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Correlation Development for the Effect of Metal Contaminants on the Thermal Stability of *Jatropha curcas* Biodiesel

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ABSTRACT: The present paper deals with the study of the effect of metal contaminants on the thermal stability of *Jatropha curcas* biodiesel (JCB). Taking pyrogallol (PY) as the most effective antioxidant based on the earlier work of the authors, JCB was mixed with different transition metals, Fe, Ni, Mn, Co, and Cu, in different concentrations. Thermal stability parameters, such as insoluble formation (Ins) and activation energy (E_a), were measured using American Society for Testing and Materials (ASTM) D6468 and thermogravimetric analysis (TGA) methods. On the basis of the results, several correlations are developed for assessing the thermal stability in terms of Ins and E_a as a function of the antioxidant and metal concentrations. A comparison between the experimental Ins and E_a values and those predicted by the correlation shows that about 95% of the predicted data points lie within ±10% deviation lines of the experimental results. This is the first study of its kind being reported showing the relationship of thermal stability with antioxidant concentration and metal contaminants. The correlations developed can be used to predict the amount of antioxidants required to stabilize the JCB.

INTRODUCTION

Nowadays, biodiesel is considered as an important alternative biofuel because of its environmental benefits and simple industrial production from renewable resources. New investment opportunities for industrial-scale plants in Europe, Asia, Australia, United States, and Brazil have led to the increased production of biodiesel using vegetable-oil-based feedstocks with the added advantage of higher lubricity compared to petrodiesel. However, such eco-friendly liquid fuels have lower oxidation stability, which ultimately affects their marketability.[1] Transesterification of oil or fats with short-chain alcohol, usually methanol and ethanol, produces a mixture of corresponding monoalkyl esters defined as biodiesel. The biodiesel has the same fatty acid compositions as the parent oils or fats, with a considerable proportion of unsaturated fatty acids. Its oxidative stability, therefore, becomes a crucial quality issue during long-term storage.[2] Ultraviolet irradiation, high-temperature exposition, and the presence of trace metallic elements can reduce the overall stability of the biofuel, thereby significantly impacting its fuel quality. On the other hand, the oxidative degradation by the air also affects some properties of the biodiesel, such as kinematic viscosity, cetane number, and acid value of the fuel.[3] A number of research groups are working to find out the chemical substances that inhibit this oxidation process and improve the quality of biodiesel.[2,4]

Different vegetable oils obtained from soybean, castor, sunflower, cotton, corn, palm, etc. are widely used for biodiesel production in different parts of the world depending upon the cultivation of oil crops. India imports about 40−50% of total domestic edible oil demand,[1,5] and therefore, it is impossible to divert these resources for biodiesel production. Therefore, attention has been directed to the use of non-edible oil resources, such as *Jatropha*, pongamia, neem, etc., for biodiesel production in the country. In India, *Jatropha curcas* plantations are under cultivation on more than 40 000 ha of land under the National Biofuel Program of the Government of India. The oil from the seeds of the *Jatropha curcas* plant will become the source of biodiesel, which is likely to offer the substitute of diesel fuel on a significant scale in India.

Mittelbach and Schober[6] have studied the influence of antioxidants on the oxidation stability of biodiesel and showed that the oxidation stability is influenced by the addition of different natural and synthetic antioxidants. Dunn et al.[7] examined the effectiveness of five antioxidants, viz., *tert*-butylhydroquinone (TBHQ), butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), propyl gallate (PrG), and α-tocopherol, in mixtures with soybean oil fatty acid methyl esters (SMEs) and found that increasing the antioxidant concentration increases the activity, also leading to an increased oxidation stability.

Sarin et al.[8] have worked on finding the optimum mix of different blends of palm and *Jatropha* biodiesel for improved oxidation stability. The effect of natural and synthetic antioxidants on the oxidative stability of palm biodiesel was examined by Liang et al.,[9] who found that crude palm oil methyl ester (CPOME) containing 600 ppm of vitamin E was found to exhibit oxidative stability of more than 6 h as per the specifications of the European standard for biodiesel (EN 14214). Sarin et al.[10] have further evaluated the influence of metal contaminants on the oxidation stability of *Jatropha* biodiesel and found that the metals had a detrimental effect on the oxidation stability. Even small concentrations of metal contaminants showed nearly the same effect on the oxidation stability as large concentrations. Cu has been found to have the strongest detrimental and catalytic effect. Fritsch et al.[11] have examined the effect of antioxidants on refined

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Table 1. Physico-chemical Properties of Biodiesel as Per Different Standards

property (unit)	ASTM D6751	ASTM D6751 limits	IS 15607	IS15607 limits	*Jatropha* methyl ester
flash point (°C)	D93	minimum of 130	IS 1448		172
viscosity at 40 °C (cSt)	D445	1.9−6.0	IS 1448		4.38
water and sediment (vol %)	D2709	maximum of 0.05	D2709	maximum of 0.05	0.05
free glycerin (% mass)	D6584	maximum of 0.02	D6584	maximum of 0.02	0.01
total glycerin (% mass)	D6584	maximum of 0.24	D6584	maximum of 0.24	0.03
oxidation stability of FAME (h)	EN 14112	3	EN 14112	minimum of 6	3.27
oxidation stability of FAME blend (h)			EN 590	minimum of 20	
free glycerol	D6584	maximum of 0.02	D6584	maximum of 0.02	0.01
total glycerol	D6584	maximum of 0.25	D6584	maximum of 0.25	0.12
acid value	D664	maximum of 0.5	D664	maximum of 0.5	0.38
ester content			EN 14103	maximum of 96.5	98.5

Table 2. Fatty Acid Composition of JCB

fatty acid	percent composition (%)
palmitic acid (P)	16.8
stearic acid (S)	7.7
oleic acid (O)	39.1
linoleic acid (L)	36.0
linolenic acid (LL)	0.2

palm oil and found TBHQ to have a better effect as an antioxidant on refined palm oil than BHT and BHA. Although numerous papers are available on the storage, thermal and oxidation stability of biodiesel, and the effect of antioxidants on the stability of biodiesel synthesized from edible oils, little work is reported on the oxidation and thermal stability of biodiesel from non-edible oil seeds and the influence of the presence of metal on the oxidation and thermal behaviors of biodiesel from non-edible oil seeds. Accordingly, there is a strong need to develop correlations that can be used to determine the amount of antioxidants actually required to be added to stabilize the biodiesel.

The present paper reported the results of the study carried out on the influence of the presence of metals on the thermo-oxidation behavior of *Jatropha curcas* biodiesel (JCB) and the effect of the antioxidant [pyrogallol (PY)] on the oxidation behavior JCB doped with metal. Different transition metals, Fe, Ni, Mn, Co, and Cu, commonly found in the alloys and metallurgy used in the manufacturing of storage tanks and barrels were blended with varying concentrations (mg/L) in JCB. The effectiveness of different antioxidants has already been reported in our earlier publication, in which PY was found to be the most effective antioxidant, and therefore, only PY is used in the present study.[12] On the basis of the results, different correlations are developed for the induction period (IP), insoluble formation (Ins), and activation energy (E_a) as a function of antioxidants and metal contaminants.

MATERIALS

All of the chemicals, including PY, were of analytical grade (AR) and purchased from Sigma Aldrich, India. Different transition metals, Fe, Ni, Mn, Co, and Cu, were also purchased from Sigma Aldrich, India. Biodiesel was prepared using two-step acid−base-catalyzed transesterification processes developed by the authors and reported in our previous publications.[13,14] Physico- chemical properties of JCB are given in Table 1. Fatty acid composition of JCB is shown in Table 2. Biodiesel is mixed with predetermined concentrations of different metal contaminants and PY. Then, biodiesel samples were subjected to the Rancimat test to measure the IP.

EXPERIMENTAL SECTION

Measurement of Thermal Stability. Thermal stability of JCB was quantified by the Ins and E_a, which were evaluated as per the American Society for Testing and Materials (ASTM) D6468 and thermogravimetric analysis (TGA) methods, respectively. The procedure is discussed below.

ASTM D6468. The thermal stability of JCB is measured using modified ASTM D6468.[15] Accordingly, JCB and their blends with diesel are heated at 150 °C for 180 min during exposure to air. After aging and cooling, the fuel samples are filtered and the average filterable insolubles are estimated by the gravitational method. The results are discussed in the next section. The test method makes use of a filter paper with a nominal porosity of 11 μm, which does not capture all of the sediment formed during aging but allows for differentiation over a broad range.

TGA. The thermogravimetric thermogram of JCB was recorded on a thermogravimetric analyzer (Perkin-Elmer Pyris 6) using alumina pans. The thermal analysis was conducted at a heating rate of 10 °C/min from 10 to 700 °C in a dry air atmosphere of 100 mL/min. A sample size of about 15 mg was used. The temperature and weight scales were calibrated using indium over a specific range of heating rates, with a calibration parameter over its respective Curie point.

Thermogravimetric data have been used to characterize the materials as well as investigate the thermodynamics and kinetics of the reactions and transitions of oil samples in oil industries. The method can also be adopted to analyze the thermal behavior of biodiesel. Currently, several methods are available in the literature that can be used to calculate the kinetic parameters.[16] The kinetic analysis used for the thermal conversion of the biodiesel is discussed below.

The rate of thermal deterioration, dx/dt, for the biodiesel is expressed by

$$dx/dt = k(f) = k(1-x)^n \qquad (1)$$

where n is the order of reaction, k is the reaction rate constant, and x is the extent of conversion

x is given by

$$x = \frac{w_o - w_t}{w_o - w_\infty}$$

where w_o, w_t, and w_∞ are the original, current, and final weights of sample, respectively.

On the basis of the TGA thermogram, reaction 1 was found to be first-order.

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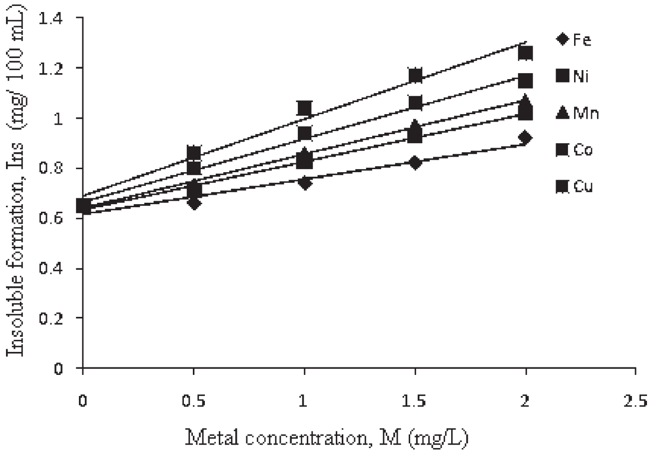

Figure 1. Effect of metal contaminants on the thermal stability of JCB.

Thus, $n = 1$, and eq 1 becomes

$$\frac{dx}{dt} = k(1 - x)$$

For the non-isothermal case, the above equation can be further modified to

$$\frac{dx}{dT}\frac{dT}{dt} = k(1 - x) \tag{2}$$

where dT/dt is the heating rate B.

According to the Arrhenius relationship, the reaction rate constant k in eq 2 can be expressed as

$$k = A\exp(-E_a/RT) \tag{3}$$

where E_a, A, and R are the activation energy, frequency factor, and ideal gas law constant (8.314 J mol^{-1} K^{-1}), respectively.

Substituting eq 3 into eq 2 yields

$$\frac{dx}{dt} = \frac{A}{B}\exp\left(\frac{-E_a}{RT}\right)(1 - x) \tag{4}$$

For the direct Arrhenius plot method for the non-isothermal kinetic parameters with a constant heating rate ($B = dT/dt$), eq 4 was rearranged to

$$\ln\left[\frac{1}{(1-x)}\frac{dx}{dt}\right] = \ln\frac{A}{B} - \frac{E_a}{RT} \tag{5}$$

The plot $\ln[1/(1 - x)dx/dt]$ versus $1/T$ should give a straight line with slope $-E_a/R$, from which the activation energy, E_a, can be calculated.

RESULTS AND DISCUSSION

Effect of the Metal Concentration on Thermal Stability. To determine the effect of metal contaminants on thermal stability, the experiments were conducted by mixing the biodiesel with different metal contaminants in predetermined concentrations with different concentrations of PY. Each experiment has been conducted twice, and the average value of the same is considered for further calculation. Figure 1 also shows that, for all of the metal contaminants, Ins values increase with the amount of metal contaminants because of accelerated oxidation of JCB in the presence of metal contaminants.[10,12]

Panels a−e of Figure 2 show the variation of Ins with varying concentrations of metal contaminants at a constant concentration of PY for Fe, Ni, Mn, Co, and Cu, respectively. The presence of these metals reduces the thermal stability of biodiesel as measured by the Ins. This may be attributed because of the acceleration of free-radical oxidation as a result of a metal-mediated initiation reaction and because of this formation of insolubles accelerated.[10,12] Cu is found to have the strongest catalytic effect, followed by Co, Mn, Ni, and Fe. The figure shows that increasing amounts of antioxidant lead to reducing values of Ins. Because the amount of metal contaminant is more in the biodiesel, the effectiveness of the antioxidant decreases accordingly. Cu is found to have a maximum catalytic effect on thermal stability. At the same time as the amount of PY increases for the same metal concentration, the Ins formed was decreased because of retardation in the oxidation process.[2,6−10,12]

Panels a−e of Figure 3 show the variation of E_a with varying concentrations of metal contaminants at a constant concentration of PY for Fe, Ni, Mn, Co, and Cu, respectively. The presence of these metals reduces the thermal stability of biodiesel as measured by E_a. The reason for this is the same because of the acceleration of free-radical oxidation as a result of a metal-mediated initiation reaction, which in turn increases the polymer formation as well.[10,12] In this case also, E_a increases as the amount of PY increases because of retardation in the oxidation process.[2,6−10,12]

Correlation Development for Thermal Stability. The results of the effect of the antioxidant and metal concentrations indicated that these parameters play a critical role in the thermal stability of biodiesel. The biodiesel producer may use a correlation to know the amount of antioxidant required to maintain the thermal stability of metal-contaminated biodiesel conforming to the international standard. A correlation is developed for Ins and E_a as a function of metal contaminant and antioxidant concentrations. A correlation development technique for Fe-contaminated biodiesel is discussed below.

The equation for Ins can be written as

$$\mathrm{Ins} = f(M, A) \tag{6}$$

where A is the antioxidant concentration and M is the metal contaminant concentration. A correlation has been statistically developed for Ins by regression analysis of the experimental data. To determine the functional relationship between Ins and the metal concentration (M), a set of data points for different values of metal concentrations has been plotted on a log−log scale, as shown in Figure 4.

Figure 4 shows a monotonic increase of Ins with an increase in the metal concentration. It is observed that the data yields straight lines with nearly the same slope, while the value of intercept of each line is slightly different. Similar plots of the ln(Ins) versus ln(M) for different sets of antioxidant concentration were drawn, and it was observed that, in all of the cases, the slopes of different lines are nearly the same, while the value of intercept of each line is slightly different. The functional relationship between Ins and the metal concentration was therefore found to follow the equation given below

$$\ln(\mathrm{Ins}) = n\ln(M) + X_1 \tag{7}$$

Equation 7 can be written as

$$\mathrm{Ins} = X_0(M)^n \tag{8}$$

where X_0 is $\exp\{X_1\}$.

The least-squares method is used to fit the best curve through all of the data points pertaining to 20 Fe-contaminated biodiesel

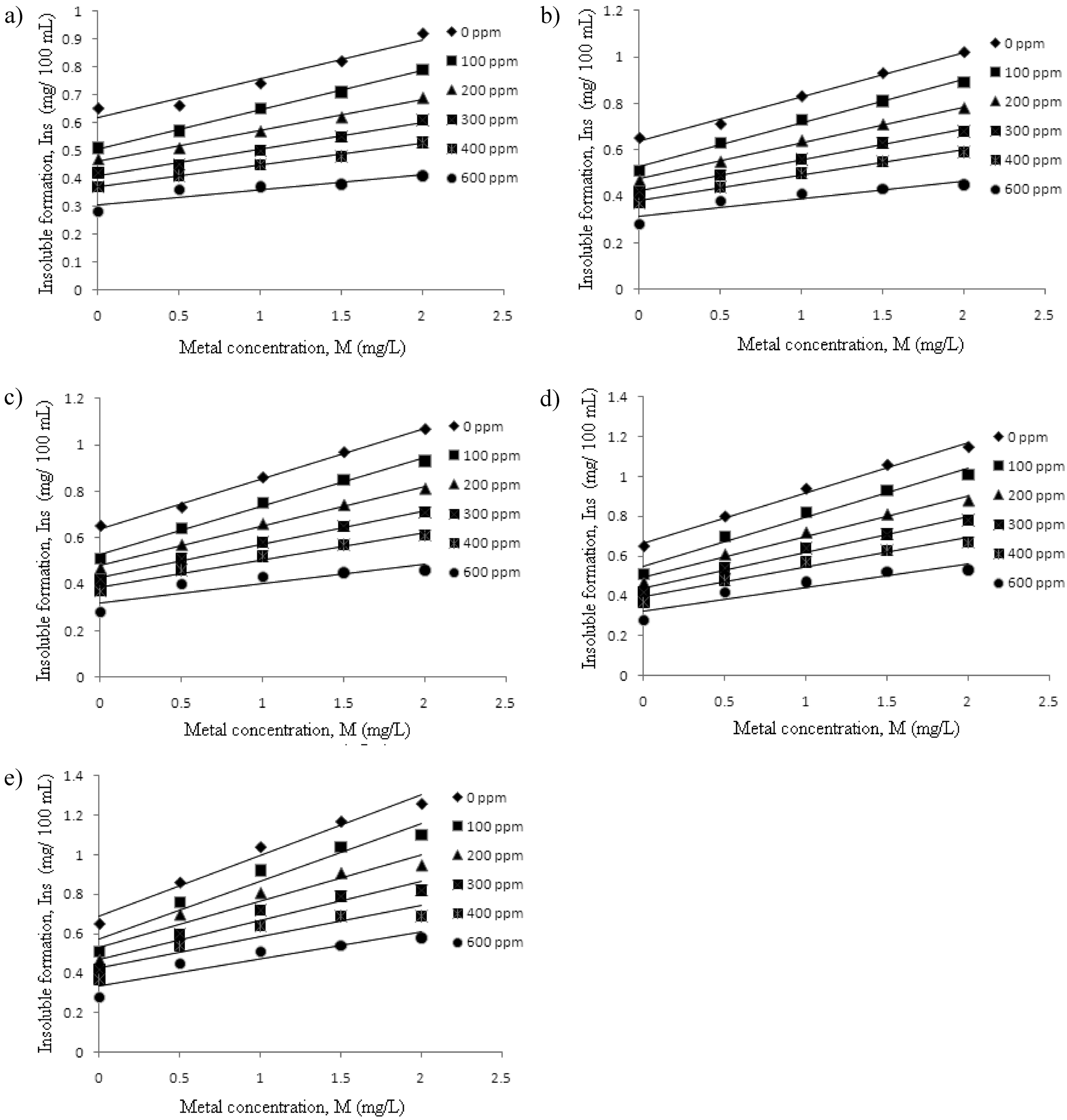

Figure 2. Variation in Ins with (a) Fe, (b) Ni, (c) Mn, (d) Co, and (e) Cu concentrations at different levels of the antioxidant concentration.

samples, as shown in Figure 4, and the relationship was obtained as

$$\mathrm{Ins} = X_0(M)^{0.1817} \tag{9}$$

In eq 9, the value of constant X_0 is a function of the metal concentration.

The functional relationship between Ins and the relative antioxidant concentration (A) was found to follow the equation given below

$$\ln(X_0) = n\ln(A) + Y_1 \tag{10}$$

Equation 10 can be written as

$$X_0 = Y_0(A)^n \tag{11}$$

where Y_0 is $\exp\{Y_1\}$.

As shown in Figure 5, a regression analysis to fit a straight line through data points yields

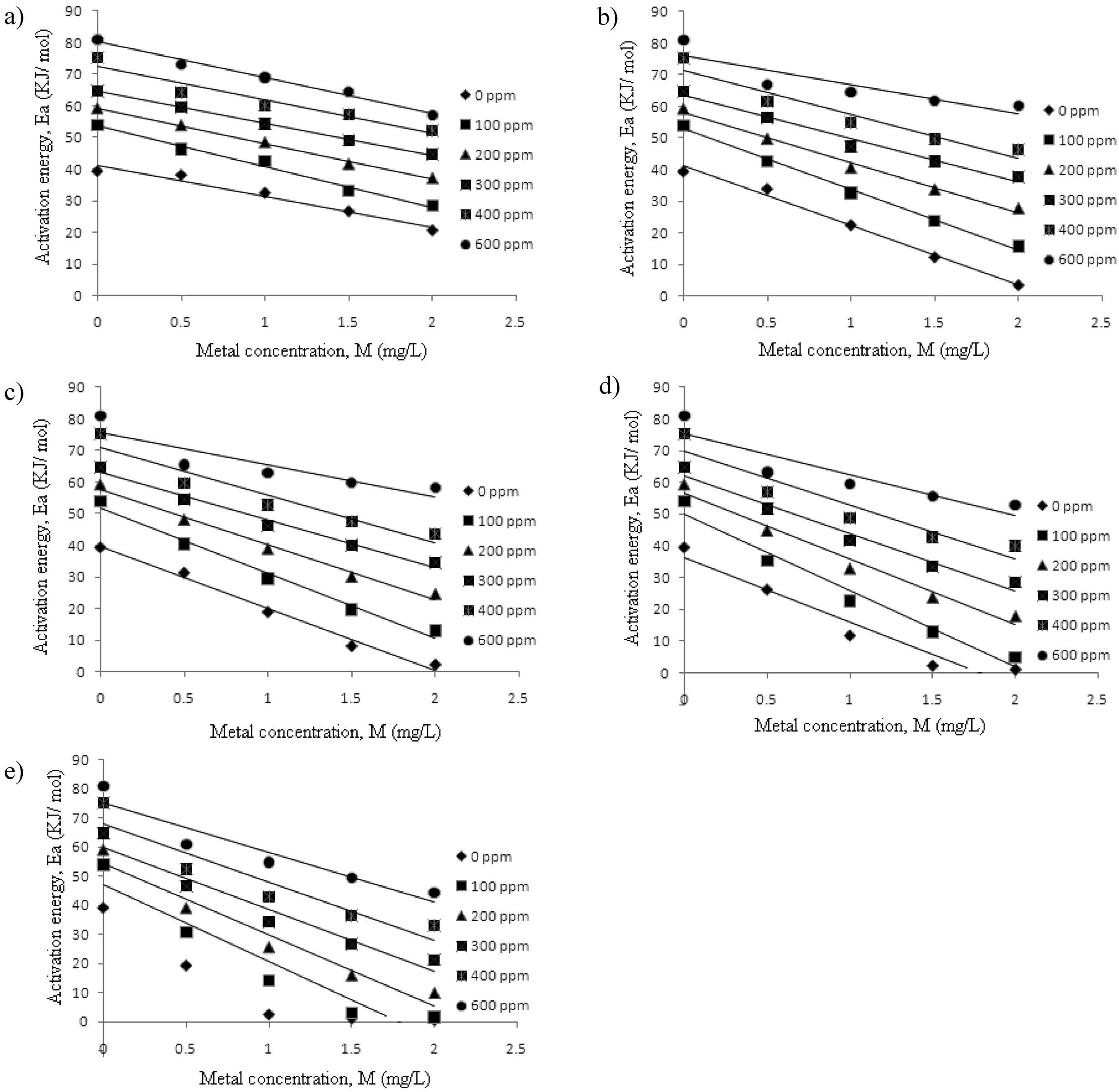

Figure 3. Variation in E_a with (a) Fe, (b) Ni, (c) Mn, (d) Co, and (e) Cu concentrations at different levels of the antioxidant concentration.

$$X_0 = Y_0(A)^{-0.3156} \tag{12}$$

Equation 12 can be written as

$$\mathrm{Ins}/(M)^{-0.1817} = Y_0(A)^{-0.3156} \tag{13}$$

$$\mathrm{Ins} = Y_0(M)^{0.1817}(A)^{-0.3156} \tag{14}$$

$$\mathrm{Ins} = 2.975(M)^{0.1817}(A)^{-0.3156} \tag{15}$$

In the same manner for activation energy

$$\ln(E_a) = n\ln(M) + X_1 \tag{16}$$

Equation 16 can be written as

$$E_a = X_0(M)^n \tag{17}$$

where X_0 is $\exp\{X_1\}$.

The least-squares method is used to fit the best curve through all of the data points pertaining to 20 Fe-contaminated biodiesel samples, as shown in Figure 6, and the relationship was obtained as

$$E_a = X_0(M)^{-0.2239} \tag{18}$$

In eq 18, the value of constant X_0 is a function of the metal concentration.

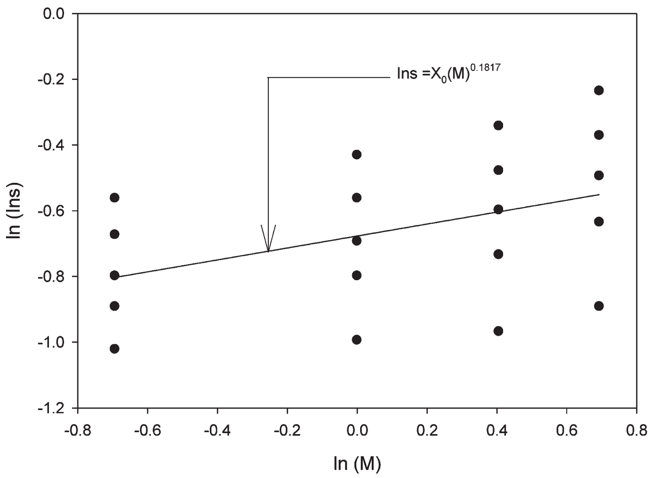

Figure 4. Plot of ln(Ins) and ln(M) for all of the experimental data for Fe-contaminated biodiesel.

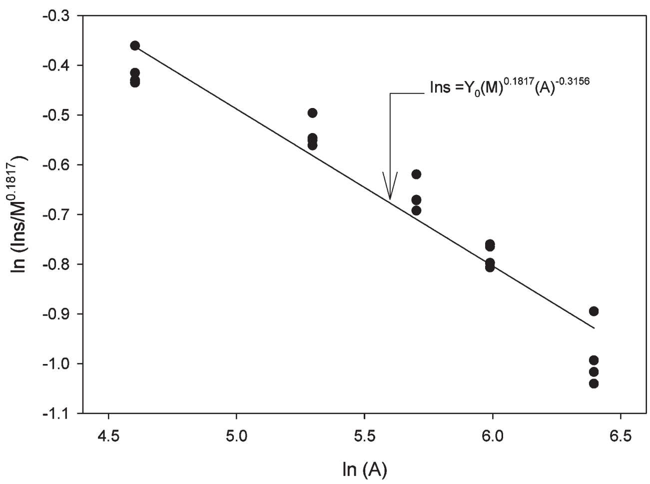

Figure 5. Plot of ln[Ins/(M)$^{0.1817}$] and ln(A) for all of the experimental data for Fe-contaminated biodiesel.

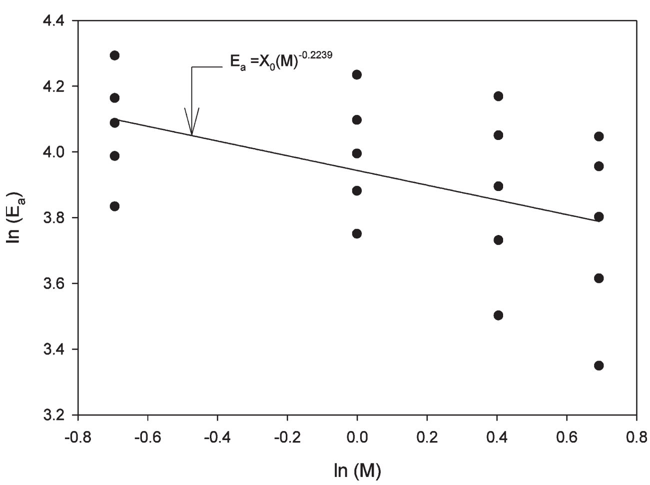

Figure 6. Plot of ln(E_a) and ln(M) for all of the experimental data for Fe-contaminated biodiesel.

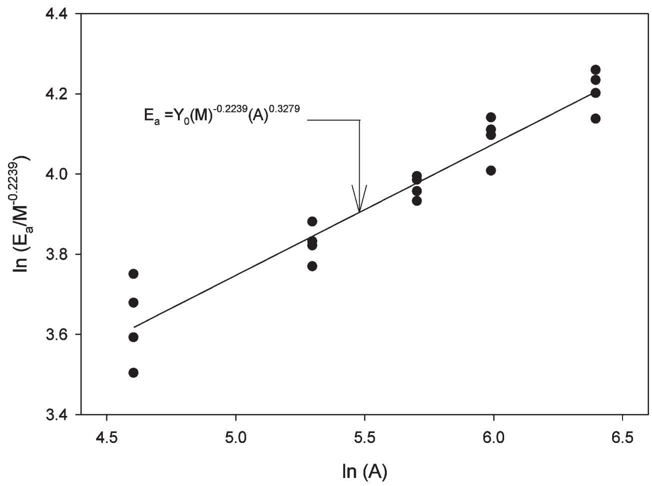

Figure 7. Plot of ln[E_a/(M)$^{-0.2239}$] and ln(A) for all of the experimental data for Fe-contaminated biodiesel.

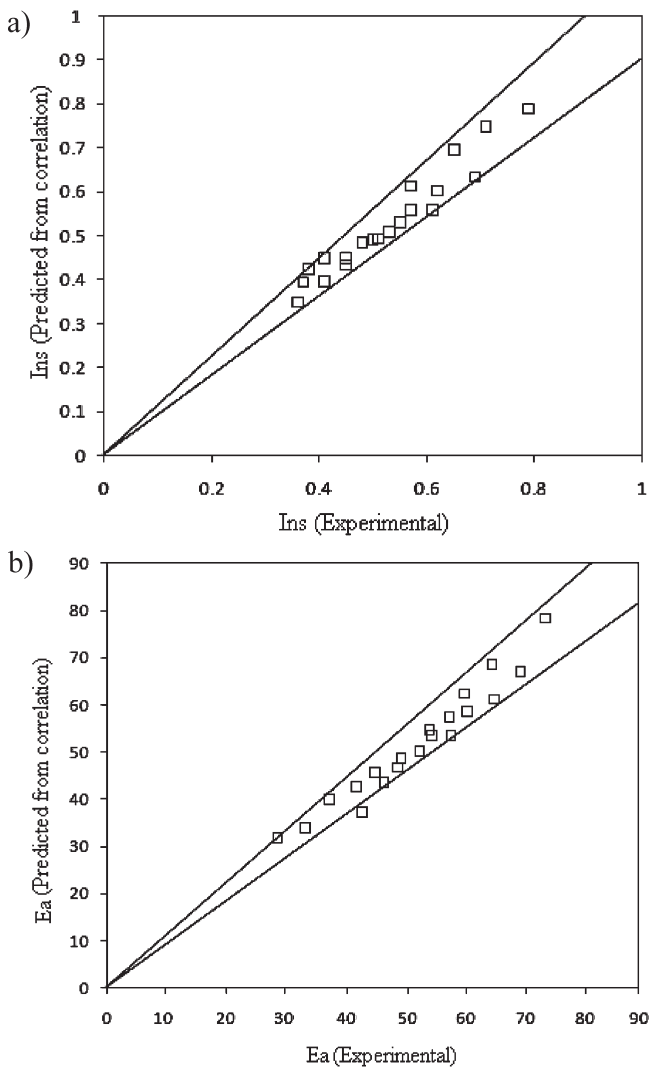

Figure 8. Comparison of experimental and predicted values of (a) Ins and (b) E_a for Fe-contaminated JCB.

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Table 3. Correlation Developed for Ins and E_a for Different Metal Contaminants

metal	correlation	regression coefficient
Fe	$\text{Ins} = 2.975(M)^{0.1817}(A)^{-0.3156}$	0.92
	$E_a = 8.23(M)^{-0.2239}(A)^{0.3279}$	0.92
Ni	$\text{Ins} = 3.534(M)^{0.2122}(A)^{-0.3277}$	0.93
	$E_a = 2.975(M)^{-0.3313}(A)^{0.4840}$	0.85
Mn	$\text{Ins} = 3.586(M)^{0.2124}(A)^{-0.3237}$	0.92
	$E_a = 2.022(M)^{-0.3796}(A)^{0.5419}$	0.85
Co	$\text{Ins} = 3.751(M)^{0.2418}(A)^{-0.3164}$	0.95
	$E_a = 0.499(M)^{-0.5474}(A)^{0.7625}$	0.77
Cu	$\text{Ins} = 4.976(M)^{0.2003}(A)^{-0.3511}$	0.90
	$E_a = 0.0395(M)^{-0.8432}(A)^{1.1645}$	0.74

The functional relationship between E_a and relative antioxidant concentration (M) was found to follow the equation given below

$$\ln(X_0) = n \ln(A) + Y_1 \quad (19)$$

Equation 19 can be written as

$$X_0 = Y_0(A)^n \quad (20)$$

where Y_0 is $\exp\{Y_1\}$.

As shown in Figure 7, a regression analysis to fit a straight line through data points yields

$$X_0 = Y_0(A)^{0.3279} \quad (21)$$

Equation 21 can be written as

$$E_a/M^{-0.2239} = Y_0(A)^{0.3279} \quad (22)$$

$$E_a = Y_0(M)^{-0.2239}(A)^{0.3279} \quad (23)$$

$$E_a = 8.23(M)^{-0.2239}(A)^{0.3279} \quad (24)$$

A comparison between Ins and E_a obtained from experimental investigation and those predicted by the correlation is shown in panels a and b of Figure 8, respectively, which shows that about 95% of the predicted data points lie within ±10% deviation lines of the experimental results. The value of the regression coefficient has been found as 0.92.

A similar procedure has been employed to develop a statistical correlation for Ins and E_a for other metal-contaminated biodiesels based on regression analysis of data obtained from the experimental investigations. These correlations are given in Table 3. The comparison between the experimental values of Ins and E_a and those predicted using the correlation for the other four metals has also been carried out, and it is found that about 95% of the predicted values of the data lie within ±10% of experimentally observed data. The regression of data for the correlation has regression coefficient values of 0.92, 0.93, 0.92, 0.95, and 0.9 for Fe, Ni, Mn, Co, and Cu, respectively, for calculating Ins. The regression coefficient for E_a is 0.92, 0.85, 0.85, 0.77, and 0.74 for Fe, Ni, Mn, Co, and Cu, respectively. Therefore, the correlations developed can be used to predict the Ins and E_a with reasonable accuracy in the range of parameters investigated for JCB. To validate the correlation, experiments were performed further and the results were found in good agreement with the predicted values from correlations. This is the first study of its kind being reported. The correlations developed can be used to predict the amount of antioxidants required to maintain the specification of thermal stability for JCB with reasonable accuracy in the range of parameters investigated.

CONCLUSIONS

In the present paper, the effect of metal contaminants on the thermal stability of biodiesel has been studied with and without antioxidants. The results indicated that Cu had the strongest catalytic effect, followed by Co, Mn, Ni, and Fe. On the basis of the results of various experiments, a number of correlations have also been developed for oxidation stability in terms of Ins and E_a as a function of the antioxidant and metal concentrations. A comparison between Ins and E_a obtained from experimental investigation and those predicted by the correlation shows that about 95% of the predicted data points lie within ±10% deviation lines of the experimental results. The value of the regression coefficient has been found to be 0.92, indicating that the correlations can be used to predict the concentration of antioxidants required to be added to biodiesel to maintain the thermal stability specifications.

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