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Role of FT-IR in Assessing Lubricant Degradation – A Study on Palm Oil Methyl Ester Blended Lubricant

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In this paper, studies were made on the palm oil methyl ester (POME) added lubricants using FT-IR for monitoring oil degradation. In order to assess the degradation characteristics of POME added lubricant by FT-IR, static oxidation test was conducted using three different blended lubricants (viz, zero percent POME, five percent POME and ten percent POME with mineral-based oil) for 280 hrs. The oxidation temperature was set at 140 °C. FT-IR quantitative data indicate an increased in oxidation products which was formed from 10% POME added lubricants after 280 hrs of oxidation test. The 5% POME added lubricant and mineral-based lubricant (without POME) showed less oxidation product after the test. From the FT-IR spectrum analysis of the oxidized oils it could be concluded that 5% POME can improve the performance of mineral-based oil by forming protective films.

Keywords : Lubricant degradation, Palm oil methyl ester, Oxidation

1. INTRODUCTION

A lubricant begins to change slowly from the moment it enters into the lubrication system of an operating engine or machine. Generally, internal combustion engines, such as diesel engines, are the most stressful on lubricating oil. In this environment, high temperature and stress promote thermal and mechanical degradation of the lubricant base stock and consume additive constituents. A number of researchers [1-5] have studied the various degradation characteristics of lubricants and also focused on the remaining useful life of the lubricant. Out of these, determination of remaining useful life of the lubricant by differential scanning calorimetry, voltametric methods and fourier transform infrared spectroscopy or FT-IR method are significant. Since the lubricant should not be used beyond its useful life, which may result in component wear and malfunctioning of the engine and machine, a periodic drain-off and recharge of lubricant is performed for engine and equipment where central lubrication system is used. However, no work has been reported on the degradation of POME added lubricants in connection with above characteristics. This paper presents experiments on the degradation properties results of the lubricants after static oxidation test using FT-IT spectrometer.

2. EXPERIMENTAL DETAILS

2.1 Oil sample

A commercial mineral-based lubricant (equivalent to SAE 40) was pre-mixed with 0%, 5% and 10% POME using a magnetic stirrer for about 10 minutes.

2.2 Static oxidation test

In order to assess the oxidation stability of POME added lubricants, static oxidation test was conducted for 280 hours. The sample oil (about 300 ml) was put into a 500 ml glass beaker and placed on a hot-plate heater. The oxidation temperature was set at 140° C. The aeration was kept constant throughout the test. At every forty (40) hours time interval, the oil sample was analyzed for degradation characteristics. The degradation characteristics were measured using FT-IR spectrometer.

2.3 Fourier Transform–Infrared (FT-IR) Spectroscopy Analysis

Infrared spectroscopy is the study of the interaction of infrared light with matter. The fundamental measurement obtained in infrared spectroscopy is an infrared spectrum, which is a plot of measured infrared intensity versus wavelength of light [6]. This new technique is widely used in oil analysis because of its capabilities to extract lubricant degradation and contamination parameters from the measured spectrum of the used oil sample within very short time. The system used for FT-IR spectroscopy is Nicolet Magna-IR 550 series II. The spectral range was maintained from 400 cm^{-1} to 4,000 cm^{-1}. For each spectrum 10 scans were done keeping the optical path difference (OPD) to 8 cm/sec. The spectrum difference between the new oil and used oil represent the products formed by oxidation of the base oil, potential presence of contaminants in the oil, and depletion of the additives.

3. RESULTS AND DISCUSSION

FT-IR qualitative analysis of degraded oil

FT-IR spectroscopy analysis test results of three different degraded lubricants are presented in Table 1. The position of the bands of the peak are: 2,700-2800 cm^{-1}, 1,600-1700 cm^{-1}, 1200-1,100 cm^{-1}, 800-900 cm^{-1}, etc. The band position of the peaks indicate that changes have taken place on bands around 819, 1,168, 1,608, 1,747, 1,748, 2,732 etc. as the wave numbers of the peaks are almost same for all spectrum. This means that there is possibility of loss of additives associated by production of contaminant functional group and also oxidation of the POME blended lubricants. Almost similar observation was obtained by Mukherjee et al. [5] where they tested heavy duty earth moving machinery oil for remaining useful life of lubricant assessment. The characteristic group frequencies are identified as follows [6, 7]:

Wave number range(cm^{-1})	*Characteristics*
2700 – 2800	Formation of aliphatic, aromatic compounds; formation of carboxylates.
1700 – 1800	Due to the presence of carbonyl

	containing degradation products of the lube oil.
Around 1600	Formation alkanes, amides, alcohols
1100-1200	Formation of sulfation products. Presence of ester including double bond (C-O-C) stretching
Around 830	Formation or presence of two adjacent hydrogen atoms.

From Table 1 it can be seen that the presence of new compounds in the region of carbonyl vibration (1760-1710 cm^{-1}) was usually associated with the presence of ketones (1715 cm^{-1}) or carboxylic acids (1710 cm^{-1}) due to the oxidation of POME added lubricants. Another peak at 1752 which was obtained from the 5% POME-lubricant indicates the possible formation of a membered ring lactones. Lactones, by their ring nature, can form hydroxy acids in the presence of Fe^{3+} ions. These acids, with peaks in the carbonyl vibration region, exhibit the ability to physisorb onto the metallic surfaces and form protective films. However, under 10% POME added lubricant, the broad feature centered around 1715 – 1730 cm^{-1} wave numbers was due to the presence of carbonyl-containing degradation products of the lubricant. This has been identified as ketones or aldehydes [8]. Ketones, although found in straight chain lengths up to 11 carbon atoms, are having low polarity and as such do not absorb readily onto metallic surfaces. The spectrums increase around 1608 wave number was associated with the presence of long chain alcohols, amides, conjugated carbonyl species [9] which are formed through oxidation of the lubricants and known as surfactants. This is because of the presence of stearic acid in POME composition, which acts as a surfactant, and readily adsorb between the mating surfaces. Therefore, it could be concluded that lower percentage of POME (preferably 5%) can improve the performance of mineral-based oil by forming protective films either by physisorbtion or by adsorption onto the mating surfaces.

4. CONCLUSIONS

Following conclusions can be drawn from the present study:

FT-IR quantitative data indicate an increased in oxidation products which was formed from 10% POME added lubricants after 280 hrs of oxidation test. The 5% POME added lubricant and mineral-based lubricant (without POME) showed less oxidation product after the test. From the FT-IR spectrum analysis of the oxidized oils it could be concluded that 5% POME can improve the performance of mineral-based oil by forming protective films either by physisorbtion or by adsorption onto the mating surfaces. For lubricant, the FT-IR method can be used which requires a small amount of sample. The method is very simple and it gives reliable results.

5. ACKNOWLEDGMENT

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Table 1

FT-IR test results of degraded oil after static oxidation test. Table shows the band position (wave number) of the peak for all spectrums.

Sample Oil	Frequency/Wave number (cm^{-1})					
	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
0% POME	3190 (0.476)	2732 (0.54)	1732 (0.472)	1605 (0.427)	1163 (0.783)	829 (0.453)
5% POME	3175 (0.361)	2730 (0.538)	1713 (0.274)	1608 (0.397)	1158 (0.482)	825 (0.413)
10% POME	3180 (0.456)	2730 (0.608)	1715 (1.453)	1603 (0.67)	1173 (1.122)	819 (0.532)

Note: Parenthesis () indicates percentage IR absorbance at different peaks.