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Evaluation of the Transesterification Process And Physio-Chemical Properties of Jojoba Biodiesel

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Abstract*: *In this investigation, Jojoba bio-oil was converted to biodiesel through a process of transesterification. In the present work methanol is used as an alcohol and potassium hydroxide is used as a catalyst. Single stage transesterification process was employed as the content of free fatty acids was 1.2%. Physiochemical properties of the biodiesel like density, specific gravity, calorific value, flash and fire point, ash content, kinematic viscosity and acid value were found out using different methods and these properties are compared with those of petroleum diesel.

Keywords: Jojoba bio-oil, transesterification, physio-chemical properties, density, heating value.

I. INTRODUCTION

With the growing population and growing technology the petroleum reserves are being greatly depleted and also the environmental issues like air pollution and greenhouse effect demand for clean and renewable alternative fuels. In modern world most of the pollution occurs from the emissions of vehicles like nitrogen, sulphur and ash particles. Bio-fuel is the alternative fuel for petroleum fuels as they are biodegradable. The bio-fuel is used in place of petroleum fuels in order to reduce the toxic gases like nitrogen and negligible emission of sulphur. They produce very less amounts of greenhouse gases like carbon dioxide and methane when burnt. Environmental protection agency (EPA) has established a rule in the year 2006 that sulphur content must be reduced from 500 to 15 ppm in highways.

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The main reason for this rule is to prevent the poisoning of catalyst which is responsible reducing the emission from the exhaust. Replacing Bio-diesel in place of petroleum diesel can control the emission of sulphur. Bio-diesel is an oxygenated fuel. The bio-diesel contains nearly 10-12 wt% of oxygen making its quality of ignition better and hence less amount of CO is produced [1-3]. International Energy Agency has stated that the carbon dioxide production is increasing daily and from 2020-2030 it is estimated that 8.6 billion metric tons of carbon dioxide will be produced. When the bio diesel is burnt a small quantity of CO_2 is produced which is balanced with the photosynthesis process.

The bio-fuels are prepared from the bio oils which are also called as bio mass pyrolysis oil. The bio-oils are extracted from both non-edible oils and edible oils like seed, vegetable oils and animal fats. Previously bio-oil is extracted from soya beans which is an edible seed but the main constraint is the cost. It is better that the bio-oil is extracted from the non-edible seeds as the cost of cultivation and maintenance will be very less. The Bio-oil which satisfies these conditions is found to be Jojoba oil. This oil is extracted from jojoba seeds through the regular process of crushing. It has high amounts of water, solids and acids. It also have high values of properties like surface tension and viscosity, and have low heating value and the thermal properties are unstable. The bio-oil is converted in bio diesel through a process of transesterification. Greenhouse gas emission includes the emission of methane (CH_4), carbon dioxide (CO_2) and nitrous oxide (NO_2) [3-5]. The greenhouse depends on the quantity of these gases produced and the potential of the gases to cause the effect. The Greenhouse gas emission factors for consumption of natural gas, Bio-diesel, jojoba biomass, fossil fuels, electricity and glycerin are mentioned in Table 2.

Generally all the vegetable oils and animal fats are triglycerides but the jojoba oil is not, this is the main reason for selection jojoba oil. Also jojoba plant grows in low fertile lands and in different climates, like it does not require heavy amount of water and it can even grow in semi dessert areas. Argentina is the first Jojoba oil producer & exporter of the world. The seed oil of Jojoba was firstly extracted in 1929. The jojoba seed has nearly 40-50 wt. % of jojoba oil. The chemical reactivity of the jojoba oil is high and the normal boiling point is very low. In traditional oil seed crops glyceride oils are produced in these oils fatty acids are around the glycerol but the jojoba oil contains no glycerides. In the jojoba oil the fatty acids are directly bound to the fatty alcohols.

The chemical structure of jojoba oil has a long chain esters, which is very similar to whale oil and human sebum. It is made of fatty acids surrounded directly to the fatty alcohols. Generally the oil has very low value of acidity and its molecular simplicity is very different at high temperature. The oil has long chain of carbon atoms of nearly 38-44 carbons. The nature of Jojoba plant is it consists of high oil content in Jojoba seeds and these are the best cultivating crops as the Jojoba plant can grow and produce seed in different conditions of the climate. The plant can withstand up to a temperature of 55 degrees centigrade and as low temperature as 5 degree centigrade in the winter. Jojoba plant is a perennial shrub which grows mostly in the Mexico region and in the United States and partially in countries like South America, Egypt, South Africa Israel, India & Argentina. Once the jojoba seeds dry they fall to the ground from there they are collected and sent for crushing. Earlier the green seeds are harvested and crushed which resulted in less production of jojoba oil and when crushed it introduced water into the oil.

Transesterification process is the main step for converting vegetable/animal oil to bio-diesel. In this process the oil is treated with alcohol along with some catalyst which gives glycerol and biodiesel as products. In the first step the alcohol is taken and catalyst is mixed in it thoroughly and this liquid is added to the jojoba oil and left for some time with continuous stirring. The mixture is allowed to settle after the reaction, which forms two layers one is biodiesel and the other is mixture of glycerol and catalyst. Earlier the process of transesterification is carried out on several vegetable and seed oils which are edible, the biodiesel was extracted but the main problem is with the cost of the biodiesel produced. For many years the study of biodiesel produced from soya beans is done in America. But here also the main issue is the cost of production. Several studies were done to minimize the biodiesel cost, one of the important method is using non-edible oil instead of edible oil. In the present study the jojoba oil is found to be a very good alternative which is produced from the seeds of jojoba. These can be grown even in semi desert areas with minimal water supply. Reasons for selecting jojoba is that jojoba oil is not a triglyceride but it is a mixture of esters of long chain and the other reasons being the jojoba seed contains 40-50 wt. % of oil and that they can be grow in severe conditions. The jojoba oil is converted into bio diesel by transesterification using methanol/ethanol, sodium methoxide/potassium hydroxide is used as catalyst. Methanol and the jojoba oil are mixed in the molar ratio of 6:1. After the process of transesterification jojoba oil is converted into methyl jojobate and Jojobyl alcohol in which methyl jojobate is the biodiesel. The physical properties of the biodiesel at cold conditions are not so suitable for the engine. The density, pour point, viscosity and the cloud point of jojoba oil are higher than the petroleum diesel. So fuels having high viscosity have less penetration and flow properties in the cold conditions [5].

The properties like calorific value, density, pour point and calorific value gets decreased because of the addition of methanol. The low cold flow properties of bio-diesel are improved by adding methanol. Jojoba Methyl Ester (JME) is produced through a process of transesterification. The solvent extraction is done by making two separate phases: the lower layer rich in biomass phase & the upper layer rich bio-diesel phase. The bio-diesel is less in density. It can be used as a transportation fuel. The results has showed that the blends showed have more thermal stability and ideal fuel properties than of normal bio-diesel which is in use.

Canoira *et al (*2006) proposed a procedure for converting jojoba oil-wax to jojoba biodiesel through transesterification process by using methanol and sodium methoxide is used as catalyst. Methanol, toluene and petroleum ether are used for transesterification is done to convert wax of jojoba oil to methyl jojobate with acid catalysis and with basic catalysis. The physiochemical properties of both the biodiesel and wax are found out [6]. Bouaid *et al (*2007) proposed on FAME (fatty acid methyl ester) from agricultural and vegetable oils from various oil seed crops. They obtained the maximum yield of esters. In the transesterification process fatty acids are produced as secondary products. A basic catalyst was used in this process [7]. Jiang *et al* (2010) proposed solvent extraction which means to the bio-fuel is upgraded in another way. They have used a method in which the bio-oil is not used as a whole instead another better oil is used to extract fuel fractions of high quality leaving the rest of the matter. After doing so the blends of these fuels form two layers the top one is rich bio-oil and the bottom one is rich bio-diesel [8]. Azad *et al* (2018) have conducted several processes to find out the emissions from the diesel engine when jojoba bio-diesel is used instead of the petroleum diesel. The results of processes conducted with bio-diesel and their blends are compared with that of petroleum diesel and the deviations are found out [9]. Shailesh *et al* (2009) studied the energy analysis for the production of biodiesel from the oil of jojoba seeds. The energy efficiency are mentioned in terms of the total energy ratio. For the analysis of production of bio-diesel from seed-oil, they analyzed the husk, waste residues and glycerin. They calculated the GHG emissions of biodiesel. John *et al* (2018) studied about the plants producing bio-diesel by using a simulation software. Both edible and non-edible oils in the vegetable oils are used in this study. They have used different kinds of catalysts in their study [17]. Gorski *et al* (2008) they have used the HYSYS software to find out the properties of only the non-edible vegetable oils with different catalysts. Vegetable oils have low volatility, high viscosity and they have these unsaturated hydrocarbon chain which are very reactive. They have taken only the non-edible vegetable oils as to prevent the usage of edible oils in bio-diesel production [11]. Al Awad *et al* (2014) conducted an experimental study to find out the effect of ethanol on jojoba biodiesel blends and on the performance and emission characteristics of a C.I engine. They prepared jojoba methyl from the transesterification process. They found out and compared the characteristics with that of standard biodiesel. For the testing purpose they have used air cooled C.I engine and the loading is given electrically. The brake thermal efficiency of jojoba biodiesel was found out. They did an emission analysis like hydrocarbon emissions on the jojoba biodiesel [12]. Huzayyin *et al* (2004) have found out the physical properties of both jojoba oil and its blends with petroleum diesel. The equations for measuring the properties of both the jojoba oil and its blends are found out. The density and viscosities of these fuels were found out using these equations [14]. Selim *et al* (2003) the viscosities of both jojoba oil along with blends and petroleum diesel are found.

Four viscosity correlations are suggested to predict the viscosity variation with temperature is found out. They used four equations for study. They are 1. Increase third order correlation 2. Hyperbolic three- parameter decay correlation 3. Rational polynomial correlation 4. Single exponential decay three-parameter equation [15]. Saleh *et al* (2009) proposed the process of converting jojoba oil to biodiesel using organic catalysts. They did experiments on the effect of different parameters on the percentage conversion of biodiesel. They determined the physical and fuel properties of jojoba biodiesel. The identification of fatty acid methyl esters GC-MS systems were done [16].

Many works have been done on the jojoba oil and also on the processes and techniques of converting jojoba oil into biodiesel. But the researches were not able to bring down the cost of the biodiesel to the cost of petroleum diesel and also they were unable to bring down the reaction time significantly during the process of transesterification. The main aim of this work is to perform transesterification process on jojoba oil to convert it to biodiesel and to find out the physiochemical properties of the biodiesel and compare them with the petroleum diesel.

II. MATERIALS AND METHODS

In the present work the jojoba biodiesel is extracted from the jojoba oil. This is done by a process called transesterification. As we are performing transesterification on jojoba oil, no pre transesterification is needed. For this work we used jojoba oil, potassium hydroxide which is used for transesterification and potassium hydroxide is used as catalyst, mild H_2SO_4 is used for neutralizing the transesterified solution and the properties of the biodiesel like viscosity, flash point, acid value, specific gravity, heating value, ash content are found out.

A. Transesterification of Jojoba Bio-oil

There are several methods of transesterification one of the effective methods is by using potassium hydroxide as catalyst and methanol as alcohol. In the first step potassium hydroxide (KOH) (1.3 wt. % of the reaction) is added with methanol to make potassium methoxide. This mixture is mixed with jojoba oil in a 6:1 molar ratio i.e. 6 moles of potassium hydroxide is mixed with one mole of jojoba oil. This mixture is taken in a 500 ml sealed-clapped glass reactor. At room temperature this reaction takes about 80 minutes for completion under continuous vigorous stirring at 600 rpm. To minimise the reaction time a heating plate is used to vary the temperature of the mixture. With the increase in temperature up to a certain level there will be a reduction in the reaction time and the percentage conversion of bio oil to biodiesel will increase. It was found that at 50^o the reaction time is 25 minutes and it gives 95% of conversion [17]. After the reaction is completed the liquid is neutralized with hydro chloric acid to reach pH content 7. To separate any catalyst residue and any excess acid present. Product was washed with distilled water. The amount of water used is 5% of total mass of the reaction. After washing the product was settled for 8 hours. After the settling time the liquid forms two layers. The top layer is the bio diesel (methyl jojobate) and the bottom layer is the mixture of both methanol and other residues such as glycerine. The second layer is Jojobyl alcohol. The two layers are easily separated and the layer of biodiesel was dried at 80^0C in an oven for removing excess water or methanol present in it. The Jojobyl alcohol and methanol mixture were distilled using the distillation apparatus for separating methanol for reuse.

$$yield\ \% = \frac{volume\ of\ biodiesel\ product}{total\ feed\ volume} \times 100$$

B. Physio-chemical properties

Viscosity of the fuel is found out using redwood viscometer the description of the equipment and the procedure for finding out the viscosity are given below. The viscometer cup is cleaned and properly dry it for removing any trace of solvent. The viscometer is levelled with levelling screws. The outer bath is filled with water to determine the viscosity and the temperature of 80^0c is maintained. The ball is used to close a valve from which the oil flows to the flask. Place a dry flask below in the line with discharging jet. A stirrer and thermometer are inserted through the lid. Heat the water slowly with continuous stirring. When the jojoba oil in the cup reaches the required temperature stop the heating. The ball is removed and the jojoba oil is allowed to flow into the flask upto the mark of 50 ml. The time for 50ml of oil to be collected is noted down and found to be 32 seconds.

The Specific gravity of a substance is defined as the ratio of density of the liquid for which the specific gravity is to be found out to the density of standard liquid or mass of the liquid for which the specific gravity is to be found out to the mass of the standard liquid which is water. There are several methods for finding the specific gravity of a liquid, the one we used to find out the specific gravity of jojoba oil is by using pycnometer. A pycnometer is simply a bottle which is filled with the sample jojoba oil up to the 50 ml mark and is weighed on the simple balance, and the same bottle is used to fill it with water which is considered as a standard liquid. The ratio of masses of jojoba oil to the water is the specific gravity of the jojoba oil [18].

The acid value is used to known free fatty acids in an oil. It is the KOH by weight needed to neutralize the oil. The jojoba oil is dissolved in toluene and then titrated with KOH which is 0.1 normal. 100 ml of jojoba oil is dissolved in 125 mL of a solute (toluene and isopropyl alcohol) which is a mixture of tolune and isopropyl alcohol. Mixture of the solvent is neutralized with KOH. The oil is mixed and titration is done until the appearance of pink color.

$$Acid\ value = ((S - B) \times N \times (56.1)/W$$

Where S - standard alkali used (ml), B-standard alkali used for titration of the blank (mL), N- normality of alkali and W is sample weight

Flash Point of a substance is the least temperature at which it gives vapours that ignite for some time when a flame is kept near it. It is done by using Pensky Marten's flash point apparatus. The oil cup is filled with the jojoba oil upto the mark. The lid is fixed which has thermometer and stirrer. The oil is stirred at a rate of 60 revolutions per minute. And the flash is checked for every 1^oC rise in the temperature of jojoba oil. For jojoba biodiesel the flash point is found to be 150^oC.

Ash content is the amount of left out materials oil in air at specific high temperature. The ash content of the oil is the measure of the amount inorganic non-combustible material it contains.

The dry crucible is taken and is weighed it was 200 grams in weight which is taken as W1. Then 2grams of jojoba oil is taken in the crucible and its mass is found out which is 202 grams and it is taken as W2. Then this sample is fired with a burner. Heat the remaining in a furnace at 775 +25 °C until all carbon materials has disappeared, it takes 20-30 mins. The crucible temperature is brought nearer to room temperature. The crucible is weighed this will be W3 we got W3 as 200.16

$$Ash\ content = \frac{W3 - W1}{W2} \times 100$$

Heating value or calorific value is the amount of heat emitted by the fuel when it is burnt. It is always mentioned as joules of heat liberated per kilogram burning of the fuel. For finding out the calorific value of a fuel a Bomb calorimeter is used. 100 grams of jojoba oil sample (X) is taken in a small crucible. A thin magnesium wire that touches the fuel sample is stretched across the electrodes. The lid of the bomb is sealed tightly [19]. The bomb is made full with oxygen at 25-30atm because the fuel is to be combusted fully. Then the BOM apparatus is inserted in a copper calorimeter containing known weight of water (W_b). The temperature (t1) of the water at the beginning is noted thorough stirring. The burning of the oil takes place once the current is switched on. The max temperature (t_2) of the water is noted down which is obtained by the heat liberated from combustion. The calorific value of the fuel can now be calculated. Water equivalent of the calorimeter, stirrer and bomb thermometer is found out (W_b).

Heat gained by water = $W(t_2 - t_1)$ cal
Heat gained by calorimeter = $w(t_2 - t1)$ cal
Total heat gained = $W(t_2 - t_1)$ cal + $W(t_2 - t_1)$ cal
= $(W + w)(t2 - t_1)$
Heat liberated by the fuel = X *L
Now, Heat liberated by the fuel = Heat gained by water and calorimeter

$$X * L = (W + w)(t_2 - t_1)$$
$$L = (W + w)(t_2 - t_1) / X \text{ cal/g.}$$

Table- I: Physiochemical Properties of Test fuels

Physio-Chemical Property	Units	Jojoba oil	Jojoba bio-dies el	Petroleum diesel
Specific gravity	-	0.865	0.877	0.739
Flash point	^oC	275	150	52-96
Kinematic viscosity	mm/s^2	24.05	5.86	1.3
Acid value	Mg KOH/g	0.71	0.22	2.9
Ash content	%by weight	0.064	0.08	0.01-0.02
Heating value	MJ/Kg	49.34	35.66	45.5

III. RESULTS AND DISCUSSION

The transesterification of the jojoba oil with methanol is carried out and the physiochemical properties of the jojoba oil before and after the transesterification process were found out using several methods mentioned above. These properties are compared with the petroleum diesel and the advantages and disadvantages of the jojoba biodiesel are discussed.

Specific gravity is directly proportional to the density of the fuel, the density of the fuel used in the engine determines the combustion and emissions, particle pollution emissions increases with increase in the density. The amount of fuel flow in the engine is also determined by the specific gravity. Specific gravity is influenced by the chemical composition of the oil and on the temperature. The specific gravity of jojoba oil is found to be 0.865, whereas the sp. gravity of jojoba biodiesel is 0.877, sp. gravity of petroleum diesel is 0.739. The specific gravity of biodiesel is increased because of the addition of different components during the process of transesterification. From the table we can see that the specific gravity of the jojoba oil and the jojoba diesel are high compared to the petroleum diesel as these oils contain bio matter in them [21].

The flash point of the fuel is an indication of how easy it may burn. The flash point of jojoba oil is found to be 275^0 C, and of jojoba biodiesel is 150^oC and for the petroleum diesel is $52\text{-}96^oC$. We know that the bio-oil has a high organic content in it making it difficult to burn. Petroleum diesel has the least flash point as it doesn't contain any organic matter in it. Having low flash point makes it easy for combustion and the performance of the engines will be smooth.

Kinematic viscosity of a bio-oil is typically measured at 40^oC to determine the fluid properties. The viscosity value of the bio-oil is used to determine the stability of liquid fuel during storage. Viscosity varies widely depending on the biomass type and the water content in the oil. The found kinematic viscosities of jojoba oil was 24.05 mm/s^2, biodiesel was 5.86 and the petroleum diesel was 1.3. The kinematic viscosity of biodiesel is decreased because of the addition of the methanol during transesterification. As mentioned above viscosity depends on the biomass content of the oil so the kinematic viscosity of the jojoba oil is highest among the three oils. Next in order is biodiesel and the petroleum diesel has lowest viscosity [22].

The acid value is the number of mg of potassium hydroxide required to neutralize the free acid in 1 gram of oil. A good oil should have a very low acid value. Increase in acid value should be taken as an indicator of oxidation of the oil which may lead to gum formation. Compared to jojoba oil and the petroleum diesel jojoba biodiesel have less acid value making it less prone to gum formation. Acid value of jojoba oil is found to be 0.71, of jojoba biodiesel is 0.22 and of petroleum diesel is 2.9.

Ash content is the amount of non-combustible carbon contents formed when the oil is burnt. Ash content depends on the organic mass present in the oil. So higher the ash content of the oil less is the pollution caused by the oil when burnt as ash is the remains of organic matter. But the presence of ash content is not good for the engine. Higher the ash content higher is the chance of seizing of the engine. The ash contents of jojoba oil as found to be 0.064, jojoba biodiesel having 0.08 and the petroleum diesel was 0.01-0.02 are and the ash content of the biodiesel is more making it less suitable for engines working for long durations [20, 23].

Heating value is the amount of heat liberated by an oil when a certain quantity of it is burnt. It determines the power produced by the oil when combustion takes place. Higher the heating value higher is the power generated by the oil. The heating values of jojoba oil is found to be 49.35 and of biodiesel is 35.66 and petroleum diesel is 45.55, biodiesel have less heating value than the petroleum diesel.

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IV. CONCLUSION

In the present work the jojoba oil is converted into biodiesel through a process of transesterification using methanol as alcohol and potassium hydroxide as catalyst. The physiochemical properties like kinematic viscosity, specific gravity, flash point, acid value, ash content, and heating value are found out and are compared to the properties of petroleum diesel. Finally, it can be concluded that the derived Jojoba biodiesel was suitable for use in CI engine with similarity in physiochemical properties to that of mineral diesel. In the future work of this project we are going to find out the effect of temperature on the percentage yield of the biodiesel, the time of the reaction and on the physiochemical properties of the oil.

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