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Effect Of Additives On Flow Properties Of Pure Biodiesels And Their Mixturesat Low Temperatures

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ABSTRACT
The effect of concentration of two chemical additives polymethacrylate (PMA) and polyalphaolefin (PAO) on the cloud point, pour point and kinematic viscosity of Jatropha biodiesel and Karanja biodiesel and their mixture has been studied. The concentration of a chemical additive has been varied in the range of 0 – 10 g/L. For the same concentration 7.5 g/L of chemical additive, the reduction in the cloud point is 4^0C with PAO compared to 2^0C with PMA. and the reduction in pour point is 10–11^0C with PAO compared to 7–8^0C with PMA. With increase in concentration of either PMA or PAO from 0 to 10 g/L, the reduction of 20 ¬ 25 percent in kinematic viscosity of pure biodiesels and their mixture has been observed. This study indicates that PAO is superior compared to PMA in improving the flow properties of Jatropha biodiesel and Karanja biodiesel and their mixture at low temperatures.

Keywords: Jatropha biodiesel, Karanja biodiesel, Cloud point, Pour point, Kinematic viscosity, polymethacrylate (PMA), polyalphaolefin (PAO)

1. INTRODUCTION

Biodiesel is defined as the monoalkyl esters of long-chain fatty acids derived from renewable feed stocks like vegetable oils or animal fats (IS 15607, 2005). India has vast resources of non-edible seeds from which oil can be leached to produce biodiesel, depending upon the potential and specific needs of the locality (Kumar and Sharma, 2011). The fossil fuel depletion and environmental degradation are the major issues for sustainable efforts being undertaken to determine the suitability of biodiesel as fuel for compression-ignition engine.
Biodiesel is obtained by transesterification of triglycerides present in vegetable oils/animal fats (Srivastava and Prasad, 2000; Otera, 1993; Srivastava and Prasad, 2002). It is technically competitive with conventional petroleum diesel fuel and offers number of advantages, such as enhanced biodegradability, reduced toxicity, lower emission and increased lubricity.However, its flow properties at low temperaturesare unfavorable. These properties are dependent on the fatty acid composition of constituent esters and the presence of minor components such as monoglycerides and steryl glycosides (Dunn, 2011).The parameters used to characterize flow properties of biodiesel at low temperatures include cloud point (CP), pour point (PP), cold filter plugging point(CFPP) and low temperature flow test (LTFT). Their definition and method of determination are given in Table 1.
Biodiesel at its CP appears cloudy due to formation of small crystals of saturated fatty acid alkyl esters and it is still able to flow freely. Biodiesel at its PP will not flow through the fuel injection system and causes operational problems, such as completely plugged lines and engine shutdown due to fuel starvation. LTFT which has been widely accepted in North America and CFPPwhich has been widely accepted in Europe are often considered the most important to characterize the suitability of a biodiesel for use in a vehicle as a function of weather. These temperatures of a biodiesel are usually between its CP and PP. Both LTFT and

CFPP focus on dynamic tests that simulate flow through filters in the fuel injection system, rather than on static physical property tests like CP and PP.

Table 1. Parameters used for characterization of flow properties of biodiesel

Parameter	**Definition**	**Method of determination**
Cloud point	Cloud point is defined as the temperature where crystals become visible (diameter exceeds 0.5 µm) forming a hazy or cloudy suspension.	IS 1448 (P:10), ASTM D-2500
Pour point	Pour point is defined as the lowest temperature where the fuel flows or can be pumped.	IS 1448 (P:10), ASTM D-97
Cold filter plugging point	Cold filter plugging point is defined as the lowest temperature where 20 mL of fuel passes safely through a 45 µm wire mesh filter under 2 kPa vacuum within 60 s.	ASTM D-6371
Low temperature flow test	Low temperature flow test is defined as the lowest temperature where 180 mL of fuel passes through a 17 µm wire mesh filter under 20 kPa vacuum within 60 s.	ASTM D-4539

The most convenient and economical way of improving the low temperature properties of biodiesel is its treatment with chemical additives which are generally referred to as pour point depressants, flow improvers or crystal modifiers. Most additives promote the formation of small (10 – 100µm) needle shaped crystals. These crystals experience significantly reduced growth and agglomeration rates as temperature decreases below cloud point. However, the rate of nucleation is promoted and causes the formation of a large quantity of the relatively small and more compact crystals. Although most of these crystals will becaught in fuel filters, the cake layer formed on the filter surface is considerably more permeable to flow of biodiesel.

Dunn and Bagby(1995)determined CP, PP, CFPP and LTFTof biodiesels prepared from soybean oil and tallow. They found that both CFPP and LTFT vary almost linearly with CP. Dunn et al. (1996) studied the effect of twelve cold flow additives on CP, PP, CFPP and LTFT of biodiesels. These additives significantly improved the PP, but did not affect the CP or kinematic viscosity significantly. Hanna et al. (1996)used various combinations of methyl tallowate (made from edible beef tallow), ethanol and diesel fuel to observe crystal formation as a function of temperature. They observed that ethanol decreased the formation of crystals in methyl tallowate at all temperatures. Chiu et al. (2004) presented studies on CP, PP and LTFTof soybean biodiesel blends in kerosene in the presence of four additives (OS-110050, Bio Flow-870, Bio Flow-875, Diesel Fuel Anti-Gel) at 0.1 - 2%. They observed that the PP of soybean biodiesel is decreased by 27^0C for a mixture of 0.2% additive, 79,8% soybean biodiesel and 20% kerosene. All additives had insignificant effect on the CP but two of the additives decreased the PP. Conley and Tao (2006)reviewed the results of CP, PP and CFPP tests of pure biodiesel formed from soy, canola, lard and edible tallow conducted by other investigators. They concluded that biodiesel prepared from soy and canola oil provided the best performance of the types of pure biodiesel considered. Joshi and Pegg (2007)measured the CP and PP of biodiesel blends made with No. 2 diesel and biodiesel derived from ethyl esters of fish oil. They developedempirical equations for predicting the CP and PP for a given blend. Lopes et al. (2007) studied the effect of polyamides on CP, PP and CFPP of biodiesels derived from rapeseed, soybean and palm oils. They found that the polyamide additive was very effective in improving the CP and PP of rapeseed methyl esters. Shrestha et al. (2008)studied the effect of four additives on the CP and PP of biodiesel blends made from soy methyl ester,mustard ethyl ester, mustard methyl ester, used vegetable oil methyl ester and No. 2 diesel. The additives tested had no effect on the CP and a significant effect on the PP particularly for blends containing 20% biodiesel and lower. They observed that the additives generally worked better with ethyl esters than methyl esters and that a higher percentage of diesel in the blend resulted in better additive effectiveness. Rafie and Attia (2008)evaluated the improvement of flow properties of pure biodiesel prepared from sunflower oil, linseed oil and mixture of soybean, sunflower and oleen oilsthrough the use of 1 wt. % ozonated vegetable oil. They found that ozonated oils maintain flow properties of the biodiesel. Bhale et al. (2009)investigated the effect of ethanol, kerosene and commercial

additives on the low-temperature properties of pure biodiesel obtained from Madhuca indica. They reported a considerable reduction in pour point with these cold flow improvers. Ethanol blended biodiesel is claimed as an alternate fuel for improved cold flow behavior and better emission characteristics without affecting engine performance. Garcia-Perez et al. (2010) evaluated the effect of mixing of bio-oil produced from pyrolysis of pine pellets and pine chips in biodiesel prepared from poultry fat. The addition of bio-oil modifies the crystallization behavior of unsaturated compounds present in biodiesel. Joshi et al. (2010) studied the effect of blending alcohols(ethanol, isopropanol and butanol) with poultry fat methyl esters on low-temperature properties. They suggested butanol as the most prudent choice when considering alcohol biodiesel blends. Boshui et al. (2010) evaluated the impact of olefin-ester copolymer, ethylene vinyl acetate copolymer and polymethyl acrylate on the low-temperature properties and viscosity-temperature characteristics of soybean biodiesel. They used a polarizing microscope to the crystal morphologies of the biodiesel at low temperatures. They found that the olefin-ester copolymer was the most effective for improving the low-temperature properties of soybean biodiesel. Torres et al. (2011) synthesized fatty acid derivatives by esterification of stearic, oleic and linoleic acids with linear and cyclic alcohols and by epoxidation of methyl oleate and subsequent ring-opening reaction with the same alcohols. Some of these fatty acid derivatives allowed a decrease in CFPP of biodiesel. Chastek (2011)described the methods for improving the low-temperature properties of canola-based biodiesel. Hereported that at 1% loading, polylauryl methacrylate lowers the pour point by 30^0C and LTFT by 28^0C.Misra and Murthy (2011) reviewed the literature on the use of various additives for improving the low-temperature properties of biodiesel. Warasanam and Pengprecha (2012) synthesized pour point depressants by the esterification of dicarboxylic acid, namely, succinic acid and azelaic acid, with methanol and isopropyl alcohol using sulphuric acid as catalyst. They found diisopropyl azelate to be the most effective in improving the low-temperature properties of biodiesel. Satapimonphan and Pengprecha (2012) investigated the effect of methyl stearate, isopropyl stearate, methyl oleate and isopropyl oleate on the CP and PP of biodiesel from white sesame seed oil. Isopropyl oleate was found to be the most effective in decreasing the CP and PP of biodiesel used.Lv et al. (2013) investigated the effect of DEP (trade name), polyglycerol ester (PGE) and PA (prepared in the laboratory) on the cold flow properties of palm methyl ester. When the ratio of DEP: PGE: PA was 3:1:1 or 2:2:1, the CFPP of palm methyl ester was decreased by 7^0C.Mohammadi et al. (2014) explored the impact of different solvents in stabilizing biodiesel-polymer solution. Among them, acetone was found to be the best stabilizer. The CP of the biodiesel-polymer-acetone fuel was found to have improved due to the inclusion of acetone.

This paper is aimed to investigate the effect of chemical additives on the flow properties of pure biodiesels obtained from Jatropha and Karanja, the important species in Indian context and their mixtures at low temperatures. Two chemical additives polymethacrylate (PMA) and polyalphaolefin (PAO) have been used in this study. Since CP and PP of biodiesel are the most important indicators for ensuring good performance at low temperatures and these are related to CFPP and LTFT of biodiesel, the effects of concentration of PMA and PAO on CP, PP and kinematic viscosity at 40^0C of pure biodiesels and their mixtureshave been investigated to study the effectiveness of the selected chemical additives.

2. EXPERIMENTAL SECTION

2.1 Materials

Jatropha oil and Karanja oilwerepurchased 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. Chemical additives PMA and PAO were purchased from M/s Pawan & Company, Delhi. The characteristics of Jatrophaoil and Karanja oil and their biodieselare given in Table 2. The characteristics of chemical additives PMA and PAO are given in Table 3.

Table 2. Characteristics of Jatropha oil and Karanja oil 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, mm^2/s	33.30	30.95	4.16	3.58
Acid value, mgKOH/g	5.19	4.25	0.81	0.61

Table 3. Characteristics of chemical additives PMA and PAO

Characteristics	Value for	
	PMA	PAO
Density, kg/m^3	0.867	0.896
Molecular weight, kg/kmol	2260	1875
Pour point, ^oC	¬ 6	¬ 9

2.2 Method

The experimental method for making biodiesel from Jatropha oil and Karanja oil is described below. Since the free fatty acid (FFA) contents of Jatropha oil and Karanja oil(given in Table 2) are above the acceptable limit of 1 wt.% for transesterification, the biodiesel from Jatropha oilandKaranja oilhas been prepared by acid-catalyzed esterification process followed by alkali-catalyzed transesterifcation process (Berchmans and Hirata, 2008).

In an acid-catalyzed esterification process, the FFApresent in Jatropha/Karanja oil is esterified with methanol in presence of sulphuric acid as catalyst. For esterification methanol to oil mole ratio of 6:1 was used with 1% sulphuric acid (based on weight of the oil). The esterification reaction was carried out at a temperature of 60^oC with stirring at 400 rpm for two hours. At the end of esterification reaction, the contents were allowed to cool and transferred to separating funnel for overnight. Methanol-water layer formed at the top was removed. The esterified product separated at the bottom was distilled under vacuum to recover methanol and its FFA content was determined by standard titrimetric method.

In an alkali-catalyzed transesterification process, the esterified product from the previous step was transesterified with methanol to produce biodiesel using methanol to esterified product mole ratio of 6:1 in the presence of 0.55 % w/v KOH (based on weight of esterified product) as catalyst. The esterified product from previous step was poured into the flask and heated to 50^oC. The solution of KOH in methanol is heated to 50^oC and then added to heated esterified product. The reaction mixture is then heated to 60^oC and stirred at 400 rpm for two hours. After completion of reaction the contents were cooled and transferred to separating funnel. The product was allowed to stand overnight for separation of glycerol layer from fatty acid methyl ester layer on top. The separated fatty acid methyl ester layer was washed with distilled water to remove alkali (Phenolphthalein test). The fatty acid methyl ester mixture was then dried with anhydrous sodium sulphate followed by filtration. The filtrate mixture was distilled under vacuum to remove unreacted methanol. The mixture left in the flask is the biodiesel which was tested for cloud point, pour point and kinematic viscosity.

The biodiesel mixtures were prepared by measuring the appropriate amounts of Jatropha biodiesel and Karanja biodiesel and then mixing in a conical flask at 45^0C for 45 minute with continuous stirring to ensure uniform mixing. Similarly the appropriate amounts of biodiesel mixture and chemical additive to produce mixture containing chemical additive in the range of 0-10 g/L were mixed in a conical flask at 45^0C for 45 minute with continuous stirring.

3. RESULTS AND DISCUSSION

The effect of concentration of two chemical additives PMA and PAO on the cloud point, pour point and kinematic viscosity of Jatropha biodiesel, Karanja biodiesel and their mixture has been studied. The concentration of chemical additives has been varied in the range of 0 – 10 g/L. The experimental results are discussed below:

Figure1 shows the effect of concentration of PMA on the cloud point of Jatropha biodiesel, Karanja biodieseland their mixture. The cloud points of pure Jatropha biodiesel and Karanja biodiesel have been found to be 3^0C and $– 1^0C$, respectively. It can be seen from this figure that the cloud point of Jatropha biodiesel, Karanja biodiesel and their mixture first decreases with increase in the concentration of PMA up to about 5 g/L and then it becomes constant with further increase in the concentration of PMA. The decrease in the

cloud point of Jatropha biodiesel, Karanja biodiesel and mixture of 50 vol. % Jatropha biodiesel and 50 vol. % Karanja biodiesel has been observed to be 2^0C for increase in the concentration of PMA from 0 to 10 g/L. The effect of concentration of PAO on the cloud point of Jatropha biodiesel, Karanja biodiesel and their mixture is shown in Fig.2. This figure shows that the cloud point of Jatropha biodiesel, Karanja biodiesel and their mixture decreases with increase in the concentration of PAO up to about 7.5 g/L. The decrease in the cloud point of Jatropha biodiesel, Karanja biodiesel and mixture of 50 vol.% Jatropha biodiesel and 50 vol.% Karanja biodiesel has been observed to be 4^0C for increase in the concentration of PAO from 0 to 10 g/L.

Figure 3 shows the effect of concentration of PMA on the pour point of Jatropha biodiesel, Karanja biodiesel and their mixture. The pour points of pure Jatropha biodiesel and Karanja biodiesel have been found to be -5^0C and -7^0C, respectively. It can be seen from this figure that the pour point of Jatropha biodiesel, Karanja biodiesel and their mixture first decreases with increase in the concentration of PMA up to about 5 g/L and then it becomes constant with further increase in the concentration of PMA. The decrease in the pour point of Jatropha biodiesel, Karanja biodiesel and mixture of 50 vol. % Jatropha biodiesel and 50 vol. % Karanja biodiesel has been observed to be $7–8^0$C for increase in the concentration of PMA from 0 to 10 g/L. The effect of concentration of PAO on the pour point of Jatropha biodiesel, Karanja biodiesel and their mixtures is shown in Fig.4. It can be seen from this figure that the pour point of Jatropha biodiesel, Karanja biodiesel and their mixtures decreases with increase in the concentration of PAO up to 10 g/L. The decrease in the pour point of Jatropha biodiesel, Karanja biodiesel and mixture of 50 vol. % Jatropha biodiesel and 50 vol. % Karanja biodiesel has been observed to be $10–11^0$C for increase in the concentration of PAO from 0 to 10 g/L.

The effect of concentration of PMA and PAO on the kinematic viscosity at 40^0C of Jatropha biodiesel, Karanja biodiesel and their mixture is identical and it is shown in Fig. 5. This figure showsthat the kinematic viscosity of Jatropha biodiesel, Karanja biodiesel and their mixture decreases with increase in the concentration of PMA as well as PAO from 0 to 10 g/L. The kinematic viscosity of pure Jatropha biodiesel and Karanja biodiesel at 40^0C is found to be 4.16 mm^2s^{-1} and 3.58mm^2s^{-1}, respectively. With increase in concentration of either PMA or PAO from 0 to 10 g/L, the reduction in kinematic viscosity of Jatropha biodiesel, Karanja biodiesel and mixture of 50 vol. % Jatropha biodiesel and 50 vol. % Karanja biodiesel of 1.07, 0.73 and 0.82 mm^2s^{-1}, respectively has been observed.

For comparing the effectiveness of PMA and PAO, the variation of cloud point and pour point with biodiesel blend composition with PMA and PAO concentration as a parameter is shown in Figs. 6 and 7. For the same concentration 7.5 g/L of chemical additive, the reduction in the cloud point is 4^0C with PAO compared to 2^0C with PMA and the reduction in pour point is $10–11^0$C with PAO compared to $7–8^0$C with PMA. From the above discussion it is clear that both PMA as well as PAO are effective for the reduction in the cloud point and pour point of Jatropha biodiesel, Karanja biodiesel and their mixture. However, PAO is superior compared to PMA in improving the flow properties of Jatropha biodiesel, Karanja biodiesel and their mixture at low temperatures.

4. CONCLUSIONS

The effect of concentration of two chemical additives PMA and PAO on the cloud point, pour point and kinematic viscosity of Jatropha biodiesel, Karanja biodiesel and their mixtures has been studied. The concentration of chemical additives has been varied in the range of 0 – 10 g/L.

Both PMA as well as PAO are effective for the reduction in the cloud point and pour point of Jatropha biodiesel, Karanja biodiesel and their mixture. For the same concentration of 7.5 g/L of these additives, the reduction in the cloud point is 4^0C with PAO compared to 2^0C with PMA and the reduction in pour point is $10–11^0$C with PAO compared to $7–8^0$C with PMA. Thus, PAO is superior compared to PMA in improving the flow properties of Jatropha biodiesel and Karanja biodiesel and their mixtures at low temperatures. With increase in concentration of either PMA or PAO from 0 to 10 g/L, the reduction in kinematic viscosity of pure biodiesels and their mixture is about 20 – 25 %

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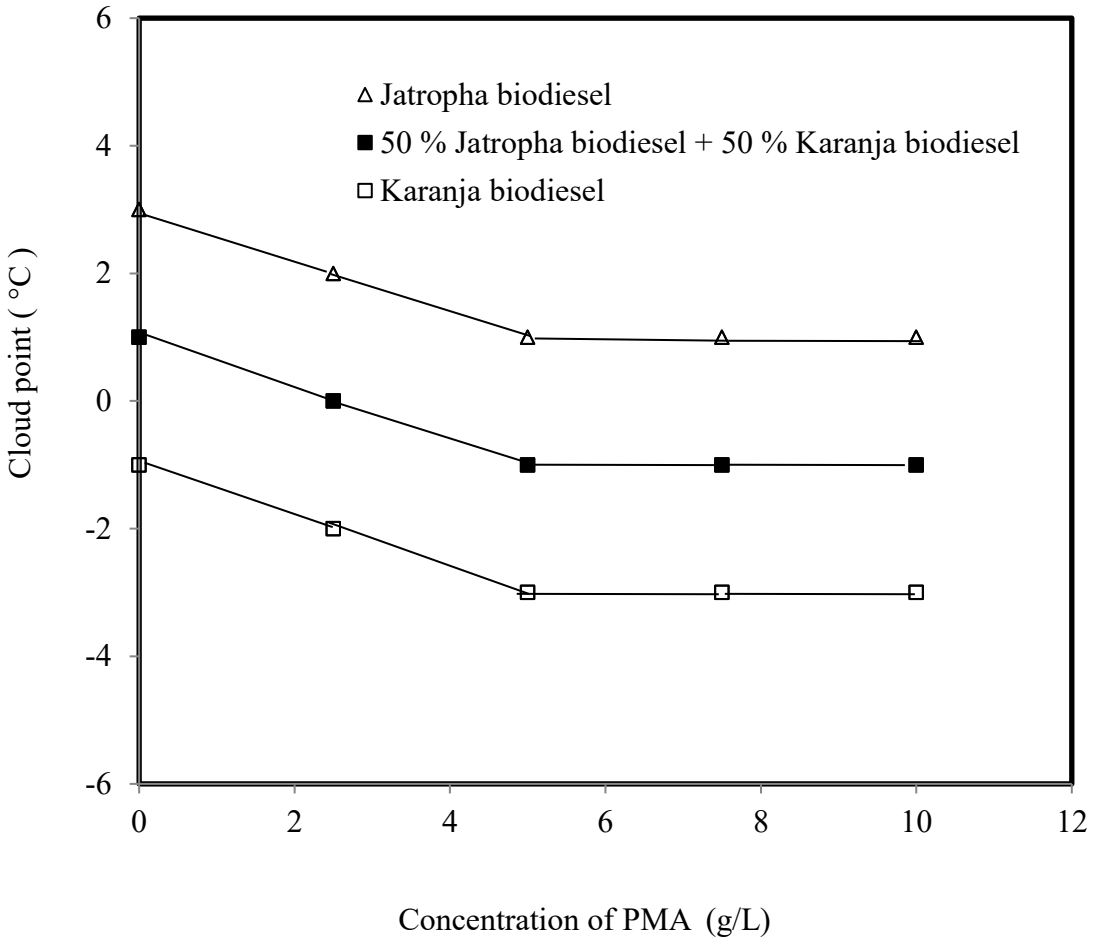

Fig.1 Variation of cloud point of pure biodiesels and their mixture with concentration of PMA.

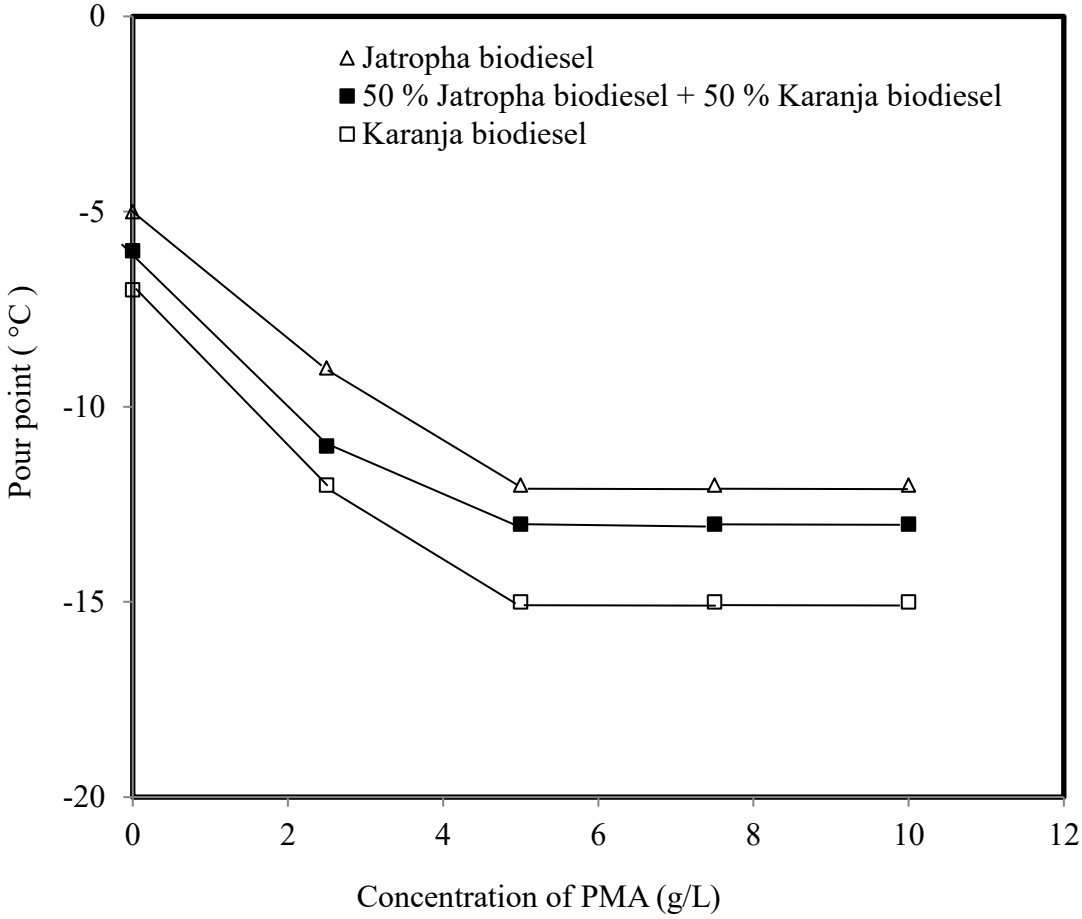

Fig.2 Variation of pour point of pure biodiesels and their mixturewith concentration of PMA.

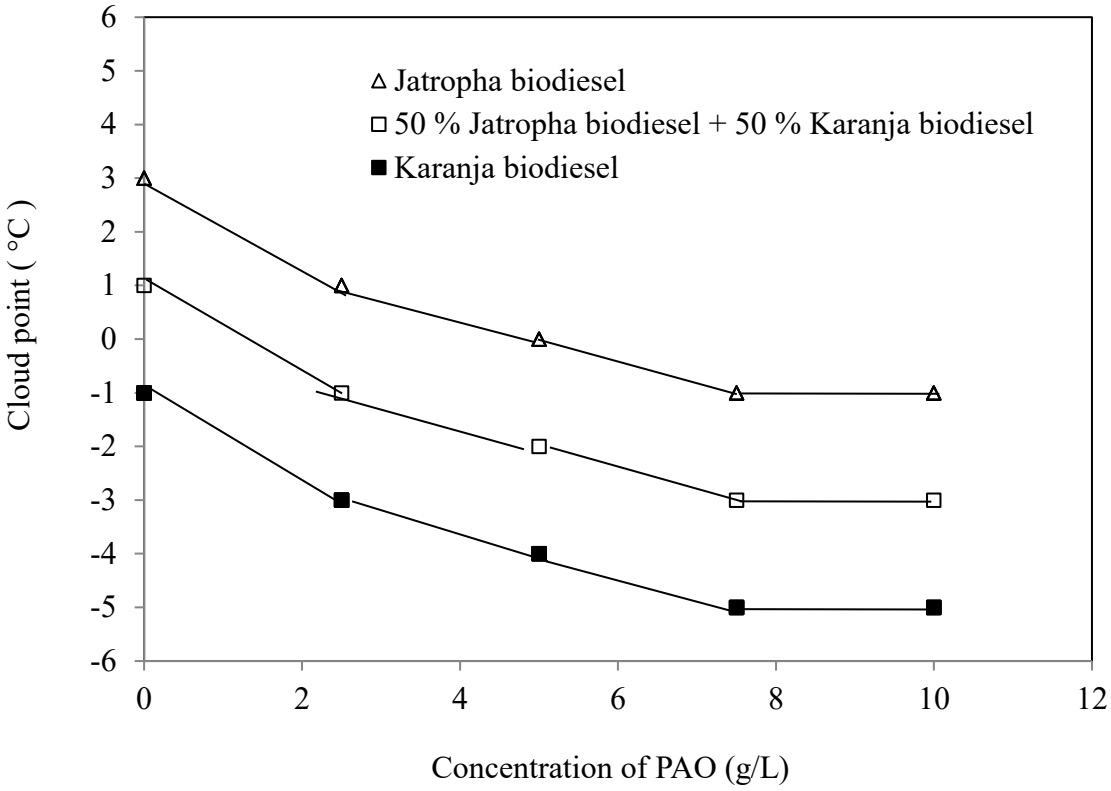

Fig.3 Variation of cloud point of pure biodiesels and their mixture with concentration of PAO.

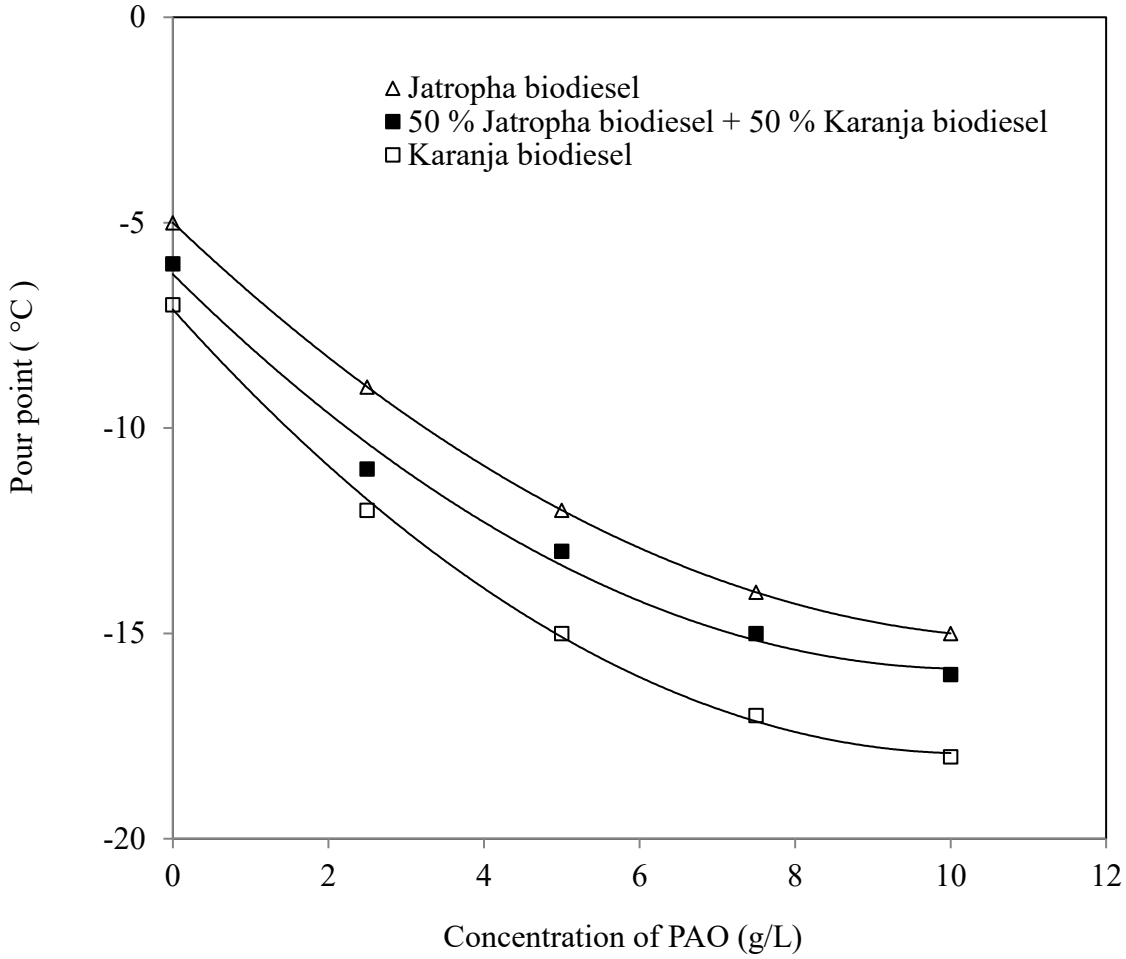

Fig. 4 Variation of pour point of pure biodiesels and their mixture with concentration of PAO.

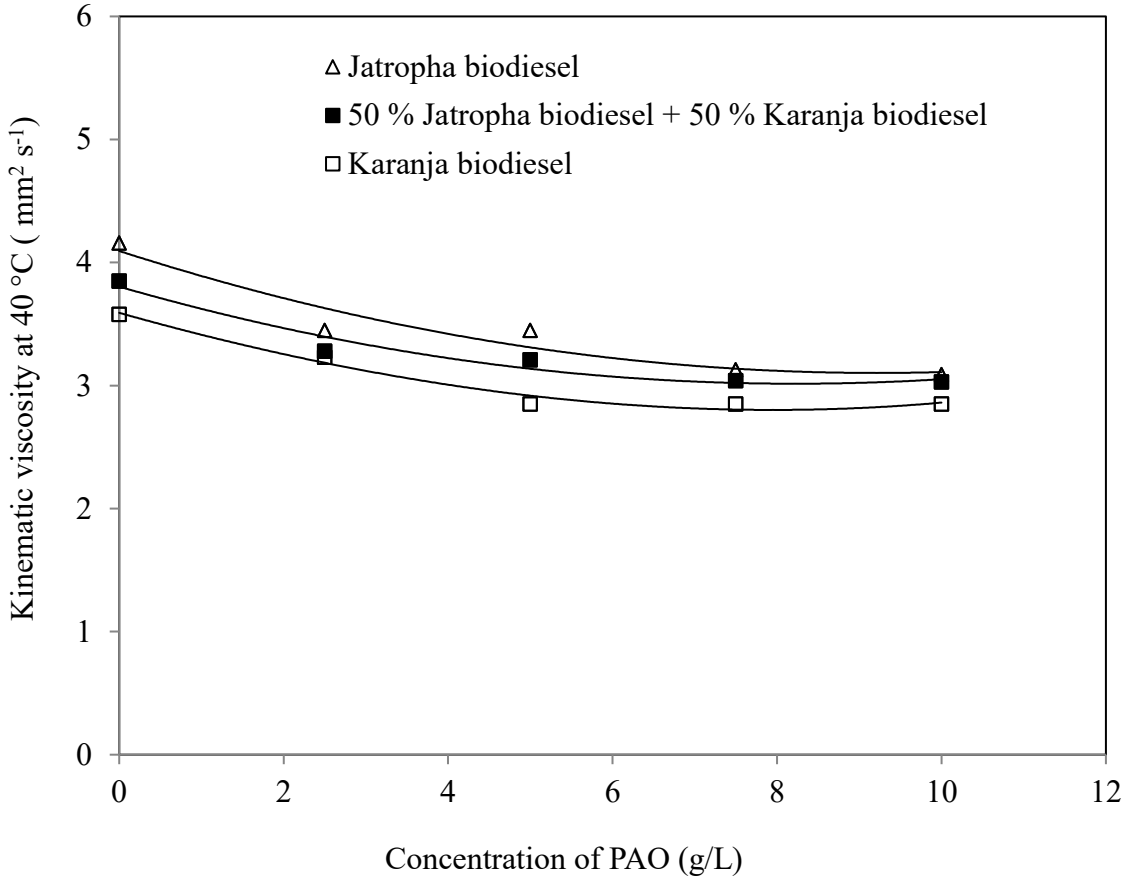

Fig. 5 Variation of kinematic viscosity of pure biodiesels and their mixture with concentrationof PAO.

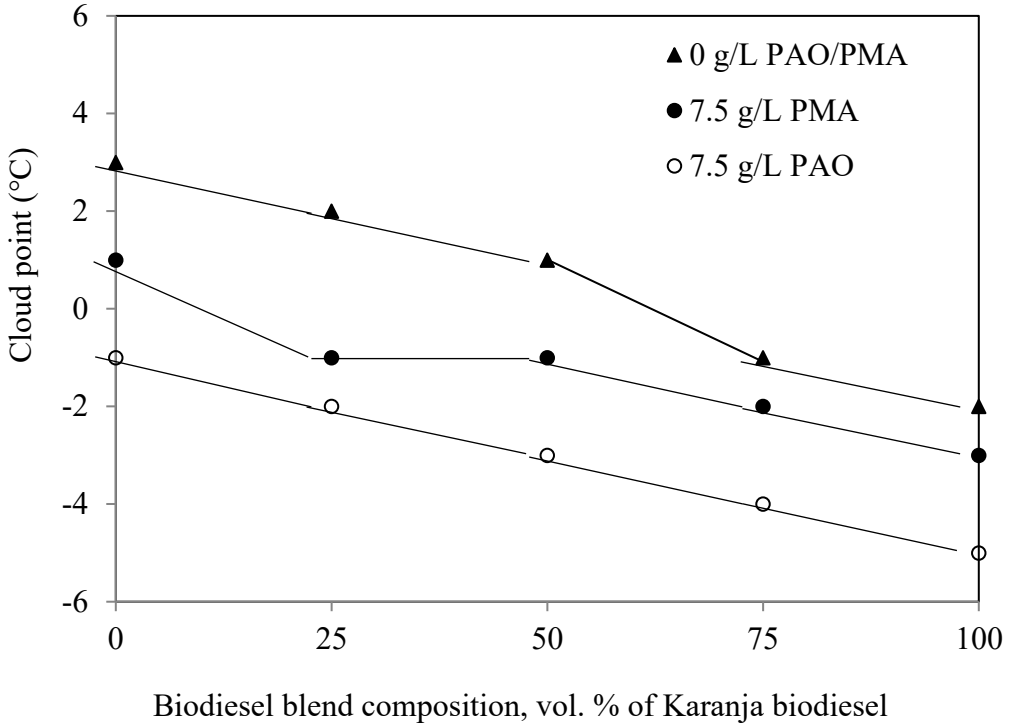

Fig. 6 Variation of cloud point with biodiesel blend composition with PMA and PAO concentration as a parameter.

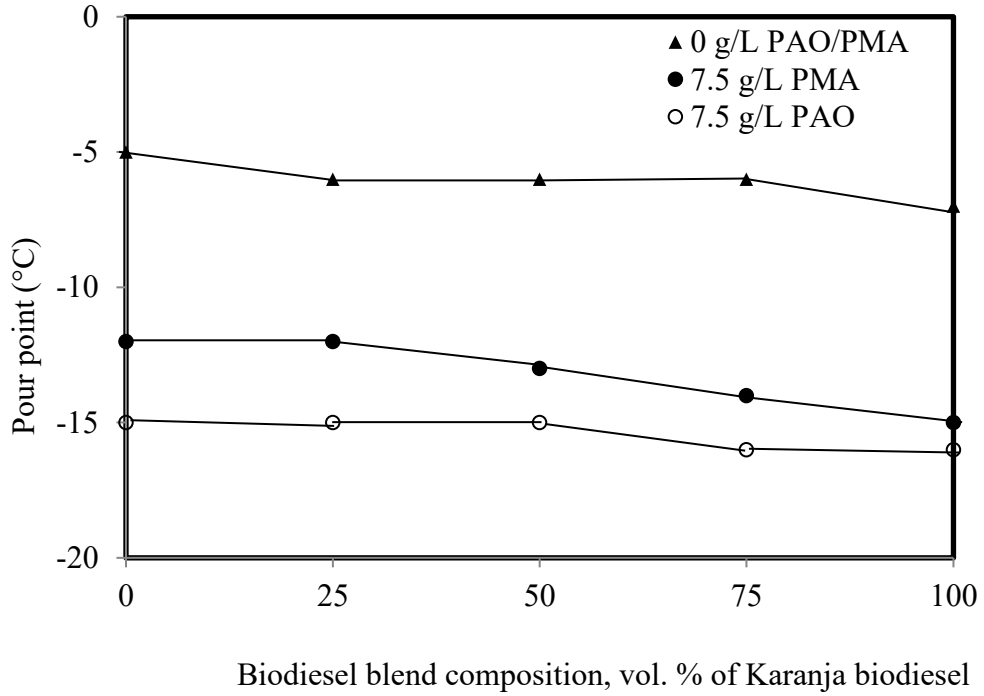

Fig. 7 Variation of pour point with biodiesel blend composition with PMA and PAO concentration as a parameter.