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The Challenges in the Development of Energy Efficient Greases

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Abstract

Under pressure from governmental legislation, original equipment manufacturers (OEMs) have been focused on improving the efficiency of engines and drive trains. There was little focus on improving the efficiency of other automotive components. With the desire to utilize every means of potential efficiency saving, all moving componentry on a typical automobile is now being scrutinized for friction and energy losses. This includes the greases used in the wheel and accessory equipment bearings, and transmission components, including universal joints (UJs) and constant velocity joints (CVJs), which transmit power and motion from the gearbox to the wheels.

There are two main contributors to energy losses in bearings: churning through the grease; and friction losses due to sliding of the cage against the rolling elements and raceways. By optimising the lubrication of the bearings, theoretical improvements in energy efficiency are possible, but as bearings are extremely efficient, measuring the losses is a significant challenge.

In components that have significant sliding contacts, such as plunging CVJs, lower friction greases have been developed. Initially they were developed to reduce the noise, vibration and harshness (NVH) profile of transmissions, but now more focus is being given to their application to improve energy efficiency.

Historically, friction coefficients of greases have typically been measured using an Optimol Instruments™ SRV tester. This has been standardized as DIN 51834 for friction measurements. However, published papers show that many users of the instrument do not follow the standardized conditions to determine the friction coefficients of greases. Other tests used to measure friction include the high frequency reciprocating rig (HFRR), the Cameron Plint TE77, and the sliding 4-ball wear tester.

Today, grease frictional losses are not just an issue for the automobile industry but also for industry in general as users seek to improve energy efficiency. This paper looks at friction and efficiency losses in greases and the application of friction modifiers to potentially reduce the frictional losses in grease- lubricated componentry in a wide variety of applications.

Introduction

The last 30 years have seen an increased focus on improving the efficiency of automobiles and commercial vehicles, both from the perspective of resource conservation and greenhouse gas reduction. Government legislation has forced vehicle manufacturers to make engines and

drivelines significantly more efficient than they were in the 1980s. Base oil viscosities of crankcase oils have fallen from a then typical SAE 20W-50 to the recently published SAE 0W-16 standard. The implications of the legislation, along with changes to hardware and operating conditions have improved crankcase and driveline lubricants efficiency. Since 1990, there has been a significant reduction (25%) in the amount of fuel consumed per mile by passenger cars and light trucks. This is despite the average vehicle now weighing approximately 15% more than it did then due to the incorporation of more safety features and ancillary equipment. In 2014, according to Wyrley-Birch (1) in his presentation at the 10th ICIS Pan American Base Oils & Lubricants Conference, we have entered the "Era of Efficiency."

While the spotlight was on the energy efficiency of engines and drive trains of vehicles, there was little or no focus on other components. With the desire to capture every means of potential efficiency saving, all moving componentry on a typical automobile is now being scrutinized for friction and energy losses. On a typical passenger car or light truck, there may be as many as 50 different greases used to lubricate components. The majority of these have no influence on the efficiency of the vehicle. An example of this would be the grease to lubricate the driver's seat rail. However greases used in the wheel and accessory equipment bearings, steering, and transmission components all have been shown to influence efficiency. Transmission components include support bearings, universal joints (UJs) and constant velocity joints (CVJs), which transmit power and motion from the gearbox to the wheels.

At the 2013 NLGI annual meeting there were three presentations (2), (3), and (4) focussed on the efficiency of industrial and automotive lubricants. The first paper (2) focussed on methods of testing and the development of low friction greases. The second paper (3) looked at methods for determining the performance of energy efficient of industrial gear oils. The third paper (4) investigated the influences on the efficiency of rolling element bearing greases. Many of the same measurement and assessment techniques were employed in all three papers. Both of the two papers focussed on grease (2) and (4) examined different aspects of the role that grease plays in respect to energy efficiency.

Yamamoto and Imai (4), as part of their development of energy efficient greases, reported that aside from the losses due to the sealing system, there are two main contributors to energy losses in deep groove ball bearings: churning through the grease; and viscous rolling resistance. In this type of ball bearing, the centre of the ball on raceway contact sees primarily a rolling motion but is a mixture of sliding and rolling on the edges of the contact patch. The ball slides against the cage pocket wall, but in these contacts the stresses are comparatively low and have little measurable effect on efficiency. Deep groove ball bearings are the primary design used in electric motors, driveshaft support bearings and accessory drive components.

Wheel bearings are a different case. Because that they have to support both radial and axial loads, deep groove ball bearings cannot be used. In many North American vehicle applications, a pair of opposed tapered roller bearings are used as wheel bearings. In other markets such as Europe, an opposed pair of angular contact bearings are used. In both types of bearing set ups, there are friction losses due to sliding contacts of their rolling elements against the cage, and the cage against the raceways. The sliding speeds, lubrication, and contact conditions are different, which leads to varying energy losses and efficiency.

According to Bowden and Tabor (5) the coefficient of friction in a sliding contact is an order of magnitude greater than for a rolling contact. If the amount of sliding in a contact could be reduced, it would contribute to lower frictional losses. Along with the current pressures of better durability, reduced size and weight, improving frictional efficiency of components is another significant challenge to designers. The other way to reduce sliding frictional losses is to reduce the coefficient of friction of the lubricant. In the 1920s, friction modifiers were first investigated (6) as a means of lowering friction under boundary lubrication conditions in liquid lubricants. In 1963, the first organic molybdenum friction modifiers were developed (7) but not widely used until the 1980s for low friction greases (8). These low friction greases were initially developed to reduce the noise, vibration and harshness (NVH) profile of plunging CVJs in front engine, front wheel drive (FF) transmissions (8). Similar organic molybdenum additives were later applied to improve the fuel efficiency of crankcase oils but now more focus is being given to their application to improve energy efficiency of automotive and industrial greases (2).

Friction

Friction is one of the most important forces in nature, but it is not fully understood by scientists. It is the reaction force that opposes the motion of two bodies in contact (5). Friction itself has been unfairly labelled as a negative force as it reduces the useable energy of machines and converts it to heat or vibrations. There are many influences on the energy efficiency of componentry, friction losses being only one source. Friction also has its benefits. Frictional forces keep people from falling over when they walk by preventing slip between the soles of their shoes and the ground. It has been widely reported that friction is the enemy of energy efficiency but this is an over-simplification. Friction losses in the driving mechanism of automobiles account for approximately 25% of the energy losses, but without friction the wheels would slip and the vehicle would be undriveable. Neither clutch mechanisms nor brake systems would function. Leonardo Da Vinci is known to have studied friction 500 years ago. The first laws of friction were devised by G. Amontons (1699) later modified by C-A de Coulomb (1785) and L. Euler. Amontons' first law of friction states that "The force of friction is directly proportional to the applied load" and his

second states that "The force of friction is independent of the apparent area of contact", (6). One of the basic assumptions made is that the contacting materials are perfectly rigid and inelastic, which is clearly not true for real world applications. A good example being automobile tires, where increasing the width provides increasing traction. Friction is dependent on the real area of contact which is different from the apparent area of contact. There are essentially two types of friction: static and dynamic (3). Static friction, also called adhesive friction, is the component of resistance to motion as the contacting bodies try to move from rest. It is not the same as inertia.

Dynamic friction, also called kinetic friction in many texts, is the component of resistance to motion of the contacting bodies as they move relative to one another. Both need to be considered and understood in order to reduce the friction between the surfaces. Both play a significant role in friction measurements determined using the SRV test. Static friction is caused by the interaction of the surfaces trying to start moving across one another and has two

components: interfacial adhesion and roughness interaction. Dynamic friction is caused by the interaction of the surfaces moving across one another and is made up of three components: interfacial adhesion, roughness interaction, and the shearing of the lubricant or surface layer separating the contacting loaded surfaces. Coulomb's law of friction states that "Dynamic friction is independent of the sliding velocity." For dry sliding without interfacial transfer this can be true but for most lubricated contacts this is not true.

The friction coefficient is typically defined as the ratio of the normal force to the tangential force and is dependent on the local tribological system. In the case of an object being pulled along, the normal force is the weight of the object.
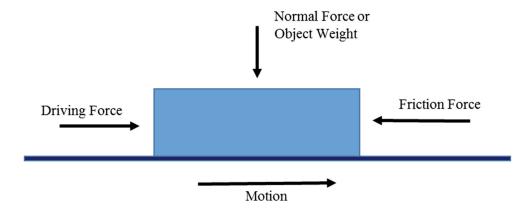

Figure 1 – Tribological system

Friction Force = Normal force x friction coefficient $F = \mu.N$ Equation 1

Both static and dynamic friction need to be considered and understood in order to manage the friction between the surfaces, especially in reciprocating friction tests and applications such as CV joints

Friction Testing
As reported in 2013 (2), there are many different standardized ways of way of measuring friction in greases. Historically the sliding 4-ball wear tester was used to measure friction. One important issue with using the 4-ball machine to measure friction is the bearing used in the apparatus influences the friction values. Some machines have air bearings and others have tapered roller bearings. The latter have higher motion resistance and this makes comparative data difficult to interpret. Cameron-Plint instruments were developed in the 1960s to measure friction and wear. The main two instruments used are the TE70 Micro Friction machine (commonly known as the HFRR) and the TE77 High Frequency Friction Machine (commonly known as the Cameron-Plint). Both machines were designed to have sinusoidal reciprocating motion. Improvements in instrumentation led to the development and use of electrical resistance / capacitance methods for the amount of metal-metal contact. Around 1990, PCS instruments introduced their version of the HFRR.

The SRV test method was developed in the 1980s by Optimol in Germany. The latest instruments have many features that older machines did not have and the upgraded drive and control systems allow for higher loads and better data acquisition. The general principle was a "Square" wave motion driven forward and springing back. The tester measures the reaction force from the motion under load and from this the maximum, minimum and average values of friction during the test run are calculated. The

test method was standardized as DIN 51834-6 (9). The software produces a friction coefficient-time trace. The output from the force transducer can be captured and digitized to give static and dynamic coefficients. A comparison of the testing parameters of the four types of test machines is included in table 1 below.

Test rig	Sliding 4-ball	HFRR	Cameron-Plint	SRV
Contact Geometry	1 rotating ball loaded on three fixed balls	Ball on smooth flat plate	Ball on smooth flat plate	Ball on smooth flat plate
Contact shape	3 points	Point	Point	Point
Contact motion	Unidirectional	Reciprocating sinusoidal motion	Reciprocating sinusoidal motion	Reciprocating square wave
Rpm / Frequency (Hz)	1200	20 to 50	20	50
Speed (m/s)	0.4	0.04 or 0.1	0.4	0.2
Duration (minutes)	60	75	120	120
Sliding distance (m)	1436	200 or 450	3110	1440
Temperature (°C)	75	No set temperature	100 or 120	50 or 80 or higher
Load (N)	392	1.96 or 4.91	200	200
Stress (GPa)	3.05	0.81 or 1.10	0.1	2.71

Table 1 – Comparison of testing parameters

As reported in 2013 (2), a whole series of commercial and experimental friction modifiers were tested in two different base greases. Based on published data (8), the most common globally used thickener systems for low friction greases are urea derivatives and simple lithium soaps. For some higher temperature applications, lithium complex greases are also used so it was decided to use lithium complex and urea-thickened greases. The first selection issue was type and viscosity of the base fluid.

Many low friction greases have base oil viscosities between ISO VG 68 and 150. The laboratory urea grease had an MDI-fatty amine thickener (~12%wt in the finished grease) and was made in a 3-litre resin flask. The base oil was a mixture of naphthenic and paraffinic mineral oils with 10%wt added PAO to give a final ISO VG of 100. The lithium complex was made in a laboratory grease kettle. It used azelaic acid as the complexing agent along with 12-

hydroxystearic acid in a group I paraffinic oil with a viscosity of 112 mm^2/s at 40 °C. Additives were tested at 2wt% on top of a model non-friction modified fully formulated package, which included zinc and phosphorus anti-wear, sulfurized EP, anti-oxidant, and corrosion inhibitor additives at normal treatment levels. Standardised SRV tests at 80 °C, 200 N load, 0.5 mm stroke amplitude and 50 Hz using a 10 mm polished bearing steel ball running against the standard surface lapped discs were carried out. The friction data was reported as being the average value over the test after running in and the end of test (EOT) in which the friction data is averaged over the last 30s of the test run. The ball wear scar diameter was measured along the direction of travel and at an angle 90° to this and averaged. The friction and wear data for the baseline greases is in table 2.

Grease	Average friction coefficient	EOT friction coefficient	Average wear scar diameter (mm)	Comments
Base urea	0.161	0.164	0.99	Very high wear
Formulated urea	0.150	0.145	0.82	High wear
Base LiX	0.134	0.124	0.95	Very high wear; lower than expected friction
Formulated LiX	0.149	0.149	0.80	High wear

Table 2 – SRV Test results for the base greases

The summary of the testing of the 25 different friction modifiers (2) showed that only organic molybdenum complexes gave low friction coefficients (<0.08). It also showed that they do not work on their own and need other additives present such as zinc dithiophosphate and or sulfurized extreme pressure additives as found in fully formulated greases. A summary of the results of SRV testing of fully formulated (FF) greases with molybdenum complex additives is in Table 3. Moly1 is a molybdenum dithiocarbamate (MoDTC) and moly2 is non-phosphorus molybdenum complex (MoX).

Grease + additives	Average friction coefficient	EOT friction coefficient	Average wear scar diameter (mm)	Comments
Urea + FF + Moly1	0.071	0.046	0.49	Long running in time
Urea + FF + Moly2	0.098	0.057	0.59	Higher wear
LiX + FF + Moly1	0.053	0.043	0.45	stable
LiX + FF + Moly2	0.071	0.045	0.60	Higher wear

Table 3 – SRV Test results for the fully F greases with molybdenum complexes

From this testing it was clear that the two molybdenum complexes reacted differently with the two different thickeners and that further optimisation of the formulations was needed. An additional 10 combinations of additives and molybdenum compounds were formulated (LF4 to LF 13). These were tested in lithium, lithium complex and urea-thickened greases. The best two candidate packages, LF4 and LF9, both containing mixtures of MoDTC, MoX and organic friction modifiers. The friction and wear scar data for these two packages are included in table 4.

Additive Package	Thickener	Average friction coefficient	EOT friction coefficient	Average wear scar diameter (mm)	Comments
LF4	Urea	0.053	0.053	0.53	Some running-in
	Lithium	0.057	0.059	0.48	Some running-in
	Lithium complex	0.046	0.050	0.47	Best response in lithium complex
LF9	Urea	0.052	0.046	0.50	Best response in urea
	Lithium	0.061	0.061	0.46	Some running-in
	Lithium complex	0.062	0.058	0.47	Some running-in

Table 4 – Test results for LF4 and LF9 additives

As seen in table 4, the package LF4 gave the best response in the lithium complex base and the LF9 in the urea-thickened greases. Most of the packages tested gave friction coefficients around 0.06 for the simple lithium grease. Adding additional MoDTC to both the LF4 and LF9 packages improved the running-in characteristics, but increased the treat level and net treat cost significantly. This work has shown that low friction can be achieved by formulating packages with combinations or molybdenum complexes, organic friction modifiers and a mixture of anti-wear and other performance additives. However for complete energy efficiency other factors need to be considered, most of which were investigated at by both Harinarain (3) and Yamamoto (4) in their development of energy efficiency lubricants papers. These factors: lubricant traction; film thickness; and rheology and consistency; along with grease fill will now be considered in turn.

Lubricant Traction

One of two definitions of traction in use today is as a measure of the internal friction of a lubricant. It was reported by both Harinarain (3) and Yamamoto (4) that measuring the traction coefficient of the base fluid was an important step in determining the energy efficiency of the lubricant. The lower the traction coefficient the less heat generation that occurs in the application. Yamamoto (4) compared the traction coefficients of a number of base fluids and traction fluids including different types of mineral oil with increasing temperature. One challenge was that the viscosities of the fluids were all different. Comparing the two naphthenic mineral oils, the traction coefficient was determined to be higher for the higher viscosity fluid and similarly for the two synthetic oils tested. The work did however confirm that when comparing different types of base fluids of the same viscosity, polyalphaolefin (PAO) fluids give lower traction than paraffinic mineral oils which in turn are lower than naphthenic.

Some recent unpublished work looked at testing select novel base fluids with extremely low traction coefficients. The fluids gave traction coefficients as measured by The PCS Instruments MTM2™ as of the order of 0.02 which is half the typical value reported for PAO fluids (4). Optical measurements to determine their elastohydrodynamic (EHD) lubrication properties showed that they did not form films thick enough to be measured. High-pressure rheology measurements taken on the fluid showed that these types of fluids were largely incompressible and had no piezo-viscous characteristics. Without suitable piezo-viscosity, lubricants are not capable of generating EHD films and cannot separate the surfaces. A 4-ball

wear test carried out on the same fluid, resulted in a wear scar of ~2 mm, compared to about 0.8 mm for a neat PAO fluid. This type of behaviour showed that going to a low traction fluid to improve energy efficiency, cannot be done at the expense of film forming. This is illustrated in the Stribeck curve in Figure 2, showing zone of optimum lubrication in which the film formed is thick enough to fully separate the surfaces, prevent wear but not so thick that churning losses predominate.

Also an attempt was made to determine the traction coefficients of some different greases. This proved to be a challenge. The first issue was controlling the temperature. As grease is an insulator with poor heat convection characteristics, heating up and controlling the temperature of the grease during the test was difficult. For greases running between room temperature and 80 °C, samples were pre- heated and incubated at temperature in an oven, prior to apply to the test pieces. Above this upper temperature, it was not possible to maintain stable temperature. The second issue was starvation. In both traction and optical EHD testing, it is very difficult to get the grease to behave like it would in a bearing. In the test, the contact pushes the grease away and as the grease is on a horizontal flat, it cannot flow back into the contact and has to be pushed back by a wiper. This creates artificial conditions, which need further development.
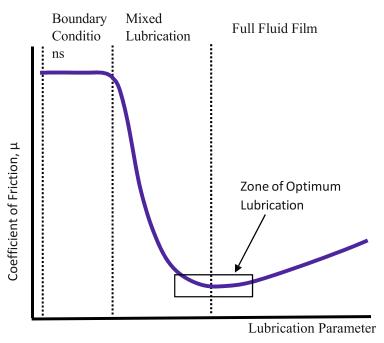

Figure 2 Stribeck curve showing zone of optimum lubrication

Film Thickness Effects

Historically it has always been reported that with greases, it is the oil that does the lubrication and the thickener plays no role in the lubrication. Worked carried by Cann and Hurley (10) showed that under the conditions of their testing that their greases built up thicker films than when testing just base oils alone. This was also supported when looking at the lubrication properties of CVJ greases using the optical EHD tester (8). A recent publication by SKF (11) suggested that at higher speeds, the film thickness equates to the viscosity of the base oil.

Looking at the SKF data (11) and that presented by Yamamoto (4), greases still form slightly thicker films than the base oil alone. The most interesting feature of both the SKF (11) and Yamamoto (4) work was that at low speed, greases formed much thicker films than was expected. Yamamoto (4) compared lithium soap greases with a urea-thickened grease and saw that at an entrainment velocity of 0.01 m/s, the base oil gave a central film thickness of 20 nm, the two lithium soaps about 60 nm and that of the urea about 120 nm. Similar thicker films were reported by SKF (11). This has important consideration when developing more energy efficient greases.

Rheology and Consistency

How stiff the grease is and how easily it flows are important considerations for the development of energy efficient greases. Another important factor is how the grease behaves under shear. Most greases soften when subject to shear, either in by working in the laboratory or in a component or bearing application. For an NLGI #2 grease, it is clear that comparing greases by their penetration grades will not give any indication about how they will affect efficiency. As greases run-in, the apparent viscosity typically becomes lower and the losses to churning are reduced. All greases behave differently and take different lengths of time and shear cycles to stabilise when subject to shear. The apparent viscosity of the grease is important, but not the whole answer, as shear history also plays a role. What is clear is that testing greases when they are newly applied to bearings or components, before they have chance to shear soften will give a different outcome of any energy efficiency testing. Figure 3 illustrates the shear softening behaviour of grease.
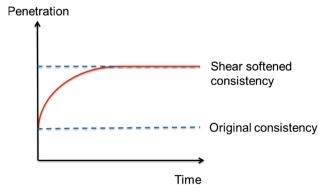

Figure 3 Shear softening behaviour of grease

It has been observed that urea greases with "grains of rice" or "rice pudding" structures can undergo temporary loss of consistency when subjected to shear. They can soften by as much as 120 penetration points when worked for 100,000 double strokes in a grease worker. Leaving the grease to sit without shear allows the grease to recover up to 100 penetration points. Urea greases can also have fibre-like structures similar to those of lithium soap greases. In the case of these fibre urea greases they shear soften up to 120 penetration points, but do not recover on standing. These fibrous types of ureas typically have lower thickener contents (~10%wt) than "rice pudding" urea greases (~12%wt). When this type of fibrous urea thickener was used for CVJ greases (6) they contributed to energy efficiency. The greases started with a grade #2 consistency. When pumped and assembled into CVJ joints the softened

to NLGI #1. When worked in the CVJ they softened to between 0 and 00 grades. Unpublished temperature data showed that the softening contributed to a lower running temperature, reduced plunging resistance and lower vibration transmission compared to greases that did not soften such as anhydrous calcium or lithium soap-thickened greases. Traditional calcium complex thickeners used in CVJ greases were developed which softened 100 penetration points and mimicked the behaviour of the fibrous urea greases. One challenge for both of these types of grease that readily softened was keeping the greases sealed in. Improved sealing systems were developed which also allowed the use of shear stable NLGI 0 grade lithium soap greases, which also contributed to energy efficiency and reduced noise and vibration. Lithium soap greases are also slightly different, depending on how they are made. If rapidly quenched lithium soap greases will have predominance of small fibres. These do not thicken the oil very well with poorer yields and higher soap contents but have lower bleed and better shear softening resistance. In terms of efficiency the higher soap content of quenched soap greases leads to higher churning losses. Slow cooling of molten lithium soap results in the formation of larger fibres. These thicken better than the smaller fibres but do not have the shear stability or lower bleed of the small fibres. Greases that bleed small amounts of oil in a consistent manner have been shown to give better lubrication properties than those that do not readily bleed oil (12). Having steady bleed of oil contributes to thicker EHD films being formed. In mixed lubrication, this reduces the metal-metal contact and in turn reduces frictional losses and improves efficiency. Manufacturing simple lithium soap greases with a partial quench results in a balance of yield, bleed and rheological properties leading to good lubrication and better efficiency. Lithium complex greases with that have been made with a well- controlled complexing reaction typically shear soften slightly by up to 30 penetration points. Higher levels of complexing acids produce higher dropping points but also higher thickener contents for the same NLGI grade, which can negatively affect the energy efficiency.

Grease Fill Effects

Another issue related to greasing is the amount of grease that is packed into the bearing or component. It is customary to pack only 20 to 50% of the available free volume of a bearing with grease. In the case of CV joints around 30% of the volume is filled. Figure 4 illustrates what happens to the running temperature and efficiency of greased bearings and components. If the grease is significantly over-packed, a thermal runaway will result and the component will fail prematurely. If the grease is slightly over-packed or a grease with too high apparent or base oil viscosity is selected, then the bearing will run hot. When bearings are packed properly, the component will heat up as it is running in. The grease thickener will shear down and additives will react to form a protective anti-wear layer. The component will then absorb less energy as it operates and it will cool down to a stable steady state running temperature. Towards the end of the greases life, it is no longer able to lubricate effectively and as is seen with most grease component tests, the temperature starts to rise as the grease breaks down. With energy efficient greases, the amount of running in and temperature rise at the start of operation is minimal. The surfaces are protected and the components have settled into stable running.
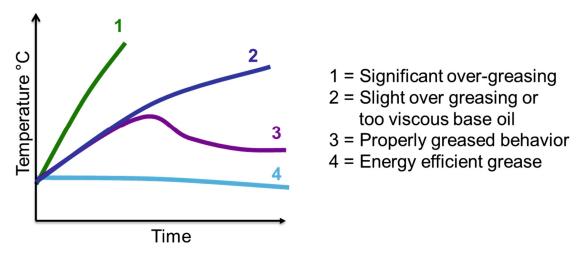

Figure 4 Grease effects on bearing running temperature

Component and Bearing Testing

The only true way of measuring the efficiency of a system is to measure the energy put in and the useful energy coming out. In the case of a bearing or automotive component it is torque in torque out. Most automotive components have air flowing over them when in service and this needs to be built into the test rig used to evaluate the components. For stationary industrial components, only the ambient air temperature would need to be controlled. A schematic of a test stand is illustrated in Figure 5.
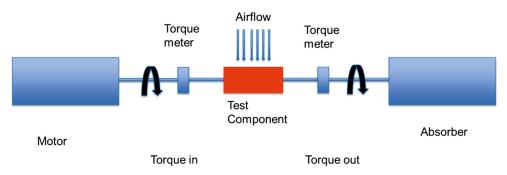
Figure 5 Energy efficiency measurement of components

Measuring the small torque losses in a single highly efficient small sized (6204) deep groove ball bearing is very challenging. Yamamoto (4) carried his measurements out on a pair of 6204 bearings to improve the sensing and separation of different greases tested. His initial results showed that after running in there was no measurable difference in the friction torque of two greases with different fibre structures, but confirmed that the amount of thickener influences the churning losses, showing that the higher the thickener content, the less efficient the grease was. He also confirmed that by optimising the base type and viscosity, thickener system and amount that improved more energy efficient greases could be developed for deep groove ball bearings.

In many applications, tapered roller bearings are used. These have significantly more sliding contacts than in deep groove ball bearings. A pair of tapered roller bearings were run for 500 hours in an FE8 test under a 20 kN load at 750 rpm to simulate a commercial vehicle wheel bearing test. The friction torque and temperatures of the two bearings were monitored throughout the test and are reported in Figure 6. The results show that it takes more than 150 hours to reach steady state in this test. A second wheel bearing grease was tested and gave similar torques and temperatures.
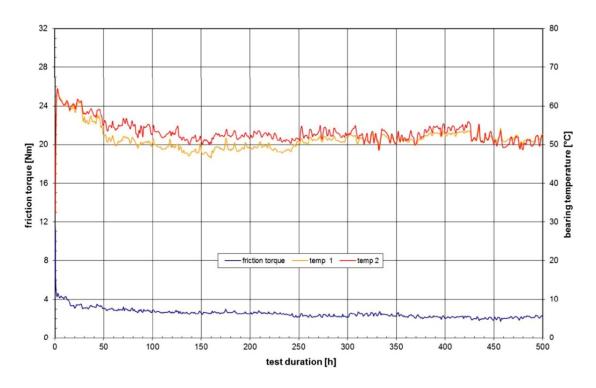
Figure 6 Friction torque and bearing temperature in an FE8 tapered roller bearing test

To try to get better discrimination between greases, it is necessary to couple more bearings together to magnify the individual losses. Work is ongoing to using a four bearing test machine, to measure the efficiency of greases used in bearings.

Figure 7 Schematic of the 4 tapered roller bearing tester

Initial work focussed on setting up the test rig to measure the effect of pre-loading on performance. The rig was originally designed to run oil and therefore oils were initially used to investigate efficiency. The bearings were first run-in to remove the effects seen in the FE8 test. An SAE 75W90 was run under different loading conditions with a speed ramp. It was observed that the lower the pre-load, the lower the torque losses. Reducing oil viscosity was investigated by running the same oil at higher temperatures. At the lower temperature the film thickness was sufficient to separate the surfaces fully but at the higher temperature, the losses increased suggesting that the surfaces were running under mixed lubrication. Comparing oils of three different viscosities showed similar results. At lower speed the thinnest fluid obtained the highest torque losses but at higher speed gave the lowest losses. Cann (10) had reported that after running-in, greases typically see a "parched" lubricant flow between that of fully flooded and starved. Some tests were run at restricted oil flow to try to simulate this behaviour. The fully flooded bearings showed higher losses than those with restricted flow. Initially work on greases showed that consistently packing the bearings is a problem. A grease packer was machined to facilitate uniform grease distribution in test bearings. A series of greases have been defined and are in the process of being made and tested. These greases look at the effects of base oil type: group II paraffinic mineral oil and synthetic hydrocarbon (PAO); viscosity ISO VG 68 and ISO VG 220; lithium complex and urea thickeners. The effect of low friction additives will also be investigated by running greases with and without the LF4 package described above. A summary of the greases being tested is in table 5. This work is ongoing and the work will be reported when completed.

Grease	Base oil type	Viscosity at 40 °C	Thickener	Additives
1	Mineral Group II	68	Lithium complex	No
2	Mineral Group II	68	Lithium complex	LF4
3	Poly alpha olefin	68	Lithium complex	No
4	Poly alpha olefin	68	Lithium complex	LF4
5	Mineral Group II	220	Lithium complex	No
6	Mineral Group II	220	Lithium complex	LF4
7	Poly alpha olefin	220	Lithium complex	No
8	Poly alpha olefin	220	Lithium complex	LF4
9	Mineral Group II	68	Di-urea	No
10	Mineral Group II	68	Di-urea	LF4
11	Poly alpha olefin	68	Di-urea	No
12	Poly alpha olefin	68	Di-urea	LF4
13	Mineral Group II	220	Di-urea	No
14	Mineral Group II	220	Di-urea	LF4
15	Poly alpha olefin	220	Di-urea	No
16	Poly alpha olefin	220	Di-urea	LF4

Table 5 - Energy efficient greases matrix

Summary

This work and that of others has shown that measuring the efficiency of greases is a particular challenge. The energy losses in small deep groove ball bearings are very small and difficult to quantify. Moving to bearings with more sliding has led to the development of a modified test machine for measuring the energy efficiency of tapered roller bearings.

In bearings and components with sliding it has also been showed that it is necessary to apply molybdenum complex containing formulations to achieve low friction coefficients. The complexes do not work on their own but have to be combined with other surface active additives.

Conclusions

By optimising the lubrication of the bearings, theoretical improvements in energy efficiency are possible, but as bearings are extremely efficient, measuring the losses is a significant challenge.

This work has shown that using molybdenum additives continues to be the best way to achieve low friction coefficients in grease, and that their combination with other molybdenum-free additives provides an effective way to formulate optimized low friction additive packages.

Acknowledgements

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Performance Case Study For Modified Calcium Suphonate Grease With Improved Pumpability And Heat Resistance Characteristics In Continuous Casting Shop In Integradted Steel Plants
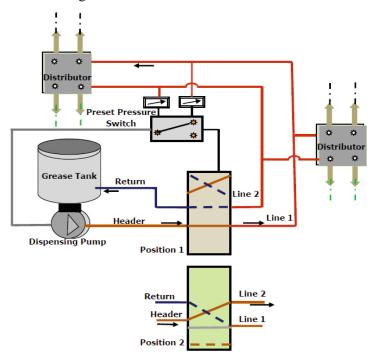
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ABSTRACT

In continuous casting mill, hot slabs passes through Mould, Rollers of Benders and Segments where rolling elements are subjected to very high temperature and ingress of large quantity of water. Traditionally Original Equipment Manufacturer (OE) recommends lithium base or lithium complex grease with or without molybdenum disulfide for lubrication of such rolling element through centralized lubrication system. Lithium complex has certain limitations in its performance in an environment where high load, extreme hot environment & likely water contamination are unavoidable. This leads to major problem of retention of grease in bearing housing, leakages in spool of grease distributor, excessive consumption, etc. Hence designing of grease for such aggressive application environment is very critical and always remains a great challenge. Balmer Lawrie through development of modified calcium sulphonate grease has created a lubrication solution to address the said problems generally encountered with lithium soap based greases in major steel plants. The modified grease with enhanced retention property , controlled pumpability, excellent temperature & water resistant characteristics was successfully field trialed & established its superior performance at integrated steel plant to improve upon grease consumption rate, bearing failure with better retention of grease in bearing housing, problem of leakages in spool of grease distributor, etc. and thereby decreasing maintenance downtime and increasing productivity in particular.

Introduction

Continuous caster route in steel production is largely followed since last few decades and today over 50% of steel is produced in continuous steel caster. In contiguous steel casting, the molten metal is poured into stationary mould and passes through bender and segments etc. The rolling elements in bender and segments are subjected to high loads and elevated temperatures with low rotational speed. They are also subjected to continuous secondary cooling water environment. In such hostile environment, proper lubrication is critical to performance and productivity of the caster. Also, centralized lubrication system is most widely method adopted in continuous steel casting roller

Schematic Dual Line Dispensing System

elements lubrication where a typically small quantity of grease is dispensed with at an increased frequency to drive out the old grease and replenish the bearing with fresh grease. In such cases, specific grease needs to be developed to affect increased productivity and overall performance of the system.

Lubrication system and Grease Design Architecture

Dual line centralized lubrication system in continuous steel caster comprises of single pump station, control panel, divider valves, distributors, end-of-line pressure switches and tubing to feed to large

no of lubrication points. The total length of tubing counts as long as 200 – 500 mtrs or more and the grease is subjected to severe pressure to travel to all such lube points through long tubing. It experiences temperature variation during the passage. Considering the requirement of typically small quantity at a time and to effect functioning end-of-line pressure switch to trigger a signal, grease in the holding line must withstand high pressure in the feed line without oil separation. Since the feed per point per shot is very small, it has to meet certain characteristics like surface adhesion, high stability to water contamination, high temperature etc to stay in place for lubrication till renewal with fresh grease. Accordingly, modification of architecture of the sulphonate grease was undertaken and necessary characteristics were suitably incorporated prior to establish in continuous steel casting roller element bearing.

Balmer Lawrie Approach in Problem Identification & Development

Modified Ca-Sulphonate grease was developed and was established in Continuous Casting shop in Strand 2 in Segments, bender & mould through rigorous trial in an Integrated Steel Plant and was subsequently extended to other strands as well to prove its integrity with the system. Centralized lubrication is the most effective way to lubricate all remotely located lube service points at a preset interval with right quantity of lubricant and is one of the most important issues in operation and maintenance of bearing roller elements. Hence the efficient running of the CLS largely depends on judicious selection of proper characteristics and texture of the candidate grease. Balmer Lawrie, prior to undertake the development of suitable grease made a thorough study of the CLS system in SMS2 with twin line double caster, nature of failures and explored all avenues to assess the possible causes of failure of OEM recommended conventional Lithium based grease as well as Sulphonated grease of different sources. Based on rigorous trial observations, the developed Ca-sulphonate grease was further modified and customized to suit the existing CLS requirement to deliver the grease to all remote lube points without compromising on performance requirement of the grease at the hostile application points.

Development Matrices of Modified Calcium Sulphonate Grease.

The major difficulties with conventional lithium base grease are repeated failure of auto cut-out switch as well as difficulties in creating a lubricant film between the bearing's rollers and raceways. It had resulted in increased level of consumptions and increased bearing failure. Though ca-sulphonate grease has reported interesting extreme pressure performance, it lacks in water spray off resistance and similar performance problem along with pumpability issue was also experienced with conventional sulphonate grease formulation. The operating temperature variance in most continuous casters ranges from 60-80 deg C to sometimes peaking at over 130 deg C. Keeping the above factors and pumpability in mind, the development was undertaken with moderately high viscosity base oil sulphonate greases for initial trial in segment /bender bearing roller elements to aid in sealing as well as stay in roller element. Also, attempt was made with development of high temperature formulation of the candidate grease for initial trial to offset effect of the water washout and rust inhibition characteristics. The initial properties of developed high temperature candidate sulphonate grease subjected to initial trial and subsequent modified Ca-sulphonate grease are tabulated as under:

TYPICAL PROPERTIES

S/N	Description	Initital Grease	Modified Grease	Test Method
1	NLGI Grade	2/1	2/1	
2	Texture	Smooth & Buttery	Smooth & Buttery	
3	Thickener Type	Ca-Sulphonate Complex	Ca-Sulphonate Complex	
4	Base Oil Type	Mineral	Mineral	
5	Worked Penetration @60X @25 degC	295 - 300	300 - 310	ASTM D 217
6	Change in Penetration @100X @25 degC	$\pm$ 13%	$\pm$ 10%	ASTM D 217
7	Dropping Point, deg C	> 320	> 320	ASTM D 2265
8	Water washout @80 degC, 1% loss	5.0	1.0	ASTM D 1264
9	Four Ball weld Load, kgs	315	400	IP 239
10	Bomb Oxidation, ΔPsi @100 hrs	5.0	4.0	ASTM D 942

Performance Result, Benefits and Interpretation

Modified candidate Ca-Sulphonate grease has shown an encouraging performance improvement in areas of difficulties with previous similar base grease and OEM recommended lithium base grease as under

1. Good pumpability ie transferring of the modified sulphonate grease from barrel to grease reservoir as well as dispensing in the dual line headers through the diversion valve and tubing to the distributors
2. Texture of the modified grease showed proper functioning of main dispensing pump and end-of-line pressure switch as per preset line pressure
3. Good water resistance characteristics and improvement in surface adhesion characteristics in the modified grease demonstrated no leakages in spool of grease distributor and consumptions have reduced to the tune of approx.15%

4. A balance high temperature properties eliminated problems of roll jamming of segments, reduced bearing failure and scale deposition
5. Improvement in water resistance properties ensured retention at the bearing roller element which established desired group current of roll drive and reduced bearing failure

Conclusion

Based on preliminary field trial at continuous steel casting in integrated steel plant using dual line grease dispensing system, candidate Ca-sulphonate grease formulation was reviewed and was modified to overcome the perennial problems being faced with OEM recommended lithium grease as well as conventional Ca-sulphonate grease. The architectural modification in the formulation in respect of its enhanced retention characteristics, water resistance and load carrying properties, balanced tackiness and most importantly its uninterrupted pumpability has eliminated existing problems and demonstrated a superior performance for such hostile environment. The modified ca- sulphonate grease, customized to specific applications is now being regularly used and has reserved the status of 'Approved Grease'.

Acknowledgement

Bio based grease from Neem oil.

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Abstract

Finite resources of crude petroleum oil and growing concern over climate change are driving investment and innovation in the sector of biofuels. Bio based lubricants are an attractive alternative to conventional petro based lubricants due to a number of their physical properties including: renewability, biodegradability, high lubricity and high flash points. Vegetable oils in comparison with mineral oils are renewable, easily available, environmentally friendly, economically cheaper than mineral oil and hence sustainable indeed. This paper presents a series of structural modifications of vegetable oil- Neem oil using various alcohols to form fatty acid esters. This fatty acid esters were then used as base oil in the formulation of grease using lithium-12-hydroxy stearate as thickener. Formulated bio-based grease was then evaluated for its tribological properties against mineral oil based grease.

Key words: Vegetable oil, Grease, Cone Penetration, Wear Scar, Roll Stability, Weld load.

1. Introduction

Global grease consumption is estimated at 1,296 KT of which industrial applications accounts for 691 KT. Mineral oil based greases account for close to 90% of the global demand, while 9 % of synthetic esters are used and only 1% of bio degradable base oils are used for manufacturing of greases. It is assumed that global consumption of grease in industrial application will be up to 758 KT till 2017[1].All over the globe usage and disposal of lubricants has to satisfy the need to protect our nature in general and of all the living beings in special. For all cases of direct contact between lubricants on one side and human beings and the nature on the other side the compatibility has to be checked. The necessity for eco-friendly nature has to be understood by all those who are working in the fields of lubricants [2].

Also limited sources of petroleum oil and its effects over climate change makes everyonelook towards renewable resources as they are easily available, environmental friendly and hence sustainable indeed. Vegetable oils are increasingly used as green raw materials in various areas of research in bio based fuels [3]. Vegetable oils in comparison with mineral oils have different properties due to presence of triglyceride in its chemical structure [4]. They have good lubricity, viscosity indices, superior anticorrosion property and affinity to metal surfaces. High value of flash point over 300ºC classify vegetable oils to non- flammable liquids [5].Industrially, most fatty acids are obtained from animal or vegetable sources. It is possible to produce several industrial products from vegetable oils and fat. Vegetable oils as lubricants are preferred because they are biodegradable and non-toxic, unlike conventional mineral oil [6].

Researcher have developed various routes to use vegetable oil based bio-lubricants by chemical modifications of the natural oil or fats. These modifications involves trans-esterification [7], Epoxidation [8], enzyme catalyzed esterification [9] and esteloid formation [10]. Traditional method for formulating greaseusingthickeners is also reported by number of authors in which mineral oils are replaced by vegetable oils and fats [11-12]. Modifications of vegetable oils improves the properties related to viscosity, adherence to the metal surfaces, stability etc. which impacts its tribological properties.

In the present work the attempt has been done to produce a base stock for lubricating grease by chemical modification of Neem oil.Neem is a medium sized tree and is found throughout India. Biologically it is named as *Azadirachitaindica*. It is easy-growing tree for tropical and sub-tropical regions and can be easily grown-up form its seeds. Oil content in its seeds varies from 20- 27%.Neem oil was converted into its methyl ester which were then further modified by trans-esterification with different alcohols viz. Hexanol, Octanol and NPG. Trans-esterified products were characterized by GC-MS. These esters of Neem oil were then thermo mechanically dispersed with lithium-12-hydorxystearate to formulate grease. All-purpose grease of mineral oil based type SN-500 was prepared andused as standard to compare various properties of bio-lubricants.

2. Materials and method

All the chemicals were procured from SD Fine chemical Baroda were of laboratory reagent grade and were used without any further purification. Neem oil was procured from Anand Agriculture University. Lithium-12-hydorxy stearate was procured from Petrochem Industries, Palghar. It was evaluated for Iodine value, Saponification value and Peroxide value as per ASTM D5554, ASTM D5558 and ISO 3960 respectively. Fatty acid profile was evaluated by Gas Chromatography. Tribological properties such as roll stability, cone penetration, drop point, wear preventative and weld load were determined by ASTM D1831 (Roll stability for 2 hours), ASTM D1403 (Quarter Cone),ASTM D566 (Drop point), ASTM D 2266 (Wear preventative) and ASTM D 2596 (Weld load) respectively.All the test samples were taken as raw sample, no additives were added in any of the samples.

2.1 Gas Chromatography analysis:

Gas chromatography of Neem oil was carried out using Perkin Elmer Auto System XL.

The column used was BP-225, having 25 meters length and 250 mm diameter

The operating conditions of GC for Neem oil are as follows:

Injector Temperature	250^0 Celsius
Detector Temperature	250^0 Celsius
Oven Temperature	60^0 for 5 minutes the rate of increase in temperature was 10^0 per minute and the sample was held at 220^0 for 10 minutes
Carrier Gas	Nitrogen
Injector Volume	1 microliter
Carrier flow	6 psi

2.2 Synthesis of Neem oil methyl esters

Neem oil was esterified with methanol to form Neem Oil Methyl Esters (NOME). Catalyst used in this process was NaOH, 1%w/v, Oil to Alcohol ratio was kept as 1:6. Temperature of the reaction mass was maintained at 60-65 degree Celsius for 60 minutes [13]. After the completion of reaction the mixture of methyl esters and glycerol was separated in a separating funnel overnight. The methyl ester thus synthesized was used for the production of lubricating grease base stock.

2.3 Synthesis of Lubricating grease base stock- Transesterification of Neem oil methyl esters

The methyl esters were further trans esterified with various alcohols such as Hexanol, Octanol, Neo-pentyl glycol to form various high molecular weight esters- lubricating grease base stock. The reaction was carried out at desired temperature and pressure conditions using 3% sodium methoxide as catalyst. Entire reaction was conducted under vacuum which simultaneously removed the byproduct. The product obtained was characterized by GC-MS and its temperature. It was further used as base oil in formulation of grease. The representative structures of methyl esters of Neem oil and Hexyl, Octyl and Neo pentyl glycol esters of Neem oil are shown in scheme 1, 2 and 3 respectively.

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Scheme 1. (a) Methyl ester formation from Neem oil.

(b) Hexyl ester formation from Methyl esters of Neem Oil.

Scheme 2. (a) Methyl ester formation from Neem oil.

   (b) Octyl ester formation from Methyl esters of Neem Oil.

(a) Esterification

Neem Oil + Methanol

Methyl ester + Glycerol

(b) Transesterification

Methyl ester + Neo pentyl glycol

Neo pentyl glycol ester + Methanol

Scheme 3. (a) Methyl ester formation from Neem oil.

(b) Neo pentyl glycol ester formation from Methyl esters of Neem Oil.

2.4 **Formulation of bio based grease**

Trans-esterified esters of Neem oil formed using Hexanol, Octanol and Neo-pentyl glycol were further used as base oil in formulating grease. Grease was prepared using lithium-12- hydroxy stearate as preformed soap. Preformed soap was thermo mechanically dispersed using high speed stirrer. 10% w/w of the preformed soap was used to formulate bio based grease, which was further tested for its tribological properties.

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2.5 Gas Chromatography-Mass Spectroscopy analysis

GC-MS analysis of all Neem oil esters was carried out on Perkin Elmer Auto system XL with turbo mass. The column used was PE-5MS

The operating conditions of GC-MS for the Neem oil esters are as follows:

Column   PE-5MS

Column Length   30 meters

Injector Temperature 250^0 Celsius

Oven Temperature 80^0 Celsius for 5 minutes, the rate of increase in temperature was of 10^0 Celsius per minute and the sample was held at 290^0 Celsius for 10 minutes

Flow Rate   1ml per minute

E.I Source Temperature 220^0 Celsius per minute

Transfer line temperature 250^0 Celsius per minute

Mass range   20 to 620 AMU

Split ratio   1:70

2.6 Drop point

The dropping point of grease is the temperature at which the thickener loses its ability to maintain the base oil within the thickener matrix. This may be due to the thickener melting or the oil becoming so thin that the surface tension and capillary action become insufficient to hold the oil within the thickener matrix. Drop point of all the test samples were determined using ASTM D566 method.

2.7 **Cone penetration**

Lubricating greases consistency is measured by ASTM D 1403 method, and is reported in terms of various NLGI grades ranging from 000 for semi fluid to 6 for solid block greases.

2.7.1 **Un-Worked Penetration Test**

All the three samples of Neem oil ester based grease were subjected to penetration test. Grease cup, cylindrical in shape with 50ml capacity at 25^0 C, was filled completely with grease and its surface was smoothened and placed on the penetrometer. Then penetrometer cone was released and allowed to sink in the grease cup under its own weight for 5 seconds.

The depth the cone has penetrated is then measured, in tenths of a millimeter. Deeper the cone penetrates in the grease cup, higher is its penetration result and the softer the grease.

2.7.2 **Worked Penetration Test**

The worked penetration test was carried out in the same manner as that of un-worked penetration test but prior to penetration the sample was been subjected to 60 double strokes in the ASTM grease worker. In this method, the disturbance of the grease was standardized by the prescribed working process and is more reliable than Un-worked penetration test because the grease has been subjected to a work load of double strokes.

The un-worked and the worked penetration tests were performed to determine the consistency of grease and its resistance to deformation when subjected to force. Significant difference in penetration of cone within un-worked and worked grease indicates its poor shear stability.

2.8 **Roll Stability of grease**

Mechanical stability of the grease was evaluated using ASTM D 1831 roll stability worker. In the Roll Stability Test agrease is rolled at 165 rpm for a 2hrs at 25^0 C using a steel cylinder which contains a 5 kg heavy round steel block. The change in cone penetration is used to measure mechanical stability. Due to the small size of the sample, working and penetrations are performed on ASTM D 1403 one-quarter or one-half scale equipment.

2.9 **Wear Preventative**

The four-ball wear-test method isperformed to determine the wear-preventing characteristic of greases under the test conditions.Three, ½ inch diameter steel balls are clamped together and covered with the test grease. The grease is heated to 75°C, and then a forth identical ball is pressed downward with a force of 40 kgf into the cavity formed by the three clamped, stationary balls. This arrangement forms a three-point contact. The top ball is then rotated at 1200 rpm for 60 minutes. Scar on the three steady balls is measured using a microscope.

2.10 **Weld load**

The test is used to determine the load carrying properties of a lubricant at high test loads. Four-Ball Test measures a lubricant's extreme pressure properties under Hertzian contact in sliding or rolling motion. Weld load ofgrease is tested in a four ball system where a rotating ball slides over three stationary balls. This test is run at higher loads till the test balls are welded together.

ASTM D2596 method is used to determine the weld load. A 12.7 mm (0.5 inch) steel ball is rotated against three stationary balls of the same description. Lubricating grease is applied over the balls. Common test conditions are 1200 rpm, 75°C, 60 minutes, and 40 kg load.

3. Results and Discussion

The carboxylic acid groups present in the fatty acid chains were converted in to methyl esters. These methyl esters were then transesterified with higher alcohols viz. Hexanol, Octanol and Neo-pentyl glycol. The resultant transesterified products were then compared with HP all- purpose grease for performance characteristics.

3.1 **Physicochemical properties**

Physicochemical properties of Neem oil viz. Saponification value, Iodine value and Peroxide value were calculated which are tabulated in table 1.

Table 1. Physicochemical properties of Neem oil.

Saponification Value	159
Iodine value	74.17
Peroxide value	02.02

3.2 **Fatty acid profile**

Fatty acid profile of Neem oil was done by Gas Chromatography. The results of fatty acid profile shows the presence of oleic acid upto 50.10%. The data of other fatty acids are tabulated in table 2.
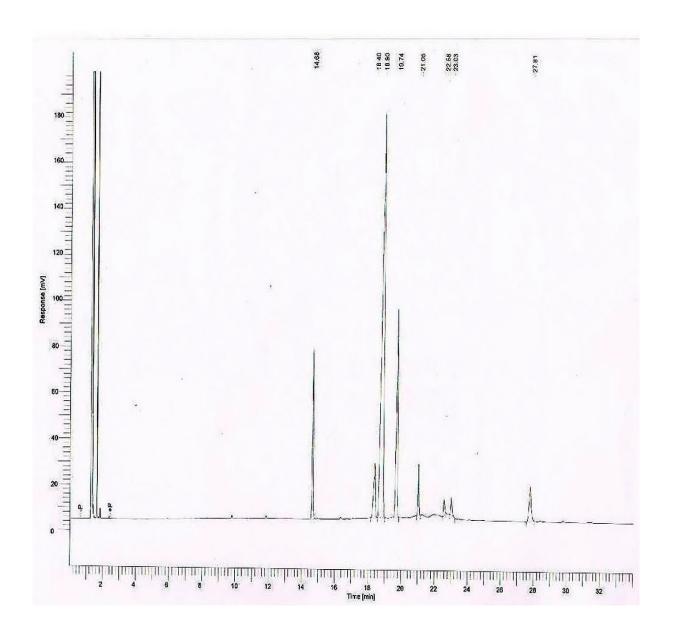
Fig. 1. Gas Chromatogram of Neem oil

Fig.1. is the gas chromatogram of Neem oil in which peak at time 14.677 is of palmitic acid, peak at 18.40 is of stearic acid, peak at time 18.902 is of oleic acid and peak at time 19.743 is of linoleic acid. Percentage of fatty acids in Neem oil are tabulated in table 2.

Table 2. Fatty acid composition in percentage

Fatty Acid	Percentage
Palmitic Acid	17.31
Stearic Acid	16.44
Oleic Acid	50.10
Linoleic Acid	13.13
Linolinic Acid	0.45
Unknown Fatty Acids	2.75

3.3 Gas Chromatography-mass spectrometry was carried out of various Neem oil esters, which are the desired products and can replace mineral oil based lubricants. The percentage conversion of methyl ester of Neem oil to various esters is shown in table 3.

Table 3. Percentage conversion of Neem oil methyl esters to various transesterified esters.

Starting material from which esters were synthesized		Yield (%)
Neem oil methyl ester	Hexanol	94.50
Neem oil methyl ester	Octanol	93.10
Neem oil methyl ester	Neopentyl glycol	95.00

3.4 **Drop Point**

Dropping point is the temperature at which the grease passes from a semisolid to a liquid state under the conditions of test. This change in state is typical of grease containing as thicken-

ers soaps of conventional types. Drop point of Neem oil ester based grease were measured using ASTM D566 method which are tabulated below in table 3.

Table 4. Drop point values of various vegetable oil based grease

Greases	Drop point
Lithium grease using Neem oil hexyl ester as base oil	167^0 C
Lithium grease using Neem oil octyl ester as base oil	178^0 C
Lithium grease using Neem oil neo pentyl glycol ester as base oil	144^0 C
All-purpose lithium grease using mineral base oil	195^0 C

From the results it is observed that lithium-12-hydroxystearate based grease from Neem oil esters have comparatively less dropping point than the mineral oil based grease.

3.5 **Cone penetration**

In the case of lubricating greases its consistency is termed as its measure of the relative hardness or softness and has some relation to its rheological properties. A proper consistency will make the grease stay in the bearing without generating too much friction. Hardness or softness of lubricating greases can be checked using ASTM D 1403 cone penetration method.On the basis of ASTM worked penetrations, NLGI has standardized a numerical scale for classifying the consistency of greases in order of increasing hardness, the consistency numbers are tabulated below:

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Table 5. NLGI grade and its standard penetration results

Classification of greases by NLGI consistency number		
NLGI number	ASTM worked penetration $(10^{-1}$ mm$)$	Appearance at room temperature
000	445–475	very fluid
00	400–430	fluid
0	355–385	semi-fluid
1	310–340	very soft
2	265–295	soft
3	220–250	medium hard
4	175–205	hard
5	130–160	very hard
6	85–115	extremely hard

Worked and unworked penetration results of the Neem oil ester based greases are tabulated below:

Table 6 Worked and Un-worked penetration results

Lithium grease from various base oils.	ASTM D 1403 Cone penetration		Difference	Percentage Change
	Unworked Penetration	Worked Penetration		
Neem oil hexyl ester	301.25	305.5	3.75	1.22
Neem oil octyl ester	140.25	237.75	97.5	69.51
Neem oil neo pentyl glycol ester	166.5	174	7.5	4.50
Mineral oil (SN 500)	234	264	30	12.82

From the above results it can be observed that grease made using Neem oil hexyl ester as well as neem oil neo pentyl glycol ester based lithium greases has goodconsistency than mineral oil based lithium grease.

3.6 Roll stability

The ability of a grease to resist changes in consistency during mechanical working is named its roll stability or shear stability.Mechanical stability is a significant characteristic of lubricating grease when dealing with the long-term service of grease-lubricated roller bearings. Consistency of greases, in service, generally decreases as it is exposed to mechanical forces caused due to churning action in rolling parts of the bearing. Poor stability will lead to degradation of the grease which results into leakage of grease through seals, or at worst a total failure of the bearing.

Table 7 Worked and Un-worked penetration results after roll stability

Lithium grease from various base oils.	ASTM D 1403 Cone penetration		Difference	Percentage Change
	Quarter cone worked penetration (60 strokes)	Quarter cone worked penetration (60 strokes) after roll stability		
Neem oil hexyl ester	74	65	9	12.16
Neem oil octyl ester	57	56	1	1.75
Neem oil neo pentyl glycol ester	40	50	10	25
Mineral oil (SN 500)	64	74	10	15.62

From the above results it can be observed that the Neem oil hexyl ester as well as neem oil octyl ester based lithium grease became thicker instead of becoming thinner, and hence its consistency is much better than mineral oil based lithium grease.

*3.7***Wear Scar**

The four-ball wear-test method is performed to determine the wear-preventing characteristic of greases under the test conditions.

Table 8 Wear Scar, Average co-efficient of friction and average torque results

Lithium grease using various base oils.	Ball 1		Ball 2		Ball 3		Average scar (mm)	Average Coefficient of friction	Average Torque
	X axis (mm)	Y axis (mm)	X axis (mm)	Y axis (mm)	X axis (mm)	Y axis (mm)			
Neem oil hexyl ester	0.927	0.972	1.017	1.033	0.969	0.982	0.983	0.106	1.924
Neem oil octyl ester	1.008	1.026	0.979	0.982	0.968	0.950	0.985	0.102	1.841
Neem oil npg ester	0.967	0.960	0.856	0.922	0.908	0.875	0.914	0.046	0.826
Mineral oil (SN 500)	0.694	0.801	0.732	0.62	0.937	0.998	0.7970	0.102	1.885

The results shown in the above table reveals that the Neem oil ester based lithium greases have satisfactory values of average scar which are comparable to mineral oil based grease sample. Hence neem oil ester based lithium greases have competitive wear preventative properties.

Co-efficient of friction and Torque generated values are also shown in the above table, from which it can be observed that Neem oil ester based lithium grease have good tribological properties and Neem oil neo pentyl glycol ester based lithium grease has the least co-efficient of friction and torque values.

3.8 **Weld load**

The test is used to determine the load barring properties of a lubricant at high test loads. Four- Ball Test measures a lubricant's extreme pressure properties under Hertzian contact in sliding or rolling motion. Weld load of grease is tested in a four ball system where a rotating ball slides over three stationary balls. This test is run at higher loads till the test balls are welded

together.The weld load of the Neem oil ester based lithium grease and that of mineral oil based lithium grease is shown in fig. 5
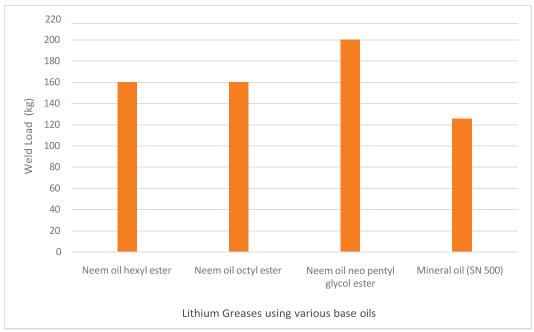

Fig. 5. Weld load values using four ball apparatus

From the above shown graph it can be observed that the weld load of all the Neem oil esters is much better than the mineral oil based grease. Amongst the three Neemoil esters, neo pentyl glycol ester of Neem oil has superior load carrying capacity. The weld load test of all the samples were taken in raw sample, no additives were added in any of the samples.

4. Conclusion:

Neem oil methyl esters can be successfully transesterified using various alcohols viz. Hexanol, Octanol and Neo-Pentyl Glycol. These transesterified esters can be used

as base oil in formulation of grease. These vegetable oil ester based grease exhibit comparative performance properties against mineral oil based grease, which can be enhanced by using various additives. Thus these esters derived from Neem oil- a renewable source, can be used as base stock to formulate lubricating grease. Use of such renewable resources for deriving a value added product like grease can eliminate various problems related to environment and depletion of finite resources; which are still in search of a legitimate solution.

5. Acknowledgement:

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