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Experimental investigations of effect of Karanja biodiesel on tribological properties of lubricating oil in a compression ignition engine

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HIGHLIGHTS

- Comparison of lube oil degradation for diesel & KOME20 in 200 h test.
- Resinous material content in lube oil increased for biodiesel fuelling.
- Comparatively higher oxidation of lube oil from biodiesel engine.
- Higher wear trace metals in lube oil from biodiesel engine after 100 h.
- Copper corrosiveness of lube oil of biodiesel engine similar to diesel.

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ABSTRACT

For large-scale implementation of biodiesel in transportation engines, its effect on lubricating oil degradation needs to be experimentally investigated. Due to differences in chemical composition of biodiesel and mineral diesel, the comparative effects on lubricating oil degradation and residual useful life for long-term application will be different. In this study, effect of 20% Karanja biodiesel blend on lubricating oil tribology was studied vis-à-vis mineral diesel in a 200 h long endurance test. Higher increase in density, carbon residue and ash content was observed for biodiesel blend fuelled engine's lubricating oil in comparison to diesel. Higher amount of resinous polymerized material in the lubricating oil of biodiesel fuelled engine indicated possibility of higher oxidation and polymerization of base-stock of lubricating oil of the biodiesel fuelled engine. After 100 h, higher increase in concentration of wear trace metals such as iron, aluminum, copper, chromium and magnesium in the lubricating oil of biodiesel fuelled engine in comparison to mineral diesel indicated significant deterioration of lubricating oil.

1. Introduction

Due to depletion of petroleum resources and resulting environmental degradation and global warming, alternative transportation fuels are getting worldwide consideration. Biodiesel is the most readily considered renewable alternative to mineral diesel at current stage of technology development, which remains the most widely used transportation fuel. Before large scale implementation of biodiesel, its effects on engine performance, emissions, durability and lubricating oil degradation needs to be investigated. Some studies have experimentally evaluated the lubricating oil degradation during long duration tests on static engines experiments and field trials [1–10]. Verhaeven et al. investigated the effect of rapeseed oil methyl ester (RME) and used vegetable oil methyl ester (UVOME) on engine durability in a demonstration study using ten vehicles operated over a distance of 100,000 km [2]. Analysis of the lubricating oil samples taken at an interval of 7500 km vehicle run confirmed that no special degradation of the engine or lubricating oil took place [2]. Lin et al. compared the effect of fuels on lubricating oil degradation over 300 h (18,000 km equivalent) operation of heavy duty diesel engine fuelled with palm biodiesel blends [11]. They reported that viscosity (@ 40 °C) of the lubricating oil for diesel fuelled engine after 300 h reduced to 95.1 cSt from the initial viscosity of 107 cSt and total alkaline number increased to 8.24 mg KOH g^{-1} from the initial value of 7.89 mg KOH g^{-1}. For biodiesel, the corresponding values of viscosity and total alkaline number after 300 h were 96.8 cST and 8.26 mg KOH g^{-1} respectively [11]. Staat and Gateau reported the results of 3 years long investigations on 2000 vehicles in France using rapeseed methyl ester (RME) as fuel [1]. For more than 50% concentration of RME in the fuel, slight reduction in lubricating oil viscosity with usage

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was observed but it was not significant enough to alter the lubricating oil change interval [1]. Agarwal investigated the effect of 20% biodiesel blend (B20) of linseed oil methyl ester on the tribological properties of lubricating oil in 512 h endurance test [7]. He reported that the fuel dilution was lower for the lubricating oil from the biodiesel fuelled engine in comparison to diesel fuelled engine by measuring viscosity and flash point [7]. Sinha and Agarwal observed lower wear trace metal concentration of Fe, Cr, Cu, Zn, Ni and Mg in the used lubricating oil from B20 fuelled engine in comparison to mineral diesel fuelled engine. Pb and Al were found in slightly higher concentration in the lubricating oil from B20 fuelled engine, which might be due to the attack of biodiesel on paints and bearings [3,6]. Agarwal et al. reported lower concentrations of trace metals such as Fe, Cu, Zn, Mg, Cr, Pb, and Co in the lubricating oil from B20 fuelled engine in comparison to mineral diesel fuelled engine [12–13].

Fuel chemistry plays an important role in the performance and useful life of lubricating oil [7,14]. Properties of lubricating oil change with its usage due to fuel dilution and addition of contaminants from the engine due to wear, carbonaceous materials and impurities entering with intake air [14]. Biodiesel is comparatively more chemically reactive than mineral diesel due to presence of oxygen in the molecular structure of biodiesel [15]. Differences in physical properties of biodiesel and mineral diesel cause different levels of fuel dilution of the lubricating oil. Hence, the effect of biodiesel on the lubricating oil degradation needs to be evaluated in long-duration engine tests before taking decision about their large-scale usage. There are very few studies reporting the long-term effects of biodiesel on the lubricating oil degradation in the open literature. Karanja oil is being considered as an important feedstock with a potential of producing biodiesel on large scale because it is well adapted to climatic conditions and is available in surplus quantities throughout South Asian region [9,10]. In this study, effect of 20% (v/v) blend of Karanja biodiesel with mineral diesel (KOME20) on the lubricating oil degradation was experimentally investigated during 200 h long endurance test vis-à-vis baseline mineral diesel. Various tribological studies on the lubricating oil samples drawn at a regular interval from mineral diesel and KOME20 fuelled engines were conducted in order to correlate the effect of fuel properties on lubricant degradation and its residual useful life.

2. Materials and methods

Effect of KOME20 on lubricating oil degradation was studied in a four stroke, four cylinder, variable speed, medium duty, transportation compression ignition (CI) engine (Mahindra & Mahindra, MDI 3000) during 200 h long endurance test in two phases. 200 h endurance test duration is equivalent to 20,000 km field trial of the vehicle. Recommended oil change interval by the manufacturer was 20,000 km. Hence 200 h endurance test duration was conducted. Technical specifications of the test engine are given in Table 1.

Table 1
Technical specifications of the test engine.

Engine type	Four stroke, naturally aspirated, water cooled
Number of cylinders	Four, in-line
Compression ratio	18
Combustion system	Direct injection, re-entrant bowl
Bore/stroke	88.9/101.6 mm
Swept volume	2520 cc
Liner type	Cast iron replaceable wet liners
Fuel injection timing	(SOI) 17 ± 1° BTDC
Max. torque	152 N m @ 1800 rpm
Oil sump capacity	7.0 l

In the first phase, engine was operated for 200 h on mineral diesel for generating baseline data under severe loading according to IS: 10,000 (Part IX) specifications [16]. In the second phase, KOME20 was used as a fuel in place of mineral diesel, keeping all other test conditions identical to the first phase. At the start of the second phase, new set of liners, pistons, piston rings, gudgeon pins and bearings were installed. Table 2 shows a 2 h engine loading cycle, which was followed during the long-term endurance test. Engine was subjected to 100 such loading cycles in each phase. For comparing the effect of KOME20 and mineral diesel on the lubricating oil, samples were drawn from the oil sump during the endurance test after a regular interval of every 20 h engine operation.

A large number of tests were conducted on the lubricating oil samples in order to evaluate the comparative performance of fuels such as measurement of density, viscosity, flash point, moisture content, pentane and benzene insoluble. Density of the lubricating oil samples was measured using a portable density meter (Kyoto Electronics, DA-130N). Viscosity of the lubricating oil samples was determined at 40 °C and 100 °C temperatures using kinematic viscometer (Stanhope-Seta, Setavis 83541-3). Flash point of the lubricating oils was measured by flash point apparatus (Sanhope-Seta, 33000-0) according to relevant ASTM procedures applicable.

For determination of ash content, lubricating oil sample taken in a silica crucible was ignited and was allowed to burn until only ash and carbon remained. The carbonaceous residue was reduced to ash by heating in a muffle furnace at 775 °C for four hours then it was cooled and weighed. TBN measurement apparatus (Kittiwake, DIGI cell) and "Reagent C" were used for determining the total base number of the lubricating oil samples. The copper corrosion bath (Stanhope Seta, Setavis 11300) was used for determining the copper corrosion potential of test fuels and lubricating oil samples. Freshly polished copper strip was immersed in lubricating oil sample for 3 h and maintained at 100 °C temperature. For determination of resinous material and oxidation of lubricating oil, pentane, benzene and toluene insolubles were measured separately. These tests were conducted using an oil centrifuge (Remi Instruments, R-19). 10 grams of lubricating oil sample was put in preweighed conical centrifuge tube and filled up to 100 ml mark with pentane. The tube was shaken until the mixture became homogeneous. Pairs of tubes were placed in the centrifuge on opposite sides of the rotating head in order to maintain balance and centrifuge was then operated at 1500 rpm to maintain relative centrifugal force (ratio of centrifugal and gravitational accelerations) between 600 and 700 for 20 min duration. The insolubles get deposited at the bottom of the tubes. Supernatant liquid was thrown without disturbing the precipitate formed in the bottom of the conical tube. This insoluble was again centrifuged with 50 ml pentane, twice 20 min each time. This precipitate was then dried for 30 min in an oven at 105 °C temperature and then the centrifuge tube was weighed to find the mass of pentane insolubles. For determining toluene insolubles, lubricating oil sample was first dissolved in pentane and centrifuged as described above. After washing the precipitate second time with pentane, toluene–ethanol solution was added up to 50 ml mark and samples were subjected to centrifugation for 20 min at 1500 rpm. This procedure was repeated again by substituting toluene–ethanol mixture with toluene. For benzene insoluble determination, toluene was

Table 2
Engine loading cycle for the endurance test (IS 10,000 Part IX [14]).

Speed (rev/m)	Load (N m)	Running time (min)
2600	105	50
1800	135	45
750	No load	5
2600	135	20

replaced with benzene. The precipitate was dried for 30 min at in an oven 105 °C temperature and centrifuge tube was weighed to find the amount of toluene/benzene insolubles.

For determination of trace metal concentrations in the lubricating oil samples, approximately 10 g lubricating oil sample was converted into ash by heating in a muffle furnace. This ash was then digested in 10 ml concentrated HNO_3 by heating on a hot plate at 200 °C for 3 h. This digested solution was diluted by milli-Q water to makeup 100 ml solution. Trace metal concentration in this diluted solution were determined by inductively coupled plasma-optical emission spectro-photometer (ICP-OES) (Thermo Fischer Scientific, iCAP DUO 6300 ICP).

3. Results and discussion

Investigation of the effects of any new alternative fuel on tribological properties of the lubricating oil is very important for assessing the suitability of new fuel for existing engines. Various lubricating oil investigations were carried out to get an assessment about the health of lubricating oil and the engine. All these tests were used for indirect interpretation of comparative performance of new fuels in unmodified engines.

3.1. Density

Fig. 1 shows the variation in density of lubricating oil samples collected at an interval of 20 h from the sump of mineral diesel and KOME20 fuelled engines. Addition of wear debris, fuel and moisture affects the density of the lubricating oil. The density of lubricating oil samples from both engines show an increasing trend with usage. Increase in density at a higher rate in the initial phase (first 20 h) of engine operation may be due to higher wear of engine components initially, due to break-in.

Lubricating oil of KOME20 fuelled engine shows higher rate of change of density in comparison to lubricating oil from mineral diesel fuelled engine in entire duration of the test. Fuel dilution decreases the density of lubricating oil because the density of fuels is lower than lubricating oil. Density of KOME20 and mineral diesel was 0.841 and 0.831 (g/cm^3) respectively therefore fuel dilution of lubricating oil by KOME20 causes lesser reduction in density of lubricating oil in comparison to mineral diesel. Due to continuous heating and exposure to moisture, polymerization of lubricating oil also takes place, which increases the density. Change in density is therefore a net resultant effect of wear debris addition, lubricating oil polymerization, moisture addition etc., which tends to increase lubricating oil density whereas fuel dilution tends to decrease the lubricating oil density. Hence density measurement alone does not provide any conclusive information regarding comparative effect of fuel chemistry on the lubricating oil.

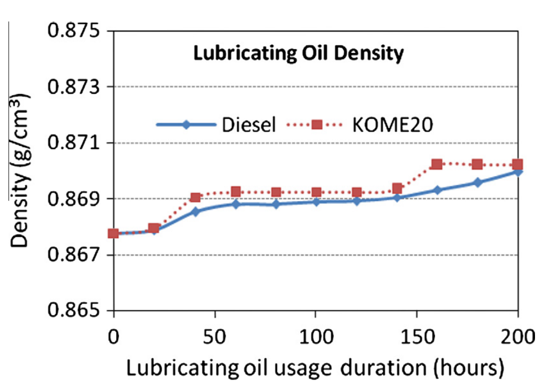

Fig. 1. Lubricating oil density.

3.2. Viscosity

Fig. 2 shows the variation of viscosity of lubricating oil samples of mineral diesel and KOME20 fuelled engines measured at 40 °C and 100 °C temperatures. These viscosities were measured after filtering the lubricating oils through 75 μm filters so that the blockage of the capillary tubes of kinematic viscometer can be avoided.

Viscosity of lubricating oil samples drawn from both engines initially increases sharply. Then rate of increase in viscosity for KOME20 fuelled engine's lubricating oil becomes higher than that of mineral diesel. Lubricating oils viscosity starts decreasing after 100 h for both fuels. Lubricating oil of KOME20 fuelled engine shows higher reduction beyond 100 h. Viscosity of lubricating oil samples measured at 100 °C was relatively higher for mineral diesel fuelled engine. Oxidation and polymerization of lubricating oil tend to increase the viscosity, whereas fuel dilution tends to decrease the viscosity. This trend of viscosity indicates that there is higher degree of fuel dilution in case of KOME20, which is causing higher rate of viscosity increase in the beginning due to 'fuel induced oxidation'. Higher viscosity, surface tension and density along with poor volatility of Karanja biodiesel in comparison to mineral diesel increase the liquid length of fuel spray in the engine combustion chamber. This leads to higher probability of fuel droplets hitting the liner walls, which in-turn increase the chances of mixing of fuel with lubricating oil, resulting in increased fuel dilution of the lubricating oil in the oil sump. When rate of increase in viscosity due to oxidation becomes lower, viscosity starts reducing due to fuel dilution.

3.3. Carbon residue and ash content

Fig. 3(a) and (b) shows the variation of carbon residue and ash content of the lubricating oil samples drawn from mineral diesel and KOME20 fuelled engines. Change in carbon residue is a qualitative indicator of polymerization of lubricating oil because increasing content of non-combustible polymerized part of higher molecular weight base-stock remains as carbon residue after combustion/pyrolysis of the lubricating oil. Measured carbon residue of the lubricating oils from both fuels was almost similar for the entire test (Fig. 3(a)). Change in ash content of lubricating oil with usage indicates metallic wear debris addition to the lubricating oil with usage. Slightly higher ash content of lubricating oil of KOME20 fuelled engine compared to mineral diesel fuelled engine suggests that KOME20 fuelled engine produces possibly higher wear debris.

3.4. Copper corrosion

Copper strip corrosion test of lubricating oil samples from mineral diesel and KOME20 fuelled engines was performed to assess corrosiveness of lubricating oil to the copper containing engine components. Copper corrosion bath and ASTM standards for copper corrosion were used for this test. Fresh lubricating oil matched grade 1a, which is light orange, almost same as freshly polished copper strip.

Copper corrosiveness of all the lubricating oil samples drawn from mineral diesel as well as KOME20 fuelled engines were also grade 1a. Hence, it can be concluded that Karanja biodiesel does not cause any additional harm/damage to the copper containing components of the engine.

3.5. Total base number

Total base number (TBN) is a measure of remaining alkalinity reserve of the lubricating oil, which is an indicator of its ability

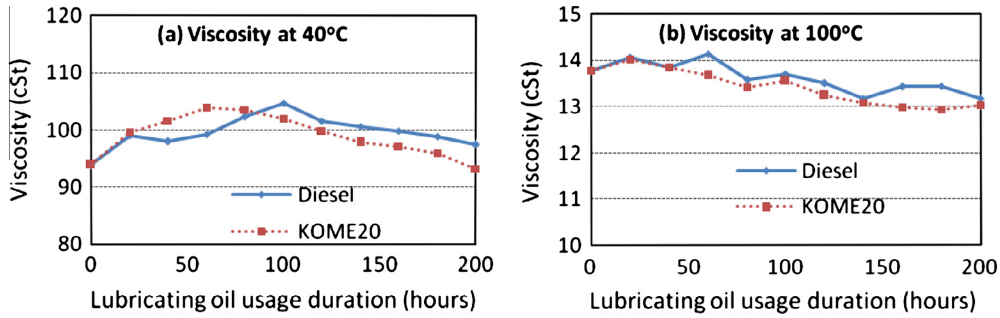

Fig. 2. Viscosity of lubricating oil at (a) 40 °C and (b) 100 °C.

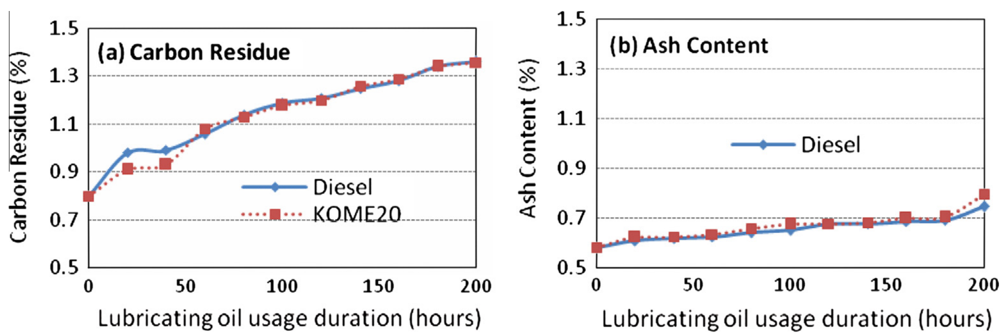

Fig. 3. Variation of (a) carbon residue and (b) ash content of lubricating oil samples.

to counter corrosive acid's adverse effects. Higher TBN indicate lower concentration of free acids in the lubricating oil [6].

Fig. 4 shows the variation of TBN of lubricating oil samples drawn from mineral diesel and KOME20 fuelled engines. It is observed that TBN decreased with lubricating oil usage for mineral diesel as well as KOME20 fuelled engines. Rate of reduction of TBN of lubricating oil samples from mineral diesel and KOME20 were identical up to 100 h. After 100 h of engine operation, TBN of lubricating oil from KOME20 fuelled engine was found to be lower in comparison to mineral diesel fuelled engines, which suggests relatively faster depletion of corrosion inhibitors (additives) in the lubricating oil of KOME20 fuelled engine, possibly as a result of its interaction with biodiesel, which is chemically more active compared to mineral diesel. Oxidation of ester molecules of biodiesel present in the lubricating oil encourage formation of organic acids, which reduce the reserve alkalinity of lubricating oil.

3.6. Flash point

Flash point of lubricating oil samples reduced with usage for both fuels (Fig. 5). Flash point of Karanja biodiesel and mineral diesel used in this study were 168 and 49.5 °C respectively. Same level of fuel dilution for KOME20 would have resulted in lower flash point depression of the lubricating oil due to higher flash point temperature of KOME20 compared to mineral diesel. Almost similar flash point after 200 h engine operation and viscosity variation (Fig. 2(a)) indicated that fuel dilution of lubricating oil was higher for KOME20 fuelled engine even though depression in lubricant's flash point was lower for KOME20 compared to mineral diesel.

3.7. Pentane, benzene and toluene insolubles

Measurement of insolubles in the lubricating oil samples assists in evaluating the performance of used oil [17]. Pentane, toluene and benzene insolubles in the lubricating oil samples of mineral diesel and KOME20 fuelled engine were measured according to ASTD D893 [17].

Pentane insolubles reflect amount of sludge formed by oil oxidation, metallic wear debris, fuel carbon and foreign particles entering the system. Fig. 6(a) shows the variation of pentane insoluble of lubricating oil with usage. Benzene and toluene, which are aromatic, can dissolve resinous material of lubricating oils also.

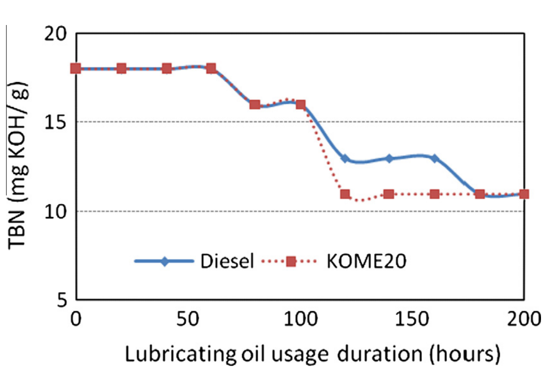

Fig. 4. Variation of total base number of the lubricating oil samples.

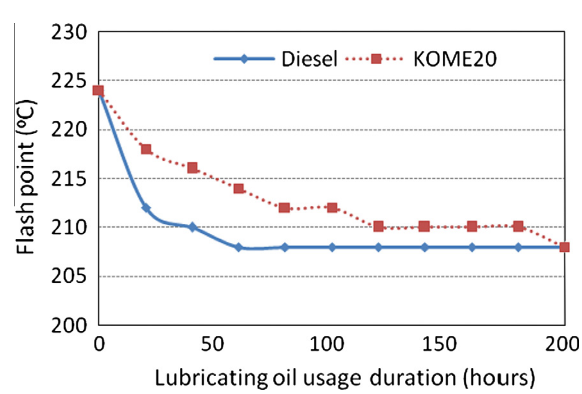

Fig. 5. Variation of flash point of the lubricating oil samples.

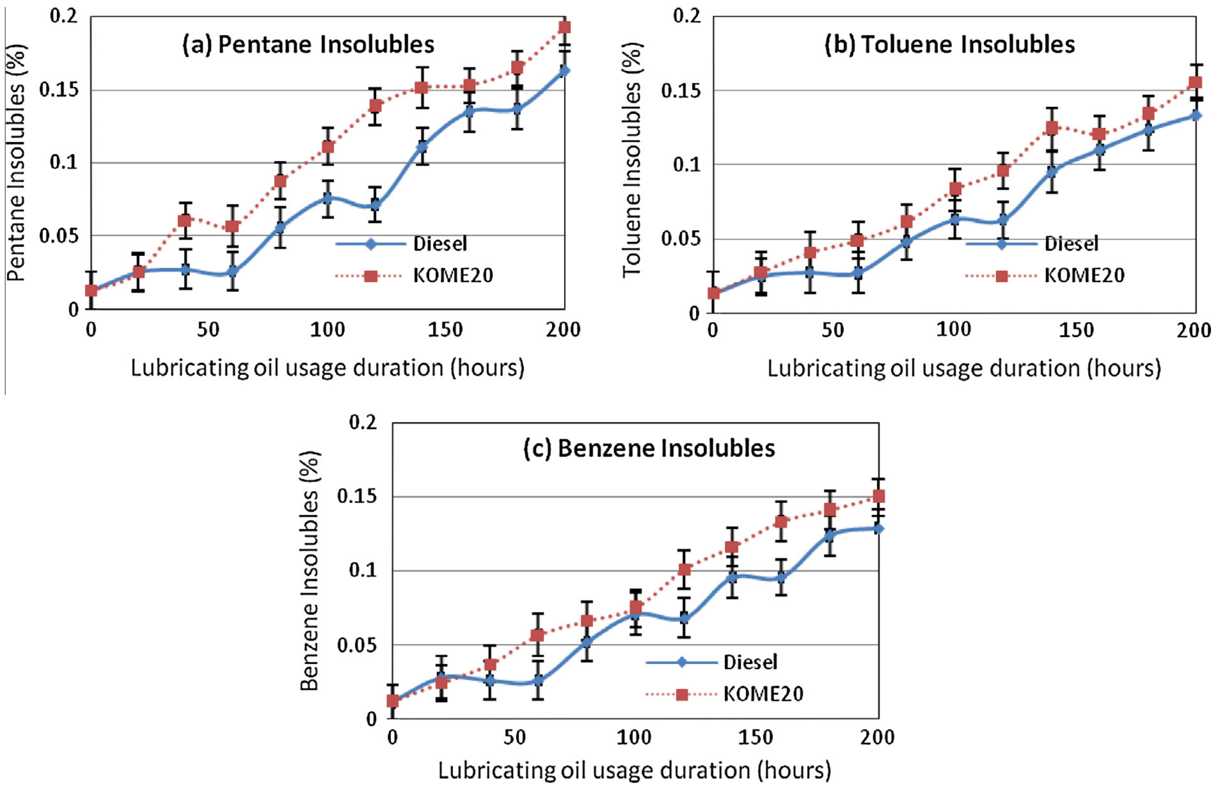

Fig. 6. Variation of (a) pentane insolubles, (b) toluene insolubles and (c) benzene insolubles in lubricating oil samples.

Hence, difference in the pentane and toluene/benzene insolubles indicates resinous material content of the lubricating oil. Higher difference between pentane and benzene insolubles indicate higher oxidation of lubricating oil resulting in higher polymerization of lubricating oil base-stock.

Resinous material content evaluated by difference of pentane-toluene insoluble and pentane-benzene insoluble is shown in Fig. 7. Higher benzene and toluene insoluble for lubricating oil samples of KOME20 fuelled engine (Fig. 6(b) and (c)) indicates higher wear debris for KOME20 fuelled engine. Higher resinous material in the lubricating oil of KOME20 fuelled engine (Fig. 7) indicates higher extent of lubricating oil oxidation and polymerization of lubricating oil base-stock due to addition of Karanja biodiesel in the fuel by fuel dilution.

3.8. Wear trace metals

Trace metal concentration in the lubricating oil increases with usage due to accumulation of wear debris. Concentration of wear metals in the lubricating oil can be used as an indicator of wear of engine components containing those metals [18]. Concentrations of wear trace metals present in the lubricating oil samples

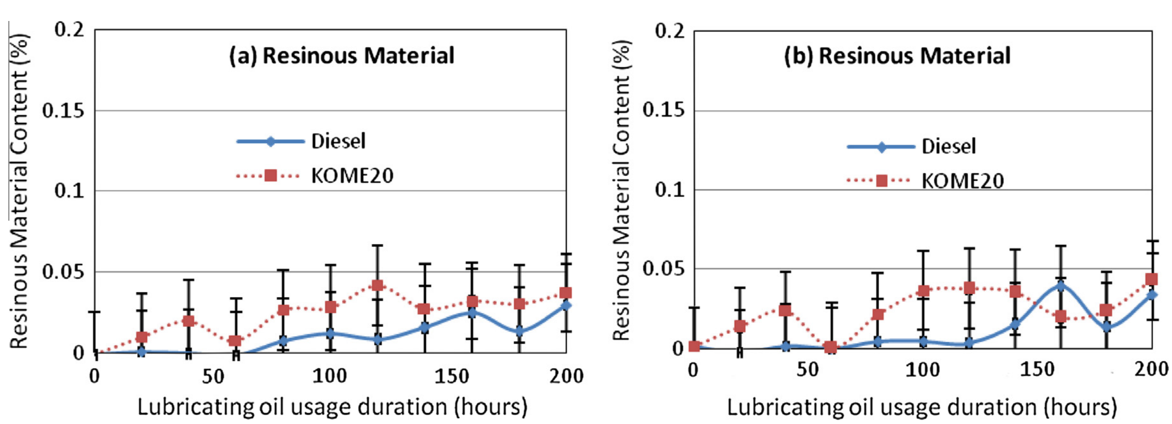

Fig. 7. Variation of (a) resinous material content (difference of pentane and toluene insolubles) and (b) Resinous material content (difference of pentane and benzene insolubles) in the lubricating oil samples.

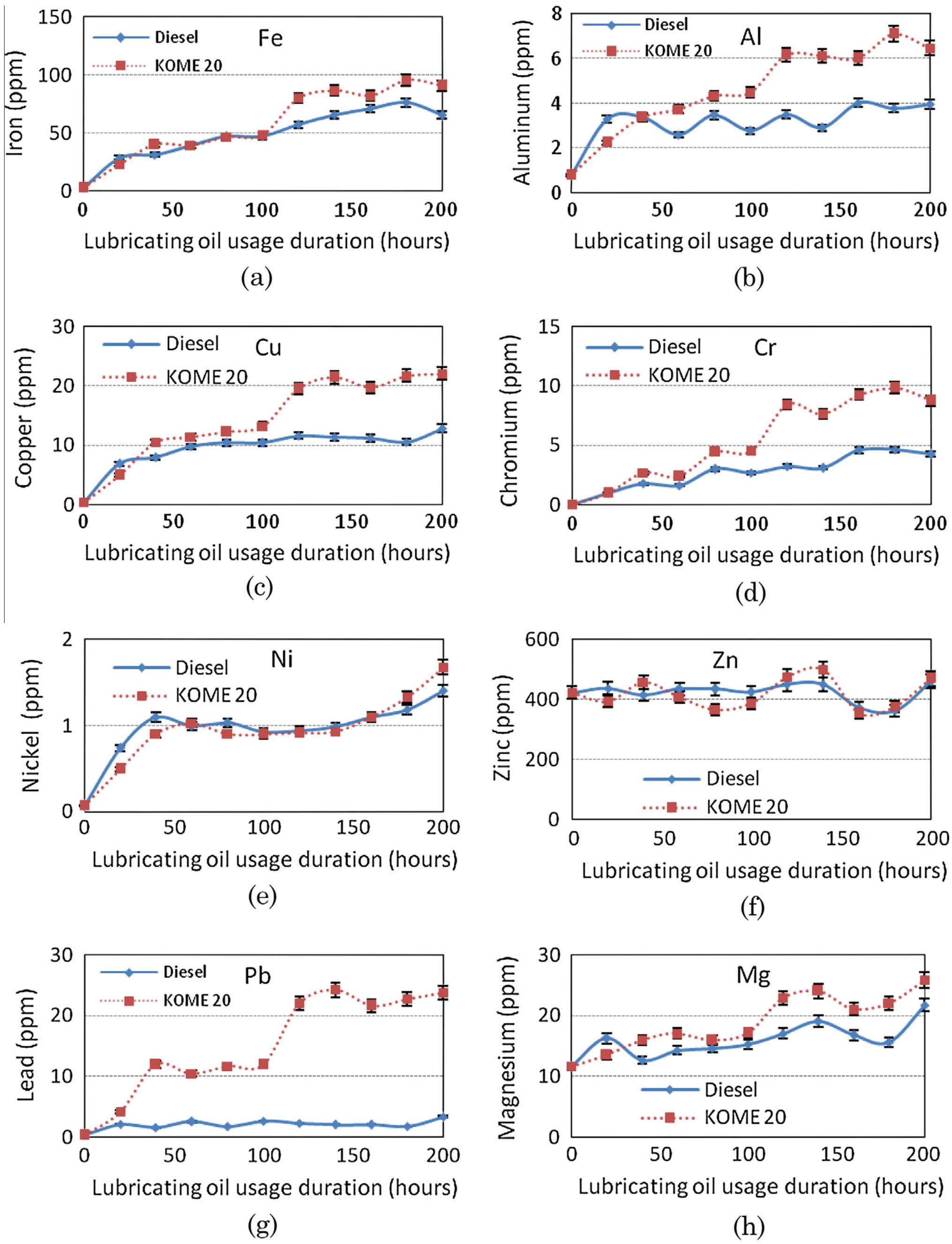

Fig. 8. Variation of wear trace metals in lubricating oil (a) iron, (b) aluminum, (c) copper, (d) chromium, (e) nickel, (f) zinc, (g) lead and (h) magnesium.

of mineral diesel and KOME20 fuelled engines were measured by ICP-OES. These results are discussed in following sub-sections for each trace metals.

3.8.1. Iron

Sources of iron in the lubricating oil are wear debris from cylinder liner, piston, rings, valves, gears, shafts, bearings, rust, and crankshaft [3,7]. It can be seen that wear of iron components in mineral diesel and KOME20 fuelled engines are almost similar up to 100 h of engine operation (Fig. 8(a)). After 100 h, iron concentration rises sharply in the lubricating oil of KOME20 fuelled engine. This indicates possibility of higher oxidation of lubricating oil from KOME20 fuelled engine, which possibly results in increased wear of iron components for KOME20 fuelled engine.

3.8.2. Aluminum

Aluminum in the lubricating oil originates from wear of piston, bearings, dirt, additives and thrust washers [3,7]. Aluminum concentration in the lubricating oil of KOME20 fuelled engine is marginally higher than lubricating oil from mineral diesel fuelled engine (Fig. 8(b)). This indicates that the wear of aluminum containing components is slightly enhanced by addition of 20% Karanja biodiesel to the mineral diesel.

3.8.3. Copper

Copper in the wear debris originate from bearings, bushes, valve guides, etc. [3]. Concentration of copper in the lubricating oil of KOME20 fuelled engine increases sharply after 100 h of engine operation (8(c)). This increased concentration indicates the possibility of relatively inferior lubrication after the oxidation of lubricating oil, which results in higher wear of bearings after 100 h of usage.

3.8.4. Chromium

Chromium in the lubricating oil could originate from wear of cylinder liner, compression rings, gears, crankshaft and bearings [3,7]. Chromium concentration is quite small for the two sample sets, up to 100 h of engine operation. However KOME20 fuelled engine demonstrates increase in chromium concentration in the lubricating oil after 100 h (Fig. 8(d)). Measurement of weight loss of piston rings has shown clearly lesser wear of the KOME20 fuelled engine's piston rings. Hence it can be inferred that this increase in the concentration of KOME20 fuelled engine is due to higher wear of bearings.

3.8.5. Nickel

Fig. 8(e) shows the concentration of nickel in the lubricating oil from mineral diesel and KOME20 fuelled engine. Wear of nickel containing bearings, valves, gear plating etc. are the main source of nickel in the wear debris of the lubricating oil samples [3,7]. Concentration of nickel was very small and was almost similar for the lubricating oil samples from mineral diesel and KOME20 fuelled engines.

3.8.6. Zinc

Zinc containing additive compound zinc di-alkyl-di-thio-phosphate (ZDDP) is added to the lubricating oil as multi-functional additives, which acts as antioxidant, anti-wear additive, detergent, and extreme pressure additive. Fresh lubricating oil contains a reasonable amount of zinc traces as organo-metallic complex. The zinc in the lubricating oil could originate from additive depletion, wear of bearings, brass components, and neoprene seals [3]. Initial zinc concentration in lubricating oil from both engines was significant however it remained largely constant with time (Fig. 8(f)).

3.8.7. Lead

Lead in the lubricating oil originates from wear of bearings, paints and grease [3,7]. Lead wear debris in the lubricating oil of KOME20 fuelled engines were higher than lubricating oil from mineral diesel fuelled engine (Fig. 8(g)).

3.8.8. Magnesium

Magnesium is added to the lubricating oils as detergent inhibitor additive. Magnesium in wear debris may originate from additive depletion, wear of cylinder liner surface, bearings, gear box housing etc. [3]. Variation of magnesium concentration in the lubricating oil is shown in Fig. 8(h). Magnesium concentration of KOME20 fuelled engine increased after 100 h, indicating the possibility of higher wear due to degradation of lubricating oil.

4. Conclusions

Effect of 20% Karanja biodiesel blend on lubricating oil tribology was studied vis-à-vis baseline mineral diesel in a 200 h long endurance test. Important conclusions from this study are as follows:

- Higher increase in density, carbon residue and ash content was observed for biodiesel fuelled engine's lubricating oil with usage.
- Variation in viscosity of lubricating oils from biodiesel and mineral diesel fuelled engines indicated possibility of higher oxidation and polymerization of lubricating oil drawn from biodiesel fuelled engine.
- Higher amount of resinous polymerized material in the lubricating oil of biodiesel fuelled engine indicated possibility of higher oxidation and polymerization of lubricating oil drawn from biodiesel fuelled engine in comparison to mineral diesel fuelled engine.
- There was no difference in the copper corrosion potential of the lubricating oils from mineral diesel and biodiesel fuelled engines.
- Higher ash content of the lubricating oil from biodiesel fuelled engine indicated possibility of higher wear trace metals, which were confirmed by measurement of wear trace metals in lubricating oil samples.
- Higher concentrations of iron, aluminum, copper, chromium and magnesium in the lubricating oil from biodiesel fuelled engine in comparison to mineral diesel fuelled engine after 100 h indicated significant deterioration of lubricating oil.

No severe operational problem was observed during 200 h endurance test of KOME20 fuelled engine in comparison to mineral diesel fuelled engine except higher oxidation of lubricating oil of KOME20 fuelled engine, which indicated the need for reformulation of lubricating oil for biodiesel fuelled engines, in case of large-scale implementation.

Acknowledgements

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