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Effect of antioxidants on physico-chemical properties of EURO-III HSD (high speed diesel) and *Jatropha* biodiesel blends

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ABSTRACT

The stability of fuel during storage is an important consideration for bulk users. Storage instability leads to solids formation which can plug nozzles and filters. This work focuses on the effect of the addition of antioxidants on **EURO-III and *Jatropha* Biodiesel blends**. The changes in physico-chemical properties were observed for **these blends** after addition of antioxidants. BHA (butylated hydroxy anisole), BHT (butylatedhydroxy toluene), TBHQ (tert-butylhydroxyquinone) and DPA (diphenylamine) were the antioxidants used for this study. The rate of change in kinematic viscosity and density of **EURO-III-*Jatropha* Biodiesel blends** with antioxidants were found to be less as compared to the neat samples. The oxidation stability of the neat samples, after addition of antioxidants, was found to increase significantly. It was also observed that addition of antioxidant significantly improved the oxidation stability of biodiesel-diesel fuel blends however in some case may act as pro-oxidants. The results showed that the addition of an antioxidant to diesel fuel blends influenced beneficially most of the important fuel properties.

1. Introduction

Biodiesel an alternate environment friendly liquid biofuel has reached an important position in the global fuel market. Increased environmental concern brought the quality of biodiesel and its blends into focus. Also it is well accepted that clean combustion in diesel engines can be accomplished only by engine modification coupled with diesel fuel reformulation or additive introduction [1,2].

Biodiesel, is a mixture of methyl esters with long chain fatty acids derived from vegetable oil and animal fats, and is similar to the commercial diesel oil in terms of fuel quality and combustion properties [3,4]. When compared to petroleum diesel, it possesses a number of advantages such as biodegradability and non-toxicity [5]. Biodiesel also has a favourable combustion–emission profile, producing much less carbon monoxide, sulphur oxides, nitrogen hydride, particulate matter, and unburned hydrocarbons compared to the petroleum-base diesel [6]. Therefore, to reduce air pollution and minimize the emission of greenhouse gas, it is beneficial to use biodiesel as an alternative fuel to substitute the petroleum-based diesel [7].

However, a major drawback with biodiesel is that it is more susceptible to oxidation degradation. The oxidation stability depends on the fatty acid composition of the oil. The oil that contains more unsaturation is more prone to oxidation [8]. For example, it has been reported that the relative rate of oxidation of methyl ester of oleic acid (18:1) linoleic acid (18:2) & linolenic acid (18:3) is in the ratio of 1:12:25 [9], that results in the formation of by product that increases viscosity [10]. During the process of transesterification of vegetable oils, the basic fatty acid chain remain same [11]. It has also been reported that the rate of oxidation is directly proportional to the number of bisallylic carbons present [12]. Oxidation is a complex process and due to which the methyl ester gets converted into a variety of species including shorter chain fatty acid and aldehyde and also to high molecular weight species through oxidative polymerization. The first step is the formation of free radical adjacent to a double bond. Free radical being highly reactive reacts with atmospheric oxygen leading to formation of peroxy radical. This peroxy free radical gets stabilized by abstracting 'H' from new fatty acid methyl ester and thereby creating new free radical. The process continues resulting in the formation of aldehyde alcohol and carbonic acid [13]. These reactions are less pronounced in the parent oil due to the presence of natural antioxidants which get partially lost during refining thereby reducing

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the oxidation stability [14]. The use of antioxidant can improve the fuel stability to a certain extent. Several studies have reported the effect of various synthetic antioxidants on oxidation stability of biodiesel [15–22]. However, limited work has been carried out on the stability behaviour of biodiesel blends with EURO-III HSD (high speed diesel) as well as on impact of antioxidants on storage stability of biodiesel/diesel blends [16,18,23–29].

The aim of the present work is to investigate the potential of different concentrations of various antioxidants on JME EURO-III HSD blends concerning the improvement of the oxidation stability of low sulphur diesel/biodiesel blends. The goal was to identify suitable antioxidant that could significantly improve the stability of biodiesel and its blend with EURO-III HSD diesel.

2. Experimental section

2.1. Blending components

EURO-III HSD/Bharat Stage III, JME (*Jatropha curcus* methyl ester) and antioxidants.

2.2. EURO-III HSD/Bharat Stage III

EURO-III HSD (a 50 L lot) corresponding to Bharat Stage III, was obtained from the retail outlet and its key physicochemical characteristics were done. The results are presented in Table 1.

2.3. Jatropha curcus methyl ester (JME)

Bulk preparation of JME under optimized process conditions and its detailed characterization: both physicochemical and chemical have been carried out [30]. The complete details of JME are given below. Table 2 shows the physicochemical characterization results. The prepared biodiesel was used to constitute the biodiesel-diesel-blends containing 5%, 10%, 20% and 40% biodiesel (i.e.JB-5, JB-10, JB-20 and JB-40) volume/volume with base diesel fuel to study their physico-chemical behaviour during long term storage.

2.4. Jatropha curcus methyl ester (JME) profile & GC

The composition of prepared JME was analysed using a GC (gas chromatograph) supplied by Nucon 5700 series with EOX column (serial no 5061; 30 m length, 0.25 mm ID and 0.25 mm outer dia). 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. FAME (Fatty acid methyl ester) peaks were identified by comparison of their retention times with authentic standards by GC and quantified by area normalization.

Table 1
Physicochemical characterization of EURO-III high speed diesel.

S.NO	Characteristics	Unit	Expected value for HSD	EURO-III requirement	Test methods
1.	Specific gravity @ 15 °C	–	0.8243	0.820–0.8450	ASTM-D 4052
2.	API @ 15 °C		40.16	36–41	Using correlation
3.	Cetane index (CI)		51	46	ASTM D 4737
4.	Aniline point	°C, °F	53, 127	–	ASTM D611-07
5.	Kinematic viscosity @ 40 °C	cSt	2.74	2.0–4.5	ASTM-D 445
6.	Flash point	°C	62	66	ASTM D 93
7.	Pour point	°C	9	15 max	ASTM D 97

Table 2
Physico-chemical characterization of JME.

S.No	Property (unit)	Biodiesel (B-100)	Std. limits	Test methods
1	Flash point (°C)	161.5	Min 100	ASTM D 93
2	Moisture content	0.041%	Max. 0.05%	ASTM-D 2709
3	Cloud point (°C)	+12	–	ASTM D 2500
4	Pour point (°C)	+3	–	ASTM D 97
5	Calorific value (KJ/kg)	39071	–	–
6	Specific gravity	0.8811	0.820–0.845	ASTM-D 4052
7	Viscosity (cSt)	4.7138	1.9–6.0	ASTM-D 445
8	Acid value		0.5 max	ASTM-D 664
9	Oxidation stability (at 140 °C, h)	4.21	6 h min	ASTM-D 7545

The GC of JME is shown in Fig. 1.

Analysis revealed that JME biodiesel contains mainly Palmitic (16:0), Palmitoleic (16:1), Steric (18:0), Oleic (18:1), Linoleic (18:2) and Linolenic (18:3) methyl esters. The detailed composition is given in Table 3. The numbers in parentheses show carbon number and number of double bond present e.g. oleic acid 18 carbon atom and one double bond. The results obtained in the study are in agreement with those reported by other workers [31,32].

2.5. AO (antioxidants) used

The antioxidants used in the present study are BHA (butylated hydroxy anisole), BHT (butylatedhydroxy toluene), TBHQ (tert-butylhydroxyquinone) and DPA (diphenylamine). These analytical grade antioxidants were procured from Sigma Aldrich, India. Fig. 2 shows structure of various antioxidants used in the present study.

2.6. Storage conditions

Biodiesel and its diesel blend samples of volume 500 mL were stored in closed Borosil glass bottles of 1 Lit capacity for 80 days and

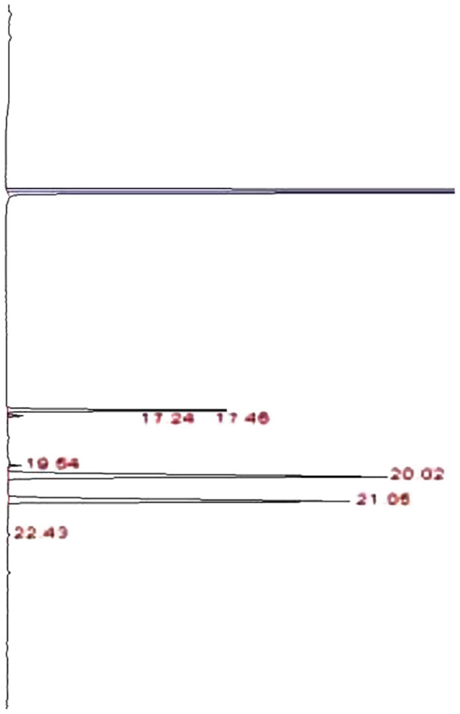

Fig. 1. Fatty acid profile of *Jatropha methyl ester.*

Table 3
Fatty acid composition of *Jatropha biodiesel* by GC.

FA profile of *JC biodiesel*		12:0 Lauric	14:0 Myristic	16:0 Palmitic	16:1 Palmitoleic	18:0 Stearic	18:1 Oleic	18:2 Linoleic	18:3 Linolenic	20:0 Arachidic	Others
Observed (Fig. 1)	Rt (min)			17.24	17.46	19.54	20.02	21.05	22.43		–
	%	–	–	15.34	0.79	0.50	43.97	39.31	0.1	–	–
Reported [18]		–	1.4	15.6	–	9.7	40.8	32.1	–	0.4	–
Reported [26]		5.9	2.7	13.5	–	6.1	21.8	47.4	–	–	2.7

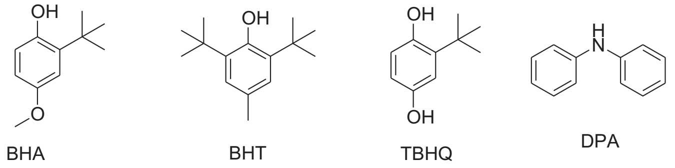

Fig. 2. Structure of antioxidants.

were kept indoors, at a room temperature of 18 °C and 28 °C. 500 mL space in the bottle was occupied by air. Samples were taken out periodically every 10 days to study the additive effects.

2.7. Approaches to oxidation status

There are several methods used to assess oxidation behaviour of biodiesel and its blends. This include acid value, rancimat period, density, kinematic viscosity etc. [33] Most of the studies have used rancimat method [34,35]. The present work is different from previous studies in this area since EURO-III is a low sulphur diesel specification and the blends with biodiesel are therefore relatively sulphur free, and till date very little information is available on these blends. Moreover, the oxidation stability was quantified in terms of rancimat period, kinematic viscosity and density. The rancimat period was measured using Petrotest "PetroOXY(e)-VERSION: 10.08.2011" instrument made in Germany. The rancimat period was estimated accordingly the standard method ASTM-D 7545-09 "Oxidation stability of fuel". Rancimat method involves measuring the time elapsed between starting a test cycle and the breaking point (reaction stoppage time) which is defined as a pressure drop of 10% below the maximum pressure developed in the test vessel as it warms up to test temperature. Rancimat period was measured using 5 ml fuel sample in hermetically sealed test chamber. The chamber was automatically pressurized with oxygen up to 700 kPa (approximately 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 pressure drop that is displayed. The length of the rancimat period is directly related to the oxidation stability of the fuel. Rancimat period were measured for biodiesel and its blends with commercial EURO-III diesel fuel using the method described above. All the determinations were performed in duplicate and the mean value is reported with an error of ±2%.

Kinematic viscosity of biodiesel and its blends, with and without antioxidants, were measured at 40 °C and 50% torque by Fungi-lab Expert Series Viscometer, according to ASTM-D 445 method.

Density of diesel samples were measured at 15 °C using Anton Paar Density meter DMA-35 Version 3, according to ASTM-D 4052 method. All the data obtained were well supported [13] and within the range as per ASTM standard.

2.8. Screening of antioxidants

For screening purpose the effects of four synthetic antioxidants (BHT, BHA, DPA and TBHQ) were investigated on neat JME. The study was done by adding 2 mL of 300, 400 and 500 ppm (w/w) of each antioxidant in JME. The results are given in Table 4 and the variation of rancimat period with different concentrations of antioxidants is shown in Fig. 3. It is clear from the data obtained that the rancimat period gets enhanced, more with TBHQ as compared to other antioxidants and the best result is obtained with 500 ppm concentration. BHT and BHA were found to be less effective compared to TBHQ and DPA was least effective and was not used for further study.

Above investigation reveals that phenolic antioxidants are more effective as compared to non-phenolic ones. Furthermore, effectiveness of TBHQ signifies that the antioxidant capability has a direct relation with the number of −OH groups present in the aromatic ring. As active hydroxyl group easily provides free proton to inhibit the formation of free radicals or interrupts the propagation of free radical and thus slows down the rate of oxidation [36–38]. Low volatility of BHT and BHA also plays a crucial role in their poor performance [37]. Thus based on the results obtained from screening study of antioxidant additives, 500 ppm of TBHQ is used as the optimized concentration for further studies for oxidation stability of diesel/biodiesel blends.

2.9. Detailed stability studies

Based on screening test blends shown in Table 5 with two different concentration i.e. 500 ppm & 600 ppm of antioxidants were examined in detail by measuring their rancimat time, density and kinematic viscosity.

Table 4
Oxidation stability of *Jatropha* biodiesel with different concentrations of various antioxidants. (The values of rancimat periods in hours).

Days	0 ppm	BHT 300 ppm	BHA 300 ppm	DPA 300 ppm	TBHQ 300 ppm	BHT 400 ppm	BHA 400 ppm	DPA 400 ppm	TBHQ 400 ppm	BHT 500 ppm	BHA 500 ppm	DPA 500 ppm	TBHQ 500 ppm
0	4.2	4.3	4.32	4.3	4.34	4.32	4.32	4.32	4.38	4.36	4.36	4.31	4.42
7	3.7	3.8	3.8	3.81	3.91	3.8	3.81	3.8	3.98	3.9	3.9	3.79	4.11
14	3.2	3.4	3.38	3.35	3.45	3.5	3.36	3.38	3.51	3.5	3.44	3.36	3.65
21	3	3.16	3.11	3.07	3.15	3.18	3.1	3.11	3.21	3.2	3.15	3.1	3.32
28	2.8	2.9	2.87	2.85	2.95	2.93	2.82	2.86	2.96	2.9	2.88	2.88	3.02
35	2.6	2.75	2.67	2.73	2.81	2.75	2.67	2.68	2.88	2.73	2.71	2.68	2.94
42	2.4	2.5	2.48	2.48	2.58	2.5	2.5	2.5	2.59	2.5	2.49	2.5	2.65

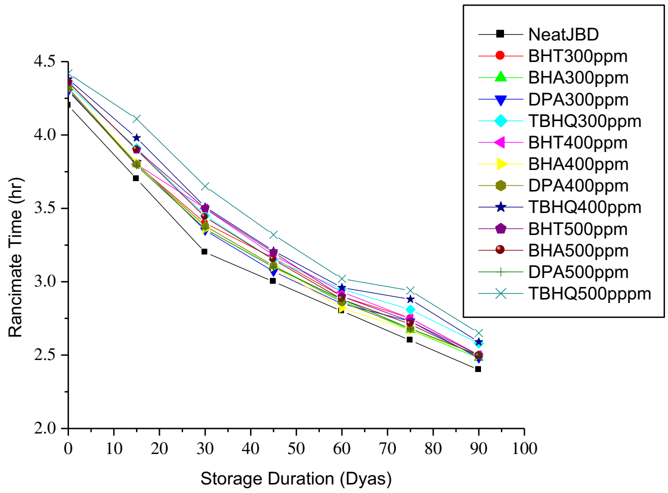

Fig. 3. Oxidation stability of *JME* with different concentrations of various antioxidants.

Initially, we studied oxidation stability of different blends without antioxidants for a period of 80 days and the result revealed that there is a strong correlation between biodiesel concentration and stability. As biodiesel concentration increases the stability is found to decrease as shown in Fig. 4. Maximum rancimat period is obtained with JB-5 and minimum with JB-40.

3. Results & discussion

The graphs given below from Figs. 5–16 show effect of different concentrations of various antioxidants on different blends JB-5, JB-10, JB-20 and JB-40 in terms of rancimat period, density and viscosity for a period of 80 days.

3.1. Rancimat period measurement of diesel biodiesel blends

Figs. 5–8 reveals performance of various samples in terms of rancimat period. For JB-5 the best performance was found with 500 ppm TBHQ with rancimat period of 47 h. With the same concentration of TBHQ improvements were also observed in viscosity as well as density. Similar pattern was observed with JB-10. So for blends with 5% and 10% biodiesel (JB-5 and JB-10) TBHQ was found to be more effective than BHA and BHT. This may be due to the presence of two hydroxyl (–OH) groups attached to aromatic ring. The function of –OH group is to provide proton that inhibit the formation of free radical or interrupt propagation of free radical thus delay the rate of oxidation. Also due to more electro negativity TBHQ offers more sites for formation of complex between free radical and antioxidant radical for the stabilization of ester chain. The poor performance of BHT and BHA can be due to their high volatility which causes their loss during the experiment. For blends with larger portion of biodiesel (JB-20 and JB-40), the results obtained are contrary to those obtained with JB-5 and JB-10, herein the stability performance of TBHQ was found to be lower than that of BHA, which may be due to pro-oxidant interaction [30] of TBHQ. The reason may be attributed to different structure of *JME* as compared to non polar hydrocarbon which may interact with the highly hindered polar phenol group of BHA to reduce their antioxidant capabilities.

Table 5
Blends of three antioxidants TBHQ, BHA, and BHT.

Blends/antioxidant (conc)	JB-5	JB-10	JB-20	JB-40
Neat sample	JB-5	JB-10	JB-20	JB-40
TBHQ	500 ppm	500 ppm	500 ppm	500 ppm
	600 ppm	600 ppm	600 ppm	600 ppm
BHA	500 ppm	500 ppm	500 ppm	500 ppm
	600 ppm	600 ppm	600 ppm	600 ppm
BHT	500 ppm	500 ppm	500 ppm	500 ppm
	600 ppm	600 ppm	600 ppm	600 ppm

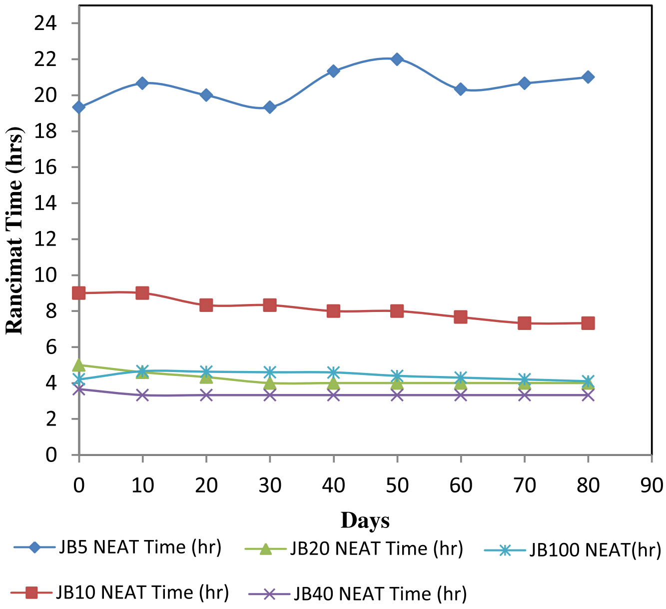

Fig. 4. Stability of various JME/EURO-III blends without antioxidants.

3.2. Density measurement of diesel biodiesel blends

A density measurement is a property for developing adequate storage methods for diesel biodiesel blends [39,40]. In diesel biodiesel blends the density of fuel increases with increasing amount of biodiesel in the mixture. The density of all the blends was observed within the range mentioned by standard ASTM-D 445.

The initial density range of neat blends (JB5, JB10, JB20 and B40) was found to be 0.8316 gm/cm^3–0.8501 gm/cm^3 with an average

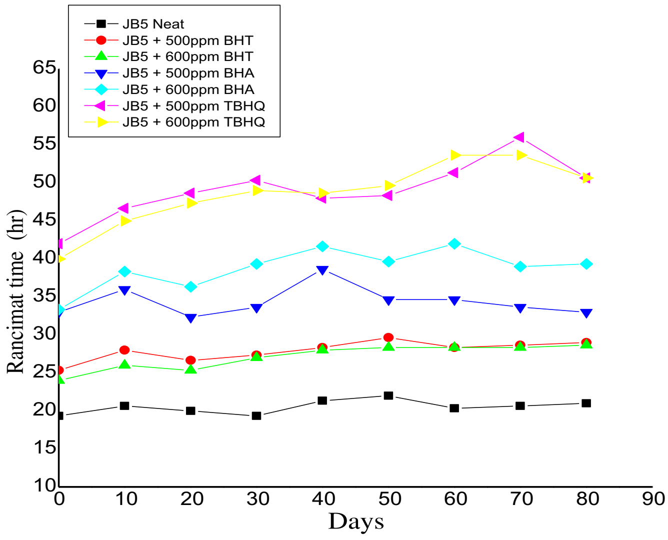

Fig. 5. Variation of rancimat period of JB-5 with various antioxidants.

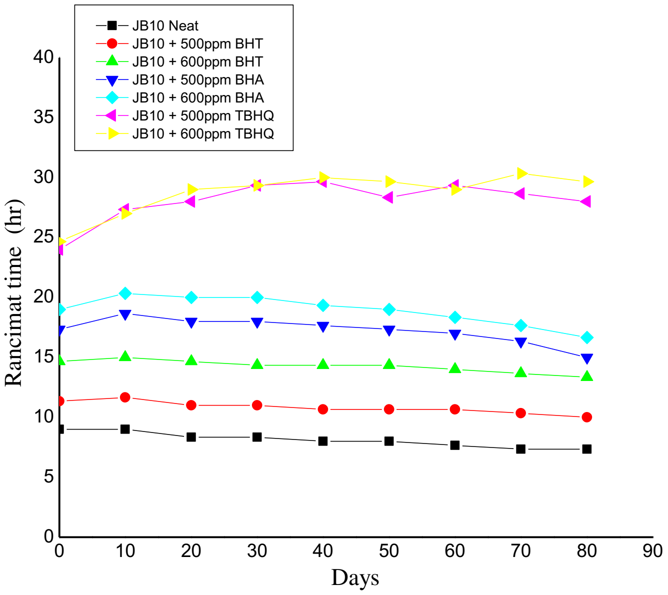

Fig. 6. Variation of rancimat period of JB-10 with various antioxidants.

initial density range of 0.838 gm/cm^3 while as the final density value for the same were ranged from 0.8334 gm/cm^3–0.8535 gm/cm^3 with an average of 0.841 gm/cm^3.

Similarly, densities of all the blends were also investigated with antioxidant and the results are shown from Figs. 9–12. The initial density of JB10 blends with additives ranged from 0.8316 gm/cm^3–0.8334 gm/cm^3 with an average of 0.832 gm/cm^3 whereas for JB-40, the initial density ranged between 0.849 gm/cm^3 and 0.852 gm/cm^3 with an average of 0.850 gm/cm$^{3.}$

3.3. *Kinematic viscosity measurement of diesel biodiesel blends*

Kinematic viscosity of all the blends were also investigated with and without antioxidant and the results are summarised in figures

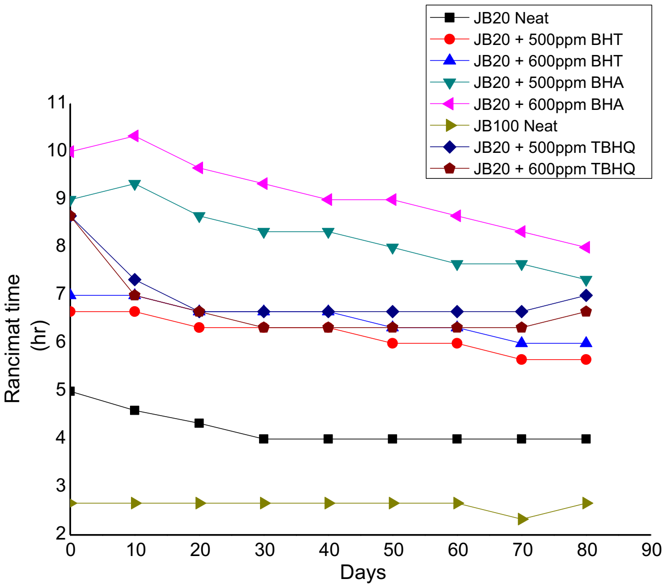

Fig. 7. Variation of rancimat period of JB-20 with various antioxidants.

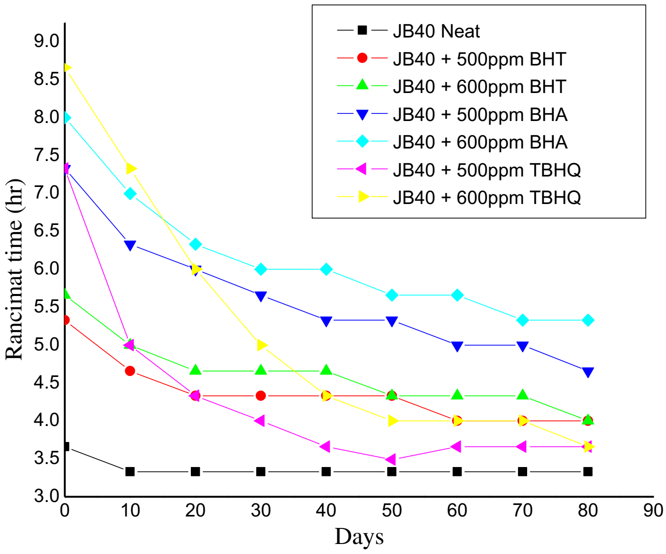

Fig. 8. Variation of rancimat period of JB-40 with various antioxidants.

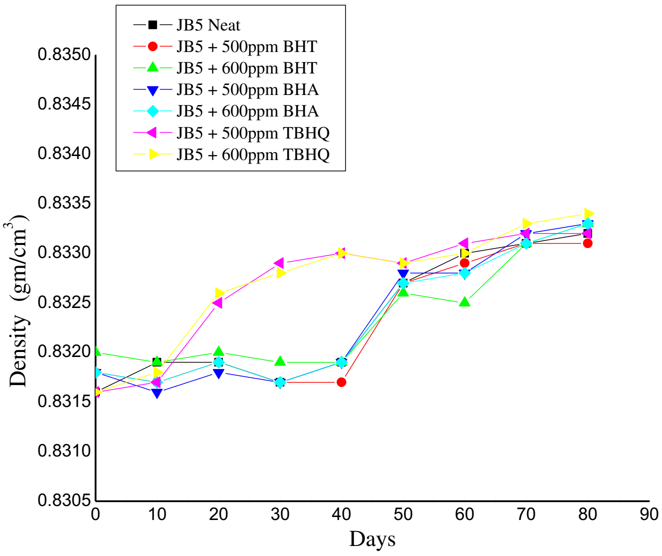

Fig. 9. Variation of density of JB-5 with various antioxidants.

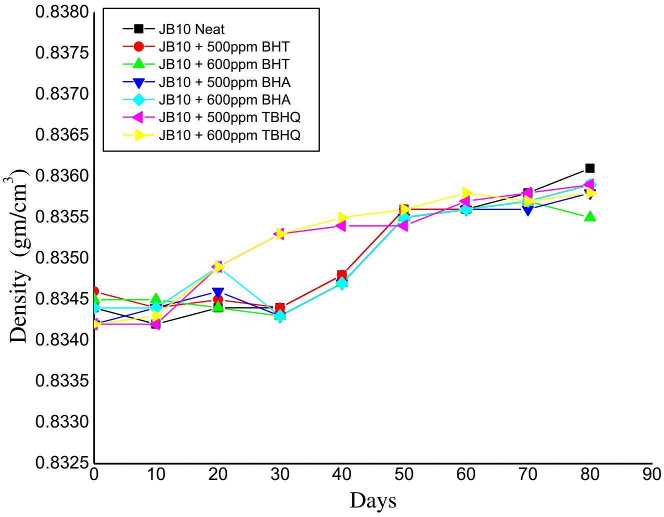

Fig. 10. Variation of density of JB-10 with various antioxidants.

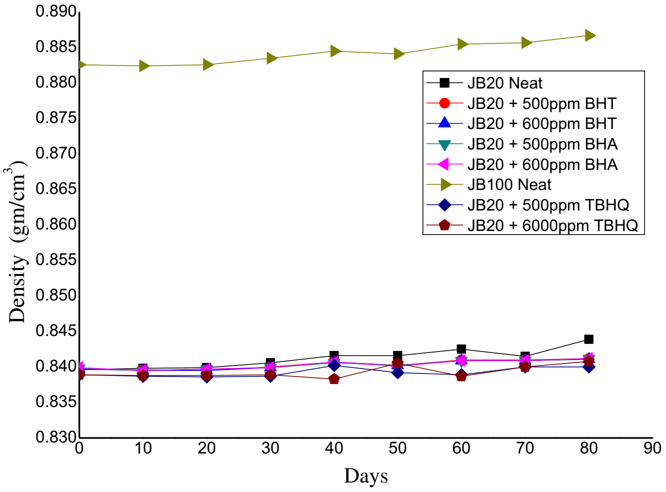

Fig. 11. Variation of density of JB-20 with various antioxidants.

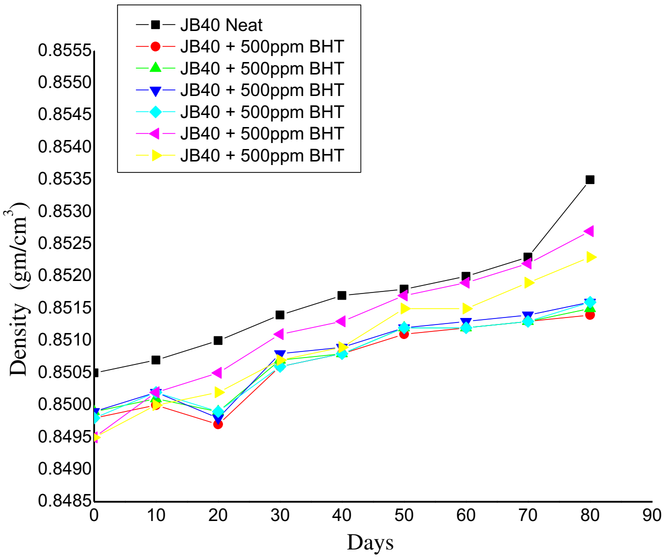

Fig. 12. Variation of density of JB-40 with various antioxidants.

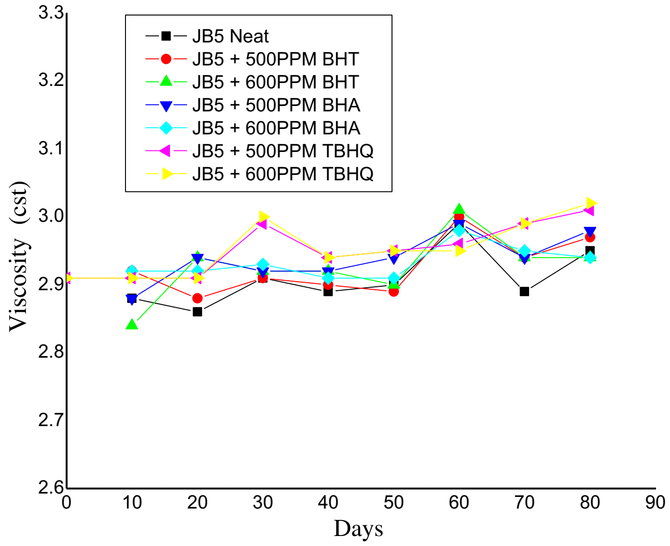

Fig. 13. Variation of viscosity of JB-5 with various antioxidants.

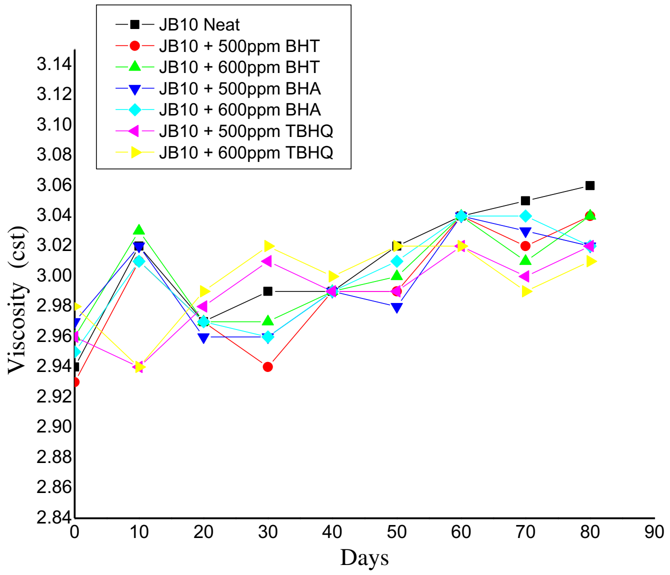

Fig. 14. Variation of viscosity of JB-10 with various antioxidants.

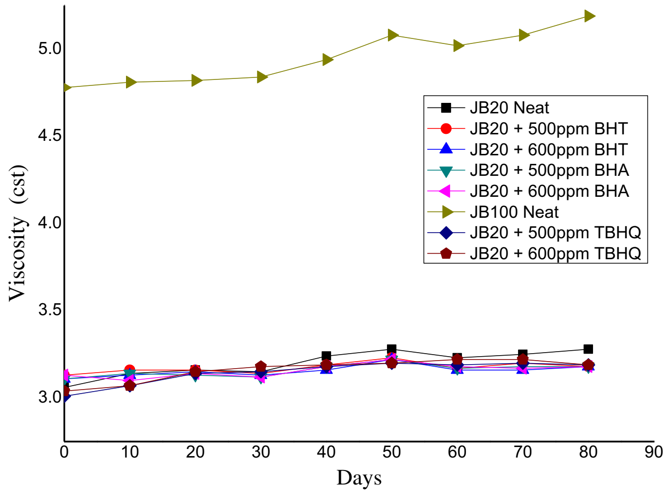

Fig. 15. Variation of viscosity of JB-20 with various antioxidants.

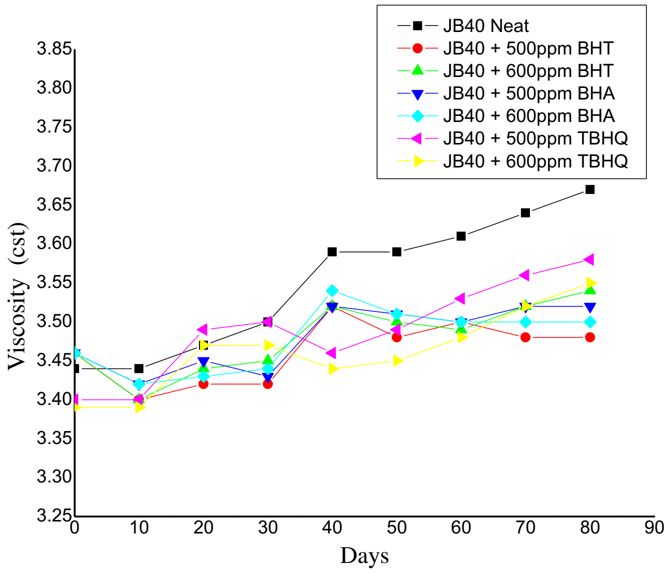

Fig. 16. Variation of viscosity of JB-40 with various antioxidants.

Table 6
Comparison of same concentration of different antioxidants on various methyl esters.

Biodiesel	IP of Neat BD (h)	Induction Period of BD with antioxidants in hr (antioxidants conc. ppm)				Ref
		TBHQ	BHA	BHT	DPA	
JB	4.2 h	8.6 h (500 ppm)	7.6 h (500 ppm)	6.2 h (500 ppm)	6.3 h (500 ppm)	Present Study
Soya biodiesel	4 h	7 h (500 ppm)	5 h (500 ppm)	4.5 h (500 ppm)	Not reported	[42]
	4 h	12 h (1000 ppm)	7 h (1000 ppm)	6 h (1000 ppm)	Not reported	[43]
Canola biodiesel	5 h	Not reported	Not reported	Not reported	13 h (100 ppm PY)	[44]

from Figs. 13–16 respectively. As during oxidation of biodiesel the viscosity increases due to formation of oxidized products which lead to the formation of sediments and gum [41].

The initial kinematic viscosity of neat diesel biodiesel blends (JB5, JB10, JB20 and JB40) ranged from 2.89 to 3.44 mm^2/s with an average value of 3.10 mm^2/s whereas the final value for the same were ranged from 2.95 to 3.617 mm^2/s with an average of 3.24 mm^2/s.

Similarly, kinematic viscosity of all the blends with antioxidants were also studied. The initial kinematic viscosity with antioxidant ranged from 2.92 to 3.43 mm^2/s, where as the final value ranged from 2.97 to 3.52 mm^2/s. The viscosity of blends were found within the range as per standard ASTM-D 445.

In Table 6, we have compared the results obtained with neat JME containing 500 ppm of different antioxidants in terms of Induction period with methyl esters from different sources as reported in the literature [42–44] with same concentration of antioxidants. From the data it is clear that with 500 ppm of different antioxidants, the maximum improvement was found with JME as compared to soya biodiesel and this may be attributed to different fatty acid structure of the methyl esters.

4. Conclusion

According to European commission the use of biodiesel should reach a minimum of 20% by 2020, therefore oxidation stability has been carried out with respect to biodiesel-EURO-III HSD blend. JME when blended with petrodiesel leads to composition having efficient and improved oxidation stability. A strong correlation was found between biodiesel concentration and stability, increasing concentration leads to decreased stability. This study revealed that with neat methyl esters and lower blends JB-5, JB-10, TBHQ was found to be most effective among all used antioxidants. Contrary to this with higher blends JB-20 and JB-40, BHA was found to be more effective. Moreover, the oxidation stability of JME showed better result as compared to that of soya biodiesel with different antioxidants.

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