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Grease For Tyre Curing Press Lubrication

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ABSTRACT
Curing of green tyre is one of the most important processes in the tyre manufacturing industries. Temperature, pressure and moisture are the main criticalities during this process. So far, no satisfactory grease has been reported for the lubrication of the bush bearings of tyre curing press. Tyre manufacturers in India use different lithium or calcium based greases for this purpose. Leakage and excess consumption were the main problems faced by the tyre manufacturers while using these greases. Indian oil"s Research and Development Centre developed a special grease for curing press mould application and the developed grease was field tried at two tyre manufacturing companies. The leakage was found reduced drastically and consumption was reduced to about half after using newly developed grease in both field trials. Details of the development of this grease and its field trials are discussed in this paper.
Key words: Green tyre, curing, leakage, bush bearing

INTRODUCTION
Tyre is an advanced engineered product made of rubber as one of the base ingredient. Fiber, textile and steel cord are some of the components that go into tyre"s inner line, body piles, bead assembly, belts, side walls and tread. Other components like carbon black, silica, sulphur, antioxidants, copper, process oils, cross-linkers etc. are also being used in tyre manufacturing. Tyre manufacturing is a very complex process. In early days, tyres were produced by merely sticking natural rubber plies on to steel frames. Tyre manufacturing mainly involves compounding and mixing, component manufacturing, assembly, curing and finishing steps. These steps are performed by a variety of machines performing mixing, extruding, calendaring, milling and moulding processes. After going through these complex processes, a mixture of these components undergoes the characteristic shape of a tyre through „curing" process" which is performed in a press moulding machine. Grease is being used in the curing press machines for providing adequate lubrication of various moving parts including bush bearings. The grease encounters high pressure, heat and moisture environment during this curing process. In order to tap this niche area of lubricants, a research programme was initiated to develop grease suitable to lubricate bush bearings of „tyre press moulding machine". This paper describes the development, evaluation, rheological studies and field performance of newly developed grease.

EXPERIMENTAL
Since, grease leakage was one of the serious problems faced in the press mould lubrication, a grease sustaining higher dropping point with excellent working stability was the best option for this application. Grease must be able to withstand high temperature, pressure and moisture. So, the formulation was designed in such a way that the grease should take care of all these parameters. Accordingly, small grease batches were prepared in a closed pressure kettle under

controlled experimental conditions such as temperature, pressure etc. Manufacturing process was established by repeated preparation of successful batches both at bench and pilot plant scale. Additives like anti-oxidant, anti-rust, extreme pressure and anti-wear were also incorporated and their respective dosages were optimized. Grease batches were tested as per standard test methods such as ASTM, IP, DIN etc. against a set of specifications framed to develop this grease. Batches meeting the physico-chemical properties were tested for performance properties also. Best performing candidate was selected for the field evaluation studies at two reputed tyre manufacturing units. Typical properties of the developed grease are given in Table 1. The grease was developed in mineral oil with NLGI 1 consistency. It possesses excellent mechanical stability (Penetration change after one lakh double strokes = 12 units). Grease has Dropping point of more than 200°C and minimal oil separation (< 5.0%) even at elevated temperature. Grease is highly water resistant. It passes copper corrosion and emcor corrosion tests. It has relatively low flow pressure suitable for easy pumping, which is a requirement for grease lubrication in curing press. It is having good tribology properties as indicated by four ball weld load (>250 kg) and four ball wear @ 40 kg (<0.60 mm) test results. The relevance of these properties in context to the field performance has been discussed later.

Table-1: Properties of Grease Developed for Tyre Curing Press Application

S. No.	Property	Typical Result
1	Consistency	NLGI Grade 1
2	Working stability, change in penetration after 10^5 strokes	12
3	Dropping Point,° C	> 200
4	Copper Corrosion Test	Pass
5	Base Oil Type	Mineral
6	Heat Stability (oil separation), % wt.	<5
7	Water Washout at 80° C, %wt	<5
8	Behavior of grease in presence of water, rating	1-90
9	Oil separation during storage, %wt	<5
10	Flow pressure at 20°C,hPa	<50
11	Four ball Weld Load, kg	>250
12	Four ball Wear Scar Diameter, mm	<0.60
13	Rust prevention, Emcor test, rating	0,0

The performance of "New Grease" was compared with two in-use greases (Grease — A and Grease — B) at tyre manufacturing plant(s). The typical characteristics of these greases are provided below:

Table-2: Test data of Grease-A

S. No	Property	Result
1	Appearance	Homogeneous
2	Penetration, worked	337
3	Dropping Point° C	93
4	Four ball Weld load, kg	225
5	Four ball Wear Scar Diameter, mm	0.75

Table-3: Test data of Grease-B

S. No	Property	Result
1	Appearance	Homogeneous
2	Penetration, worked	325
3	Dropping Point° C	192
4	Four ball Weld load, kg	250
5	Four ball Wear Scar Diameter, mm	0.65

RHEOLOGICAL STUDIES

Rheological studies of developed grease were carried out at different temperatures and time to compare with the flow behavior of greases used by two different tyre manufacturing companies. The shear rate was kept 30 sec^{-1}. Coaxial cylinder measuring system was used for the study. First apparent viscosity of the grease vs. time at constant shear and temperature was studied. Temperature was kept at 50 ^{0}C. All greases have shown downward trend in viscosity with time. However, New Grease had shown slower decrease in viscosity as compared to remaining two in-use greases (Fig-1).

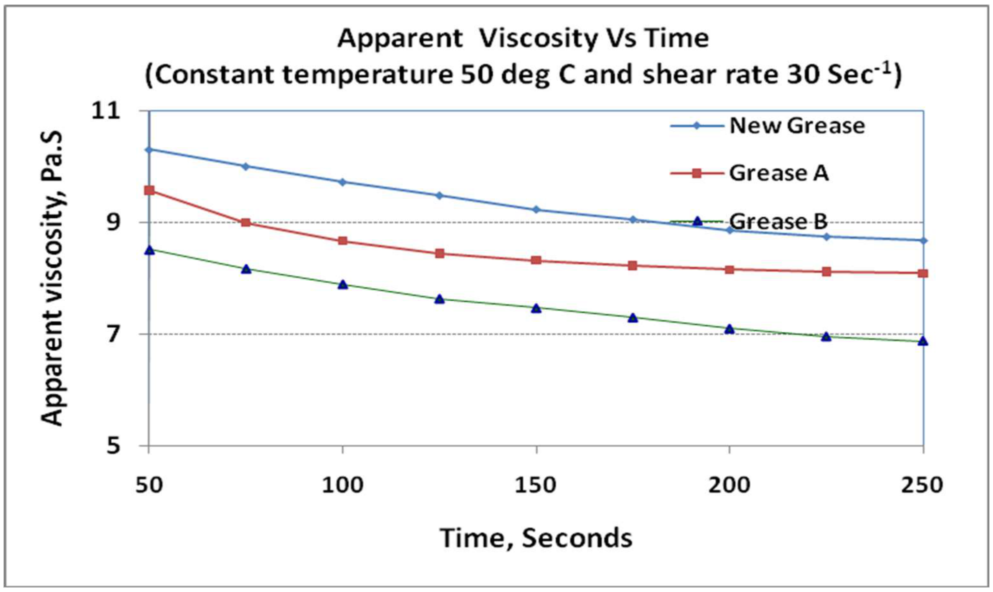

Fig-1

In the second study shear was kept constant and temperature was increased from 40 ^{0}C to 110 ^{0}C and determined the apparent viscosity of all the three greases. It was found that Grease A & Grease B showed a downward trend in viscosity when temperature was increased but the viscosity of new grease was least affected especially at the initial stage of temperature rise (upto 60 ^{0}C) (Fig-2).

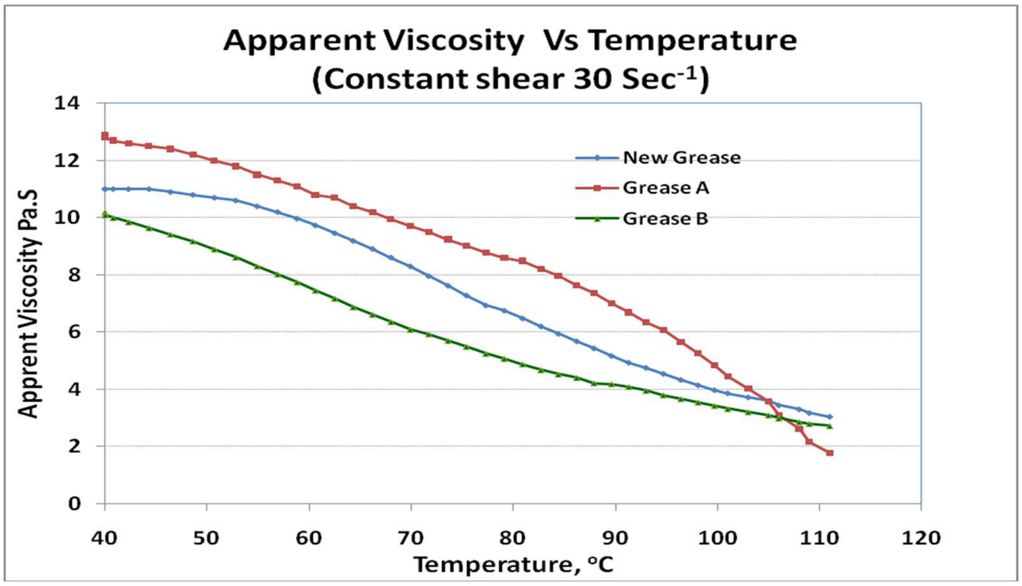

Fig-2

Change in apparent viscosity with respect to time shows that Grease A has the maximum change in viscosity and the new grease has the minimum (Fig-3). Change in viscosity is almost constant for the newly developed grease which shows its superior stability during shear, indicating that the developed grease must perform better in field and will combat leakage during application.

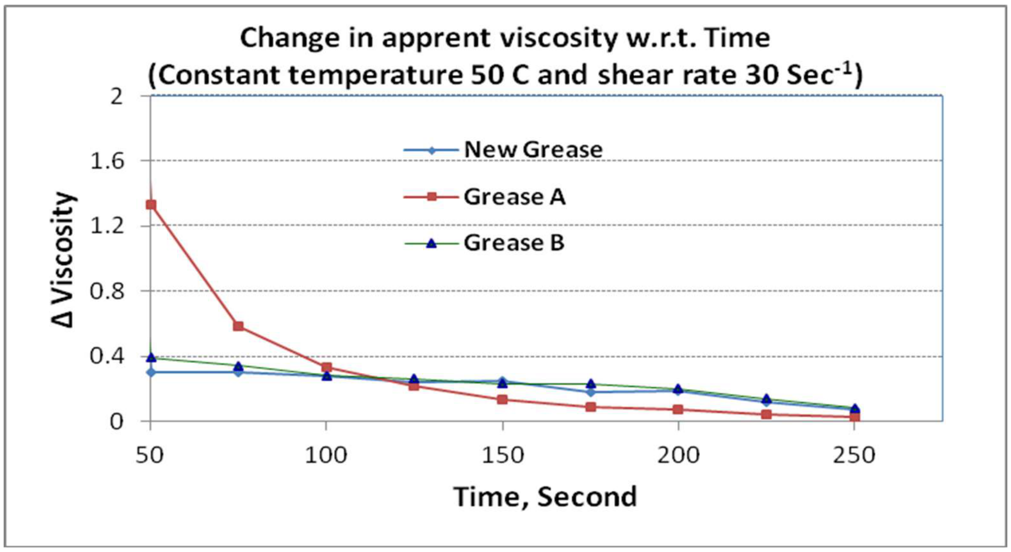

Fig-3

FIELD VALUATION

Performance of developed grease was field tested at two reputed tyre manufacturing units. Product was evaluated in a typical single press moulding machine. The machine mainly comprises of frame, mould opening/closing device, mould adjusting device, center mechanism, curing chamber, tyre loading device, tyre unloading device, hydraulic system, electrical system, pipeline system and post cure inflator etc. A 37 KW motor operates this ~ 38 tones machine. Mould (220 — 520 mm in height) consists of two circular rings which open / close with help of hydraulically driven pistons. Grease lubricates the bush bearings of this moulding press. Grease from a reservoir is fed to sealed mould bearings through a pump at around 2000 psi pressure**.** In the curing process the green tyre is transferred on to the lower mould bead seat. A rubber bladder is inserted into the green tyre and the mould is closed. The bladder is then inflated with high pressure steam. As the mould closes and is locked the bladder pressure increases so as to make the green tyre flow on to the mould, taking on the tread pattern and side wall engraved into the mould. Temperature inside the tightly secured mould reaches to about 175°C with pressure around 350 psi. Curing reaction takes place for around 15 minutes to few hours depending on the tyre specifications. At the end of the reaction the pressure is released and mould is opened up. Tyre is stripped off the mould for finishing process.

Before the introduction of the new grease the old grease was flushed out with the help of some petroleum oil and cleaned the system thoroughly. New grease was then introduced into the system through a Lincoln make air operated single stroke pump. Following were the observations:

Viscosity of the base oil and consistency of the developed grease (NLGI 1) was found optimum for easy pumping of the grease from reservoir. Grease could be smoothly fed to the bearings at 2400 psi. No corrosion of copper or any other metallic part of the machine was reported for the period of trial. Typical mould closing force of 1700 KN was also supported by bearings of the mould. Grease gave satisfactory performance even at high pressures of >250 psi. No seal rupture or bearing failure was reported. Minimum grease change time was found to increase from 12 days (for earlier grease in use) to 25 days. Grease was found to be compatible with seals and grease transporting hose material. Leakage from the side arm bearing and guide roller bearing virtually stopped when new grease was used replacing the old grease, which is evident from the figures 4-7. Grease consumption was found reduced to almost half when new grease was used to lubricate the bearings.

Fig-4: Side arm bearing: Old grease **Fig-5**: Side arm bearing: Newly developed grease

Fig-6: Guide roller bearings: Old grease **Fig-7:** Guide roller bearings: Newly developed grease

CONCLUSION

An NLGI 1 grade consistency grease was developed for lubrication of „*tyre curing press'* and it was successfully evaluated at two reputed tyre manufacturing units. During its evaluation, leakage was reduced significantly and grease consumption came down to half as compared to in-use greases. Additionally, decrease in leakages and spillages of grease provided cleaner shop floor also.

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Study on the Effect of Composition of Polymeric Additives used in Multigrade Gear Oils in Correlation with their Performance

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ABSTRACT

In recent years, there is a ever growing demand on gear oils in both industrial and automotive with numerous products of varying viscosity range available to end users. Among the gear oils, multigrade oils are being most popular and are replacing the monograde gear oils. This is mainly attributed to better thermal behavior against the temperature fluctuations. The performance of the oil is mainly due to selection of appropriate polymeric additives used as a viscosity index improver with optimum shear stability property in the gear oils. In the present work, an attempt has been made to study the composition of few selected polymeric additives as a VI improver (type & molecular weight & its distribution) in tailor made gear oils with shearing effect tested by KRL tester for performance. Effect of shearing was also observed by measurement of viscosity of the oil before and after the test. FTIR spectral and Gel permeation chromatographic analysis were used to characterize the polymeric VI additives used. The study is also extended to few brands of similar gear oils available in the market. The information obtained from the above study will be useful in understanding the type of VI improver suitable for a given applications.

INTRODUCTION

With the advent of newer technology, various changes in machinery & equipment's were taken place in both industrial and automotive applications. This has made significant requirements on various lubricants including engine oils, gear oils, transmission oils, and hydraulic oils. etc. Gear oil is one of the most important lubricants which are available in both monograde to multigrade with trend shifting from mineral oil origin to synthetics. Multigrade oils differ from monograde oil with inclusion of viscosity Index Improver- a specialized macromolecules like polymers which can absorb the temperature fluctuations on usage. Multigrade oils are preferred by users for these characteristic properties for long life performance. Suitable high molecular weight polymers which are shear stable are being used for the end gear applications. Polymers based on hydrocarbon type olefinic copolymer OCP), polybutene polymers, polyacrylate esters, etc are being widely used in formulating gear oils by lubricant manufacturers.

In the present work , an attempt has been made to study the composition of few selected polymeric additives as a VI improver (type & molecular weight & its distribution) in tailor made gear oils with shearing effect tested by KRL tester for performance. Effect of shearing was observed by measurement of viscosity of the oil before and after the test. FTIR spectral and Gel Permeation Chromatographic analysis were used to characterize the separated polymeric VI additives present in the gear oil sample .under study The study is extended to few brands of similar Gear Oils available in the market. The information obtained from the above study will be useful in understanding the type of VI improver suitable for a given end applications.

EXPERIMENTAL

Chemicals: All chemicals employed for analysis were of analytical reagent grade. Acetone, Hexane, GPC Standards - Polystyrene with known molecular weights required for GPC molecular weight calibration.

Lab wares: Standard glassware of Borosil make was used for analysis. Whatman 40 (12.5 cm) filter paper, Oven, Vacuum Desiccators, Beaker 250ml & 500ml capacity.

Instruments: Thermo Nicolet makes Fourier Transform Infrared Spectrometer model iS10 (FTIR), Waters make Modular HPLC system with GPC option. Kinematic Viscometer Bath for 40 & 100 ^{0}C with tubes, Ducom make KRL Four Ball Tester TR - 30H Model.

PROCEDURE

500 ml of eight samples of gear oil including monograde and multigrade for both industrial & automotive applications marked as (A to H) were collected and subjected to above study by adopting the following procedures.

I. Separation and Characterization of Polymeric Additive from the Gear Oil samples under study:

15-20 gms of each of the oil samples was taken in a 500 ml beaker and dissolved the oil sample in 100 ml of hexane, added 100ml of acetone. The solution become hazy on addition of acetone and the solution was kept for overnight to separate the polymeric additive. The solution was carefully decanted with polymeric additive retained at the bottom of the beaker. Washing of polymeric residue obtained initially with 100 ml 1:1 hexane to acetone to remove any adhering oil. Separated polymeric residue was then dried in an oven maintained at 100 ^{0}C and kept in vacuum desiccators. The dried residue obtained was weighed and estimated quantitatively. Further the polymeric residue obtained was further subjected to FTIR Spectral analysis for the type/nature of polymeric additive and Gel Permeation Chromatographic analysis for molecular weight and its molecular weight distribution.

a) FTIR Spectral Analysis:

Infrared spectrum of each of above samples of gear oils along with its separated polymeric residue were recorded in Potassium Bromide cell windows without spacer in a IR demountable cell. The Infrared spectral range used was 4000 cm^{-1} - 400 cm^{-1}. Number of Scans: 32, Resolution: 1 cm^{-1} Thermo Nicolet Fourier Transform Infrared Spectrometer

model iS10 (FTIR) was used for study. Infrared Spectral analysis provides the information on the nature of polymeric additive used in the gear oil samples.

b) Gel Permeation Chromatographic Analysis (GPC):

Gel permeation chromatograms of each of separated polymeric VI improver were recorded using Refractive Index Detector (model 2414, Attenuation x4). Molecular weight and its distribution of each separated additives were obtained using calibration curve. A known polystyrene standards with peak molecular weights covering 4,000 to 4 lakhs was used for calibration. Waters make modular HPLC system with GPC option with Breeze-2 Software was used for the analysis. The samples (200 - 400µl) solution made in THF solvent was injected into Universal Injector U6K. Three Standard 35 cm long Ultrastyragel columns packed in tetrahydrofuran (THF) of pore size 100, 1000 & 10000A^0 were connected in series were used. Binary HPLC pump model 1525 was used to pump at flow rate 1ml/minute of solvent (THF) for analysis.

II. Shear Stability Test by KRL Four Ball Testers as per Method (CEC-L-45-A-99):
About 40 ml of each of gear oil samples under study was taken and subjected to shear stability test using instrumental conditions: Speed RPM - 1475, Load condition 5000N, Temperature 60 ^{0}C and Test Duration - 20 hrs run. After test completed, gear oil was removed and viscosity at 100 ^{0}C was measured and compared with fresh oil viscosity at similar temperature. The percentage change in viscosity at 100^{0}C against fresh oil was calculated and which is a measure of shear stability of the oil.

a) Viscosity Data of Gear Oil Samples under study:
Viscosity of the each of the gear oil samples before and after the KRL test was measured by as per method ASTM D 445. Kinematic viscosity at 40 ^{0}C & 100 ^{0}C, Viscosity index were obtained for all the gear oil samples.

RESULTS & DISCUSSIONS:

Table 1 shows the results of separated polymeric additive content by above mentioned wet chemical procedure. From the Table 1, it was observed that different samples of gear oil have different polymeric additive contents. This reflects that there is a distinct variation in composition offered by different manufacturers of gear oils for similar grade and end application.

Table 1

S.No	Sample Description	Gear Oil SAE Grade	Separated polymeric/VI Improver content (wt%)
1	A	90	--
2	B	90	--
3	C	80W	0.92
4	D	80W90	8.26
5	E	80W90	4.12
6	F	85W140	9.19
7	G	85W140	6.48
8	H	85W140	3.96

a) Fourier Transform Infrared Spectral Analysis of Gear Oil Samples under study:

The samples of gear oil under study were recorded for infrared spectra along with its separated polymeric additive. It was observed that IR spectra of samples as such did not give clearly the presence of VI improver additive therefore require clear separation of the polymeric additive from gear oil under study. Fig.1, 2 & 3 shows IR spectra of typical separated polymeric additive from the gear samples under study:

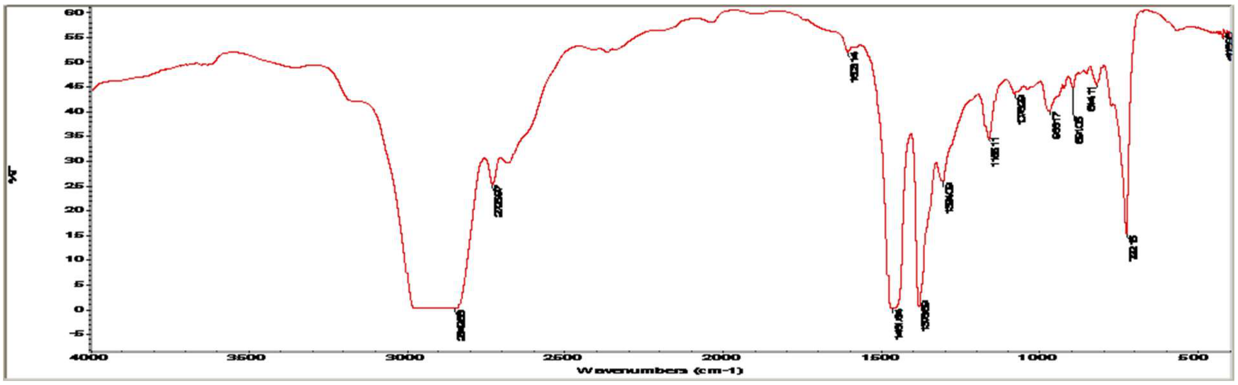

Fig. 1 FTIR spectrum of separated polymeric additive (olefinic copolymer) from Gear Oil samples

However, IR spectral analysis of the entire polymeric additive from gear oil samples under study has indicated the peaks characteristic of type polymeric additive used.

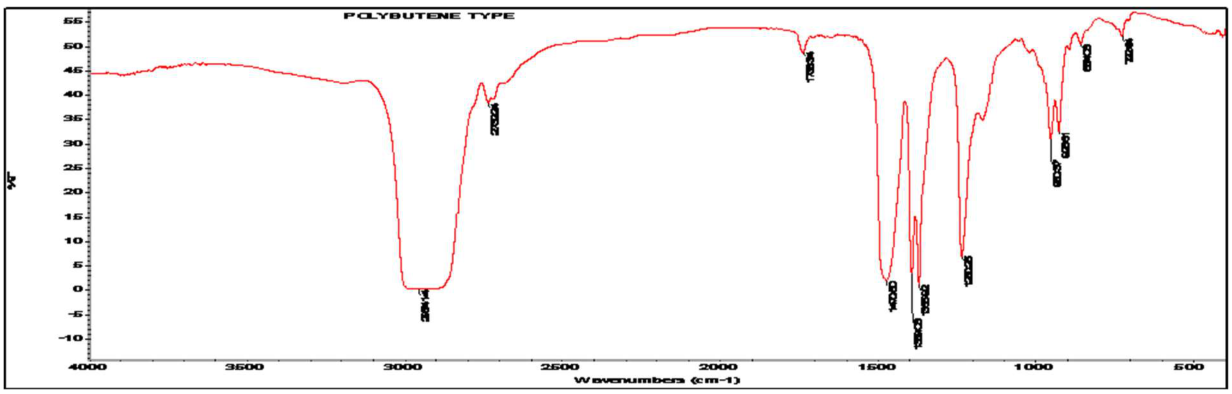

Fig. 2 FTIR spectrum of separated polymeric additive type (polybutene) from Gear Oil samples.

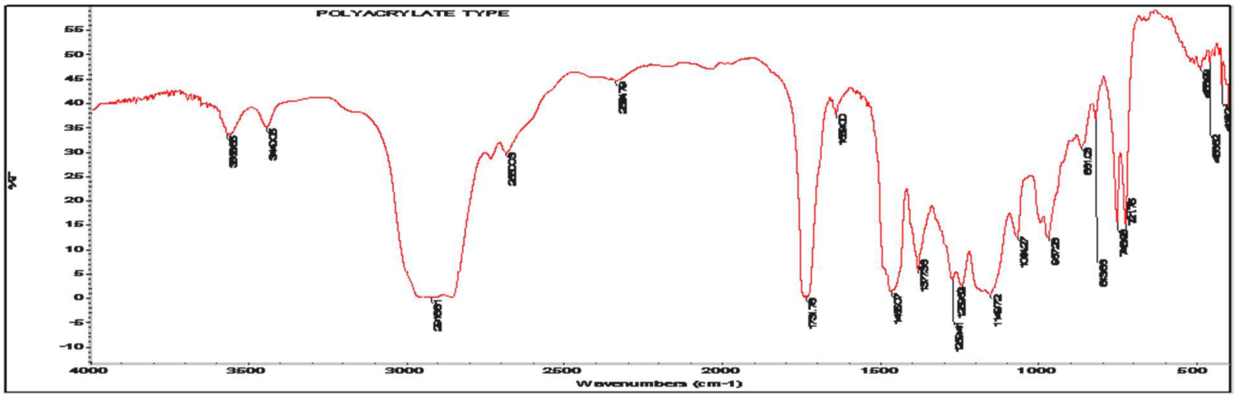

Fig. 3 FTIR spectrum of separated polymeric additive type (polyacrylates ester) from samples.

From the Table 2, FTIR Spectral analysis of separated polymeric additive in gear oil samples under study

Table 2

S. No	Sample Description	Gear Oil SAE Grade	Type of Separated Polymeric additive from Gear Oil samples under study
1	A	90	Absence of polymeric additive
2	B	90	Absence of Polymeric additive
3	C	80W	Presence of Olefinic Copolymer (OCP Type) 3000-2700 cm^{-1}, bifurcated peaks 1474 cm^{-1}&1377 cm^{-1}, 1155 cm^{-1}, 966 cm^{-1} and 721 cm^{-1}
4	D	80W90	Presence of Polybutene Type -3000-2700 cm^{-1}, bifurcated peaks 1476 cm^{-1}& 1377 cm^{-1}, 1230 cm^{-1}, 986 cm^{-1} , 922 $cm^{=1}$ and 722 cm^{-1}
5	E	80W90	Presence of Olefinic Copolymer (OCP Type) 3000-2700 cm^{-1}, bifurcated peaks 1477 cm^{-1}&1377 cm^{-1}, 1154 cm^{-1}, 965 cm^{-1} and 722 cm^{-1}
6	F	85W140	Presence of Polybutene Type -3000-2700 cm^{-1}, bifurcated peaks 1476 cm^{-1}&1377 cm^{-1}, 1229 cm^{-1}, 986 cm^{-1} 965 cm^{-1} and 722 cm^{-1}
7	G	85W140	Presence of Olefinic Copolymer (OCP Type) 3000-2700 cm^{-1}, bifurcated peaks 1476 cm^{-1}& 1375 cm^{-1}, 11565cm^{-1}, 966 cm^{-1} and 721 cm^{-1}
8	H	85W140	Presence of Olefinic Copolymer (OCP Type) 3000-2700 cm^{-1}, bifurcated peaks 1476 cm^{-1}&1376cm^{-1}, 1155 cm^{-1}, 962 cm^{-1} and 722 cm^{-1}

Fig.1 (sample C, E, G & H) and Fig. 2 (sample D, F) depicts the difference in the IR spectra observed for gear oil samples for given end applications. Gear Oil samples under study did not indicate the presence of polyacrylates esters type of VI improvers as observed in Fig. 3

b) Gel Permeation Chromatographic Analysis of separated polymeric additive from the Gear Oil Samples under study:

Fig. 4 and 5 shows a typical GPC chromatogram of separated polymeric additives

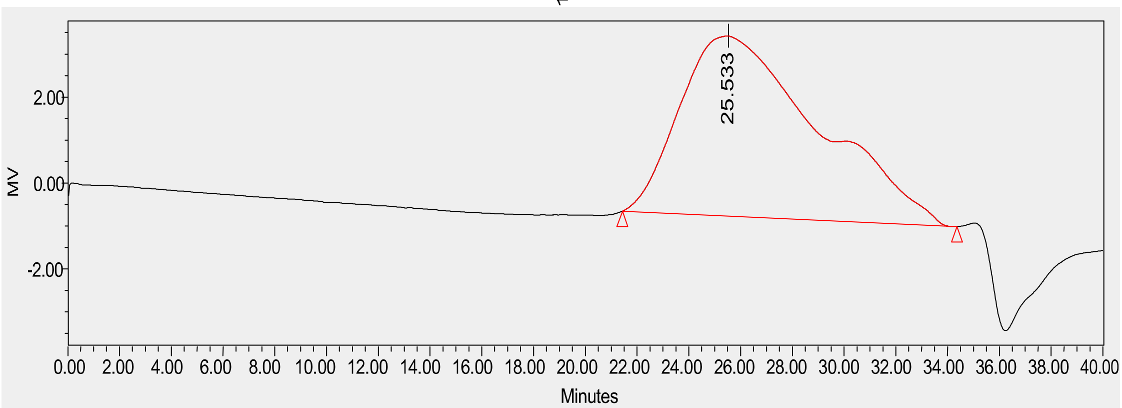

Fig. 4 GPC of Separated polymer from Multigrade oil Sample D

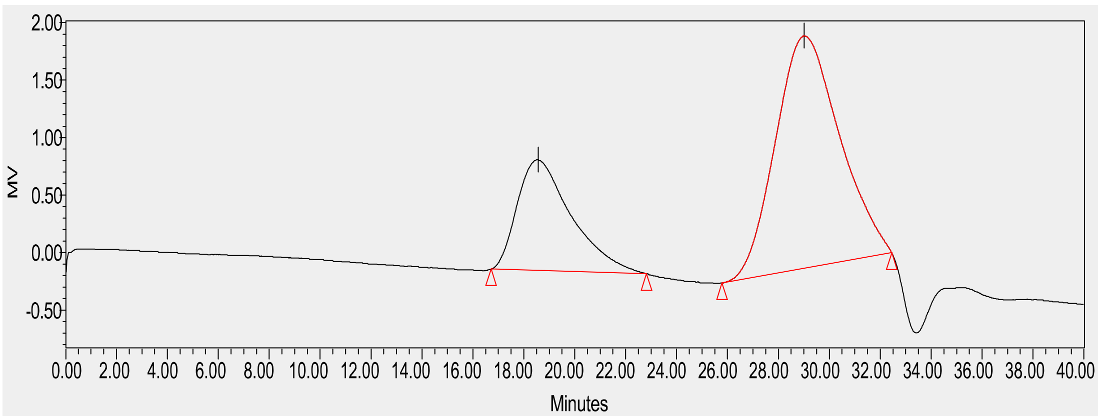

Fig. 5 GPC of Separated polymer from Multigrade oil Sample G

of Polybutene type and Olefinic Copolymer type from gear oil under study: Table 3 indicates GPC analysis of separated polymeric additive from gear oil samples under study.

Table 3

S. No	Sample Descrip-tion	Gear Oil SAE Grade	Peak Molecular Weight ob-tained from GPC	Remarks if any
1	A	90	-	Absence of polymeric additive
2	B	90	-	Absence of Polymeric additive
3	C	80W	27,040	Presence of Olefinic Copolymer OCP Type) very broad molecular weight distribution
4	D	80W90	9,402	Presence of Polybutene Type – very broad molecular weight distribution
5	E	80W90	50,275	Presence of Olefinic Copolymer OCP Type) very broad molecular weight distribution
6	F	85W140	8, 977	Presence of Polybutene Type – very broad molecular weight distribution
7	G	85W140	2,66,685	Presence of Olefinic Copolymer OCP Type) very broad molecular weight distribution
8	H	85W140	95,940	Presence of Olefinic Copolymer (OCP Type) very broad molecular weight distribution

II Shear Stability Test by KRL Four Ball Tester as per Method (CEC-L-45-A-99) on Gear Oil samples under study:

Table 4 indicates the test results of the gear oil samples under study. Performance of the polymeric additive present in multigrade oil for shear stability was carried out on KRL Four Ball Test Rig.

Table 4

S.No	Sample Description	K.V at 40 ^{0}C cSt, Before Test	K.V at 100 ^{0}C cSt, Before Test	VI (Calc)	K.Vi at 100 ^{0}C cSt, After Test	Percentage Change in Viscosity (%) at 100 ^{0}C
1	A 90	190.1	16.96	95	16.74	1.30
2	B 90	183.39	16.77	96	16.59	1.07
3	C 80W	73.50	9.83	114	9.64	1.93
4	D 80W90	134.74	15.58	120	15.22	2.32
5	E 80W90	137.96	14.25	100	19.97	3.99
6	F 85W140	273.34	26.86	129	25.59	4.69
7	G 85W140	384.21	27.19	96	24.32	10.55
8	H 85W140	367.77	26.99	99	26.60	1.45

The viscosity of gear oil before and after test at 100 ^{0}C was measured by Standard ASTM D445 method. The percentage change in viscosity after the test was calculated which is measure of shear stability of polymeric VI additive used in the gear oil samples under study. Gear Oil is better performing if the percentage changes in viscosity within 5% maximum. From the above Table 4, it was observed that among the respective grade 85W140 of multi-grade oil sample 'G' has observed very high viscosity change due to higher shear despite having high molecular weight OCP type polymer. It was also observed that lower molecular weight polybutene type of polymeric additive gave better results with change in viscosity due to better shear stability (within 5%).

CONCLUSIONS:

The above study is based on correlation of type, molecular weight & its distribution of polymeric VI improver used in multigrade gear oils under study with performance of VI improver in terms of its shear stability. It was concluded that there is a distinct variation in use of polymeric VI additive in terms of nature as well as its molecular weight in different market samples of multigrade gear oil s for a given end application. FTIR spectral analysis provided useful information on the nature/type of VI improver used only after separating it from the gear oil samples under study. GPC analysis gave very useful information on peak molecular weight & distribution type - narrow/ broad of separated polymeric additive from the samples under study. Samples having polybutene type-low molecular weight polymer with broad distribution gave better results than high molecular weight OCP type polymeric VI improver. Among the multigrade gear oils samples, sample 'D'&'H' is expected to perform better as the its VI improver is very shear stable with less percentage change in viscosity. Both nature and its molecular weight are important parameters for deciding to select a VI improver for multigrade gear oils as evident in sample 'D'& 'E'. Shear stability of the polymer is important parameter of selecting polymer not the molecular weight as observed in sample of multigrade oil 'G' Monograde oils 'A' & 'B' have taken for study observed low values in percentage change in Viscosity mainly attributed to base fluid is not relevant as they do not have polymeric additive.

The procedure adopted for separation of polymeric additives such as VI improvers followed by characterization for type and molecular weight and correlation with shear in property of polymeric additive will help in understanding the long life performance of the multigrade gear oils. The procedure for separation of VI improver is important which can be easily adopted in the laboratory for qualitative and quantitative purpose.

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Food grade greases for high temperature applications

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Abstract

The modern machinery used to prepare and process foodstuffs, cosmetics, and pharmaceutical products need lubricants, grease and oil for lubrication, heat transfer, power transmission and corrosion protection of machinery, machine parts, equipment and instruments. Incidental contact between lubricants and food cannot always be fully excluded and may result in contamination of the food product. In all cases where product contact cannot be fully excluded food grade lubricants must be used.

In order to ensure that safety in food processing areas, Foodsafe lubricants have been designed not only to meet the FDA /NSF statuary requirements but also to meet performance levels required for the application.

In this technical paper we would be analysing the high temperature performance of Food grade greases in controlled environment. High temperature food grade greases play an important role in manufacture of Ready to eat food, chocolates, wafers, biscuits and packaging machines where the greases need to stay in grade even at high temperatures .We have studied the high performance related properties by various test methods in order to understand the performance characteristics of our food grade greases

Introduction

In order to meet the high temperature lubrication requirements, high viscosity synthetic base oils have been used in high temperature industrial lubricants for many years. They are effective up to 220 C (380 F) and PFPE's upto 280'C (500' F) show little evaporation or oxidative degradation compared to other common lubricant basestocks. Raj petro has introduced a series of Synthetic Food grade greases that has earned the prestigious NSF H1 approval for use in indirect food contact applications. The wide industrial acceptance of synthetic oils & PFPE's suggests that food processing plants will benefit from the long lasting, clean lubrication they provide. This paper describes high temperature food grade greases made from these unique base stocks.

Background

The food industry represents one of the most demanding challenges at high temperatures for a lubricant. The lubricant must not only perform the familiar functions of reducing friction, wear, and corrosion, but it must conform to rigorous standards to ensure food safety is never compromised. In today scenario food safety is the first responsibility of any company that manufactures edible products. Consumers have every right to expect food plants will comply with government regulations and industry best practices at every stage of the process.

There are many governing bodies around the world that are involved in the safety of food products because of the obvious public health considerations and associated repercussions of contamination. In the United States, the regulatory environment begins with the US Food and Drug Administration (FDA). Interpreting the Federal regulations can be a monumental task, so NSF International provides guidance that helps food processors understand which products are approved for use in food plants and how they are best applied. In India Food Safety Act has been enacted in 2011 which has necessitated the use of foodgrade lubricants for Food processing applications

Food grade lubricants are made from base fluids and additives that are safe for food manufacturing. Regulations also require food grade lubricants to be tasteless, odourless and colourless. Until 1998, USDA Food Safety and Inspection Service, published “the white book”, a list of proprietary substances and non-food compounds that were food grade or USDA H1 approved. The USDA cancelled all its activity to review and approve lubricants in September 1998. After this cancellation, NSF International adopted the procedures.NSF International, a not-for-profit; non-governmental organization helps the FDA classify and manage approvals for lubricants in the food industry. NSF International provides a conformity assessment for the FDA and then categorizes the lubricant appropriately. NSF adopted these procedures to certify lubricants as H1 and H2. NSF defines H1 lubricants as “lubricants for incidental food contact special approval criteria based on FDA List 21 CFR Part 178.3570”.

NSF H1 food grade lubricants are appropriate in situations where potential risk scenarios exist such that small amounts of lubricant that could potentially come in contact with edible products. NSF H1 lubricants offer the food industry a sense of security and safety from incidental contamination in food from more toxic, non-food grade lubricants.

The primary mechanisms for oil degradation at high temperatures are oxidative and/or thermal breakdown, and polymerization. Breakdown, in which breakdown of the lubricant molecule occurs, leads to the formation of lower molecular weight volatile compounds. Evaporation of these compounds can cause changes in viscosity, oil loss, and the production of excessive smoke. This can lead to poorer lubrication, higher cost, reduced cleanliness of the plant, poorer product quality, and higher exposure to potentially toxic organic compounds. Polymerization will lead to formation of insoluble gums and varnishes that can build up in the work environment or even contaminate food products. Cleaning these deposits requires additional maintenance and generates chemical waste materials for disposal. Further, production is lost as machinery is taken out of service for cleaning.

Objective:

The purpose of this paper is to assess the performance of high temperature food grade grease by focusing on the oil/thickener system. We started by making laboratory samples of food grade synthetic greases using different thickeners as shown below.

Raj Petro Name	Density	Kin Vis @ 40’C	Kin vis @ 100	Flash Point ‘C	Pour Point ‘C
Food grade grease A	0.950	ISO VG 100	12.5	250	-40
Food grade Grease B	1.042	ISO VG 460	➢ 35	220	-40
Food grade grease C	0.850	ISO VG 100	10.8	240	-21
Food grade Grease D	1.821	ISO VG 460	> 42	> 340	-40

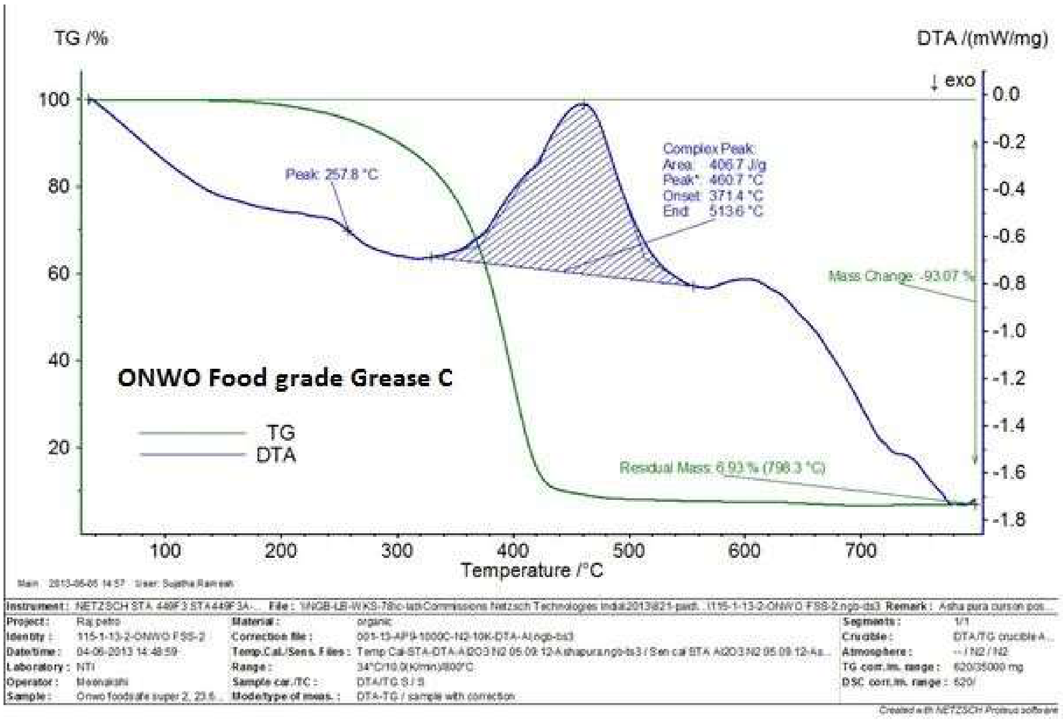
Experimental:

This test is not meant to simulate any specific working environment, but provides a simple comparison of how high temperatures affect both the thickener and base oil of the grease. A two gram sample of each grease is weighed into a 100 mm stainless steel pan and then covered with a 70 mm cover. A group of up to four greases are set on a pan with cover and placed in an oven for 20 hours at the specified temperature. At the end of the test, the specimens are removed from the oven and allowed to cool for one hour. The pans and covers are weighed again to determine weight loss of the grease and weight increase in the cover pan attributed to solid varnish deposited from oil vapours. They are also photographed to document the appearance of the grease and pans. Testing started at 400 F (204’C).for 4 / 6 / 8 / 12 hrs. intervals If the grease survived, a fresh sample was tested at a higher temperature.

The above experimental method is also verified using TGA / DTA analysis

Definition: Thermogravimetric Analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere. A TGA consists of a sample pan that is supported by a precision balance. That pan resides in a furnace and is heated or cooled during the experiment. The mass of the sample is monitored during the experiment. A sample purge gas controls the sample environment. This gas may be inert or a reactive gas that flows over the sample and exits through an exhaust.

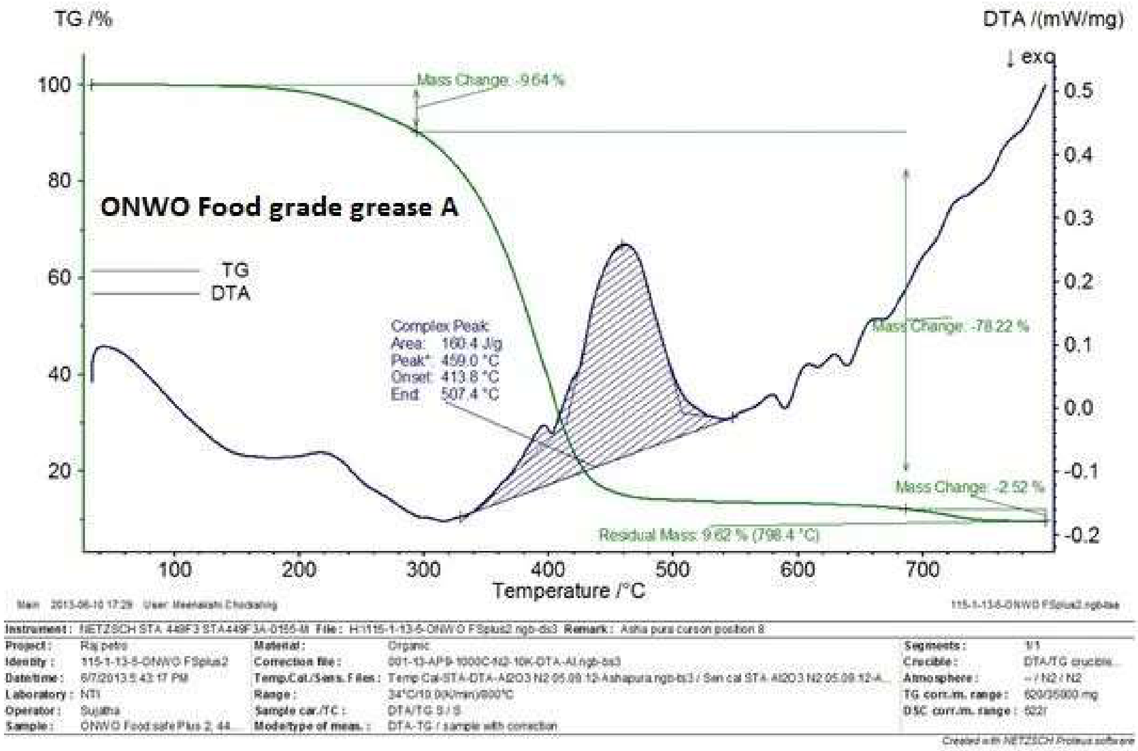
Fig 1 : TGA /DTA analysis of ONWO Food grease C



In the above TGA /DTA the following are observed

1. The melting point starts around 210 ‘C and the 93% of the mas change occurs at 420’C
2. The DTA curve indicates an endothermic reaction with a peak at 475’C due to the decomposition ofsolids used in the grease.
3. The above characteristic clearly indicates that the solids provide lubrication above 200 'C

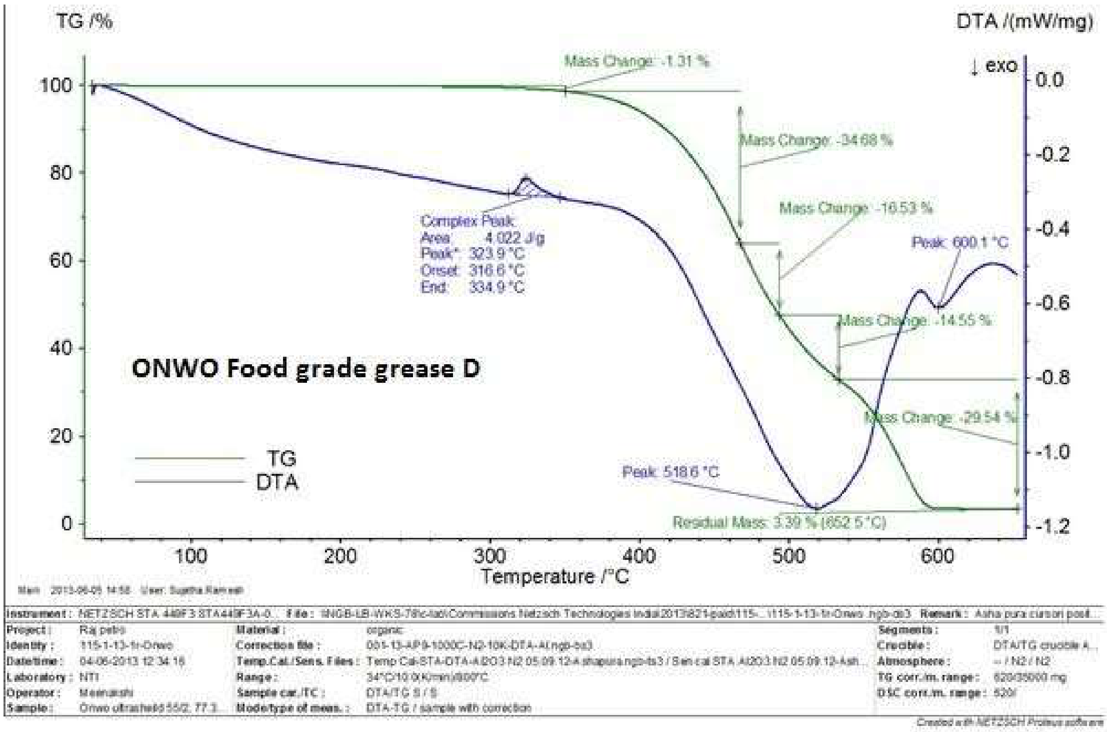
Fig 2 : TGA /DTA analysis of ONWO Food grease A



In the above TGA /DAT the following Observations are Observed.

1. The mass decomposition starts around 200'C and a reduction of 10% is noticed till 275'C.
2. There is an endothermic reaction between 320'C and 550'C due to the solid lubricants present in thegrease.
3. **The combination of synthetic oil & solids provides lubrication upto 250 'C**

Fig 3 : TGA /DTA analysis of Food grade grease D



In the above TGA /DTA the following are observed

1. The mass decomposition starts around 340'C with its melting point noticed around 320'C by the peak of the DTA curve.
2. The above DTA /TGA graph indicates that the grease performs continuously upto 280'C.

Conclusion of hot studies conducted in the oven .

Further studies have been conducted in the lab by studying the grease under varying temperatures and timeintervals

- At 204 'C All the three Foodsafe greases retained their greasy structure after 12 hrs
- At 232'C All the three Foodsafe greases were darkened but vapour formation was still neglible after12 hours.
- At 260'C 4 hours – no drop out was observed / heavy deposit noticed /top plate amber stains noticed
- At 260'C 12 hours – no drop out observed / heavy deposit noticed /brown stains noticed
- At 260'C -20 hrs – no drop out observed / heavy deposit noticed / dark stains noticed.

In conclusion we find that the three Foodsafe Greases work satisfactorily upto 204'C and in the case of PFPEgrease it works upto 260'C.

Annexure 1

Studies on high temperature Greases

FOOD GRADE GREASE B
TIME: 12 HRS TEMP: 204^{0}C

FOOD GRADE GREASE B
TIME: 12 HRS TEMP: 232^{0}C

FOOD GRADE GREASE C
TIME: 12 HRS TEMP: 204^{0}C

FOOD GRADE GREASE C
TIME: 12 HRS TEMP: 232^{0}C

FOOD GRADE GREASE A
TIME: 12 HRS TEMP: 204^{0}C

FOOD GRADE GREASE A
TIME: 12 HRS TEMP: 232^{0}C

Temp: 204 deg C , Time : 12 Hrs				
ID	**Thickener**	**Skinning**	**Weight loss**	**Vapor Deposit (mg)**
FOOD GRADE GREASE B	**Greasy texture**	**light Skin**	**< 10%**	**0.5**
FOOD GRADE GREASE C	**Greasy texture**	**light skin**	**< 12%**	**1.2**
FOOD GRADE GREASE A	**Greasy texture**	**light skin**	**< 8.5%**	**0.8**

Temp: 232 deg C , Time : 12 Hrs				
ID	**Thickener**	**Skinning**	**Weight loss**	**Vapor Deposit (mg)**
FOOD GRADE GREASE B	**Greasy ,Heavy Deposit**	**Heavy Skin**	**24**	**1.8**
FOOD GRADE GREASE C	**Greasy , Heavy Deposit**	**Heavy skin**	**26.5**	**2.6**
FOOD GRADE GREASE A	**Greasy , Heavy Deposit**	**Mild Skin**	**25.5**	**2.4**

 **FOOD GRADE GREASE B** TIME: 4 HRS TEMP: 260^{0}C	 **FOOD GRADE GREASE B** TIME: 12 HRS TEMP: 260^{0}C	 **FOOD GRADE GREASE B** TIME: 20 HRS TEMP: 260^{0}C
 **FOOD GRADE GREASE C** TIME: 4 HRS TEMP: 260^{0}C	 **FOOD GRADE GREASE C** TIME: 12 HRS TEMP: 260^{0}C	 **FOOD GRADE GREASE C** TIME: 20 HRS TEMP: 260^{0}C
 **FOOD GRADE GREASE A** TIME: 4 HRS TEMP: 260^{0}C	 **FOOD GRADE GREASE A** TIME: 12 HRS TEMP: 260^{0}C	 **FOOD GRADE GREASE A** TIME: 20 HRS TEMP: 260^{0}C

Temp: 260 deg C , Time : 4 Hrs				
ID	**Thickener**	**Skinning**	**Weight loss**	**Vapor Deposit (mg)**
FOOD GRADE GREASE B	No Drop, Heavy Deposit	Heavy Skin	29%	30.7
FOOD GRADE GREASE C	No Drop, Heavy Deposit	Heavy skin	49.8%	10.2
FOOD GRADE GREASE A	No Drop, Heavy Deposit	Mild skin	36.5%	41.7

Temp: 260 deg C , Time : 12 Hrs				
ID	**Thickener**	**Skinning**	**Weight loss**	**Vapor Deposit (mg)**
FOOD GRADE GREASE B	**No Drop, Heavy Deposit**	**Heavy Skin**	**48**	**31.8**
FOOD GRADE GREASE C	**No Drop, Heavy Deposit**	**Heavy skin**	**54.8**	**92.6**
FOOD GRADE GREASE A	**No Drop, Heavy Deposit**	**Mild Skin**	**38.5**	**33.2**

Temp: 260 deg C , Time : 20 Hrs				
ID	**Thickener**	**Skinning**	**Weight loss**	**Vapor Deposit (mg)**
FOOD GRADE GREASE B	**No Drop, Heavy Deposit**	**Heavy Skin**	**48**	**40.3**
FOOD GRADE GREASE C	**No Drop, Heavy Deposit**	**Heavy skin**	**58.1**	**100.5**
FOOD GRADE GREASE A	**No Drop, Heavy Deposit**	**Mild Skin**	**39.9**	**42.3**

References:

Food Grade Grease Improves High Temperature PerformanceBy Tyler Housel and Sarah Plimpton Liebowitz