

Effect of nanoparticles on Tribological Study of Eco-Friendly Grease Made with Soybean Oil, Beeswax and Acacia Gum

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Abstract: The lubricating grease is simply a lubricating fluid which is gelled with a thickening agent such that the lubricant can be retained more readily in the specific required area. It is made of two primary ingredients: base oils and thickeners. Various performance additives are blended frequently to grease to tailor their characteristics. The grease used in industries is made of mineral oil which is non-renewable, non-biodegradable, toxic, bio-accumulative, and hence environmentally unfriendly. A potential, sustainable and economical eco-friendly alternative to harmful petroleum-based lubricants is a big challenge for the 21st-century lubricant industry. The present study explores, for the very first time, grease produced from all natural, ecofriendly material such as Soybean oil (SO), Beeswax (BW) and Acacia gum (AG). Various samples are prepared by varying the components ratios. Application of grease depends on the physiological properties of the grease. This paper mainly focuses on properties like cone penetration, NGLI Grade, drop point, Wear Test. The tests performed for evaluating the properties are Cone penetration (ASTM-D 217-2021-a), NLGI grade (ASTM-D 217-2020), Drop point (ASTM-D -2265-2020) and Wear preventive Characteristics (ASTM D2266).

Keywords - grease, soybean oil, beeswax, acacia gum, cone penetration, nlgi grade, drop point test, wear test, tribology.

I. INTRODUCTION

The principal function of any lubricant is to extend the life and improve the efficiency of mechanical devices by reducing friction and wear. Alternate functions include heat dissipation, corrosion protection, power transmission, and contaminant removal. Normally, fluid lubricants are difficult to keep at the point of application and must be replenished frequently. If, however, a fluid lubricant is thickened, its confinement is improved, and

lubrication intervals can be protracted. The lubricating grease in simple words is a lubricating fluid which has been blended with a thickening agent so that the lubricant can be retained more readily in the required area. This is not to say that the thickener does not play a part in the lubrication. Some thickeners will contribute in the lubrication usually depends upon the type of thickener being used and the lubricating regime [1]. Grease was previously defined as a gelled lubricating fluid. In spite of this simplistic definition conveys the general concept of grease, a more extensive discussion is required to provide a fuller understanding of what constitutes lubricating grease. Lubricating grease is a semi-fluid to solid product of a dispersion of a thickener in a liquid lubricant. Additives, either liquid or solid, are usually included to improve grease properties or performance [2]. By definition, grease is a lubricant. It is essentially a two-phase system - a liquid-phase lubricant into which a solid-phase finely divided thickener is uniformly dispersed. The liquid is immobilized by the thickener dispersion that must maintain stability with respect to time and usage [3]. At operating temperatures, thickeners are insoluble or, at most, only slightly soluble in the liquid lubricant. There should be some affinity between the solid thickener and the liquid lubricant in order to form a stable, gel-like structure. The thickener can be constituted of fibers (such as various metallic soaps), or plates or spheres (such as certain non-soap thickeners) [4]. The essential requirements are that the particles be extremely small, uniformly dispersed, and capable of forming a relatively stable, gel-like structure with the liquid lubricant [5,6].

Manufacturing of grease requires considerably more processing, usually including synthesizing the thickener in the fluid [7]. A strict time, temperature, and mixing profile must be followed to properly synthesize the thickener. Next, thorough mixing in the desired additives and blending at the proper time and temperature has to be done before the final finishing and processing [8]. The finishing and process steps includes homogenization, where the grease is passed through a mill to disperse the thickener and additives, or deaerating, to remove entrained air or both where needed. In the definition of lubricating grease given here, the liquid component of the grease might be a mineral oil or any fluid that has lubricating properties. The thickener may be any material that, in combination with the selected fluid, will produce the solid or semi-fluid structure. The other ingredients are additives or modifiers which are used to modify existing properties or impart special ones [9].

A. Fluid Components-

Most of the greases produced today have mineral oils as their fluid components. These oils may range in viscosity from as light as kerosene up to the heaviest cylinder stocks. In the case of some specialty greases, products such as waxes, petrolatum, or asphalts may be used [10].

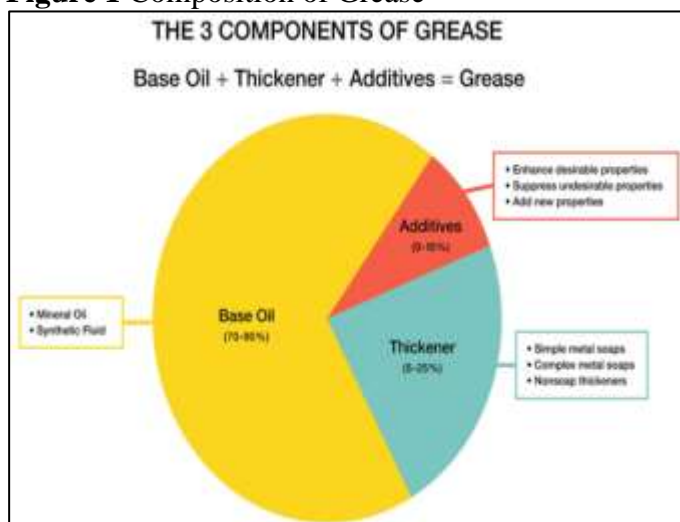
Although these latter materials may not be too accurately described as "liquid lubricants," they perform the same function as the fluid components in conventional greases. Greases made with mineral oils provide satisfactory performance in most automotive and industrial applications at very low or high temperature applications or in applications where temperature may vary over a wide range [11].

B. Thickeners-

The principal thickeners used in greases are metallic soaps. The earliest greases were made with calcium soaps, then greases made with sodium soaps were introduced. Later, soaps such as aluminum, lithium, and barium came into use. Some greases which are made with mixtures of soaps, such as sodium and calcium, are usually referred in as mixed base greases. Soaps made with other metals have been proposed for use in greases but have not received commercial acceptance, either because of cost or performance problems. Some lead soap are used as thickeners, but most lead and zinc soaps are used as modifying agents in greases. Modifications of metallic soap greases, called "complex" greases, are becoming increasingly popular. These complex greases are made by using a mixture of a conventional metallic soap forming material with complexing agent. These complexing agents might be either organic or

inorganic and may or may not comprise of another metallic constituent. Among the most successful of the complex greases are the calcium complex greases [12]. These are made with a synthesis of conventional calcium soap forming materials and an organic acid having low molecular weight as the complexing agent. Greases of this type are characterized by very high dropping points, usually above 500 (250°C), and may also have excellent load carrying properties. Other complex greases aluminium, barium, and lithium are also manufactured for certain applications. A number of non soap thickeners are in use, primarily for special applications. Modified bentonite (clay) and silica aerogel are used to manufacture non melting greases for high temperature applications. Since oxidation can still cause the oil component of these greases to deteriorate, regular relubrication is required. Thickeners such as polyurea, pigments, dyes, and various other fabricated materials are used to some extent. However, due to their higher costs, their use is somewhat restricted to applications where performance requirements are critical [13].

Figure 1 Composition of Grease



C. Additives And Modifiers-

Additives and modifiers commonly used in lubricating greases are oxidation or rust inhibitors, pour point depressants, extreme pressure anti-wear agents, lubricity or friction reducing agents, and dyes or pigments. Most of these materials have much the same function as similar material added to lubricating oils[14].

Unfortunately, the current grease industry is corrupted to a vast extent with mineral oil-based products[15], which are non-renewable, non-biodegradable, toxic, bio-accumulative, and hence environmentally unfriendly [16]. Synthetic oil-based greases (petroleum-derived) are biodegradable but non-renewable and highly expensive [17]. Apart from the base oils, the popular thickeners (metal soaps, metal complex soaps, and polyurea) [18] and additives [19] further contribute to the harmfulness of the grease. The only justification for using these harmful ingredients lies in the superior performance of the resulting products[20-22]. However, it should be understood that the recurrent production and consumption of such greases pose high risks to the land and aquatic life [123], as the former eventually end up in (or interact with) the environment. Further, they significantly contribute to depleting non-renewable petroleum resources at a rapid rate [24].

The lubricating grease is semi solid type of lubricant. This grease consists of a base oil, thickener and additives. This grease mainly consists of mineral oils as base oil. The thickeners used are lithium soap, sodium soap, calcium soap, aluminium soap, complex soaps, etc [25]. These traditional greases are found non-biodegradable which makes them harmful to the environment [26, 27]. So an initiative was with an aim to replace these grease components with biodegradable ones. Vegetable oils were tested for replacing mineral oil keeping the

same thickeners and additives [28-30]. Another study considered use of beeswax as thickener, rapeseed oil as base oil and using conventional additives [31]. Artificial polymers are produced from non-renewable petroleum resources. They include proteins and polysaccharides. One of the most vital polysaccharides is gum which is significantly used in a wide range of applications due to its unique properties [32,33]. Properties of Gum acacia, a naturally available material were studied which concluded that it can be adopted in greases as an additive [24].

Table 1 Physico-chemical properties of Soybean oil[24].

Characteristic	Value
Viscosity @ 40°C, (mm ² /s)	36
Viscosity @ 100°C, (mm ² /s)	10
Viscosity index	192
Saponification Value	170
Flash point (°C)	324
Pour point (°C)	246

Table 2 Characteristics of commercial grease used as the benchmark

Characteristic	Value
Base oil	Mineral
Thickener	Lithium
NLGI Grade	2
Worked Penetration, 60st @ 25°C (10 ⁻¹ mm)	285
Drop point, (°C)	198
Operating temperature range, (°C)	-20 to +130
Base Oil Viscosity @ 40°C, (mm ² /s)	150

Soybean oil is a widely available vegetable oil derived from soybeans, making it a sustainable and renewable resource. It possesses favorable lubricating properties, including good viscosity characteristics and high lubricity. Soybean oil is biodegradable, making it an environmentally friendly alternative to petroleum-based oils. Beeswax is a natural wax produced by honeybees. It offers several beneficial properties for grease formulations. Beeswax acts as a thickening agent, improving the consistency and structure of the grease. It provides excellent water resistance and protects equipment from moisture related corrosion. Beeswax also contributes to the adhesive properties of the grease, aiding in its retention on surfaces and reducing the frequent reapplication.

Now, discussing about the additive, Acacia gum, also called as gum arabic, is a natural gum derived from the sap of various Acacia tree species. It is commonly used as a stabilizer and emulsifier in food and cosmetic applications, but it can also serve as a thickening agent in grease formulations. Acacia gum helps improve the consistency and structure of the grease, enhancing its tackiness and adhesion properties. It can contribute to the overall stability and structural integrity of the grease, allowing it to withstand various operating conditions. The present study explores beeswax as thickening agent and gum acacia as additive in grease based on soybean oil. The prime focus is to investigate the friction and wear performance of

the developed grease. The performance of this developed grease is benchmarked with that of a mineral oil and lithium soap based commercial grease.

II. EXPERIMENTAL

A. Materials

Refined soybean oil, utilized as the base oil, was purchased from a local supermarket. The characteristics of the oil are mentioned in table 1. Natural Beeswax was obtained from Shivay Trading Company, which is used as thickening agent. the acacia gum used as an additive was procured from PC Industires.

Chrome alloy steel balls (AISI E-52100) (diameter = 12.7 mm, Rockwell C hardness 64–66, grade = 25 extra polish) were obtained from Ducom material characterization systems for conducting experiment [34]. Extreme pressure commercial grease was purchased to act as a benchmark. The characteristics of the commercial grease are mentioned in Table 2.

Table 3 Various proportions of Components of grease and their corresponding NLGI Grades.

Sample	Base oil proportion (% w/w)	Beeswax proportion (% w/w)	Additive proportion (% w/w)
1	90	10	0
2	80	20	0
3	70	30	0
4	60	40	0
5	60	40	0.5
6	60	40	1
7	60	40	4
8	60	40	10

B. Strategy for grease formulation

Keeping in mind the practical application of greases, the proportion of ingredients was selected (Table 3) so as to develop greases with bearing-grade consistency (i.e., NLGI 2). A preliminary consistency-based approach was used to determine the correct ratio of the base oil to the thickener (i.e. 60:40) to develop a grease of NLGI 2-grade consistency. In practice, the base oil's to the thickener ratio is kept constant for additive-based greases to achieve similar consistencies as the additives' concentrations vary. The same practice was followed. The concentration of additives (AG) was varied in a broad range of 0.5–10 % w/w (0.5%, 1%, 4%, and 10%) to explore their behavior comprehensively. The maximum doping was kept only constrained to 10 % w/w.

C. Development of eco-friendly grease

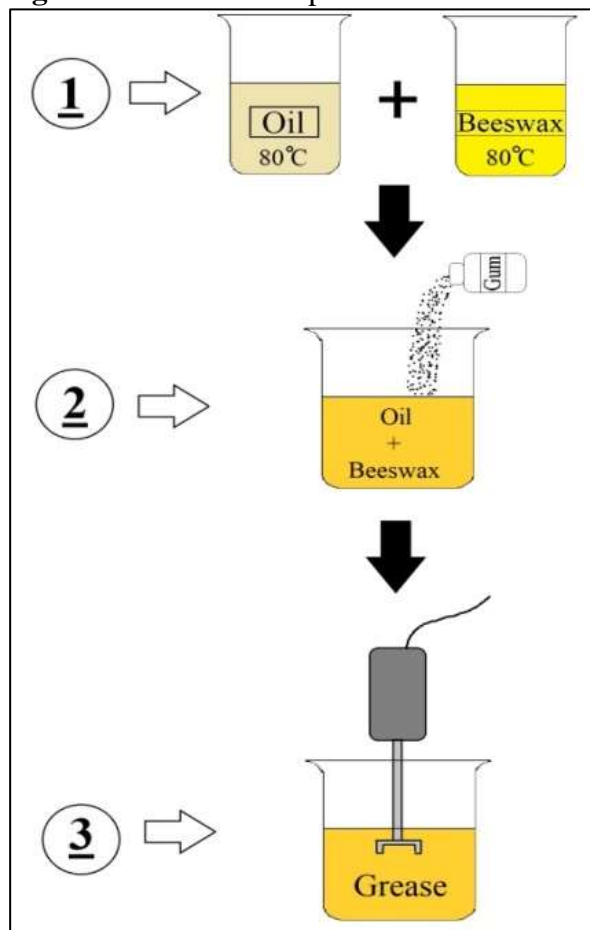
Considering the above discussed alternatives for the new grease, soybean oil is selected as base oil. Beeswax is constituted as thickener and Acacia gum as an additive. Soybean oil and beeswax are heated in separate containers to 80°C. Soybean oil and beeswax are mixed together. Acacia gum is grinded to make powder as mixing it with oil and wax becomes easier. Acacia gum is added to the mixture. This mixture is then stirred with help of hand blender at 1200 rpm for 2 minutes until it cools down and thick grease is formed. 8 samples with varying proportions of the components were prepared to form the grease. These various sample proportions are shown above in table 3. Respective properties of the developed grease are discussed in table 4.

D. Cone Penetration Testing (ASTM-D 217-2021-a)

For worked penetration, the sample is brought to 25 °C ± 0.5 °C (77 °F ± 1 °F) and stationed in the worker cup. The sample is subjected to 60 double strokes in the grease

worker. The penetration is determined immediately by releasing the cone assembly from the penetrometer and allowing the cone to drop freely into the grease for $5 \text{ s} \pm 0.1 \text{ s}$ [35]. Three determinations are made and averaged to give the reported result.

Figure 2 Process of Preparation



E. NLGI grade Testing (ASTM-D 217-2020)

These cone penetration tests not only evaluate the consistency of lubricating greases over the full range of NLGI numbers from 000 to 6, but also evaluate the consistency of stiff greases having penetration numbers less than 85. Worked penetration results are required to determine to which NLGI consistency grade a grease belongs. Undisturbed penetration results provide a means of evaluating the effect of storage conditions on grease consistency [35].

F. Drop point Testing (ASTM-D -2265-2020)

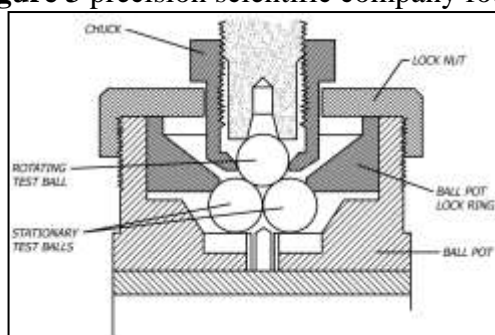
A grease sample in a grease test cup is supported in a test tube placed in an aluminum block oven at a preset constant temperature. A sample thermometer is placed in the tube and so positioned that it measures the temperature in the sample cup without coming in contact with the grease. As the temperature increases, at some point a drop of material will fall from the cup to the bottom of the test tube. The reading on the sample thermometer is recorded to the nearest degree as the observed dropping point. Simultaneously, the temperature of the aluminum block oven is also recorded to the nearest degree. The dropping point helps in identifying the grease as to type and for establishing and maintaining bench marks for quality control [36].

Table 4 Classification and description of grease according to penetration [36].

NLGI Number	Penetration (1/10,mm)at25°C	Description
000	445-475	Fluid
00	400-430	Semi-fluid

0	355-385	Very fluid
1	310-340	Soft
2	265-295	Medium
3	220-250	Medium-hard
4	175-205	Hard
5	130-160	Very hard
6	85-115	Block

Figure 3 precision scientific company four-ball test arrangement[34].



G. Tribological Testing

A Four-ball tester (Ducom Instruments, India) TR-30L model was utilized to record the wear behavior of the greases as per the ASTM D 2266 standard. The test involves relative sliding of steel balls (12.7 mm diameter) where a ball rotates over the three stationary balls clamped in a ball pot, thus making a three-point sliding contact as shown in Fig. . The ball pot is filled with sufficient grease to cover the contacting points. The fourth ball is rotated at a speed of 1200 ± 60 RPM, a temperature of $75 \pm 2^\circ\text{C}$, under a fixed applied load of 40kgf ($390 \pm 2\text{N}$) and for a duration of 60 ± 1 min. The wear scars on surfaces of the three stationary balls are then observed with the help of image acquisition system and winducom software [34].

III. RESULTS AND DISCUSSIONS

For this study the Cone penetration tests, NLGI Grade tests, Drop point tests were conducted at Chemtech Laboratories, Pune and the Wear tests were conducted at Mechanical Engineering Department, PREC, Loni. All the test results are discussed as follows,

A. Cone Penetration analysis

From the table 4. it can be interpreted that as the proportion of beeswax in the base soybean oil increases i.e. from 10%w/w to 40%w/w, the penetration of the cone of penetrometer in the grease goes on decreasing from 424 to 295 (1/10mm). Furthermore when the grease (SO + 40% BW) is doped with the additive Acaia gum varying from 0.5%w/w to 10%w/w, the penetration of the cone is observed to be decreasing from 293 to 277 (1/10mm). The cone penetration of the commercial grease is 285 (1/10mm), which falls under the same penetration range that of developed grease.

B. NLGI Grade Analysis

From the table 4, it can be noticed that as the proportion of beeswax in the base soybean oil increases the NLGI grade the developed grease changes from 00 to 2. As we are comparing the developed grease with the commercial grease which is of NLGI grade 2, the obtained NLGI grade is satisfactory and further useful for comparison of the performances of these greases.

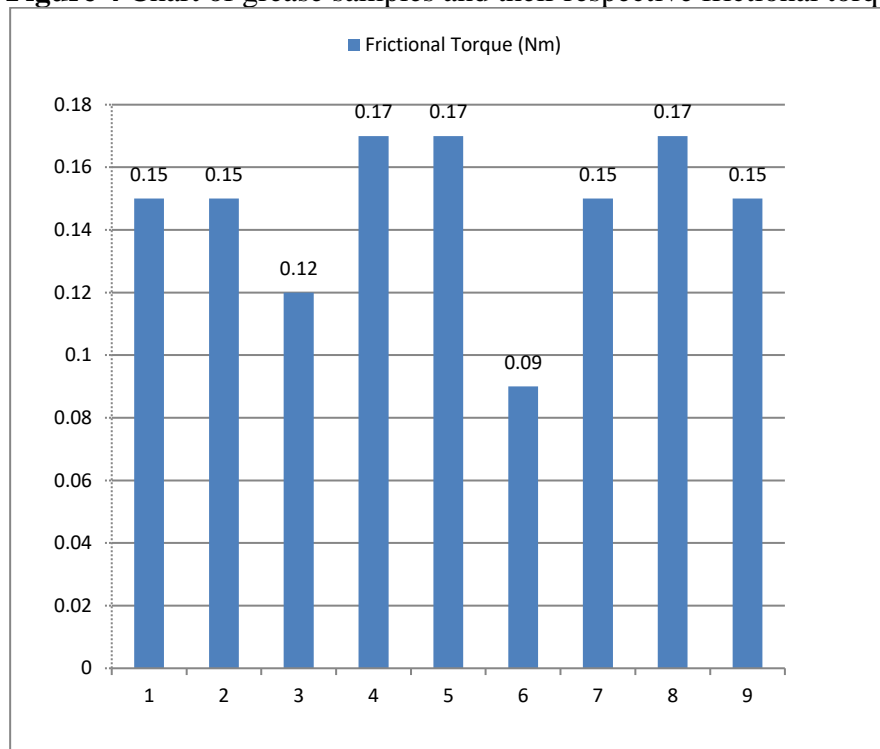
C. Drop Point Analysis

As depicted in table 4., the drop point of the developed grease varies from 65°C to 70°C. Meanwhile, the drop point of the commercial grease is 198°C, which is pretty much higher than that of the developed grease. As the drop point of the developed grease is low, while performing the wear test the grease completely transformed in liquid form which is undesirable. The developed grease may fall short in comparison of the commercial grease in terms of high temperature applications.

D. Tribological Analysis

The most important tribological characteristics of the lubricating grease i.e. friction losses and wear scar were investigated. The measurements of frictional torque illustrate the tribological behavior of friction pair during 3600s tests are shown in the chart Fig. 4. The Commercial grease used as a benchmark shows frictional torque of 0.15 Nm with coefficient of friction 0.08150. Where, the developed samples depict frictional torque ranging from 0.17 Nm to 0.09 Nm. Out of the prepared samples of grease, Sample 3[SO + 30% BW] performs than commercial grease by measuring frictional torque of 0.12 Nm with coefficient of friction being 0.06700.

Figure 4 Chart of grease samples and their respective frictional torques.



Sample 6[SO + 40% BW + 1% AG] shows the greatest reduction in the frictional torque i.e. 0.09 Nm with coefficient of friction being 0.05054. Thereafter, commercial grease leads to mean wear scar diameter of 371 μm and average scar area of 0.168 mm^2 . Sample 6 also shows the least mean wear scar diameter of 668.3(μm) and average scar area of 0.251 mm^2 out of the developed grease samples.

In fig. 5 and fig. 6 scars and respective scar areas are shown for different grease samples. Similarly graphs for comparison between coefficient of friction of different grease samples are illustrated in fig. 7, fig. 8, and fig. 9.

Fig. 7 shows comparison between samples 1, 2, 3, and 4, out of which sample 3 shows best results. Fig. 8 shows comparison between samples 5, 6, 7 and 8, out of which sample 6 shows best results. Finally in fig. 9 comparison between sample 3, 6 and sample 9 (benchmark grease) can be seen.

Table 5 Test results of grease samples

Sample Number	Cone Penetration (1/10,mm)	NLGI Grade Number	Drop Point (°C)
1	424	00	70
2	331	1	65
3	330	1	68
4	295	2	68
5	293	2	67
6	289	2	66
7	285	2	69
8	277	2	70
9	285	2	198

Figure 5 Wear scar Measurements of sample 1

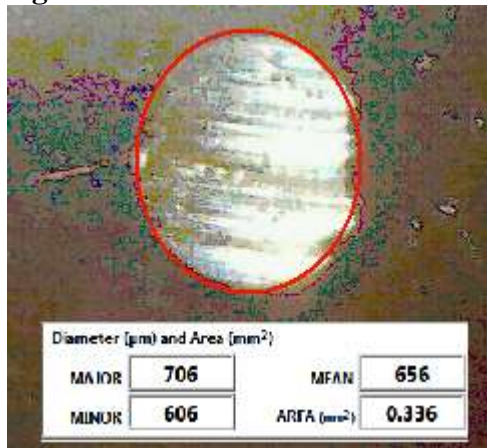


Figure 6 Wear scar Measurements of sample 2

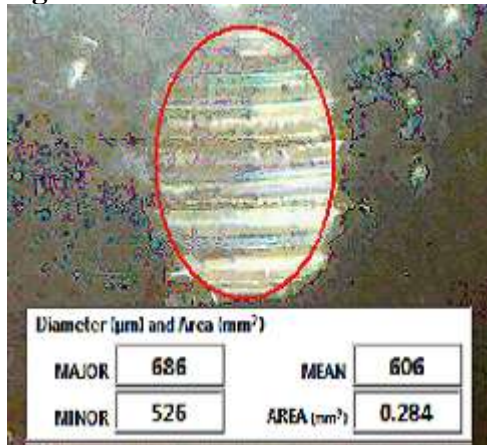


Figure 7 Wear scar Measurements of sample 3

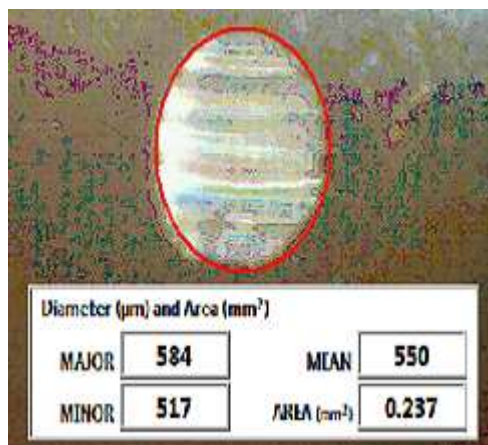


Figure 8 Wear scar Measurements of sample 4

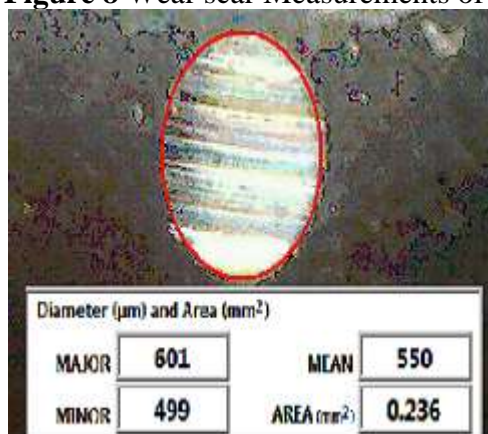


Figure 9 Wear scar Measurements of sample 5

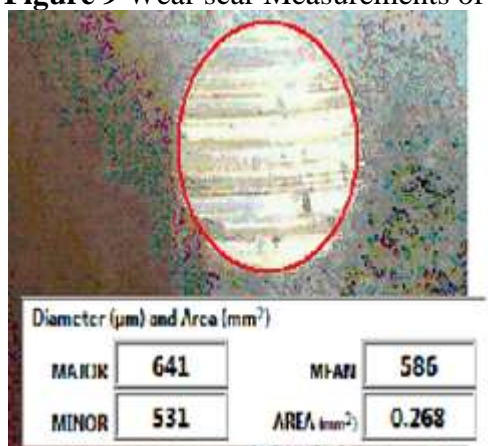


Figure 10 Wear scar Measurements of sample 6

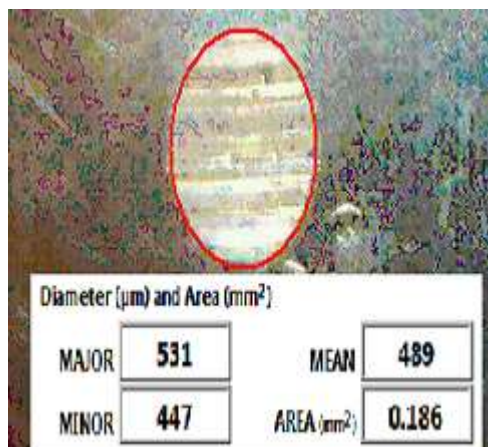


Figure 11 Wear scar Measurements of sample 7

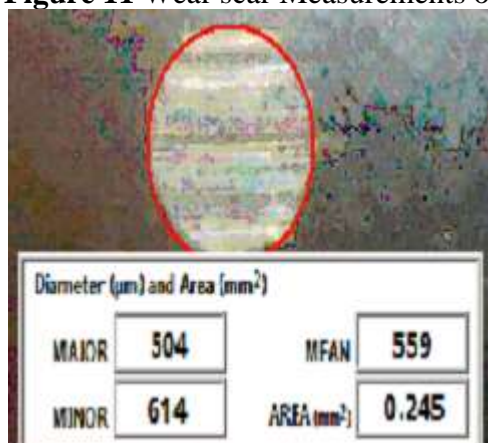


Figure 12 Wear scar Measurements of sample 8

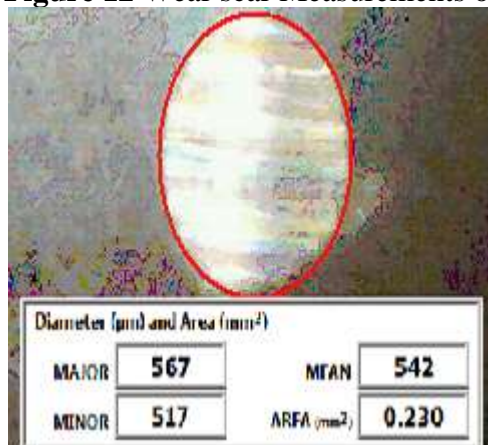


Figure 13 Wear scar Measurements of sample 9

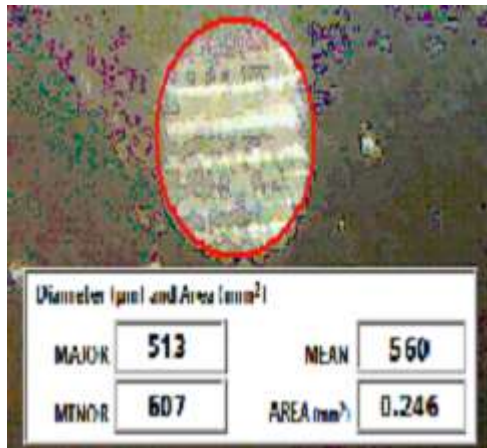


Figure 14 Wear test view for comparison of coefficient of friction between the samples 1, 2, 3, and 4.

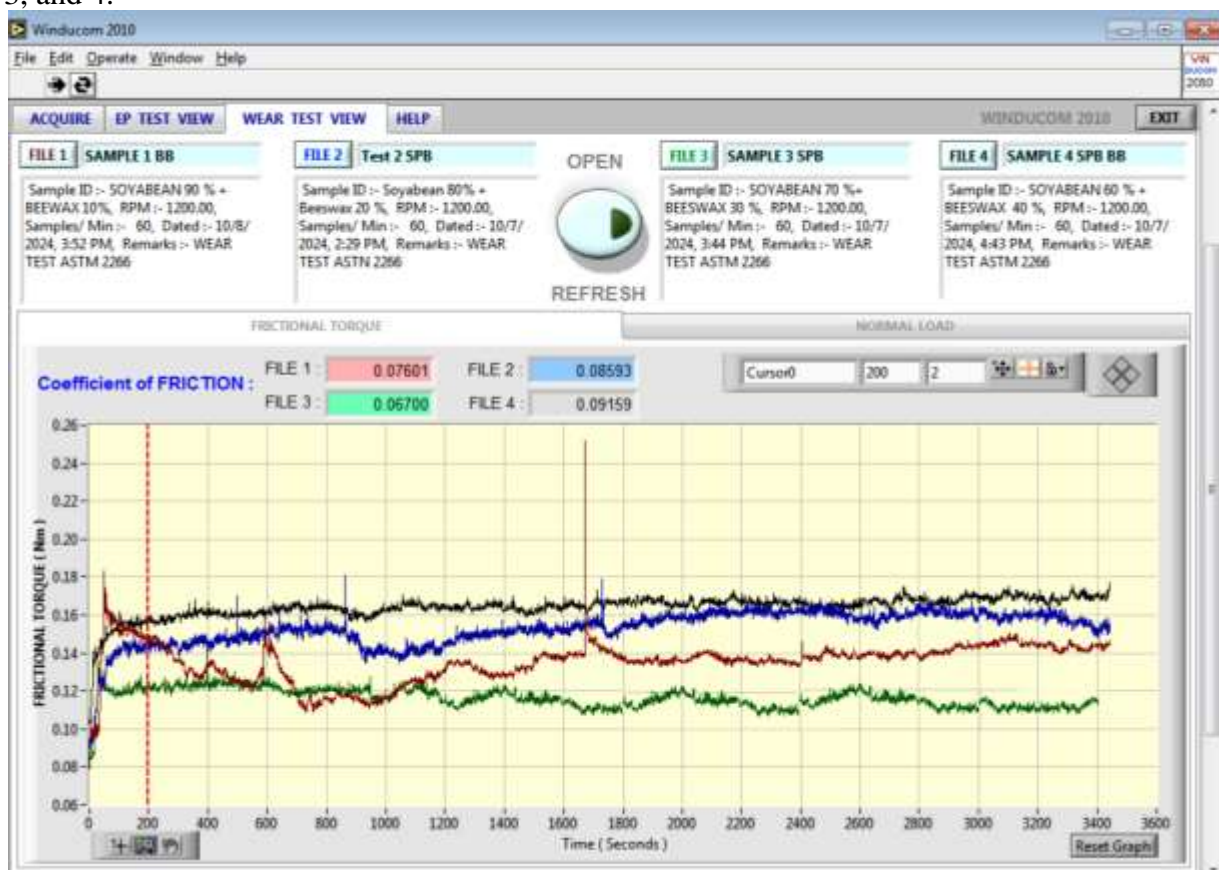


Figure 15 Wear test view for comparison of coefficient of friction between the samples 5, 6, 7 and 8.

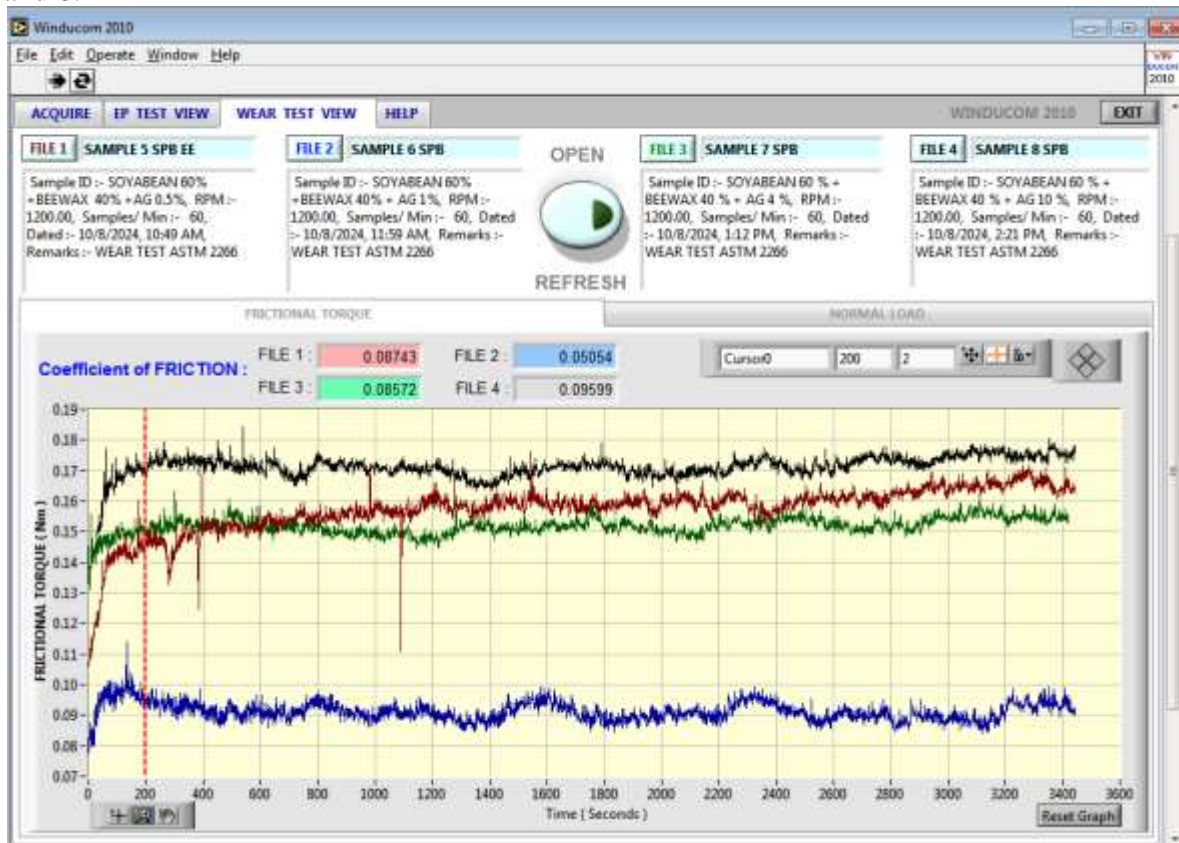


Figure 16. Wear test view for comparison of coefficient of friction between the samples 3, 6 and 9.

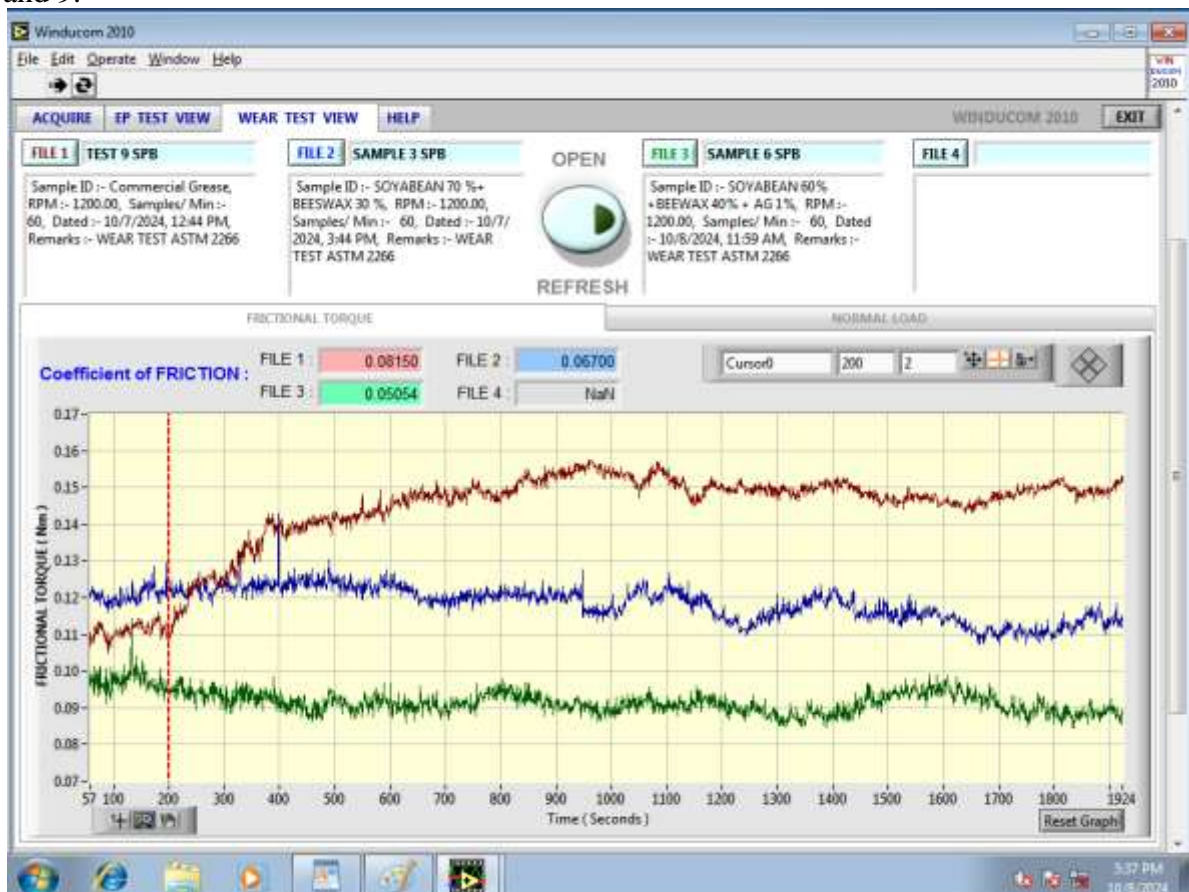


Table 6 Wear scar diameters, coefficients of friction, average scar areas and frictional torques of grease samples.

Sample	Average Mean scar Diameter (μm)	Average Scar Area (mm^2)	Frictional Torque (Nm)	load (N)
1	668.3	0.348	0.15	395
2	655.33	0.335	0.15	399
3	569.3	0.253	0.12	401
4	583	0.265	0.17	402
5	593.66	0.275	0.17	404
6	562.66	0.251	0.09	401
7	578.66	0.262	0.15	393
8	580.33	0.264	0.17	396
9	575.33	0.259	0.15	407

IV. CONCLUSIONS

In this study, basics of the lubricating grease were studied. These basics include a rough idea of what grease is, composition of grease and tribological characteristics of grease. This study helps to understand that commercially available grease is made from mineral oil and various metal soaps which pose a threat to the environment as they are not eco-friendly. This study proposes new eco-friendly grease which is produced from soybean oil as base oil, beeswax as thickener and acacia gum as additive. The NLGL Grade of this grease ranges from 00 to 2.

The cone penetration in this grease varies from 277 to 424 (1/10, mm) and drop point changes from 65°C to 70°C. The developed grease [Sample 6(SO + 40% BW + 1% AG)] shows promising results leading to low coefficient of friction and low wear scar area than other prepared samples. But the developed grease has low drop point which is undesirable so cannot be used in high temperature applications. This could be overcome by doping the developed grease with other additives to improve the thermal performance. It is possible to develop lubricating grease from naturally available renewable sources to replace the non eco-friendly synthetic grease.

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