Oxidation Stability, Engine Performance and Emissions Investigations of Karanja, Neem and Jatropha Biodiesel and Blends

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Deepak Khurana
Indian Institute of Technology Roorkee

Avinash Kumar Agarwal
Indian Institute of Technology Kanpur

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Deepak Khurana and Avinash Kumar Agarwal
Indian Institute of Technology Kanpur

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ABSTRACT

Poor oxidation stability is the central problem associated with the commercial acceptance of the biodiesel. The EU standard (EN14214) specifies a minimum value of 6 h for biodiesel induction period at 110°C, measured with Rancimat instrument. Most of the freshly prepared biodiesel generally have lower induction periods than prescribed by the standards. Anti-oxidants are therefore added to enhance the oxidation/ storage stability of biodiesel. Oxidation is an exothermic process, and the reaction heat evolved makes it possible to use thermo gravimetric analysis (TGA).

In the present study, the oxidation stability of methyl esters derived from Karanja oil and Neem oil, stabilized with anti-oxidant pyrogalol (PY) was studied by DSC. Onset temperature of freshly prepared Karanja biodiesel (KOME) and Neem biodiesel (NOME) was observed to be 148 and 153°C respectively. The stability increases with increasing anti-oxidant dosage. A comparative analysis of oxidation stability of biodiesel/diesel blends was done on biodiesel derived from Karanja (*Pongamia pinnata*), Neem (*Azadirachta indica*) and Jatropha (*Jatropha curcas*) oil using Rancimat instrument. Jatropha biodiesel (JOME)/ diesel blends were observed to be most stable compared to KOME and NOME/ diesel blends. Further in this study, emissions and performance tests were conducted on an IDI diesel engine for diesel, B100, and biodiesel dosed with 1000 ppm of PY to experimentally evaluate adverse effects of anti-oxidants on biodiesel performance and emissions. With anti-oxidants dosed biodiesel, THC and CO were found to be slightly higher compared to diesel and B100. However the difference was not significant. Thermal efficiency and BSFC of anti-oxidants dosed biodiesel was comparable to diesel and B100.

INTRODUCTION

Vegetable oils and their esters (Biodiesel) are the promising alternative fuels for diesel engine. The biodiesel feedstock varies from country to country depending on the availability of vegetable oil in the vicinity and the Government policies. Traditional raw materials for biodiesel production in the west are Soybean oil, Rapeseed oil, Sunflower oil, Palm oil, Rice-bran oil etc. Since India is a net importer of edible oils, these cannot be used for production of biodiesel. Still, India has the potential to be a leading global producer of biodiesel, as biodiesel can be produced from non-edible oils like Jatropha, Karanja, Neem, Mahua, Castor, Linseed, Kusum etc. Many of these oils are not being utilized fully. Out of these feed-stocks, India is focusing on Jatropha and Karanja, which can grow in arid zones and wastelands of the country. India has about 80-100 million hectares of wasteland, which can be used for the cultivation of Jatropha and Karanja. India is also one of the largest producers of Neem oil, which is an untapped resource in India. Oxidation of these esters in presence of air, high temperature and metal exposure is a major hindrance for its commercialization. The ill effects of oxidation of biodiesel are increased viscosity, peroxide value, acid number, and gum deposits. An increase in these properties of biodiesel cause problems in the engine like damage to fuel delivery system, filter plugging, injector coking, corrosion, hardening of rubber components, fusion of moving components and engine deposits etc. Therefore the main objective of this study is to investigate the oxidation stability, engine performance and emissions of biodiesel derived from non-edible oils of sub-continental origin such as Karanja, Neem and Jatropha.

The European Committee of Standardization (CEN) in its standard EN14214 has specified a minimum of 6 h induction period at 110°C for biodiesel, measured with Rancimat instrument [1, 2]. The heat content of vegetable oils and their esters is nearly 90% that of diesel fuel [3, 4, 5]. The stability of biodiesel is classified as oxidation stability, thermal stability and storage stability. However all these are broadly termed as oxidation stability. Thermal instability is related to the increased rate of oxidation at higher temperature which in turn increases the weight of the molecules due to the formation of insolubles. In an engine, extra fuel from the injector returns back to the fuel tank. Being in contact with hot fuel injector installed in the cylinder head, the fuel gets exposed to heat for a considerable duration. This could be a problem in case of biofuels, such as biodiesel, which start changing their properties, once they are exposed to heat. Biodiesel should therefore be able to retain its thermal stability at higher temperatures. There is no thermal stability limit specified for biodiesel in any standard prescribed internationally. However from a practical stand point, a biodiesel sample, which maintains its thermal stability up to 150°C can be regarded as thermally stable. Differential Scanning Calorimetry (DSC) is an accelerated method, which finds the temperature at which the oxidation reaction takes place. This temperature is termed as oxidation onset temperature [6].

The oxidation stability of biodiesel can be improved with addition of anti-oxidant additives. All the anti-oxidants have an aromatic ring with at least one hydroxyl (−OH) group. The hydroxyl (−OH) group of the anti-oxidant is very active so that hydrogen is abstracted from −OH and donated to the oxidized free radical to inhibit the rate of oxidation (Figure 1). The resulting anti-oxidant is a stable radical that can react with other fatty acid free radicals and further contribute to oxidation inhibition [7-8].

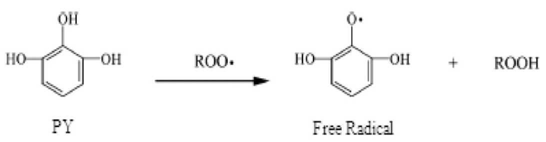

Figure 1. Mechanism of anti-oxidant activity.

LITERATURE REVIEW

Operating diesel engine produces hazardous emissions which may affect the environment and adversely affect the human health [9, 10, 11] e.g. Oxides of Nitrogen (NO_X) irritate the eyes and throat, and create breathing problems; Carbon monoxide (CO) reduces the oxygen carrying capacity of the blood; Sulfur dioxide (SO_2) reduces the lung resistance causing chronic bronchitis and also acid rain; Poly-cyclic aromatic hydrocarbons (PAH) can cause cancer; Carbon dioxide (CO_2) leads to global warming; and Particulate Matter (PM) results in respiratory diseases like asthma and emissions of hydrocarbon (HC) results in loss of fuel. Biodiesel is however a clean burning fuel with little or no SO_2, lower THC, CO, PM and CO_2 emissions with possibly slightly higher NO_X. But for practical feasibility of biodiesel, anti-oxidants are added to increase the storage stability of biodiesel. This study is aimed at experimental assessment whether these additives affect the emission characteristics of biodiesel significantly.

Sharma et al. [12] used DSC method for determining the oxidation stability of lubricating oil with reasonable accuracy and repeatability. Perez et al. [13] used DSC method to understand the oxidation stability and cold flow properties of various blends with mineral diesel. Robert O Dunn [14] investigated the effectiveness of five anti-oxidants on the oxidation stability of Soybean oil methyl esters (SME). Anti-oxidant examined were 2-tert butyl hydroquinone (TBHQ), 6-di-tert butyl-4-methyl phenol (BHT), 2-tert butyl-4-methoxy phenol (BHA), 3,4,5-tri hydroxy benzoic acid (PG) and α-Tocopherol (α-T). The anti-oxidants activity was determined in terms of increasing oxidation onset temperature by using a non-isothermal pressure differential scanning calorimeter (P-DSC). Increasing anti-oxidant loading showed a sharp increase in stability up to 1000 ppm followed by smaller increase in stability at higher concentrations. As such, there is no specification limit for onset temperature for thermal oxidation stability measured by DSC. Though a stability of 150°C in dynamic mode and 160°C in static mode showed a quite stable biodiesel. PG and BHA showed higher thermal stability in static mode at all concentrations followed by TBHQ, BHT and α-T respectively. Whereas in dynamic mode, PG and BHT showed higher oxidation onset temperature followed by BHA, TBHQ and α-T respectively. An anti-oxidant loading of 2000 ppm showed an onset temperature of nearly 150°C for anti-oxidants except α-T in dynamic mode and nearly 160°C in static mode. The onset temperature at 2000 ppm of α-T was 146°C in static and 128°C in dynamic mode. Effectiveness of anti-oxidants in static mode was in the order BHA∼PG>TBHQ>BHT>α-T and in dynamic mode the order was BHT∼PG> BHA>TBHQ>α-T.

Polvaka et al. [15] studied the effect of 1,2,3 tri-hydroxy benzene (PY) and BHT on the oxidation stability of biodiesel derived from Rapeseed oil and Waste frying oil using Rancimat method as well as differential scanning calorimeter (DSC). Both distilled and undistilled samples of Rapeseed oil and waste frying oil were analyzed. The effectiveness of BHT and PY was analyzed in the concentration range of 0-1000 ppm. It was observed that the oxidation onset temperature increases with an increase in concentration of the anti-oxidant. The onset temperature for untreated distilled waste frying oil was 185°C. The onset temperature with PY was higher than BHT at all concentrations. At 500 ppm

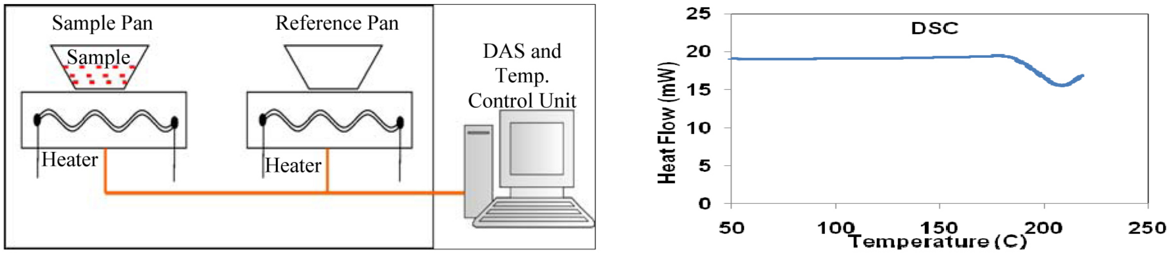

Figure 2. Principle of Differential Scanning Calorimetry (DSC).

concentration the onset temperature was 189°C and 197°C for BHT and PY respectively, while at 1000 ppm, values were 191°C and 198°C for BHT and PY respectively. Analysis was also done on the effects of ramp rate on the onset temperature of Rapeseed oil biodiesel. With the increasing ramp rate from 1°C/min to 15°C/min, the onset temperature increases. Again PY and BHT showed higher onset temperature than untreated Rapeseed oil. In the same study, the effectiveness of anti-oxidants on the induction period was also evaluated using Rancimat method. A constant concentration of 400 ppm of PY and 500 ppm of BHT was analyzed with Rapeseed oil methyl esters and Waste frying oil methyl esters- both distilled and undistilled. The induction periods of distilled samples were lower than corresponding undistilled samples. None of the untreated distilled samples had the stability limit of 6 h while neat distilled samples were having stability more than 6 h. Maximum stability (25.3 h) was observed for undistilled Rapeseed oil methyl ester with 400 ppm of PY anti-oxidant. Samples treated with BHT also obtained the EN 14214 limit except with distilled Waste frying oil methyl ester samples (5.35 h).

Experiments have been done to find the exhaust emissions of biodiesel dosed with anti-oxidants [16]. It was observed in these studies that there was no noticeable increase in PM, THC, CO and CO_2 emissions compared to pure biodiesel. Surprisingly NO_x emissions were reported to have reduced by using anti-oxidant dosed biodiesel [16]. It was inferred that the anti-oxidants which are responsible for stabilizing the biodiesel for longer time, when applied to practical conditions, have no noticeable negative effect of exhaust emissions.

MATERIALS AND METHODS

For the present study, Karanja, Neem and Jatropha oils were procured. Karanja oil methyl ester (KOME), Neem oil methyl ester (NOME) and Jatropha oil methyl ester (JOME) were produced using transesterification process. High grade methanol (6:1 molar ratio) mixed with alkali (NaOH) catalyst (1% w/w_{oil}) was added to the reactor containing pre-heated vegetable oil. The transesterification reaction was carried out at 55-60°C at a stirring speed of 700-800 rpm for 1 hour. After completion of the reaction, reaction products were transferred to a separating funnel, where upper phase (biodiesel) was separated from lower phase (glycerol) followed by subsequent washing (by distilled water) and drying of the final product i.e. biodiesel.

NOME was produced by a two-step process due to very high free fatty acid (FFA) content of the Neem oil. With feedstock containing high FFA, conventional biodiesel production by base catalyzed transesterification process is not suitable. A two-step process is used. It includes pre-treatment of the high FFA vegetable oil with sulfuric acid i.e. esterification process proceeded by conventional transesterification process. In the first step, esterification is done to reduce the FFA content of the Neem oil by converting FFA into esters. The second step is transesterification process as described earlier, in which triglyceride portion of the oil reacts with methanol in presence of NaOH to form esters and glycerol [17-18].

Thermal stability of biodiesel can be measured by DSC instrument. DSC is a thermo-analytical technique, in which, the difference in the amount of heat required to increase the temperature of a sample and reference are measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment (Figure 2). Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time.

The basic principle underlying this technique is that, when a sample undergoes a physical transition such as phase transformation or oxidation, relatively higher or lower heat flow will be required compared to the reference in order to maintain both at the same temperature. Whether higher or lower heat must flow to the sample depends on whether the process is exothermic or endothermic. Since the oxidation reaction for biodiesel is exothermic, therefore DSC can be used to study the oxidation stability of biodiesel. A differential scanning calorimetry instrument (Model: DSC-6; Make: Perkin Elmer) was used for measuring the oxidation stability of biodiesel samples. Fuel samples of 10±0.5 mg were weighed into open aluminum pans and placed in the sample chamber of the equipment. Temperature was programmed from 30 to 300°C at the ramp rate of 10°C/min. Purified oxygen (99.8%) was passed through the sample enclosure as a purge gas at a flow rate of 60 ml/ min.

For this experimental study, the anti-oxidant used was 1,2,3 tri-hydroxy benzene (PY-purity grade >99%), supplied by Merck Specialties Pvt Ltd, India. A biodiesel Rancimat (Model: 873; Make: Metrohm, Switzerland) was used for measuring the oxidation stability of biodiesel/ diesel blends (Figure 3).

Figure 3. Biodiesel Rancimat Instrument.

During the measurement of oxidation stability of biodiesel by Rancimat, a stream of air is passed through the fatty acid methyl ester sample contained in a sealed and heated reaction vessel at constant temperature (e.g. 110°C at the air flow rate of 10 1/h as per EN 14214). This treatment results in oxidation of the fatty acid methyl ester molecules in the sample, with peroxides initially being formed as the primary oxidation products. After some time, the fatty acid methyl esters disintegrate completely; the secondary oxidation products formed include low-molecular organic acids in addition to other volatile organic compounds. These are transported in the stream of air to a second vessel containing distilled water. The conductivity in this vessel is recorded continuously. The organic acids can be detected by the increase in conductivity. The time that elapses until these secondary reaction products appear is known as induction time or induction period.

The performance and emission characteristics of the fuel (treated with and without anti-oxidants) were performed on an Indica IDI four cylinder engine coupled with an eddy current dynamometer. This is water cooled, indirect injection, four strokes, and four cylinder diesel engine. The eddy current dynamometer is equipped with a dyno controller capable of loading the engine at desired speed/load combination (Figure 4). Tests were performed at constant speed and varying engine load. The exhaust gas emissions (CO, THC, NO_x and CO_2 and O_2) were measured by a Horiba Raw Exhaust Gas Emission Analyzer (Model: EXSA-1500, Make: Horiba, Japan).

Figure 4. Experimental Setup of IDI Engine for Performance and Emissions

Detailed specifications of the test engine are given in Table 1:

Table 1. Specifications of the Test Engine

Model	Tata Indica 475
Type	Water cooled, IDI, 4-Stroke
Number of Cylinders	4
Compression Ratio	22:1
Stroke /Bore	79.5 mm/75 mm
Max. Power Output	53.5 hp @ 5000 rpm
Max. Torque	85 Nm @ 2500 rpm
Loading Device	Eddy Current Dynamometer

The performance and emission test were conducted to find the suitability of anti-oxidant dosed biodiesel fuel for diesel engine. Performance test includes measurement of thermal efficiency, brake specific fuel consumption and exhaust gas temperature at different engine operating conditions. The following parameters were used for performance and emission characteristics from the engine experimental setup:

Performance Parameters: Thermal Efficiency, Brake Specific Fuel Consumption (bsfc), and Exhaust Gas Temperature (EGT);

Emission Parameters: mass emissions of Oxides of Nitrogen (NO_x), Total Hydrocarbon (THC), and Carbon Monoxide (CO).

RESULTS AND DISCUSSION

The important fuel properties of NOME, KOME and JOME such as density, viscosity, flash point, calorific value and oxidation stability were evaluated and are given in Table 2. KOME and JOME satisfy the viscosity range of 1.9-6.0 cSt at 40°C as specified by ASTM-D 6751 and, while the viscosity of NOME was slightly above the viscosity limits prescribed.

Table 2. Important physical properties of biodiesel samples.

Property	Unit	KOME	NOME	JOME
Density at 15°C	kg/m^3	871	887	882
Viscosity at 40°C	cSt	4.89	6.17	5.27
Flash Point	°C	142	127	161
Calorific Value	MJ/kg	39.56	39.87	39.44
Oxidation Stability	h	1.82	1.39	3.05

The experiments were performed on Perkin Elmer DSC to find the oxidation onset temperature of NOME and KOME, without anti-oxidant as well as with anti-oxidant. The anti-oxidant used was PY in two concentrations (500 and 1000 ppm respectively) for both biodiesel samples.

Thermal Stability Measured with DSC

Figure 5 represents the oxidation onset temperature for biodiesel samples with and without anti-oxidants measured with DSC. The onset temperature of NOME (NB100) and KOME (KB100) (without anti-oxidants) were observed to be 148°C and 153°C respectively.

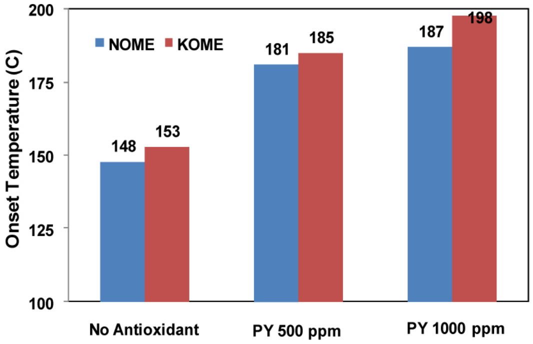

Figure 5. Oxidation onset temperature of KOME and NOME measured with DSC.

It was observed that the oxidation onset temperature increases with increasing dosage of anti-oxidant. At 500 ppm of PY, the onset temperature of NOME increased from 148°C to 181°C and for KOME it increased from 153°C to 185°C. Further increase in oxidation onset temperature was observed at 1000 ppm concentration of PY. The oxidation onset temperature values at PY 1000 ppm was 187°C for NOME and 198°C for KOME respectively (Figure 5).

Oxidation Stability of Biodiesel/ Diesel Blends

The Rancimat instrument (Figure 3) was used to find the oxidation stability of the three biodiesels (B100) and their various blends with diesel. Diesel has no unsaturated compounds in their structure therefore its oxidation stability is very high. It may therefore be expected that the stability of biodiesel will increase when blended with diesel. Higher the percentage of diesel in the blend, higher will be the stability of biodiesel blend. The blending of biodiesel may therefore eliminate the requirement of anti-oxidant for its long-term storage. The oxidation stability of various biodiesel (KOME, NOME and JOME) blends such as BIO, B20, B50 and B100 was measured using Rancimat tests. The results are shown in figure 6.

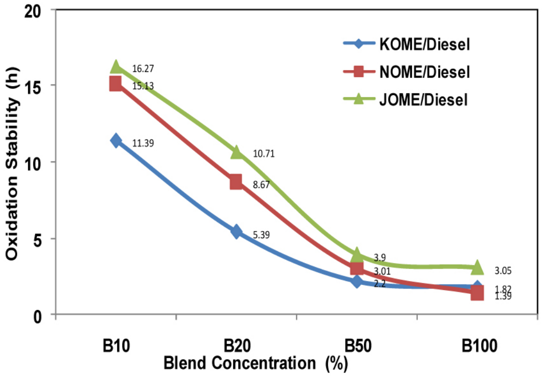

Figure 6. Oxidation Stability of Biodiesel Blends.

It was observed that BIO had the highest oxidation stability and B100 the least, for all biodiesel samples. The increase in stability of JOME was observed to be from 3.05 to 10.71 and 16.27 h at a blend ratio of B100 to B20 and B10 respectively. The blends of JOME were most stable followed by NOME and KOME blends. The increase in stability of NOME was from 1.39 to 8.67 and 15.13 h at B100 to B20 and B10 respectively. At B20, NOME and JOME satisfied the oxidation stability criterion while for KOME, only B10 satisfied the oxidation stability criterion. The induction period of KOME (B10) was observed to be 11.39 h.

Engine Performance and Emissions

Engine performance and emission tests were conducted on diesel, 100% KOME, and PY1000 ppm dosed KOME on an IDI engine at 2000 rpm (engine rated speed) at the varying load conditions of 0, 20, 40, 60, 80 and 100% rated engine loads. Graphs were plotted for performance and emission parameters against brake mean effective pressure (BMEP). Various engine performance parameters such as brake specific fuel consumption (BSFC), brake thermal efficiency, and exhaust gas temperature (EGT) are measured, calculated and reported in figure 7:

Figure 7 shows the trend for BSFC versus BMEP for different fuels. For all fuels the BSFC decreases with increase in engine load. Biodiesel showed higher BSFC compared to diesel owing to lower calorific value of biodiesel compared to diesel. At lower engine loads, the BSFC of anti-oxidants dosed biodiesel is lower while at higher loads, it is higher than untreated biodiesel. The brake thermal efficiency for biodiesel is lower compared to diesel. The maximum brake

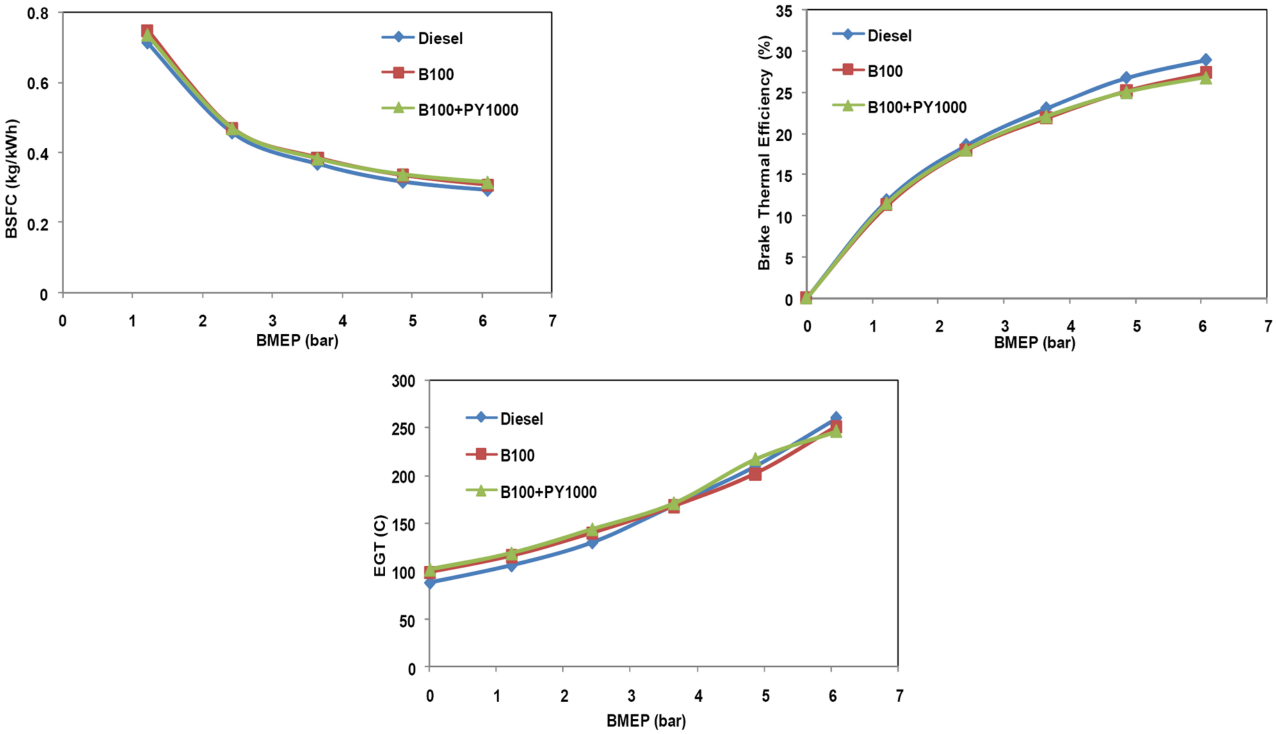

Figure 7. Engine Performance characteristics for diesel, biodiesel, and biodiesel with anti-oxidant.

thermal efficiency obtained was 28.9% for diesel followed by 27.36% for biodiesel and 26.79% for PY1000 dosed biodiesel. Figure 7 also shows the variation of exhaust gas temperature versus BMEP for test fuels. The exhaust gas temperature increases with increasing engine load. At lower engine loads, the exhaust gas temperature of treated and untreated biodiesel is higher compared to diesel while at higher loads, the exhaust gas temperature of diesel is higher compared to treated and untreated biodiesel.

The formation of NO_X with increasing BMEP is shown in Figure 8. The most significant factor that causes NO_X is high combustion temperature zones within the combustion chamber. As the load increases, the in-cylinder temperature also increases and thus higher NO_X formation (raw emission) takes place. The NO_X formation per unit power (mass emission) however decreases with increasing engine load, showing that for unit power output, lesser NO_X is emitted at higher engine loads compared to lower engine loads. This is because at higher engine loads, turbulence and atomization increases leading to faster combustion and lower residence time for species in the high temperature zone. Biodiesel showed relatively higher NO_X (slightly) compared to diesel especially at higher engine loads.

The possible reason may be relatively slower burning characteristics of more viscous biodiesel, due to which the residence time of the fuel in high temperature zone is higher. NO_X emissions are observed to be highest with PY1000 ppm dosed biodiesel.

The variation of THC emissions with BMEP is shown in Figure 8. It can be seen that THC decreases when the engine is fueled with biodiesel. The cetane number plays an important role in ignition process. Since the cetane number of biodiesel is higher than diesel, it exhibits a shorter ignition delay period and results in improved combustion. High cetane number and presence of oxygen in the molecule of biodiesel results in relatively lower THC and CO emissions. The least THC emissions are obtained with PY1000 ppm dosed biodiesel. At low loads, the mass emission of THC is higher because of lower turbulence and inadequate mixing of fuel and hence improper utilization of oxygen. At higher engine loads, the THC mass emissions are observed to be higher. At higher engine loads, more fuel is injected in the cylinder, which leads to richer combustion and lower availability of oxygen to oxidize hydrocarbons.

Figure 8 also shows the comparison of CO emissions for different fuels at different BMEP. Biodiesel emits lower CO in comparison to diesel at all loads. In biodiesel, some of the CO produced during combustion of biodiesel might have converted to CO_2 by reacting with extra fuel oxygen present in the molecules of biodiesel leading to reduced CO emissions. The CO emissions from PY1000 dosed biodiesel is also lower than diesel. The CO mass emissions are higher at low engine loads for the same reason as that of THC

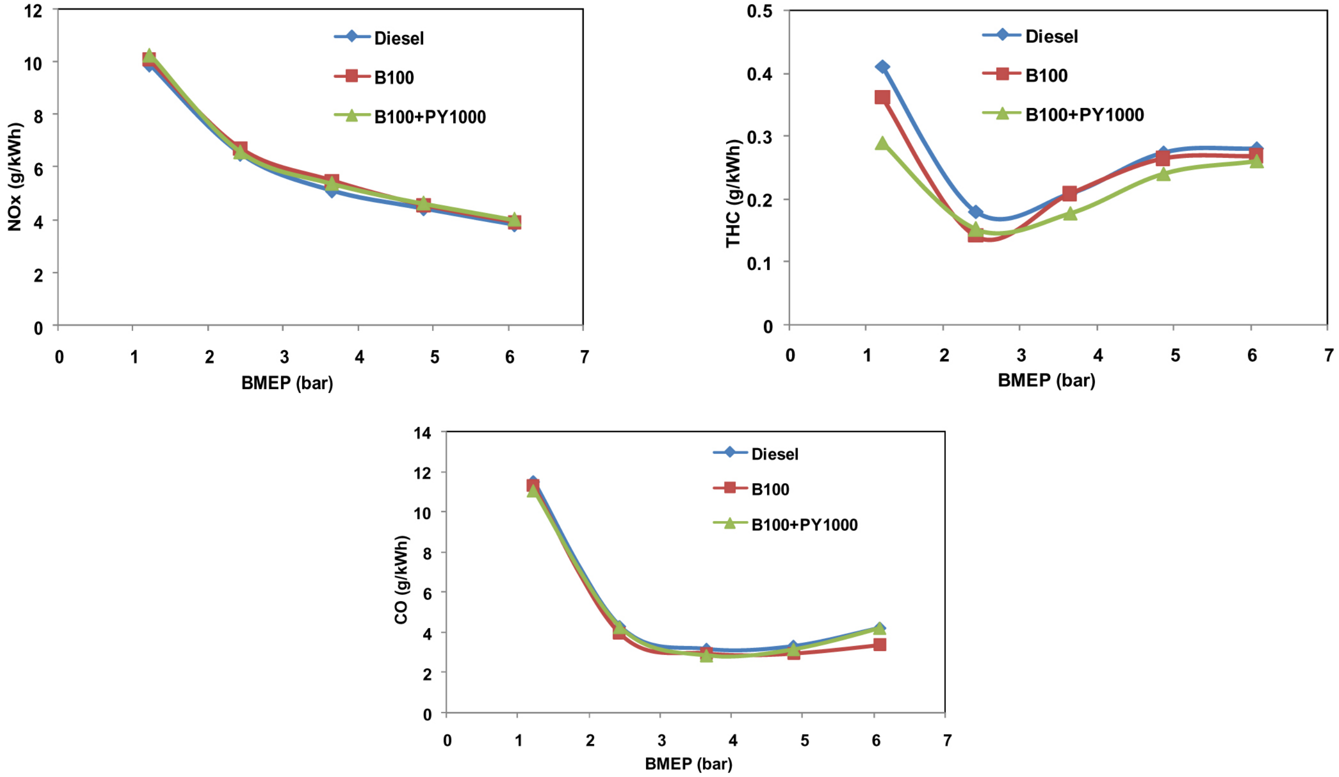

Figure 8. Engine emission characteristics for diesel, biodiesel, and biodiesel with anti-oxidant.

emissions i.e. lower turbulence and inadequate mixing of fuel and air. As the engine load increases, the temperature in the cylinder becomes higher, which improves atomization, resulting in improved combustion and lower CO mass emissions. At full load, CO increases slightly due to relatively richer combustion.

SUMMARY/ CONCLUSIONS

Anti-oxidants are invariably added to enhance the oxidation stability of biodiesel. Rancimat and DSC are the accelerated methods for measuring the oxidation stability. NOME and KOME samples were observed to be quite thermally stable in a DSC test. The oxidation onset temperature of freshly prepared NOME and KOME was observed to be 148 and 153°C respectively. The DSC was observed to be quick and reliable method for finding the oxidation stability of biodiesel. The effect of anti-oxidants in enhancing thermal stability was clearly observed in DSC test. With addition of PY500, the NOME and KOME had the thermal stability of 181 and 185°C respectively. The corresponding values at PY1000 were observed to be 187 and 198°C respectively.

Blending of biodiesel with diesel eliminated the need of anti-oxidants to satisfy the EN 14214 limit of 6 h stability measured with Rancimat. It was observed that B10 had the highest stability and B100 the least among the blends investigated. The increase in stability of JOME was observed to be from 3.05 to 10.71 and 16.27 h at a blend ratio of B100 to B20 and B10 respectively. The blends of JOME were most stable followed by NOME and KOME blends. At B20, NOME and JOME were found to have stabilized while KOME was found to have stabilized at B10. The induction period of KOME (B10) was observed to be 11.39 h.

Performance and emissions tests were conducted on an IDI diesel engine for diesel, B100, and biodiesel dosed with PY1000 to experimentally evaluate any adverse effect of anti-oxidants on biodiesel performance and emissions. It was observed that CO and THC were lower for untreated biodiesel compared to diesel. Untreated biodiesel showed higher NO_X emissions compared to diesel especially at higher loads. NO_X emissions were highest with PY1000 ppm dosed biodiesel. Biodiesel showed higher BSFC compared to diesel because of relatively lower calorific value of biodiesel compared to diesel. At lower engine loads, the BSFC of anti-oxidants dosed biodiesel was slightly lower while at higher loads, it was slightly higher than untreated biodiesel. The brake thermal efficiency for biodiesel was lower compared to diesel. The maximum brake thermal efficiency obtained was 28.9% for diesel followed by 27.36% for biodiesel, and 26.79% for PY1000 dosed biodiesel. The exhaust gas temperature increased with increasing load. At lower loads, the exhaust gas temperature of treated and untreated biodiesel was relatively higher compared to diesel while at higher loads; the exhaust gas temperature of diesel was higher compared to treated and untreated biodiesel.

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CONTACT INFORMATION

Deepak Khurana was a Graduate student working with Dr. Agarwal on his Master's dissertation in mechanical engineering from IIT Kanpur. He completed his M.Tech. from IIT Kanpur in Mechanical Engineering in July 2010 and joined Lovely Professional University (Punjab) as faculty. His areas of interest include internal combustion engines, biodiesel, and emission control.

Dr. Avinash Kumar Agarwal is currently working as Associate Professor of Mechanical Engineering at Indian Institute of Technology, Kanpur. He is a 1994 graduate of Mechanical Engineering from MREC Jaipur. His area of doctoral research at IIT Delhi was developing biodiesel and related engine tribology investigations. Dr Agarwal joined Engine Research Center, University of Wisconsin, Madison, USA for pursuing his Post-Doctoral research from August 1999 - February 2001. His main areas of current interest are combustion phenomenon study in IC engines, automobile emissions, biodiesel development and characterization, laser diagnostic techniques, PIV, lubricating oil consumption phenomenon, lubricating oil tribology, development of micro sensors, alternative fuels for diesel engines etc.

The Engineering Meetings Board has approved this paper for publication. It has successfully completed SAE's peer review process under the supervision of the session organizer. This process requires a minimum of three (3) reviews by industry experts.

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