Estimation and Correlation Developed for Viscosity of Lubricating Oil Using Fourier Transform Infrared Spectroscopy

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Abstract: Proper lubrication in the machine is very important. Without proper lubrication in the machine reduces the performance and life of machine. The working of machine in good condition depend on the quality of lubricants and condition of machine. The lubricating properties of lubricants decide the time interval of lubrication. Hence rather than the conventional way of changing the lubricant at the fixed interval, it is recommended that the oil be changed based on their lubricating properties. Viscosity is a very important properties which affect the useful life of the oil. Viscosity decides the load bearing capacity of the oil film. With time the viscosity of the oil may both increases as well decreases depending upon the conditions. Tom (2007) described how FTIR can be used for oil analysis and suggested the selection of correct parameters for specific applications. The present paper proposes to use Fourier Transform Infrared (FTIR) spectroscopy to find the viscosity of the oil. In this present paper, the trends in viscosity with the transmittance of selected peak will be found out and the equations of the trend line will give the viscosity at any particular value of transmittance of FTIR spectra.

Keywords: Viscosity, FTIR, Lubricant, Transmittance

1. Introduction

Lubricants lose their properties with the use. Once the lubricants are stripped off their lubricating properties, they are drained off and new oil is poured in the machine. The time interval between pouring in of fresh oil and draining out the used oil is the useful life of the lubricant. Traditionally when the used oil will be poured out and fresh oil will be poured in is a fixed interval of time or fixed hours of running of the machine. This time interval is either decided by the oil supplier or by the original manufacturer of the equipment.

This should not be the recommended technique. The oil is subjected to different rate of deterioration for different conditions of the machines and also the working conditions into which the machine has been put. So the useful life of lubricant will be different conditions of machine and different work environment. There are two disadvantages of oil change at conventional fixed time interval:

- If the condition of machine is good and it is working in a good condition, there is always a possibility that the oil when it is being changed may be still having some useful life left with the lubricant. This results in loss of lubricant.
- If machine is old, not maintained properly and are working in a rugged environment, then the oil on being subjected to rough conditions, may lose its lubricating properties before the time interval decided for the oil change. This may result in under lubrication of the equipment. It is detrimental for the machine.

Hence rather than the conventional way of changing the lubricant at the fixed interval, it is recommended that oil be changed based on their lubricating properties.

The properties of oil at a pre-decided fixed interval be analyzed and when the properties fall down below a certain level then the oil will be changed. Viscosity, Viscosity Index, Total Acid Number(TAN), Total Base number(TBN), flash point, pour point, water content, total undisolved contamination etc. are some of the properties of oil which may be used for this purpose. Viscosity isa very important properties of the lubricants which affect the useful life of the oil. It decides the load bearing capacity of the oil film. Higher the viscosity more is the load bearing capacity of the oil film. But, higher viscosity causes flow related problems. So the viscosity of oil being used must be within an optimum level. With time the viscosity of the oil may both increases as well decreases depending upon the conditions.

2. Proposed Method

The present paper proposes to use Fourier Transform Infrared (FTIR) spectroscopy to find the viscosity of the oil. A relationship between percentage transmittance vs wave number could be obtained from FTIR spectra. Based on the available literature prominent peaks would be selected and values of their percentage transmittance at different hours of running will be noted. The correlation between the percent transmittance and the hours of use would be calculated. The peak with highest correlation coefficient will be the main cause of oil deterioration. The trends in viscosity with the transmittance of selected peak will be found out and the equations of the trend line will give the viscosity at any particular value of transmittance of FTIR spectra.

3. Plan of work
The FTIR Spectra of the oil samples were recorded on Perkin Elmer FT-IR Spectrum 2000. (Figure-10). In each case the oil sample without any treatment was spread between the slide of KBr (6 mm thick) and the reflectance spectrum was recorded. There are many other methods for FTIR Spectroscopy analysis, but in those methods the oil samples are processed / filtered before analysis. Spectrum reflectance method was applied because in this method pre-processing of oil sample is not required. Thus more accurate results are obtained indicating the state of oil at its operating condition. Percentage transmittance of the engine oil was measured using FTIR Spectrometer.

4. Experimental Set-Up

The viscosity of engine and gear oil was measured in centipoises (cP) at ambient temperature using SV-10 Vibro Viscometer (Make: A & D India). The sample or samples to be examined should be in the viscosity range of 0.3-10,000 cP. This viscometer works on the principle of tuning-fork vibration method to measure viscosity. Vibro Viscometer has a unit to detect viscosity of a sample, which is composed of two thin sensor plates that vibrate as shown above. It drives the sensor plates to vibrate at uniform Sine-Wave vibration in reverse phase, like a tuning-fork. The density of the oil was measured using specific gravity bottles and chemical balance. Kinematic viscosity is commonly used to represent the viscosity of lubricating oils. It was derived by dividing the viscometer reading twice by density.

5. Result and Discussion

Most of the literature on oil analysis was available on analysis of engine oil. The present work engine oil from a Dumper used in the open cast coal mine was collected. Details of the dumper and the engine oil are as follows:

- Engine make: Caterpillar
- Capacity: 100 Tonne
- Type: Diesel, four stroke, turbo charged after cooled
- Gross power: 1000 HP
- Net power: 938 HP
- Bore: 17.02 cm
- Engine oil Type: CH4 15W40
- Engine oil make: Mak
- Oil capacity: 125 litres

Values of kinematic viscosity for different samples have been shown in Table -3.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Sample</th>
<th>Hours of running</th>
<th>Kinematic Viscosity (c.St)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fresh</td>
<td>0</td>
<td>80</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>100</td>
<td>109</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>196</td>
<td>86</td>
</tr>
<tr>
<td>4</td>
<td>3</td>
<td>250</td>
<td>88</td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td>270</td>
<td>86</td>
</tr>
</tbody>
</table>

The value of kinematic viscosity against time has been plotted and shown in the graph -1.
The viscosity of oil initially increased followed by a drop. Then the variation in viscosity of oil was small.

**FTIR**

Fourier Transform Infrared (FTIR) spectrum of each of the oil samples have been given in, from graph-2 to graph-6. The superimposed spectrum of all the samples including fresh oil has been shown in graph-7.
These spectra are the relationship between percent transmittance vs wave number (cm\(^{-1}\)). The wave number of prominent peaks in the spectra were selected on the basis of available literature (Mukherjee et al., 2000; Kumar et al., 2005). Values of percentage transmittance at corresponding hours of running for these selected peaks were tabulated and have been shown in Table -2.

### Table 2: Percent transmittance

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Wave No.</th>
<th>Inference</th>
<th>Percent transmittance at different hours of running</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>O hrs.</td>
</tr>
<tr>
<td>1</td>
<td>725</td>
<td>Sulphonic acid group</td>
<td>Fresh</td>
</tr>
<tr>
<td>2</td>
<td>1379</td>
<td>(\text{S=O})</td>
<td>61.778</td>
</tr>
<tr>
<td>3</td>
<td>1469</td>
<td>(\text{N=O and CH}_2) bending</td>
<td>49.239</td>
</tr>
<tr>
<td>4</td>
<td>1620</td>
<td>Amides and nitro compounds</td>
<td>80.932</td>
</tr>
<tr>
<td>5</td>
<td>1750</td>
<td>Oxidation products, carbonyl region</td>
<td>84.167</td>
</tr>
<tr>
<td>6</td>
<td>2856</td>
<td>(\text{OH and CH}_3) stretching</td>
<td>21.47</td>
</tr>
<tr>
<td>7</td>
<td>3500</td>
<td>Water</td>
<td>82.399</td>
</tr>
</tbody>
</table>

**Graph 5:** FTIR spectra of sample 3  
**Graph 6:** FTIR spectra of sample 4  
**Graph 7:** Superimposed FTIR spectra of all samples
The values of viscosity at different hours of running were taken from Table 1 and transmittance values from Table 2. The correlation coefficient of viscosity with the peak values of the transmittance at different wave numbers were calculated using MS Excel. The values are given in the Table No. 3.

Table 3: Correlation coefficient with viscosity

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Wave No</th>
<th>Inference</th>
<th>Correlation coefficient with viscosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>725</td>
<td>Sulphonic acid group</td>
<td>0.879</td>
</tr>
<tr>
<td>2</td>
<td>1379</td>
<td>S=O</td>
<td>0.904</td>
</tr>
<tr>
<td>3</td>
<td>1469</td>
<td>N=O and CH₂ bending</td>
<td>0.842</td>
</tr>
<tr>
<td>4</td>
<td>1620</td>
<td>Amides and nitro compounds</td>
<td>0.764</td>
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<td>5</td>
<td>1750</td>
<td>Oxidation products, carbonyl region</td>
<td>0.984</td>
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<tr>
<td>6</td>
<td>2856</td>
<td>OH and CH₃ stretching</td>
<td>0.37</td>
</tr>
<tr>
<td>7</td>
<td>3500</td>
<td>Water</td>
<td>0.903</td>
</tr>
</tbody>
</table>

Table 3 shows that viscosity of the lubricating oil at different hours of running correlates well with the respective transmittance. Except one at the wave number 2856 (OH and CH₃ stretching) all the peaks have high values of correlation coefficient. The correlation coefficient of viscosity with the transmittance value at wave number 1750 cm⁻¹ is highest. It indicates that the oxidation of the oil was the main cause of change in viscosity of the oil. At elevated temperatures, oil exposed to oxygen from the air, will oxidize (chemically combine with oxygen) to form a variety of compounds. The majority of these are Carbonyl containing compounds (C=O) such as Esters, Ketones and Carboxylic acids. Some of these compounds are dissolved by the oil, or remain suspended owing to dispersive additives in the oil. The net effect of polymerized oxidation is that chemically, the oil becomes acidic causing corrosion; while physically an increase in viscosity occurs.

It can be easily inferred from the above discussion that oxidation of the oil was the main reason behind oil deterioration.

From Table 3, it is observed that changes in the viscosity of the oil can be best correlated with changes in percent transmittance of FTIR at wave number 1750 cm⁻¹.

Graph 8: Viscosity Vs Transmittance at wave number 1750 cm⁻¹

To investigate the above correlations, viscosity of the oil has been plotted against transmittance at 1750 cm⁻¹ in graph 8. In graph 8, a best fit polynomial curve has been derived using MS excel. If, x be the percent transmittance at wave number 1750 cm⁻¹, y be the viscosity of the oil in c.St. Then the viscosity will be given by equation

\[ y = 0.154x^2 - 27.48x + 1298 \]

Where viscosity is in c.St. and x is the percent transmittance of FTIR spectrum at wave number 1750 cm⁻¹.

Determining viscosity conventional methods in the laboratory require a large amount of sample, are time consuming and costly. Using the suggested methods the viscosity can be derived from the FTIR spectra itself. It requires very small amount of sample (a small droplet only) and gives result in seconds.

The above correlation study also suggested that oxidation of the oil was main reason for its deterioration. Correlation between percent transmittance of FTIR of seven prominent

6. Conclusion

Deterioration in the lubricating oil is reflected in the changes in viscosity, decrease in TBN value and structural changes in oil as reflected in FTIR spectrum.

The analysis of correlation of viscosity with percent transmittance of FTIR spectrum suggested that viscosity of the oil could be expressed in terms of transmittance value. The derived equations is:

\[ y = 0.154x^2 - 27.48x + 1298 \]

Determining viscosity conventional methods in the laboratory require a large amount of sample, are time consuming and costly. Using the suggested methods the viscosity can be derived from the FTIR spectra itself. It requires very small amount of sample (a small droplet only) and gives result in seconds.

The above correlation study also suggested that oxidation of the oil was main reason for its deterioration. Correlation between percent transmittance of FTIR of seven prominent
peaks and viscosity gave the highest correlation coefficient for oxidation and nitrization of the oil.

References


