



Preparation of Methyl Ester (Biodiesel) from Karanja (Pongamia Pinnata) 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. In India, there are many trees bearing oil like ratanjot (*Jatropha curcus*), mahua (*Madhuca indica*), pilu (*Salvodara oleoids*), nahor (*Mesua ferralina*), kokam (*Garcinia indica*), rubber seed (*Hevea brasiliensis*) and karanja (*Pongamia pinnata*) etc. Among these species, which can yield oil as a source of energy in the form of biodiesel, *Pongamia pinnata* has been found to be one of the most suitable species due to its various favorable attributes like its hardy nature, high oil recovery and quality of oil, etc. As the acid value of this 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 karanja oil which gives 907ml of karanja oil methyl ester (KOME) and 109ml of glycerol using methanol (13%) and sodium hydroxide as a catalyst (1%). The properties like density, viscosity, flash point, cloud point and pour point have been determined as per ASTM standards for accessing the fuel quality of KOME.

Keywords: Karanja oil, biodiesel, transesterification and esterification.

Introduction

Chemically biodiesel is referred as mono-alkyl esters of long chain fatty acid derived from renewable biological sources. It can be directly used in the compression ignition engine. Biodiesel fuel is a clean burning alternative fuel that comes from 100% renewable resources. Many people believe that Biodiesel is the fuel of the future. Sometimes it is also known as Biofuel. Biodiesel which is derived from triglycerides by transesterification and from the fatty acids by esterification has attracted considerable attention during the past decade as a renewable, biodegradable, eco-friendly and non-toxic fuel. Biodiesel is recently used as a substitute for petroleum based diesel due to environmental considerations and depletion of vital resources like petroleum and coal. The possible use of renewable resources as fuels and as a major feedstock for the chemical industry is currently gaining growth. Further as petroleum is a fast depleting natural resource, an alternative renewable way to petroleum is a necessity. Now serious efforts are being made on the production and utilization of biodiesel in India. A lot of research work has been carried out to use vegetable oil both in its neat form and modified form. Studies have shown that the usage of vegetable oils in neat form is possible but not preferable¹. The high viscosity of vegetable oils and the low volatility affects the atomization and spray pattern of fuel, leading to incomplete combustion and severe carbon

deposits, injector choking and piston ring sticking. The methods used to reduce viscosity are - blending with diesel, emulsification, pyrolysis, transesterification.

Among these, the transesterification is the commonly used commercial process to produce clean and environmental friendly fuel². Methyl / ethyl esters of sunflower oil^{3,4}, rice bran oil⁵, palm oil⁶, mahua oil⁷, jatropha oil^{8,9}, karanja oil¹⁰, soyabean oil¹¹, rapeseed oil¹² and rubber seed oil^{13,14} have been successfully tested on C.I. engines and their performance has been studied. The sunflower oil, soybean oil and palm oil are edible oils and also are expensive. Hence they are not suitable for use as feedstock for biodiesel production in economical way. Hence non-edible oil like karanja (*Pongamia pinnata*) is attractive due to easy availability and low cost production.

Although its heat of combustion is slightly lower than that of the petro-diesel, there is no engine adjustment necessary and there is no loss in efficiency¹⁵. Methyl esters are non-corrosive and are produced at low pressure and low temperature conditions. Concentrated (about 80%) glycerin is obtained as a by-product during transesterification process. Bradshaw¹⁶ stated that 4.8:1 molar ratio of methanol to vegetable oil leads to 98% conversion. He noted that ratio greater than 5.25:1 interfered with gravity separation of the glycerol and added useless expense to the separation. Freedman, et al.¹⁷ studied the effect

of molar ratio of methanol to oil and effect of changes in concentrations of tri-, di- and monoglyceride on ester yield. Freedman et. al.¹⁸ obtained the results for methanolysis of sunflower oil, in which the molar ratio varied from 6:1 to 1:1 and concluded that 98% conversion to ester was obtained at a molar ratio of 6:1. Biodiesel from karanja oil shows no corrosion on piston metal and piston liner whereas biodiesel from jatropha curcas has slight corrosive effect on piston liner¹⁹. In the present investigation, biodiesel was prepared from karanja oil and its properties were analyzed to a certain its suitability as biodiesel.

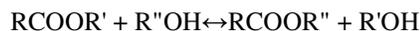
Benefits of Biodiesel: i. Easy to handle, ii. No need of modification in diesel engine, iii. Low carbon emission, iv. It is renewable energy source, v. Easy to blend with petrodiesel fuel, vi. High cetane number, vii. High lubricity, viii. Environment friendly, ix. It is a future fuel, x. Biodegradable and non-toxics.

Pongamia pinnata, a Renewable and Sustainable Source of Biodiesel: At a time when society is becoming increasingly aware of the declining reserves of oil for the production of fossile fuels, it has become apparent that biofuels are destined to make a substantial contribution to the future energy demands of the domestic and industrial economies. Pongamia pinnata will impact most significantly through the extraction of seed oil for use in the manufacture of biodiesel. The potential of P. pinnata oil as a source of fuel for the biodiesel industry is well recognized^{20,21,22}. Moreover, the use of vegetable oils from plants such as P. pinnata has the potential to provide an environmentally acceptable fuel, the production of which is greenhouse gas neutral, with reductions in current diesel engine emissions²³. Importantly, the successful adoption of biofuels is reliant on the supply of feedstock from non-food crops with the capacity to grow on marginal land not destined to be used for the cultivation of food crops. In this regard P. pinnata is a strong candidate to contribute significant amounts of fuel feedstock, meeting both of these criteria. Existing feedstocks such as palm oil and canola are costly, making the production of biodiesel economically marginal. Sources such as tallow and waste oil from food outlets are seen as variable in availability and/or of low quality. Pongamia pinnata belongs to the sub family Fabaceae (Papilionaceae). It is also called Derris indica and Pongamia glabra. It is a medium sized evergreen tree with a spreading crown and a short bole. The tree is planted for shade

and is grown as ornamental tree. It is one of the few nitrogen fixing trees producing seeds containing 30-40% oil. The natural distribution is along coasts and river banks in lands and native to the Asian subcontinent. It is also cultivated along road sides, canal banks and open farm lands.

Material and Methods

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 water¹⁵ this process has been widely used to reduce the high viscosity of triglycerides. The transesterification reaction is represented by the general equation as below



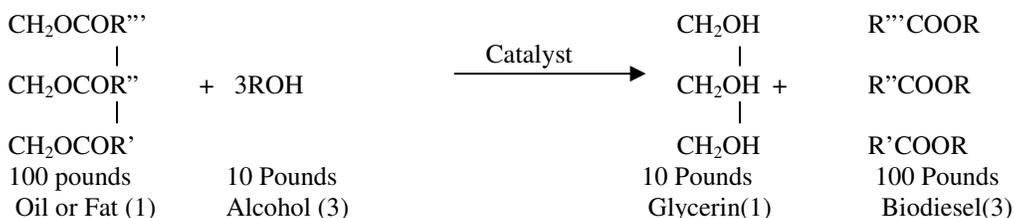
Scheme-1

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, do 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.

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 catalyst²⁴. 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 3.



Scheme-2

General equation for methanolysis of triglycerides

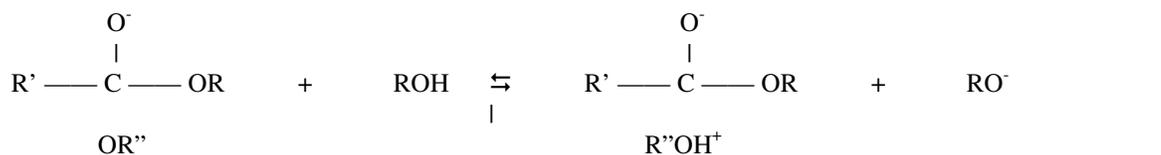
Reaction mechanism –



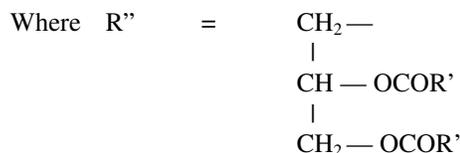
Step 1.



Step 2.



Step 3.



$\text{R}' =$ Carbon chain of fatty acid

$\text{R} =$ Alkyl group of Alcohol

Scheme-3
Mechanism of base catalyzed transesterification

Experimental set up: The experimental set up is shown in figure 1. 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.

Materials: i. Feedstock: karanja crude oil, ii. Acid catalyst: H_2SO_4 , iii. Base Catalyst: NaOH of 1% w/w of oil, iv. Reactant:

Methanol to oil ratio -molar ratio is 13%, v. Reactor vessel, vi. Electricity and timer.

Pretreatment: In this method, the karanja 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)²⁵. After this demoisurisation of oil we removed available wax, carbon residue, unsaponifiable matter and fiber. These are present in a very small quantity and carried out some important tests of oil that are given in table-2.

Esterification: Karanja oil contains 6%- 20% (wt) free fatty acids²⁶⁻²⁹. The methyl ester is produced by chemically reacting karanja oil with an alcohol (methyl), in the presence of catalyst

(KOH). A two stage process is used for the transesterification of karanja oil^{30,31}. The first stage (acid catalyzed) of the process is to reduce the free fatty acids (FFA) content in karanja 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 karanja 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 700 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 completion³². After dewatering the esterified oil was fed to the transesterification process³³

Transesterification- Base catalyzed reaction: Mixing of alcohol and catalyst: The catalyst used is typically sodium hydroxide (NaOH) with 1% of total quantity of oil mass. It is dissolved in the 13% of distilled methanol (CH₃OH) using a standard agitator at 700 rpm speed for 20 minutes. The alcohol - catalyst solution was prepared freshly in order to maintain the catalytic activity and prevent the moisture absorbance. After completion it is slowly charged into preheated esterified oil. The quantity of chemicals used and time for both the reaction are given in observation table-1

Transesterification Reaction: When the methoxide was added to oil, the system was closed to prevent the loss of alcohol as well as to prevent the moisture. The temperature of reaction mix was maintained at 60 to 65°C (that is near to the boiling point of methyl alcohol) to speed up the reaction. The recommended reaction time is 70 min. The stirring speed is maintained at 560-700rpm. Excess alcohol is normally used to ensure total conversion of the fat or oil to its esters. The reaction mixture was taken each after 20 min. for analysis of FFA. After the confirmation of completion of methyl ester formation, the

heating was stopped and the products were cooled and transferred to separating funnel.

Settling and Separation: Once the reaction is complete, it is allowed for settling for 8-10 hours in separating funnel. At this stage two major product obtained that are glycerin and biodiesel. Each has a substantial amount of the excess methanol that was used in the reaction. The glycerin phase is much denser than biodiesel phase and is settled down while biodiesel floted up. The two can be gravity separated with glycerin simply drawn off the bottom of the settling vessel. The amount of transesterified karanja biodiesel (KOME) and glycerine by this process are given in table-3.

Alcohol Removal: Once the glycerin and biodiesel phases were been separated, the excess alcohol in each phase was removed by distillation. In either case, the alcohol is recovered using distillation equipment and is re-used. Care must be taken to ensure no water accumulates in the recovered alcohol stream.

Methyl Ester Wash: Once separated from the glycerin and alcohol removal, the crude biodiesel was purified by washing gently with warm water to remove residual catalyst or soaps. The biodiesel was washed by air bubbling method up to the clear water was drained out. This shows the impurities present in biodiesel was removed completely.

Dring of Biodiesel: This is normally the end of the production process to remove water present in the biodiesel which results in a clear amber-yellow liquid with a viscosity similar to petro diesel. In some systems the biodiesel is distilled in an additional step to remove small amounts of color bodies to produce a colorless biodiesel.

Product quality: Prior to use a commercial fuel, the finished biodiesel must be analyzed using sophisticated analytical equipment to ensure it meets ASTM specifications. The most important aspects of biodiesel production to ensure trouble free operation in diesel engines are: complete reaction, removal of glycerin, removal of catalyst, removal of alcohol, absence of free fatty acids.

**Table-1- Observation table
Oil taken for reaction = 1 Lit. Karanja oil**

Esterification Reaction			
Reaction time	Reaction temperature	chemicals	quantity
60- 80min.	60 -65°C	H ₂ SO ₄	0.5%
		Methanol	13%
Transesterification Reaction			
After esterification to check Fatty acid level (<1 % suitable for transesterification).			
60- 65 min.	60 -65°C	NaOH	1%
		Methanol	13%

Process flowchart¹⁵

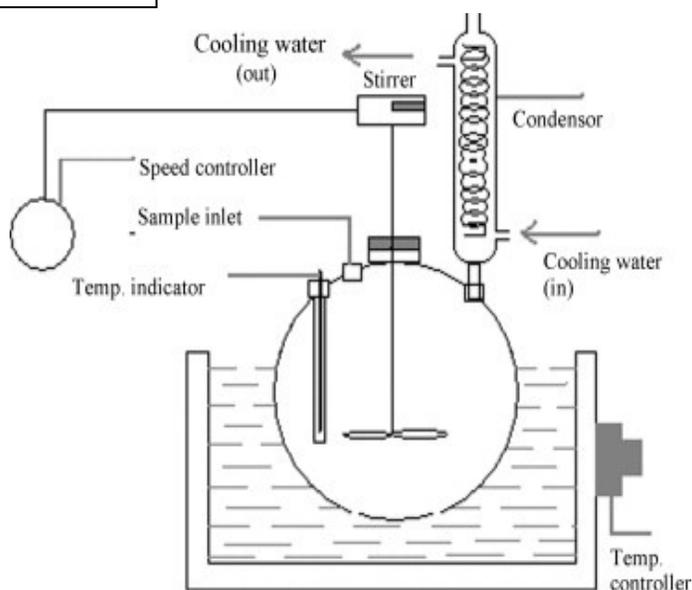
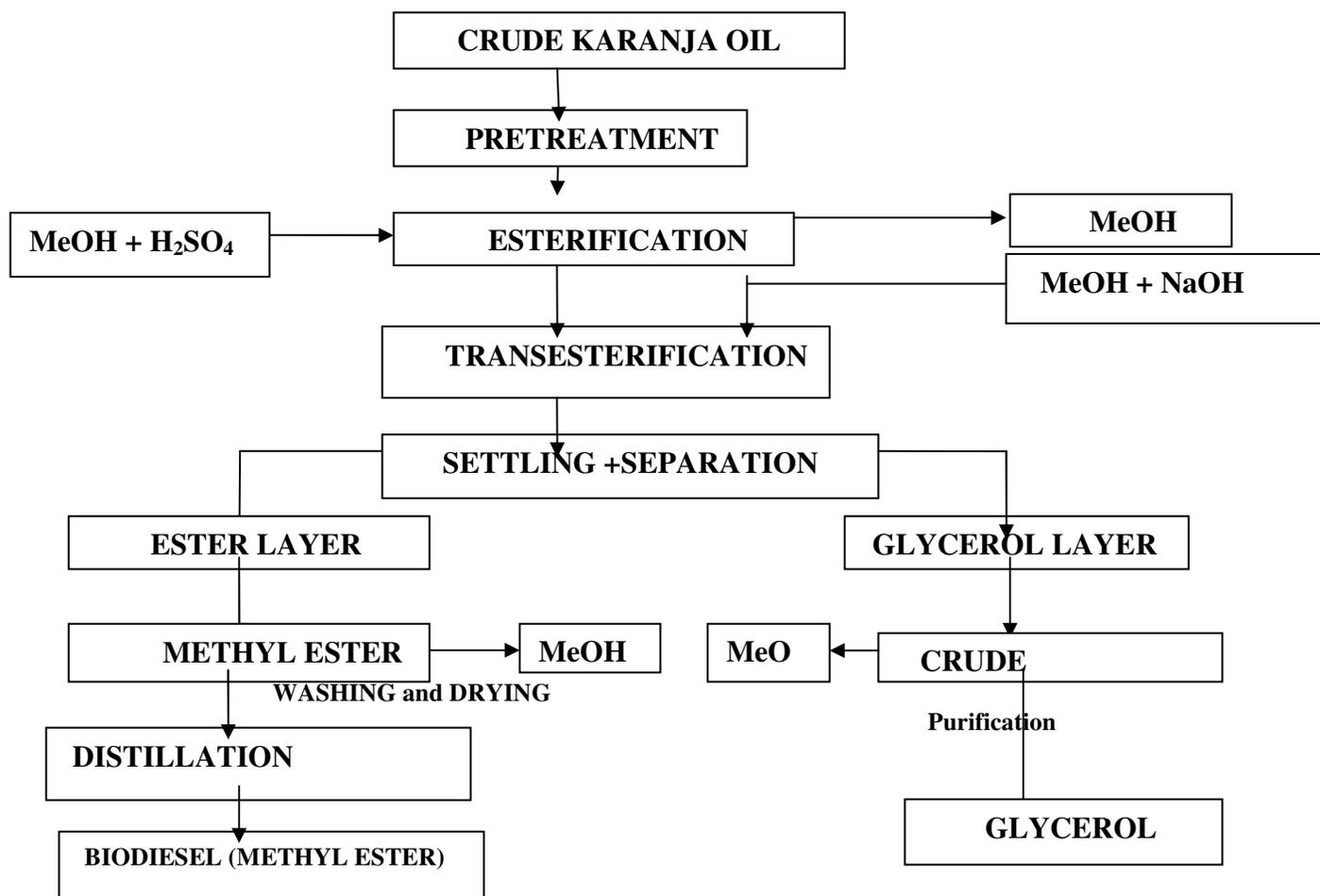


Figure-1
Experimental setup for preparation of karanja methyl ester (Biodiesel)

Results and Discussion

Oil extraction: The extraction of oil from karanja seeds were done by Mechanical expelling method, yield obtained 24 %.

Physico-chemical properties of karanja oil: Extracted karanja oil consisted of 94% pure triglyceride esters and the rest were free fatty acids and lipid associates, particularly flavonoids, which is measure of unsaponifiable matter. The physicochemical properties were determined as per BIS and ASTM method and the results were reported in table-2.

Table-2
Physico-chemical properties of karanja crude oil

Properties	Unit	Test values
Density	gm/cc	0.927
Kinematic Viscosity @ 40°C	mm ² /s	40.2
Acid value	mgKOH/gm	5.40
Pour point	°C	6
Cloud point	°C	3.5
Flash point	°C	225
Calorific value	MJ/Kg	8742
Saponification value		184
Carbon residue	wt%	1.51
Specific gravity		0.936
Copper corrosion test		No

Free fatty acid present in karanja oil: Extracted oil consisted of 94.09% pure triglyceride and rests were free fatty acids and lipid associates, which is the measure of unsaponifiable matter.

Table-2
The Fatty acid composition of karanja oil

Fatty acid name	Molecular formula	Composition (%)
Palmitic acid	C ₁₆ H ₃₂ O ₆	11.65
Stearic acid	C ₁₈ H ₃₆ O ₂	7.50
Tetracosanoic acid	C ₂₄ H ₄₈ O ₂	1.09
Dosocasoic acid	C ₂₂ H ₄₄ O ₂	4.45
Oleic acid	C ₁₈ H ₃₄ O ₂	51.59
Linoleic acid	C ₁₈ H ₃₂ O ₂	16.64
Eicosanoic acid	C ₂₀ H ₄₀ O ₂	1.35

Table-3
Biodiesel Recovery in Transesterification Reaction

Particulars	Karanja oil (ml)	
	Biodiesel	Glycerine
Batch I	910	105
Batch II	905	113
Batch III	908	109
Mean	907	109

Biodiesel recovery in Transesterification Reaction: From the table-3 it is clear that, the per liter biodiesel recovery from karanja oil was 907ml and the glycerine recovery per liter karanja oil was 109ml. The biodiesel recovery by using developed biodiesel processor was nearly 90%. Difference was

observed in the amount of glycerin and methyl ester is due to the quality of feedstock oil.

Karanja methyl ester Properties as per ASTM Standard: Table-4 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 product of incomplete transesterification and separation may produce biodiesel of low quality. Thus the reaction should be completed and the glycerol and methyl ester layers should be separated completely and also the flavonoids are to be removed from the product. Thus the reaction condition needs to be optimized in order to get high yield of biodiesel and also complete reaction. Also the reaction depends upon the raw materials. For base catalysed transesterification reaction, all the substances should be anhydrous and FFA content of oil should be low.

Table-4
Comparison of karanja biodiesel (KOME) with Diesel fuel

Properties	Unit	Karanja methyl ester	Diese l
Density	gm/cc	0.860	0.840
Kinematic Viscosity @ 40°C	Cst	4.78	2.98
Acid value	mgKOH/gm	0.42	0.35
Free glycerin	wt%	0.015	-----
Cloud point	°C	6	-16
Flash point	°C	144	74
Cetane number		41.7	49.0
Calorific value	Kcal/KG	3700	4285
Iodine value		91	-----
Saponification value		187	-----
Moisture	%	0.02	0.02
Carbon residue	%	0.005	0.01
Ash content	wt %	0.005	0.02

The above listed fuel properties from experimental results indicate that the karanja oil methyl ester (KOME) 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 karanja 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 karanja. 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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