# Production, Properties and Improvement of Cold-Flow Properties of Karanja Biodiesel Blends

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- **ABSTRACT**

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 for biodiesel fuel production have been developed, among which transesterification using alkali as 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 a number of countries. In India, non-edible oils like karanja oil and jatropha oil are available in abundance, which can be converted to biodiesel. In the present studies, biodiesel has been prepared from karanja oil. As the acid values of this oil were more than 3, hence it can be converted to biodiesel by esterification followed by transesterification process. The methyl ester produced by these methods was analyzed to acertain their suitability as diesel fuels. Then the comparison of physicochemical properties of karanja oil, karanja oil methyl ester and biodiesel was done.The various properties of methyl esters are found to be comparable with that of diesel fuel. However it requires further studies for considering the product as a suitable biodiesel. Biodiesel fuel has become more attractive recently because of its environmental benefits and the fact that it is a product made from renewable resources. However the less favorable cold flow properties or the low temperature

operability of biodiesel fuel compared to conventional diesel is a major drawback limiting its use. The poor flow properties of biodiesel at cold temperatures are mainly due to biodiesel fuel being composed of long-chain fatty acids with an alcohol molecule attached. If the double bond of unsaturated fatty acids in these long-chain fatty acids could be ruptured selectively, then the cold flow properties of biodiesel fuel would be enhanced by reducing its viscosity.

**Keywords***: Karanja oil, Biodiesel, Esterification, Transesterification, Cloud point, Pour point and Kinematic Viscosity

## NOMENCLATURE

KBD                  Karanja biodiesel

KO                   Karanj oil

PD                   Petroleum diesel

PMA                  polymethacrylate

PAO                  poly alpha olefin

## 1. INTRODUCTION:

Biodiesel is defined as the fatty acid alkyl esters of vegetable oil, animal fat or waste oils. It is a technically competitive and environmentally friendly alternative to conventional fossil-derived diesel fuel for use in compression-ignition engines. Biodiesel is biodegradable, non-toxic, possesses inherent lubricity and  emissions in comparison to regular diesel. Although pure biodiesel can be used in modern unmodified diesel engines, it is more commonly encountered as a blend component in petrodiesel such as B20 (20% biodiesel by volume). Currently, blends of up to B5 are allowed in IS: 15607:2005, the diesel fuel standard. In addition, IS: 15607:2005 was recently adopted for petrodiesel blends from B6 to B20. Biodiesel must be certified under IS: 15607:2005, the B100 standard, before it may be used as a fuel or blend component.

The main process for obtaining biodiesel is the transesterification reaction[1,2,3,4], which consists of an alcoholysis of triglycerides to obtain methyl esters and glycerol as a byproduct. Transesterification does not alter the fatty acid composition of the feedstock, but this composition plays an important role in influencing certain Critical parameters of the biodiesel. The types of vegetable oils used for biodiesel production may vary according to climate and the availability of raw material. Generally, the most abundant vegetable oil in a particular region is the most commonly used feedstock for biodiesel production**.**

Biodiesel is susceptible to start-up and performance problems when vehicles and fuel systems are subjected to cold temperatures. In winter, crystallization of high melting saturated fatty acid methyl esters may lead to the plugging of filters and tubes. Cold flow properties of diesel fuel are usually characterized by the following three temperature measures: cloud point (CP), cold filter plugging point (CFPP) and pour point (PP). Initially, cooling temperatures cause the formation of solid wax crystal nuclei that are submicron in scale and invisible to the human

eye. Further decreases in temperature cause the crystal nuclei to grow**.** The temperature at which crystals become visible is defined as the cloud point (CP) because the crystals usually form a cloudy or hazy suspension. The temperature at which crystal agglomeration is sufficiently extensive to prevent free pouring of fluid is defined as pour point (PP). The cold filter plugging point (CFPP) is then defined as the lowest temperature at which 40 mL of oil safely passes through the filter within 60 s.

Previous studies concerning the cold flow properties of biodiesel have determined that the lengths of the hydrocarbon chains and the presence of unsaturated structures significantly affect its low-temperature properties [5]. Peanut oil contains higher concentrations of long-chain saturated compounds (arachidic, behenic and lignoceric) than other oils [6]. The crystallization of these compounds may lead to plugged filters and tubes.

The nature of biodiesel suggests that crystallization fractionation may be a useful technique for decreasing CP by reducing the saturated alkyl ester content. Crystallization fractionation involves the separation of the components of lipids (vegetable oils, fats, fatty acids, fatty acid esters, monodiglycerides and other derivatives) based on differences in crystallization temperatures. The traditional fractionation process includes two stages. The crystallization stage consists of selective nucleation and crystal growth under a strictly controlled cooling rate combined with gentle agitation. Once well-defined crystals with a narrow distribution of specific sizes and characteristics are formed, the resulting slurry is transferred to the second stage for separation into solid and liquid fractions, typically by filtration or centrifugation**.** Three oil fractionation processes have led to industrial applications: dry fractionation through batch crystallization of the oil by controlled cooling and subsequent continuous filtration on a belt filter; lanza fractionation through batch crystallization of the oil under controlled cooling and

separation of the fractions by centrifugation after the addition of a surface active agent; and solvent fractionation through continuous crystallization of the oil in a solvent followed by separation of the liquid and the solid fraction through a continuous drum filter. Two of these processes, namely dry and solvent fractionation, have been used in studies of biodiesel. Winterization has also been employed to reduce the pour point of biodiesel by lowering its saturated fatty acid methyl ester components. To achieve significant reductions in pour point, several winterization steps are required to achieve a yield of higher than 25–26% and thereby render this technique viable. The other option is winterizing methyl esters in various solvents. Because of the poor yields of the winterized unblended methyl esters from common oils, researchers have attempted to dilute the esters with various solvents. Methanol, acetone, chloroform, and hexane have been explored as diluting solvents. Methanol offers the advantage that the winterization may be easily integrated into the industrial biodiesel production facilities [7].

While most of the properties of the biodiesel are comparable to petroleum based diesel fuel, improvement of its low temperature flow characteristic still remains one of the major challenges while using biodiesel as an alternative fuel for diesel engines. The biodiesel fuels derived from fats or oils with significant amounts of saturated fatty compounds will display higher cloud points and pour point.

The cloud point, which usually occurs at a higher temperature than the pour point, is the temperature at which a liquid fatty material becomes cloudy due to the formation of crystals and solidification of saturates. Crystallization of the saturated fatty acid methyl ester components of the biodiesel during cold seasons causes fuel starvation and operability problems as solidified material clog fuel lines and filters. With decreasing temperature more solids form and materials

approaches the pour point, the lowest temperature at which it will cease to flow. It has been well established that the presence of higher amount of saturated components increases the cloud point and pour point of biodiesel.

One of the major technical obstacles confronting the use of the biodiesel is its poor cold-flow properties in comparison with petroleum-based diesel. The cloud point and pour point of biodiesel are 15-25°C higher than that of petroleum-based diesel. Methyl and ethyl esters of soybean oil will crystallize and separate from diesel fuel at temperature often encountered in winter time operation. So the formation of crystals at relatively high temperature can plug the fuel lines and filters causing problems in fuel pumping and engine performance during winter operation.

Consider the case of petroleum middle distillates[8,9]. As ambient temperature cools to a point slightly higher than the cloud point, n-paraffin's ($C_{18}$- $C_{30}$ n-alkenes) in the fuel reach their saturation temperature. Under this condition, the fuels become a suspension of wax crystals in a mixture of shorter-chained n-alkenes, olefins, and aromatics. Although the crystals are initially submicron in size and invisible to the human eye, they grow in the size as temperature drops further. When the particle size reaches 0.5 µm, the crystals become visible, and the temperature at this point is defend as the cloud point. If unchecked, the crystals continue to the grow into large flat plate like structures. As temperature drops below the cloud point, the crystals become large enough (.5-1 mm) to fuse together into large agglomerates. These results in restricted flow though fuel lines and blocked filters and eventually in fuel starvation and stalled engines. In general, petroleum-based diesel develops operability problems when ambient temperature drops to a level between cloud point and pour point of diesel. With respect to long-chain fatty acid methyl esters, the problem is further aggravated because their cloud point is significantly higher

than that of petroleum-based diesel [10]. As temperature approaches freezing point, crystals large enough to cloud the mixture become visible. These crystals are primarily composed of methyl octadecanoate. The growth of the crystals depends on the composition of saturated methyl esters other than ocatadecanoate (E.g. hexadecanoate) and morphology relative to the nature of remaining liquid portion of the mixture. Another complication for the methyl esters may result in the relatively small difference between cloud points eg for methyl soyate, pour point is only approximately 2˚C below cloud point).

The viscosity of a fluid plays a major role in its pumping and flow within an engine. Generally, methyl esters have a Newtonin behavior at typical working temperature. However methyl esters from soybean and mustered oils present a pseudolastic behavior when temperature reaches values below 5 $^{0}$C.It is proposed to examine the potential of processing of biodiesel by winterization to improve it low temperature properties of pure biodiesel and its blends with petroleum-based diesel by evaluating yield and comparing cloud point and pour point data. Use of additive- with respect to petroleum distillates cold flow improvers significantly alter the size and habit of wax crystal formation, producing smaller and more compact crystal. When these crystals become trapped in the fuel filter, they form permeable cake layer that allows liquid fuel to flow to the injectors. This study will examine the suitability of additives marketed for petroleum distillates to improve low-temperature properties of biodiesel and its blends with petroleum-based diesel by standard petroleum industry methodologies, including cloud point, pour point, and kinematic viscosity.

The present work deals with influence of chemical additives PMA (polymethacrylate) and PAO (poly alpha olefin) on the flow properties of biodiesel made from Jatropha oil and its blends in petroleum based diesel fuels and anhydrous ethanol at low temperatures. The effects of

these chemical additives on the cloud point, pour point and kinematic viscosity of biodiesel and its blends in petroleum based diesel fuels at low temperatures in the range of 25-0 °C have been tabulated and discussed.

## 2 EXPERIMENTAL SECTIONS

### 2.1 Materials

Karanja oil was purchased from M/s Jatropha Vikas Sansthan, Delhi. Methanol 99.9% (LR grade) and sodium hydroxide 99.8 (AR grade) were obtained from M/s Ranbaxy Laboratories Limited, Delhi. Petroleum diesel was purchased from Indian Oil Corporation Depot, Kanpur. Chemical additives PMA and PAO were purchased from the Pawan & Company, Delhi. The characteristics of these additives and Karanja and jatropha oils and its blends are given Table 1 and 2.

**Table1. Characteristics of PMA and PAO [11]**

| Characteristics | Value for | |
|---|---|---|
| | PMA | PAO |
| Density, kg/m$^3$ | 0.867 | 0.896 |
| Molecular weight, kg/kmol | 22600 | 1875 |
| Pour point, °C | -6 | -9 |

**Table 2 Characteristics of Jatropha and Karanja Oils and their biodiesel**

| Characteristics | Jatropha oil | Karanja oil | Biodiesel prepared from | |
|---|---|---|---|---|
| | | | Jatropha oil | Karanja oil |
| Free fatty acid, wt.% as oleic acid | 5.2 | 2.5 | 0.31 | 0.42 |
| Cloud point, °C | 2 | 5 | 3 | -1 |

| Pour point, °C | 1 | 1 | -5 | -7 |
|---|---|---|---|---|
| Kinematic viscosity at 40°C, mm²/s [13] | 32.64 | 15.2 | 3.41 | 1.52 |
| Acid value, mg/KOH/g | 5.19 | 4.25 | 0.81 | 0.61 |

## 2.2 Preparation of Biodiesel

## 2.3 Acid -Catalyzed Esterification Process

The free fatty acid present in Jatropha oil is esterified with methanol in presence of sulfuric acid as catalyst. For esterification 6:1 mole ratio of methanol to oil has been used with Sulphuric acid (1% based on the oil weight) and reaction was carried out at temperature 60°C for two hours, contents were allowed to cool and transferred to separating funnel for overnight. Methanol-water layer formed at the top was removed. The FFA content of esterified product was determined by standard titrimetry method. The acid value of the esterified product separated at the bottom was also determined. This should be below 2 mg KOH/ g for the use in transesterification process otherwise soap formation occurs.

## 2.4 Base-Catalyzed Transesterification Process

After acid pretreatment conditions generate for the use of methanol to oil with a molar ratio 6:1 in the presence of 0.55% w/v KOH as catalyst. The esterified product from previous step was poured into the flask and heated at 50 °C. The solution of KOH in methanol based on oil weight is heated to 50 °C before addition and then added to heated oil. Excess alcohol was normally used to ensure total conversion of the fat or oil to its esters. The reaction mixture is heated and stirred at 60-65 °C and 400 rpm for two hours. After completion of reaction the contents were cooled and transferred to separating funnel. The product was allowed to settle overnight before

separation of glycerol layer from methyl ester layer of fatty acids on top. KBD is separated and washed with distilled water to remove alkali (Phenolphthalein test). KBD mixture was then dried with anhydrous sodium sulphate followed by filtration. The mixture was distilled to remove unreacted methanol. The residue in the flask was only KBD. This KBD was tested cloud point, pour point and kinematic viscosity.

The transesterification reaction is represented by the general equation as [1]:

$$RCOOR' + R''OH \leftrightarrow RCOOR'' + R'OH$$
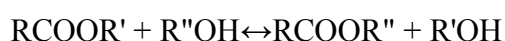
If methane is used in this process it is called methanolysis. Methanolysis of glyceride is represented:
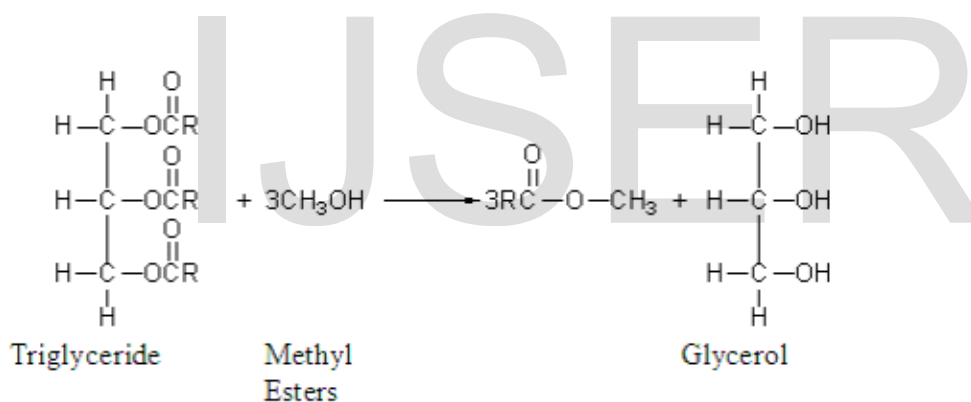


**Scheme 1 : Formation of Methyl Ester from Triglycerides**

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

The mechanism of alkali-catalysed transesterification is described below. The first step involves the attack of the alkoxide ion to the carbonyl carbon of the triglyceride molecule,

$$ROH \;+\; B \;\rightleftharpoons\; RO^- \;+\; BH^+ \qquad (1)$$

$$(2)$$

$$(3)$$

$$(4)$$

**Scheme 2: Mechanism of base catalysed Transesterification**

This results in the formation of a 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

applicable to di-glyceride and monoglyceride.

## 2.5 Preparation of Blends

Biodiesel produced in the laboratory has been used for the preparation of its blends with

petroleum based diesel by blending technique. Biodiesel and petroleum-based diesel will be

blending in a conical flask under continuous stirring to ensure uniform mixing at 45 ˚C for 45 to

60 minute. These blends are on volume basis and they will be stored in amber glass bottles.

### 2.5.1 Preparation of biodiesel-diesel-additive blends

Karanja biodiesel was mixed with cold-flow improvers (the additive purchased from the Pawan & company Delhi) before adding it to diesel. The appropriate amount of Karanja biodiesel were measured in masses of 20, 15, 10 and 5 ml and placed into glass flask. The appropriate amounts of cold-flow improver were added to each flask (0, 0.25, 0.5, 0.75 and 1g). Biodiesel petroleum-based diesel-additive will be blending in a conical flask under continuous stirring to ensure uniform mixing at 45 °C for 45 to 60 minute.

## 3. Determination of Flow Properties:

### 3.1 Cloud Point [12]

Cloud point is defined as the temperature at which a cloud of crystal (of wax or esters) first appears in a liquid when it is cooled under controlled conditions during a standard test. The same cooling procedure as described in IS: 1448 [P: 10] was followed; the sample were examined at intervals of 1 °C, until any cloud was observed at the bottom of the test jar. The cloud point was reported as a multiple of 1°C.

### 3.2 Pour Point [12]

IS: 1448[P: 10] pour point procedure that is least 9 °C the expected pour point. The sample was immersed into an -18 °C cooling bath. If the sample had not ceased to flow when its temperature has cooled to -6 °C the sample was then transferred to – 30 °C cooling bath.

Reading was taken for every 3 °C decrease in the temperature, until the sample totally ceased flow (the sample was held in a horizontal position for 5 s). Reading of the test thermometer was taken and 3 °C was added to the temperature recorded as the result of the **IS: 1448[P: 10]** pour point.

### 3.3 Kinematic viscosity determination [13]

Kinematic viscosity ($\upsilon$, mm/s$^{-1}$) was measured with a Saybolt viscometer at 40 °C according

All experiment were run in triplicate (Table 1). To IS: 1448 (P: 25)-1976 [13]. All experiment were run in triplicate (Table2).

## 3.4 Acid Value and Free Fatty Acid Determination[15]

Acid value (AV, mg/KOH/g Sample) titration were performed as described in the IS: 548-1964 Part-1[15]. The titration end point was determined by the instrument and visually verified using a phenolphthalein indicator. FFA, wt% as oleic acid as described in the IS: 548-1964 Part-1[15]. Experiment were run in duplicate and mean value are reported (Table 2).

## 3.5 Cloud and pour point set up

The assembly used for the cloud and pour point measurement is shown schematically in fig. 1.The glass jar was immersed in an ice-salt bath. The glass test jar was thermally isolated from the polished brass cylinder by means of a cork support, stopper and ring assembly. The entire assembly was secured in the bath so as to isolate it from any vibration, because vibrations to the test sample may lead to low and erroneous results. Three times repeated for each sample to check the consistency of the results. The difference among all the three measurement was near more than 1k.
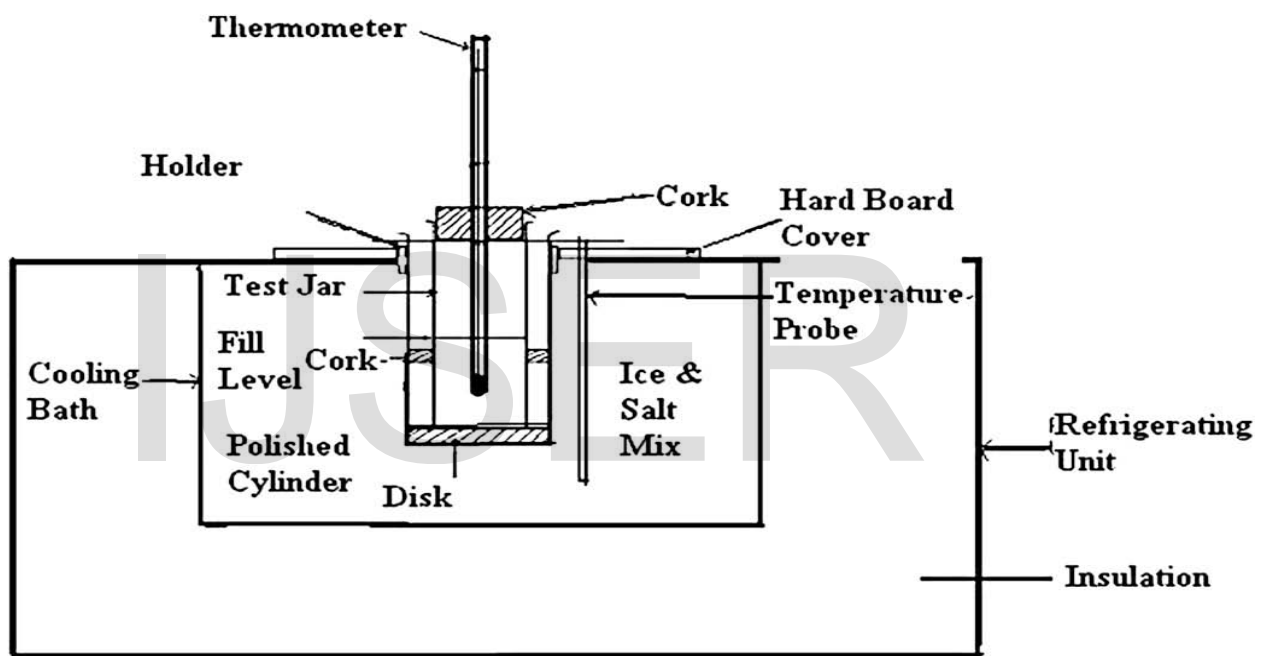
**Fig.1 Cloud and pour point set up**

## 4   RESULTS AND DISCUSSION:

### 4.1 Fatty Acid composition of Karanja oil:

The percentage composition of fatty acids present in karanja oil was determined by

GCMs and is represented in the table-3 below

| Fatty Acid | Structure and Molecular Formula | Fatty acid % |
|---|---|---|
| Palmitic | 16:0   $CH_3(CH2)_{14}C00H$ | 11.56 |
| Stearic | 18:0   $CH_3(CH2)_{16}COOH$ | 7.4 |
| Eicosanoic acid | 20:0    $CH_3(CH2)_{18}COOH$ | 1.35 |
| Dosocasnoic acid | 22:0   $CH_3(CH2)_{20}COOH$ | 4.45 |
| Tetracosanoic acid | 24:0   $CH_3(CH2)_{22}COOH$ | 1.08 |
| Oleic | 18:1    $CH_3(CH2)_{14}(CH=CH)COOH$ | 52.3 |
| Linoleic | 18:3  $CH_3(CH2)_{12}(CH=CH)2COOH$ | 16.46 |
| Residual | | 5.40 |

### 4.2 Comparison of fuel properties of Karanja oil, Karanja oil methyl ester and Diesel:

Comparison of fuel properties of karanja oil, karanja oil methyl ester was done with diesel and the results are shown in the table-4 given below:

**Table-4: Comparison of fuel properties of karanja oil, karanja oil methyl ester and diesel**

| Properties | Karanja oil | Karanja oil methyl ester | Diesel |
|---|---|---|---|
| Density@15kg/m$^3$ | 0.905 | 0.797 | 0.850 |
| Viscosity@40˚C, mm$^2$/s | 15.2 | 1.52 | 1.86 |
| Flash Point ˚C | 205 | 97.8 | 70.0 |
| Cloud Point ˚C | 5 | -1 | -1 |
| Pour Point ˚C | 1 | -7 | -18 |
| AcidValue mg/KOH/g | 4.25 | 0.81 | 0.35 |
| Iodine Value | 90.8 | 91 | ------- |
| Saponification Value | 189.7 | 180.0 | ------------ |
| Calorific Value  Kcal/kg | 8742 | 3712 | 4290 |
| Cetane Number | 38.0 | 42.9 | 46 |

Cloud point, pour point and kinematic viscosity studies were conducted for KBD blends with and without cold-flow improvers, in blends of KBD. Although most of the properties of biodiesel fuels are comparable with that of diesel fuel but cloud point and pour point indicate poor cold-flow behavior.

It can be seen Fig. 1 shows variation of cloud point of biodiesel blends from 0 to 20 Vol. %. PAO of average molecular weight of 1875 has been use as cold flow property improver. Its concentration has been varied from 0 to 1 g per 100 ml. At a given concentration of PAO cloud point increases with increment in biodiesel composition. For a given composition of biodiesel blends, Cloud point decreases with increment concentration of PAO.
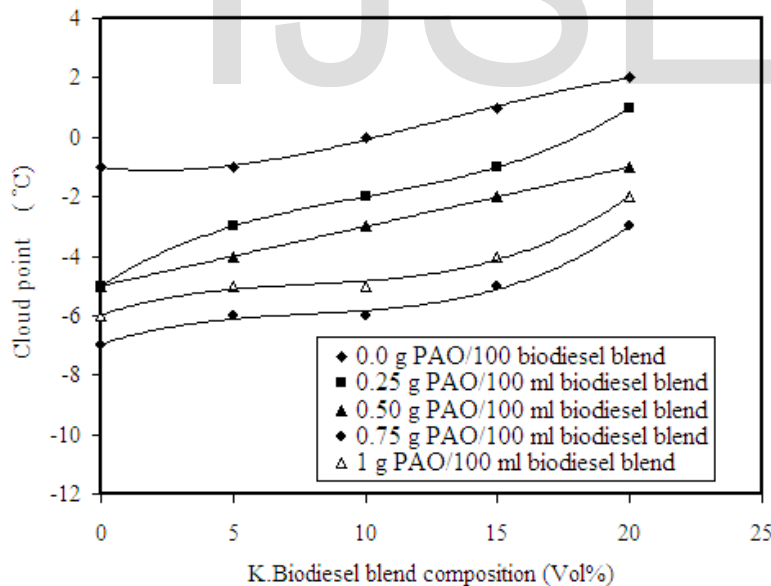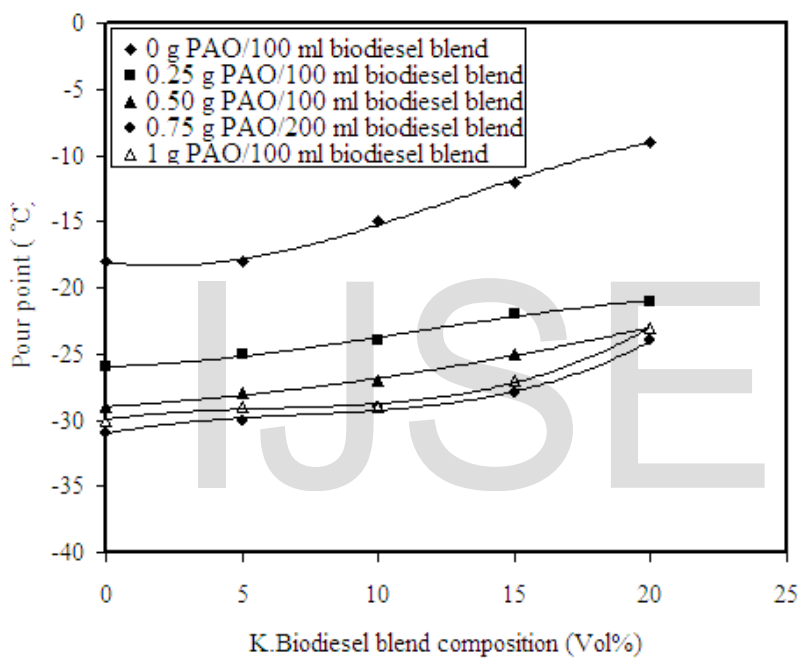


Fig.1 Variation of cloud point with biodiesel blend composition with PAO concentration as a parameter.

It can be seen Fig. 2 shows variation of pour point of biodiesel blends from 0 to 20 Vol. %. PAO of average molecular weight of 1875 has been use as cold flow property improver. Its concentration has been varied from 0 to 1 g per 100 a given concentration of PAO pour point increases with increment in biodiesel composition. For a given composition of biodiesel blends, pour point decreases with increment concentration of PAO.
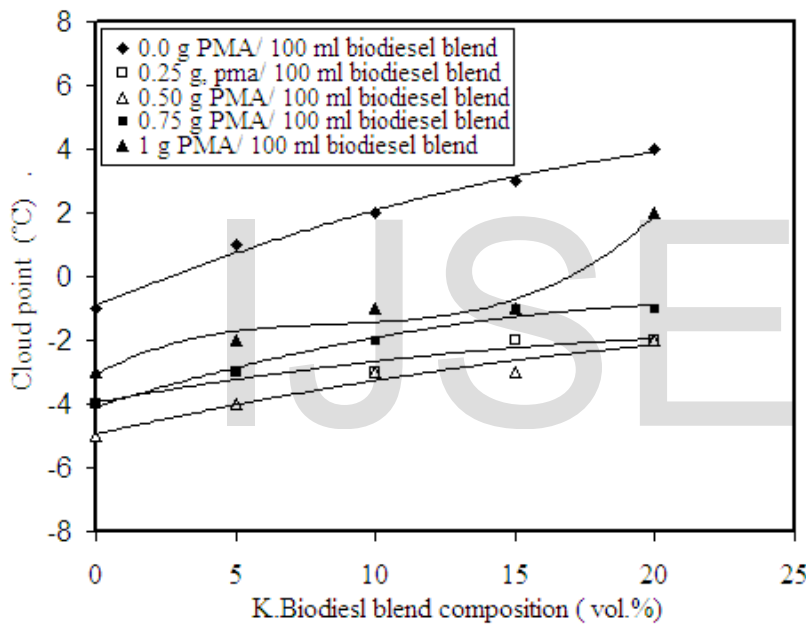


Fig. 2 Variation of pour point with biodiesel blend composition with PAO concentration as a parameter.

It can be seen Fig. 3 shows variation of cloud point of biodiesel blends from 0 to 20 Vol. %. PMA of average molecular weight of 22600 has been use as cold flow property improver. Its concentration has been varied from 0 to 1 g per 100 ml. At a given concentration of PMA cloud point increases with increment in biodiesel composition. For a given composition of biodiesel blends, Cloud point decreases with increment concentration of PMA.



Fig. 3 Variation of cloud point with biodiesel blend composition with PMA concentration as a parameter.

It can be seen Fig. 4 shows variation of pour point of biodiesel blends from 0 to 20 Vol. %. PMA of average molecular weight of 22600 has been use as cold flow property improver. Its concentration has been varied from 0 to 1 g per 100 ml. At a given concentration of PMA pour point increases with increment in biodiesel composition. For a given composition of biodiesel blends, pour point decreases with increment concentration of PMA.
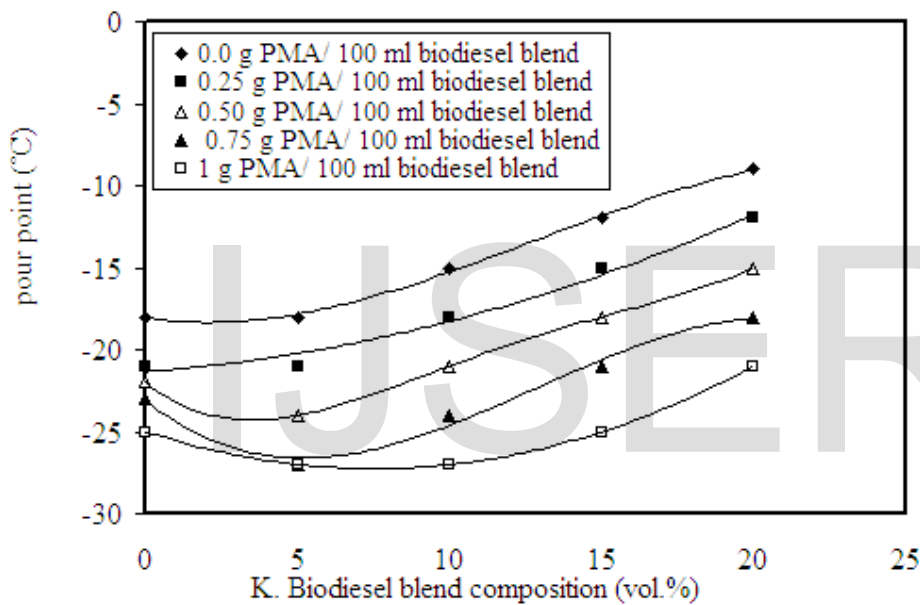


Fig.4 Variation of pour point with biodiesel blends composition with PMA concentration as a parameter.

From Figure [5] it can be seen that effect of PAO on the kinematic viscosity of jatropha biodiesel. Variation of kinematic viscosity of biodiesel blends from 0 to 20 Vol. %. PAO of average molecular weight of 1875 has been use as cold-flow property improver. Its concentration has been varied from 0 to 1g per 100 ml. At a given concentration of PAO kinematic viscosity increases with increment in biodiesel composition. For a given composition of biodiesel blends, kinematic viscosity increases with increment concentration of PAO.
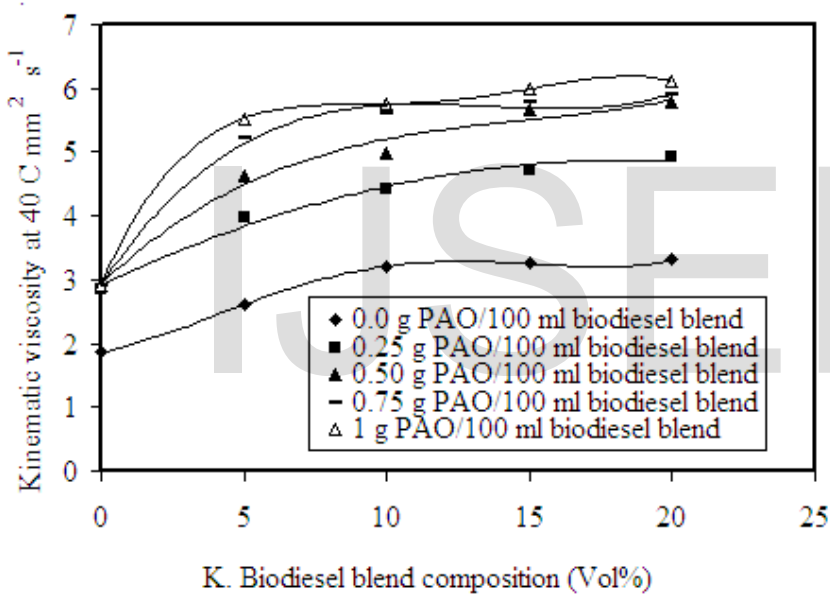


Fig. 5 Variation of kinematic viscosity with biodiesel blend composition with PAO concentration as a parameter

From Figure [6] it can be seen that effect of PMA on the kinematic viscosity of jatropha biodiesel. Variation of kinematic viscosity of biodiesel blends from 0 to 20 Vol. %. PMA of average molecular weight of 22600 has been use as cold-flow property improver. Its concentration has been varied from 0 to 1 g per 100 ml. At a given concentration of PMA kinematic viscosity increases with increment in biodiesel composition. For a given composition of biodiesel blends, kinematic viscosity increases with increment concentration of PMA.
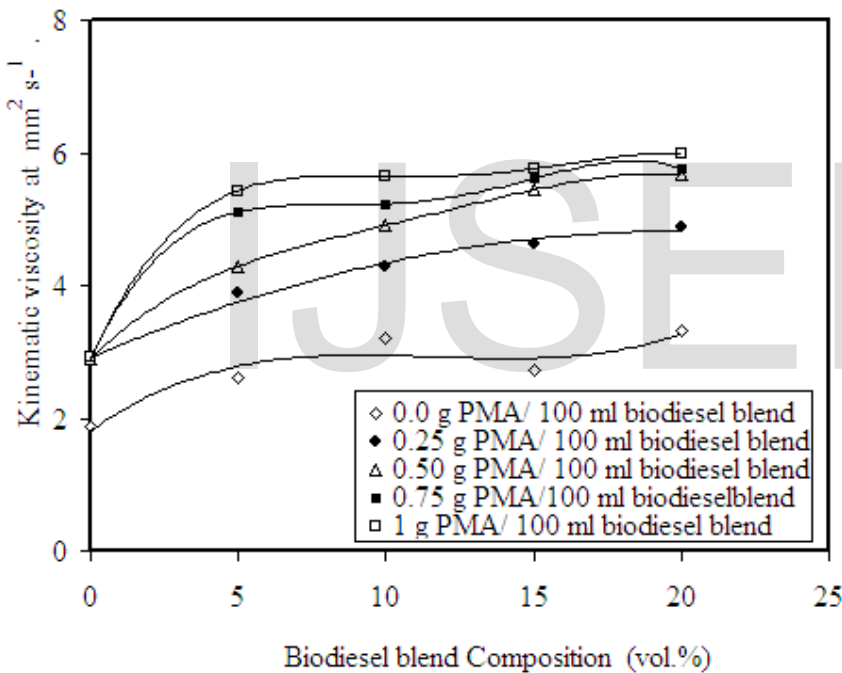


Fig. 6 Variation of Kinematic viscosity with biodiesel blend composition with PMA concentration as a parameter.

**Table 5: Sample total volume and blend uncertainties [14]**

| Blend | Total Volume(± Uncertainty) | B100 Volume Fraction(± Uncertainty) |
|---|---|---|
| B100 | 100± 0.5 | 1.0 |
| B20 | 100± 0.5 | 0.2±0.0050 |
| B15 | 100± 0.5 | 0.1±0.0050 |
| B10 | 100± 0.5 | 0.1±0.0020 |
| B5 | 100± 0.5 | 0.05±0.0012 |
| Diesel | 100± 0.5 | 0.0 |

**Table 5.Uncertainty in Additive g**

| Blend | PMA and PAO | | | |
|---|---|---|---|---|
| | 2.5 | 5.0 | 7.5 | 10 |
| B10 | ±1.0000 | ±0.0015 | ±1.0002 | ±1.0002 |

## 5. CONCLUSION:

Thus this study suggests that the karanja oils can be used as a source of triglycerides in the manufacture of biodiesel by esterification and or transesterification. 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 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. There has been a considerable interest in developing biodiesel as an alternative fuel in recent year due to its environmental benefits and because it is derived from renewable resources like vegetable oils or animal fats. However, the freezing and gelling of many biodiesel formulations are limiting their applications. Formulations that are high in the alkyl esters of saturation fatty acids, like the methyl esters of beef tallow, can gel up at temperature near $0~^0C$. Jatropha and Karanja-based methyl esters, the most common in the India will become cloudy and pour point at temperature near $-1^0$ C and $-5~^0C$, $-7~^0C$.

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