Parametric studies on Pyrolysis of pungam oil cake in electrically heated fluidized bed research reactor

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Abstract

Fluidized bed flash pyrolysis experiments have been conducted on a sample of pungam oil cake to determine particularly the effects of particle size, pyrolysis temperature and nitrogen gas flow rate on the products yield. The particle size, pyrolysis temperature and nitrogen gas flow rate were varied from 0.3 to 1.4 mm, 400°C to 550°C and 1.25 m³/ h to 2.4 m³/ h. The maximum oil yield of 54.8 wt % was obtained at a nitrogen gas flow rate of 2.0 m³/ h, particle size of 1.0-1.18 mm and pyrolysis temperature of 500°C. The maximum gas yield of 46.0 wt % was obtained at particle size of 0.3-0.6 mm, nitrogen flow rate of 1.75 m³/ h and at 500°C. The maximum char yield of 14.1 wt % was obtained at 400°C, particle size of 1.18-1.4 mm, and at nitrogen gas flow rate of 1.25 m³/ h. The gross calorific value of pyrolysis oil was found to be 37.45 MJ/Kg. The calorific value of pungam oil cake was found to be 14.3 MJ/Kg. The gas fractions were obtained using gas chromatography. The pyrolysis gas fractions were found to contain hydrogen, methane, nitrogen, oxygen, carbon dioxide and carbon monoxide. The liquid may be used as a source of low-grade fuel or it may be upgraded to higher quality liquid fuel by thermo catalytic cracking or trans-esterification processes.

Key words: pungam oil cake, fluidized bed reactor, pyrolysis oil, and gas chromatography

Introduction

The diminishing reserves and apparent negative effects such as green house gases and acid rain of fossil fuel have led the world community to recognize the importance of renewable and cleaner energy in recent years. The use of biomass as an energy source is an issue of great importance, as it constitutes part of an alternative solution for the replacement of fossil fuels. Even though raw biomass has significantly less energy content than petroleum, it has certain other advantages compared to fossil fuels. First, biomass is the only renewable organic and most abundant resource. Second, biomass fixes carbon dioxide balance in the atmosphere by photosynthesis.

Biomass feedstock's, such as agricultural residues, wastes from the paper, forestry and food industries, municipal wastes and energy crops have attracted great attention as renewable energy sources available world-wide[1]. Because of the ease of production and supply advantages, certain crops have been adopted as renewable sources in the production of liquid fuels. Soybean and rape seed, sunflower, cotton, euphorbia species and safflower are among the most promising renewable sources that have already been studied from the pyrolysis parameters and fuel properties point of view. It is necessary to develop technologies which make possible the conversion of biomass to a more suitable form, such as solid, liquid or gas. The nature and characteristics of the feedstock's plays an important role in the design of a thermo chemical conversion system.

The recovery of energy from a renewable source like biomass involves chemical, biochemical and thermo chemical processes, depending on the nature of the source. The main advantages of using biomass are its negligible sulphur, nitrogen and metal content. The utilization of biomass for energy generation has lead to reduced carbon dioxide, sulphur dioxide emissions and importantly prevents the green house effect and acid rain. The net flow of carbon dioxide to the atmosphere, and thereby the global potential, is reduced when fossil fuels are replaced with sustainably produced biomass[2]. Consumption of

agricultural residues for energy production would also reduce the environmental damage.

Among the thermo chemical processes, pyrolysis has become an attractive alternative because of the ease of operation. Its suitability to produce fuel for energy production and as feedstock's for chemical industries, relatively few polluting emissions, carbon dioxide neutral cycle and ease of reproduction makes pyrolysis oil a favourable option. The proportion of gas, liquid and solid products depend very much on the pyrolysis technique used and on the reaction parameters. Depending on the operating conditions, the pyrolysis process can be divided into three sub classes such as conventional pyrolysis (carbonization), fast pyrolysis and flash pyrolysis. Slow pyrolysis processes are performed at a low-heating rate and a long residence time. The longer residence times can cause secondary cracking of the primary products reducing yield and adversely affecting bio oil properties. In additions, a low heating rate and long residence time may increase energy input[4,5]. All of these are not attractive for commercial application of liquid fuel production. The preferred technology for production of oily products is fast or flash pyrolysis at high temperatures with very short residence time. The pyrolysis oil from biomass waste was found to be highly oxygenated and complex, and chemically unstable. Thus, the liquid products still need to be upgraded by lowering the oxygen content and removing residues.

The solid char can be used as a fuel in the form of briquettes or as a char–oil/water slurry or it can be upgraded to activated carbon and used in purification processes. The gases generated have a low to medium heating value, but may contain sufficient energy to supply the energy requirements of a pyrolysis plant.

Pyrolytic oil has strategic value because, as a liquid with high calorific value, its handling, storage, transportation, and utilization are similar to that of oil. It can be upgraded to obtain light hydrocarbons for transport fuel. Furthermore, compared to most coal- and petroleum-derived fuels, biomass oils contain low levels of aromatics and sulfur.

Karanja tree (Pongamia glabra) is wonderful tree almost like neem tree. In south part of the indian peninsula the karanja oil / cake are also used same like neem oil and neem cake. This Can be considered same like neem oil and neem cake in pest and fertilizer management in organically.

Pongamia pinnata (Karanja), grows extensively across India. The seed oil has been used by the natives of India for hundreds of years. It is used in Ayuvedic and Siddha traditional medicine systems to treat various skin conditions from eczema and psoriasis to leprosy, and for skin protection, as it is believed to enhance the UV absorbing properties of conventional sunscreens. A paste made of Karanja and lime is used to relieve rheumatic pain, and Karanja infused baths are used for cleaning skin ulcers and sores.

Pharmaceutical preparations based on Karanja Oil are used for treating skin diseases including acne, herpes, rosacia and leucoderma (partial or total loss of skin pigmentation, often occurring in patches, also known as vitiligo.). Roots of the tree are used for cleaning gums, teeth, and ulcers. Bark is used internally for bleeding piles. Juices from the plant, as well as the oil, are antiseptic. Karanj seeds yield bitter, non-edible (24-40%) oil with odd odour. This oil is used for illumination, leather dressing, soap making, lubrication and in other related purposes. This oil possesses insecticidal properties. The oil is also used as medicinal oil, both internally and externally. The cake left after extraction of oil from Karanja, is excellent organic manure with insect controlling properties. Parker Karanja Cake having minimum karanjan content of 1800 ppm and provides the crop with essential nutrients, while significantly reducing the number of soil insects and nematodes.

Materials

Prior to use, the sample (pungam oil cake) was air dried and ground in a ball mill and then screened to give fractions 0.3-0.6, 0.71-1.0, 1.0-1.18 and 1.18-1.4 mm in size. The values for volatiles, fixed carbon, ash, moisture (wt % as-received), elemental

composition (dry, ash-free basis) and calorific value (MJ/kg) were determined for pungam oil cake. The main characteristics of the pungam oil cake are given in table 1. Table.2 shows the Specification for Karanja Cake table.3 shows the Specification for Karanja oil.

Table-1: The main characteristics of pungam oil Cake

Proximate analysis	Composition
Moisture	08.12%
Ash	04.40%
Volatile Matter	70.00%
Fixed Carbon	17.48%
Ultimate Analysis	**Composition**
Carbon	56.71%
Hydrogen	05.30%
Oxygen	33.70%
Nitrogen	04.29%
Gross calorific Value	14.3 MJ/Kg

Table-2: Specification for Karanja Cake

Nitrogen	4.0%
Phosphorous 205	0.9%
Potassium 20	1.3%
Sulphar	Nil
Oil Content	5%

Methods

Pyrolysis reactor: Fig 1 shows the layout of an electrically heated fluidized bed reactor. The pyrolysis experiments were conducted in a tubular reactor under nitrogen gas atmosphere. The reactor was heated electrically using 230 V AC power supply through an autotransformer. The temperature was measured with the help of thermocouples, located at five different points, in the reactor bed. Nitrogen gas was used as the fluidizing gas and flow rate was measured with the help of a rotameter. The fluidizing gas velocity was maintained greater than the minimum fluidization velocity. The particle size of sand used for all the experiments were 0.3-0.6 mm. The amount of sand used was 500 g. The amount of pungam oil cake used was 600 g.

The experiments were carried out in three steps. The first one was to determine the effect of pyrolysis temperature on product yields. The temperatures used for this study were 400^oC, 450^oC, 500^oC and 550^oC. When the reactor reached the selected pyrolysis temperature, a screw feeder was used to feed the sample at the rate of 30 g/ min.

Table-3: Specification for Karanja Oil

S.No.	Parameters	Result
1	Karanjan Content	min 20,000 ppm
2	Specific Gravity	0.925 - 0.940
3	Refractive Index	1.4734 - 1.4790
4	Acid Value	20
5	Saponification Value	186 - 196
6	Iodine Value	80 - 90
7	Unsaponificable Matter	3.0
8	Organic Matter	80 - 90%
9	**Fatty acid composition**	**in %**
i	- Palmitic Acid	3.7 - 7.9
ii	- Stearic Acid	2.4 - 8.9
iii	- Arachidic Acid	2.2 - 4.7
iv	- Behanic Acid	4.2 - 5.3
v	- Lignoceric Acid	1.1 - 3.5
vi	- Oleic Acid	44.5 - 71.3
vii	- Linoleic Acid	10.8 - 18.3
viii	- Eicosenoic Acid	9.5 - 12.4

The other two steps of experiment were performed to establish the effect of particle size and the effect of sweep gas (nitrogen) velocity on the pyrolysis yields. These experiments were conducted using four different particle sizes of ranges 0.3-0.6, 0.71-1.0, 1.0-1.18 and 1.18-1.4 mm and sweep gas flow rates of 1.25, 1.75, 2.0 and 2.4 m^3/ h. Totally 64 runs were conducted by varying the process parameters such as temperature, particle size, and nitrogen gas flow rates.

When heat was applied externally thermal degradation takes place in the absence of air. The pyrolysis vapours were allowed to pass through a cyclone separator. The particulate matters were collected in a cyclone separator. Then the vapour was allowed to pass through a shell and tube condenser.

The vapour was condensed and collected in a flask. The liquid phase consisting of aqueous and oil phases were separated and weighed. The solid char was collected and weighed. Then the gas yield was calculated from the mass balance. The sample of non condensable gases were collected and analyzed by Gas chromatography. The oil used in this study was obtained under experimental condition that gives maximum oil yield. Prior to the study, the pyrolysis liquids were first decanted and then centrifuged for 15 minutes at about 2000 rpm, in order to separate an organic phase from aqueous phase and char traces.

Result and Discussion

Effect of Temperature on Product yield: Fig.2 shows the product yield of jatropha oil cake in relation to pyrolysis temperatures of 400, 450, 500 and 550ºC for the particle size of 1.0-1.18 mm under a sweep gas velocity of 2.0 m³/ h. The maximum oil yield of 54.8 wt% was obtained at 550ºC. The yield of oil increases from 39.4 wt % to 54.8 wt % when the pyrolysis temperature was increased from 400ºC to 550ºC. As shown in fig.2 the pyrolysis temperature affected the yield of the product significantly. In general, the yield increased to an optimum value with an increase in temperature and then decreased.

The operation also gives reduced liquid, char and increased gas yield at 550ºC. It is known that increasing the pyrolysis temperature increases carbon conversion to gas[6]. It may be concluded that secondary reactions of the liquid fraction of the volatiles and further decomposition of the char particles proceeded in the reactor with increasing pyrolysis temperatures. Similar behaviours with different biomass samples were also obtained in the literature[7,8,9].

At the pyrolysis temperature of 400ºC the decomposition was relatively slow and gas and char yield were 39.2 wt % and 10.2 wt %. At a higher pyrolysis temperature of 550ºC the oil yield decreased to 48.9 wt %. The char yield decreased when the pyrolysis temperature was increased. The char yield decreases by 8.3 wt % as the temperature

was raised from 400ºC to 550ºC. If these results are investigated with respect to maximum oil yield, it is clear that the optimum pyrolysis temperature is 500ºC under the experimental conditions used.

Water is formed by dehydration. With rising pyrolysis temperature, the water content of the liquids increases due to generation of water of reaction during decomposition of the jatropha oil cake. Literature shows that, the oil yield has reduced with the increase in pyrolysis temperature from 550ºC to 700ºC but the gas yield increases. Literature study shows that the reason why liquid product yields decreased at pyrolysis temperatures of 550ºC to 700ºC was the decrease of the organic and specific product and secondary volatiles decomposition at temperature above 500ºC[10,11].
Effect of Particle size on Product yield

The second set of experiments was performed to determine the effect of particle size on product yield. On varying the particle size from 0.3-1.18 mm with the sweep gas flow rate of 2.0 m³/ h, the yield of oil increases from 49.0 wt % to 54.8 wt %. For the particle size of 1.0-1.18 mm, the yield of oil obtained was 54.8 wt % .The char yield increases as the particle size increases[12]. This result suggested that mass and heat transfer restrictions had a profound influence at a larger particle size of 1.18-1.40 mm resulting in decrease in oil yield. The gas yield obtained was found at the level of 30.0 to 42.4 wt % for all the particle sizes investigated at nitrogen gas flow rate of 2.0 m³/ h and at 500°C. The effects of particle size on the yield of pyrolysis product are shown in fig.3.

Particle size is known to influence pyrolysis product yield[13]. Our experimental result show that particle size had significant effect on product yields. If the particle size is sufficiently small it can be heated uniformly which results in high oil yield. The maximum gas yield of 42.4 wt % was obtained at particle size of 0.71-1.00 mm. Reaction Completion time increases with increasing size of particle. The rate of heat transfer from outer to inner surface for large particle size is less[14]. The water contents in the pyrolysis oil resulting from the original moisture in

the feedstock and as a product of the dehydration reactions occurring during pyrolysis could be up to 11.0 wt % at nitrogen gas flow rate of 2.0 m³/ h, particle size of 0.3-0.6 mm and at 500°C.

Effect of Nitrogen gas flow rate on Product yield: Fig.4 shows the effect of nitrogen gas flow rate on the yield of pyrolysis products. The increase in nitrogen gas flow rate from 1.25 to 2.00 m³/ h increased the oil yield from 48.9 wt % to 54.8 wt %. The gas yield was decreased from 32.3 wt % to 30.0 wt %. The short residence time of the volatiles in the reactor as the sweep gas (nitrogen gas) velocity increased causes relatively minor secondary decomposition of higher molecular weight products[15,16]. At a pyrolysis temperature of 500°C, the maximum oil yield obtained was 54.8 wt % with particle size of 1.0-1.18 mm at a sweep gas flow rate of 2.00 m³/ h. The maximum gas yield of 32.6 wt % was obtained at a sweep gas flow rate of 2.4 m³/ h.

The char yield decreases as the nitrogen gas flow rate increases. It was observed that oil yield increased by 5.9 wt % upon increasing the nitrogen gas flow rate from 1.25 to 2.0 m³/ h. The char yield decreased by 2.5 wt % when reaching a nitrogen flow rate of 1.25 to 2.4 m³/ h. Our experimental results show that the yield of oil and non-condensable gases were affected by the nitrogen gas flow rate. The nitrogen gas flow rate had significant effect on the char and water yield also. As reported in the literature, the sweep gas (nitrogen gas) removed the volatiles from the pyrolysis environment [17,18]. Therefore, secondary reactions such as thermal cracking, re-polymerization and re-condensation were kept to a minimum for maximum liquid yield.

Analysis of pyrolysis oil: The properties of pyrolysis oil obtained using flash pyrolysis of pungam oil are given in table 4. The properties of pyrolysis oil were compared with diesel. The calorific value of diesel is 42.151 MJ/kg and that of pungam cake pyrolysis oil was found to be 37.45 MJ/kg. Calorific value of pyrolysis oil indicates that the energy content was close to diesel. The higher value of Conradson carbon residue indicates that

carbon deposits will occur in engine parts. Viscosity of fuel is an important parameter in diesel engines. In nature of pyrolysis oil makes it difficult to be handled by a standard diesel fuel pump and filter and the spray of fuel injected into the cylinder was affected, causing the formation of larger droplets; the result is reduced combustion efficiency and increased carbon accumulation inside the engine and on the injector.

In the pure form, pyrolysis oils are not suitable for use in modern diesel engines[19]. A reduction in viscosity greatly reduces engine operation problems.Thermo catalytic cracking and trans-esterification are the various techniques used to solve this problem related to high viscosity[20]. The high flash point suggested that the oil could be safely stored at room temperature but it leads to the starting problem of the engine. The presence of ash in the oil can cause erosion and corrosion problems[21]. The main source of ash in pyrolysis oil is the solid char particles carried over by pyrolysis vapors. The higher the value of acidity, the higher is the problem in the engine parts.

Analysis of pyrolysis gases: The gas fractions were obtained using gas chromatography. It can be observed that the concentration of combustible component in the gas increased as the particle size increased from 0.3-1.18 mm. The pyrolysis gas fraction at nitrogen gas flow rate of 1.25 m³/ h is shown in fig.5. The maximum percentage of hydrogen was 3.1 at nitrogen gas flow rate of 1.25 m³/ h. The maximum percentage of methane was 1.24 at nitrogen gas flow rate of 1.25 m³/ h.

Fig.6 shows the pyrolysis gas fractions at particle size of 0.3-0.6 mm. The maximum percentage of hydrogen was found to be 0.014 at particle size of 0.3-0.6 mm. The maximum percentage of methane was found to be 0.329 at particle size of 0.3-0.6 mm. The pyrolysis gas fraction at 400°C is shown in fig.7. The maximum percentage of hydrogen was 2.109 at 400°C. The maximum percentage of methane was 0.364 at 400°C.

Conclusions

In this study, pyrolysis experiments of pungam oil cake were performed in an electrically heated fluidized bed reactor in a nitrogen atmosphere. The maximum oil yield of 54.8 wt % was obtained at a pyrolysis temperature of 500°C, particle size of 1.0-1.18 mm and nitrogen gas flow rate of 2.0 m^3/ h. The yield of oil increases from 39.4 wt % to 54.8 wt % when the pyrolysis temperature was increased from 400ºC to 500ºC. It was observed that oil yield increased by 15.4 wt % upon increasing the temperature from 400ºC to 500ºC. The char yield decreases when the pyrolysis temperature was increased. The char yield decreased by 8.3 wt % as the temperature was raised from 400ºC to 550ºC.

The increase in nitrogen gas flow rate from 1.25 to 2.00 m³/ h increased the oil yield from 48.9 wt % to 54.8 wt %. The gas yield was decreased from 32.3 wt % to 30.0 wt %. The char yield decreases as the nitrogen gas flow rate was increased from 1.25 to 2.0 m^3 / h. It was observed that oil yield increased by 5.9 wt % upon increasing the nitrogen gas flow rate from 1.25 to 2.0 m^3 / h. The char yield decreased by 2.5 wt % when reaching a nitrogen flow rate of 1.25 to 2.4 m^3 / h. Our experimental results show that the yields of oil and non-condensable gases were affected by the nitrogen gas flow rate. The nitrogen gas flow rate had significant effect on the char and water yield also. The pyrolysis gas can be used as source of heat for reactor.

For the particle size of 1.0-1.18 mm, the yield of oil obtained was 54.8 wt %. The yield of oil increases from 49.0 wt % to 54.8 wt % when the particle size was increased from 0.3-0.6 mm to 1.0-1.18 mm. It was observed that oil yield increased by 5.8 wt % upon increasing the particle size from 0.3-0.6 mm to 1.0-1.18 mm. The char yield increases as the particle size increases. The char yield increases by 4.1 wt % as the particle size was increased from 0.3-0.6 mm to 1.18-1.4 mm. This result suggested that mass and heat transfer restrictions had a profound influence at a larger particle size of 1.18-1.40 mm resulting in decrease in oil yield. The gas yield obtained was found at the level of 30.0 to 42.4 wt % for all the particle sizes investigated at nitrogen gas flow rate of 2.0 m³/ h and at 500°C. The maximum gas yield of 42.4 wt % was obtained at particle size of 0.71-1.00 mm. Particle size is known to influence pyrolysis product yield. Our experimental results show that particle size had significant effect on product yield. If the particle size is sufficiently small it can be heated uniformly resulting in high oil yield.

The calorific value of pyrolysis oil was found to be 37.45 MJ/kg. The Calorific value of pyrolysis oil indicates that the energy content is close to diesel. Higher value of Conradson carbon residue indicates that carbon deposits will occur in engine parts. The liquid may be used as a source of low-grade fuel or it may be upgraded to higher quality liquid fuel by thermo catalytic cracking or trans-esterification processes. In the pure form pyrolysis oils are not suitable for use in modern diesel engines. A reduction in viscosity greatly reduces engine operation problems. Thermo catalytic cracking and trans-esterification are the techniques used to solve the problem related to high viscosity. The presence of carbon residue and ash are responsible for corrosion and forming the residue on the engine parts which will affect the engine life. These values should be as small as possible. The high flash point suggested that the oil could be safely stored at room temperature but it leads to the starting problem of the engine. Flash point is important from safety view point; this temperature should be as high as practical. Pour point is important for cold weather operation. For satisfactory working, the value should be well below freezing point of the oil used.

The pyrolysis gas fractions were analyzed by gas chromatography. The pyrolysis gas fractions were found to contain hydrogen, methane, nitrogen, oxygen, carbon dioxide and carbon monoxide.

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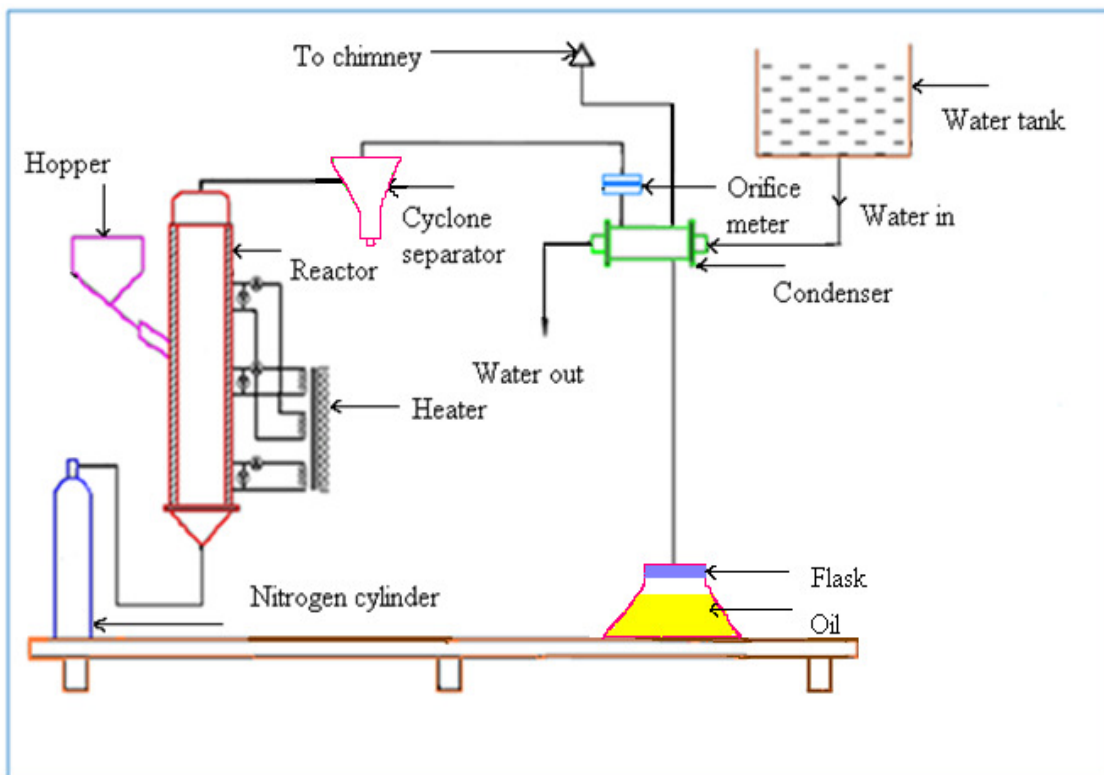
Fig-1: An Electrically heated fluidized bed reactor

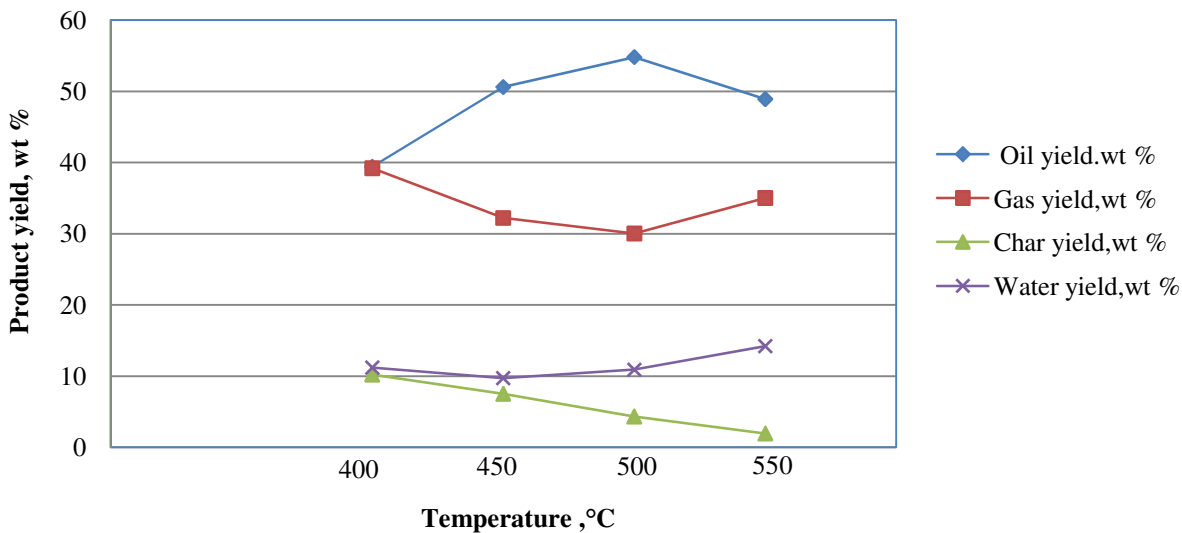**Fig-2: Temperature Vs Product yield**
(Particle size 1.0-1.18 mm and Nitrogen gas flow rate 2.00 m^3/h)
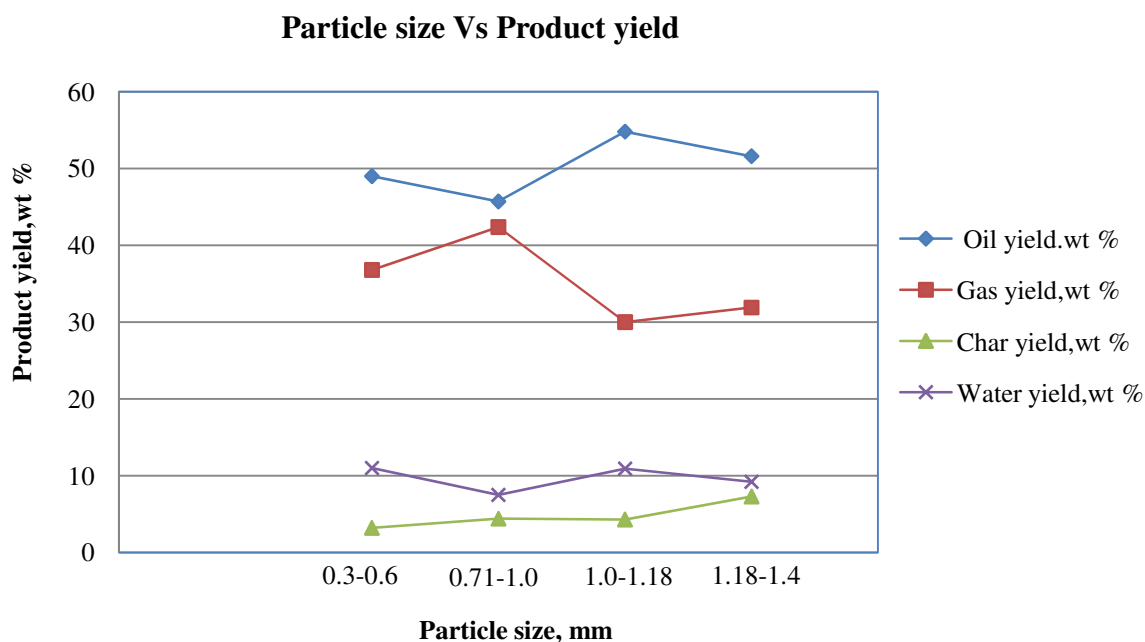
Particle size Vs Product yield

Fig-3: Particle size Vs Product yield
(Nitrogen gas flow rate : 2.00 m^3/h and Temperature: 500^0C)

Nitrogen gas flow rate Vs Product yield
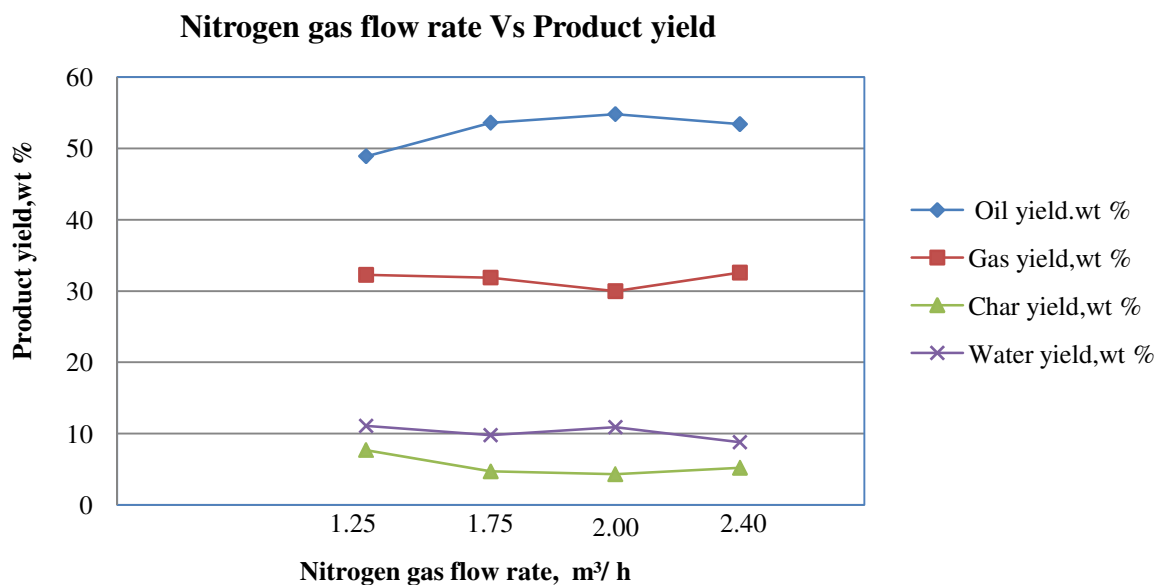
Fig-4: Nitrogen gas flow Vs Product yield
(Particle size 1.0-1.18 mm and Temperature: 500^0C)

Table-4: Comparison of properties of pyrolysis oil with diesel

Properties	Diesel	Pyrolysis oil
Calorific value	42.151 MJ/kg	37.45MJ/kg
Conradson carbon residue	0.30 %	15.32 %
Kinematic viscosity at 40ºC	2.0 to 4.5 cSt	7.8 cSt
Flash point	80ºC	135ºC
Pour point	2ºC	5ºC
Ash content	0.01 to 0.1 %	0.17 %
Acidity as mg of KOH/gm	0.20	89.34
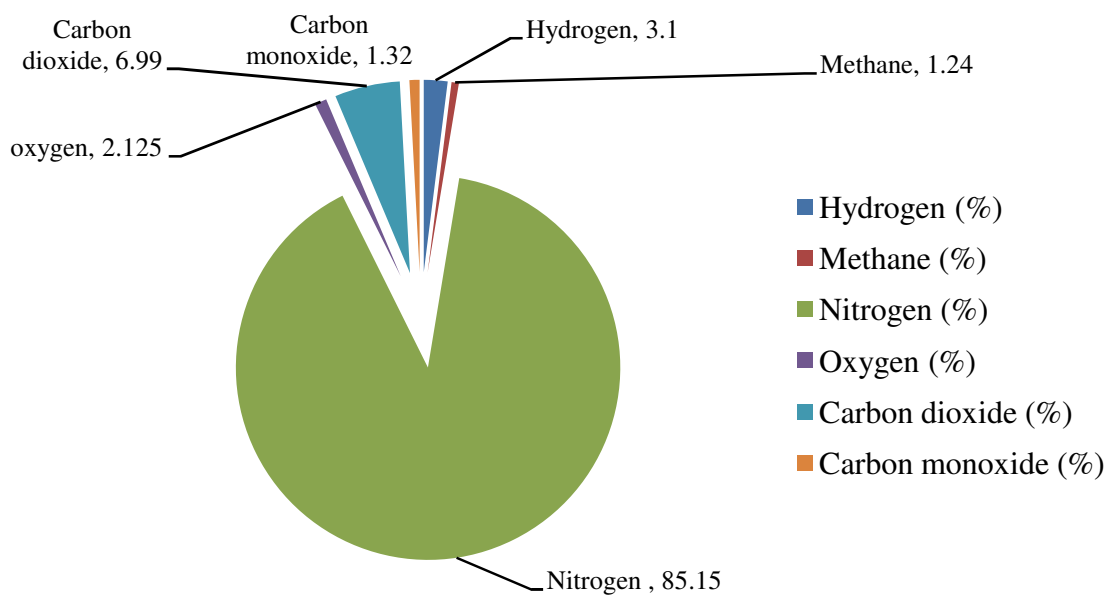
Figure-5: Pyrolysis gas fractions at nitrogen gas flow rate of 1.25m^3/h
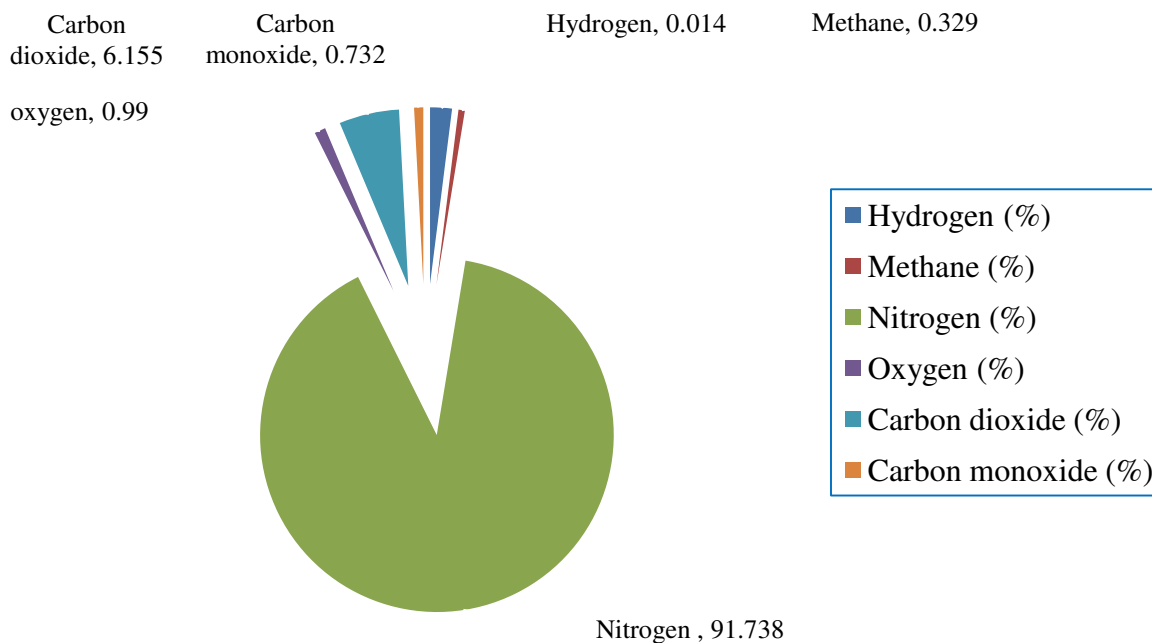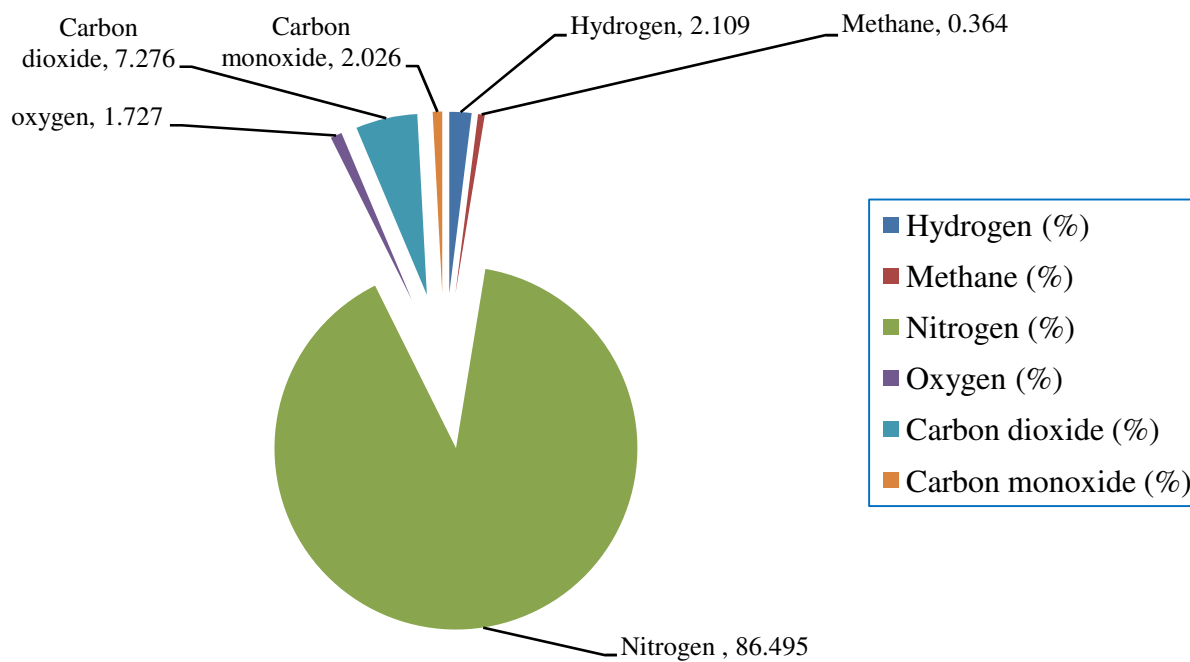
Figure-6: Pyrolysis gas fractions at Particle size 0.3-0.6mm

Figure-7: Pyrolysis gas fractions at Temperature 500^{0}C