THE BIOPETROL SYNTHESIS FROM PALMITIC ACID-HETEROGENOUS CATALYTIC CRACKING WITH ZEOLITE CATALYST

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ABSTRACT

Biofuel is defined as fuel produced from derivation of vegetable oils and specifically, biopetrol is defined as fuel which has the same characteristic with the petrol, but is produced from palmitic acid that is dominated in palm oil where the conversion of palmitic acid is done to get the molecular formula and structure of isooctane. Due to depletion of fossil fuel, environmental issues, and rising of petrol price, biopetrol can be the alternative fuel to the fossil fuel. Catalytic cracking method is conducted in this research due to the low yields from thermal cracking method. Futhermore, Malaysia is plenty with palm oil which palmitic acid is dominated in palm oil composition. In recent years, there have been several other studies on the production of hydrocarbons from palm oil mainly bio-gasoline (biopetrol) which have been carried out using cracking catalysts. Zeolites have shown excellent performance as solid acid cracking catalysts due to their higher selectivity. Many researchers have studied that dynamic catalytic cracking method will produce much higher yields than static cracking method and thermal catalytic cracking method. Through catalytic cracking process, palmitic acid is catalytic crack with 5g zeolite and 4 different rotation speed which is 600rpm, 780rpm, 960rpm and 1140rpm. The distillated product is diluted with mixture solution hexane at the ratio of 1:24. All the product samples are analyzed with Gas Chromatographer (GC). The isooctane concentrations are increased when the speed of reaction are increased, but not obviously. The lowest percentage concentration of isooctane obtained is 16.63376% and the highest percentage concentration of isooctane obtained is 23.09627%. These yields are much higher than the yields produced from static cracking method which is 20.7210% to 23.09627 %. It showed that the dynamic catalytic cracking method will produce much higher yields for biopetrol synthesized compared to thermal cracking method.

ABSTRAK

Biofuel ditakrifkan sebagai bahan api yang dihasilkan daripada terbitan oleh minyak sayuran. Secara spesifiknya biopetrol juga ditakrif sebagai bahan api yang mempunyai sifat yang sama dengan petrol tetapi ia dihasilkan daripada asid palmitik di mana komposisinya banyak terdapat pada minyak sawit. Pertukaran asid palmitik dilakukan dengan mendapat formula molekul dan struktur isooktana. Oleh kerana sumber bahan api yang semakin berkurangan, isu alam sekitar iaitu pencemaran alam, dan kenaikan harga petrol, biopetrol boleh menjadi bahan api alternatif kepada bahan api fosil. Kaedah penghuraian bermangkin digunakan dalam kajian ini disebabkan hasil yang dijana daripada kaedah penghuraian haba adalah rendah. Tambahan pula Malaysia kaya dengan sumber minyak sawit di mana asid palmitik mendominasi komposisi di dalamnya. Kini, terdapat kajian berkenaan penghasilan biopetrol daripada hidrokarbon minyak sawit. Kajian dijalankan menggunakan penghuraian bermangkin. Mangkin zeolite telah menunjukkan hasil yang cemerlang sebagai mangkin penhuraian asid pepejal kerana kadar selektif dan pemilihannya yang tinggi. Banyak penyelidik telah menemui bahawa kaedah penghuraian bermangkin dapat menghasilkan produk yang lebih tinggi daripada kaedah penghuraian haba penghuraian berkatalis, asid palmitik dihuraikan secara bermangkin dengan 5g zeolite serta 4 kelajuan putaran yang berbeza 600rpm, 780rpm, 960rpm dan 1140rpm. Produk disulingkan dicairkan dengan larutan campuran heksana pada nisbah 1:24. Semua sampel produk dianalisis dengan Gas Chromatographer (GC). Kepekatan isooktana meningkat ketika kelajuan reaksi meningkat, tetapi ketara peningkatannya. Kepekatan isooktana terendah diperolehi isooctane 16.63376% dan kepekatan peratusan tertinggi yang diperolehi isooktana 23.09627%. Keputusan ini jauh lebih tinggi daripada hasil yang dihasilkan dari kaedah penghuraian bermangkin tetap yang 20.7210% menjadi 23.09627%.. Eksperimen ini dijalankan dalam keadaan

dinamik untuk mendapatkan hasil yang lebih tingi berbanding kaedah penghuraian bermangkin tetap.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

1.0 General

Biopetrol is one type of biofuels those were defined as fuel derived from vegetable oils. Biopetrol is specifically defined as fuel derived from palmitic acid as dominative component in palm oil, and it has the same characteristic with the petrol, based on its molecular chemical formula and structure of isooctane. Biopetrol is synthesized as alternative of the fossil fuel that currently used by petrol-used vehicles. Biopetrol and other biofuels are the best way of reducing the emission of the greenhouse gases. They can also be looked upon as a way of energy security which stands as an alternative of fossil fuels that are limited in availability. Today, the use of biofuels has expanded throughout the globe. Some of the major producers and users of biogases are currently in Asia, Europe and America. Theoretically, biofuels can be easily produced through any carbon source however several photosynthetic plants are the material sources those most commonly used for biofuel production. Almost all types of materials derived from the plants are used for manufacturing biogas. One of the greatest problems that are being faced by the researchers in the field is the conversion process of biomass and biogas into the liquid biofuel.

1.1 Identification of Problem

1.1.1 Rising of Petrol Oil Prices

Petrol oil prices have been spiralled annually. It will cause many domino effect in terms of goods and services cost and will interrupt the global economic growth and stability. The highest crude oil price in ever history until now that has been recorded on Jun 30, 2008 that is 143 US Dollar per barrel. This will also increase all the petroleum-based product prices, which diesel and petrol were mostly affected. Figure 1.1 shows the price of global crude oil from 1994 to March 2008 and Figure 1.2 shows the petrol price in Malaysia from May 2004 to Jun 2008.

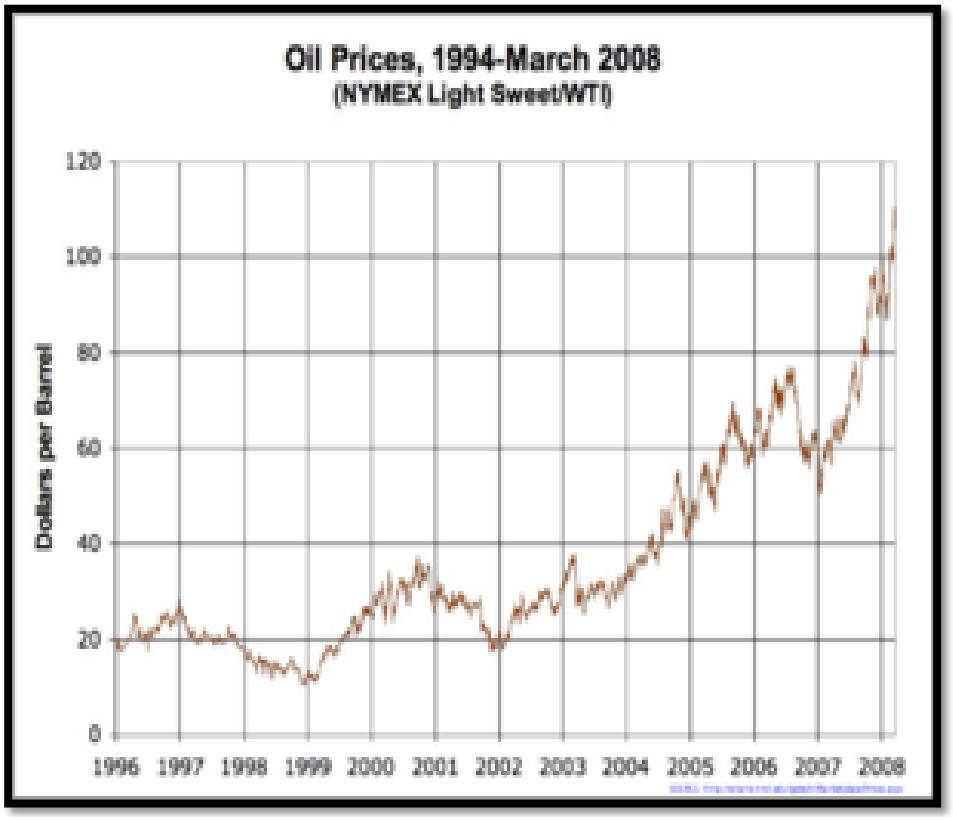

Figure 1.1: Price of World Crude Oil from 1994 to March 2008

Figure 1.2: Petrol Price in Malaysia from May 2004 to Jun 2008

One of the major factors that caused this increasing of petroleum oil prices is the limitation of the fossil fuel reserves and high demand for the petroleum-based product. Fossil fuel are non-renewable energy resources because the formation fossil fuels will take millions of years, and the fossil fuel reserves are being depleted much faster than new one being formed. According to Malaysia's situation, if Petroliam Nasional Malaysia Berhad (PETRONAS) does not discover new fuel oil reserve, the Malaysia oil reserve will end around 20 until 22 years later and fully become net importer petroleum country.

Due to the rising of the development growth around the world, the demand of petroleum-based product was increased especially gasoline (greatest demand of gasoline caused over 50% of the crude oil be converted into it). The drastic economic growth from the India and China will affect one of the major petroleum limited supply according to the recent global situation. Worldwide energy demand, driven by the population growth and industrialization of the developing world, will expand by 40% in the next 20 years. This will result the unbalance of demand and supply. The helps to monitor increasing fuels' prices were reports that petroleum production is at (Deffeyes

Kenneth S, 2007) or near full capacity (Gold Russell and Davis Ann, 2007). Global consumption of oil rose from 30 billion barrels (4.8×10^9 m^3) in 2004 to 31 billion in 2005 (Wikipedia, 2008).

Many series of unstable geopolitical issues and wars occur around the world. These including the collapse of World Trade Centre (WTC) on US on September 11, 2001; the war in Iraq; the crisis between Israel and Lebanon; the nuclear brinkmanship between US and Iran and other unstable geopolitical issues. It is not refused also that the rising of oil prices is due to the speculative activities from someone party(s) (Hassan Marican, 2008).

1.1.2 Green House Gasses Increasing

The increase in the concentration of carbon dioxide, one of the three major atmospheric contributes to the greenhouse effect has been carefully documented at the Mauna Loa Observatory in Hawaii. The 1990 rate of increase was about 0.4% per year. The interesting cyclic variations represent the reduction in carbon dioxide by photosynthesis during the growing season in the northern hemisphere. Those gas molecules in the Earth's atmosphere with three or more atoms are called "greenhouse gases" because they can capture outgoing infrared energy from the Earth, thereby warming the planet. The greenhouse gases include water vapor (H_2O), ozone (O_3), carbon dioxide (CO_2), and methane (CH_4). Also, trace quantities of chloro-fluoro-carbons (CFC's) can have a disproportionately large effect.

1.1.3 Petrol Is Much More Consumed Than Diesel

The consumption of petrol is much higher than consumption of diesel in Malaysia. This is because usually in Malaysia, consumer or citizens in Malaysia use more petrol than diesel like all their cars is petrol engine. On the other hand, petrol

demand in Malaysia is getting higher day by day. Related to fuel crisis that source of fuel (petrol, diesel, petroleum) is limited and the price of petrol is increasing. The choice between Petrol cars and Diesel cars is highly debated right from the inception of these two engine types.

1.2 Rational and Signification

The problems stated in section 1.1 above have led to an intensified search for viable alternative sources of energy global. This research can resolve the problem by synthesizing biopetrol from palmitic acid using catalytic cracking method. Biopetrol can be an alternative choice to the petrol from the fossil fuel. So, the dependent on petroleum uses can be reduced. It might be wise for Malaysia to adopt an implementation in order to use the renewable fuel resources. In this case, Malaysia exploits further utilization of its crude palm oil in automotive sector through research and development by authorities related with palm oil industry. The result is the engine oil and biodiesel. Today, the biodiesel production from palm oil in Malaysia has been established, industrialized in big scale and commercialized to Europe (Yusof B, 2006). However, the biodiesel used is limited for diesel-used vehicles only, so the same approach must be done for petrol-used vehicles by biopetrol.

Malaysia currently accounts for 51 % of world palm oil production and 62% of world exports, and therefore also for 8% and 22% of the worlds total production and exports of oils and fats. As the biggest producer and exporter of palm oil and palm oil products, Malaysia has an important role to play in fulfilling the growing global need for oils and fats in general (aseansources.com, 2008). So, Malaysia has big opportunity to produce biopetrol from palmitic acid because it has enough resources to produce in large scale. Palm oil can become the top choice of biofuel producers because world have plenty of palm oil.

Biopetrol is an environmental-friendly alternative liquid fuel. There has been renewal interest in the use of vegetable oils for synthesizing biopetrol due to its less polluting and renewable nature as against the conventional petroleum diesel fuel. The biggest difference between biofuels and petroleum feedstocks is oxygen content. Biofuels have oxygen levels from 10% to 45% while petroleum has essentially 0%, which makes the chemical properties of biofuels very different from petroleum. All fuels have very low sulfur levels and many of them have low nitrogen levels (Trabzon, 2007). The CO_2 reduction potential of biofuels is enormous in comparison with fossil fuels. Renowned institutes for example, the Institute for Energy and Environmental Research, Germany, have formulated clear statements on CO_2 reduction via biofuels. Taking into account all the energy expended in the cultivation, transport and processing of biodiesel, this reduction resulting from replacing a single liter of diesel with biodiesel amounts to 2.2 kg CO_2 (Biopetrol Forum, 2007). So the same effect to the biopetrol compared to the biodiesel. Biopetrol could be an answer to the future air emission control. Biopetrol also is the renewable energy based, which is not only environmental-friendly, but also economic to produce. Since using thermal cracking method is not producing much yields of biopetrol, so the catalytic cracking method is carried on to produce more yields of biopetrol. This method is more effective.

1.3 Objective

1. To synthesize biopetrol as alternative fuel for petrol-used vehicles from fatty acids.
2. To find and determine concentration of synthesized biopetrol obtained.

1.4 Scope

1. Conversion of fatty acids to form desired isooctane in biopetrol through catalytic cracking method.
2. Selection of various reaction movements or resolution of active catalyst for performing catalytic cracking method applied to the fatty acids.
3. Qualitative and quantitative analysis of biopetrol through gas chromatography analysis using mixture of hexane and pure isooctane as standard calibration.

CHAPTER 2

LITERATURE REVIEW

2.0 Definition of Fuel

A simple definition for fuel oil is a lighter type of oil, or a liquid byproduct of crude oil, which is used for energy, especially in regard to heating. When petroleum is refined, there are two main categories under which it is classified. One is distillate oils, which includes diesel, and the other is residual oils, which includes things like kerosene. Different types of fuel oil are classified under both categories. Distillate fuel oil is the type generally used for home heating. While gasoline is also a byproduct of the petroleum refining process, it is far less stable than the fuel oil used to heat homes and commercial properties. Even so, the rise and fall of gas prices is generally a good indicator of the stability, or lack thereof, of fuel oil prices. Since the price of crude oil directly affects the price of fuel oil, consumers should be prepared to pay more to heat their homes as the worldwide demand for oil and gas continues to increase.

2.1 Fuel Types by Period of Natural Renovation

2.1.1 Fossil fuels

Fossil fuels or mineral fuels are fossil source fuels, that is, hydrocarbons found within the top layer of the Earth's crust. They range from volatile materials with low carbon: hydrogen ratios like methane, to liquid petroleum to nonvolatile materials composed of almost pure carbon, like anthracite coal. Methane can be found in hydrocarbon fields, alone, associated with oil, or in the form of methane. Fossil fuels are non-renewable fuel sources that are found within the top layer of the Earth's crust. They were formed millions of years ago from the organic remains of prehistoric plants and animals. The dead plant and animal matter sank to the bottom of swamps and oceans and were covered by sand and rock, and as more rock weighed down on them they eventually squeezed out with water. The production and use of fossil fuels raise environmental concerns. A global movement toward the generation of renewable energy is therefore under way to help meet increased energy needs.

2.1.2 Biodiesel

Biodiesel is an alternative fuel derived from palm oil and can be used in compression ignition engines i.e. diesel engines without any modifications. It refers to methyl esters derived from palm oil through a process known as 'transesterification'. Biodiesel, however, is an alternative fuel to petroleum-based diesel, which can be manufactured from vegetable oils such as palm, rapeseed (canola), soy, linseed, jatropha and coconut oil. Biodiesel can also be manufactured from animal fats (including tallow) and used cooking oils. The use of Biodiesel does not require any major changes in the fuel distribution system, avoiding the need for expensive additional infrastructure. In many instances, Biodiesel is mixed with petroleum-based

diesel to create a Biodiesel blend, which can then be sold into the general diesel market.

2.1.3 Biofuel

Biofuel is defined as solid, liquid or gaseous fuel obtained from relatively recently lifeless or living biological material and is different from fossil fuels, which are derived from long dead biological material. Also, various plants and plant-derived materials are used for biofuel manufacturing. Globally, biofuels are most commonly used to power vehicles, heat homes, and for cooking. Biofuels is the best way of reducing the emission of the greenhouse gases. They can also be looked upon as a way of energy security which stands as an alternative of fossil fuels that are limited in availability. Theoretically, biofuel can be easily produced through any carbon source; making the photosynthetic plants the most commonly used material for production. Almost all types of materials derived from the plants are used for manufacturing biogas. One of the greatest problems that is being faced by the researchers in the field is how to covert the biomass energy into the liquid fuel.

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

2.1.4 Biopetrol from Palmitic Acid

Palmitic acid or hexadecanoic acid (CH_3 $(CH_2)_{14}COOH$) is a fatty acid which is found in animal fats and vegetable oils. To produce isooctane from palmitic acid, catalytic cracking process is needed. Catalyst is used and heat is supplied at palmitic acid's melting point within a range of 63 ^{0}C – 64 ^{0}C to melt the solid palmitic acid. After it turns to the liquid, the heating is continuous at isooctane's boiling point of 98 ^{0}C by using heating manner to form new arrangements of carbon compounds including isooctane. The sample produced will have lots of hydrocarbon chains because of the heat that breaks the carbon chain randomly. Although alkanes from C5 until C9 are categorized as gasoline, C8 will be the major component in this study.

2.1.5 Vegetable Oil and Biodiesel

Vegetable oil is used in several old diesel engines that have indirect injection systems. This oil is also used to create biodiesel, which when mixed with conventional diesel fuel is compatible for most diesel engines. Used vegetable oil is converted into biodiesel. Sometimes, water and particulates are separated from the used vegetable oil and then this is used as a fuel.

Its composition is just like mineral diesel. When biodiesel is mixed with mineral diesel, the mixture can be used in any diesel engine. It is observed that in several nations, the diesel engines under warranty are converted to 100% biodiesel use. It has also been proved that most people can run their vehicles on biodiesel without any problem. A large number of vehicle manufacturers recommend the use of 15% biodiesel mixed with mineral diesel. In Europe, a 5% biodiesel blend is generally used at gas stations.

2.1.6 Bioalcohol

Bioalcohols are biologically produced alcohols. Common among these are ethanol and rare among these are propanol and butanol. Biobutanol can be used directly in a gasoline engine and hence is considered a direct replacement for gasoline. The butanol can be burned straight in the existing gasoline engines without any alteration to the engine or car. It is also claimed that this butanol produces more energy. Also, butanol has a less corrosive effect and is less soluble in water than ethanol.

2.1.7 Bioethanol/Ethanol Fuel

Ethanol fuel is the most commonly used biofuel in the world and particularly in Brazil. Ethanol can be put to use in petrol engines as a substitute for gasoline. Also, it can be mixed with gasoline in any ratio. The contemporary automobile petrol engines can work on mixtures of gasoline and ethanol that have 15% bioethanol. This mixture of gasoline and ethanol has more quantity of octane. This indicates that the engine would burn hotter and more efficiently. In high altitude spots, the mixture of gasoline and ethanol is used as a winter oxidizer and thereby atmospheric pollution is decreased. The ethanol fuel has less British thermal unit energy content. Thus, to drive the same distance, more fuel is required. Also ethanol has a corrosive effect on combustion chambers, aluminum, rubber hoses and gaskets and fuel systems.

2.1.8 Biogas

Biogas is created when organic material is anaerobically digested by anaerobes. During production, there is a solid byproduct called digestate. This can be used as a biofuel or fertilizer. Biogas consists of methane. Landfill gas is created in

landfills due to natural anaerobic digestion and is a less clean form of biogas. Dried manure, charcoal and wood are examples of solid biofuels.

2.2 Catalytic Cracking

Cracking is the name given to breaking up large hydrocarbon molecules into smaller and more useful bits. This is achieved by using high pressures and temperatures without a catalyst, or lower temperatures and pressures in the presence of a catalyst.The source of the large hydrocarbon molecules is often the naphtha fraction or the gas oil fraction from the fractional distillation of crude oil (petroleum). These fractions are obtained from the distillation process as liquids, but are re-vaporised before cracking. The hydrocarbons are mixed with a very fine catalyst powder. These days the catalysts are zeolites (complex aluminosilicates) - these are more efficient than the older mixtures of aluminium oxide and silicon dioxide.The whole mixture is blown rather like a liquid through a reaction chamber at a temperature of about 500°C. Because the mixture behaves like a liquid, this is known as fluid catalytic cracking (or fluidised catalytic cracking).Although the mixture of gas and fine solid behaves as a liquid, this is nevertheless an example of heterogeneous catalysis - the catalyst is in a different phase from the reactants. The catalyst is recovered afterwards, and the cracked mixture is separated by cooling and further fractional distillation. There isn't any single unique reaction happening in the cracker. The hydrocarbon molecules are broken up in a fairly random way to produce mixtures of smaller hydrocarbons, some of which have carbon-carbon double bonds. One possible reaction involving the hydrocarbon $C_{15}H_{32}$ might be:

Figure 2.1: Reaction of hydrocarbon $C_{15}H_{32}$

Or, showing more clearly what happens to the various atoms and bonds:

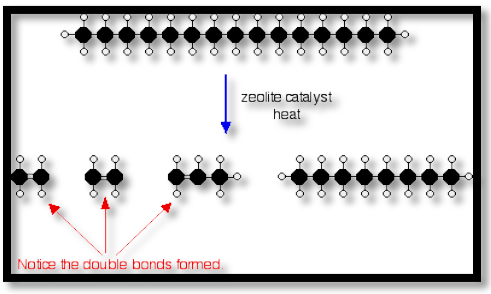

Figure 2.2: Reaction involving the Hydrocarbon $C_{15}H_{32}$

This is only one way in which this particular molecule might break up. The ethene and propene are important materials for making plastics or producing other organic chemicals. The octane is one of the molecules found in petrol (gasoline).

2.3 Chemicals

2.3.1 Palmitic Acid

Palmitic acid**,** also called hexadecanoic acid**,** is one of the most common saturated fatty acids found in animals and plants. It is a white solid that melts at 63.1°C and its chemical formula is CH_3 $(CH_2)_{14}COOH$. As its name tells us, it is found in palm oil but also in butter, cheese, milk and meat. Palmitate is a term for the salts or esters of palmitic acid. The palmitate anion is the observed form of palmitic acid at physiological pH. Palmitic acid is the first fatty acid produced during lipogenesis (fatty acid synthesis) and from which longer fatty acids can be produced. Palmitate negatively feeds back on acetyl-CoA carboxylase (ACC) which is responsible for converting acetyl-ACP to malonyl-ACP on the growing acyl chain, thus preventing further palmitate generation. Palmitate is an antioxidant and a vitamin A compound added to low fat milk to replace the vitamin content lost through the removal of milk fat. Palmitate is attached to the alcohol form of vitamin A, retinol, in order to make vitamin A stable in milk. Derivatives of palmitic acid were used in

World War II to produce napalm. Reduction of palmitic acid yields cetyl alcohol. Palmitic acid is the first fatty acid produced during lipogenesis and from which longer fatty acids can be produced.

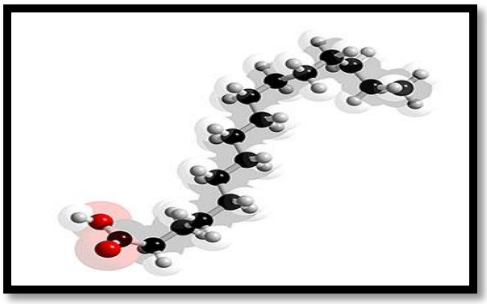

Figure 2.3: Palmitic acid structure

Figure 2.4: Skeleton structure of palmitic acid

Table 2.1: Physical and chemical properties of palmitic acid

Other names	Cetylic acid, hexadecylic acid, hexadecanoic acid
Appearance	White chips, crystal or powder
Molecular formula	$C_{16}H_{32}O_2$
Molecular weight	256.42 g/mol
Melting point ^{0}C	61 – 64°C
Boiling point ^{0}C	352°C
Density	0.853 g/cm^3 at 62 °C
Solubility	Insoluble in water

2.3.2 Isooctane

2,2,4-Trimethylpentane, also known as isooctane, is an octane isomer which defines the 100 point on the octane rating scale. It is an important component of

gasoline. Isooctane is targeted as product of biopetrol because petrol itself is dominated by isooctane, with small amount of heptane and a little presence of benzene. Isooctane is derived through isomerization of octane with certain conditions and the presence of catalyst, as performed in petroleum industries. The octane number used as petrol production's parameter to measure the tendency of petrol to auto-ignite and knock in petrol-used engines. Since the petrol consists mixture of isooctane and heptane, so the octane number is graded based on composition of both alkanes in petrol. The higher octane number determined represents the higher composition of isooctane in petrol, which gives low tendency to auto-ignite, little knock and smooth burning, which is assigned as petrol with high quality.

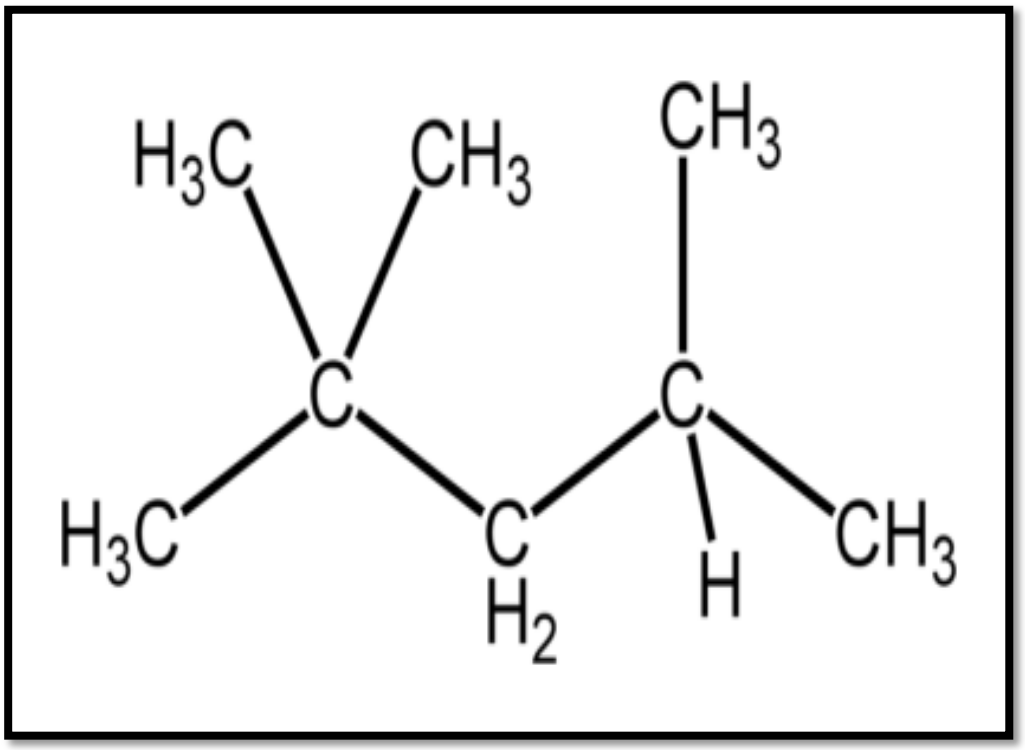

Figure 2.5: Structure of isooctane

Table 2.2: Physical and chemical properties of Isooctane

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

2.3.3 Octane Rating

The octane rating of a spark ignition engine fuel is the knock resistance (anti-knock rating) compared to a mixture of iso-octane (2,2,4-trimethylpentane, an isomer of octane) and n-heptane. By definition, iso-octane is assigned an octane rating of 100 and heptane is assigned an octane rating of zero. An 87-octane gasoline, for example, possesses the same anti-knock rating of a mixture of 87% (by volume) iso-octane and 13% (by volume) n-heptane. This does not mean, however, that the gasoline actually contains these hydrocarbons in these proportions. It simply means that it has the same autoignition resistance as the described mixture.

A high tendency to auto-ignite, or low octane rating, is undesirable in a spark ignition engine but desirable in a diesel engine. The standard for the combustion quality of diesel fuel is the cetane number. A diesel fuel with a high cetane number has a high tendency to auto-ignite, as is preferred.

It should be noted that octane rating does not relate to the energy content of the fuel, nor the speed at which the flame initiated by the spark plug propagates across the cylinder. It is only a measure of the fuel's resistance to auto-ignition. It is for this reason that one highly branched form, or isomer, of octane (2,2,4-trimethylpentane)