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Oxidation Stability of Biodiesel Produced from Non-Edible Oils of African Origin

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ABSTRACT

Mono alkyl esters of long-chain fatty acids derived from renewable lipid feedstock, such as vegetable oils or animal fats, also known as biodiesel are well positioned to replace mineral diesel. The outstanding technical problem with biodiesel is that it is more susceptible to oxidation owing to its exposure to oxygen present in the air and high temperature. This happens mainly due to the presence of varying numbers of double bonds in the free fatty acid molecules. The chemical reactivity of esters can therefore be divided into oxidative and thermal instability, which can be determined by the amount and configuration of the olefinic unsaturation in the fatty acid chains. Many of the plant-derived fatty oils contain polyunsaturated fatty acids that are more prone to oxidation.

Increasing production of biodiesel from vegetable oils (edible) places strain on food production, availability and price and leads to food vs. fuel conflict. Hence, this study evaluates biodiesel derived from low cost non-edible oils, primarily available in African continent, namely croton *megalocarpus*, moringa *oleifera* and jatropha oils. The fuel related properties and oxidation stability of croton oil methyl ester (COME), moringa oil methyl ester (MOME) and jatropha oil methyl ester (JOME) were determined and compared with global biodiesel standards such as ASTM D6751 and EN 14214. Oxidation stability of COME, MOME and JOME with and without antioxidants was analyzed using Rancimat method. The results showed that most of the

properties of COME, MOME and JOME fulfilled the minimum requirements specified in the ASTM D6751 and EN 14214 biodiesel standards. However, COME and MOME did not fulfill the oxidation stability requirements specified in EN 14214 (6 h), while JOME displayed a remarkably high oxidation stability of 10.43 h. Also, this study examined the effectiveness of three antioxidants namely 1, 2, 3 trihydroxy benzene (PY), 3, 4, 5-tri hydroxy benzoic acid (PG) and 2-tert butyl-4-methoxy phenol (BHA) on COME, MOME and JOME. The result showed that, the effectiveness of these antioxidants was in the order of PY>PG>BHA.

INTRODUCTION

As the petroleum prices continue to rise, diesel supply is becoming scarce and unreliable. There is a growing concern for cleaner environment; hence scientists have invested considerable efforts in searching for renewable and sustainable substitutes of diesel fuel. Biodiesel is a non-toxic, biodegradable and renewable fuel that can be used in diesel engines, and is becoming one of the fastest growing fuels in the global fuel market [1-2]. Biodiesel is produced from transesterification reaction of vegetable oils or animal fats with primary alcohols in presence of a catalyst. It mainly consists of fatty acid esters, which have fuel characteristics similar to mineral diesel. Despite many advantages, biodiesel's chemical nature makes it more susceptible to oxidation compared to mineral diesel during long-term storage. The sensitivity to oxidation varies depending on the raw material, presence of naturally occurring antioxidants and

storage conditions such as exposure to atmospheric oxygen, daylight, contaminants (metals that speed up oxidation) and high temperature [3].

The biodiesel stability generally depends on the fatty acid profile of the feedstock oil. Therefore biodiesel with high unsaturated fatty acids such as oleic (C18:1), linoleic (C18:2) and linolenic (C18:3) acids are especially prone to oxidation. The relative oxidation rates of these unsaturated esters are linolenic > linoleic > Oleic [4]. The stability of biodiesel can be further classified as oxidation stability, thermal stability and storage stability however, all these are broadly termed as oxidation stability. Figure 1 shows the typical oxidation reactions of biodiesel. Oxidation of biodiesel starts with the removal of hydrogen from a carbon atom to produce a carbon free radical.

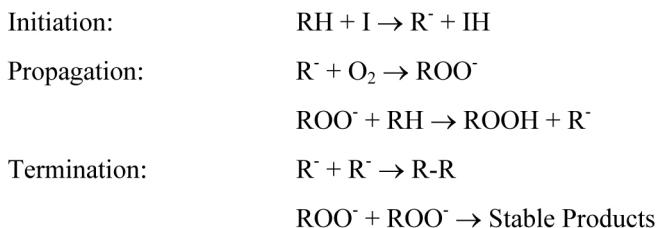


Figure 1. Typical Oxidation Reaction of Biodiesel [5]

If diatomic oxygen is present, the subsequent reaction to form a peroxy radical is extremely fast. The peroxy free radical is not as reactive as the carbon free radical, but is sufficiently reactive to quickly abstract hydrogen from a carbon to form another carbon radical and a hydroperoxide (ROOH). The new carbon free radical can then react with diatomic oxygen to continue the propagation cycle. This chain reaction terminates when two free radicals react with each other to yield stable products like aldehydes, shorter chain carboxylic acids, insolubles, gum and sediments. When biodiesel containing these oxidation products is used in the engine, it impairs the engine performance due to fuel filter plugging, injector fouling and deposit formation in engine combustion chamber and various components of the fuel system [5].

Several studies related to the stability of biodiesel have been reported in the literature. A number of researchers investigated the oxidation stability of biodiesel synthesized from edible oils [6, 7, 8, 9, 10, 11]. However increase in production of biodiesel from edible vegetable oils lead to strain on food production, its availability and prices, and finally leading to food vs. fuel conflict. Hence, this study evaluates biodiesel derived from less common non-food feedstock, primarily available in African continent, namely croton *megalocarpus*, moringa *oleifera* and jatropha oils. There are few studies reported on oxidation stability of biodiesel from less common feedstocks such as tree-borne non-edible oil seeds. Sarin et al. [12] reported oxidation oil stability of biodiesel derived from tree-borne non-edible oil

seeds such as Jatropha and Karanja and compared it with biodiesel derived from edible oils such as Palm and Sunflower oils. It was found that the trend of induction period has direct correlation with the percentage of saturated fatty acids in biodiesel. For example, palm oil biodiesel exhibited higher oxidation stability (13.37 h) than that of jatropha, karanja and sunflower biodiesels which give induction periods of 3.23, 2.35 and 1.73 h respectively. The higher oxidation stability recorded by palm oil biodiesel was due to higher saturated fatty acids (43.45%) compared to 21.1, 16.0 and 11.6% respectively of jatropha, karanja and sunflower oils. Also, various anti-oxidants were investigated for improving oxidation stability of these biodiesels especially Jatropha biodiesel. Among antioxidants evaluated, 2, 6-di-tertiary-butyl hydroxyl-toluene (antioxidant) was found to be most effective. Sarin et al. [13] also studied the influence of metal contaminants on oxidation stability of jatropha biodiesel, and concluded that copper contamination shows strongest detrimental and catalytic effect on the oxidation stability of biodiesel. Freire et al. [14] conducted thermal investigation on vegetable oil and biodiesel derived from jatropha curcas. Thermo-gravimetric analysis (TGA) and pressurized differential scanning calorimetry (PDSC) were used in order to determine the thermal stability of physic nut biodiesel. It was found that the physic nut oil and ethyl biodiesel from different crops were thermally stable until 203 °C, showing its suitability to be used as mineral diesel substitute. Rashid et al. [15] evaluated moringa *oleifera* oil as a possible source of biodiesel. The oxidation stability of moringa oil using Rancimat test was found to be 15.32 h, which was due to the presence of antioxidants occurring naturally in the oil and low polyunsaturated fatty acid content. The oxidation stability of moringa methyl ester (3.61 h) was significantly lower as compared to the oil itself. The explanation given was that the antioxidants naturally present in moringa *oleifera* oil were either deactivated during the transesterification process and/ or removed during the subsequent purification or separation procedures. There were other studies reported on oxidation stability of biodiesel from less common feedstock such as karanja [16], tung [17], rubberseed [18] and moringa [19] oils.

The objective of the present study is to evaluated the oxidation stability of biodiesels derived from croton *megalocarpus*, jatropha and moringa *oleifera* oils, which are less common feedstocks of African origin using Rancimat method as per EN 14112. The plants are native to East Africa, and are widely found in the mountains of Tanzania, Kenya and Uganda. Rashid et al. [15] and Aliyu et al. [20] reported origin, family and climatic conditions for moringa *oleifera* and croton *megalocarpus* plants respectively. The present study is one of the first studies investigating the oxidation stability behaviour of biodiesel from croton *megalocarpus* oil. The production of biodiesels from these vegetable oils was carried out using optimized conditions of transesterification process. The fuel related properties and

Table 1. Fuel properties of biodiesels and mineral diesel.

Property (Units)	ASTM D6751 Limits	EN 14214 Limits	COME	MOME	JOME	Diesel
Density at 15 °C (Kg/m ³)	--	860-900	880	890	870	840
Viscosity at 40 °C (mm ² /s)	1.9-6.0	3.5-5.0	4.70	4.78	4.35	2.45
Acid Value (mg KOH/g)	0.5 max	0.5 max	0.20	0.16	0.16	--
Flash point (°C)	130 min	>101	160	166	175	65.5
Heating Value (MJ/kg)	--	--	37.24	38.34	37.87	42.34
Lubricity (μm)	--	--	202	228	224	--
Cetane number	47 min	51 min	47.52	62.12	59.24	54.60
Cloud point (°C)	Report	--	-4	10	1	-16
Oxidation stability at 110 °C (h)	3 min	6 min	4.04	5.05	10.43	--

oxidation stability with and without antioxidants of croton oil methyl ester (COME), moringa oil methyl ester (MOME) and jatropha oil methyl ester (JOME) were determined and compared with global biodiesel standards such as ASTM D6751 and EN 14214.

BIODIESEL SYNTHESIS AND CHARACTERISATION

The free fatty acid content (FFAs) of croton, moringa and jatropha oils was 5%, 3.5% and 8.2% respectively, which was above 2% recommended for the application of a two-step transesterification process to convert the high FFA oils to its mono-esters. The acid esterification process followed by transesterification was then used to prepare the methyl esters. The former was performed using acid catalyst (w/w_{oil}: 0.5%H₂SO₄) and 6:1 methanol/ oil molar ratio [21]. After acid esterification, the transesterification was carried out at the following standard conditions: 6:1 methanol/ oil molar ratio (mol/ mol), 1.0 % w/ w_{oil}) potassium hydroxide, 55-60°C reaction temperature, 400 rpm agitation speed and 60 min reaction time [15]. After completion of the reaction, the mixture was transferred to a separating funnel and allowed to cool down to room temperature without agitation, leading to separation of two distinct phases. The upper phase consists primarily of methyl ester (biodiesel) while the lower phase contains glycerol, excess methanol and catalyst, soap formed during the reaction, some entrained methyl esters and partial glycerides. The upper phase i.e. biodiesel was washed with deionised water at 50°C repeatedly until the washing water becomes clear to remove traces of glycerin, unreacted catalyst and soap formed during the transesterification. The ester was then subjected to heating at 110°C to remove excess alcohol and moisture. The final product (biodiesel) formed was a clear, light yellow liquid.

The synthesized biodiesel samples were tested for physical and chemical properties and compared with diesel fuel obtained from local filling station in Budapest, Hungary. Kinematic viscosity was obtained using the miniature u-tube viscometer (TV 4000) as per ASTM D445. The cetane

number was determined using a CFR (Waukesha F-5) engine as per ASTM D613. The cloud point was determined as per ASTM D2500. The calorific value of COME, MOME and JOME were determined by Bomb Calorimeter as per ASTM D240. The density of COME, MOME and JOME were obtained using a density meter (DA-130N) as per ASTM D941. The acid value of COME, MOME and JOME was determined as per ASTM D974. The flash point in the present study was measured using a Pensky martens flash point tester (Stanhope-SETA, 34000-0 U) as per ASTM D93. The lubricity was determined by utilizing a high-frequency reciprocating rig (HFRR). The measurements were performed as per the ISO 12156 HFRR Test method. The oxidation stability investigations were carried out using Rancimat instrument. All measurements were carried out three times for each sample and the average results are reported in table 1.

Tables 1 summarize important fuel properties of COME, MOME and JOME. It is clear from the table that most of the properties satisfy the American and European biodiesel standards (ASTM D6751 and EN 14214). Kinematic viscosity of COME MOME and JOME was found to be 4.70, 4.78 and 4.35 mm²/s at 40 °C respectively, which was slightly higher than mineral diesel however within the global biodiesel specifications. COME, MOME and JOME showed significantly higher flash point compared to mineral diesel (roughly 100°C higher) making biodiesel safer in terms of handling, transport and storage. Acid value of COME MOME and JOME was 0.20, 0.16 and 0.16 mg KOH/g respectively and was less than that specified in ASTM D6751 standard (0.5 max) and EN 14214 (0.5 max). Density of COME, MOME and JOME was 879, 890 and 840 kg/m³ respectively. This was higher than that of mineral diesel but was within the range specified in EN 14214 standards of 860-900 kg/m³. The lower calorific value of COME, MOME and JOME was found to be 37.24, 38.34 and 37.87 MJ/kg respectively which was lower than that of mineral diesel (42.34 MJ/kg). This is because of higher oxygen content in biodiesel. The cetane number (CN) of COME was 47.52, which was lower than that of MOME (62.12) and JOME (59.24). Generally, it is

Table 2. Fatty acid composition of COME, MOME and JOME

Fatty acid composition (wt. %)	COME	MOME	JOME
Myristic acid methyl ester (C14:0)	--	0.2	--
Palmitic acid methyl ester (C16:0)	5.7	12.7	15.7
Palmitoleic acid methyl ester (C16:1)	--	1.1	0.9
Stearic acid methyl ester (C18:0)	3.9	4.9	6.7
Oleic acid methyl ester (C18:1)	11.8	73.0	39.7
Linoleic acid methyl ester (C18:2)	71.6	2.5	36.5
Linolenic acid methyl ester (C18:3)	6.9	0.8	0.3
Arachidic acid methyl ester (C20:0)	--	3.8	--
Eicosenoic acid methyl ester (C20:1)	--	0.9	0.2
Saturated Fatty acids	9.6	21.6	22.4
Monounsaturated fatty acids	11.8	75.0	40.8
Polyunsaturated fatty acids	78.5	3.3	36.8

understood that biodiesel has a higher cetane number than conventional diesel, but cetane number of mineral diesel fuel used in this study was higher than that of COME. The higher cetane number of diesel is an indication that it was likely to have a cetane number enhancing additive in it, known as cetane improvers. This suggests that there is a need of using cetane number improver additive, while using COME as mineral diesel replacement. The lubricity of both COME (202 μm), MOME (228 μm) and JOME (224 μm) was well within the acceptable limits of 460 μm and 520 μm as prescribed in mineral diesel standards EN 590 and ASTM D975 respectively. COME, MOME and JOME displayed a cloud point -4 , 10 and 1°C respectively. The remarkably excellent low temperature flow properties of COME were attributed to lower proportions of saturated fatty acids and a high composition of linoleic and linolenic acid methyl ester (71.6% and 6.9% respectively; Table 2).

The fatty acid composition of biodiesels was analyzed by using a gas chromatography mass spectroscopy (GC MS) (Agilent 6890N). Gas Chromatograph was coupled to an inert mass-selective detector (MSD) (Agilent 5973). 1 μl biodiesel sample was injected using a split ratio (200:1) in an auto sampler at 24.79 psi at an inlet temperature of 250°C . The GC was equipped with a polyethylene glycol column (HP Innowax) of 60 m length, 250 μm i.d. and 0.25 μm film thickness. The oven temperature was kept at 60°C for the first 10 min, and increased at a ramp rate of $4^\circ\text{C}/\text{min}$ up to 220°C and held for 10 min and then ramped at $1^\circ\text{C}/\text{min}$ to 250°C . Helium was used as carrier gas at a constant flow of 1.2 ml/min. The composition of the oil for individual components was obtained from electronic integration measurements using flame ionization detection (FID). All relative percentages determined by GC for each fatty acid methyl ester sample are an average of three runs. These are reported in table 2.

The fatty acid profile of COME, MOME and JOME are summarized in Table 2. COME possessed high percentage of linoleic and linolenic acid methyl ester (71.6% and 6.9% respectively). Oleic acid methyl ester (73%) is the dominant

in MOME, which agrees with previous studies [15]. Generally, COME contains high percentage of polyunsaturated fatty acid methyl esters (78.5%) with only 9.6% of saturated fatty acid methyl esters. MOME contains a low amount of polyunsaturated fatty acid methyl ester (3.3%) with high percentage of saturate fatty acid methyl ester (21.6%). JOME contains 40.8% monounsaturated fatty acid methyl ester and 36.8% polyunsaturated fatty acid methyl ester. JOME possessed 22.4% of saturated fatty acid methyl ester.

OXIDATION STABILITY OF BIODIESEL

Oxidation stability of COME, MOME and JOME with and without different dosages of antioxidants was studied by Rancimat equipment (Metrohm, Switzerland, 873). The working principle of Rancimat instrument is shown in figure 2. A stream of purified air at a flow rate of 10 l/h was passed through the biodiesel sample (10 ml) kept at a constant temperature (110°C) in the Rancimat. The vapours released during the oxidation of biodiesel sample along with the air were passed through the flask containing distilled water, which contains an electrode for measuring the conductivity. The electrode is connected to a measuring and recording device (Figure 2) which indicates the end of the induction period when the conductivity of water begins to increase rapidly. The acceleration of conductivity is caused by the dissociation of volatile carboxylic acids produced during the oxidation process of biodiesel and these volatile organic acids are absorbed in the water. When the conductivity of this solution is recorded continuously, an oxidation curve is obtained, whose point of inflection is known as the induction period; and this provides an excellent parameter for the oxidation stability.

The oxidation stability of COME, MOME and JOME met the ASTM D6751 specifications (3 h) (Table 1) however COME and MOME did not meet the EN 14214 specifications (6 h) as shown in Figure 3.

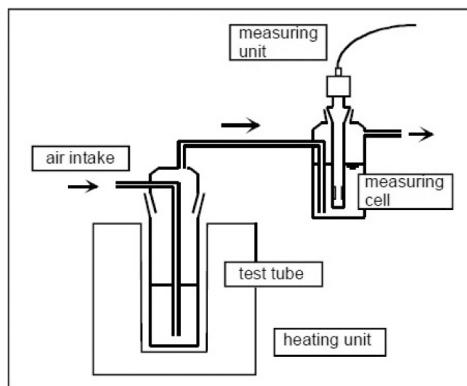


Figure 2. Principle of Rancimat instrument.

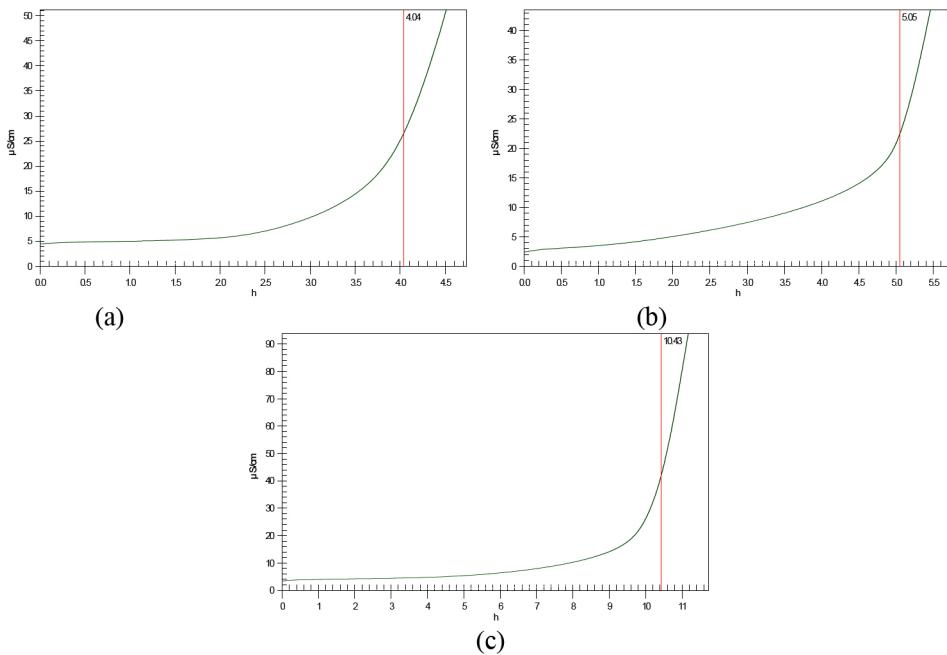


Figure 3. Oxidation stability of Methyl Esters (a) COME and (b) MOME and (c) JOME.

COME recorded oxidation stability of 4.04 h, which is less than that of MOME (5.05 h) and JOME (10.43 h). JOME displayed very high oxidation stability (10.43 h) well within specified oxidation stability standards, possible due to presence of 22.4% saturated fatty acid methyl ester (Table 2) and natural occurring antioxidants, which give excellent oxidation stability properties to JOME. In this study, it was found that the trend of Induction Period had direct correlation with the percentage of saturated fatty acid. For example, MOME with 21.6% saturated fatty acid showed higher oxidation stability than COME (9.6%). MOME showed lower oxidation stability than JOME possible due to higher oleic acid methyl ester (73%) whereas JOME had only 39.7% oleic acid methyl ester with higher saturates (22.4%) compared to MOME (21.6%), but JOME had high linoleate (36.5%) than MOME (2.5%). Further research is required to determine the

natural antioxidants (tocopherols) present in each of these oil, which will affect the oxidation characteristics of the biodiesel. Possible JOME contains higher natural antioxidants and benefits from them. Therefore in order to meet the oxidative stability specifications, a dosage of synthetic antioxidants was required to be added to COME and MOME.

OXIDATION STABILITY OF BIODIESEL SAMPLES WITH ANTIOXIDANTS

The oxidation stability of biodiesel samples was found to be lower than specified by the EN 14214 standards (6 h), except JOME. To increase the oxidation stability, the methyl esters (COME, MOME, and JOME) were doped with stabilizers or synthetic antioxidants. The antioxidants were selected on the

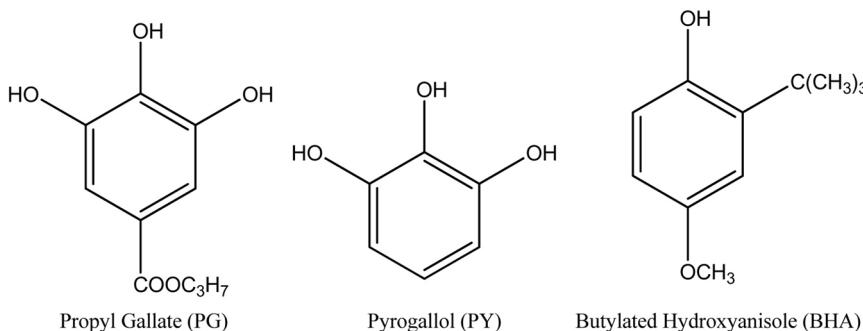


Figure 4. Chemical Structure of the Antioxidants.

basis of their chemical structure, quality, cost, availability and previous studies on biodiesel and vegetable oils used in food industry. Three antioxidants chosen for this study were 1, 2, 3 tri-hydroxy benzene (Pyrogallol, PY), 3, 4, 5-tri hydroxy benzoic acid (Propyl Gallate, PG) and 2-tert butyl-4-methoxy phenol (Butylated Hydroxyanisole, BHA). All these antioxidants have an aromatic ring with at least one hydroxyl ($-OH$) group (Figure 4). PY and PG possess three $-OH$ groups attached to their aromatic rings. The hydroxyl ($-OH$) group of the antioxidant is very active so the hydrogen is abstracted from $-OH$ and donated to the oxidized free radical to inhibit the rate of oxidation in methyl esters. The resulting antioxidant is a stable radical that can react with other fatty acid free radicals and further contribute to oxidation inhibition (Figure 1).

All the three antioxidants were doped at 200, 500 and 1000 ppm dosage for the three methyl esters and tested in the Rancimat to observe their effectiveness. It was found that, oxidation stability increased with the increase in dosage for these antioxidants. The experimental results are shown in figure 5 for these three anti-oxidants dosed biodiesel samples from the three oils.

From these results, it was observed that, the antioxidant PY and PG were more effective than BHA in all dosage for COME, MOME and JOME (Figure 5). PY and PG displayed induction period above specified standards at 200 ppm for all biodiesel samples to meet EN 14214 standards of 6 h. PG showed induction period of 7.59, 7.63, and 21.07 h for COME, MOME and JOME, well above limits at 200 ppm. The induction period for 200 ppm of PY for COME, MOME, and JOME were 12.13, 9.94 and 28.53 h respectively. BHA was found to be the least effective antioxidants. For COME, it required 1000 ppm of BHA to meet the standards. Overall, the effectiveness of these antioxidants was in the order of PY>PG>BHA. PY and PG were more effective than BHA because they possess three $-OH$ groups in their aromatic rings as shown in Figure 4 while BHA has only one $-OH$ group in its molecular structure.

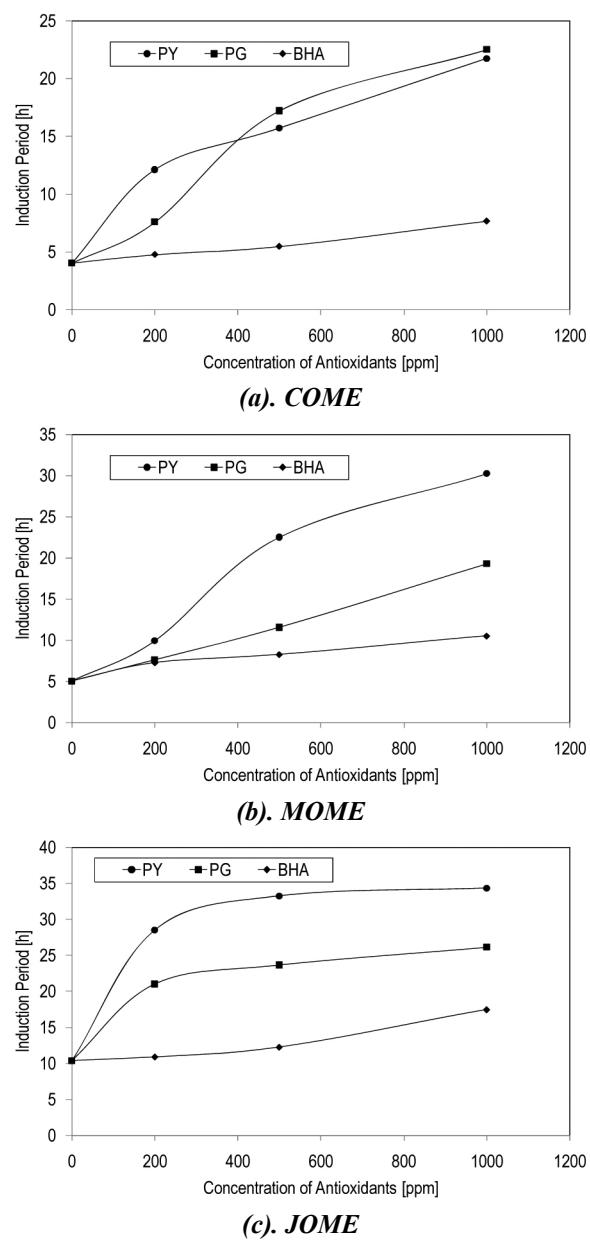


Figure 5. Oxidation stability of the three biodiesel samples with different dosages of antioxidants.

SUMMARY/CONCLUSIONS

The following conclusions can be drawn from the experimental results. Most of the properties of COME, MOME and JOME fulfilled the minimum requirements specified in the ASTM D6751 and EN 14214 biodiesel standards. However the oxidation stability of the biodiesels (COME, and MOME) did not meet the EN 14214 specifications (6 h), because of presence of unsaturated fatty acid, which is the primary reason for lower oxidation stability of biodiesels. Also, antioxidants naturally present in vegetable oils were either deactivated during the transesterification process and/ or removed during the subsequent purification or separation procedures. Therefore addition of synthetic antioxidants was required to improve the oxidation stability of biodiesel samples. Among the three antioxidant used in the present study, PY and PG were found to be more effective compared to BHA in all dosage ranges. Only 200 ppm of both PY and PG was able to satisfy the minimum requirement of oxidation stability as specified in the ASTM D6751 and EN 14214 biodiesel standards for COME and MOME. In summary, the biodiesel derived from African origin non-edible oils such as COME, MOME and JOME can be utilized as partial substitute for mineral diesel. Additionally, further research is required on biodiesels derived from vegetable oils of African origin such as exploring the effect of antioxidants on CI engine performance, exhaust emissions and combustion characteristics.

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