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Production of Bio-Fuel from Neem Oil by Pyrolysis Method

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ABSTRACT
Green energy used to be the poster child of a fringe crowd, but not any longer. Today, average consumers are considering greener alternatives to meet their energy needs, whether for fueling their vehicles of heating their homes. But why make the switch? There are a number of reasons alternative fuels can be an excellent fit for your lifestyle, both in direct and indirect ways. With the trend toward greener living, the opportunity to make the switch to alternative fuels is greater than it has ever been .The contribution of neem as a contributor of biodiesel production will be of great importance in future. In India neem tree is a widely grown up termed as a divine tree due to its wide relevance in many areas of study. neem is a large tree growing about 25 m in height with semi-straight to straight trunk, 3 m in girth and spreading branches forming a broad crown, starts fruiting after 3-5 years. From the tenth year onwards it can produce up to 50 Kg of fruits annually. The tree has adaptability to a wide range of climatic, factors. It grows well in dry, stony shallow soils and even on soils having hard calcareous or clay pan, at a shallow depth .It grows on almost all types of soil including clayey, saline and alkaline soil, but does well on black cotton soils and deep well drained soil with good sub-soil water. This paper deals with Biodiesel production from neem oil by pyrolysis method. It has high lubricity, clean burning fuel and can be a fuel component for use in existing unmodified diesel engine. The fuel properties of biodiesel including flash point-and fire point were examined.
Key words: *Divine, Pyrolysis, Calcareous, Bio diesel, Emission, Saline*
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INTRODUCTION

Biodiesel, an alternative diesel fuel, is made from renewable biological sources such as vegetable oils and animal fats. It is biodegradable and non-toxic. It also has low emission profiles and so is environmentally beneficial. Importance of biodiesel increases due to Availability and renewability of biodiesel - Biodiesel is the only alternative fuel with the property that low concentration bio fuel–petroleum fuel blends will run well in unmodified conventional engines. It can be stored anywhere petroleum diesel fuel is stored. Biodiesel can be made from domestically produced, renewable oilseed crops such as soybean, rapeseed and sunflower. The risks of handling, transporting and storing biodiesel are much lower than those associated with petroleum diesel. Biodiesel is safe to handle and transport because it is as biodegradable as sugar and has a high flash point compared to petroleum diesel fuel. Biodiesel can be used alone or mixed in any ratio with petroleum diesel fuel. Lower emission from biodiesel - Biodiesel mainly emits carbon monoxide, carbon dioxide, oxides of nitrogen, sulfur oxides and smoke. Combustion of biodiesel alone provides over a 90% reduction in total unburned hydrocarbons (HC) and a75–90% reduction in polycyclic aromatic hydrocarbons (PAHs). Biodiesel further provides significant reductions in particulates and carbon monoxide over petroleum diesel fuel. Biodiesel provides a slight increase or decrease in nitrogen oxides depending on engine family and testing procedures [1-5]. Because biodiesel is made from renewable sources, it presents a convenient way to provide fuel while protecting the environment from unwanted emissions Biodegradability of biodiesel - Biodegradable fuels such as biodiesels have an expanding range of potential applications and are environmentally friendly. Therefore, there is growing interest in degradable diesel fuels that degrade more rapidly than conventional petroleum fuels. Biodiesel is non-toxic and degrades about four times faster than petroleum diesel. Its oxygen content improves the biodegradation process, leading to an increased level of quick biodegradation Different methods use for production of biodiesel Direct use/blending- Vegetable oil can be used directly as diesel fuel without any changes in the engine. The very first engine (by Rudolf Diesel) was tested using vegetable oil as fuel. The primary concern with vegetable oil as fuel is its high viscosity (atomization of vegetable oil is difficult) which leads to problem in long run as there is carbon deposits, coking and trumpet formation on injectors, thickening, gelling and oiling sticking. **Micro emulsions**- It is defined as colloidal dispersion of fluid microstructures (1-150 nm) in solvents forming two immiscible phases. The common solvent used is methanol and ethanol. Micro emulsion is a probable solution to high viscosity of vegetable oil. Their atomization is relatively easy due to lower viscosity.

Pyrolysis, strictly defined, is the conversion of one substance into another by means of heat or by heat with the aid of a catalyst . It involves heating in the absence of air or oxygen (Sonntag, 1979b) and cleavage of chemical bonds to yield small molecules. Chemistry involved in pyrolysis is difficult to characterize because of the variety of reaction paths and the variety of reaction products that may be obtained from the reactions that occur. The pyrolyzed material can be vegetable oil, animal fat, natural fatty acids or methyl esters of fatty acids. The pyrolysis of fats has been investigated for more than 100 years, especially in those areas of the world that lack deposits of petroleum. Many investigators have studied the pyrolysis of triglycerides to obtain products suitable for diesel engines [6-8].

MATERIALS AND METHODS

Materials

Here one non-edible seed has been used namely neem seed (also known as *Mellia azadirachta*) which was bought from a local fodder shop in Rourkela, Orissa, India. Both the seed were crushed using a household grinder, so that we could feed maximum seed, almost up to the capacity so that there would be minimum void and hence less oxygen for oxidation since pyrolysis is a process of heating substance in absence of oxygen. Below are the figures of neem seed.


Thermo gravimetric analysis Pyrolysis is heating of a substance in absence of air at a particular temperature. Therefore, the temperature for effective pyrolysis of the neem seed has to be determined. For this purpose, thermo-gravimetric analysis (TGA) of the sample cake was done using a DTG60 instrument. Around 20-30 milligrams of sample cake was taken and heated up to a final temperature of 800°C and a residence time of 1 minute at 800°C was allowed. TGA (Thermo-gravimetric weight) was performed in air atmospheres at a heating rate of 10°C/Min.

Extraction of Neem oil by Neem seeds:

Low tech method of extracting neem seed oil

There is a way to make your own neem seed oil. It will not give you high yields, and it will not be of the best quality. But it will give you some oil...Crush, grind, pound or otherwise smash up the seed kernels.Put them in a bowl or bucket and cover with water. The oil floats on top and can be skimmed off.

Making a watery neem seed extract

To make a strong enough extract with water you need about 500 g of seed kernels to 10 l of water. (Of course you can adjust the overall amount as required.)Crush, grind, pound or otherwise smash up the seed kernels. Put them in a cloth bag and suspend it over a big enough bucket. Add the water through the cloth bag and catch the extract in the bucket .Alternatively you could just put the bag inside a bucket or tub and steep it over night, but the above method will give a more powerful extract. The extract can be used as is. However if you want to use it for spraying you will need to filter it first or it will clog up your sprayer .This by the way is the most common method of using neem in agriculture in third word countries. 20 - 30 kg of kernels usually treat about one hectare. That means you need about 50 liters to treat 1000 m^2.

Alcohol Extraction

Azadirachtin and the other limonoids (the substances responsible for neem's insecticidal properties) are highly soluble in alcohol. Alcoholic extracts are over 50 times as concentrated as watery extracts .To make an alcoholic extract you soak the kernels in either ethanol of methanol and then strain and filter the brew.

The concentration of the resulting extract can vary greatly, from 0.2% to over 6% active ingredients .Unless you purify the crude extract, and adjust the concentration and the pH, the solution will not be stable. The active ingredients in neem usually decompose rapidly. I would say leave the oil pressing and alcoholic extraction to the big processors and buy pure neem seed oil and extracts, of known concentration, from reputable, organic neem product suppliers

Experimental setup and procedure

The setup consist of a semi-batch reactor in which the seeds are placed and that is then closed very tightly so as to avoid any leakage of gas as the result of pyrolysis. The outlet of the reactor is connected to a condenser (circulating water is the cooling medium) which condenses the gases/vapor coming of the outlet. Just at the other end of the condenser a measuring cylinder is placed where the gases being condensed is collected. The reactor is heated using an electric furnace. The temperature is controlled via PID controller. The seeds are fed into the reactor and it is closed very tightly using screw and bolt. Once the heating is started and after reaching a suitable temperature the reaction begins and the vapors that are released comes out of the reactor outlet which is connected to the condenser where the vapors are condensed and the collected in the test tube. Most of the non-condensable vapors are simply released. The product mainly consists of pyrolytic oil and water which then is separated based on density difference.

Experimental setup

Procedure

•Firstly approximately 25 gm of sample is taken and fed in the reactor.

•Then the desired temperature is set and the pyrolysis is started.

•Once the reaction begins the reaction time and the yield is noted down.

•This is done for the entire range of temperature with 50°C interval.

•Then the temperature is identified at which there is maximum yield.

•Rest of the extraction is carried out at this temperature.

Sample pyrolysis run

To determine the temperature at which there is maximum yield in the temperature range obtained by TGA, sample pyrolysis run were done. In the sample pyrolysis run, we took approx. 25 gm of the seed and pyrolysis was done at different temperature in that range at the interval of 50°C, to determine the temperature at which maximum yield of liquid product is obtained. During sample runs various data like reaction time, yield of char, and yield of liquid product were noted down. Variation in yield of char, liquid product and gas (volatiles) with respect to temperature is plotted. Variation in reaction time with temperature was also plotted.

Characterization of raw material and char

The neem seed and char of these seeds were analyzed in order to observe the change in the properties of the solid material as a resultof pyrolysis.

Proximate analysis

It provides information on moisture content, ash content, volatile matter content and fixed carbon content of the material. It was carried out using ASTM D3172 - 07a method.

Calorific value

Calorific value of a material is the amount of heat liberated when 1Kg of that material is burnt. It was determined for both seed and char using a bomb calorimeter.

Scanning electron microscopy

The surface of the char obtained was viewed under aScanning Electron Microscope (Model: JEOL-JSM-6480LV SEM) at different magnification values to have a clear view on pore density and size.

Physical characterization of bio-oil

Physical properties such as density, specific gravity, viscosity, conradson carbon, flash point, fire point, pour point, cloud point, calorific value, sulphur content, distillation boiling range and cetane index of the bio-oil was determined using the following standard methods:

Physical properties Method

Density -ASTM D1298 - 99
Kinetic viscosity- ASTM D445 - 11
Conradson carbon -ASTM D189 - 06(2010)e1
Flash point -ASTM D6450 - 05(2010)
Fire point- ASTM D1310 - 01(2007)
Pour point -ASTM D5853 - 09
Calorific value- ASTM D5468 - 02(2007)
Distillation boiling range- ASTM D2887 - 08
Cetane index -ASTM D4737 - 10

Chemical characterization of bio-oil :-In order to determine the functional groups present in the pyrolytic oil, Fourier Transform Infrared spectroscopy of the oil was analyzed in a Perkin-Elmer infrared spectrometer

. Gas Chromatography–Mass Spectrometry of the pyrolytic oil was performed using a GC-MS-OP 2010[SHIMADZU] analyzer in Sargam Laboratory, Chennai to determine the chemical compounds present in the oil.

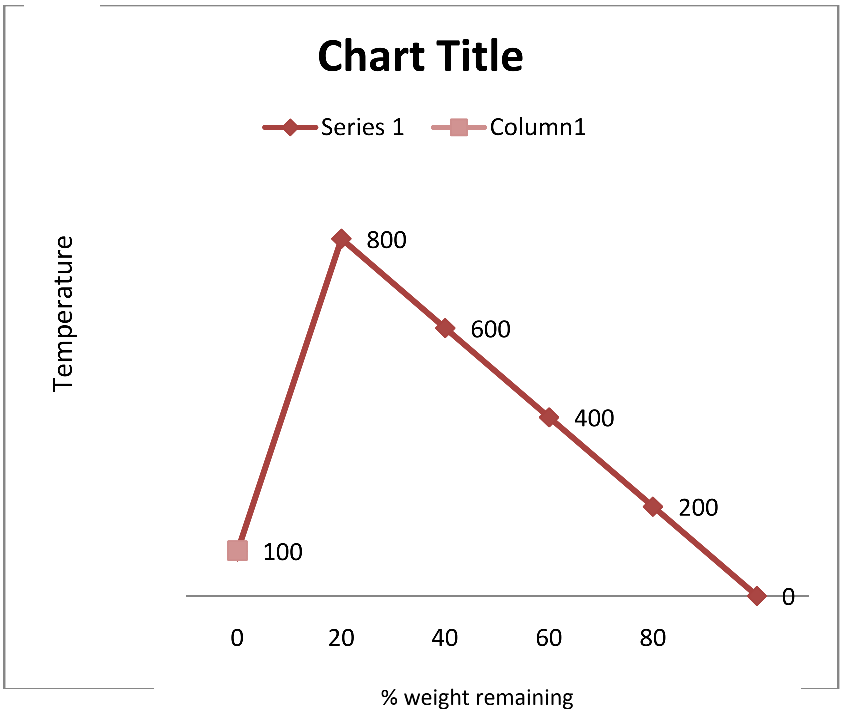
RESULT AND DISCUSSION

Thermo gravimetric analysis (TGA)

Thermo gravimetric analysis (TGA) measures the amount and rate of change in the weight of a material as a function of temperature. TGA is helpful in determining the range of temperature under which the moisture and the volatile content of the substance is driven out which is then condensed and the pyrolysed oil is recovered. Here the TGA is carried in normal atmosphere with the rate of heating being 10°C/minute and the range being room temperature to 800°C.

1) **Neem seed**

TGA plot for neem seed



The plot shows that maximum thermal degradation took place between ranges of 200°C to 600°C. The initial degradation can be explained by the fact that initially the moisture is driven away as the temperature is increased. Then the removal of volatile matter consisting of relatively less complex molecules accounts for the further degradation in weight which can be said for the range of 400°C to 550°C. The further degradation can be explained by the fact that much more complex molecules are driven away as the heating progresses.

CONCLUSION

1. The seeds used here are non-edible and hence they are perfect for biomass. It can also be explained by the fact India do not produce enough edible oils which can be used for this purpose.
2. Maximum yield of the neem oil are at 500°C and 475°C.
3. As the heating progresses the oil production increases at first and then after certain extent it start to decrease.
4. Reaction time decreases as the pyrolysis temperature is increased but char decreases with increase in pyrolysis temperature.
5. The fixed carbon increases in the char as most of volatile matter is driven away due topyrolysis. Also the volatile matter in seeds is good which accounts for the production of fuel on pyrolysis.
6. The oil obtained has comparable calorific value to most of the fuel used in day to day life but it has comparable higher pour point , density and kinematic viscosity, so one can say that transportation and piping of these fuels can be a tough task especially in cold areas.
7. Seeing the physical properties one can say that it is moderate grade fuel and can be used as blends with other major fuels.
8.Neem oil consist of over forty compounds over twenty compounds functional groups such as alkenes, alkenes, alkynes, alcohols, ketones, aldehydes ,aromatics rings, amides, nit riles and nitro compounds.

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