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Characterization of Non Soap Thickener based Lubricating Greases by Attenuated Total Reflectance (ATR) Spectroscopic Methodology

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Abstract :

With the advent of newer technological development both in machinery as well as end applications, many greases based on various Thickeners fortified with additives & base oils are available to meet the stringent & extremes of high load and high temperature conditions . Thickeners play very important role for holding the oils and additives in greases and are of two categories - one category is based metal Soap Thickener and second category is Non soap thickeners which are different and some of them are Organo Polyurea , Organo Sulphonate Complex, Fumed Silica , Organo Clay and PTFE. Among the analytical techniques extensively used for characterising lubricating greases are AAS/ICPOES for elemental composition , Soxhlet extraction method for separation of thickener and base oils with additives and characterisation of greases and separated components by Fourier Transform Infrared Spectrometer (FTIR) for chemical composition. All these techniques have been extended to non soap thickener based greases as well.Conventional IR /FTIR technique most commonly used based on IR spectral transmission mode provides information on molecular composition but as a limitation for handling lubricating greases having components such as Non Soap Thickener which reduces optical transmission and limits the characterisation of such lubricating greases. Secondly, variation in repetitive results due to Variation in transmission affects quantitative analysis as surface of cell windows used for sample are effected by moisture in a conventional FTIR analysis . With advent of modern FTIR instrument which combines Transmission Mode as well as Reflectance Accessories such as Attenuated Total Reflectance (ATR), Multi Reflection Technique etc .which overcomes above limitations. In the present study , we have made an attempt to characterise the lubricating greases containing different non soap thickener such as Overbased Sulphonate complex , Polyurea ,Teflon (PTFE), Organic Clay , Organic Silica as such and with solid lubricants such as Graphite and molybdenum disulphide powders by using Horizontal Attenuated Reflectance (HATR) spectroscopy.The effect of consistency of lubricating greases containing non soap thickener , type of greases on the qualitative & quantitative estimation of non soap thickener as well as solid lubricants were studied.. The methodology adopted is rapid with the results obtained are comparable with conventional wet chemical method.

Introduction

With advent of newer technology ,various changes in machinery & equipments were observed in both industrial and automotive applications. This has made significant requirements on various lubricating greases to meet the conventional to stringent, extreme and specialized applications. Wide range of soap and non soap thickeners , base fluids , additives , solid compounds such as solid lubricants are available to the manufacturers of lubricating greases to meet the desired requirements of end applications. Thickeners play very important role for holding the oils and additives in lubricating greases and are of two categories - one category is based metal Soap Thickener and second category is Non soap thickeners which are different and some of them are Organo Polyurea , Organo Sulphonate Complex, Fumed Silica , Organo Clay and PTFE Etc. .Some of the products may contain solid lubricants like Graphite , Molysulphide powder are the predominant materials used which reduces coefficient of friction and wear of rubbing parts preventing direct contact between their surfaces even under high loads conditions.

It is observed that a very few analytical methods are reported in the literature . Limitation of conventional FTIR etc . to characterise the lubricating greases containing non soap thickener without and with containing these solid lubricants. With advent of modern FTIR instrument which can combine Transmission as well as reflectance accessories has helped to handle poor transmission /highly opaque materials . It was felt that multiple reflection spectroscopic technique such as Horizontal Attenuated Total Reflectance (ATR) /FTIR would find useful in characterising the characterise the lubricating greases containing non soap thickener without and with these solid lubricants.. Horizontal Attenuated Total Reflection (ATR) is a sampling technique used in conjunction with infrared spectroscopy which enables samples to be examined directly in the solid or liquid state without furtherpreparation. Light undergoes multiple internal reflections in the crystal of high refractive index, shown in yellow. The sample is in contact with the crystal. ATR uses a property of total internal reflection resulting in an evanescent wave. A beam of infrared light is passed through the HATR crystal in such a way that it reflects at least once off the internal surface in contact with the sample. This reflection forms the evanescent wave which extends into the sample. The penetration depth into the sample is typically between 0.5 and 2 micrometres, with the exact value being determined by the wavelength of light, the angle of incidence and the indices of refraction for the ATR Zinc Selenium 45^0 crystal and the medium being probed. The number of reflections may be varied by varying the angle of incidence. The beam is then collected by a detector as it exits the crystal and IR spectrum is obtained .Most modern infrared spectrometer - FTIR can be converted to characterise samples via ATR by mounting the ATR accessory in the spectrometer's sample compartment. The accessibility of ATR-FTIR has led to substantial use by the scientific community.

In the present study , an attempt has been made to characterise the lubricating greases containing the non soap thickener without and with solid lubricant component such as molybdenum disulphide and graphite powder by using Horizontal Attenuated Reflectance (HATR) spectroscopic methodology. Optical Spectroscopic techniques has limitation of analysinglubricants with dark black / grey color because of poor transmission properties and conventional wet chemical method is time consuming and involves hazardous solvent & organic acid for total solid lubricant content in lubricating greases. These limitations can be overcome by using Horizontal Attenuated Total Reflectance Accessory attached to FTIR instrument . HATR technique has been used to characterise lubricating greases with non soap thickener as well as with solid lubricants.. In the present study, samples of different non soap thickener based lubricating grease were taken along with samples containing solid lubricants such Molybdenum Disulphide (MoS_2) powder & Graphite Powder and Non Soap lubricating grease with mixed graphite & molybdenum disulphide powders were also taken for quantitative estimation of solid lubricants using ATR technique.

The effect of consistency of lubricating greases , type of greases and the quantitative estimation of solid lubricants were also studied. The information obtained from the above study shall be useful in both qualitative and quantitative estimation of solid lubricants in different types of non soap thickener based lubricating greases. The methodology adopted is rapid with the results obtained are comparable .

EXPERIMENTAL

Chemicals : All chemicals employed for analysis were of Analytical Reagent Grade Hexane , Toluene and Acetic Acid, , Anhydrous Sodium Sulphate, Graphite and Molybdenum Disulphide super fine powder forms of Purity 99.9%

Labware : Standard glassware of Borosil make were used for analysis. Beaker 250ml & 500 ml capacity, Glass rods& Filter Papers

Instruments : Thermo Nicolet make Fourier Transform Infrared Spectrometer model iS10 (FTIR) with Horizontal ATRaccessory with Zinc Selenium Trough with angle 45^0.

PROCEDURE :

Samples of Different type of Non Soap lubricating Greases based on Organo Polyurea , Organo Sulphonate Complex, Fumed Silica , Organo Clay and PTFE Solid were taken for study. Initially , All the above samples were subjected to both conventional FTIR system and Multiple Reflection Accessory such as Attenuated Total Reflectance Accessory (ATR) attached with FTIR system and compared . Besides, All these lubricating greases with loaded with solid lubricant –graphite & molybdenum disulphide were subjected to both conventional FTIR & ATR Spectral Analysis and compared . Samples of Non Soap thickener of Lubricating greases with and without solid lubricant of consistency NLGI 2 & 3 were taken for study.

I **Sampling Procedure for Characterization of Non Soap Thickener Lubricating Greases** :

A) Samples of Non Soap Thickener Lubricating Greases under study were analysed by conventional FTIR system usingPotassium Bromide cell windows by spacing samples in between cell windows placed ina demountable cell sample holder
.No Teflon spacer were used.Instrumental condition : Resolution 4cm^{-1}, Scan range : 4000cm-1 to 650cm-1 , Number ofScans : 32.Back ground correction is done before every samples were analysed.

B) Sampling Procedure for Characterization of Non Soap Thickener Lubricating Greases using ATR Accessory . All thesamples under study were loaded one by one as per procedure given below :
- About 2 to 2.5 gm of each of the lubricating greases containing graphite and molybdenum disulphide was spread on the Zinc Selenium Crystal with angle 45 ^{0} ATR trough for recording IR spectrum through ATR accessory attached to FTIR Instrument .Standard Instrumental condition used : Resolution 4 cm^{-1}, Scan range : 4000cm-1 to 650cm-1 , Number of Scans
: 32. Before recording the IR spectrum of each of the samples of lubricating greases , background of blank HATR accessorywith cleaned Zinc Selenium trough was recorded.IR spectrum of sample obtained through background correction. In additionaddition , samples of lubricating greases with and without solid lubricant of different concentration MoS2 \ & Graphite weresubjected to ATR analysis and compared .This will identify qualitatively which type of solid lubricant is used . Before quantitative analysis is done, the consistency of the lubricating greases to be checked and based on which calibration can beselected for quantitative analysis .

Figure 1 and 2 shows Horizontal Attenuated Total Reflectance (HATR) Accessory and Horizontal Zinc Selenium Crystal withangle 45 ^{0} ATR trough.

Figure 1 ATR Accessory

Figure 2 Zinc Selenium Angle 45 ^{0}Trough

II Methodology adopted for Quantitative Analysis of solid lubricant Graphite & MoS2 content in Non Soap thickener containing lubricating greases using HATR accessory attached to FTIR :

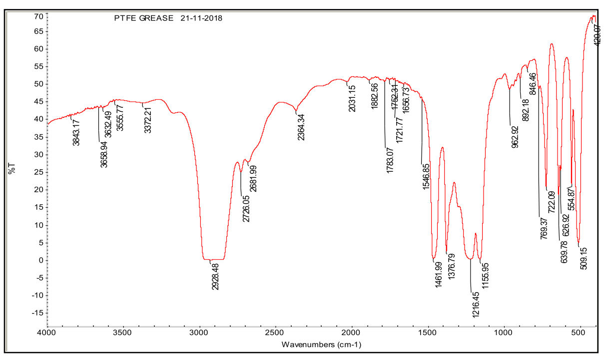
Non Soap thickener based Lubricating grease samples containing only graphite, molybdenum Disulphide only and mixed graphite & Molybdenum disulphide were taken for quantitative estimation of solid lubricant in the lubricating grease . In present study one base non soap thickener lubricating grease was taken to demonstrate the calibration. Sulphonate Complex Grease with consistency NLGI 2 and 3 were taken for calibration of varying amount of solid lubricant in the base Sulphonate Complex Grease Separate calibration was constructed for solid lubricant-Molybdenum Disulphide (MoS_2) with varying amount 0.5%, 1.5% & 3.0% in both the consistency of base lubricating grease NLGI 2 &3 . Similarly, Separate calibration was constructed with solid lubricant –graphite with varying amount of graphite 1.5% , 3.0%, 6.0% & 10.0 in both the consistency of base Sulphonate Complex grease NLGI 2 & 3 . IR spectrum of each of the standard was also recorded by using ATR accessory and measured %Transmittance .
% Transmittance (T) of base Sulphonate Complex grease of both the consistency were also measured as a blank with no solid lubricant .Calibration plot was obtained by difference as %ΔT (%T(Blank Grease – %T Grease with % Solid Lubricant) . IR spectra of unknown lubricating grease samples containing only molybdenum disulphide , only graphite were recorded in a similar manner using ATR accessory and measured their transmittance . In similar manner , Calibration study can be constructed with varyand extended to unknown grease identified qualitatively - the type of lubricating greases of non soap thickener either Poly ureas grease type , Fumed Silica type , Organic Clay type , PTFE type with and without solid lubricants such as molybdenum disulphide and graphite . Unknown content of solid lubricant can be determined from constructing respective calibration with consistency known of non soap lubricating greases.

Results and Discussions :

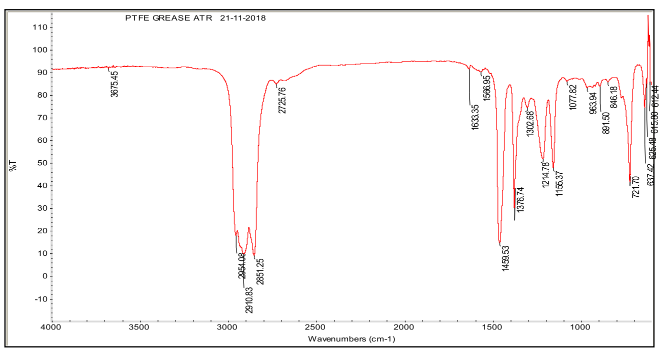
I Characterisation of Non Soap Thickener containing Lubricating Greases by ATR Accessory with FTIR System :

A) A comparison of IR Spectral differences observed IR spectrum taken by conventional recording using KBr cell windows by direct transmission mode and by using rultiple reflectance accessory such as ATR- FTIR System.

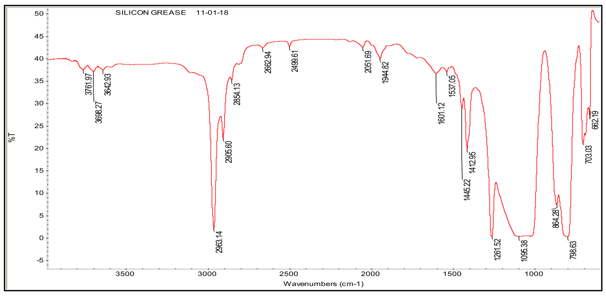
It was observed as shown in **Figure 1** that FTIR spectrum recorded by conventional transmission mode shows noises level high which need automatic smooth processing to reduce noise level. This is due to non soap thickener particulates reflect IRbeams in transmission mode. Loading less and scan speed improves IR spectrum of this greases in conventional transmissionmode. This requires time consuming exercise which is overcome by reflectance accessory such as Attenuated Total Reflectance(ATR) where loading has no impact and noise level is reduced to very minimum. Better IR spectrum obtained when compared with IR spectrum of same sample with conventional transmission mode .



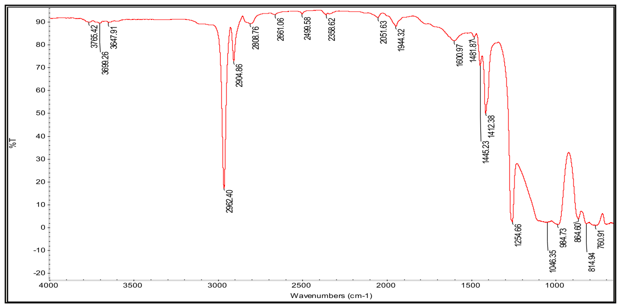
PTFE Grease in conventional FTIR System



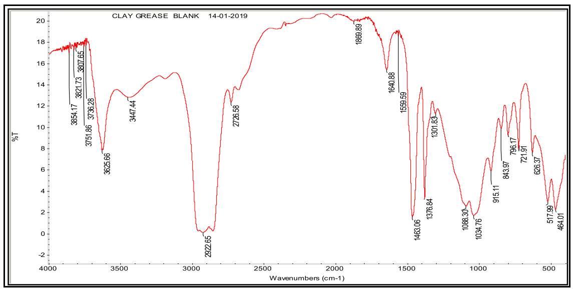
PTFE Grease in ATR- FTIR System



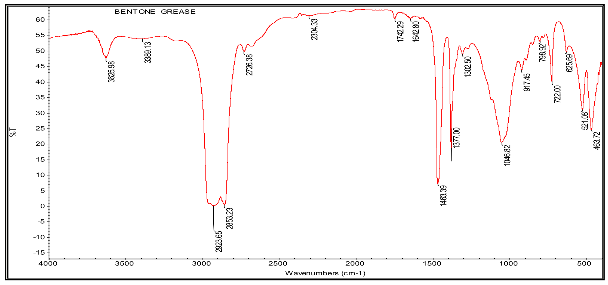
Silicon Grease by conventional FTIR System



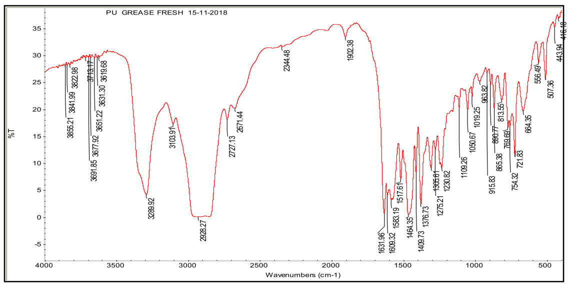
Silicon Grease by ATR - FTIR System



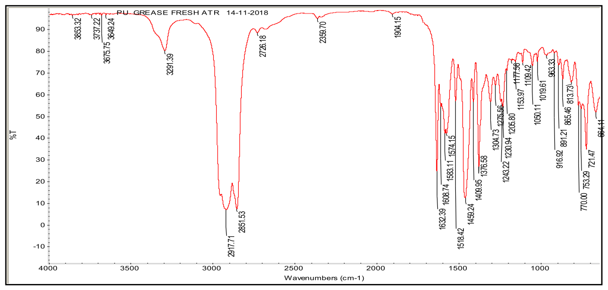
Organic Clay Grease by conventional FTIR System



Organic Clay Grease by ATR - FTIR System

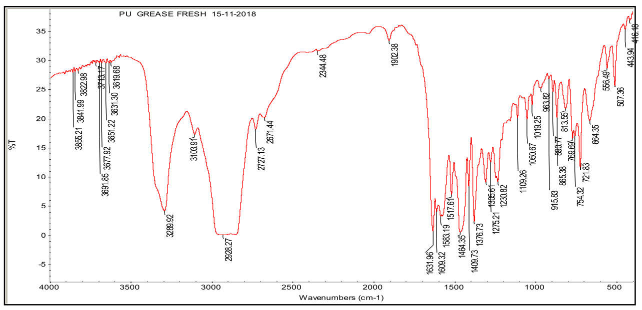


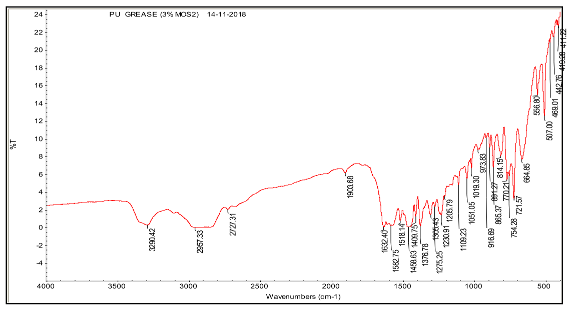
Poly urea grease by conventional FTIR System



Polyurea grease by ATR- FTIR System

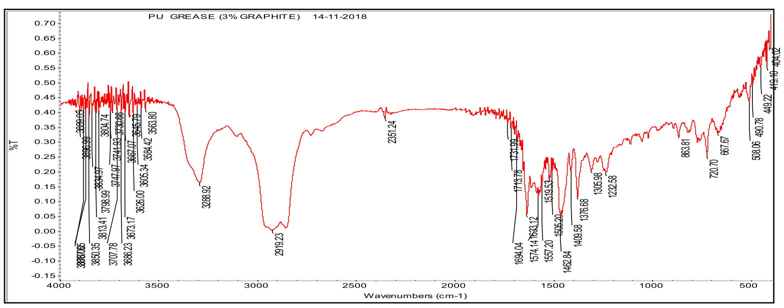
Figure 1shows a comparison of Non Soap Thickener based greases in both Conventional FTIR system and ATR –FTIR system.



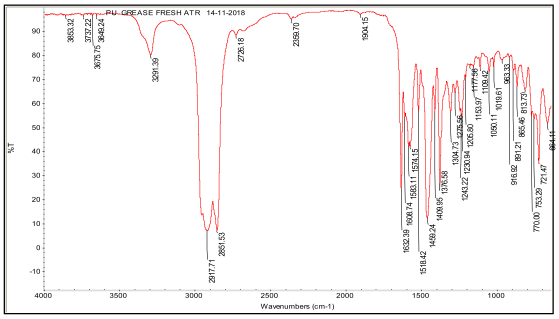


Poly urea grease by conventional FTIR System

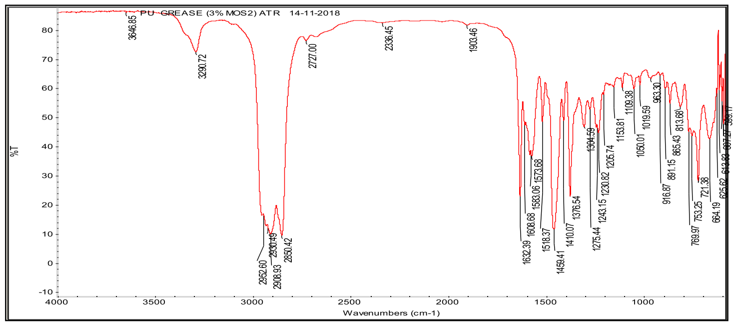
Poly urea grease with SL-MoS_2 by conventional FTIR System



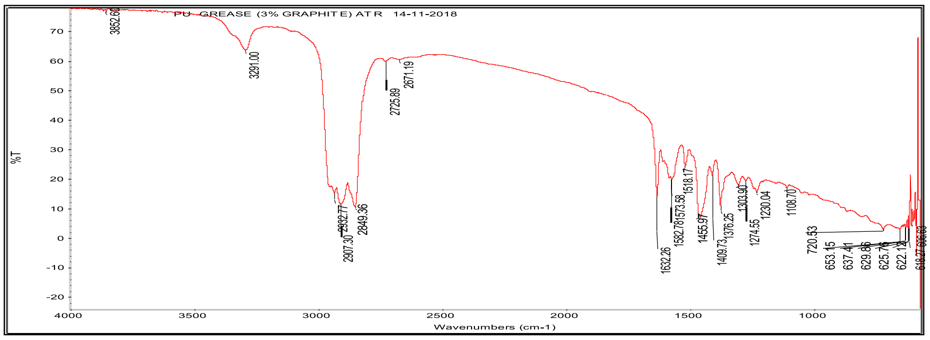
Poly urea grease with SL-Graphite by conventional FTIR System



Poly urea grease by ATR - FTIR System



Poly urea grease with SL-MoS_2 by ATR-FTIR System



Poly urea grease with SL-Graphite by ATR-FTIR System

Figure 2 shows a comparison of Non Soap Thickener based greases (Poly Urea) with Solid Lubricants(SL) in bothConventional FTIR system and ATR –FTIR system.

It was observed from the above **Figure 2** that FTIR spectra of Non Soap Thickener based greases (Poly Urea) recorded by conventional transmission mode using KBr Windows shows noises level very high which need automatic smooth processing to reduce noise level which is time consuming . This is due to non soap thickener particulates and incorporation of solid lubricants which are opaque & dark materials reflect IR beams and brings down %T in transmission mode. Loading less and increasing scan speed does not improve the quality of IR spectrum of this greases in conventional transmission mode. This requires time consuming exercise which is overcome by the use of reflectance accessory such as Attenuated Total Reflectance(ATR) where loading has no impact and noise level is reduced to very minimum. Better IR spectra of samples loaded with solid lubricants such as graphite and molybdenum disulphide ($MoS2$) obtained when compared with IR spectra of same samples loaded in conventional transmission mode .

B) Qualitative method for Characterizing Non Soap Thickener based greases with and without containing solid lubricants -Graphite and Molybdenum Disulphide .

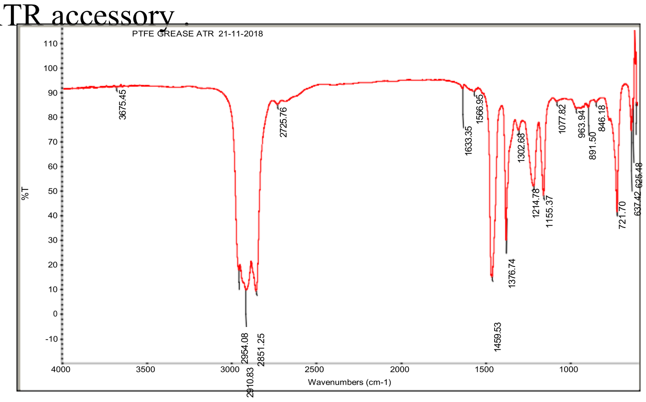
Table 1 shows for qualitative analysis ,from the recorded IR spectrum of Non Soap Thickener containing lubricating greases assuch inand in ATR accessory to identified the type of non soap thickener present in the different lubricating greases through Characteristic Peaks and type of solid lubricant also present in the lubricating greases containing Non Soap thickener.

Table 1

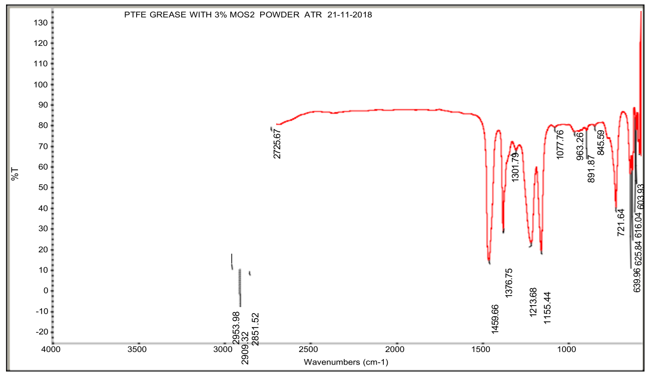
S.No.	Sample Description	Non Soap Thickener Type / Base Oil	Solid Lubricant Type	Characteristics Peaks of Non Soap Thickener in Lubricating Grease By ATR Accessory / FTIR
1	A	Poly urea / Mineral oil	---	**3400-3250cm-1**,**1632cm-1, 1609cm-1, 1573cm-1,1510cm-1**,1110cm-1, 865cm-1 814cm-1, 753cm-1&723cm-1.
2	B	Poly urea / Mineral oil	MoS_2	**3400-3250cm-1**,**1632cm-1, 1609cm-1, 1573cm-1,1510cm-1**,1110cm-1, 865cm-1 814cm-1, 753cm-1&723cm-1**Intensity fall of peaks is very less.**
3	C	Poly urea / Mineral oil	Graphite	**3400-3250cm-1**,**1632cm-1, 1609cm-1, 1573cm-1,1510cm-1**,1110cm-1, 865cm-1 814cm-1, 753cm-1&723cm-1. **Intensity fall of peaks is very high with some of the peaks disappears.**
4.	D	Sulphonate Complex Grease /Mineral Oil	----	**1578cm-1**, broad peak at **1611to 1377**cm-1, 1194cm-1, 1141cm-1, 1058cm-1 **884**cm-1, 668cm-1
5	E	Sulphonate Complex Grease /Mineral Oil	MoS_2	**1578cm-1**, broad peak at **1611to 1377**cm-1, 1194cm-1, 1141cm-1, 1058cm-1 **884**cm-1, 668cm-1. **Intensity fall of peaks is very less**.
6	F	Sulphonate Complex Grease /Mineral Oil	Graphite	**1578cm-1**, broad peak at **1611to 1377**cm-1, 1194cm-1, 1141cm-1, 1058cm-1 **884**cm-1, 668cm-1**.Intensity fall of peaks is drastic due**
				to SL - Graphite
7	G	PTFE / Mineral Oil	-----	**1214cm-1 (–C-F) ,1155cm-1**, 963cm-1, 891cm-1, 846cm-1 , peaks region below 650cm-1
8	H	PTFE /Mineral Oil	MoS_2	**1213cm-1 (–C-F) ,1155cm-1**, 963cm-1, 891cm-1, 846cm-1 , peaks region below 650cm-1 .**There is no fall in base line intensity of the peaks in the presence of SL - MoS2. %T is not much effected.**
9	I	PTFE /Mineral Oil	Graphite	**1213cm-1 (–C-F) ,1155cm-1**, 963cm-1, 891cm-1, 846cm-1 , peaks region below 650cm-1 .**There is distinct fall in intensity of the peaks in the presence of Solid Lubricant Graphite . %T is very much effected.**
10	J	Organic Clay /Mineral Oil	---	**3625cm-1** Sharp peak, , **1046cm-1** (–Si-O)- broad peak overlapped with 917 small Sharp peak and other peaks below 600cm-1.
11	K	Organic Clay /Mineral Oil	MoS_2	**3625cm-1** Sharp peak, , **1046cm-1** (–Si-O)- broad peak overlapped with 917 small Sharp peak and other peaks below 600cm-1. **There is small fall in base line intensity in the presence of SL - MoS2. %T is not much effected.**
12	L	Organic Clay /Mineral Oil	Graphite	**3625cm-1** Sharp peak, , **1046cm-1** (–Si-O)- broad peak overlapped with 917 small Sharp peak and other peaks below 600cm-1. **There isdistinct fall in base line intensity in the presence of SL – Graphite . %T is highly reduced and much effected.**
13	M	Silica Grease / Silicon oil	----	**3700cm-1-3600cm-1** , bifurcated peaks at **2963cm-1& 2905cm-1**, bifuracated peak **1261cm-1&1095cm-1**(strong,broad), bifurcated peaks-**864cm-1 &798cm-1, 703 cm-1.**
14	N	Silica Grease / Silicon oil	MoS_2	**3700cm-1-3600cm-1** , bifurcated peaks at **2963cm-1& 2905cm-1**, bifuracated peak **1261cm-1&1095cm-1**(strong,broad), bifurcated peaks-**864cm-1 &798cm-1, 703 cm-1.%T of base line is not much effected. There is little drop in %T.**
15	O	Silica Grease / Silicon oil	Graphite	**3700cm-1-3600cm-1**, bifurcated peaks at **2963cm-1& 2905cm-1**,bifuracated peak **1261cm-1&1095cm-1**(strong,broad), bifurcated peaks-**864cm-1 &798cm-1, 703 cm-1..%T of base line is very much reduced & much effected. There is substantial drop in %T.**

It was observed from the recorded IR spectra of lubricating grease containing using ATR accessory depends on the type of solid lubricant used MoS2 or graphite .The fall in percentage transmission is more fall in graphite based lubricating grease than MoS2 based grease with similar content of solid lubricant . It is also observed that increase in solid lubricant content , the drop in percentage transmission is more prominent in graphite based lubricating grease than in molybdenum disulphide lubricating grease . With increase in high content of graphite in lubricating greases , IR spectrum shows poor transmission with information on thickener with number of peaks reduces .This information provides dosage level & type of Solid Lubricant used in the unknown sample .Lubricating grease with mixed type containing both graphite and molybdenum sulphide shows similar pattern as that of graphite based lubricating grease .

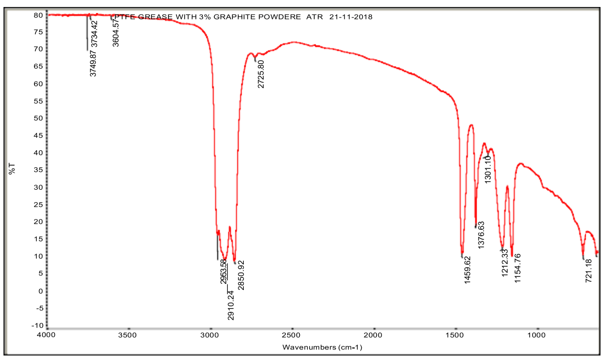
Figure 3 shows IR Spectra of some of lubricating greases with and without solid lubricants recorded by ATR accessory .



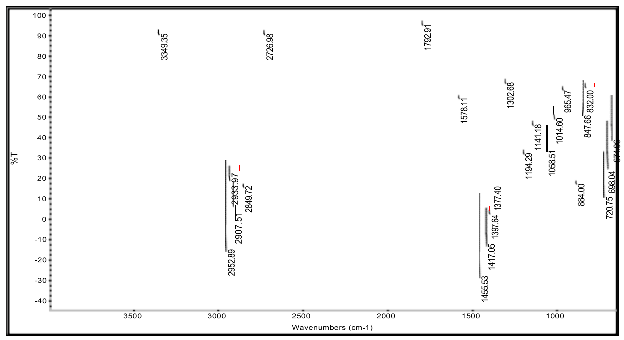
PTFE Grease



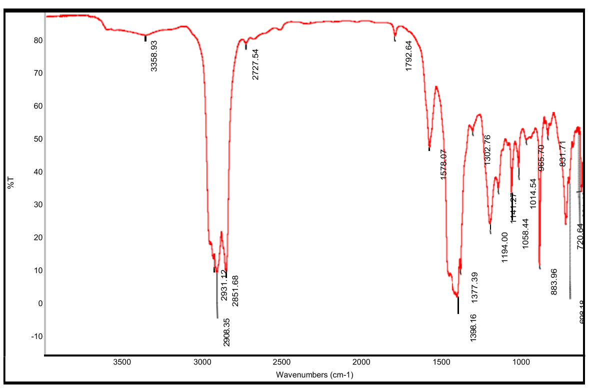
PTFE Grease with MoS2



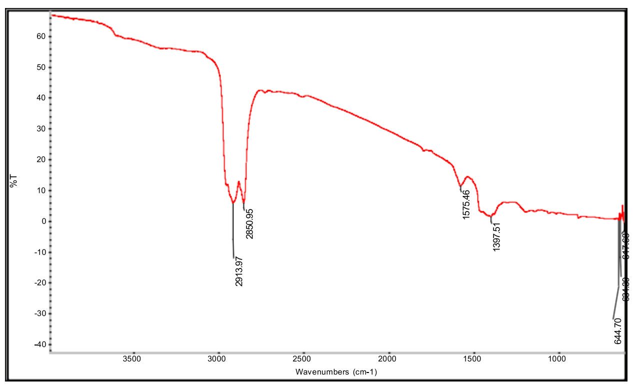
PTFE Grease with SL- Graphite



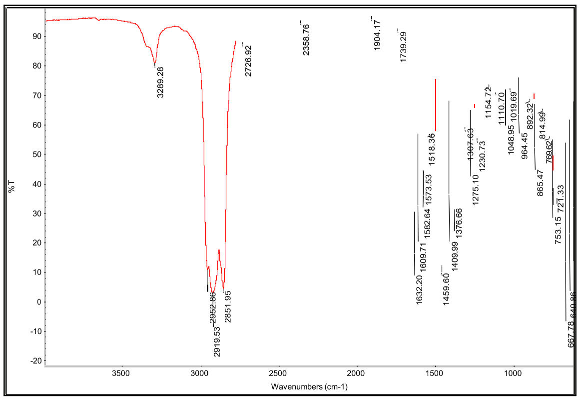
Sulphonate Complex Grease



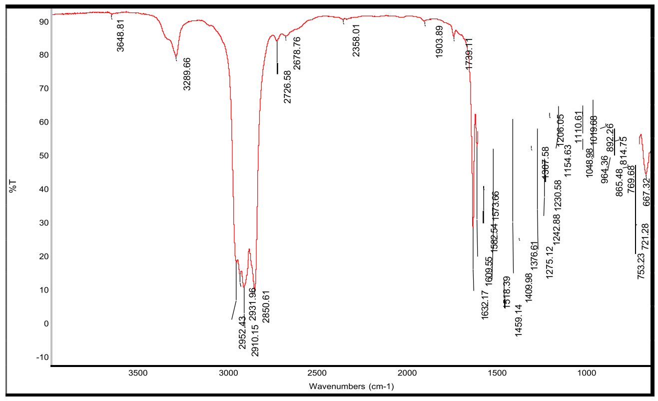
Sulphonate Complex Grease with SL- MoS_2



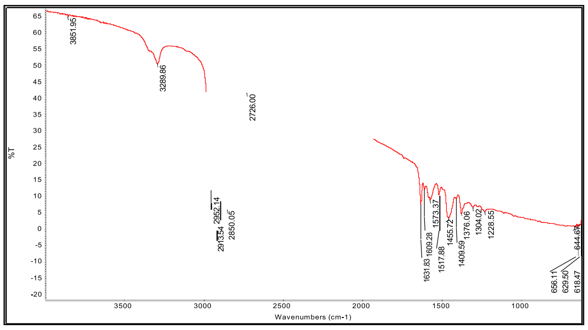
Sulphonate Complex Grease with SL- Graphite



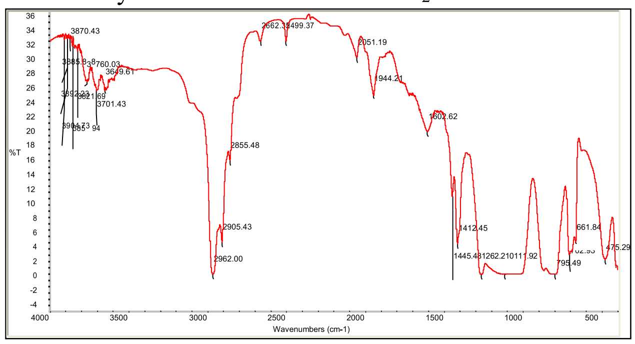
Polyurea Grease



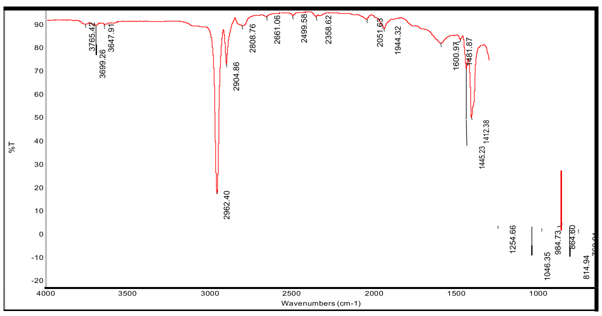
Polyurea Grease with SL- MoS_2



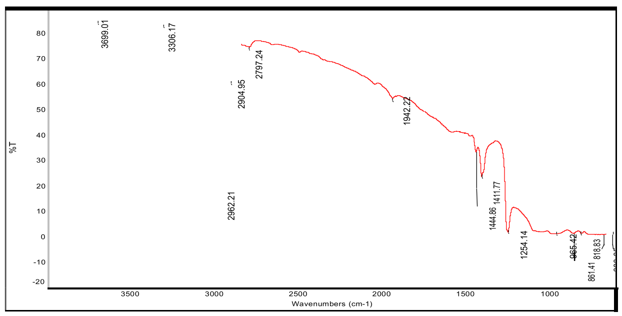
Polyurea Grease with SL- Graphite



Silicon grease



Silicon grease with SL- MoS_2

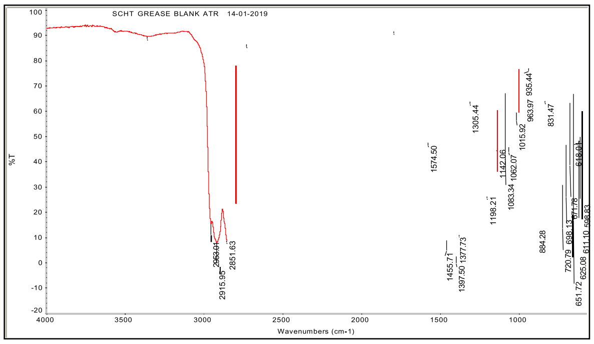


Silicon grease with SL- Graphite

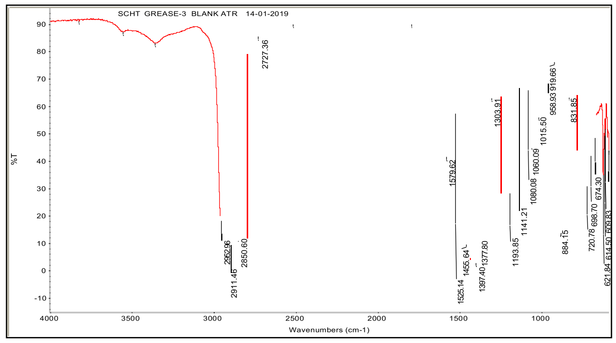
Figure 4 . IR Spectra of Lubricating greases with and without solid lubricants recorded by ATR –FTIR

The effect of consistency of lubricating grease with solid lubricant in the percentage transmission in the IR spectrum recorded in ATR accessory was also studied . It was observed that lubricating grease containing solid lubricant with NLGI 2 consistency shows better % transmittance than lubricating grease containing solid lubricant with NLGI 3 consistency. This is applicable to both the solid lubricants- Graphite and MoS2 containing lubricating greases. This observation was taken into consideration for quantitative estimation of solid lubricants subsequently.

Figure 5.shows Comparative IR Spectra of Non –Soap thickener based lubricating grease with two different consistency of NLGI 2 and 3 .



Sulphonate Complex Grease NLGI -2 (%T 92.64)



Sulphonate Complex Grease NLGI -3 (%T 90.69)

II Methodology adopted for Quantitative Analysis of Solid Lubricant Graphite & MoS2 content in Lubricating Greases containing solid lubricant using ATR accessory attached to FTIR :

Basis on qualitative analysis as mentioned above , from the IR spectral characteristics , the type of non soap thickener based lubricating grease is obtained .If the non soap thickener based lubricating grease contains Solid Lubricants –Graphite or Molysulphide based or mixed type , from the type of IR spectra obtained with base line shift with % T Transmission will indicate which type solid lubricant is present whether it is Graphite based or Molybdenum Disulphide based.

As observed in study, % T obtained from IR spectrum of non soap thickener based lubricating grease containing solid lubricants depended on Consistency of the lubricating grease. Accordingly , Quantitative methodology is adopted by taking consistency of lubricating grease under study into consideration .

The present study is restricted to Sulphonate Complex non soap thickener based lubricating grease of Consistency both NLGI 2 & 3 by constructing a separate calibration plot - **% age difference ΔT (%TFresh -%T with SL)** vs % age Solid Lubricant(SL)
-Molybdenum disulphide & Graphite . A separate calibration plot is obtained for both the consistency and two solid lubricants - MoS2 and Graphite.

Unknown Non Soap thickener based lubricating grease samples containing solid lubricant either MoS2 or Graphite or mixed type
, were subjected to same instrumental condition as that carried out with standards and recorded IR spectrum of the sample. %T was measured from the recorded IR spectrum. Using the calibrations plots , % age solid lubricant content either MoS2 or Graphite contents can be determined .

In a similar methodology can be extended and adopted to study few other types of non soap thickener based lubricating greases such as Organic Clay based , Poly urea based, Silicone based, and Teflon based lubricating greases with and without solid lubricants. The study also cover quantitative estimation of mixed solid lubricants in lubricating greases containing both the solid lubricants with graphite content is more , Molybdenum disulphide can be determined through molybdenum content obtained by AAS/ ICPOES techniques. If the Molybdenum disulphide content is determined to be absent , From the plot of graphite , graphite content in the non soap thickener based lubricating greases is determined. .The results can be compared with standard method for estimation of total solid lubricant by wet chemical method-Toluene Acetic acid method & by AAS/ICPOES methods.

Table 2 shows Sulphonate Complex Grease NLGI 2 with varying amount of Solid Lubricant Molybdenum Disulphide for calibration plot

Table 2

Sl. No.	Sample Description	%MoS_2 in Grease	**%T**	**ΔT (%T_{Fresh} -%$T_{with\ SL}$)**
1	Base Grease Blank	0	92.64	-----
2	Standard 1(Base Grease + MoS_2)	0.5	91.74	0.90
3	Standard 2(Base Grease + MoS_2)	1.5	90.23	2.41
4	Standard 3(Base Grease + MoS_2)	3.0	85.84	2.24

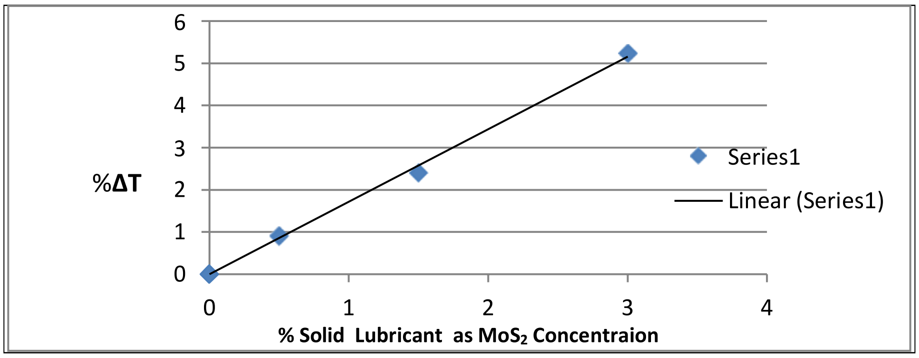


Figure-6 Calibration Plot - with NLGI-2 consistency with varying amount of Solid Lubricant – MoS2

Table 3 shows samples of Lubricating Greases with NLGI 2 Consistency with unknown concentration of Solid Lubricant MoS2

Table 3

Sl. No.	Sample Description	Grease without Solid Lubricant MoS_2(**%$T_{WO\ SL}$**)	Grease with Solid Lubricant MoS_2((**%$T_{with\ SL}$**)	ΔT (**%$T_{WO\ SL}$- %$T_{with\ SL}$**)	**Results (%) ATR Method**	**Results (%) Expected Value**
1	Sample A	92.64	91.58	1.06	0.61	0.5
2	Sample B	92.64	90.63	2.01	1.16	1.0
3	Sample C	92.64	87.24	1.96	1.13	1.0
4	Sample D	92.64	91.24	3.99	2.31	2.0

Table 4 shows Sulphonate Complex Grease with NLGI-3 consistency with varying amount of Solid Lubricant MoS2

Table 4

Sl. No.	Sample Description	%MoS_2	%T	**ΔT (%T_{Fresh} -%$T_{with\ SL}$)**
1	Base Grease (Blank)	0.0	90.69	-----
2	Standard 1(Base Grease + MoS_2)	0.5	89.72	0.97
3	Standard 2(Base Grease + MoS_2)	1.5	88.68	2.01
4	Standard 3(Base Grease + MoS_2)	3.0	86.72	3.97

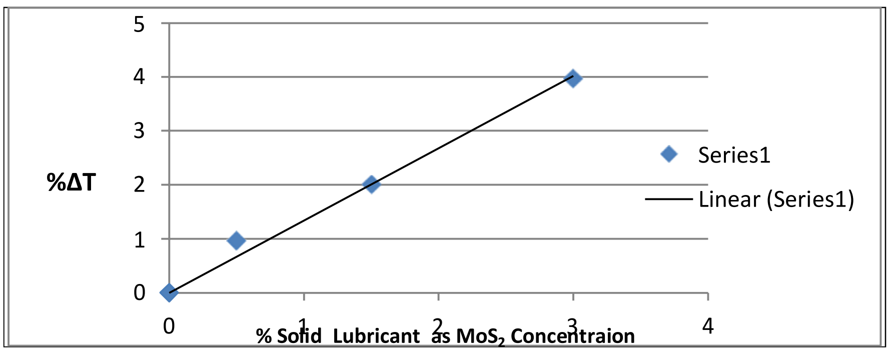


Figure-5 Calibration Plot –Sulphonate Complex Grease with NLGI-3 consistency with varying amount of Solid Lubricant –MoS2

Table 5 shows samples of Lubricating Greases with unknown concentration of Solid Lubricant MoS2

Table 5

Sl. No.	Sample Description	Grease without Solid Lubricant MoS_2(**%T$_{WO\ SL}$**)	Grease with Solid Lubricant MoS_2((**%T$_{with\ SL}$**)	**ΔT (%T$_{WO\ SL}$ - %T $_{with\ SL}$)**	**Results (%) ATR Method**	**Results (%) Expected value**
1	Sample A	90.69	86.60	4.02	3.0	3.0
2	Sample B	90.69	88.71	1.98	1.48	1.50
3	Sample C	90.69	89.67	1.02	0.76	0.75
4	Sample D	90.69	87.78	3.90	2.91	3.0

From the above Table 5 shows the results obtained from lubricating Greases of unknown MoS2 content is close agreement with expected value . Generally ,Lubricating grease with solid lubricant MoS2 contains MoS2 in the range of 0.5% to 3.0%. Accordingly calibration is selected.

Table 6 shows Sulphonate Complex Grease NLGI 2 with varying amount of Solid Lubricant - Graphite for calibration plot

Table 6

Sl. No.	Sample Description	%Graphite in Grease	**%T**	**ΔT (%T$_{Fresh}$ -%T $_{with\ SL}$)**
1	Base Grease (Blank)	0.0	92.64	-----
2	Standard1(Base Grease +Graphite)	1.5	84.37	8.27
3	Standard1(Base Grease +Graphite)	3.0	77.80	15.84
4	Standard1(Base Grease +Graphite)	6.0	61.25	31.39
5	Standard1(Base Grease +Graphite)	6.0	40.98	51.66

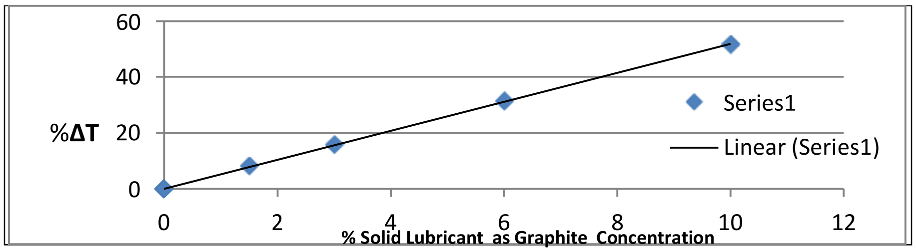


Figure-6 Calibration Plot –Sulphonate Complex Grease with NLGI-2 consistency with varying amount of SolidLubricant –Graphite .

Table 7

Sl. No.	**Sample Description**	**Grease without Solid Lubricant-Graphite (%T$_{WO\ SL}$)**	**Grease with Solid Lubricant - (Graphite) (%T$_{with\ SL}$)**	**ΔT (%T$_{WO\ SL}$-%T $_{with\ SL}$)**	**Results (% Graphite Content) HATR Method**	**Results (% Graphite Content Expected Values**
1	Sample A	92.64	84.01	8.64	1.66	1.75
2	Sample B	92.64	78.12	14.52	2.79	2.75
3	Sample C	92.64	70.82	22.82	4.39	4.50
4	Sample D	92.64	76.92	15.72	3.02	3.00

From the above **Table 7** shows the results obtained from non soap lubricating Greases of Graphite is close agreement with .expected value . Generally ,Lubricating grease with solid lubricant graphite contains Graphite in the range of 0.5% to 10.0%.

Table 8 shows Sulphonate Complex Grease NLGI 3 with varying amount of Solid Lubricant - Graphite forcalibration plot .

Table 8

Sl. No.	**Sample Description**	**%Graphite**	**%T**	**ΔT ($\%T_{Fresh}$ - $\%T_{with\ SL}$)**
1	Base Grease (Blank)	0	90.69	-----
2	Standard1(Base Grease +Graphite)	1.5	85.16	5.53
3	Standard1(Base Grease +Graphite)	3.0	80.47	10.22
4	Standard1(Base Grease +Graphite)	6.0	67.67	23.02
5	Standard1(Base Grease +Graphite)	10.0	56.32	34.37

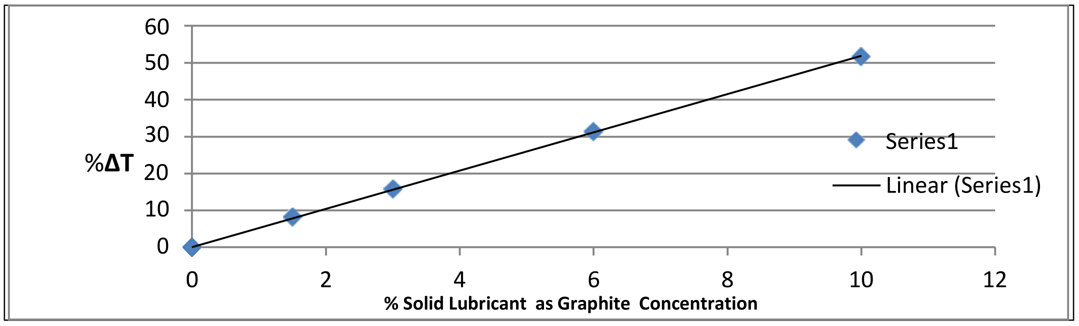


Figure 7 Calibration Plot –Lubricating Grease with NLGI-3 consistency with varying amount of Solid Lubricant – Graphite

Table 9

Sl. No.	Sample Description	Grease without Solid Lubricant Graphite($\%T_{wo\ SL}$)	Grease with Solid Lubricant Graphite (($\%T_{with\ SL}$)	ΔT ($\%T_{wo\ SL}$ - $\%T_{with\ SL}$)	**Results (% Graphite content) HATR Method**	**Results (%) Expected Value**
1	Sample A	90.69	80.32	10.37	2.93	3.0
2	Sample B	90.69	85.10	5.59	1.58	1.5
3	Sample C	90.69	67.42	23.27	6.58	6.5
4	Sample D	90.69	56.48	34.21	9.68	10.0

From the above **Table 9** shows the results obtained from non soap lubricating Greases of Graphite is close agreement with expected value . Generally , Lubricating grease with solid lubricant graphite contains Graphite in the range of 0.5% to 10.0%.

Conclusions :

In the present study, attempt has made to use instrumental technique such as Attenuated Total Reflectance (ATR) accessory attached to FTIR instrument to characterize strongly absorbing non soap thickener based lubricating greases with and without solid lubricants such as Molybdenum disulphide (MoS_2) and Graphite.

The study includes brief comparison of recorded IR spectrum of non soap thickener based lubricating greases obtained with conventional IR /FTIR System vis a vis Multiple Reflectance technique such as ATR technique.

It was observed that ATR is better alternative technique which overcomes the limitation of conventional transmission based IR / FTIR tool using potassium bromide cell windows for characterizing solid lubricant based lubricants. ATR technique is fast and easy with sample preparation requires minimum time and very much reduced noise level.

The study effect of consistency of non soap thickener based lubricating greases NLGI 2 and NLGI 3 has effect on %T of base line of the IR spectrum obtained from ATR system which is very important for constructing a calibration curve for quantitative analysis.

IR spectrum pattern of solid lubricant based lubricating greases depends on the type solid lubricant - MoS2 and graphite The non soap thickener lubricating greases containing solid lubricant shows different IR Spectral pattern with same content of respective solid lubricant MoS2 and Graphite . %Transmittance drop is high in graphite than in Molybdenum disulphide for same amount of solid lubricant in lubricating greases.

For Quantitative analysis of solid lubricant content in lubricating greases , consistency of the Lubricating grease is important parameter for creating calibration plot . Secondly, %T of fresh grease without solid lubricant as a blank along with %T of solid lubricant containing lubricating grease to be measured .The difference %ΔT of % T blank grease & %T with solid lubricant is plotted against known % age amount of solid Lubricant in the lubricating grease keeping in mind consistency of lubricating grease . Separate calibration plot were made for MoS2only and Graphite only . Solid lubricant MoS2or graphite content in different solid lubricant based lubricating greases having unknown content of solid lubricant was determined from the calibration plot of same consistency . The results were found to be in close agreement with expected values which is determined by combination of conventional wet chemical gravimetric Toluene Acetic Acid method and AAS/ ICPOES method .

The developed method is extended to lubricating greases containing both the solid lubricants graphite and MoS2 and results were found to be encouraging by selecting suitable calibration plot and in agreement with expected value. Work is in progress to analyze more market samples of non soap thickener based lubricating grease with solid lubricants with unknown content.

The information obtained from the above analytical study will be useful in characterizing non soap thickener based lubricating grease with and without the solid lubricant with type of soap used and nature of solid lubricant whether MoS2 or graphite or both .This study also used rapidly estimating the solid lubricant content in lubricating greases using ATR with FTIR technique . The method is fast and easily adoptable for laboratory with minimum sample preparation time .

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High Performance Lubricants for Electric Vehicles

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ABSTRACT

Despite limited penetration of Electric Vehicles, the trend towards electrification of road passenger transport looks likely to gain significance over the next decade as more countries & municipalities around the world implement measures to reduce carbon, particulate & other emissions. Recent signing of Paris agreement along with rising airquality concerns in different countries & the diesel emissions scandal has all helped precipitate diesel or fossil fuel restrictions. Many countries will ban all Gasoline & diesel considering electric vehicle targets in years to come.

In tandem with the development of Electric Vehicle, the concept of mobility is being redefined. A key advantage of EVs over ICEs is the ability to generate maximum torque at zero RPM. Conversely, an ICE requires higher revolutions to deliver sufficient rotational force. However, the torque capacity of EVs can cause wear issues& may necessitate torque limitations. Modern vehicles have electric traction control, which already limits wheel slip. These limitations have clear implications for lubricationsuch as range extension & temperature control.

Looking to future, an important consideration is compatibility with new materials, particularly copper & other winding materials. Ensuring the 'structures that hold thesethings in place' remain light weight is vital. Rise in the use of advance polymer may beone of the solution. The scenario would have a strong impact on the demand of light vehicle lubricants because of penetration of BEVs (which do not use engine oil at all)affects lubricant the most.

At the same time, OEMs are divesting from ICEs, adding momentum to the transition,largely because it believes compliance with carbon emission regulations would be difficult to achieve at reasonable cost. These & other similar moves around the world,combined with technology driven cost & performance improvements, mean electric vehicles are likely to gain market share from ICE vehicles in upcoming decade, morequickly than has so far been the case.

When the development of completely electric vehicles intensifies, Engine Oil and otherlubricants product groups will be affected. Whole new demand will be placed on gearoil, coolant & greases partly because they will be in contact with electric modules, sensors, circuits and will be affected by electrical current & electromagnetic fields. Thelubricant must be compatible with everything from copper wares, electric modules to special plastics & insulation materials. This means, they will have to be more specialized to cope with lubrication in these environments. Typical temperature rangesspecified by most vehicles are -40-degree C to +40-degree C, though certain situationstemperatures can exceed these values. Thus, the lubricant should be able to tolerateexcursions beyond temperature limits for a short period.

This paper deals with High performance lubricant suitable for transmission, gear box,wheel bearing & chassis applications of Electric Vehicles for quickly adapting to new demands by OEM. The paper will also give insight about legislations on pollution norms imposed by different countries to get a cleaner picture of likely trend.

Background:

An Electric vehicle (EV) is driven using a battery and an electric motor. While general vehicles use an internal combustion engine and gasoline as fuel, electric vehicles use electrical energy that is charged inside the high voltage battery. As aresult, electric vehicles are eco-friendly in that they do not require fuel and do notemit exhaust gases. As far as Lubricant is concerned, EV requires Gear Transmission fluid and Greases.

Characteristics of Electric Vehicles

1. It is driven using the electrical energy that is charged inside the high voltage battery. This method prevents air pollution since fuel, like gasoline, is not required, negating the emission of exhaust gases.

2. A high performance motor is used in the vehicle as well. Compared to standard, internal combustion engine vehicles, engine noise and vibrations are much more minimal when driving.

3. When decelerating or driving downhill, regenerative braking is utilized to charge the high voltage battery. This minimizes energy loss and increases the distance to empty.

4. When the battery charge is not sufficient, AC charge, DC charge and trickle charge are available.

The vehicle is composed of a high voltage battery that drives the motor and air-conditioner, and an auxiliary battery that drives the lamps, wipers, and audio system.

Main Components of Electric Vehicle

• **On-Board Charger (OBC*)** : A device that charges the high voltage battery by converting AC power of the power grid to DC power.

• **Inverter** : Transforms direct current into alternate current to supply power to the motor, and transforms alternate current into direct current to
charge the high voltage battery.

• **LDC*** : Transforms power from the high voltage battery to low voltage (12 V) to supply power to the vehicle (DC-DC).

• **Motor** : Uses electrical energy stored inside the high voltage battery to drive the vehicle (functions like an engine in a standard vehicle).

• **Reduction gear** : Delivers rotational force of the motor to the tires at appropriate speeds and torque.

• **High voltage battery (lithium-ion polymer)** : Stores and supplies power necessary for the electric vehicle to operate (12 V auxiliary battery provides power to the vehicle features such as lights and wipers).

- (*OBC : On-Board Charger, LDC : Low Voltage DC-DC Converter)

Global Scenario:

The number of electric and plug-in hybrid cars on the world's roads exceeded 3 million in 2017, a 54% increase compared with 2016, according to the latest edition of the International Energy Agency's Global Electric Vehicles Outlook.

China remained by far the largest electric car market in the world, accounting for half sold last year. Nearly 580,000 electric cars were sold in China in 2017, a 72% increase from the previous year. The United States had the second-highest, with about 280,000 cars sold in 2017, up from 160,000 in 2016.

Nordic countries remain leaders in market share. Electric cars accounted for 39% of new car sales in Norway, making it the world leader in electric vehicle (EV) market share. In Iceland, new EV sales were 12% of the total while the share reached 6% in Sweden. Germany and Japan also saw strong growth, with sales more than doubling in both countries from their 2016 levels.

Electric mobility is not limited to cars. In 2017, the stock of electric buses rose to 370,000 from 345,000 in 2016, and electric two-wheelers reached 250 million. The electrification of these modes of transport has been driven almost entirely by China,which accounts for more than 99% of both electric bus and two-wheeler stock, though registrations in Europe and India are also growing.

Charging infrastructure is also keeping pace. In 2017, the number of private chargers at homes and workplaces was estimated at almost 3 million worldwide. In addition, there were about 430,000 publicly accessible chargers worldwide in 2017, a quarter of which were fast chargers. Fast chargers are especially important in densely populated cities and serve an essential role in boosting the appeal of EVs by enablinglong-distance travel.

The growth of EVs has largely been driven by government policy, including public procurement programmes, financial incentives reducing the cost of purchase of EVs, tightened fuel-economy standards and regulations on the emission of local pollutants,low- and zero-emission vehicle mandates and a variety of local measures, such as restrictions on the circulation of vehicles based on their pollutant emission performances.

The rapid uptake of EVs has also been helped by progress made in recent years to improve the performance and reduce the costs of lithium-ion batteries. However, further battery cost reductions and performance improvements are essential to improve the appeal of EVs. These are achievable with a combination of improved chemistries, increased production scale and battery sizes, according to the report. Further improvements are possible with the transition to technologies beyond lithium-ion.

Innovations in battery chemistry will also be needed to maintain growth as there aresupply issues with core elements that make up lithium-ion batteries, such as nickel, lithium and cobalt. The supply of cobalt is particularly subject to risks as almost 60%of the global production of cobalt is currently concentrated in the Democratic Republicof Congo.

Additionally, the capacity to refine and process raw cobalt is highly concentrated,with China controlling 90% of refining capacity. Even accounting for ongoing developments in battery chemistry, cobalt demand for EVs is expected to be between 10 and 25 times higher than current levels by 2030.

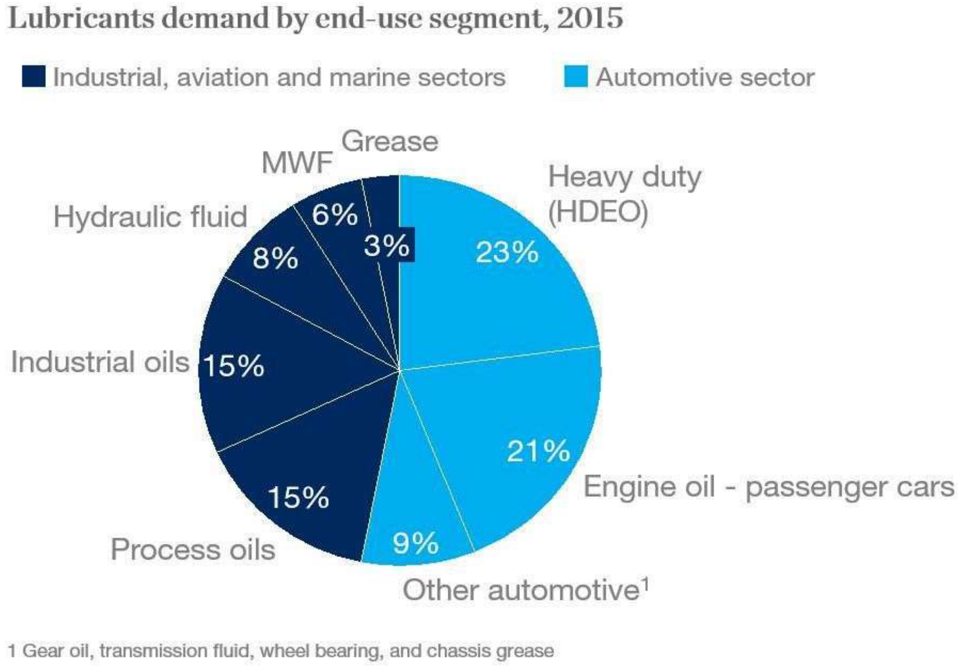
The report notes that ensuring the increased uptake of EVs while meeting social and environmental sustainability goals requires the adoption and enforcement of minimum standards on labour and environmental conditions. The environmental sustainability of batteries also requires the improvement of end-of-life and material recycling processes.

Looking forward, supportive policies and cost reductions are likely to lead to continued significant growth in the EV market. In the IEA's New Policies Scenario, which takes into account current and planned policies, the number of electric cars isprojected to reach 125 million units by 2030. Should policy ambitions rise even furtherto meet climate goals and other sustainability targets, as in the EV30@30 Scenario,the number of electric cars on the road could be as high as 220 million in 2030.

The IEA's latest Tracking Clean Energy Progress report shows that EVs are one ofthe 4 technologies out of 38 that are on track to meet long-term sustainability goals.

Impact of Electric Vehicles on lubricants demand:

In 2015, there were about 1.1 billion light (passenger) vehicles, of which 0.9% were electric, while the remainder were internal combustion engine (ICE) vehicles. In particular, battery electric vehicles (BEV) were 0.1% of the light vehicles, while 0.8%were hybrid electric vehicles (HEV/PHEV).



Despite the limited penetration of electric vehicles in 2015, the trend towards electrification of road passenger transport looks likely to gain significance over the next decade as more countries and municipalities around the world implementmeasures to reduce carbon, particulate, and other emissions. France will ban all gasoline and diesel vehicles by 2040, while Athens, Madrid, and Mexico City have announced plans to ban all diesel cars and vans by 2025. China is also considering electric vehicle targets. Although India is also considering EV as one of the alternatives to curve pollution, it will take some more time to materialise, due to different socio economic reasons.

At the same time, original equipment manufacturers (OEM) are divesting from ICEs, adding momentum to the transition. Volvo, for example, has announced it will no longer develop new diesel engines after 2019, largely because it believes compliancewith carbon emission regulations would be difficult to achieve at reasonable cost.

These and other similar moves around the world, combined with technology-driven cost and performance improvements, mean electric vehicles are likely to gain marketshare from ICE vehicles in the upcoming decade, more quickly than has so far been the case.

In 2015, 52% of world lubricant demand—totalling about 9 million metric tonnes— came from the automotive industry, so any change as we switch to electric vehicles would be significant to the lubricant market as a whole.

In the light vehicle segment, lubricants demand is dominated by engine oil, which accounts for the majority of automotive lubricants demand and for 21% of total lubricants demand in 2015. It is changed every 3,000–10,000 km.

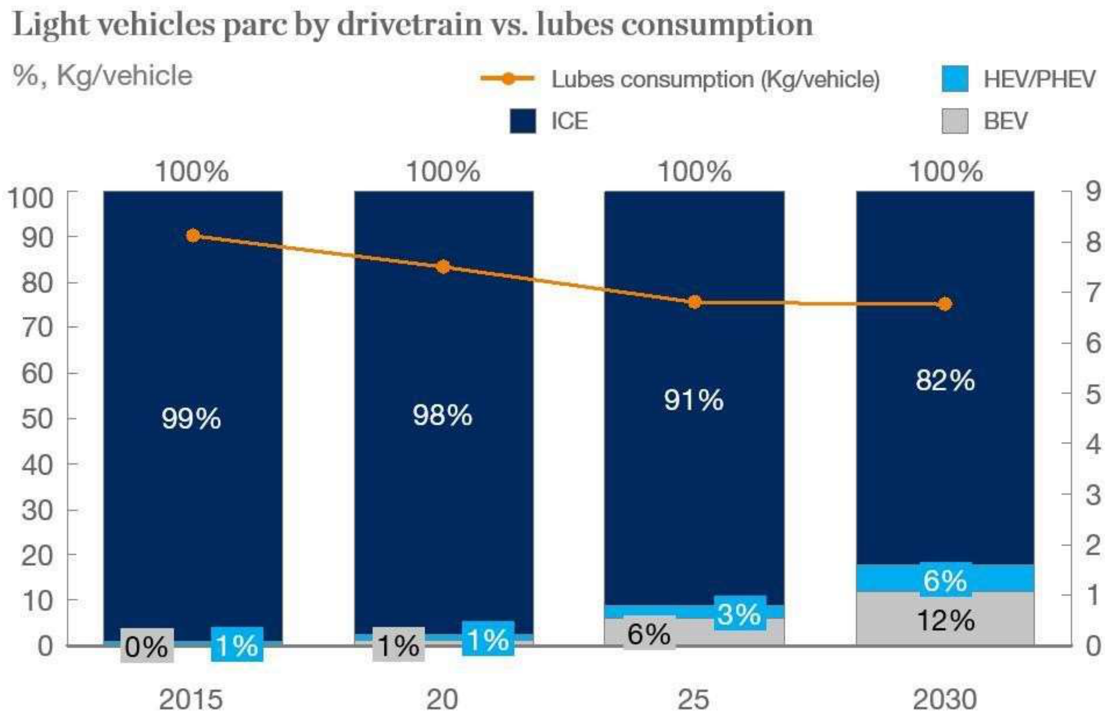
After engine oil, lubricants demand falls into three smaller categories:

- Wheel bearing and chassis grease, which is mostly sealed and changedevery 130,000 KM, if at all.
- Transmission fluid, which is changed every 150,000 KM.
- Gear oil, which is changed every 50,000–60,000 km. At times, only rear axle oil isrequired for EV with Oil life recommendation of 1,20,000 KM or more.

  Since they do not have an ICE, BEVs do not use engine oil, and they use only a small amount of greases and other secondary products. HEV/PHEVs (whichhave both an ICE and powertrain battery) do use engine oil. Compared to conventional ICE vehicles, HEV/PHEVs also require additional higher performance grade lubricants—representing a new and valuable market.

By 2030, we expect that the number of light vehicles will have risen to about 1.6 billion (an increase of 500 million from 2015), with an estimated 18% of the fleet (290 million cars) electric. At that point, only around 5% of the vehicle fleet could be HEV/PHEV, while up to 12% of the vehicle fleet could be BEV—up from 0.1%in 2015 and representing growth of 38% p.a. between 2015 and 2030.

This scenario would have a strong impact on the demand for light vehicle lubricants, because penetration of BEVs (which do not use engine oil at all) affects lubricants the most . Total lubricants demand in 2030, led by Asia, would still grow 1.5% p.a. to about 11 million metric tonnes. But, despite the overall increase, demand declines by around 1% p.a. in Europe and North America, and overall growth is far lower than it would be without the expected EV growth. After 2030, the impact is likely to be more pronounced.



This slowing of growth, and especially the decline in demand in Europe and North America, has some serious implications for lubes companies. In order to avoid shrinking along with the market, they can attempt to maintain growth by expanding market share in Asia and other developing markets; or by focusing on higher margin products such as synthetic lubes and/or high-grade lubes for the growing HEV/PHEV market. Alternatively, players could consider growth through diversification into new or growing related sectors.

The key uncertainty going forward will be the proportion of the two types of electric vehicles, as well as the general rate of uptake. Lubricants companies need to keep a careful eye on proposed legislation and uptake rates over the next five to ten years toget a clearer picture of the likely trends.

Work done by Author's Company for EV Lubricants:

Development of Rear Axle Oil (Transmission Oil/Gear Oil)

EV Gear Oil SAE 75W85 (Gear T Oil A) is being developed with following details:

Sr. No.	Properties	Test Method	Gear T Oil A
1	Density@15 deg C, gm/cm3	ASTM D 4052	>0.8300
2	Appearance	Visual	Bright & Clear
3	KV @ 40 deg °C, cSt	ASTM D 445	Range 60-66
4	KV @ 100 deg °C, cSt	ASTM D 445	<11.75
5	VI	ASTM D 2270	>180
6	Brookfield vis, @-40 deg C, cP	ASTM D 2983	With OEM
7	Pour Point deg C	ASTMD 97	More than (-45)
8	Rust Test	ASTM D 665	Pass
9	Copper Corrosion, 121 Deg C, 3hrs	ASTM D 130	1A
10	Foaming Test , ml/ml	ASTM D 892	With OEM

Oil is non-reactive to copper wires and electrical modules to special plastics and insulationmaterials.

Work done by Author's Company for EV Lubricants:

Development of Grease:

Developed Grease B (Synthetic Grease)

1. **Properties of blended oil used:**

Special Test: Reaction with electric modules, sensors, circuits etc-Nil.

Sr No.	Parameters	Test results
1	Colour (ASTM D 1500)	<3
2	K Viscosity at 40 DegC, cSt	>230
3	K Viscosity at 100 DegC, cSt	<20.0
4	Flash point0C	>260
5	Pour point DegC	<0
6	TAN mg KOH/gm	<0.03
7	Viscosity index	>100
8	Water content, (% by volume)	<0.1

9	Sulphur content (% by mass)	<0.80
10	Sulphated ash (by mass)	<0.01
11	% Paraffins	With OEM
12	% Naphthenes	With OEM
13	% Aromatics	With OEM
14	% Olefins	With OEM

Base Oil as well as Grease is non-reactive to copper wires and electrical modules to specialplastics and insulation materials.

S.NO.	**Parameters**	**Test Results**
1	Soap Base	Li complex
2	Color	Brown
3	Consistency Number	NLGI 2
4	Penetration (1/10 mm) at 25ºC (60 strokes)	Range 265-295
5	Drop point	>280
6	Water Wash Out Test, wt % loss	<3
7	% Oil Separation During storage	<1.5
8	Oxidation Stability , Pressure Drop at 100 hrs (kPa)	>30
9	Rust Preventive Properties	Pass
10	Copper Corrosion Test	1a
11	Wheel Bearing leakage (grams loss)	<2
12	Low temperature torque, in Nm, Starting torque @ -20ºC	With OEM
13	Aging of Nitrile Rubber at 120 ºC for 70 h	
	a) Change in Shore A hardness	With OEM
	b) % Change in Volume	With OEM
14	Aging of Poly-acrylic at 150 ºC for 70 h	
	a) Change in Shore A hardness	With OEM
	b) % Change in Volume	With OEM

Thermal Management method (Cooling) for Electric vehicle

Electric vehicles use large batteries to store energy. The energy flowing into the battery pack as it is charged either from regenerative braking or from the grid and discharged from the pack to power the vehicle and its accessories is measured by electrical current and voltage. The flow of current causes heating in the battery cells and their interconnection systems proportional to the square of the current flowing multiplied by the internal resistance of the cells and the interconnect systems. The higher the current flow, the heating effect will be more.

The performance of Lithium-Ion battery cells is greatly impacted by their temperature, they suffer from the Goldilocks effect, they do not perform well when too cold or too hot, which can lead to permanent and extreme damage of the cells or accelerated degradation. So in addition to cooling, heating of the cells may also be required at lower ambient temperatures to preventdamage during fast charging when the cells are too cold; this is because the internal resistanceof the cells rises when they are cold. Most lithium battery cells cannot be fast charged when they are less than 5 Deg C and cannot be charged at all when they are below 0 Deg C. Lithiumcells also begin to degrade quickly when their temperature is above 45 deg C.

In the past, the largest battery packs did not necessarily need any special cooling as the physical size of the packs was sufficient and the relative flow of current was not large comparedto the overall capacity of the pack. As ever faster battery charging rates are demanded with recharge power of over 200kW to deliver times of 30 minutes or less, higher performance electric vehicles with a requirement for consistent performance and adequate durability in global markets has meant that special thermal management methods for the battery pack arenow required.

There are 3 common battery thermal management methods used today:

- Convection to air either passively or forced.
- Cooling by flooding the battery with a dielectric oil which is then pumped out to a heat exchanger system.
- Cooling by circulation of water based coolant through cooling passages within the battery structure.

Conclusion

With Increase of Electric vehicles, the demand of Lubricants will be impacted heavily. About 52% of total lubricant demands comes from the Automotive industry which is majorly dominatedby Engine Oil. Since the electric vehicles will not require Engine Oil, hence the overall demand of Lubricants will decrease. Also, OEMs are developing oils with high drain intervals which will further reduce the demand of Lubricants in the automotive industry. Only Gear Transmission Oils & Grease will be required for lubrication of Electric Vehicles. Thus, reducing the maintenance cost of the Electric Vehicles.

Lubricant marketers should keep in mind that new oil developed should be in contact with electric modules, sensors and circuits and will be affected by Electrical current and electromagnetic fields. Lubricating Oil and Grease must be compatible with everything from copper wires and electrical modules to special plastics and insulation materials. This means, they will have to be more specialised to cope with Lubrication in these environments. Over greasing may result in higher operating temperatures, premature bearing failure and increasedrisk of contaminant ingression.

Moreover, motors in electric cars also emit a lot of heat, which will need to led away from the electric modules. Hence, effective cooling concepts will be increasingly important. It is also likely that electric motors will be driven at higher and higher speeds in order to increase efficiency. The transition is a major challenge for developers of Lubricants, since it entails a considerable change in lubricant specifications.

Auto Industry's search for cleaner, more fuel conserving technologies is seen as a positive movement. For one thing, the switch to Electric is going to take a long time, so there is a time to prepare.

Source on EV data:

1. Impact of EVs on lubricants demand (Google).
2. IEA Global EV outlook 2018 (google)
3. Hyundai Owner's Manual for EV.
4. IOC R&D

ACKNOWLEDGEMENT

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Tata steel Improve the reliability of Bolster Joint in Hot Metal Logistic through proper Lubrication

Manjunath. S, Ajit Verma,

Manuscript

Torpedoes are used in steel plants to receive hot metal (1500°C) from blast furnace and to deliver it to LD shops. Torpedo consist of two parts - Upper Bolster and Lower Bolster, which is connected through a joint called **Bolster joint** which has two spherical sheets bolted through a pin in which about 400 ton of load (200 ton of dead weight of torpedo and 200 ton of molten metal) is transferred.

During the movement of torpedo on the rail track, certain amount of clearance is given to the spherical sheets in the bolster joint to negotiate the movement of torpedo on the curve path. Lubrication of the spherical sheet plays a vital role as the component is subjected to heavy load along with very high temperature. Wear and tear of the spherical sheets pose major threat if the lubrication in between the spherical sheets gets dried up. Jamming of two halves spherical sheet increases the friction which can result **in shearing of the bolster joint pin and can result in the de-railing of the torpedo.**

Why lubrication is required ?

- Lubrication of the spherical sheet plays a vital role as the component is subjected to heavy load along with very high temperature and reduce the friction between upper and lower bolster joint while turning . Lack of lubrication generate wear particles as well as restrict the movement of the bolster joint causes one of the reason for derailment.
- Due to high temperature and load, lubrication couldn't be done frequently.

Lubrication points have been provided in the spherical sheet but points and pipes gets choked so manual greasing is done at interval of 1 month, by jacking. Grease in between the spherical sheets gets dried up or squeeze out due to high load and temperature. This can lead to wear and tear of spherical sheets, shearing of the bolster joint pin and even De-railing of the torpedo.

Problem statement:

1)Drying and solidification of grease in bolster joint, which created **problem in movement of Torpedo on curve path which could lead to derailment.** The existing grease was having inorganic thickener with mineral base oil 220 cst viscosity and working temperature was up to 160ºc which is not suitable for this application. The NLGI grade of existing grease was 2 which does not have fluidity as per requirement.

2)Manual greasing of bolster joint frequently by **Jacking** as grease pipelines were jammed**.** It was very time taking and also had a lot of **safety** concern.

Objective:

1)To introduce a new grease which would have a very high load carrying capacity as well as a very high oxidation stability and increased re-lubrication interval due to seldom availability of torpedoes for maintenance work.

2)Replacement of jammed grease pipeline with new pipeline to eliminate **Jacking**

Tribological requirement of Bolster joints

- Lubricant should having high base oil viscosity.
- Should have very high static load carrying capacity.
- Should have high adhesion property.
- Should have high oxidation stability.
- Should have high dropping point.

To meet tribological requirement, we have validated the properties of existing grease **:-**

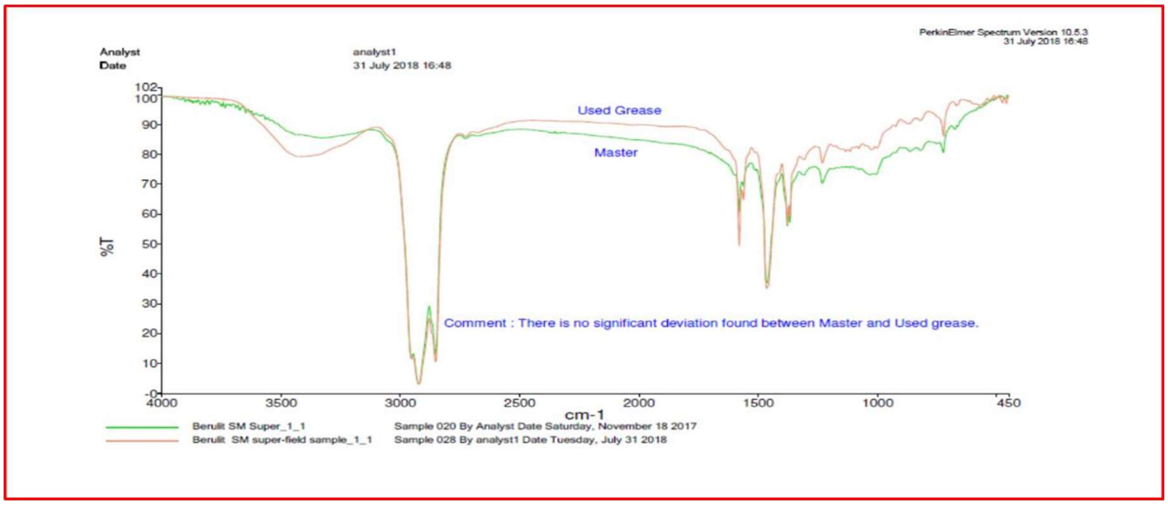
Parameters	Unit	Existing grease	Validation
THICKENER		Inorganic thickner	ok
Type of oil		Mineral	ok
BASE OIL VISCOSITY	cst @ 40 °	220	220 viscosity is not suitable for excessive load. Need grease with high viscosity base oil for this application
CONSISTENCY	NLGI grade	2	NLGI 2 grade is little harder for this application. Need thinner grease for better fluidity for this application
4 BALL WELD LOAD	KG	-	
DROP POINT	°C	None	
working temp. range	°C	up to 160 ° c	Temperature of application area is very high . Not suitable for this application. Require high working temp grease.
Solid Lubricant		Yes	Solid lubricant is ok for high load.

Solution:

Thorough study was done and alternate product was searched for that application which would take the high load as well as sustain in high temperature. we also kept in mind the fluidity of the grease. Proposed grease is semisynthetic base oil viscosity of 2500 cst with 800 kg four ball weld load and working temperature is up to 200ºc. The NLGI grade of proposed grease is 1.

Parameter	Unit	Existing grease	Proposed greases for trial
			Grease A
Thickener		Inorganic thickner	Metal soap complex
Type of oil		Mineral	Semi synthetic
Base oil viscosity	cst @ 40 °	220	2500
Consistency	NLGI grade	2	1
4 Ball Weld Load	KG	-	800
Drop Point	°C	None	-
working temp. range	°C	up to 160 ° c	up to 200 ° c
Solid Lubricant		Yes	Yes

Trial was done and performance was monitored. FTIR was done after 3 months, and result was found ok, also even after 3 months,



Traces of grease was present in pin and spherical sheets. Jammed grease pipe lines were replaced with new pipes and greasing was tried through it and was successful.

Benefits:

1) Successfully eliminated the problem of grease **drying and solidification.**

2) Re-greasing interval increased from 1 month to 3 months

3) problem of jacking was eliminated.

Tata Steel Improve The Reliability Of Re- Coiler Mandrel By Providing Improved Grease:

Ajit Verma & Manjunath.S

Manuscript

There was a chronic problem in Mandrel segment, Grease solidification and wear and tear were observed in mandrel segment in CRM BARA.

Coil wrapping and unwrapping is the main function of the mandrel. Mandrel is consist of set of three segments which expand and collapse through hydraulic cylinder with the help of cam mechanism.

The expand diameter of the mandrel is 610 mm and collapse dia is 585mm. Load carrying capacity of mandrel is approx. 20 to 27 tones. The temperature of the coil is approx. 300 to 400 °c. Due to high temperature of the coil and load, oil was getting separated from the thickener causing improper lubrication, solidification and wear and tear of the segment.

The tribological condition of the mandrel are:

- Wedges subjected to high load and sliding friction.
- Metal to metal contact hence always in Boundary Lubrication regime.
- High temperature in the system hence should have good oxidation stability.
- Long re-lubrication interval reducing man-machine interface.

To fulfil the tribological condition of the mandrel, we have checked the properties of existing grease

The existing grease having lithium soap with 110 cst viscosity mineral base oil was not capable to handle the temp and load. The greasing was done only in shut down by manual process. We were doing greasing every shut down but due to blockage of points, it took time. Cleaning of points was time taking and unsafe due to hot surface of mandrel.

The objective of the project is to address the following observation.

- Wear mark observed on segments
- Solidification of the lubricant
- Jamming of Mandrel
- Frequent greasing required which leads to unsafe activity
- Dripping of oil from grease and affect the product quality

Properties of existing grease:-

Property	Existing grease
Base oil	Mineral
Thickener	Lithium
Viscosity at 40°C	110 cst
Four ball weld Load	180KG
Solid Lubricant	NO
Working temp	120°c

Lithium-Soap	
Temperature range:	… 140 °C
Advantages:	good water resistance up to 90°C good corrosion protection good walk stability readily pumpable
Disadvantages:	no water steam resistance Not suitable for high load application and sliding surface

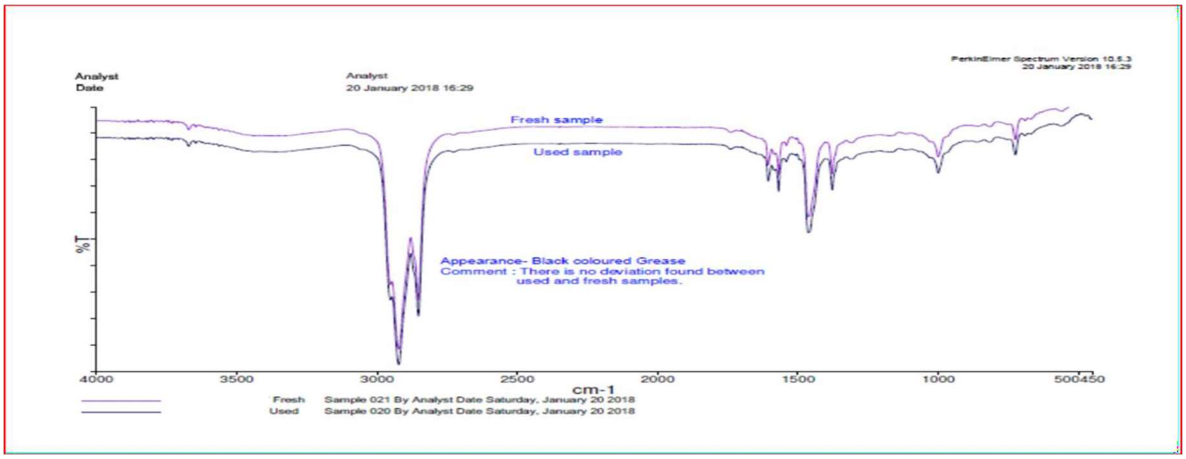
Thorough study was done and alternate product was searched for that application which can take the load as well as sustain in high temperature. We require a grease having high dropping point and good tackiness property. We have also kept in mind that the grease should not react with brass material. Finally, we have selected a grease having 400 cst mineral base oil with Calcium complex soap. Proposed grease also carrying solid lubricant which bear the high load.

Proposed Grease

Property	Proposed grease
Base oil	Mineral
Thickener	Calcium Complex
Viscosity at 40°C	600 cst
Four ball weld Load	600KG
Solid Lubricant	YES
Working temp.	160°c

Calcium-Complexsoap	
Temperature range:	… 150 °C
Advantages:	water and steam resistance very good corrosion protection very good load carrying properties readily pumpable
Disadvantages:	decomposes above 160°C might harden during longer storage time

Trial was started and performance was monitored. Regular FTIR and ferrography was done to monitor the equipment health and it has been found that there is no deviation.



In last shut down, we have checked the mandrel and no wear and tear was observed in mandrel segment. Also there was no grease solidification observed.

Benefit: -

1. Cleaning activity timing has been reduced from 3 hrs. to ½ hrs. Wasteful activity has been eliminated
2. Proper lubrication has been observed. There is No solidification of grease.
3. Grease path jamming issue has been eliminated.
4. Wear and tear in the mandrel segment has reduced.