

# Characterization of Calophyllum Oil Biodiesel—Alternative Fuel to Diesel Engines



Rahul Krishnaji Bawane, Chetan Choudhary, A. Muthuraja,  
and G. N. Shelke

**Abstract** A preliminary investigation on the fitness of Calophyllum (non-edible) oil for the development of its fractions for biodiesel/Calophyllum Oil Methyl Ester and its characterization is carried out. The Calophyllum seeds considered, which shows the second-highest productivity (after palm) of 4680 kg oil per hectore. According to the oil yield, Calophyllum was identified as the future potential feedstock for biodiesel with the productivity of 4680 lit oil per hectore, the second-highest after palm oil. The highest biodiesel yield 67% was found at 1.5:1 oil to methoxide ratio at reaction temperature 50–60 °C among the ratio of 1:1, 1.5:1 and 2:1 for the temperature range 40–50, 50–60 and 60–70 °C. The investigation revealed that the fatty acid methyl ester of Calophyllum seed oil met all the major biodiesel requirements in the USA ASTM D 6751-02, ASTM PS 121-99, European Union EN 14214, Germany DIN V 51606 and Indian Standards IS 1448. And in fuel blend, the increase in content of biodiesel, there is increases in density, kinematic viscosity at the same time there is reduction in calorific value, cetane number, flash point and fire point.

**Keywords** Biodiesel · Blend · Calophyllum · Characterization · Transesterification · Yield

## 1 Introduction

Now a day government stringent emission regulations with adopting BS-IV emission norms which were already adopted in developed countries as Euro 6 norms to minimize the emissions from automobiles, also fuel crisis in the upcoming years enforced to search for unconventional sources of fuel. Many countries are critically dependent on petroleum fuels for agricultural machinery and transportation sector,

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R. K. Bawane (✉)

Department of Mechanical Engineering, Sandip University, Nashik, MS, India

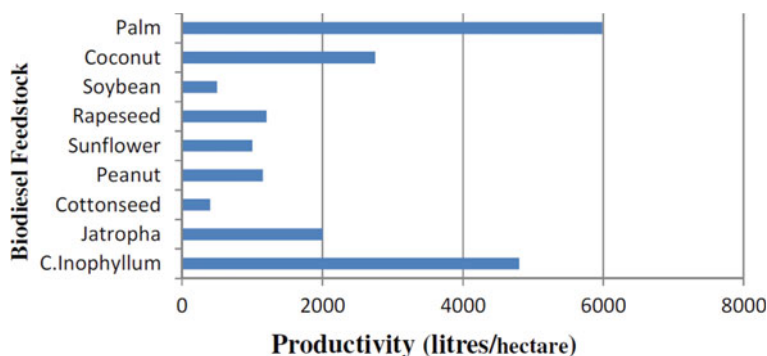
e-mail: [rahul.bawane@pccoer.in](mailto:rahul.bawane@pccoer.in)

C. Choudhary · A. Muthuraja · G. N. Shelke

Department of Mechanical Engineering, Sandip Institute of Engineering and Management, Sandip Foundation, Nashik, MS, India

few countries together geared up to the bulk of petroleum and this leads to high price variation and uncertainties in supply for the remaining countries. India mainly depends on the import of petroleum and about 2/3rd of its demand met through imports and the emissions from their use have an opposing impact on the environment and human health [1, 2]. In such a scenario, biomass is become known as one of the promising environmental friendly renewable energy substitutes, if the conventional energy sources, petroleum oils, coal, and gases happen to exhaust. This biomass treated thermo-chemically or biologically and converted into liquid and gaseous biofuels [3]. In the recent decade, biodiesels are gained importance and recognition due to their biodegradability, renewability, and environment-friendly [4]. However, biodiesel from such vegetable/crops inherently contains oxygen 10–45% by mass which is not available in the case of petroleum fuel. This promotes chemical reaction, better and complete combustion of biodiesel. Also, biodiesels reported very low sulfur content and many have a low nitrogen level which makes it more environmental friendly [2, 3]. There has been greater wakefulness on biodiesel in developing countries these days, along with boosting the economy, especially in rural areas. Biodiesel is a renewable and biodegradable fatty acid methyl ester extracted from various kinds of vegetable oil and animal fats. Edible oils to be used as fuel is not significant due to food crops are more expensive and terrific demand in food/cooking uses. Therefore researchers are made their research focused on extracting biodiesel from non-edible oil crops, non-edible oils established themselves as promising crude oils in the area of alternative fuel to the conventional petroleum diesel fuel [5]. Biodiesel has features like compatible with the diesel engine systems without any modification, also it can be blended with petroleum diesel fuel in any proportion and can be extended to neat biodiesel, and it shows a higher flash point, improved cetane number and lesser harmful emissions [1].

India is producing about  $6.73 \times 10^6$  tons of non-edible oils such as Linseed, Castor, Karanja, Neem, Palash, Kusum and many more other plant-based forests derived oils which may have a much higher production potential. The present work is focused on non-edible vegetable oil from *Calophyllum* and characterization and formulating its properties to study to see that the modified fuels can be utilized in the existing diesel engine without any substantial hardware modifications [6]. However, *Calophyllum* (Beauty Leaf Tree) is a large evergreen tree in the family Caryophyllaceae and widely available in countries like India, Australia, and East Asia. It is commonly cultivated in all tropical regions of the world. Because of its decorative leaves, fragrant flowers, and spreading crown, it is best known as an ornamental plant. This tree often grows in coastal regions, as well as nearby lowland forests. However, it has also been cultivated successfully in inland areas at moderate altitudes. It tolerates varied kinds of soil, coastal sand, clay, or even degraded soil. In India it is found in Andaman & Nicobar Islands, Lakshadweep, Karnataka, Kerala, Odisha, Maharashtra, and Tamil Nadu [6]. The average oil yield is 11.7 kg oil/tree which is about 4680 kg oil per hectore, which is second highest after palm (Fig. 1).

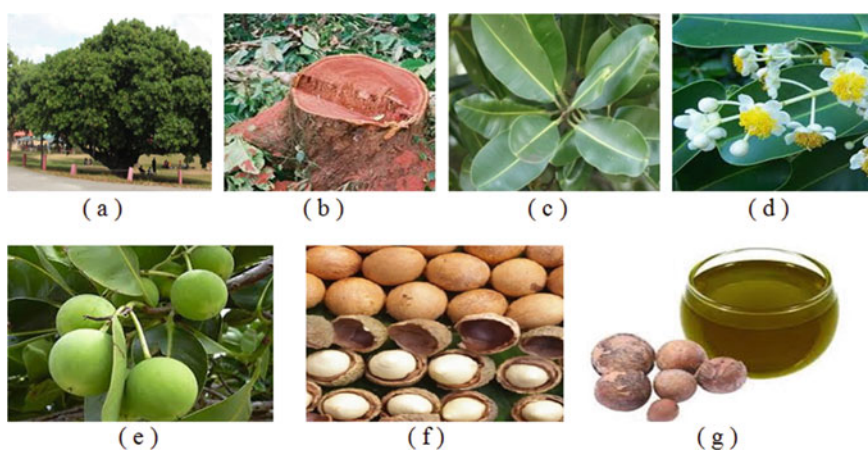


**Fig. 1** Biodiesel feedstock oil productivity yield

## 2 Material and Method

Calophyllum species extensively dispersed in tropical Asia (India, Srilanka, Thailand, Indonesia, Malaysia, Philippines), including the Hawaiian and other Pacific Islands [7]. In Maharashtra state Calophyllum available in the wild region of Kokan (Dapoli, Ratnagiri, Harihareshwar) [8], and seeds are collected mostly from May to August. The fresh seeds contain moisture 12%, therefore, fruits/seeds are dried in sunlight for 2–3 days. It was found that after drying oil yield is 1 lit per 100–150 seeds. These dried seeds are heated at 105 °C for 24 h, the kernel was then separated from the shell, and then oil yield is found to be around 70% [7, 8].

Calophyllum oil can be extracted from the seed kernel by either Full Press Extraction method wherein an electric powered screw press used or chemical oil extraction method wherein n-hexane as the solvent is used (Fig. 2).



**Fig. 2** Calophyllum and parts; **a** tree, **b** bark, **c** leaves, **d** flowers, **e** fruits, **f** seeds, **g** oil [4, 9]



**Fig. 3** Oil extraction; **a** Mechanical Screw Press, **b** chemical process [2, 3]

The Full Press Extraction Process where mechanical screw presses are used to extract the oil by squeezing the oil under high pressure. In the Chemical Oil Extraction Process where a solvent is mixed with the material to separate the oil, in this method, dried seed kernels were ground to a fine consistency, then this powder were put into conical flasks in which hexane was added at a ratio of two parts of hexane and one part of kernel powder, this mixture is then placed on the orbital mixer and left to run for at least eight hours. The hexane-oil mixture was collected, filtered and decanted into aluminum foil containers for solvent evaporation when hexane had been fully evaporated, the oil was transferred into containers for analysis [3] (Fig. 3).

The Full Press Extraction Process is generally used for the smaller capacity process, typically under 200 tons per day, and the Solvent Extraction Process is generally used for larger capacity systems.

The fresh extracted Calophyllum oil is greenish-yellow and it gets darkened during the storage and it having disagreeable odor and bitter taste (Table 1).

### 3 Experimentation and Procedure

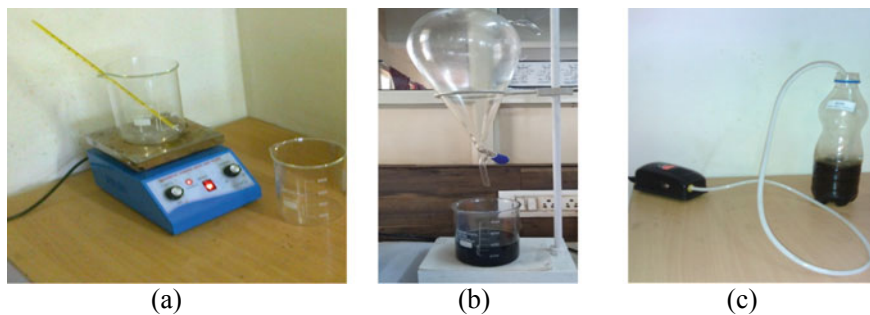
The Calophyllum Oil Methyl Ester (biodiesel) conversion is made by using transesterification of raw Calophyllum oil, and it is carried out as,

- Preparation of methoxide solution
- Sample preparation of raw Calophyllum oil with methoxide
- Reaction under Heating and continuous stirring
- Separation of biodiesel and glycerol
- Removal of excess methanol
- Biodiesel washing and drying
- Test Sample/Fuel Blends.

**Table 1** Characteristics of calophyllum oil

Description	ASTM method	Unit	Calophyllum oil				Author
			Jahirul et al. [2]	Chavan et al. [8]	Atabani et al. [10]	Sahoo and Das [11]	
Density at 40 °C	D1298	kg/m <sup>3</sup>	964	910	924.9	896	944.3
Kinematic viscosity at 40 °C	D445	cSt	56.74	38.17	55.478	71.98	65.29
Cloud point	D2500	°C	–	–	–	–	–
Pour point	D97	°C	–	–	–	–	9
Flash point	D93	°C	–	224	236.5	221	216
Calorific value	–	MJ/kg	38.10	32.50	38.51	39.25	38.37
Refractive index at 25 °C	–	°C	–	–	–	–	1.3–1.5
Iodine value	–	–	–	–	–	–	14.207
Saponification value	–	–	–	203	–	–	81.72
Peroxide value	–	–	–	–	–	–	4.01
Acid value	–	–	36.26	–	–	44	70 max

The raw/crude Calophyllum oil which is contributed by Keynote International, Mumbai, and as a catalyst is Potassium Hydroxide (KOH), Methanol. The expected product is Calophyllum Oil Methyl Ester and the by-product is Glycerol [12] (Fig. 4).



**Fig. 4** Transesterification setup; **a** hot plate with magnetic stirrer, **b** separating funnel, **c** air bubbler [12]

**Table 2** Test sample prepared [Author]

Sr. no.	Sample	Ratio	Description	Total mixture (ml)
1	Sample-1	1:1	1000 ml Calophyllum oil + 1000 ml Methoxide	2000
2	Sample-2	1.5:1	1500 ml Calophyllum oil + 1000 ml Methoxide	2500
2	Sample-3	2:1	2000 ml Calophyllum oil + 1000 ml Methoxide	3000

### 3.1 Preparation of Methoxide Solution

Most commonly NaOH or KOH are used as catalyst to speed up reaction and reduce transesterification time, and the concentration of the catalysts to methanol is maintained 0.5 to 1% by weight, as on increasing concentration it will raise the Total Acid Number also this reduces the yield of biodiesel conversion [13–15]. However, each crude oil transesterification reaction got its optimum catalyst concentration, if exceed then it participates in saponification which reacts with triglyceride to form soap and water and lead to a reduction in biodiesel yield.

In the present study, 0.1% by weight KOH catalyst is used, thus 0.79 gm of potassium hydroxide (KOH) pellets were added to 1 lit (0.791 kg) 99% pure methanol (CH<sub>3</sub>OH) and stirrer until KOH pellets get dissolved completely into methanol and form the Methoxide Solution.

### 3.2 Sample Preparation

Test sample were prepared where the raw Calophyllum oil is mixed with the methoxide solution in three different ratios as (Table 2).

### 3.3 Reaction Under Heating and Continuous Stirring

A raw Calophyllum oil is preheated up to 40 °C using a heating plate with a magnetic stirrer, and then methoxide solution is poured into it and continue stirring with heating for 90 min. under the varying temperature ranges from 45 ± 5, 55 ± 5, and 65 ± 5 °C. During the experiment, the author found below 40 °C and above 70 °C, the reaction could not complete to the level where the separation of biodiesel and glycerol take place. Table 3, shows sets of parameters used and findings of the experiments carried.

**Table 3** Characterization of biodiesel yield [Author]

Ratio	Temp. range (°C)	Total mixture (ml)	Yield	
			Biodiesel (ml)	%
1:1	40–50	2000	400	40
	50–60		550	55
	60–70		520	52
1.5:1	40–50	2500	530	35
	50–60		1000	67
	60–70		870	58
2:1	40–50	3000	300	15
	50–60		480	24
	60–70		370	18.5

### 3.4 Separation of Biodiesel and Glycerol

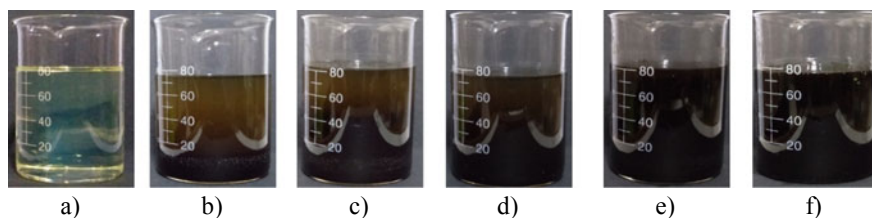
After transesterification reaction time 90 min. the mixture prepared is transferred to separating funnel to settle down, within 10 min. biodiesel separation is observed, but for complete separation, it took for 24 h. wherein the separated upper layer is of biodiesel and the bottom layer is of glycerol. Glycerol is separated out and thus biodiesel is collected for the further treatment of purification.

### 3.5 Removal of Excess Methanol

The biodiesel may have content of excess methanol which is removed out by using the thermostatic bath or rotary evaporator at a temperature 60–65 °C

### 3.6 Biodiesel Washing and Drying

The biodiesel is then washed with the help of warm water to remove any residual methanol, catalyst and glycerol traces from the saponified reaction. Water wash was carried out in successive steps to avoid trapping any air bubbles, washed water due to higher density get collected at bottom and biodiesel separated at the top layer which is then separated. The finished biodiesel was heated again to about 50 °C in the oven for 15–20 min. to remove any moisture traces left during water washing. In some cases, air washing is also preferred.



**Fig. 5** Fuel samples blends; a B00, b B20, c B40, d B60, e B80, f B100

### **3.7 Test Samples/Fuel Blends**

The Calophyllum oil biodiesel (COME) thus produced is used to prepared test fuel samples as, B00—this is neat diesel, B20—this is the biodiesel blend where mixture of 80% diesel and 20% COME, B40—this is the biodiesel blend where mixture of 60% diesel and 40% COME, B60—this is the biodiesel blend where mixture of 40% diesel and 60% COME, B80—this is the biodiesel blend where mixture of 20% diesel and 80% COME and, B100—this is neat biodiesel (Fig. 5).

The specific gravity reduces after transesterification, viscosity of raw Calophyllum oil from 65.29 cSt to COME 3.7 cSt, which is acceptable as per ASTM norms for Biodiesel. Flash point and fire point are important temperatures specified for safety during storage, handling and transport. The flash point and fire point of biodiesel was found to be 142 °C and 34 °C respectively. Flash point of Calophyllum oil 216 °C observed decreases after transesterification, which shows that its volatile characteristics had developed and it is also safe to handle.

## **4 Result and Discussion**

### **4.1 Biodiesel Yield**

The variation Calophyllum oil biodiesel yield influence by solution temperature maintained during transesterification process. In this experiment temperature ranges in three categories as 40–50, 50–60 and 60–70 °C and the raw oil and methoxide solution ratio varies from 1:1, 1.5:1 and 2:1. The Fig. 6 and Table 3, shows that Calophyllum oil biodiesel yield is found highest 67% at 1.5:1 oil to methoxide ratio at reaction temperature 50–60 °C for a time period of 90 min (Table 4).



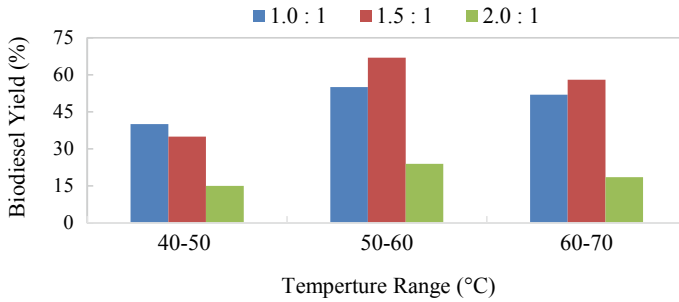


Fig. 6 Variation of biodiesel yield

### 4.2 Characteristics of Calophyllum Oil Biodiesel

Density is the very important property of any fuel and its combustion efficiency. For any liquid, density can be defined as the mass per unit volume and measured in the unit kg/l. The density of fuel in compression ignition engine play a key role because of it gives the signal of the delay between the fuel injection and combustion of the fuel in a compression ignition engine and also indicate the energy per unit mass of fuel. As in compression ignition engine, fuel injection and system are worked on the volume and thus higher density fuel result in more mass injected than required and vise-versa. Therefore the Calophyllum oil biodiesel blend and its property variations are discussed below.

**Variation of Kinematic Viscosity.** Table 5 shows the properties of Calophyllum Oil Methyl Ester as per ASTM standards, wherein, viscosity plays important role in fuel injection characteristics, it may disturbs the operation of fuel injector especially at low temperature conditions. High kinematic viscosity result in poor fuel atomization which lead to the more engine deposits and formation of soot The vegetable oil shows highest values and it is important to control it within the acceptable level to avoid adverse effect on fuel injector system and performance. The author found kinematic viscosity of the biodiesel derived is 3.7 cSt which is closed to the diesel value.

The increase of biodiesel content in fuel blend with diesel, the kinematic viscosity increases, also there is increase in density of the fuel blend (Fig. 7).

The relationship between kinematic viscosity and density is shown by the regression equation,

$$Kinematic\ Viscosity = 0.0282 * Density - 20.974$$

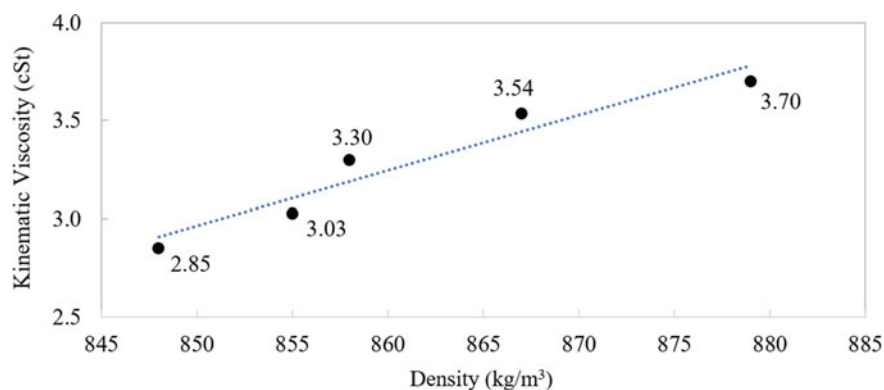
**Variation of Calorific Value.** The calorific value is the major parameter in the selection of a fuel. It indicates is the amount of heat energy present in the fuel, which is determined by the complete combustion of specified quantity at constant pressure and in normal conditions. Table 5 shows the value of calophyllum oil biodiesel is

**Table 4** Characterization of Calophyllum Oil Methyl Ester (Biodiesel)

Description	ASTM method	Unit	ASTM limits [10, 16]	Diesel	COME				Author		
				Ashok et al. [17]	Atabani et al. [10]	Sahoo and Das [11]	SathyaSelvabala et al. [18]	Sahoo et al. [19]		Rizwanul Fattah et al. [20]	Ashok et al. [17]
Density @ 40 °C	D1298	kg/m <sup>3</sup>	820-900	815	877.6	880.6	-	-	868.6	864	879.1
Kinematic viscosity @ 40 °C	D445	cSt	1.9-6.0	2.3	5.54	5.72	4	4.92	4.71	5.58	3.7
Cloud point	D2500	°C	-	-	12	-	13.2	13.2	10	-	12
Pour point	D97	°C	-	-	13	-	-	4.3	8	-	-
Flash point	D93	°C	55-101	53	162.5	151	140	140	141.5	160	142
Calorific value	-	MJ/kg	-	42.5	39.51	-	-	38.66	38.39	39.8	37.24

**Table 5** Characterization of calophyllum oil biodiesel blends [Author]

Properties	Unit	B00	B20	B40	B60	B80	B100
Density @ 15 °C	kg/m <sup>3</sup>	840	847.6	854.6	857.5	867.3	879.1
Kinematic viscosity @ 40 °C	cSt	3.12	2.85	3.03	3.3	3.54	3.7
Flash point	°C	57	179	172	163	154	142
Fire point	°C	66	188	180	171	163	151
Calorific value	MJ/kg	44.34	43.86	41.21	40.13	39.02	37.24
Cetane number	–	48	56	55	54	53	52

**Fig. 7** Variation of Kinematic viscosity

37.24 kJ/kg which is lesser than diesel because of inherently vegetable oil having lesser carbon content, thus lower energy density.

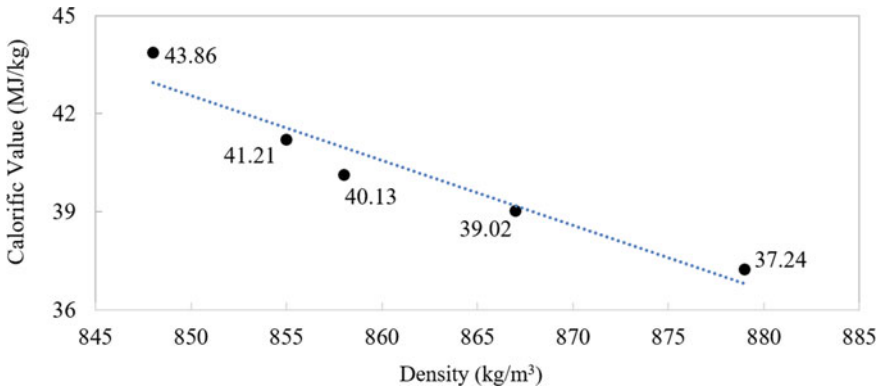
The increase of biodiesel content in fuel blend with diesel, the calorific value of fuel blend decreases, along with the increase in density of the fuel blend (Fig. 8).

The relationship between calorific value and density is shown by the regression equation,

$$\text{Calorific Value} = -0.1988 * \text{Density} + 211.57$$

**Variation of Cetane Number.** Cetane number is an inverse function of a fuel ignition delay which is the time period between the start of injection and the first identifiable pressure increase during combustion of the fuel. In a particular diesel engine, higher cetane fuels will have shorter ignition delay periods. Cetane number is used to measure of ignition quality of diesel fuels. It influences both gaseous and particulate emissions. The fuels with higher auto ignition temperatures are more likely to cause diesel knock.

Table 5 shows the cetane number of neat biodiesel is 52 and on increasing of biodiesel content in fuel blend, the value of cetane number goes on decreasing for



**Fig. 8** Variation of calorific value

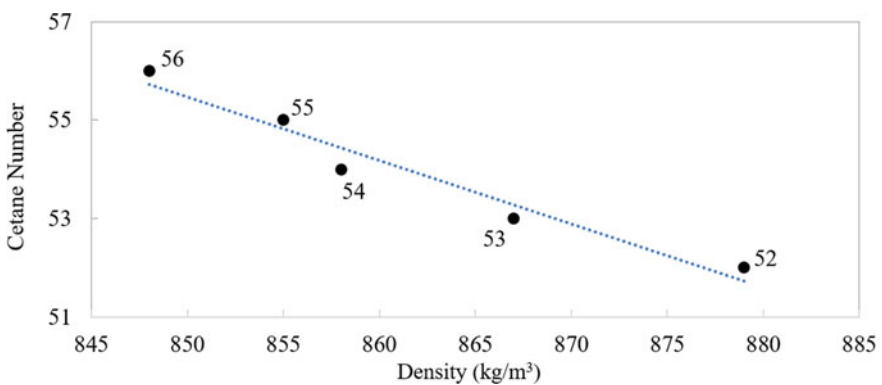
the fuel blend, also it is observed that the increase of biodiesel content increases density of the test fuel blend (Fig. 9).

The relationship between cetane number and density is shown by the regression equation,

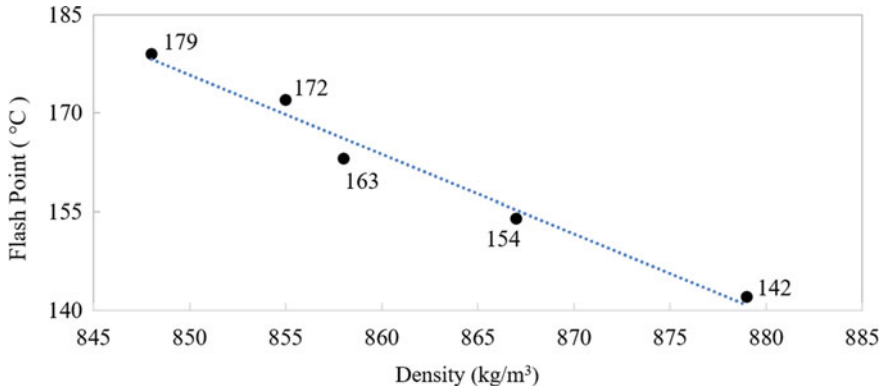
$$CetaneNumber = -0.1291 * Density + 165.21$$

**Variation of Flash Point.** This is the property that indicates the lowest temperature at which fuel ignites when it is exposed to a flame. From Table 5, the flashpoint of neat biodiesel reported by the author is 142 °C which is higher than petroleum diesel, which favors safe transportation.

The increase of biodiesel content in fuel blend with diesel, the flash point value of fuel blend reduces, along with the increase in density of the fuel blend (Fig. 10).



**Fig. 9** Variation of cetane number



**Fig. 10** Variation of flash point

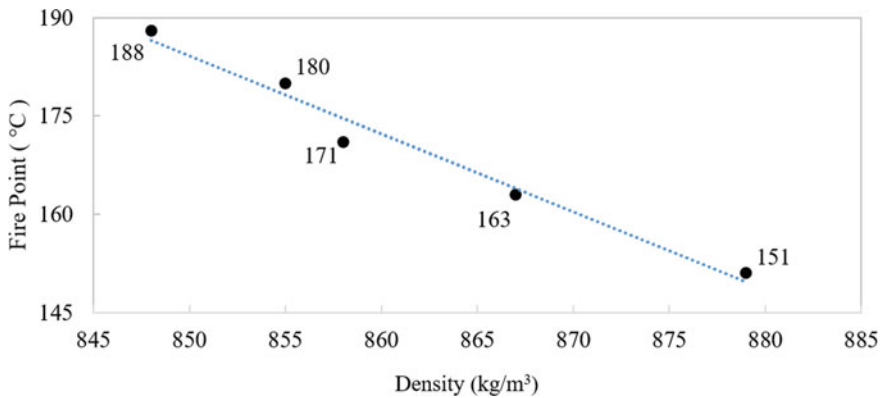
The relationship between calorific value and density is shown by the regression equation,

$$FlashPoint = -1.2073 * Density + 1201.9$$

**Variation of Fire Point.** The fire point is the lowest temperature at which the vapors keep burning after the ignition source is removed. It is higher than the flash point, because at the flash point more vapor may not be produced fast enough to sustain combustion.

From the Table 5, the fire point of the neat biodiesel is 151 °C. The increase of biodiesel content in fuel blend with diesel, the fire point of the fuel blend reduces, also it is observed that there is increase in the density of the fuel blend (Fig. 11).

The relationship between calorific value and density is shown by the regression equation,



**Fig. 11** Variation of fire point

$$\text{Fire Point} = -1.1902 * \text{Density} + 1195.8$$

## 5 Conclusions

- The analysis shows that Calophyllum oil biodiesel met the major biodiesel requirements as per standards ASTM D 6751-02, ASTM PS 121-99, EN 14214, DIN V 51606 and IS 1448.
- Highest triglyceride conversion rate of 67% was achieved after 90 min. of reaction at temperature 50–60 °C with 1.5:1 molar ratio.
- Transesterification of Calophyllum oil help to reduce the kinematic viscosity to 3.7cSt, density to 879.1 kg/m<sup>3</sup>.
- Alcoholises of Calophyllum oil lower its calorific value from 43.86 to 37.24 MJ/kg.
- Calophyllum oil biodiesel reported cetane number 52, flash point 142 °C, and fire point 151 °C.

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