Study of Lubricating Oil Deterioration Using Fourier Transform Infrared Spectroscopy

Pooja Nand[1], Nitish Kumar Sah[2], Manoj Kumar[3]

[1,2]M.Tech Scholar, Mechanical Engineering Department, BIT Sindri, Jharkhand, India
[3]Professor, Mechanical Engineering Department, BIT Sindri, Jharkhand, India

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Abstract: *Lubrication controls the friction and wear between moving surfaces in contact, by the introduction of a friction-reducing film. It can be a Solid, fluid, or plastic substance. All types of lubricants are susceptible to degradation while in use and depending upon the engine oil condition, it should be changed after certain interval of time. Viscosity of lubricating oil is the detrimental property to define the oil characteristics after hours of running of an engine. Analytical methods for the analysis of lubricating oil such measuring viscosity, TAN/TBN takes much time and not so accurate results are obtained, also large amount of lubricating oil is used in this viscosity measurement method. In the present work, Engine oil used was multi grade SAE 15 W 40 from a Generator set provides power to E-1400-16 drilling rig used for land drilling rigs for oil rig applications in Oil & Natural Gas Corporation (ONGC). Fourier-transform infrared (FTIR) spectroscopy is used for the analysis of oil. Viscosity of the lubricants were found by Stabinger Viscometer at 20º C. Correlation between viscosity and percentage transmittance were created to find out which compound is more responsible for the degradation of oil. A trend line equation was generated at a selected peak to know the viscosity value at the corresponding value of percentage transmittance. Advantage of FTIR is, it's running cost is less as no additional chemical are required to get the spectra.*

Keywords: Lubricant, FTIR, Viscosity, Transmittance

1.INTRODUCTION

Lubricants create thin film between moving surface in contact due to which function and wear of engine can be controlled and life of engine is prolonged. Lubricants can be used in any form such as fluid or solid.

Lubricating oil has large number of products which can be classified on the basis of base chemical and additives. Generally lubricating oil are belonging to either mineral based or synthetic. Constituents of petroleum based lubricating oils are 80-90 % of petroleum hydrocarbon and 10-20% of additives. All internal combustion engine is preserved from wear and corrosion because of this petroleum based lubricating oils.

The substance which plays main role in increasing the equipment life and improves the working of lubricating oil is additives, which is used for refinement of the anti-friction, chemical and physical properties of the base oil (minerals, synthetic, vegetable or animal) [1].There are various types of lubricating additives such as friction modifiers which reduces coefficient of friction due to which less fuel is consumed (some of them are graphite, molybdenum disulfide, boron nitride, tungsten disulfide, PTFE).

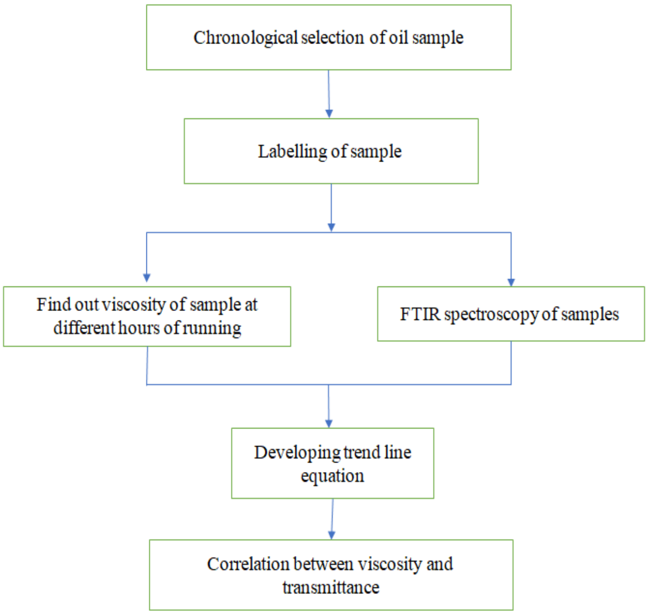
Various properties of lubricating oil are higher viscosity, thermal stability, demulsibility, hydraulic stability, corrosion prevention, high boiling point and low freezing point and high resistance to corrosion [3]. The property of lubricating oil for the center of attention in this research is viscosity. Viscosity is due to inter molecular interaction. This property of lubricating oil is affected by oxidation, nitration and formation of various other compounds which changes the composition of lubricating oil after long hour of running. Due to these changes in composition, the quality of lubricating oil changes[4]. FTIR spectroscopy is used to give the percentage of transmittance for various functional groups at different hours of running. FTIR is widely used in many industries for monitoring degradation, contaminants and additives levels in lubricants of various working lubricating oil. So, we could know that when the oil needs to be changed to prevent the engine from damage and increases its life.

2.PROPOSED METHOD

In the present work, it is proposed to analyses the viscosity of lubricating oil using Fourier Transform Infrared (FTIR) spectroscopy. FTIR spectroscopy of sample gives the relation between percentage transmittance and wave number. The prominent peaks in the graph would be selected on the basis of available research literature. The major cause of oil degradation could be defined from correlation developed between transmittance of running use of the lubricating oil and viscosity. A trend line equation would be generated between viscosity and percentage transmittance value of FTIR spectra.

International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 09 Issue: 06 | June 2022 www.irjet.net p-ISSN: 2395-0072

3.PLAN OF WORK



The FTIR Spectra of the oil samples were recorded on Nicolet iS50 FTIR spectrometer. The reflectance spectrum was recorded when lubricating oil were spread between the slides of KBrwithout any treatment. In this method of spectrum analysis pre-processing of oil sampling is not required. Thus, it can be assumed that obtained value of results is more accurate as the oil was in its operating conditions. Analysis of samples were done using FTIR spectroscopy, percentage transmittance was obtained.

4.EXPERIMENTAL SETUP

In order to calculate the kinematic viscosity, dynamic viscosity and density of the sample must be known. SVM 3000 machine were used for measuring dynamic viscosity and density which is based on a modified Couette principle in which an inner measuring bob rotates very slowly in a rapidly rotating outer tube. Kinematic viscosity (mm2/s) was calculated using dynamic viscosity and density formula. IR-Prestige 21 machine were used for FTIR analysis of lubricants.

5.RESULTS AND DISCUSSIONS

In the present work, sample were collected from Generator set provides power to E-1400-16 drilling rig used for land drilling rigs for oil rig applications in Oil & Natural Gas Corporation (ONGC).

Engine Make: Cummins

Type: 4-stroke, turbocharged or after cooler, V-16-cylinder Diesel engine. Model: KTA50G with capacity of 1430 KVA.

Engine oil used: SAE 15 W 40.

Five samples were collected including fresh lubricating oil.

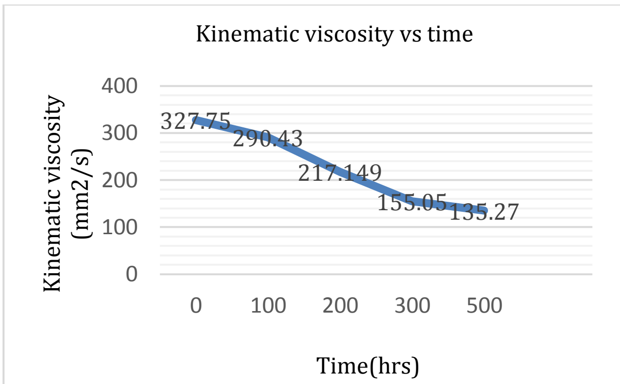
Kinematic viscosity of the different lubricating oil samples has been shown in Table 1.

Table-1: Kinematic Viscosity

Sl.No.	Sample	Hours of running	Kinematic Viscosity(mm^2/sec)
1	Fresh	0	327.75
2	1	100	290.43
3	2	200	217.149
4	3	300	155.05
5	4	500	135.27

The kinematic viscosity of oil shows very small change after 100 hrs. of running then found a major drop after 200 and 300 hrs. of running, not much variation was found between 300 and 500 hrs. of running.

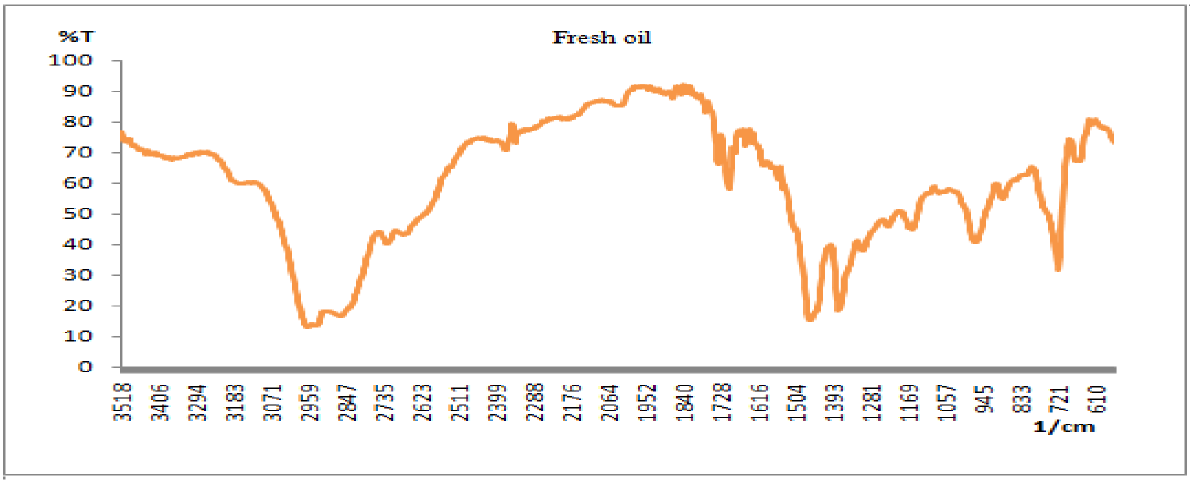
Fig -1: Stabinger Viscometer



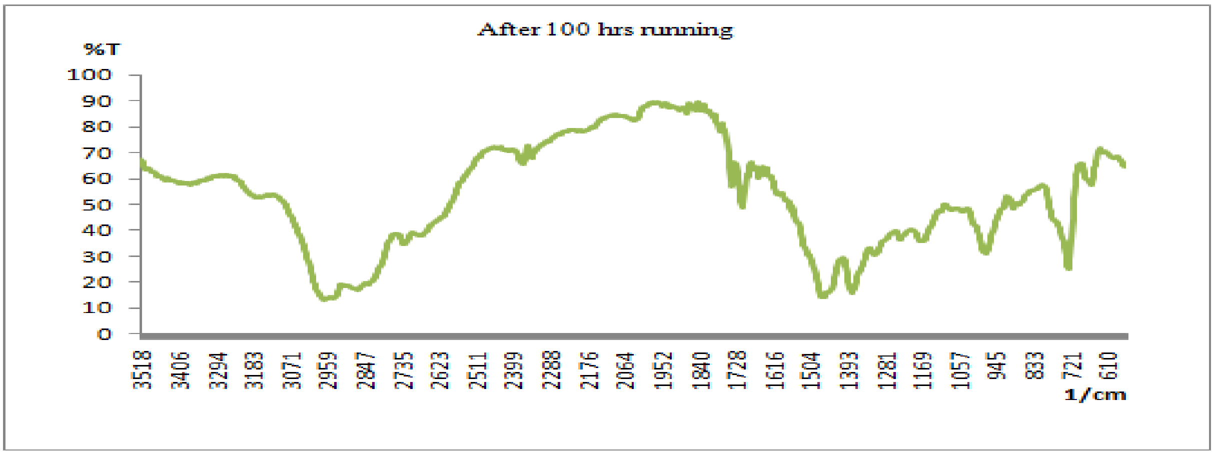
Graph-1: Variation of kinematic viscosity of oil

5.1 FTIR Analysis

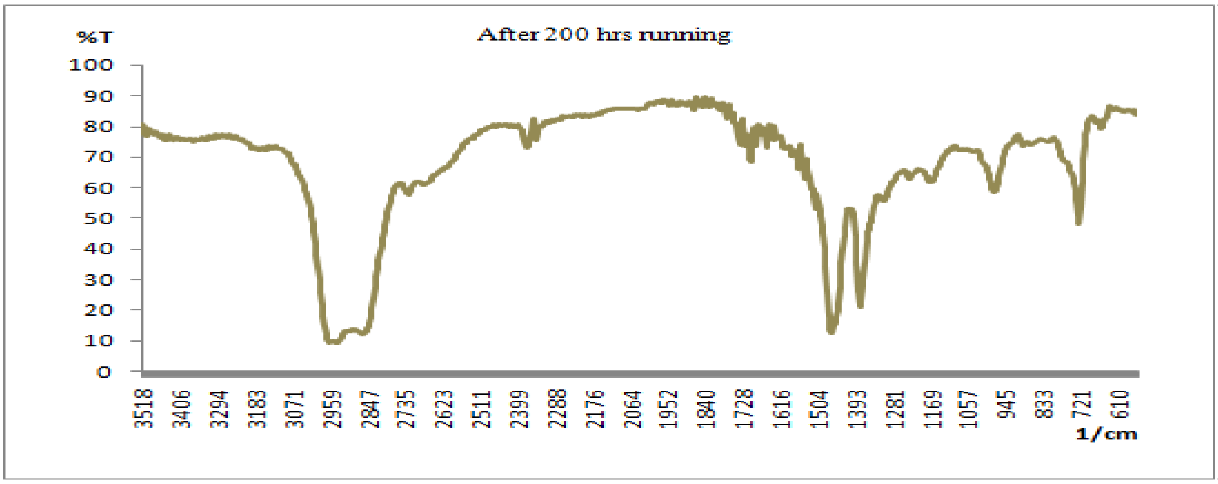
Fourier Transformation Infrared (FTIR) spectrum of each of lubricating oil samples is given in, from graph-2 to graph-7



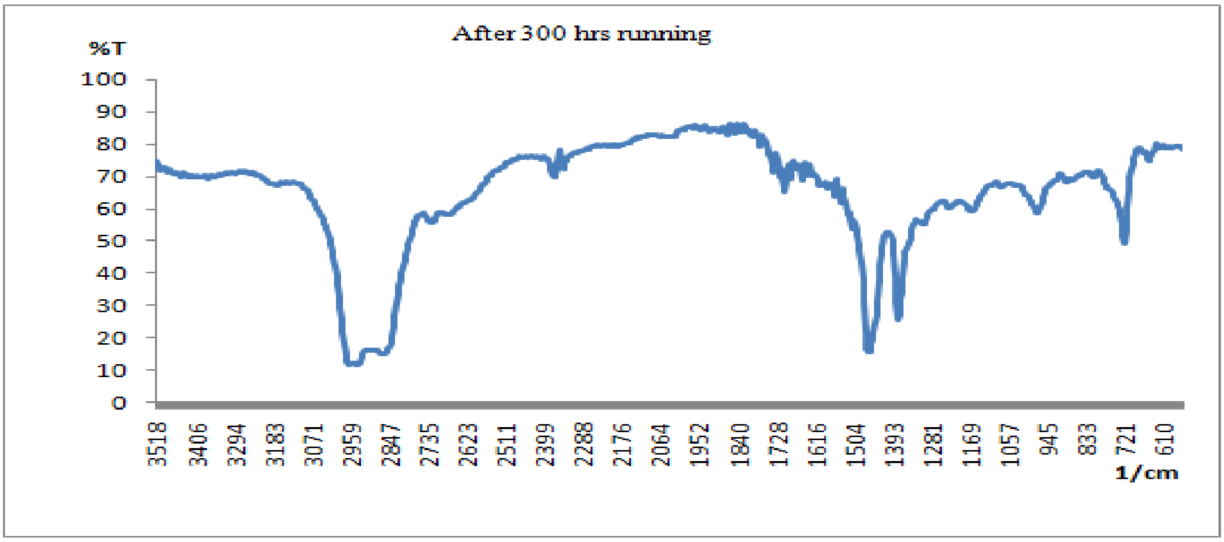
Graph-2: FTIR spectra of fresh oil



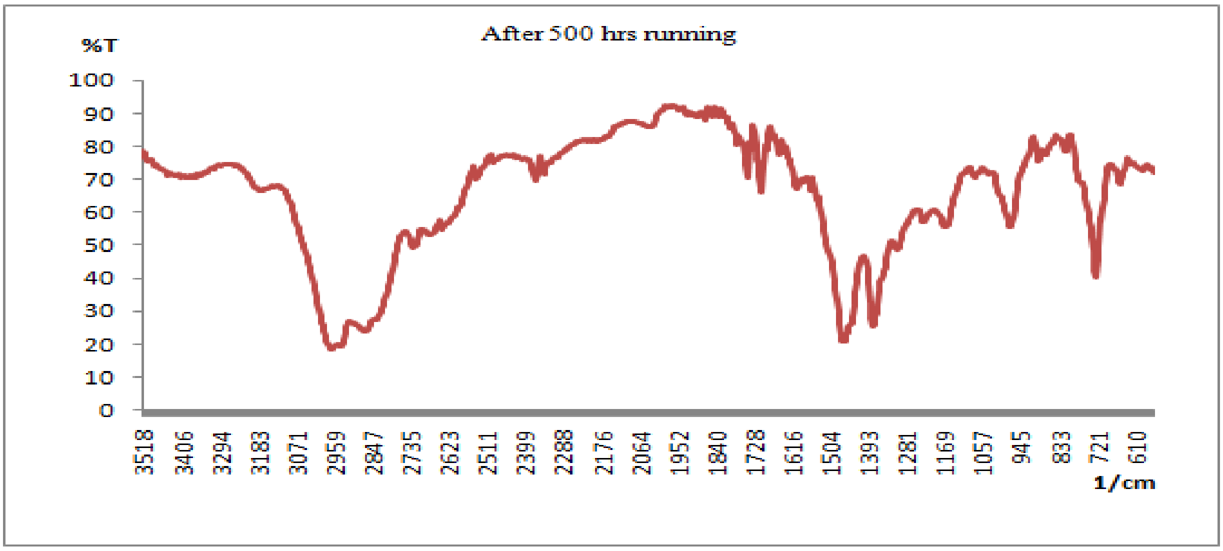
Graph-3: FTIR spectra of oil after 100 hrs. of running



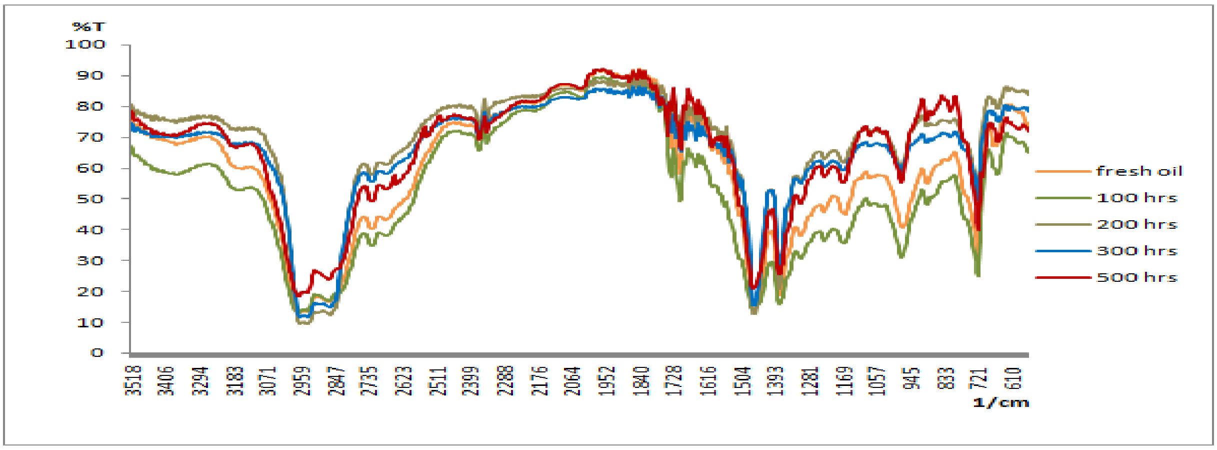
Graph 4: FTIR spectra of oil after 200 hrs of running



Graph 5: FTIR spectra of oil after 300 hrs of running



Graph 6: FTIR spectra of oil after 500 hrs. of running



Graph 7: Merged FTIR spectra of oil

Table -2: Percentage Transmittance

Sl. No.	Wave No.	Inference	Percent transmittance at different hours of running				
			0 Hrs. Fresh	100 Hrs. Sample 1	200 Hrs. Sample 2	300 Hrs. Sample 3	500 Hrs. Sample 4
1	725	C=C bending	33.225	26.352	50.733	51.374	42.261
2	975	Antiwear additives	41.481	31.314	58.710	58.766	55.585
3	1373	S=O stretching	19.013	16.110	23.028	27.109	25.963
4	1460	N=O and CH2 bending	15.428	14.257	12.792	15.513	20.975
5	1700	Oxidation products, Carbonyl region	59.368	49.946	68.275	65.243	66.115
6	2866	OH and CH_3 stretching	16.652	17.024	12.746	14.946	23.768
7	2950	OH and CH_3 stretching	13.736	14.141	10.030	12.045	19.764
8	3350	Water	67.955	58.449	75.570	70.065	71.125

These FTIR spectra of the samples show the relationship between percentage transmittance and wave number (cm^{-1}). The wave number of important and noticeable peaks is taken from a paper by Mukherjee et al., and Kumar et al., 2005.The percentage transmittance at different hours of working for the selected peaks were tabulated and shown in Table-4.

The values of viscosity at different running hours are being taken from table 5.1 and percentage transmittance from table 5.2. The correlation coefficient of viscosity with the prominent peaks of transmittance at different wave number was calculated using Micro Soft Excel. The correlation values are given in Table 5.3.

Table 3: Correlation analysis between viscosity and transmittance

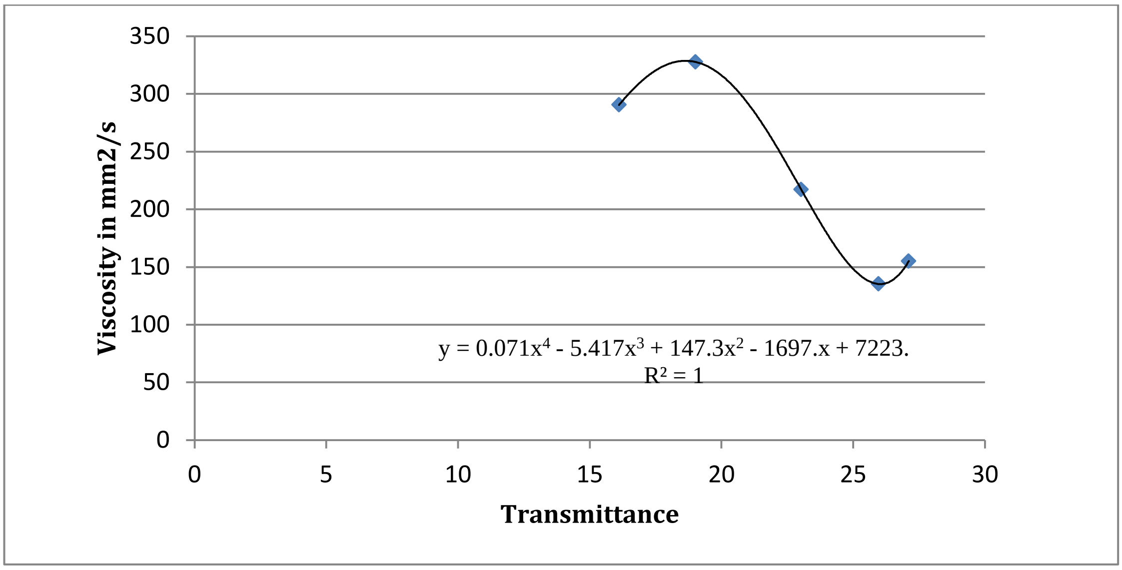
Sl. No.	Wave No.	Inferences	Correlation Coefficient with Viscosity
1	725	C=C bending	0.731
2	975	Antiwear additives	0.801
3	1373	S=O stretching	0.912
4	1460	N=O and CH2 bending	0.541
5	1700	Oxidation products, Carbonyl region	0.690
6	2866	OH and CH_3 stretching	0.336
7	2950	OH and CH_3 stretching	0.302
8	3350	Water	0.526

International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 09 Issue: 06 | June 2022 www.irjet.net p-ISSN: 2395-0072

The above correlation table shows that the Kinematic viscosity of Lubricants has a good correlation with the transmittance at corresponding hours of running. The value of correlation coefficient for most of the peaks is moderate to high. The highest value of coefficient of correlation is 0.912 at the wave number 1373 cm^{-1}, which indicates formation of sulfonate compound [5]. Generally, Sulphur compound added in lubricants acts as an antioxidant which prevent acid and sludge formation. They also form a thin film on the metal surface which protects the surface from acid and peroxide attack. Sulphur contained lubricants are frequently exposed to severe operating conditions results in corrosion and decrease in viscosity of lubricating oil [6].

Another higher peak was found at wave number 725 cm^{-1} and at 975 cm^{-1} indicating reactions with gasoline and with antiwear additives.

From the above table and graph, it can be inferred that presence of (S=O) compound has a detrimental effect on the degradation of selected lubricating oil. However, presence of other compound such as OH and CH_3 stretching does not have significant role on the viscosity of oil after long hours of running. The above correlation was investigated by plotting a graph between viscosity and transmittance at wavenumber 1373cm^{-1} and polynomial curve were derived using Micro Soft Excel.



Graph 8: Relationship between viscosity and transmittance

Let y be the viscosity of lubricating oil in mm^2/s and x be the transmittance percentage at wave number 1373 cm^{-1}. Then the relationship between viscosity and transmittance will be given by

$$y = 0.071x^4 - 5.417x^3 + 147.3x^2 - 1697.x + 7223.$$

Where y be the viscosity of lubricating oil in mm^2/s and x be the transmittance percentage at wave number 1373cm^{-1}.

The conventional methods for finding viscosity require large amount of sample, are costly and time consuming. By the proposed methods viscosity could be derived from the FTIR spectrum in few seconds during which very small amount of sample is required (only a small droplet).

6.CONCLUSION

Deterioration of lubricating oil is due to changes in viscosity whereas FTIR spectrum is due to structural changes in oil. The analysis of correlation of viscosity with percent transmittance of FTIR spectrum shows that viscosity of the oil could be expressed in terms of percentage transmittance value. The derived equation is-

$$y = 0.071x^4 - 5.417x^3 + 147.3x^2 - 1697.x + 7223.$$

The above correlation study also implied that formation of sulfonate compounds in the oil was main reason for its deterioration. Correlation between percent transmittance of FTIR of eight prominent peaks and viscosity gave the highest correlation coefficient for formation of sulfonate compound in oil.

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