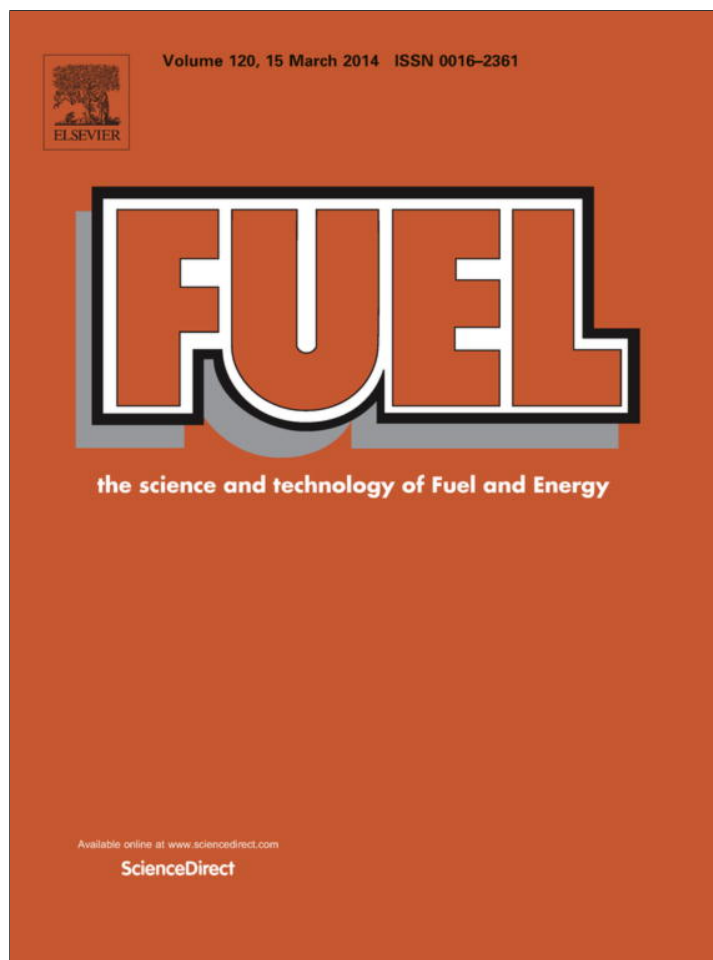


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Impact of additives on storage stability of *Karanja* (*Pongamia Pinnata*) biodiesel blends with conventional diesel sold at retail outlets



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HIGHLIGHTS

- Effect of the addition of antioxidants on storage stability blends of diesels with *KOME*.
- Strong correlation between *KOME* concentration and storage stability.
- Oxidation stability of neat *KOME* and its diesel blends were found to increase significantly with addition of antioxidants.
- PY and PrG were found most effective antioxidants among all.
- Difference in physicochemical properties shown variation in the storage stability of diesel biodiesel blends.

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ABSTRACT

Biodiesel has been recognized as a “green fuel”, being the most attractive substitute to the conventional petro-diesel. The unsaturated fatty acids present in biodiesel are susceptible to oxidation. One of the main criteria used for the quality assessment of biodiesel is ‘storage oxidation stability’. Oxidation of the esters during the long-term storage can lead to problems for the utilization of biodiesel in the engine directly. Therefore, the addition of additives is required to protect the oxidation of biodiesel. This study investigates the effect of antioxidant additives on oxidation stability of neat biodiesel [*Karanja* oil methyl ester (*KOME*)] and its diesel blends. *KOME* was prepared by methanolic KOH catalyzed transesterification of *Karanja* oil, which was produced from a non-edible oil feedstock from Indian sub-continent. Various diesel–biodiesel blends (B5, B10, B15, B20, B25 and B40) were prepared with conventional diesels sold at retail outlets of Northern India. Butylated hydroxy anisole (BHA), Butylated hydroxy toluene (BHT), *tert*-Butylhydroxyquinone (TBHQ), Propyl-gallate (PrG) and Pyrogallol (PY) antioxidant additives were selected for this study. Significant improvement in oxidation stability as well as in density and kinematic viscosity of diesel–biodiesel blends was observed with all antioxidants studied. PY, PrG and BHA were found most effective among all antioxidants tested, and their use in diesel/biodiesel blends showed a greater stabilizing potential. All the samples were stored at room temperature in air-tight bottles under ambient conditions. The aim of this study is to find the minimum required concentration of most effective antioxidant to meet the storage oxidation stability specifications.

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1. Introduction

Increase in petroleum prices, depletion of oil reservoirs and stiff regulation on exhaust emission are the major factors responsible for the search of biofuels and renewable energy resources in recent years [1,2]. It is well accepted that in diesel engines, clean combustion can be fulfilled only by engine development coupled with diesel fuel formulation [3,4]. One of the environmental friendly renewable energy sources is biodiesel [5]. Biodiesel is a mixture of methyl esters of long chain fatty acids derived from vegetable

oil and animal fats, and is similar to the commercial diesel in terms of fuel quality and combustion properties [6,7]. The biodiesel production from edible oil resource in India has not received much attention as the indigenous edible oil production is much less than its actual demand. Therefore non-edible oils (e.g. *Jatropha*, *Pongamia* (*Karanja*), *Mahua*, and *Sal*) seems the only possible source of biodiesel in India [8].

Biodiesel is non-flammable, nonexplosive, biodegradable, non-toxic and free from sulphur and aromatics fuel source. Biodiesel also provides less harmful emissions compared to petroleum diesel fuel [9,10] which makes biodiesel a good alternative to substitute the petroleum diesel [11]. However, the long term storage of biodiesel is being a problem. The presence of unsaturated fatty acids ester in biodiesel makes it more susceptible to oxidation or

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Table 1
General properties of base diesels and biodiesel.

S. no.	Property (unit)	D1	D2	D3	Karanja biodiesel	Std. limits		Test method
						Diesel	Biodiesel	
1	Flash point (°C)	68.0	79.0	84.0	148.3	55 min 35 min	100 Min ≤100 Min	ASTM D 93 IS 1448, P:20
2	Moisture content %(v/v)	0.004%	0.010%	0.011%	0.039%	Max. 0.02%	Max. 0.05%	ASTM-D 2709
3	Cloud Point (°C)	+3	0	0	+6	–	–	IS1448, P:40 ASTM–D 2500
4	Pour point (°C)	0	–3	–3	+3	3 °C winter and 15°C summer		IS 1448, P:10 ASTM–D 97
5	Total sulphur (ppm)	340 (±2)	310 (±2)	336 (±2)		350 max		IS 1448, P:10 ASTM D 1266 & IP 336 IS1448, P:83
6	Calorific value (kJ/kg)	43358	41929	42848	36871			
7	Density (g/cm ³) at 15 °C	0.8309	0.8288	0.8373	0.892	0.820–0.845 0.820–0.860	0.880–0.890	ASTM-D 4052 IS 1448, P:16
8	Kinematic viscosity (mm ² /s) at 40 °C	3.07	2.88	2.82	4.92	2.00–4.50	1.90–6.00	ASTM-D 445
9	Oxidation stability (IP, at 140 °C, hr)	–	–	–	2.98	2.00–5.00	3 h (min)	IS 1448, P:25 ASTM-D 7545
						–	6 h (min)	EN14214, prEN16091 and IS 15607

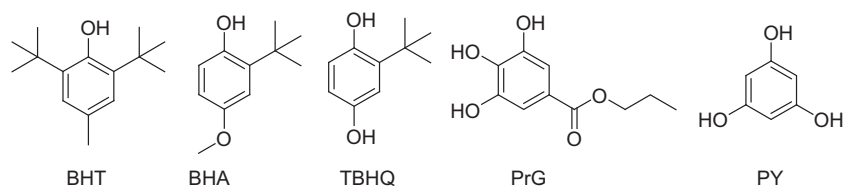


Fig. 1. Used antioxidants additives.

autoxidation during long term storage [12,13]. It is well reported in literature that oxidation stability does not correlate with the total number of double bond, but with the total number and position of allylic and bis-allylic carbon that are adjacent to double bond [14]. These oxidation processes are less pronounced in the parent oil due to the presence of natural antioxidants which get partially lost during refining [15]. Reports have been found stating that, after oxidation of biodiesel and its diesel blends, the acid value, density and viscosity increased, while iodine value decreased with increasing storage time [16]. Thus biodiesel instabilities results the formation of sediment and gum along with the fuel darkening, which causes filter plugging, injector fouling, depositions in the engine combustion chamber and malfunctions in various components of the fuel system [17,18]. The use of antioxidant additives not only slow down the oxidation processes but also improve the fuel

stability up to a certain extent [19]. Several reports have been found on the stabilities of diesel biodiesel blends [20–27]. However, limited reports are available on the impact of antioxidant additives on oxidation behavior of biodiesel from nonedible feedstock particularly from *Karanja* seed and its diesel blends [8,28–34].

In continuation of our ongoing research on optimization of additive concentration for storage stability of diesel–biodiesel blends [33,34] the aim of present study is to provide the experimental results on the effects of antioxidant additives on *KOME* and its blends with diesel fuel sold at retail outlets of Northern India. The effectiveness of selected antioxidants on oxidation stability, density and kinematic viscosity of the neat biodiesel and its diesel blends was investigated. The goal was to find out the minimum optimum concentration of additives which could significantly improve the storage stability of both, the neat biodiesel

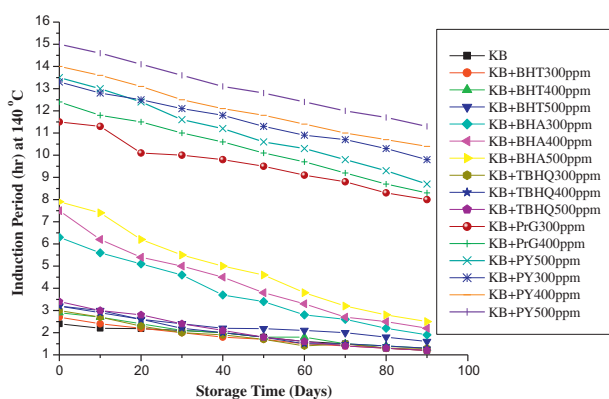


Fig. 2. Oxidation stability of *Karanja* biodiesel with antioxidants.

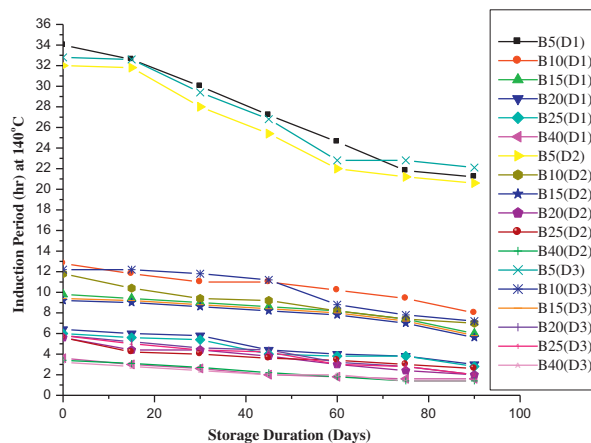


Fig. 3. Oxidation stability of diesel/biodiesel blends without antioxidants.

and its diesel blends. These results will help to support the development of biodiesel specification and technology.

2. Experimental section

2.1. Base diesel fuel

Three commercial diesels (D1, D2 and D3) were selected as base fuel. These diesels were purchased from the retail outlets of different oil companies in Northern India. The diesel sold by these outlets was received from respective refineries and might be of

different origins. The main physical properties and fuel characteristics of these diesels are listed in Table 1. The difference in their physical properties may be due to the difference in origin of crude oil, its processing and quality control.

2.2. Karanja biodiesel (KOME)

KOME was used as blending stock for collected diesel samples, and was prepared from the base catalyzed transesterification of Karanja oil. The main physical properties of biodiesel are listed in Table 1. The GC–MS analysis of FAME (biodiesel sample) was carried

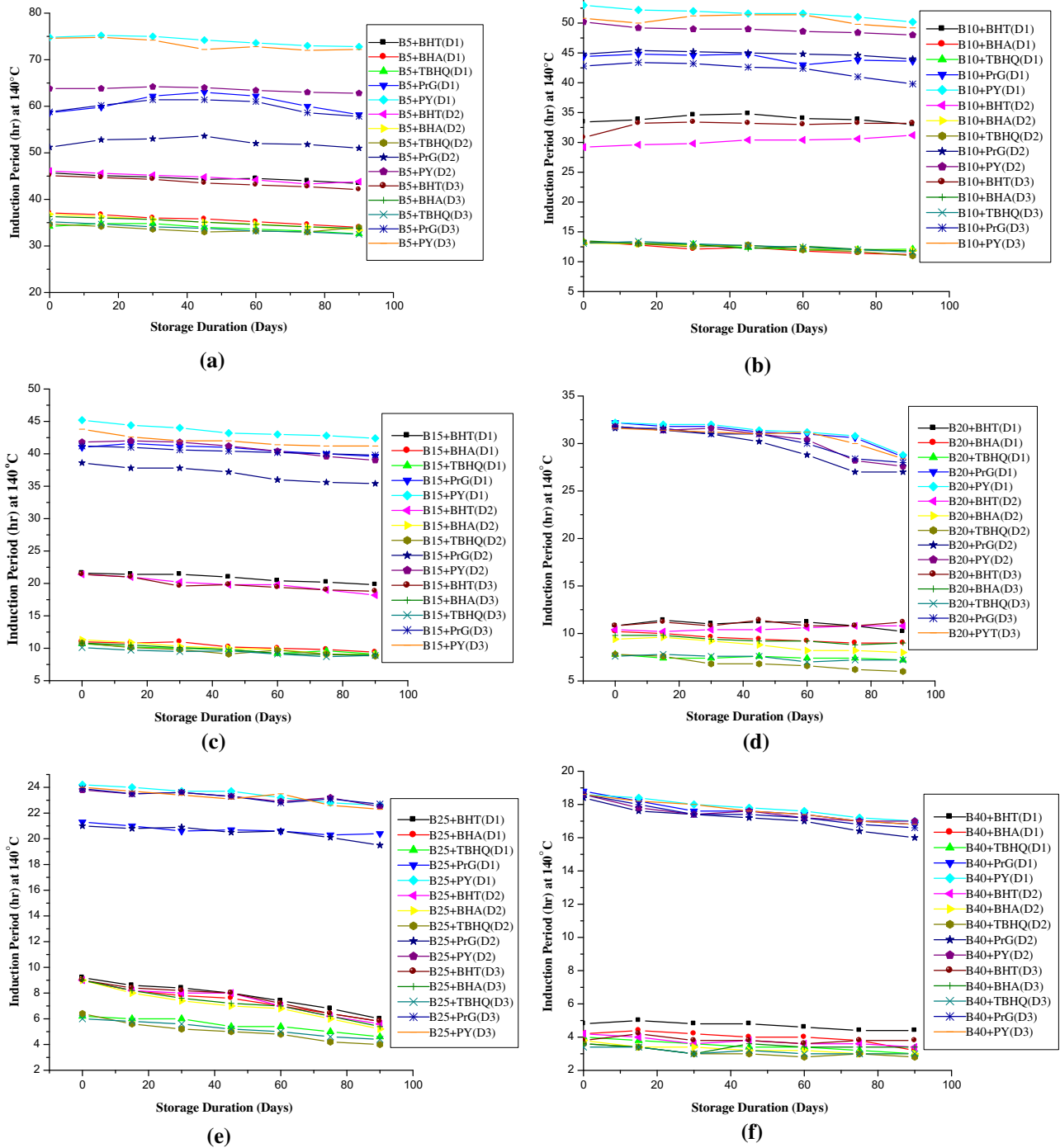


Fig. 4. Additive effects on oxidation stability of Karanja biodiesel blends with diesels D1, D2 and D3. (a) Oxidation stability of B5 blends of D1 D2 and D3, (b) oxidation stability of B10 blends of D1 D2 and D3, (c) oxidation stability of B15 blends of D1 D2 and D3, (d) oxidation stability of B20 blends of D1 D2 and D3, (e) oxidation stability of B25 blends of D1 D2 and D3 and (f) oxidation stability of B40 blends of D1 D2 and D3.

out on a QP-2010 gas chromatography mass spectrometer (GC-2010 coupled with GC-MS QP-2010) equipped with an auto sampler (AOC-5000) from Shimadzu (Japan) using a RTX-5 fused silica capillary column, 30 m × 0.25 mm × 0.25 μm (Rastek). Helium (99.9% purity) was used as the carrier gas with a column flow rate of 1 ml/min and a pre-column pressure of 49.7 kPa. The column temperature regime was 40 °C for 3 min, followed by a 5 °C/min ramp up to 230 °C, followed by 40 min at 230 °C. The injection volume and temperature were 0.2 μl and 240 °C and the split ratio was 1/30. The mass spectrometer was operated in electron compact mode with electron energy of 70 eV. Both the ion source temperature and the interface temperature were set at 200 °C. FAME peaks were identified by comparison of their retention times with authentic standards by GC-MS post run analysis and quantified by area normalization. Analysis revealed that the prepared KOME contains methyl esters of *Palmitic acid* (16:0) [2.2%], *Stearic acid* (18:0) [4.8%], *Oleic acid* (18:1) [61.2%], *Linoleic acid* (18:2) [25.5%], *Eicosanoic* (20:0) [2.1%], *Docosanoic* (22:0) [2.8%], *Tetracosanoic* (24:0) [1.4%]. The GC-MS results obtained were also supported by the existing literature [35,36]. The prepared biodiesel was used to constitute the diesel-biodiesel blends with B5, B10, B15, B20, B25 and B40 (volume/volume) with diesel fuel to study their physico-chemical properties (oxidation stability, viscosity and density) during long term storage.

2.3. Antioxidants additives

Butylated hydroxy toluene (BHT), Butylated hydroxy anisole (BHA), tert-Butylhydroxyquinone (TBHQ), Propyl-gallate (PrG) and Pyrogallol (PY) were used as antioxidant additives (Fig. 1). All additives were analytical grade and procured from Sigma Aldrich, India and used as received.

2.4. Storage conditions

500 mL sample of biodiesel and its diesel blend were stored in closed Borosil glass bottles of 1 L capacity for 90 days and were kept indoors, at a room temperature of 20 °C and 30 °C. 500 mL space in the bottle was occupied by air. Samples were taken out periodically every 15 days to study the additive effects.

2.5. Oxidation stability measurements

The oxidation stability (induction period i.e. IP) of neat KOME and its diesel blends were investigated by Petrotest "Petro-OXY(e)-VERSION: 10.08.2011" instrument made in Germany. The IP of biodiesel and its diesel blends was estimated according to the ASTM-D 7545-09 and prEN 16091 "Oxidation stability of fuel". IP was calculated for 5 ml fuel sample in hermetically sealed test chamber. The chamber was automatically pressurized with oxygen up to 700 kPa (~7 bar/101.5 psi) and heated to a temperature of 140 °C. This initiates a very fast oxidation process. As the fuel oxidizes, it consumes the oxygen in the sealed test chamber resulting in a 10% pressure drop that is displayed. The length of the induction period is a measure of how long the antioxidant will protect the biodiesel and its diesel blends from oxidation. The obtained IP values were converted to their corresponding Rancimate time by multiplying the obtained Petrotest time with a correction factor 20 (as recommended by the test method and was automatically displayed in the instrument). All determinations were performed in duplicate and the mean value is reported.

2.6. Density, kinematic viscosity, flash point and sulphur content measurements

Density of KOME and diesel blends were analyzed at 15 °C by Anton Paar density meter DMA-35 Version 3, according to

ASTM-D 4052 method while as the kinematic viscosity of the biodiesel and its diesel blends were analyzed at 40 °C temperature and 50% Torque by Fungi-lab expert series viscometer, according to ASTM-D 445 method. Flash point of the diesel and KOME samples were analyzed by Penske Martene Flash point apparatus with close cup, according to ASTM-D 93 method. Total sulphur content of diesel fuel samples were estimated by Lamp method for sulphur determination in petroleum products according to ASTM D 1266-107 and IP 336. All the data obtained were well supported by the reported/standard values.

3. Result and discussion

3.1. Effect of antioxidants on the oxidation stability of biodiesel samples

As per IS-15607 and prEN-16091 standards the biodiesel must retain its fuel characteristics over a minimum period of 6 h. under test conditions. The oxidation stability of neat biodiesel (KOME) was analyzed by Petrotest method and it gave an induction period of 2.98 h. which indicates that, it is not possible to use neat biodiesel (KOME) as an alternate fuel directly. However, improvement in induction period can be achieved by the use of antioxidant additives. Therefore effect of antioxidants on the oxidation stability of the neat KOME sample was investigated for 90 days indoor storage conditions. The antioxidants were screened by adding 300 ppm, 400 ppm and 500 ppm of each antioxidant in 500 mL of neat KOME and samples were taken out periodically every 15 days to study the additive effects. The results are shown in Fig. 2. A significant difference in the efficiencies of tested antioxidants was observed.

It is clear from the Fig. 2 that, after the addition of antioxidants, a significant improvement in the induction period of biodiesel was observed. Screening study was also revealed that the 500 ppm concentration of additive was the optimum concentration at which maximum stability was achieved. Therefore 500 ppm additive concentration was preferred for further studies of oxidation stability of diesel/biodiesel blends. From the screening data obtained, PY was found the most effective antioxidant with maximum induction period of 15.0 h whereas TBHQ was found to be the least effective during the course of study. On the basis of screening study the effectiveness of antioxidants used was observed in order of PY > PrG > BHT > BHA > TBHQ. Similar additive response was also observed by other researchers [21,30,32]. The screening study also revealed that the phenolic antioxidants were found more effective. As the active hydroxyl group provides free proton easily to inhibit the formation of free radicals or interrupt the propagation of free

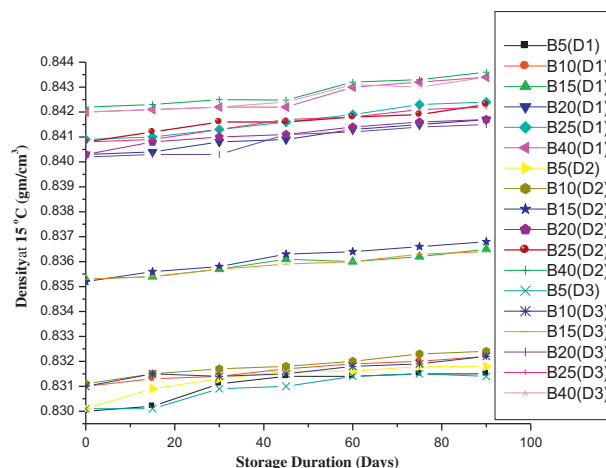


Fig. 5. Density of neat Karanja biodiesel blends of diesels D1, D2 and D3.

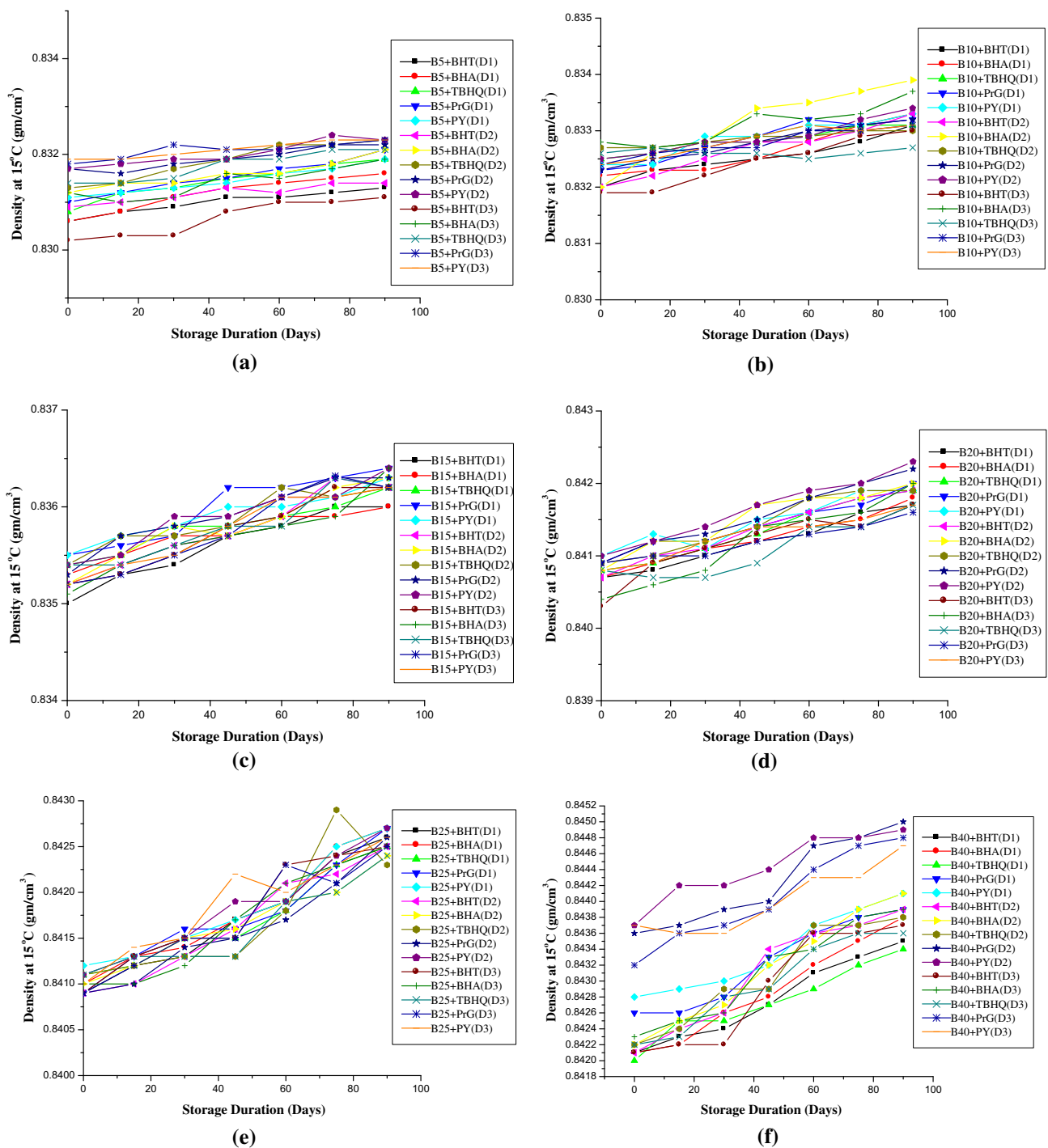


Fig. 6. Additive effects on density of *Karanja* biodiesel blends with diesels D1, D2 and D3. (a) Density of B5 blends of D1, D2 and D3, (b) density of B10 blends of D1, D2 and D3, (c) density of B15 blends of D1, D2 and D3, (d) density of B20 blends of D1, D2 and D3, (e) density of B25 blends of D1, D2 and D3 and (f) density of B40 blends of D1, D2 and D3.

radical and thus slow down the rate of oxidation, also the phenolic additives offer more sites for the formation of the complex between the free radical and antioxidant radical for the stabilization of the ester chain [37,38]. It can also be stated that the stability of neat *KOME* is lower in compare to *Jatropha* methyl ester due to the presence of ~86% of unsaturated fatty acid.

3.2. Effects of antioxidants on the oxidation stability of diesel biodiesel blends

Diesel–biodiesel blend stability is best described by determination of its induction period (IP). Blends with an IP of ≥ 20 h has

been demonstrated to be sufficiently stable for standard usage conditions and considered as EN 590:2009 limit. Induction period of neat diesel biodiesel blends (B5, B10, B15, B20, B25 and B40) was investigated under the test conditions and the results obtained are shown in Fig. 3.

From Fig. 3 it is clear that only B5 blends of all diesel samples were shown the induction period more than 20 h, whereas B10, B15, B20, B25 and B40 blends were failed to meet the minimum induction period (EN 590 limit). However, a sharp drop in the induction period of B5 blend was also observed for next 15, 30, ..., 90 days storage duration due to rapid degradation of biodiesel in blended samples. Further to see the antioxidant additives

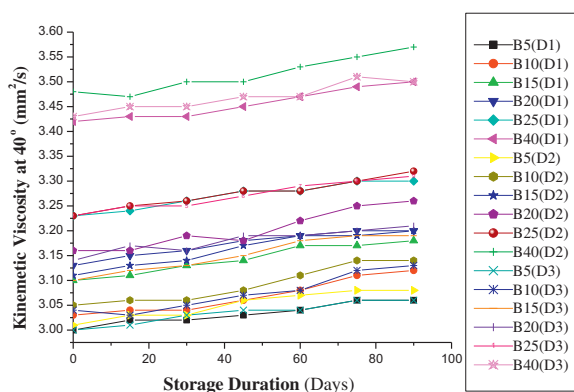


Fig. 7. Kinematic viscosity of diesel/biodiesel blends without antioxidants.

response on oxidation stability of the blends, the optimized amount (500 ppm) of antioxidants was added in 500 mL of each test solution and the oxidation stability was measured.

Fig. 4 shows the additive effects on oxidation stability of B5, B10, B15, B20, B25 and B40 diesel–biodiesel blends. Fig. 4a shows the effect of antioxidant additives of oxidation stability of B5 blends of D1, D2 and D3 diesel samples. It is clear from the Fig. 4a that, the maximum induction period of ~ 75 h was shown by the diesel D1 and D3 with PY. However, B5 blends of all three diesels were stable during the 90 days storage time with all additives and has shown the induction period more than 20 h (EN 590 limit). The B10 blends of D1, D2 and D3 (Fig. 4b) with antioxidants PY, PrG and BHT were found stable up to 90 days as all the B10 blends with these three additives has shown the induction period >20 h. However, these blends were failed to meet the minimum induction period limit (20 h as per EN590 limit) with BHA and TBHQ.

The B15 blends of D1, D2 and D3 were found stable up to 90 days with antioxidant PY and PrG, whereas these blends could be stored up to maximum ~ 75 days with BHT (Fig. 4c). However, BHA and TBHQ were not shown the desired effectiveness on long term storage of B15 blends. When the additive effect studies were performed for B20 and B25 blends of diesels D1, D2 and D3 (Fig. 4d and e), it was observed that both; B20 and B25 blends of diesels D1, D2 and D3 with antioxidants PY and PrG were stable up to 90 days study duration. B20 blend has shown the maximum induction period of ~ 32 h with both PY and PrG antioxidants, on the other hand induction period of ~ 24 h and ~ 23 h has shown by B25 blend with PY and PrG antioxidants respectively. However, these blends with BHT, BHA and TBHQ were failed to meet the minimum induction period (20 h; EN590 limit). Finally, oxidation stability of B40 blends of D1, D2 and D3 (Fig. 4f) was investigated and it was observed that with the optimized doses of antioxidants none of the blend were meet with the minimum stability limit.

Among the antioxidants investigated PY PrG and BHT has shown a greater stabilizing effect on the oxidation stability of diesel biodiesel blends of D1, D2 and D3. This was expected because all three additives have already shown their stabilizing potential with neat *Karanja* biodiesel. BHA and TBHQ were failed to mark their effectiveness in all the diesel–biodiesel blends tested. The difference in properties of diesel fuel samples (Table 1) may be the reason for the variation in oxidation stabilities of similar blends. Additionally, the lower sulphur in the base diesel may responsible to decrease the oxidation stability of the final blend [21]. From Table 1, diesel D1 and D3 have more sulphur content in compare to the diesel D2, therefore the blends of D1 and D3 have shown comparatively more induction period. It needs further study to know

the effect of diesel fuel properties on the oxidation stability of its biodiesel blends.

3.3. Density measurement of diesel biodiesel blends

A density measurement reflects stability and consistency of a fuel sample. It is a property for developing adequate storage methods for diesel biodiesel blends [39,40]. In diesel biodiesel blends the density of fuel increases with the increase of amount of biodiesel in the mixture. The density of all the blends was observed within the limit as mentioned in ASTM-D 445 and IS 1448, P:16 standards. The initial density value for neat blends (B5, B10, B15, B20, B25 and B40) of D1, D2 and D3 ranged from 0.830 gm/cm^3 to 0.844 gm/cm^3 with an average density value of 0.8365 gm/cm^3 ; while as the final density value for these blends were ranged from 0.831 gm/cm^3 to 0.843 gm/cm^3 with an average of 0.8379 gm/cm^3 (Fig. 5).

Similarly, density of all the diesel blends was also investigated with antioxidant additives and the results are shown in Fig. 6. The initial densities of the blends were ranged between 0.831 gm/cm^3 and 0.844 gm/cm^3 , whereas the final densities were found within the range of $0.833\text{--}0.846 \text{ gm/cm}^3$.

3.4. Kinematic viscosity measurement of diesel biodiesel blends

Kinematic viscosity of all the blends was also investigated with and without antioxidant additives and the results are summarized in Figs. 7 and 8 respectively. During oxidation of biodiesel the viscosity increases due to the formation of oxidized products which lead to the formation of sediments and gum [31].

The initial kinematic viscosity of neat diesel biodiesel blends ranged from 3.0 to $3.48 \text{ mm}^2/\text{s}$ with an average value of $3.17 \text{ mm}^2/\text{s}$ whereas the final value for the same were ranged from 3.06 to $3.57 \text{ mm}^2/\text{s}$ with an average of $3.24 \text{ mm}^2/\text{s}$ (Fig. 7). Similarly, the initial kinematic viscosity of the blends were ranged between $3.01\text{--}3.47 \text{ mm}^2/\text{s}$, whereas the final densities were found within the range of $3.06\text{--}3.59 \text{ mm}^2/\text{s}$ (Fig. 8). The viscosity of all the blends was observed within the limit as mentioned in ASTM-D 445 and IS 1448, P:25 standards.

4. Conclusion

In present work the *Karanja* biodiesel was blended with diesel obtained from the retail outlets of three different Oil companies in northern India, and the effectiveness of five antioxidants on the storage stability (oxidation stability, density and kinematic viscosity) of these blends were studied over a period of 90 days. The B5, B10, B15, B20, B25 and B40 diesel biodiesel blends were tested under the present study. The experimental results revealed that the 500 ppm dosages of PY, PrG, and BHT were most effective in neat biodiesel as well as its diesel blends. The neat biodiesel with 500 ppm of PY has shown the maximum stability (IP = 15 h). Except B40, all other blends with 500 ppm concentration of PY could be stored up to 90 days duration. However, B5 blend with 500 ppm of PY was found to be the most stable among all. Regarding biodiesel blends it was observed that with increasing the biodiesel concentration the oxidation stability decreases. Also the increase in density and viscosity of diesel biodiesel blends revealed that storage stability can be affected by the storage condition and time, leading to induction times below the minimum specification limit (EN590) of 20 h after 5–6 weeks. Studies showed that the induction period of blended fuel were not consistent. These variations may be due the composition of biodiesel, nature of antioxidant additives, and quality of diesel fuel. Diesel with higher sulphur content was found the most suitable for blend preparation in respect of

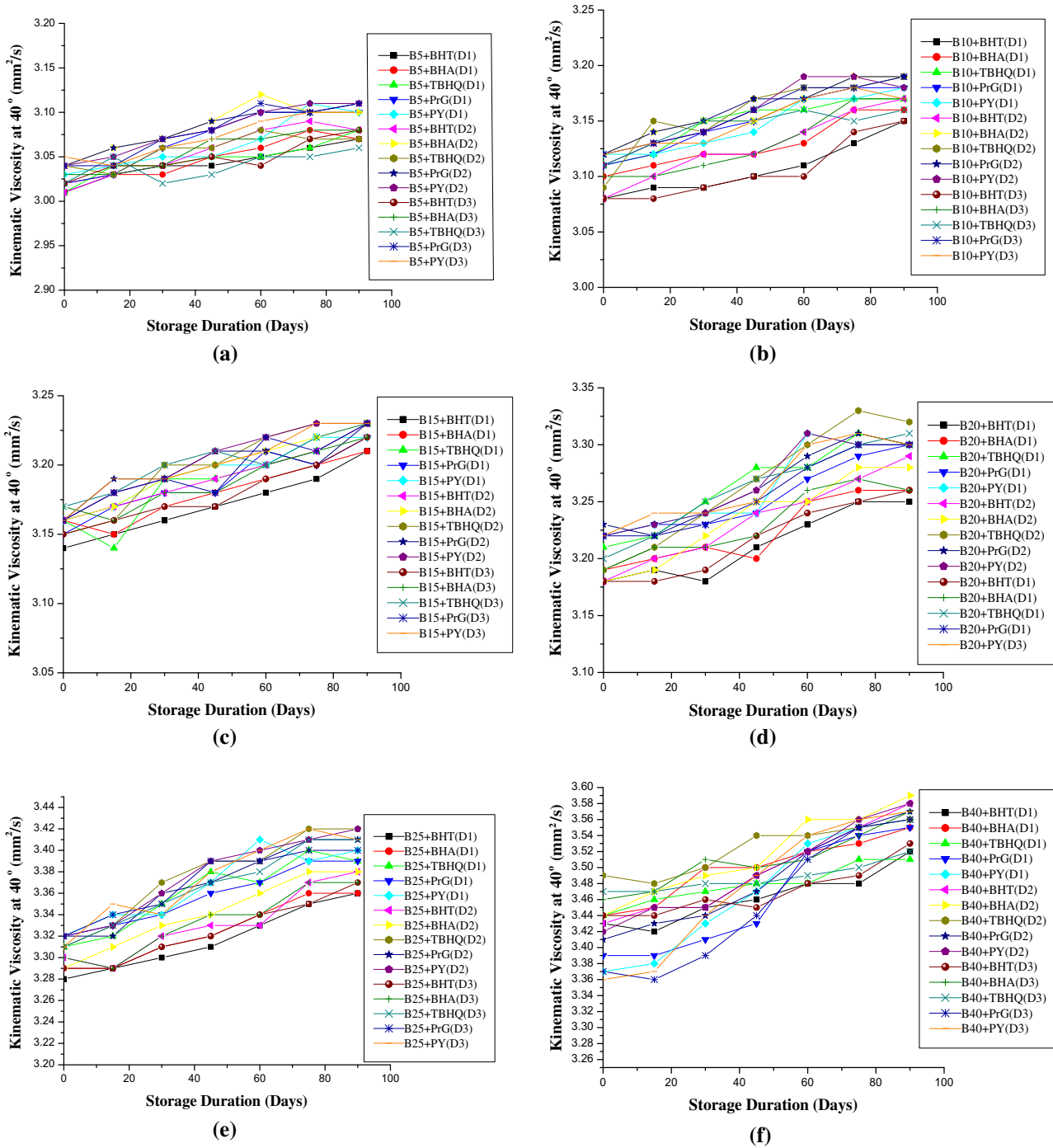


Fig. 8. Additive effects on kinematic viscosity of *Karanja* biodiesel blends with diesels D1, D2 and D3. (a) Kinematic viscosity of B5 blends of D1, D2 and D3, (b) kinematic viscosity of B10 blends of D1, D2 and D3, (c) kinematic viscosity of B5 blends of D1, D2 and D3, (d) kinematic viscosity of B10 blends of D1, D2 and D3, (e) kinematic viscosity of B5 blends of D1, D2 and D3 and (f) kinematic viscosity of B10 blends of D1, D2 and D3.

oxidation stability. However, further study is required to understand the role of the diesel fuel in the oxidation stability of diesel biodiesel blends, especially when there is a difference in the physical properties of the diesel fuel used for the blend preparation.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.fuel.2013.12.010>.

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