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Original Research Paper

Effect of Oxidation on Fuel Characteristics of Palm Oil Biodiesel During Storage

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ABSTRACT

The expected scarcity of petroleum supplies and the negative environmental consequences of fossil fuels have spurred the search for renewable and non-traditional energy sources and sustained conservation of fossil fuel. Biodiesel is an alternative fuel for diesel engines that can be produced from renewable feedstocks such as vegetable oil and animal fats. These feedstocks are with an alcohol to produce alkyl monoesters that can be used in conventional diesel engines with little or no modification. Previous research has shown that biodiesel fuelled engines produce less carbon monoxide, unburned hydrocarbons, and particulate emissions compared to diesel fuel. One drawback of biodiesel is that it is more prone to oxidation than petroleum-based diesel fuel. During long term storage, oxidation due to contact with air (auto-oxidation) presents a legitimate concern with respect to maintaining fuel quality of biodiesel. This work reported the results of the study of oxidation on the physico-chemical properties of biodiesel produced from Palm oil (*Elaeis guinensis* Jacq) and its blend with diesel through the different period of storage life. This oxidation results in increase in kinematic viscosity, relative density and acid value. While the flash and fire point and calorific value are decreased as oxidation proceeds.

INTRODUCTION

Biodiesel is an oxygenated, sulphur-free, biodegradable, non-toxic, and environmental friendly alternative diesel fuel. Biodiesel is defined as the alkyl monoesters of fatty acids from renewable resources, such as vegetable oils, animal fats, and waste cooking oils. Biodiesel has positive performance attributes such as increased cetane, high fuel lubricity, and high oxygen content. However, biodiesel still has many disadvantages related to their long term storage and thermal stability. Biodiesel is a mixture of fatty acid mono alkyl esters with relatively high concentrations of long-chain mono and poly-unsaturated compounds present. Unsaturated compounds are significantly more reactive to oxidation than saturated compounds; increasing the degree of unsaturation further increases reactivity (Natarajan 2012). The unsaturated and polyunsaturated fatty acids in biodiesel are significantly more reactive to oxidation than saturated compounds, this is because the unsaturated fatty acids contain the most reactive sites which are particularly susceptible to the free radical attack, particularly oxygen (Sarin et al. 2009). The stability of biodiesel generally depends on the nature of fatty acid composition of the parent oil as well as certain functional groups present in biodiesel molecule. All biodiesel have significant amount of esters of oleic, linoleic or linolenic acids and the trend of increasing stability is linolenic<linoleic<oleic etc. (Jain & Sharma 2011). The stability of biodiesel is inferior to petro-diesel and therefore, the blending of biodiesel with petrol-diesel will affect its fuel stability significantly (Dunn & Knothe 2003). In a similar work on biodiesel degradation due to feedstock composition, Maria et al. (2009) studied the influence of raw material composition on biodiesel quality, they found that properties can be strongly affected if the biodiesel is improperly stored and transported. They concluded that biodiesel fuel quality could be harmed by the oxidation products, which are corrosive to engine chambers and may lead to clogging of the injection pumps and filters, besides increasing the biodiesel viscosity. When biodiesel runs bad, there are some measurable effects, for example, acid number increases as the fatty acid breaks down into shorter chain acids, the kinematic viscosity increases as the fatty acids polymerizes and clump together, the peroxide value increases as free radicals oxidize into peroxide and hydroperoxide, and finally the sediments increases as the polymerization increases (Jain & Sharma 2011).

From the literature review, it can be deduced that biodiesel is prone to oxidation and hence the storage of biodiesel over a long period of time should be a subject of serious concern, that will require a possible solution in order to sustain biodiesel quality. Generally, non-edible oil composed by high number of double carbon chain (polyunsaturated acid) indicated that non-edible oil has a greater degree of unsaturated fatty acid than saturated carbon chain acid (Rizwanul et al. 2013). Therefore, this structural fatty acid composition will influence the physico-chemical properties of biodiesel such as cetane number, heat of combustion and viscosity more than edible oil (Atabani et al. 2013, Canakci & Sanli 2008). The palm vegetable oil can be chosen among different vegetable oils for its high yielding capacity and presence of high level of methyl oleate. Various studies on characterization properties of biodiesel have suggested that biodiesel with a high level of methyl oleate (monounsaturated fatty acid) may have excellent characteristics in ignition quality, fuel stability and flow properties at low temperature (Pinzi et al. 2009). The methyl ester of palm oil has been used satisfactorily for partial replacement of diesel fuel (Deepanraj & Dhanesh 2011). Therefore the determination of storage stability in this work is an attempt to evaluate measurable effect of oxidation on fuel properties of methyl ester of palm oil and its blend with diesel when it deteriorates as a result of a long period of storage.

MATERIALS AND METHODS

Raw Material

Palm oil: The biodiesel produced from palmolein used in this study was purchased from the market. The percentage content of different fatty acids in palm oil is given in Table 1. The FFA content of palm oil was found to be 0.811% (1.63 mg KOH/g of oil), therefore, this oil can be used directly for base catalyzed transesterification reaction. The maximum percentage of FFA content allowed is 1%. Here, the abbreviations MEPO will be used for methyl ester of palm oil. The commercial petroleum-based diesel fuel used as a reference fuel was purchased from a gas station.

Preparation of fuel blends: Methyl ester of palm oil-diesel blends were prepared by blending 10 to 30 % of methyl ester with diesel on volume basis. The biodiesel were stored in dark closed containers at room temperature (temperature variation between 25 and 30°C) for the period of 18 months, during which time the chemical properties were analysed after each 6th month.

Table 1: Composition of palm oil used as a reactant.

Fatty acid	Percentage by weight	Reference
Palmitic Acid	33.73	32-45
Stearic Acid	4.0	2-7
Oleic Acid	50.0	38-52
Linoleic Acid	2.76	5-11
Myristic Acid	2.5	0.6-1.6
Others	7.0	-

Analysis of chemical properties: The biodiesel was characterized for kinematic viscosity, relative density, heating value, acid value (AV) and flash and fire point. The acid value was determined by titration method according to ASTM D 664. The viscosities of biodiesel were tested using Redwood viscometer at 40°C according to IS: (P: 25): 1976. The densities of biodiesel were measured at 15°C using Pykno-meters according to IS: 1448 (P: 32): 1992 and the heating values were recorded as the high heating value (HHV) by the Toshniwal Microprocessor Bomb Calorimeter according to IS: 1448(P: 6): 1984. The flash point and fire point of the test oil samples was determined as per IS: 1448(P: 32):1992 by a Pensky Martin flash point (closed) apparatus.

RESULTS AND DISCUSSION

The studies on the oxidation stability of biodiesel was carried out to assess the level of deterioration of methyl ester of palm oil and its blend with diesel at different proportion during 18 months of storage period. In this test of oxidation stability due to aerial oxidation, certain changes in physical and chemical properties such as kinematic viscosity, relative density, gross heat of combustion, acid value and flash and fire point were evaluated on certain intervals. The results of the study are shown in Figs. 1-5.

Kinematic viscosity: Kinematic viscosity is an important property of fuel as it indicates the ability of fuel to atomize into small droplets in the combustion chamber. At the molecular level, the increase in viscosity is also an indicator for the formation of larger molecules in biodiesel. Kinematic viscosity increases with the carbon chain length in biodiesel containing free fatty acids and hydrocarbons. The viscosity of methyl ester of palm oil and diesel was observed to be 5.471 and 2.64 cS, respectively and is given in Table 2. The viscosity of their blends mixed in 10:90, 20:80 and 30:70 proportions were observed to be 3.01, 3.211 and 3.516, respectively. The viscosity of methyl ester of palm oil was 2.07 times higher than that of diesel. The higher viscosity could be due to its molecular composition and structure, increased carbon chain length compared to diesel fuel as suggested by Goering et al. (1982). The Table 2 shows the changes in viscosity with storage time. However, the viscosity of diesel is lower, and the increasing trend in viscosity over time is lower as diesel is less oxygenated than biodiesel. The viscosity of diesel was observed to be 2.64, 2.68 and 2.75 cS, respectively, after six months, 12 months and 18 months of storage. But, the changes in viscosity of methyl ester of palm oil and its blend with diesel mixed in

10:90, 20:80, 30:70 and 100:00, proportion were found to be 3.112, 3.295, 3.568 and 5.521 cS, respectively after six months of storage; 3.156, 3.301, 3.591 and 5.925 cS, respectively after 12 months of storage and 3.472, 3.517, 4.075 and 6.545 cS, respectively after 18 months of storage. The increasing trend in viscosity was due to the effect of oxidation. The oxidation process leads to the formation of free fatty acids, saturation and the production of higher molecular weight molecules resulting into viscosity increase with increasing oxidation by a longer storage period (Krutika et al. 2014). The kinematic viscosity of diesel at 38°C may range between 1.9 to 6.0 cS ASTM-D95 (ASTM 2006). The viscosity of neat methyl ester of palm oil was 6.545 cS after 18 months of storage which crossed the standard range.

Mathematical models were developed by regression analysis to predict the values of kinematic viscosity (Y), taking storage time period (x_1) and blend of fuel (x_2) as independent variables by using the results of this study. The best fit model was found to be full a quadratic model in the following form:

$$Y = a + b\,x_1 + c\,x_2 + d\,x_1^2 + e\,x_2^2 + f\,x_1\,x_2 \qquad \dots (1)$$

where,

X_1 = time period, X_2 = fuel blend, Y = kinematic viscosity,
a = 2.896, b = -0.028, c = 0.017, d = 0.002, e = 0.00007, f = 0.0004
Standard error = 0.099
Coefficient of determination (r^2) = 0.994

Relative density: The density of fuel has some effect on the break up of the fuel injected into the cylinder. In addition, more fuel is injected by mass as the fuel density increases. All biodiesel fuels regardless of produced from vegetable oils or from fats are denser and less compressible than the diesel fuel (Knothe 2005). Table 3 shows the relative density of diesel, methyl ester of palm oil and different blends. The relative density and API gravity of Palm biodiesel were 0.881 and 29.11, respectively whereas diesel had relative density and API gravity were 0.842 and 36.52, respectively. As storage time periods increased the relative density of different blends of fuel increased, but the API gravity was decreased. It can be seen that the relative density of fresh methyl ester of palm oil and its blend with diesel mixed in 100:00, 10:90, 20:80 and 30:70 proportion was 0.881, 0.844, 0.852 and 0.858, respectively. Table 2 shows the changes in relative density with storage time. However, the relative density of diesel was observed to be 0.843, 0.843 and 0.846, respectively after six months, 12 months and 18 months of storage. But, the changes in relative density of methyl ester of palm oil and its blend with diesel mixed in 10:90, 20:80, 30:70 and 100:00 proportion were found to be 0.849, 0.858, 0.867 and 0.897, respectively after six months of storage; 0.851, 0.853, 0.871 and 0.897, respectively after 12 months of storage and 0.852, 0.858, 0.892 and 0.932, respectively after 18 months of storage.

The maximum increase in percentage of relative density was found after 18 months of storage. The increase in density was caused by the formation of oxidation products, including insoluble sediments. The increase in density of methyl ester of palm oil was more due to the presence of saturated fatty acid percentages in the fuel. The fuels containing shorter chain hydrocarbon and more saturated fatty acid are more prone to be crystallized. This causes reduction of its volume and consequently increase in the density. Simultaneously the mass of the fuel is increased as the consequence of oxidation products as well (Krutika et al. 2014).

Regression analysis was made and various mathematical models were developed and tested to predict the values of relative density with variation in storage time for different fuel blends. The best fit model was found to be rational model in the following form:

$$Y = \frac{(a + bx_1 + cx_2)}{(1 + dx_1 + ex_2)} \qquad \dots (2)$$

Where,

Y = relative density, X_1 = time period, X_2 = fuel blend
a = 0.84, b = -0.036, c = 0.004, d = -0.043 and e = 0.004 are the constants
Standard error = 0.005
Coefficient of determination (r^2) = 0.966

Heat of combustion: The heating value is higher for fuel with a longer chain and higher degree of saturation (Demirbas 1998). A greater heat release from a hydrocarbon fuel means that a smaller fuel mass can be used to attain the same engine power output, and is thus more favourable. The amount of heat released from burning the neat methyl ester of palm oil and diesel was found to be 38.84 MJ/kg and 47.6 MJ/kg, respectively, as shown in Table 3. The calorific values of their blends mixed in 10:90, 20:80 and 30:70 proportions were observed to be 43.64, 41.64, 40.40 and 38.84 MJ/kg, respectively. The calorific value of methyl ester of palm oil was 1.22 times lower than that of diesel. Factors that influence the energy content of biodiesel include the oxygen content and carbon to hydrogen ratio. Generally, as the oxygen content of FAAE is increased, a corresponding reduction in energy content is observed (Moser 2009). The calorific value of diesel was observed to be 47.41, 47.21, and 46.87 MJ/kg, respectively after six months, 12 months and 18 months of storage. But, the changes in calorific values of methyl ester of palm oil and its blend with diesel mixed in 10:90, 20:80, 30:70 and 100:00 proportion were found to be 41.53, 40.01, 38.23 and 37.52 MJ/kg, respectively after

Table 2: Fuel properties of blends of methyl ester of palm oil and diesel.

Sl. No.	Fuel type	Kinematic viscosity (Cs)	Relative density	API gravity	Calorific value (MJ/kg)	Acid value (mg KOH/ g of oil)	Flash point (°C)	Fire point (°C)
1	Diesel	2.64	0.842	36.55	47.6	0.23	57	63.2
Fresh (methyl ester of palm oil: diesel)								
2	10:90	3.010	0.844	36.05	43.64	0.25	57	60
3	20:80	3.211	0.852	34.42	41.64	0.35	59	63
4	30:70	3.516	0.858	33.41	40.40	0.38	60	66
5	100:00	5.471	0.881	29.11	38.84	0.67	173	178
After six months (methyl ester of palm oil: diesel)								
6	00:100	2.64	0.843		47.41	0.24	58	63
7	10:90	3.112	0.849	35.16	41.53	0.29	56	62
8	20:80	3.295	0.858	34.61	40.01	0.38	59	63
9	30:70	3.586	0.867	31.65	38.23	0.42	60	64
10	100:00	5.521	0.897	26.17	37.52	0.69	171	175
After 12 months (methyl ester of palm oil: diesel)								
11	00:100	2.68	0.843		47:21	0.30	56	62
12	10:90	3.156	0.851	35.17	37.97	0.90	55	62
13	20:80	3.301	0.853	34.81	37.71	1.15	58	64
14	30:70	3.591	0.871	31.78	37.41	1.14	58	63
15	100:00	5.925	0.897	26.95	36.43	1.93	166	170
After 18 months (methyl ester of palm oil : diesel)								
16	00:100	2.75	0.846		46.87	0.41	56	62
17	10:90	3.472	0.852	34.78	37.39	0.98	53	59
18	20:80	3.517	0.858	33.48	37.64	1.23	56	60
19	30:70	4.075	0.892	29.51	37.17	1.56	55	63
20	100:00	6.545	0.932	25.35	36.38	2.01	150	155

six months of storage; 37.97, 37.71, 37.41 and 36.43 MJ/kg, respectively after 12 months of storage and 37.39, 37.64, 37.17 and 36.38 MJ/kg, respectively after 18 months of storage. This is primarily due to water forms in biodiesel through the per-oxidation chain mechanism following the occurrence of oxidative instability, and hence the amount of water in the samples increased with the extent of oxidative instability. The amount of heat released thus also decreased with storage time in the samples of all blends because of the production of water during the per-oxidation chain reaction (Ndana et al. 2012).

The predicted values were obtained by using a mathematical equation which has been obtained by using the regression analysis where actual values of two independent variables of time periods and blends were taken to find the effect on the dependent variable of calorific values. The best fit model among the different mathematical models that had been tested was rational model. The mathematical equation developed for calorific value was in following form:

$$Y = \frac{(a + bx_1 + cx_2)}{(1 + dx_1 + ex_2)} \quad \ldots (3)$$

Where, Y = calorific value, X_1 = time period, X_2 = fuel blend a = 48.32, b = 2.29, c = 2.21, d = 0.007 and e = 0.06 are constants

Standard error = 0.661,

Coefficient of determination (r^2) = 0.927

Acid value: The acid value is a measure of the amount of acidic substances in the fuel. During storage, the hydro peroxide produced from the oxidative degradation can undergo the complex secondary reactions, including a split into more reactive aldehydes, which further oxidize into acids, leading to an increase in acid value (Bouaid et al. 2007). Another reason for the increase in acid value is the hydrolysis of methyl ester by the reaction of moisture in the ambient air with methyl ester (Leung et al. 2006). The amount of acid value of neat palm-oil biodiesel was 0.67 mgKOH/g, which is higher than the diesel of 0.23 mgKOH/g as shown in Table 3. It can be seen that the acid value of fresh methyl ester of palm oil and its blends with diesel mixed in 100:00, 10:90, 20:80 and 30:70 proportion was 0.67, 0.25, 0.35 and 0.38 mgKOH/g, respectively. The acid value of diesel was observed to be 0.24, 0.3 and 0.41 mgKOH/g, respectively after six months, 12 months and 18 months of storage. Diesel fuel showed little variation in term of acid values as oxidation affects it. But, the changes in acid values of methyl ester of palm oil and its blend with diesel mixed in

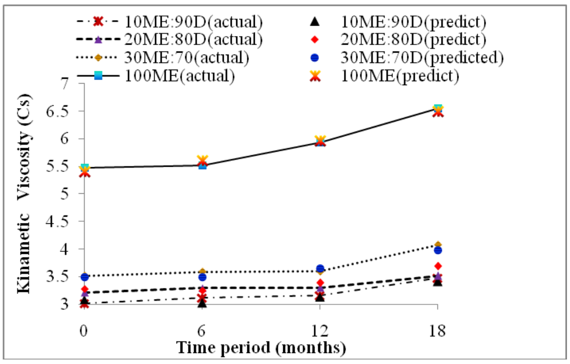

Fig. 1: Variation of actual values of kinematic viscosity from predicted ones at different blends of fuel.

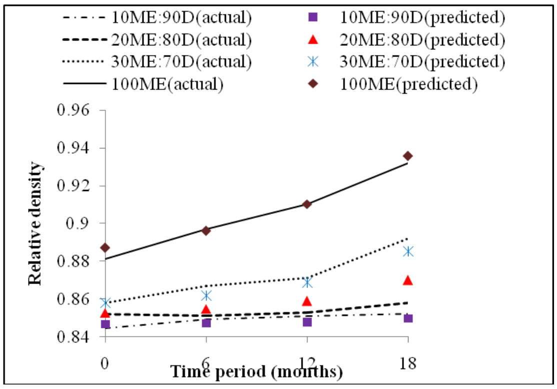

Fig. 2: Variation of actual values of relative density from predicted ones at different blends of fuel.

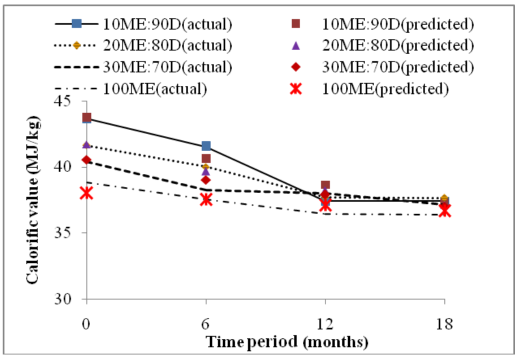

Fig. 3: Variation in actual calorific values from predicted ones at different blends of fuel.

10:90, 20:80, 30:70 and 100:00, proportion were found to be 0.29, 0.38, 0.42 and 0.69 mgKOH/g, respectively after six months of storage; 0.90, 1.15, 1.14 and 1.93 mgKOH/g, respectively after 12 months of storage and 0.98, 1.23, 1.56 and 2.01 mgKOH/g, respectively after 18 months of storage. The acid value was found to be increasing significantly with storage period for the entire sample. The acid value of neat methyl ester of palm oil and its blend are not in the range of ASTM standard for acid value of biodiesel 0.80 mgKOH/g after 12 months of storage.

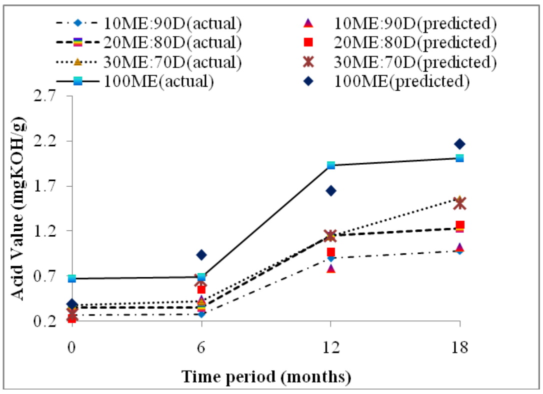

Fig. 4: Variation in actual acid values from predicted ones at different blends of fuel.

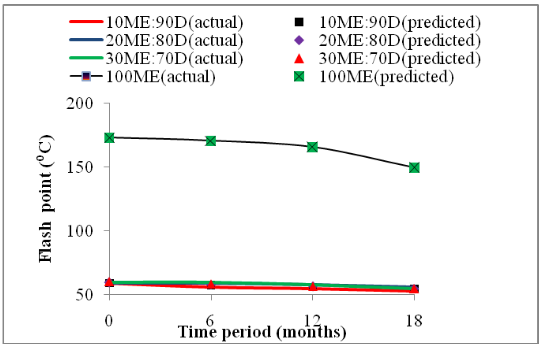

Fig. 5: Variation in actual flash point from predicted ones at different blends of fuel.

Regression analysis was made and various mathematical models were developed and tested to predict the values of acid value with variation in storage time for different fuel blends. The best fit model was found to be sigmoid model in the following form:

$$Y = \frac{a}{(1+e^{b-cx_1})(1+e^{d-ex_2})} \quad \ldots (4)$$

Where, Y = acid value, x_1 = time period, x_2 = fuel blend
a = 2.57, b = 1.68, c = 0.19, d = 0.58 and e = 0.04 are constants
Standard error = 0.201
Coefficient of determination (r^2) = 0.909

Flash point: Flash point measure the tendency of the sample to form a flammability mixture with air. It is expected that a good fuel should have a low auto-ignition temperature, especially in a diesel engine, since it has no extra mechanism to ignite the fuel in the combustion chamber.

The auto-ignition temperature of a fuel is the lowest temperature at which the fuel could spontaneously ignite without an external source of ignition. Fuels with a flash point above 62°C can be considered to be safer fuels; therefore, biodiesel is a safer fuel for handling and storage. It can be seen that the flash point of methyl ester of palm oil and its blend with diesel mixed in 100:00, 10:90, 20:80 and 30:70 proportion was 173, 57, 59 and 60°C, respectively as shown in Table 3. As the storage time period increased, the flash point of all blends decreased gradually. The flash point of all blends mixed in 10:90, 20:80, 30:70 and 100:00 proportion was 56, 59, 60, and 171°C, respectively after 6 months of storage; 55, 58, 58 and 166°C, respectively after 12 months of storage; 53, 56, 55 and 150°C, respectively after 18 months of storage. The maximum decrease in flash point was found after 18 months of storage. This occurred because, over time, the oxidative degradation converts unsaturated fatty acids to primary and secondary oxidation products such as free fatty acids, hydro peroxides, polymers with a high molecular weight, and water. Bio-lipids that contain more free fatty acids or higher water content, have lower flash points (Ndana et al. 2012).

The predicted values are obtained by using a mathematical equation which has been obtained by using the regression analysis, where observed values of two independent variables of time periods and blends were taken to find the effect on the dependent variable of flash point. The best fit model among the different mathematical models that have been tested was rational model. The mathematical equation developed for flash point was in following form:

$$Y = \frac{(a + bx_1 + cx_2)}{(1 + dx_1 + ex_2)} \quad \dots (10)$$

Where, Y = acid value, x_1 = time period, x_2 = fuel blend
a = 58.54, b = -0.31, c = -0.51, d = -0.001 and e = -0.009 are constants
Standard error = 1.171
Coefficient of determination (r^2) = 0.999

CONCLUSION

It may be concluded that the blends containing 10 to 30% of methyl ester of palm oil had their fuel properties compatible with diesel. But as the storage time period increased its properties started deteriorating. It can also be concluded that methyl ester of palm oil and its blend had the highest potential to prevent oxidation by retaining the properties of the fuel during storage period. The adverse effects of oxidation were observed after 12 months of storage in all methyl ester blends in terms of viscosity, acid value and calorific value. Its value exceeded the limiting value of the standard specification.

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