Biofuel production from Mesua ferrea L seed oil

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***Abstract*— Biofuels have become an attractive alternative fuel because of their possible environmental benefits and the current concern over the depletion of fossil fuel sources. Mesua ferrea L seed oil is potential feedstocks for production of biofuel available in northeastern (NE) region of India. Catalytic cracking is one of the most efficient methods to produce biofuel, especially biogasoline, by cracking of vegetable oil in the presence of suitable catalyst. The catalytic cracking of non-edible oils requires proper cracking catalysts and reactors for the production of biogasoline. The experiment shows that catalytic cracking of Mesua ferrea L seed oil can be done to get yield like biogasoline, gases and some quantity of coke. From the analysis, it is found that the optimum operating conditions for catalytic cracking process using 5g of heterogeneous alumina hydro silicate as catalyst are 450^{0}C temperature and 80 min of reaction time. This condition produces the highest yield of biogasoline fraction which is equivalent to 91.67 wt. %. Similarly optimum operating conditions for catalytic cracking process using 50g of heterogeneous sodium carbonate are 450^{0}C, 120 min of reaction time and at this operating condition 91.67 wt.%. of biogasoline fraction produces. It is confirmed by GC-MS analysis. The physical and the chemical properties of the Mesua ferrea oil are determined using ASTM method. Fractional distillation technique is used for the separation of mixtures of volatile components from Mesua ferrea L seed oil. It is seen from the experiment that catalytic cracking process takes less time then fractional distillation process in terms of producing yield i.e. biogasoline. Moreover, catalytic cracking process is performed in presence of catalysts and fractional distillation is completed in absence of it. Three types of products namely biogasoline, biodiesel and bioresidual oil are obtained by the fractional distillation process at three different temperatures. The product sample is then analyzed to characterize its properties by GC-MS and CHNO analyzer. The analyzed results of the product sample are methylthiane, pentylthiane, tritetracontane, heneicosane, benzopyran and methylbenzo.**

***Index Terms*— Catalytic cracking, Biocrude, Batch reactor, Fractional distillation.**

I. INTRODUCTION

The production of liquid biofuels from vegetable oils has been gaining popularity recently, because of environmental concerns and diminishing petroleum reserves. Bio-fuel is a non-polluting, locally available, accessible and reliable fuel obtained from renewable sources. There are several methods for the conversion of vegetable oils to biofuels, such as pyrolysis, fermentation, transesterification, thermal cracking, catalytic cracking, and hydrocracking [1] – [6].

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Pyrolysis is a thermochemical decomposition of organic material at elevated temperatures in the absence of oxygen. It involves the simultaneous change of chemical composition and physical phase. Fermentation is a metabolic process in which an organism converts a carbohydrate, such as starch or a sugar, into an alcohol or an acid. Transesterification is otherwise known as alcoholysis. It is the reaction of fat or oil with an alcohol to form esters and glycerin. A catalyst is used to improve the reaction rate and yield [7]. Catalytic cracking of triglyceride materials represents an alternative method of producing renewable bio-based products suitable for use in fuel and chemical applications. Catalytic cracking is one of the routes for obtaining biogasoline and other products from vegetable oils. Catalytic cracking breaks complex hydrocarbons into simpler molecules in order to increase the quality and quantity of lighter, more desirable products and decreases the amount of residuals. This process rearranges the molecular structure of hydrocarbon compounds to convert heavy hydrocarbon feedstock into lighter fractions [8] - [10]. Thermal cracking is a process in which hydrocarbons such as crude oil are subjected to high heat and temperature to break the molecular bonds and reduce the molecular weight of the substance being cracked. This process is used to extract usable components, known as fractions, which are released during the cracking process. It is one among several cracking methods used in the petroleum industry to process crude oil and other petroleum products for commercial use. Hydrocracking is process by which the hydrocarbon molecules of petroleum are broken into simpler molecules, as of gasoline or kerosene, by the addition of hydrogen under high pressure and in the presence of a catalyst.

Among these methods, the catalytic cracking of vegetable oils to produce biofuels has been widely studied by many researchers all over the world [11]- [14].

The catalytic cracking process shows several clear advantages in comparison with the pyrolysis process. Firstly, the temperature of catalytic cracking process (450˚C) is lower than pyrolysis process (500–850˚C). The quality of product derived from pyrolysis is strongly dependent on the type of feedstocks used. Production of ethanol via fermentation requires necessary pretreatment of feedstocks such as saccharification and hydrolysis. In addition, fermentation requires much longer reaction time than the reaction time required for catalytic cracking [15]. Transesterification is only used for production of biodiesel whereas catalytic cracking can be applied for the production of kerosene, gasoline and diesel. Catalytic cracking has significant advantages over transesterification such as lower processing costs, compatibility with infrastructure, engines and fuel standards, and feedstock flexibility. More importantly, the final product is similar to diesel fuel. In thermal cracking, hydrocarbons are heated to 750-900˚C in the absence of oxygen. On the other hand, hydrocarbons are heated to the comparatively low temperature of 450-600˚C in the presence of a catalyst in catalytic cracking [16].

The objective of this paper is to study the catalytic cracking of Mesua ferrea L seed oil in presence of known catalysts and compare the cracking process with fractional distillation process using Masua ferrea L seeds oil as feedstocks.

II. FEEDSTOCKS

Mesua ferrea L. is an evergreen tree grows naturally in the northeastern part of the India. A normal 15 to 20-year-old tree produces in an average of 30 kg of seeds. It is estimated that 5500 tons of mesua ferrea L. seeds are annually available from Lakhimpur and sibsagar districts of Assam.The seed contains about 55-57 wt % nonedible, reddish-brown-colored oil which had been traditionally used as a fuel. The mesua ferrea L. tree has many medicinal properties. The fresh as well as the dried flowers of the mesua ferrea L. are used as a remedy for dysentery, itching, nausea and other ailments.

III. EXPERIMENTAL

A. Materials

About 50 kg of Mesua ferrea seeds were collected from forests of Assam and dried at 50˚C in a hot air oven. The dried seeds were shelled and milled. The oil was extracted from the milled kernels using the soxhlet extraction method. The solvent was removed from the oil content with the help of rotary vacuum evaporator and was found to be 75% by weight of milled kernel. About 5 litres of oil were extracted and filtered to remove the husk or peel present in it. The filtered Mesua ferrea oil was then stored in glass bottles for further experiments.

B. Methods

1) Physical and Chemical Characteristics

Fatty acid composition and different properties of mesua ferrea seed L oil such as kinematic viscosity, density, specific gravity, calorific value, acid value, pour point, cloud point, peroxide number and ash content were investigated in the present study. Fatty acid composition of the oil was determined by gas chromatography technique using standard procedure (AOCS Official Method 1998) at Indian Institute of Technology Delhi, New Delhi, India. Other properties of mesua ferrea seed L oil viz. kinematic viscosity, density, calorific value, acid value, flash point, pour point and carbon residue were determined as per ASTM standards at Tezpur University, Tezpur, Assam.

2) CHNO Analysis of Mesua Ferrea Seed L Oil

CHNO analysis gives the percentage of carbon, hydrogen and nitrogen by mass present in the molecular structure of a molecule. Carbon and hydrogen were determined using CHNS-O rapid analyzer (Haraeus Model No. 50002651). Nitrogen was quantified using Kjhaldhal method prescribed in IS: 1350 part (5); section 2. These tests were conducted at Department of Chemical Science, Tezpur University, Assam.

3) Catalytic Cracking

Catalytic cracking is most important and widely used refinery process for converting heavy gas oils into more valuable gasoline and lighter products. Catalytic cracking was done in a batch reactor at North East Institute of Science and Technology (NEIST), Jorhat, Assam. The filtered mesua ferrea seed L oil is put in a batch reactor by adding catalyst in it. In this study, Alumina hydro Silicates (A) and Sodium Carbonate (B) are used as catalysts. The hot mesua ferrea seed oil feeds at 260- 425°C which is contacted with the catalyst in the batch reactor. Average reactor temperature is in the range of 480 to 540°C. The cracking process produces carbonaceous material (referred to as catalyst coke) which remains on the catalyst. Since the cracking reactions produce some carbonaceous material that deposits on the catalyst and very quickly reduces the catalyst reactivity, the catalyst is regenerated by burning off the deposited coke with air blown into the regenerator. The regenerator operates at a temperature of about 715 °C. The combustion of the coke is exothermic and it produces a large amount of heat that is partially absorbed by the regenerated catalyst and provides the heat required for the vaporization of the feedstock (mesua ferrea seed L oil) and the endothermic cracking reactions take place.

4) *Fractional Distillation Process*

Fractional distillation is done in biofuel laboratory of Department of Energy, Tezpur University, Assam. Fractional distillation technique is used for the separation of mixtures of volatile components. It is used to separate compounds that have boiling points closer to each other. In a fractional distillation, mesua ferrea seed L oil is put into the round bottomed flask and the fractionating column is fitted into the top. The fractional distillation column is set up with the heat source at the bottom on the still pot. As the distance from the still pot increases, a temperature gradient or temperature difference is formed in the column; it is coolest at top and hottest at the bottom. As the vapor moves in upward direction, some of the vapor condenses and revaporizes. Each time the vapor condenses and vaporizes, the composition of the more volatile component in the vapor increases. This distills the vapor along the length of the column, and eventually the vapor is composed solely of the more volatile component. The vapor condenses on the glass platforms inside the column, and runs back down into the liquid below, refluxing distillate. The most volatile component of the mixture exits as a gas at the top of the column. The vapor at the top of the column then passes into the condenser, which cools it down until it liquefies.

IV. RESULTS & DISCUSSION

A. Physical and Chemical characteristics

The fatty acid profile of Mesua ferrea seed L oil is presented in Table I. Mesua ferrea seed L oil consists of 27.13% saturation comprising of palmitic, stearic and myristic acids and 72.67% unsaturation comprising mainly of oleic, arachidic and linoleic acids.It is reported that increasing unsaturation level in oil and decreasing chain length lead to decrease of viscosity, melting point, cetane number and calorific value of oil [17] – [19].

Vegetable oil is characterized by its density, viscosity, calorific value, cloud and pour points, acid value and peroxide number. The characteristics or physico-chemical properties of Mesua ferrea seed L oil obtained in the present investigation in terms of these parameters are presented in Table II along with the results reported by earlier investigators. The results pertaining to density, viscosity, peroxide number, pour point and cloud point are discussed below [20], [21].

I: Fatty acid profile of mesua ferrea seeds L oil

Sl. No	Fatty acid	Wt (%)
1	Myristic acid (C14:0)	2.10
2	Palmitic acid (C16:0)	10.85
3	Stearic acid (C18:0)	14.18
4	Oleic acid(C18:1)	55.90
5	Linoleic acid(C18:2)	13.85
6	Arachidic acid(C20:1)	2.92

II: Physio-chemical properties of Mesua ferrea seed L oil

Properties	Test Method	Measured Values	Values obtained from literature
Density at 15˚C ,kg/ m^3	ASTM D 287	930	930[20]
Kinematic viscosity at 40˚C, cSt	ASTM D 445	26.20	26.0[20]
Calorific value, MJ/kg	ASTM D240	39.56	39.84[20]
Pour point, ˚C	ASTM D 97	0	-1.3, -1.2, -1.0, -1.1[21]
Cloud point, ˚C	ASTM D 2500	5	5.8, 6.0, 6.0, 5.5[21]
Acid value, mg KOH/g	ASTM D 664	16.2	16.40[20], 11.87, 9.64, 10.38, 11.13[21]
PON, meq/kg	ASTM D 1832	5.21	3.58, 3.60, 3.64, 3.62[21]

Calorific value (CV) - The standard measure of the energy content of a fuel is its heating value (HV), sometimes called the calorific value or heat of combustion. The heating value is obtained by the complete combustion of a unit quantity of solid fuel in an oxygen-bomb colorimeter under carefully defined conditions. The gross heat of combustion or higher heating value (GHC or HHV) is obtained by oxygen-bomb colorimeter method as the latent heat of moisture in the combustion products is recovered. The higher heating value is one of the most important properties of a fuel. The higher heating value of a fuel increases with increasing carbon number in fuel molecules and also increases as the ratio of carbon and hydrogen to oxygen and nitrogen increases. Calorific value increases with chain length and decreases with an increasing unsaturation. The calorific value or calorific content is the energy content of the oil. Fuels with more unsaturation generally have lower energy (on a weight basis) while fuels with greater saturation have higher energy content. Denser fuels provide greater energy per gallon and since fuel is sold volumetrically, the higher the density, greater the potential energy[22]. From Table II, it is seen that the CV of Mesua ferrea seed L oil (measured) is 39.56 MJ/kg which is almost similar to the literature value.

Kinematic Viscosity- Viscosity is a measure of the resistance offered by a fluid to flow. Viscosity may be considered the integral of the interaction forces of molecules. When heat is applied to fluids, molecules can then slide over each other more easily making the liquid to become less viscous[23]. The vegetable oils are all extremely viscous. The oil viscosity generally increases with concentration of saturated fatty acids, and decreases with polyunsaturated content [24]. Oil viscosity decreases nonlinearly with temperature [24]. The measured value of kinematic viscosity of the oil is 26.2 cSt which is almost similar to the literature value seen in the table II.

Density- Density is an important physical characteristic of any substance, and is a measure of the mass per unit of volume of that substance. The density of a vegetable oil depends on its molecular weight, free fatty acid content, and temperature [24], [25]. It is an accepted fact that vegetable oil density decreases linearly with increasing temperature. The fatty acid composition of biodiesel fuels affects their physical properties including density. It has been reported that the highly unsaturated fatty acid methyl ester content in biodiesel fuels lowers their density [26]. The measured value is same with the literature value for the same oil (table II).

Pour point & Cloud point- Two important parameters for low temperature applications of a fuel are cloud point (CP) and pour point (PP). The CP is the temperature at which a sample of the fuel starts to appear cloudy, indicating that wax crystals have begun to form. At even lower temperatures, diesel fuel becomes a gel that cannot be pumped. The PP is the temperature below which the fuel will not flow. Saturated fatty compounds have significantly higher melting points than unsaturated fatty compounds and in a mixture they crystallize at higher temperature than the unsaturated. Thus fats or oils with high concentration of saturated fatty compounds will display higher cloud points and pour points. The measured value of pour point is 0°C whereas measured value of cloud pt is 5°C which are close to the literature values for the same oil (table II).

Peroxide number (PON) – Peroxide number is determined in milliequivalents of peroxide per kilogram of fuel. Peroxide number indicates the content of hydroperoxides, which triggers oxidation process [27]. This parameter influences cetane number (CN). Increasing PON increases CN, an effect that may reduce ignition delay time [28].The PON of the oil is 5.21 meq/kg which is close to the literature values(table II).

Acid value- Acid value (AV) is an important indicator of vegetable oil quality. AV is expressed as the amount of KOH (in milligrams) necessary to neutralize free fatty acids contained in 1g of oil. Under unsuitable conditions of treatment and preservation, vegetable oils, mainly consisting of unsaturated fatty acids, tend to decompose slowly in contact with the atmosphere or lipidases and release their fatty acid constituents, namely free fatty acids (FFA), which are extremely susceptible to oxidation, leading to the typical unpleasant smell and taste. Therefore, the acid value (AV) is one of the most frequently determined quality indicators during vegetable oil production, storage and marketing [29], [30]. The acid value of the oil is 16.4 mg KOH/g which is close to the literature values (table II).

A. CHNO Analysis of Mesua Ferrea Seed L Oil

The CHNO analysis is performed at Department of Chemical Science, Tezpur University, Assam, India for Mesua ferrea seed L oil to reveal its molecular formula. The chemical composition of the oil sample, mainly carbon (C), hydrogen (H), nitrogen (N) and oxygen (O) is determined by CHN analyzer. It is found that percentage of C, H, N, and O is 75.36%, 11.10%, 0.01%, 12.36% respectively. The molecular formula for Mesua ferrea seed L oil is found to be $C_{15}H_{29}O_2$.

B. Catalytic Cracking

Catalytic cracking process(experiment II) is done in presence of alumina hydro silicates (catalyst A) at an operating temperature 450 °C, residence time 80 minutes and catalyst-to-oil ratio of $1gg^{-1}$ and obtains different products namely bio oil, light hydrocarbon gas and coke. Similarly catalytic cracking process (experiment I) is also done with sodium carbonate (catalyst B) at an operating temperature 450 °C, residence time 120 minutes and a catalyst-to-oil ratio of $1gg^{-1}$ and products are obtained namely bio oil, hydrocarbon gas and coke. 53.2% bio oil is obtained using catalyst A whereas with the help of catalyst B, 56.5% bio oil is produced. It can be seen from table 3 that the highest gasoline fraction yield of 38.4 wt% is achieved with catalyst B and 36.1 wt% is achieved with catalyst A.

III: Catalytic cracking using sodium carbonate and alumina hydro silicates as catalysts

Feed	Mesua ferrea seeds L	
Catalyst	Sodium Carbonate	Alumina hydro Silicates
Conversion (%)	83.6	79.5
organic liquid product (OLP)	56.5	53.2
Gasoline Fraction	38.4	36.1
Kerosene Fraction	12.5	12.0
Diesel Fraction	5.6	5.1
Gaseous Product (wt %)	20.0	20.1

D. GC-MS Analysis of The Product of Catalytic Cracking

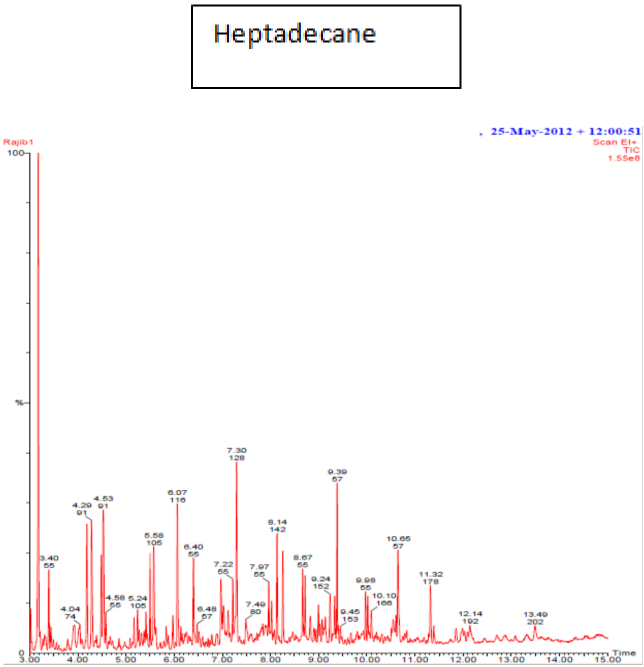

Fig 1: GC-MS analysis of Bio oil
(Using Alumina Hydro Silicates catalyst)

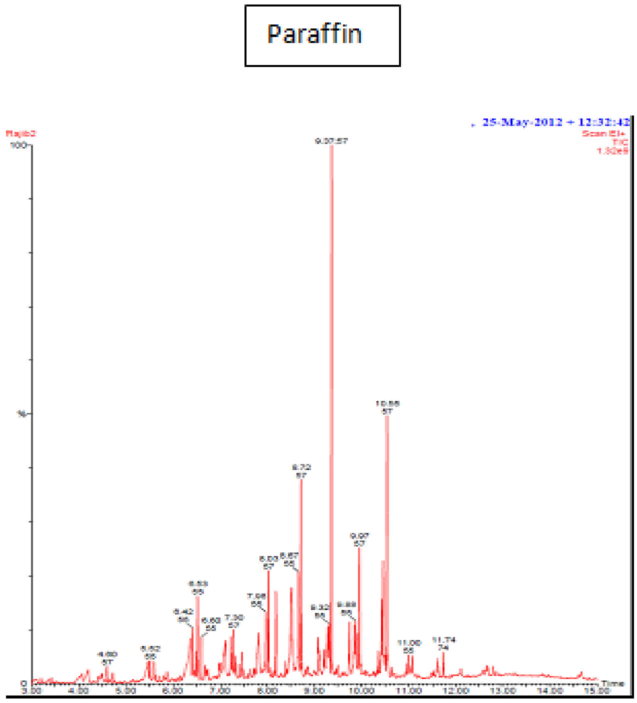

Fig 2: GC-MS analysis of Bio oil
(Using Sodium Carbonate catalyst)

It is seen from Fig 1 & Fig 2 that the main product obtained from catalytic cracking is biogasoline which is similar to the refinery gasoline. Comparison of biogasoline with refinery gasoline is presented in the table IV. The other components present in the bio oil as analyzed with the help of the GC-MS are toluene, paraffin, phenyl ethyl alcohol, heptadecane, benzene acetaldehyde, heneicosane and lighter hydrocarbon.

IV: The comparison of physio-chemical properties of biogasoline with refinery gasoline

Properties	Test Methods	Biogasoline	Refinery Gasoline
Density gm/cc	ASTM D 287	0.719	0.75
Sp.Gravity	ASTM D4052	0.71	0.71-0.77
Calorific value MJ/kg	ASTM D240	46.5	47.3

E.GC-MS Analysis of The Product of Fractional Distillation

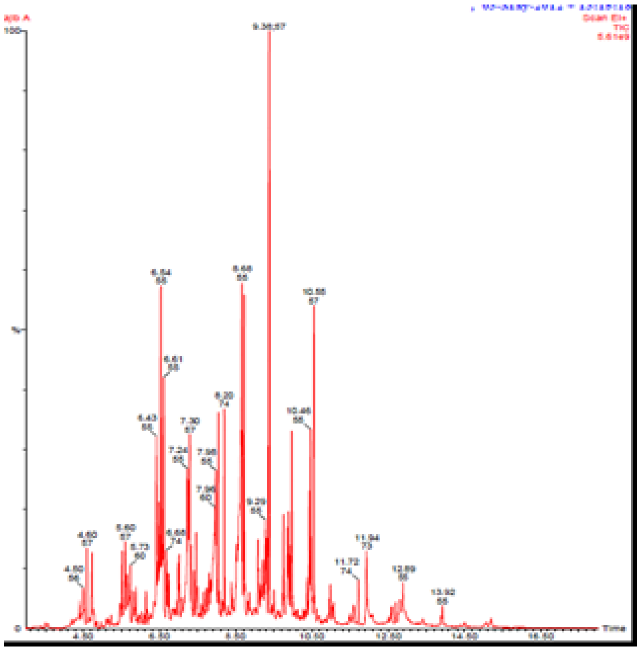

FIG 3: GC-MS ANALYSIS OF MESUA FERREA L SEED OIL

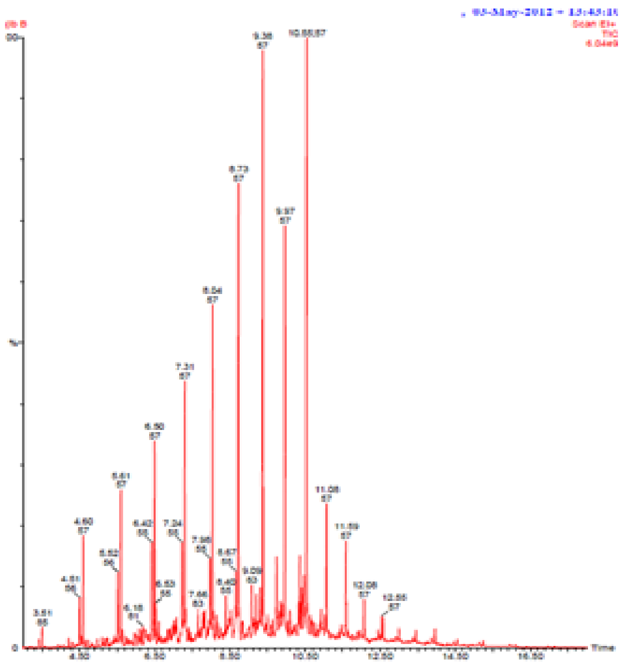

Fig 4: GC-MS analysis of Mesua Ferrea L seed oil

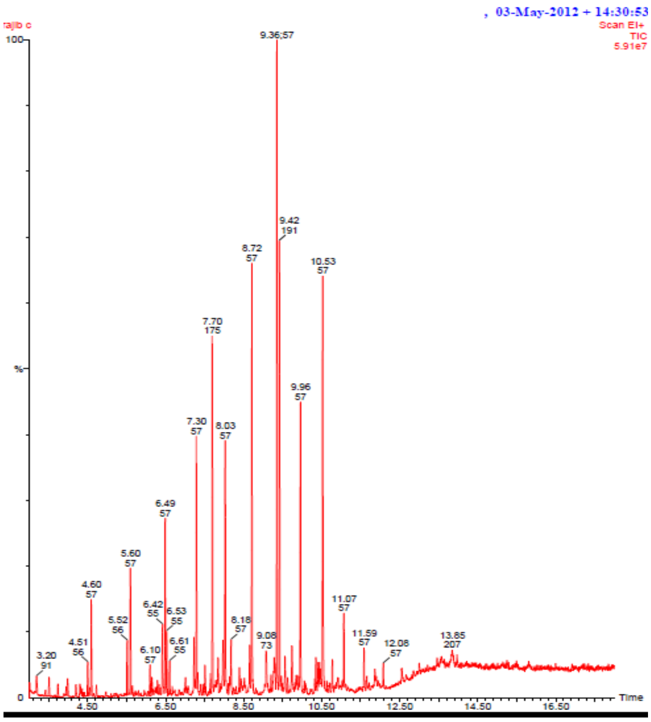

Fig 5: GC-MS analysis of Mesua Ferrea L seed oil

Fig.3 shows that the product obtained from fractional distillation is heavy aromatic hydrocarbon oil namely petrol whose operating temperature is 50°C and calorific value is 47.9 MJ/kg. The major components present in the product as analyzed through GC-MS are methylthiane, pentylthiane.

Fig.4 shows that the product obtained from fractional distillation is light aromatic hydrocarbon oil namely diesel whose operating temperature is 65°C and calorific value is 45.5 MJ/kg. The major components present in the product as analyzed through GC-MS are tritetracontane, heneicosane etc.

Fig.5 shows that the product obtained from fractional distillation is residual oil whose operating temperature is 85°C and calorific value is 43.2 MJ/kg. The major components present in the product as analyzed through GC-MS are benzopyran, methylbenzo etc.

V. CONCLUSION

Since the refinery gasoline obtained by the direct distillation process of crude petroleum is insufficient to meet the demand of the supply of present society so catalytic cracking process used to obtain biogasoline or biopetrol from Mesua Ferrea L seed oil is very useful for this society. Though fractional distillation technique is used for the separation of mixtures of volatile components from Mesua Ferrea L seed oil but it is seen from the experiments that catalytic cracking process takes less time then fractional distillation process in terms of producing yield i.e. biogasoline. Sodium carbonate catalyst (catalyst B) used in catalytic process shows better result in terms of biogasoline production than alumina hydro silicate catalyst (catalyst A). The density, calorific value and specific gravity of the biogasoline are found as 0.719 gm/cc, 46.5 MJ/kg and 0.71 respectively which are quite close to the refinery gasoline.

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