). Progress and recent trends in bio-fuels. Prog. Energy Combus, Sci.33, 1-18.

Demirbas A, Urkmez A. (2006), Biomass based combined heat and power (CHP) systems in Turkey, Energy Sources Part B, 1:245–53.

Encyclopedia Britannica, Metabolism; August 2006.

Ghandhi, V.M. Cheriak, M. and Mulky, M.J. (1990): Nutritional and toxicological evaluation of rubber seed oil, Journal of American Oil Chemical Society 67, pp. 883-886.

Hansford, R.C., 1983, Development of the theory of catalytic cracking. In: B. Davis and W.P. Hettinger Jr. (Editors), Heterogeneous Catalysis- Selected American Histories, ACS Symposium Series No. 222; Washington, D.C., p. 247.

Haydn H. Murray, 2000,Traditional and new applications for kaolin, smectite, and palygorskite: a general overview.

Hardjosuwito, B., Hoesnan, A. (1978), Rubber Seed Oil Abstr. 3(7), 17.

Helmut Knozinger, Karl Kochloefl, Heterogeneous catalysis and solid catalysts in Ullmann's Encyclopedia of Industrial Chemistry 2002, Wiley-VCH, Weinheim.

Houdry, E., J., A., 1956. Bull Assoc. Ft. Tech Pet., 117.

Ikhuonia, E.U. Aigbodion, A.I. and Okieimen, F.E (2005), Preparation and characterization of water-reducible alkyds with fumarized rubber seed oil, Progress in organic coatings 52, pp. 238-240.

International Energy Annual 2006, Retrieved 2009-02-08.

James. G. Speight (2006), The Chemistry and Technology of Petroleum (4th ed.). CRC Press.

James H. Gary and Glenn E. Handwerk (2001), Petroleum Refining: Technology and Economics (4th ed.).CRC Press.

J.N. Chheda, G.W. Huber, J.A. Dumesic, Angew. Chem. Int. Ed. 46 (2007) 7164–7183.

Knothe G., R.O. Dunn and M.O. Bagby, 1998. Biodiesel: the use of vegetable oils and their derivatives as alternative diesel fuel.

Laurence M. Harwood, Christopher J. Moody, Experimental organic chemistry: Principles and Practice (Illustrated edition), pp, 47–51.

Miguez JL, Lopez-Gonzalez LM, Porteiro J, Paz C, Granada E, Moran JC, (2006) Contribution of renewable energy sources to electricity production in Galicia (Spain), Energy Sources Part A. 28:995–1012.

Ni, M., Leung, D. Y. C., Leung, M. K. H., Sumathy, K. (2006), An overview of hydrogen production from biomass. Fuel Process. Technol. 87, 461-472.

Njoku, O. U., Ononogbu, I. C., Owusu, J. Y. (1996). An investigation of oil of rubber (Hevea bransiliensis). J. of Rubber Res. Inst. Sri Lanka 78, 52-59.

Nwoko, E.Kills, D.D and Kanhoi, J. (1988). Serum and liver lipids of rats feed rubber seed oil. Plant foods for Human Nutrition 38, pp. 145-153.

Njoku, O.U., Ononogbu, I.C., Owusu, J.Y., 1996. An investigationof oil of rubber (He6ea bransiliensis). J. of Rubber Res. Inst. Sri-Lanka 78, 52–59.

O.U. Njoku I.C., Ononogbu, O.E. Ikwuagwu , 1999 Production of biodiesel using rubber [Hevea brasiliensis (Kunth. Muell.)] seed oil.

Oblad, A.G, 1983. The contributions of Eugene J. Houdry to the development of catalytic cracking. In: B. Davis and W.P. Hettinger Jr. (Editors), Heterogeneous Catalysis—Selected American Histories. ACS Symposium Series No. 222; Washington, D.C., p. 61.

Paul Mann, Lisa Gahagan, and Mark B. Gordon (Accessed 22 June 2009). "Tectonic setting of the world's giant oil and gas fields," in Michel T. Halbouty (ed.) "Giant Oil and Gas Fields of the Decade, 1990-1999, Tulsa, Okla". American Association of Petroleum Geologists, p.50.

Ramadhas, A.S., Muraleedharam, C. and Jayaraj. (2005), Performance and emission evaluation of a diesel fueled with methyl esters of rubber seed oil, Renewable Energy 30, pp, 1789-1800.

Randall von Wedel, Ph.D, (April 22, 1999), Marine Biodiesel and Education Project for San Francisco Bay and Northern California Second Edition prepared for the National Renewable Energy Laboratory U.S. Department of Energy. CytoCulture International, Inc.

Reza Sadeghbeigi (2000), Fluid Catalytic Cracking Handbook (2nd ed), Gulf Publishing.

R.H.S. Robertson. Voltura Press, Hythe, Kent, 1986. Fuller's Earth. A History of Calcium Montmorillonite.

Rock Talk Vol 7 No.2 Colorado Geological Survey.

S.N. Hassan, N.L.A. Rahman and M.S.M. Shek, 2009, Concerntration yeild of biopetrol from oleic acid.

Siriwardene, J.A. Des and Nugara, D. (1972), Nutrive value of rubber seed meal, Ceylon Veterinary Journal 20:61.

Westermann P., Jorgensen B., Lange, L., Ahring, B. K., Christensen, C. H. (2007), Maximizing renewable hydrogen production from biomass in a bio/ catalytic refinery, Int. J. Hydrogen Energy 32, page 4135-4141.

APPANDIX A

GC Analysis Of Standard Solution

```
Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL0000001.D
 Sample Name: s 1
    ======================================================================
    Acq. Operator   : fiza30nov                      Seq. Line :   1
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 30/11/2011 10:00:11                Inj :   1
                                                    Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 30/11/2011 09:58:21  by fiza30nov
    Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 13/12/2011 15:10:48  by fiza30nov
                      (modified after loading)
    Method Info     : isooctane/pam

    ======================================================================
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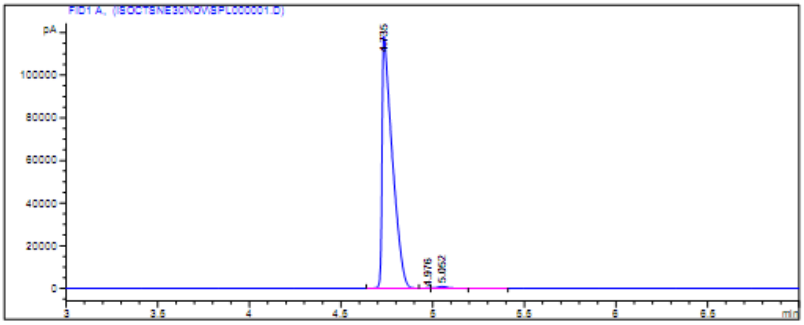



```
    ======================================================================
                          Area Percent Report
    ======================================================================

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


    Signal 1: FID1 A,

    Peak RetTime  Type   Width     Area      Area     Name
      #   [min]           [min]    [pA*s]       %
    ----|-------|------|-------|----------|--------|--------------------
       1   2.185          0.0000    0.00000  0.00000
       2   2.253          0.0000    0.00000  0.00000
       3   2.308          0.0000    0.00000  0.00000
       4   2.359          0.0000    0.00000  0.00000
       5   2.762          0.0000    0.00000  0.00000
       6   4.735 VB S     0.0525 4.56593e5   99.31207
       7   4.976 BV X     0.0349  140.91379  0.03065 ?
       8   5.052 VV X     0.0555 3021.87866  0.65728 ?

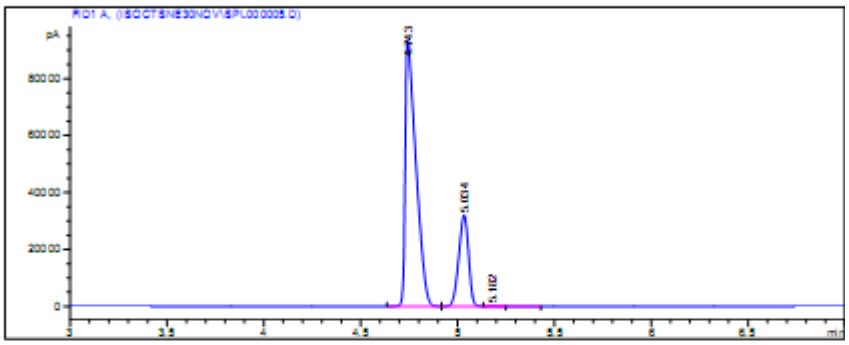
    Totals :                    4.59756e5
```

GC FID Analysis : 0% standard isooctane solution

Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL000005.D
Sample Name: s5

Acq. Operator : fisa30nov Seq. Line : 5
Acq. Instrument : Instrument 1 Location : Vial 5
Injection Date : 30/11/2011 11:30:10 Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 30/11/2011 09:58:21 by fisa30nov
Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 13/12/2011 15:03:56 by fisa30nov
(modified after loading)
Method Info : isooctane/pan



Area Percent Report

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

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Area %	Name
1	2.185		0.0000	0.00000	0.00000	
2	2.253		0.0000	0.00000	0.00000	
3	2.308		0.0000	0.00000	0.00000	
4	2.359		0.0000	0.00000	0.00000	
5	2.762		0.0000	0.00000	0.00000	
6	4.743	VV S	0.0541	3.57789e5	76.06407	
7	5.034	VB S	0.0545	1.12430e5	23.90208	?
8	5.182	BV ?	0.0468	159.23813	0.03385	?

Totals : 4.70378e5

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
Warning : Calibrated compound(s) not found

Summed Peaks Report

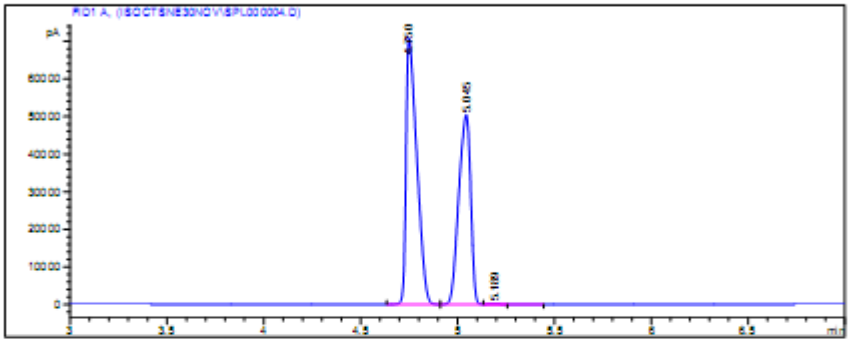
Signal 1: FID1 A,

GC FID Analysis : 20% standard isooctane solution

Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL0000004.D
Sample Name: s4

Acq. Operator : fisa30nov Seq. Line : 4
Acq. Instrument : Instrument 1 Location : Vial 4
Injection Date : 30/11/2011 11:07:43 Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 30/11/2011 09:58:21 by fisa30nov
Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 13/12/2011 15:03:56 by fisa30nov
(modified after loading)
Method Info : isooctane/pan



Area Percent Report

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

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Area %	Name
1	2.185		0.0000	0.00000	0.00000	
2	2.253		0.0000	0.00000	0.00000	
3	2.308		0.0000	0.00000	0.00000	
4	2.359		0.0000	0.00000	0.00000	
5	2.762		0.0000	0.00000	0.00000	
6	4.750	VV S	0.0524	2.72851e5	55.84981	
7	5.045	VB S	0.0708	2.15359e5	44.08180	?
8	5.189	BV T	0.0491	334.12787	0.06839	?

Totals : 4.88544e5

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
Warning : Calibrated compound(s) not found

Summed Peaks Report

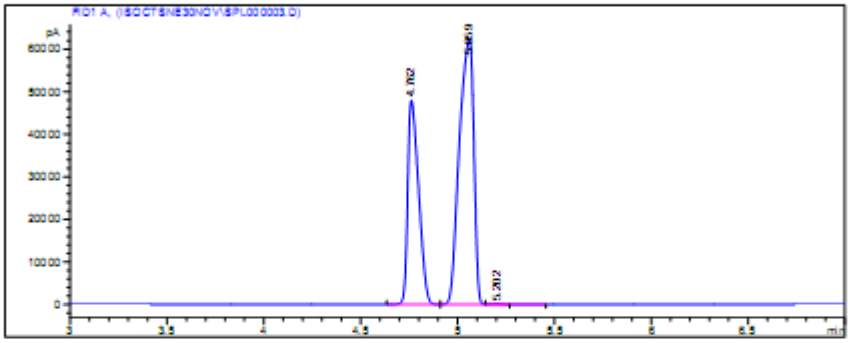
Signal 1: FID1 A,

GC FID Analysis : 40% standard isooctane solution

Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL0000003.D
Sample Name: s3

Acq. Operator : fisa30nov
Acq. Instrument : Instrument 1
Injection Date : 30/11/2011 10:45:10
Seq. Line : 3
Location : Vial 3
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 30/11/2011 09:58:21 by fisa30nov
Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 13/12/2011 15:03:56 by fisa30nov
(modified after loading)
Method Info : isooctane/pan



Area Percent Report

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

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Area %	Name
1	2.185		0.0000	0.00000	0.00000	
2	2.253		0.0000	0.00000	0.00000	
3	2.308		0.0000	0.00000	0.00000	
4	2.359		0.0000	0.00000	0.00000	
5	2.762		0.0000	0.00000	0.00000	
6	4.762	VV S	0.0590	1.86902e5	36.68721	
7	5.059	VB S	0.0766	3.22048e5	63.21528	?
8	5.202	BV T	0.0502	496.78217	0.09751	?

Totals : 5.09446e5

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
Warning : Calibrated compound(s) not found

Summed Peaks Report

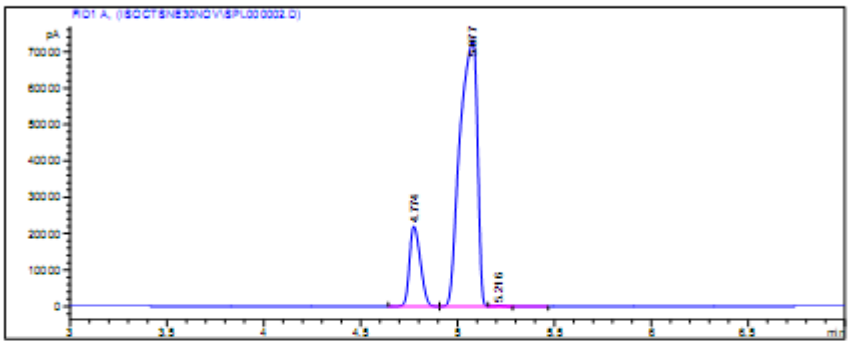
Signal 1: FID1 A,

GC FID Analysis : 60% standard isooctane solution

Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL000002.D
Sample Name: s2

Acq. Operator : fisa30nov Seq. Line : 2
Acq. Instrument : Instrument 1 Location : Vial 2
Injection Date : 30/11/2011 10:22:40 Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 30/11/2011 09:58:21 by fisa30nov
Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 13/12/2011 15:09:52 by fisa30nov
(modified after loading)
Method Info : isooctane/pan



Area Percent Report

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

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Area %	Name
1	2.185		0.0000	0.00000	0.00000	
2	2.253		0.0000	0.00000	0.00000	
3	2.308		0.0000	0.00000	0.00000	
4	2.359		0.0000	0.00000	0.00000	
5	2.762		0.0000	0.00000	0.00000	
6	4.774	BV S	0.0602	8.61105e4	15.85754	
7	5.077	VB S	0.0828	4.56253e5	84.02050	?
8	5.216	BV ?	0.0503	662.29755	0.12196	?
Totals :				5.43028e5		

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
Warning : Calibrated compound(s) not found

Summed Peaks Report

Signal 1: FID1 A,

GC FID Analysis : 80% standard isooctane solution

APPANDIX B

GC Analysis for Sample

Data File C:\CHEM32\1\DATA\ISOOCTANE271211SECOND\007F0401.D
Sample Name: R1N2sp1

Acq. Operator : wan127dec
Acq. Instrument : Instrument 1
Injection Date : 27/12/2011 12:55:32
Seq. Line : 4
Location : Vial 7
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
last changed : 20/12/2011 09:21:16 by fisa20nov
Analysis Method : C:\CHEM32\1\METHODS\B4ASHUTDOWN.M
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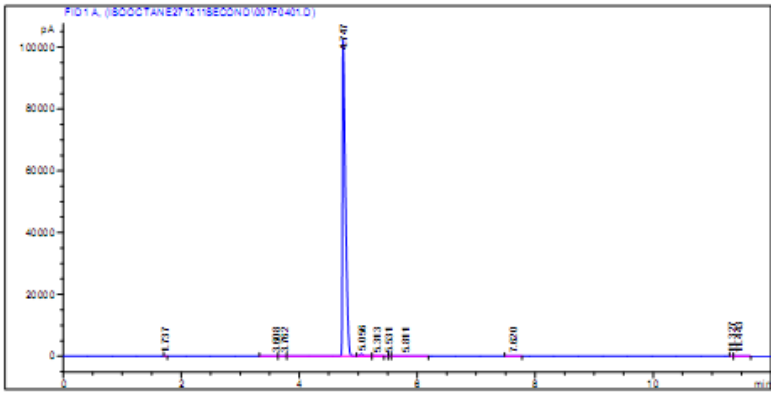
(modified after loading)
Method Info : std testing



Area Percent Report

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

Signal 1: FID1 A,

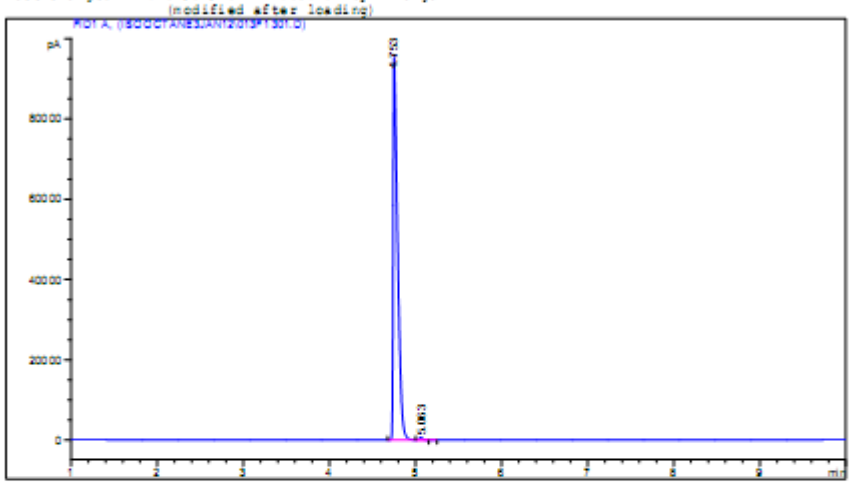
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.737	BV	0.0403	2.36669	7.59738e-1	0.00066
2	3.608	BV	0.1007	4.38034	5.28639e-1	0.00122
3	3.762	VV	0.0998	2.92638	3.56229e-1	0.00082
4	4.747	VB S	0.0505	3.56232e5	1.00227e5	99.29216
5	5.096	BV X	0.0515	2482.07080	752.68481	0.69183
6	5.313	VB X	0.0540	22.23389	6.48354	0.00620
7	5.531	BV	0.0371	1.27774	4.62801e-1	0.00036
8	5.811	VB	0.3528	13.55651	4.87372e-1	0.00378
9	7.620	VB	0.0886	1.61238	2.55711e-1	0.00045
10	11.327	BV	0.0386	1.61582	5.45543e-1	0.00045
11	11.443	VB	0.1496	4.23536	3.56503e-1	0.00118
12	12.299	VB	0.1002	1.80216	2.62215e-1	0.00050
13	15.334	VB	0.1045	1.45472	1.72306e-1	0.00041

Instrument 1 28/12/2011 10:32:14 wan127dec Page 1 of 2

GC FID Analysis (1:2): 1% isooctane dilution

Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\013F1301.D
Sample Name: RUN2 5%

Acq. Operator : fisa3jan Seq. Line : 13
Acq. Instrument : Instrument 1 Location : Vial 13
Injection Date : 03/01/2012 13:55:54 Inj : 1
Inj Volume : 1 µl
Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 20/12/2011 09:21:16 by fisa20nov
Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
Last changed : 04/01/2012 12:03:10 by fisa4jan
(modified after loading)



Area Percent Report

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

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.753	BB S	0.0535	3.39732e5	9.54853e4	99.38378
2	5.063	BB T	0.0525	2106.47607	638.16516	0.61622
Totals :				3.41839e5	9.61235e4	

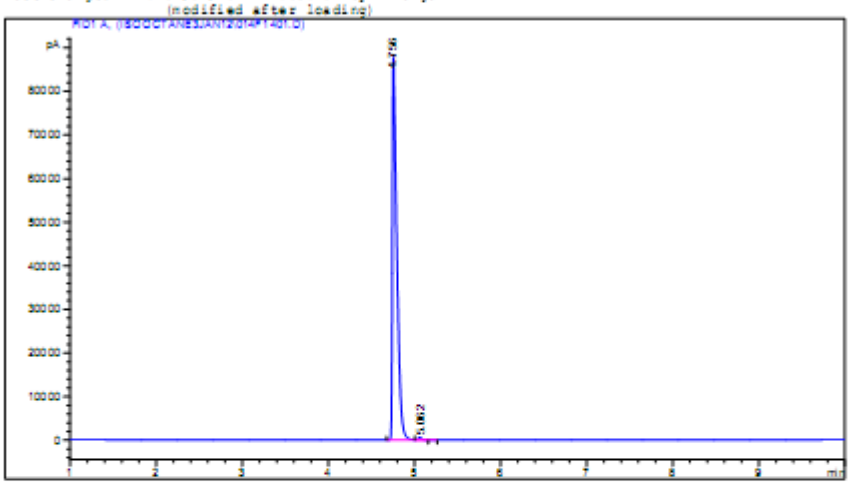
*** End of Report ***

Instrument 1 04/01/2012 12:17:38 fisa4jan Page 1 of 1

GC FID Analysis (1:2): 5% isooctane dilution

Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\014F1401.D
Sample Name: R1N2 10%

Acq. Operator : fisa3jan Seq. Line : 14
Acq. Instrument : Instrument 1 Location : Vial 14
Injection Date : 03/01/2012 14:18:25 Inj : 1
Inj Volume : 1 µl
Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE23OCT08.M
Last changed : 20/12/2011 09:21:16 by fisa20nov
Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
Last changed : 04/01/2012 12:03:10 by fisa4jan
(modified after loading)



Area Percent Report

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

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.756	BB S	0.0508	3.14293e5	8.78094e4	99.39440
2	5.062	BB T	0.0517	1914.94385	577.08661	0.60560

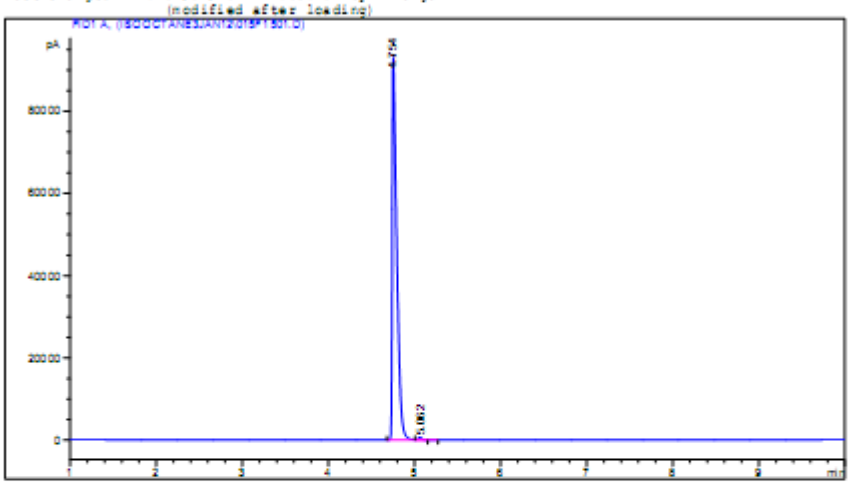
Totals : 3.16208e5 8.83865e4

*** End of Report ***

GC FID Analysis (1:2): 10% isooctane dilution

Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\015F1501.D
Sample Name: R1N2 15%

Acq. Operator : fisa3jan Seq. Line : 15
Acq. Instrument : Instrument 1 Location : Vial 15
Injection Date : 03/01/2012 14:40:57 Inj : 1
Inj Volume : 1 µl
Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed : 20/12/2011 09:21:16 by fisa20nov
Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
Last changed : 04/01/2012 12:03:10 by fisa4jan
(modified after loading)



Area Percent Report

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

Signal 1: FID1 A,

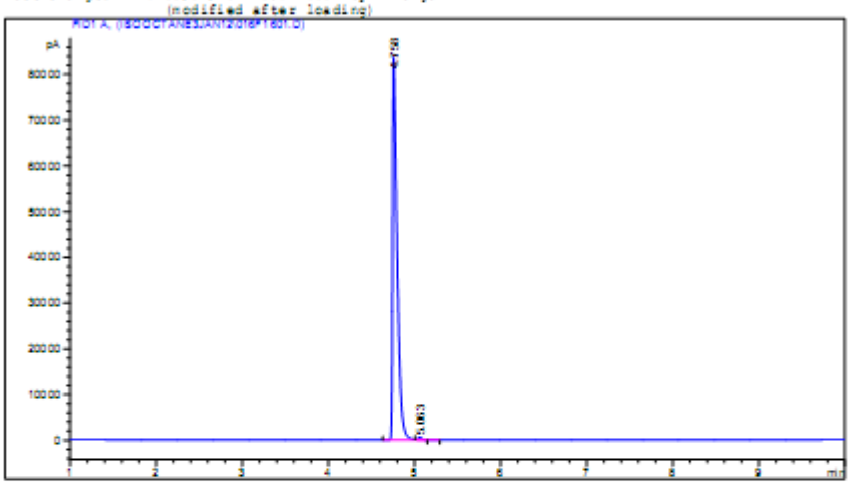
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.754	BB S	0.0507	3.39177e5	9.26994e4	99.40076
2	5.062	BB T	0.0518	2044.72559	614.73822	0.59924
Totals :				3.41221e5	9.33141e4	

*** End of Report ***

GC FID Analysis (1:2): 15% isooctane dilution

Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\016F1601.D
Sample Name: R1N2 20%

Acq. Operator : fisa3jan Seq. Line : 16
Acq. Instrument : Instrument 1 Location : Vial 16
Injection Date : 03/01/2012 15:03:33 Inj : 1
Inj Volume : 1 µl
Acq. Method : C:\CHEM32\1\METHODS\ISOOCTANE23OCT08.M
Last changed : 20/12/2011 09:21:16 by fisa20nov
Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
Last changed : 05/01/2012 09:24:08 by fisa4jan
(modified after loading)



Area Percent Report

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

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.758	BB S	0.0539	3.06836e5	8.34964e4	99.42172
2	5.063	BB X	0.0500	1784.68894	547.61108	0.57828

Totals : 3.08620e5 8.40440e4

*** End of Report ***

GC FID Analysis (1:2): 20% isooctane dilution

APPENDIX C

Material Safety Data Sheet (MSDS)

Safety data for hexane

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.

Safety data for ethanol

Identification

Product Name: Ethanol

Other Names: Ethanol**,** Ethyl alcohol

Product Code: 10476

UN Number: 1170

Dangerous Goods Class &Subsidary Risk: 3

Hazchem Code: 2SE

Poisons Schedule Number: None allocated

Use: General laboratory reagent EEC #: 200-578-6

Packaging Group: II

Physical Description and Properities

Form: liquid

Boiling point: 78 ºC

Melting point: -117 ºC

Vapor pressure: (20 ºC) ~ 59 mbar

Flashpoint (Open Cup): 12 ºC

Upper flammability limit: 15 Vol%

Lower flammability limit: 3.5 Vol%

Solubility in Water: water (20 ºC) soluble

Ignition temperature: 425 ºC

Colour:colourless

Odour: characteristic

Vapor density: 1.59

Density: (20 ºC) 0.81 g/cm3

Other information: Highly flammable.

Conditions and substances to be avoided:

Conditions to be avoided

Strong heating: - (Explosible with air in a vaporous/gaseous state.)

Substances to be avoided

alkali metals, alkaline earth metals, alkali oxides, strong oxidizing agents.

Health Hazard Information:

Toxicological information:

Acute toxicity

LD50 (oral, rat): 7060 mg/kg Sub acute to chronic toxicity

An embryo toxic effect need not be feared when the threshold limit value is observed.

After inhalation of vapors:

Slight mucosal irritations.Risk of absorption.

After eye contact:

Slight irritations.

After ingestion of large amounts: nausea and vomiting. Systemic effect: euphoria. After absorption of largequantities: dizziness, inebriation, narcosis, respiratory paralysis.

After ingestion:

After ingestion: Damage to: mucous membrane. Rapid absorption.

First Aid Information:

Eye contact:

Rinse out with plenty of water with the eyelid held wide open.

Inhalation:
Fresh air. Consult doctor if feeling unwell.

Skin contact:
Wash off with plenty of water. Remove contaminated clothing.

Ingestion:
Drink plenty of water. Induce vomiting. No emetics. No animal charcoal.

Advice to doctors:
Treat symptomatically.

Precautions for use:

Engineering controls:
Person-related precautionary measures: Do not inhale vapours/aerosols.Procedures for cleaning / absorption: Take up with liquid-absorbent material. Forward for disposal. Rinse away remainder with water.

Personal protection

Respirator:
required when vapours/aerosols are generated. Filter A (acc. to DIN 3181) forvapours of organic compounds
Ensure respirator is clean, well-fitting and in good working order. All respirators should comply with Australian Standard AS 1716 and be used in accordance with AS 1715.

Gloves:
required

Eye protection:
Required

Special risks:
Combustible.Vapours heavier than air.Formation of explosible mixtures possible withair. Take measures to prevent electrostatic charging.

Suitable extinguishing media:
Water, CO2, foam, powder.

Other precautions:
Change contaminated clothing. Wash hands after working with substance.