



Preparation of Methyl Ester (Biodiesel) from *Jatropha Curcas* Linn Oil

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Abstract

*Self reliance in energy is vital for overall economic development of our country. The need to search for alternative sources of energy which are renewable, safe and non-polluting assumes top priority in view of the uncertain supplies and frequent price hikes of fossil fuels in the international market. Biodiesel (fatty acid methyl ester) which is derived from triglycerides by transesterification, has attracted considerable attention during the past decade as a renewable, biodegradable and nontoxic fuel. Several processes of biodiesel fuel production have been developed, among which transesterification using alkali as a catalyst gives high level of conversion of triglycerides to their corresponding methyl ester in a short duration. This process has therefore been widely utilized for biodiesel fuel production in number of countries. As the acid value of *Jatropha curcas* oil is high, so that we have to reduce it by the process of esterification followed by transesterification. The methyl ester produced by this way gives the good result. The present study deals with transesterification of *Jatropha curcas* oil which gives >83% of methyl ester and >17% of glycerol using molar ratio 6:1 (methyl alcohol to oil) and 0.5wt % of sodium hydroxide at 65^oC for 90 minutes and allowed to settle overnight. As per ASTM 6751 (American Standards For Testing and Material) the properties like density, viscosity, flash point, cloud point and pour point have been carried out at Indian Biodiesel Corporation, Baramati for accessing the fuel quality of *Jatropha* oil methyl ester. (JOME)*

Keywords: *Jatropha* oil, biodiesel, transesterification and esterification.

Introduction

The concept using vegetable oil as a fuel dates back to 1895 when Dr. Rudolf Diesel developed the first diesel engine to run on vegetable oil. Rudolf Diesel stated: "the use of vegetable oil for engine fuels may seem insignificant today. But such oil may become in source of time as important as petroleum and the coal tar products of the present time Biodiesel is a non petroleum based fuel defined as fatty acid methyl ethyl esters derived from vegetable oil or animal fats and it is used in diesel engines and heating systems. Thus this fuel could be regarded as mineral diesel substitute with the advantage of reducing greenhouse emissions because it is renewable resource¹.

The extracted oil could not be used directly in diesel because of its higher viscosity. High viscosity of pure vegetable oil would reduce the fuel atomization and increase the fuel spray penetration, which would be responsible for high engine deposits and thickening of lubricating oil. The use of chemically altered vegetable oil called biodiesel does not require modification in engine or injection system or fuel lines and is directly possible in any diesel engine. Biodiesel can be produced from vegetable oils or animal fats via transesterification. The transesterification is the reaction between oil and fat, with a short chain alcohol (methanol, ethanol, and propanol) in the presence of suitable catalysts in the transesterification reaction, as they give high production yield².

Few researchers have worked feedstock having higher FFA levels using alternative processes. But there are certain exceptional cases wherein direct trans-esterification cannot be performed. Such cases appear in raw vegetable oils (non edible oils) like karanja^{3,4}, *Jatropha*⁵, mahua, neem, castor⁶ and simaroubae⁷ etc. because these non edible oils possess high free fatty acids (FFA). For determining whether the raw vegetable oils can be trans-esterified directly, the acid value is the most important property that must be known.

Oils of high free fatty acids content can be converted into biodiesel via dual step transesterification process. In the first step, the oil is treated by an acid dissolved in methanol to reduce FFA content, whereas in the second step the preheated oil is transesterified with methanol in the presence of base catalyst to form ester and glycerol.⁸

Advantages of Biodiesel: i. Biodiesel is the only alternative fuel that runs in any conventional, unmodified diesel engine. ii. It is biodegradable, nontoxic and free of aromatics. iii. It is a renewable fuel, based on oil crops or trees. iv. 100% domestic fuel. v. Based on Ames Mutagenicity tests, Biodiesel provides a 90% reduction in cancer risks. vi. Cetane number is significantly higher than that of conventional diesel fuel. vii. Lubricity is improved over conventional diesel fuel. viii. Has high flash point (more than 120^oC) compared to that of conventional diesel, which has a flash point of 65-70^oC. ix. Less consumption of environmental resources compared to fossil

diesel and therefore, lowers environmental protection costs. x. Reduction in mineral oil imports and or petroleum diesel fuel imports.

Jatropha curcas linn, is a renewable and sustainable source of Biodiesel: The *Jatropha curcas* Linnaeus plant *J. curcas* L. belongs to the family *euphorbiaceae*. The genus name *Jatropha* derives from the Greek *jatros* (doctor), *trophe* (food), which implies medicinal uses, hence the plant is traditionally used for medicinal purposes. It is a hardy shrub that can grow on poor soils and areas of low rainfall. Hence its being promoted for ideal plant for small farmers⁹. It is a drought resistant, perennial plant and living upto fifty years and has a capability to grow on marginal soils. It requires very little irrigation and grows in all types of soils. It is rapidly growing tree and easily propagated. *Jatropha* usually grows below 1400 meters of elevation from sea level and requires a minimum rainfall of 250mm, with an optimum rainfall between 900-1200mm¹⁰. It is non-edible oil being singled out for large-scale for plantation on waste lands *Jatropha* plant can thrive under adverse condition. The oil content of *Jatropha* seed ranges from 25% to 30% by weight. Fresh *Jatropha* oil slow-drying, order less and color less oil, but it turns yellow after aging¹¹⁻¹⁴.

The cultivation of the *Jatropha* plant: *Jatropha curcas* L. has various socio-economic benefits which makes it more economical when cultivated on commercial scale. Indian Biodiesel Corporation, Baramati (IBDC), Baramati is working for plantation of all types of non edible oil seeds plant since last ten years. *Jatropha* can be propagated from seeds as well as from cuttings. Seeds or cutting can be directly planted in the main field otherwise, seedlings grown in poly bags are transplanted in the field. With spacing of 3*2m under irrigated condition accommodating 1666 plants/ha. A hectare of *Jatropha* plantation is reported to yield 0.25-0.30 MT/ha/yr in the third year and increases sharply to 2.5-4.0 MT/ha/yr from the sixth year onwards¹⁵. The plantation programme, fruit mature fruit bunch and seeds are shown in figure 1, 2 and 3.

***Jatropha* as a plant of many uses:** Rural energy problems in developing countries and are linked with other rural problems. These problems need an integrated approach to reach solution. The *Jatropha* plant has four main contributions to rural development and poverty eradication in general renewable energy promotion of women poverty reduction and soil erosion control. The *Jatropha Curcas* has many products and potential contributions to rural community development. The products of the *Jatropha* plant are the plant itself fruits hulls. The seeds produce seed oil, seed cake, and seed shells. The oil processes also produce sediments from oil purifications. The *Jatropha* plant itself can used in erosion control if planted across the hills and against the wind. The plant can also be used as firewood. The fact that it grows very fast means *Jatropha* can be used to solve the problem of deforestation in many developing countries The *Jatropha* plant also provides a source of employment to many rural areas, which in turn helps to reduce urban migration in developing countries.



Figure-1



Figure-2



Figure-3

Figure-1, 2 and 3
***Jatropha* Tree Plantation, *Jatropha* Fruit and *Jatropha* Seeds By IBDC, Baramati**

Toxicity of the Jatropha plant: The toxicity of the *Jatropha curcas* is an advantage on one side and disadvantage emanates from the fact that the other. The advantage emanates from the fact that the plant leaves can't be browsed by animals and could act as an excellent fence. The disadvantage comes from the fact that the equipment, such as raw presses that are used to press *Jatropha* seeds, could not be used to press other edible seed oil from plants like sunflower unless a thorough cleaning is done which would take a lot of environmental resources. The claims that there are some varieties of non-toxic *Jatropha* plants need more investigation.

Characteristic of Jatropha Oil: Non-edible oil generally contains about 3-4 % wax and gum. De-waxing and degumming of plant oils is required not only for smooth running of the CI engine but also to prevent engine failure even if plant oils are blended with diesel. It is therefore necessary to remove wax and gum from the fresh oil before it could be used in CI engine. Analysis of *Jatropha* seeds revealed that the percentage of crude protein, crude fat and moisture were 24.60, 47.25 and 5.54% respectively. Characterization of diesel and *Jatropha* oil is as per the table-1.

Crude *Jatropha* oil, a non-edible vegetable oil shows a greater potential for replacing conventional diesel fuel quite effectively as its properties are compatible to that of diesel fuel¹⁶. It is however found from researches that the neat *Jatropha* oil can be used to run the engines in mini-vans for rural transportation haulage trucks, farm tractors and other agricultural machinery, with or without modification in engine¹⁷.

Material and Methods

Feedstock preparation: The fresh seeds are collected from IBDC, Baramati, Maharashtra. The healthy seeds are selected and are cleaned, dried at 100-105°C. For 30 minutes. The dried seeds were taken for oil extraction through ordinary mechanical oil extractor.

Pretreatment: In this method, the *jatropha* oil is first filtered to remove solid material then it is preheated at 110°C for 30 min to remove moisture (presence of moisture responsible for saponification in the reaction)¹² After demulsification of oil we removed available wax, carbon residue, unsaponifiable matter and fiber that are present in a very small quantity and carried out some important tests for available free fatty acids oil that are given in table-2.

Esterification: *Jatropha* oil contains 6%- 20% (wt) free fatty acids¹³⁻¹⁶. The methyl ester is produced by chemically reacting *jatropha* oil with an alcohol (methyl), in the presence of catalyst. A two stage process is used for the transesterification of *jatropha* oil^{17,18}. The first stage (acid catalyzed) of the process is to reduce the free fatty acids (FFA) content in *jatropha* oil by esterification with methanol (99% pure) and acid catalyst sulfuric acid (98% pure) in one hour time at 57°C in a closed reactor vessel.

The *jatropha* crude oil is first heated to 50°C and 0.5% (by wt) sulfuric acid is to be added to oil then methyl alcohol about 13% (by wt) added. Methyl alcohol is added in excess amount to speed up the reaction. This reaction was proceed with stirring at 650 rpm and temperature was controlled at 55-57 °C for 90 min with regular analysis of FFA every after 25-30 min. When the FFA is reduced upto 1 % , the reaction is stopped. The major obstacle to acid catalyzed esterification for FFA is the water formation. Water can prevent the conversion reaction of FFA to esters from going to completion¹⁹. After dewatering the esterified oil was fed to the transesterification process²⁰.

Experimental set up: The experimental set up is shown in figure 4. A 2000 ml three necked round-bottom flask was used as a reactor. The flask was placed in heating mantle whose temperature could be controlled within ± 2 °C. One of the two side necks was equipped with a condenser and the other was used as a thermo well. A thermometer was placed in the thermo well containing little glycerol for temperature measurement inside the reactor. A blade stirrer was passed through the central neck, which was connected to a motor along with speed regulator for adjusting and controlling the stirrer speed.

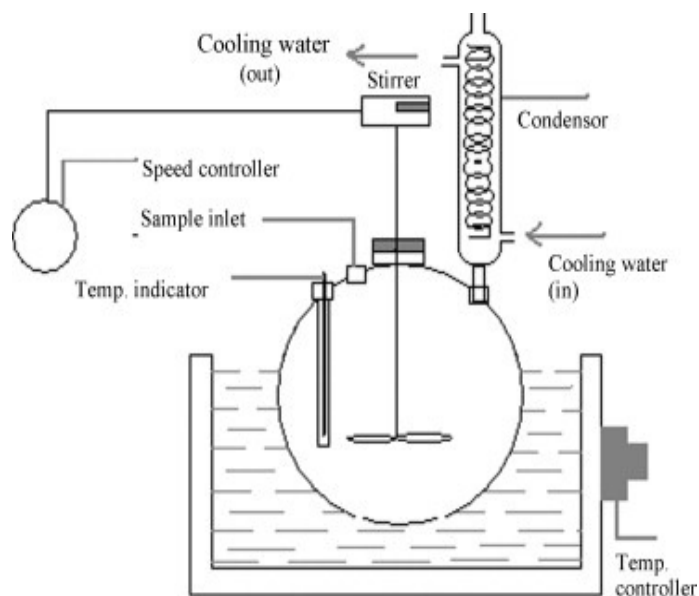


Figure 4
Experimental Set up For Transesterification of Jatropha crude oil

1000ml of *Jatropha* oil was measured using measuring cylinder, then poured into a 2000 ml three necked round bottom flask. This oil was heated upto 60°C.

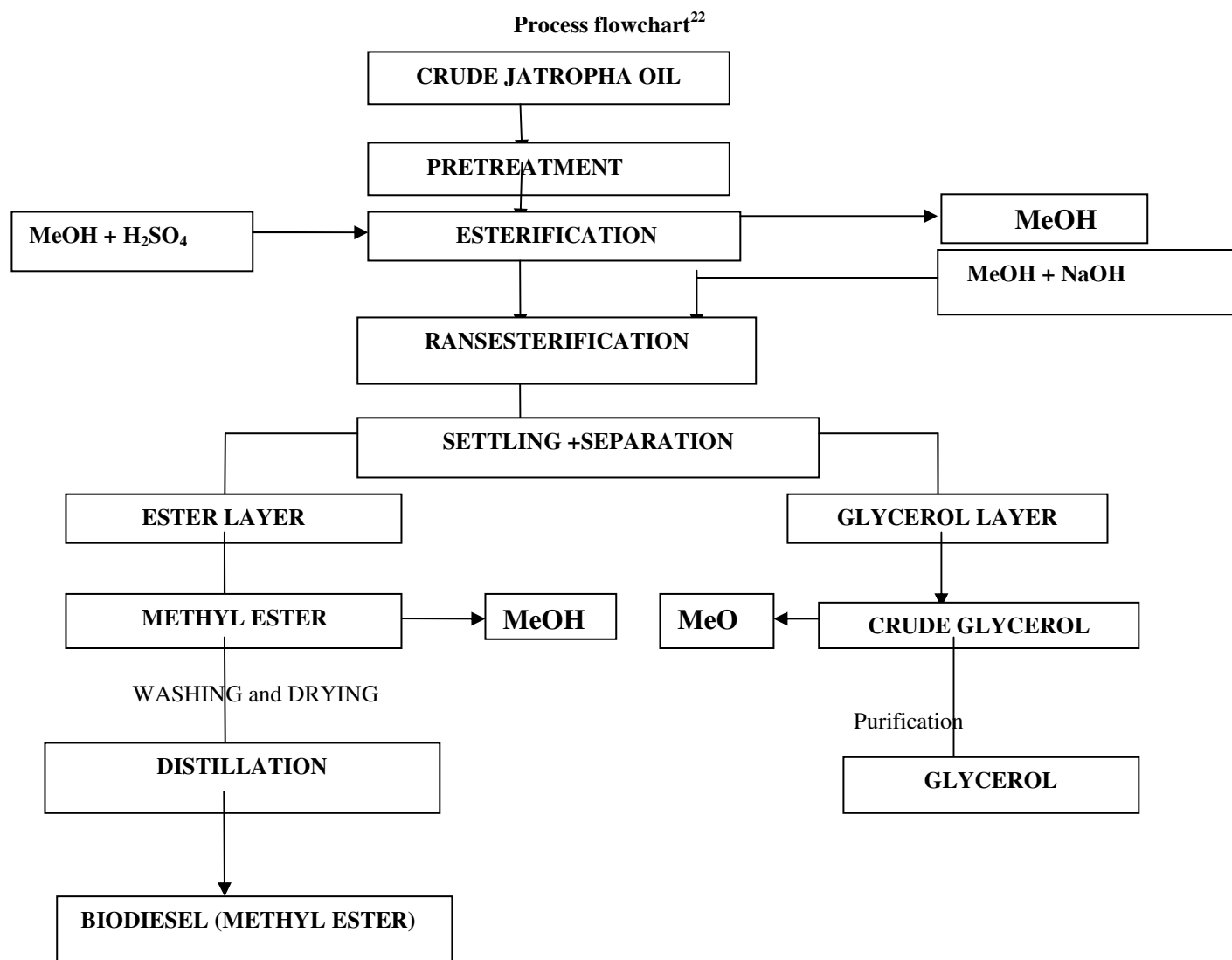
In 250ml beaker a solution of potassium methoxide was prepared using 5g (catalyst concentration 0.5%) sodium hydroxide pellet and 214 ml (mole ratio of methanol to oil, 1:6) of methanol. The solution was properly stirred until the potassium hydroxide pellet was completely dissolved. The

solution was then heated upto 60°C and slowly poured into preheated oil. The mixture was stirred vigorously for one and half hour. Finally FFA was checked and mixture was allowed to settle for 24 hours in a separating funnel. Thereafter, upper layer biodiesel was decanted into a separate beaker while the lower layer which comprised glycerol and soap was collected from the bottom of separating funnel. To remove any excess glycerol and soap from the biodiesel, hot water was used to wash it and then allowed it to remain in separating funnel until clear water was seen below the biodiesel in the separating funnel. The pH of biodiesel was then checked. The washed biodiesel sample was then dried by placing it on a hot plate and excess water still in

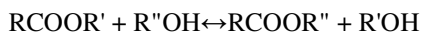
the biodiesel removed²¹. The process flow chart is shown in scheme-1.

Transesterification Reaction: Transesterification or alcoholysis is the displacement of alcohol from an ester by another in a process similar to hydrolysis, except an alcohol is used instead of water²².

This process has been widely used to reduce the high viscosity of triglycerides. The transesterification reaction is represented by the general equation as shown in scheme 2.



Scheme-1
 Press Flow Chart For Transesterification Of Jatropha Crude Oil



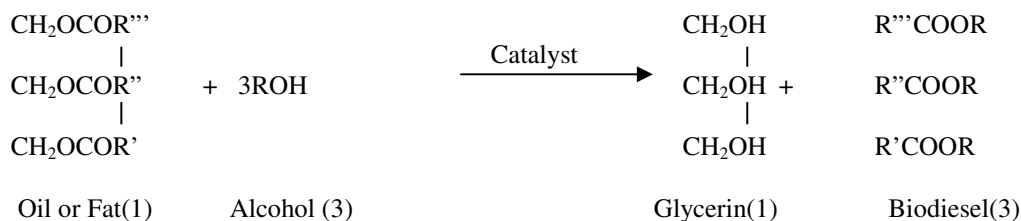
Scheme-2
 General equation of transesterification

Some feedstock must be pretreated before they can go through the transesterification process. Feedstock with less than 5 % Free Fatty Acid, may not require pretreatment. When an alkali catalyst is added to the feedstock's (With FFA > 5 %), the Free Fatty Acid react with the catalyst to form soap and water as shown in the reaction below:

If methane is used in this process it is called methanolysis. Methanolysis of glyceride is represented (scheme 3).

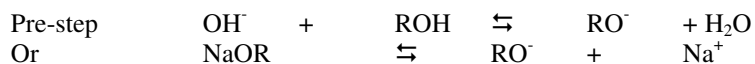
Transesterification is one of the reversible reactions. However, the presence of a catalyst (a strong acid or base) accelerates the

conversion. In the present work the reaction is conducted in the presence of base catalyst²³. The mechanism of alkali-catalyzed transesterification is described below. The first step involves the attack of the alkoxide ion to the carbonyl carbon of the triglyceride molecule, which results in the formation of tetrahedral intermediate. The reaction of this intermediate with an alcohol produces the alkoxide ion in the second step. In the last step the rearrangement of the tetrahedral intermediate gives rise to an ester and a diglyceride. The same mechanism is applied to diglyceride and monoglyceride. The reaction mechanism of transesterification is shown in scheme-4.

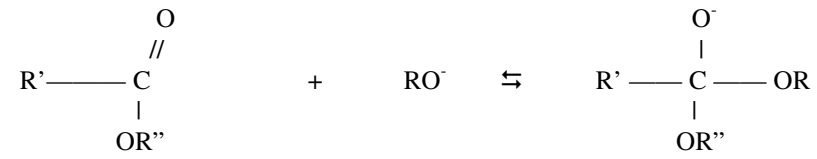


Scheme-3
 General equation for methanolysis of triglycerides

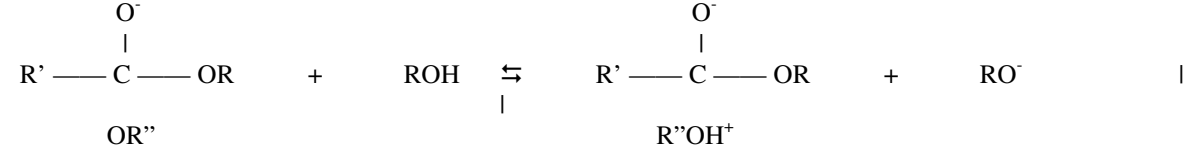
Reaction mechanism –



Step 1.



Step 2.



Step 3.



Where $\text{R}'' = \begin{array}{c} \text{CH}_2 - \\ | \\ \text{CH} - \text{OCOR}' \\ | \\ \text{CH}_2 - \text{OCOR}' \end{array}$
 $\text{R}' = \text{Carbon chain of fatty acid}$
 $\text{R} = \text{Alkyl group of Alcohol}$

Scheme-4
 Mechanism of base catalyzed transesterification

Effect of Different parameters: Effect of Molar ratio: Sharma and Singh³ used similar two step transesterification and took 8:1 molar ratio for acid esterification and 9:1 molar ratio for alkaline esterification for optimum yield of biodiesel production from karanja oil. Meher et al.²⁴ carried out investigation with 6:1 molar ratio during acid esterification and 12:1 molar ratio during alkaline esterification. Karmee and Chanda²⁵ used a single step transesterification and have achieved 92% conversion by taking 10:1 molar ratio. Presence of sufficient amount of methanol during the transesterification reaction is essential to break the glycerine-fatty acid linkages²⁶. But excess of methanol should be avoided. Increasing the molar ratio of methanol/oil beyond 6:1 neither increases the product yield nor the ester content, but rather makes the ester recovery process complicated and raised its cost. Leung and Guo²⁷ suggested that methanol has polar hydroxyl group which can act as an emulsifier causing emulsification. Thus separation of the ester layer from the water layer becomes difficult.

Effect of moisture and water content on the yield of biodiesel: Kusdiana and Saka²⁸ observed that water could pose a greater negative effect than presence of free fatty acids and hence the feedstock should be water free. Romano²⁹ and Canacki and Van Gerpen³⁰ insisted that even a small amount of water (0.1%) in the transesterification reaction would decrease the ester conversion from vegetable oil. Demirbas³¹ too reported a decrease in yield of the alkyl ester due to presence of water and FFA as they cause soap formation. Consume catalyst and reduce the effectiveness of catalyst. Srivastava and Verma³² removed the moisture content from the vegetable oil by heating in oven for 1 h at 383 K. Meher et al.²⁵ too reported a precautionary step to prevent moisture absorbance and maintenance of catalyst activity by preparing the fresh solution of potassium hydroxide and methanol. Ellis et al.³³ found that even a small amount of water in the feedstock or from esterification reaction producing water from FFA might cause reduction in conversion of fatty acid methyl ester and formation of soap instead.

Effect of free fatty acids: Free fatty acids (FFAs) content after acid esterification should be minimal or otherwise less than 2% FFAs. These FFAs react with the alkaline catalyst to produce soaps instead of esters.

Effect of temperature: The temperature maintained by the researchers during different step range between 60-65°C. It is near to boiling point of methanol. Temperature higher than this will burn the alcohol and will result in much lesser yield. A study by Leung and Guo²⁸ showed that temperature higher than 65°C had a negative impact on the product yield for neat oil but had a positive effect for waste oil with higher viscosities.

Effect of Stirring: Stirring can play an important role in the yield of biodiesel production. Mehar et al.²⁵ conducted the transesterification reaction with 180, 360 and 600 revolution per minute (rpm) and reported incomplete reaction with 180

rpm. Sharma and Singh³ reported that mode of stirring too plays a vital role in the transesterification reaction. The yield of biodiesel increased from 85% to 89.5% when magnetic stirrer (1100 rpm) was replaced with mechanical stirrer (1100 rpm).

Results and Discussion

Oil Extraction: The extraction of oil from jatropha seeds were done by Mechanical expelling method, yield obtained 22%.

Table-1
Properties of Jatropha Crude Oil

Sr. No	Parameter	Jatropha Curcas Oil
1	Density (gm/cc), 30 ⁰ C	0.93292
2	Kinematic viscosity (mm ² /s), 30 ⁰ C	55
3	Flash Point (⁰ C)	180
6	Fire Point (⁰ C)	256
7	Pour Point (⁰ C)	6
8	Saponification Value	187
9	FFA (%)	13.7
10	Cetane value	40
11	Sulphur (%) by Wt	0.0.13
12	Oxygen (% w.w)	11.06
13	Carbon (% w/w)	76.11
14	Hydrogen (% w/w)	10.52
15	Ash Content (% w/w)	0.03±0.0

From the above table 1, Density, cloud point and pour point of Jatropha oil are found to be higher than diesel. Higher cloud and pour point reflect unsuitability of Jatropha oil as diesel fuel in cold climatic condition but the flash and fire points of Jatropha oil is very high compared to mineral diesel. Hence Jatropha oil is extremely safe to handle. Higher carbon residue from Jatropha oil may possibly lead to higher carbon deposits in combustion chamber of the CI engine. Low sulphur content in Jatropha oil result in lower Sox emissions. Presence of Oxygen in fuel improves combustion properties and emission but reduces the calorific value of the fuel³⁴. Jatropha Oil has approximately 90% calorific value compared to diesel. Nitrogen content of the fuel also affects the NOx emissions. Higher viscosity is the major problem in using vegetable oil as fuel for diesel engines. Viscosity of Jatropha biodiesel is 4.84cSt at 40⁰C. It is observed that viscosity of Jatropha oil decreases remarkably with increasing temperature and it becomes close to diesel at temperature above 90⁰C³⁵.

Free fatty acid present in Jatropha curcas oil oil: Extracted oil consisted of pure triglyceride and rests were free fatty acids and lipid associates, which is the measure of Unsaponifiable matter.

From the table-4 it is clear that, the per liter biodiesel recovery from jatropha oil was 832ml and the glycerine recovery per liter jatropha oil was 123ml. The biodiesel recovery by using above mentioned process was near 83%. Difference was observed in the amount of glycerin and methyl ester is due to the quality of feedstock oil.

Table-2
The Fatty acid composition of Jatropha crude oil

Sr.	Fatty acid name	Molecular formula	Composition (%)
1	Palmitic acid	C ₁₆ H ₃₂ O ₆	15.6
2	Stearic acid	C ₁₈ H ₃₆ O ₂	9.7
3	Oleic acid	C ₁₈ H ₃₄ O ₂	40.8
4	Linoleic acid	C ₁₈ H ₃₂ O ₂	32.1
5	Other acids		1.8

Table-4
Biodiesel Recovery in Transesterification Reaction

Particulars	Jatropha Biodiesel (ml)	Glycerine (ml)
Batch I	810	160
Batch II	860	100
Batch III	824	170
Mean	832	123

Table-5
Properties of Jatropha Methyl Ester as Per ASTM 6751-9B Standard

Sr. No	Parameter	Diesel	Jatropha Methyl Ester
1	Density (gm/cc), 30 ⁰ c	0.836-.850	0.8796
2	Kinematic viscosity (cSt), 30 ⁰ c	2.2	4.2
3	Flash Point (°c)	80	168
4	Fire Point (°c)	78	164
5	Pour Point (°c)	-6	-1
6	Moisture	-	0.02
7	Cetane value	40-55	42

Table-5 shows the fuel properties of biodiesel determined as per ASTM standards. Among the general parameters for biodiesel, the viscosity controls the characteristics of the injection from the diesel injector. The viscosity of vegetable oil derived biodiesel can go to very high levels and hence it is important to control it within acceptable level to avoid negative impact on fuel injector system performance. Therefore viscosity specifications proposed are nearly same as that of the diesel fuel. It is further reduced with increase in petroleum diesel amount in the blend.

Flash point of a fuel is the temperature at which it ignites when exposed to a flame or spark. The flash point of biodiesel is higher than the petro diesel, which is safe for transport purpose.

Cold filter plugging pint (CFPP) of a fuel reflects its cold weather performance. At low operating temperature, fuel may thicken and might not flow properly thereby affecting the performance of fuel lines, fuel pumps and injectors. CFPP defines the fuels limit of filterability having a better correlation than cloud point for biodiesel as well as petro diesel.

The above listed fuel properties from experimental results indicate that the jatropha oil methyl ester (JOME) is the best

suited as per American Standards for Testing and Material (ASTM) norms for using as biodiesel in pure as well as in blending form.

Conclusion

Thus this study suggests that the jatropha oils can be used as a source of triglycerides in the manufacture of biodiesel by transesterification reaction. The biodiesel from refined vegetable oils meets the Indian requirements of high speed diesel oil. But the production of biodiesel from edible oil is currently much more expensive than diesel fuels due to relatively high cost of edible oil. There is a need to explore non-edible oils as alternative feed stock for the production of biodiesel non-edible oil like jatropha. It is easily available in many parts of the world including India and it is cheaper compared to edible oils. Production of these oil seeds can be stepped up to use them for production of biodiesel. The production of biodiesel from this non edible oil provides numerous local, regional and national economic benefits. To develop biodiesel into an economically important option in India some innovations required for modification into the process to increase the yield of ester.

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