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Sustainable-Grease from Rerefined Base Oil: A Formulation Study with Ecofriendly Solids for high-Performance Lubricating Greases

Rajasekhar Mettlu
Department of Polymer Science and Technology, Sri Jayachamarajendra College of Engineering, Mysore-570006, India

Abstract - Used engine oil (UEO) disposal is a major challenge due to stringent environmental restrictions around the world. Reusing or recycling spent engine oil is a sustainable way to save the environment. This research study investigates a practical approach for upcycling used engine oil (UCUEO) into high-performance lubricating grease with sustainable ecofriendly solids additives. The study includes three important steps UCUEO characterisation, grease formulation and performance evaluation. Three different concentrations of calcium carbonate and Talc ecofriendly solids are added to upcycled UEO oil-based lithium grease to obtain blends containing 0.5 wt.%, 1.0 wt.% and 1.5 wt. Basic properties like penetration and Oil separation of blend samples studied in comparison with base grease sample as per ASTM standards. The tribological assessment of blends has been performed using a four-ball wear test and load carrying capacity test according to the ASTM test methods. The obtained results for blends are compared to samples of base grease. Test results showed a remarkable enhancement of wear properties capacity of ecofriendly solids blend samples by 0.5–1.5% as compared to base grease. By observing wear scar in rolling elements, the blend samples lowered the average wear scar diameter (WSD) by 30–46%. Base grease samples showed the highest coefficient of friction (COF) values between 0.265. These values are reduced to 0.095 and 0.98 for grease with the blend samples reaching their minimum in the case of 1.5 wt.%. of calcium carbonate These outcomes are found consistent with the enhancements in driving power saving values. The results proved the competitiveness and suitability of the calcium carbonate and Talc ecofriendly solids improving the tribological performance of upcycling used engine oil (UCUEO) greases.

1.INTRODUCTION:

Lubricating grease plays vital role for keeping machinery running smoothly by reducing friction and wear. Grease comprises basic components which are thickener, base oil, and additives [1][2]. Generally, the base oil present in grease is petroleum based mineral oil, sourced from non-renewable petroleum resources. Mineral oil shows effective performance but it is toxic to environment due to its poor biodegradability. [3][4]. UEO is a very hazardous polluting product to the environment. There are significant amounts of heavy metals and polynuclear aromatic hydrocarbons (PAH) in it. It is commonly known that PAHs, like benzopyrene, have a high carcinogenicity. UEO contains significant levels of heavy metals, which are extremely harmful to living things [5]. The disposal of UEO (by burning, filling up land, spilling accidentally, leaking pipelines, and migrating into surface or ground water) degrades the environment. Human life as well as the lives of other living things will be in danger due to this degradation. UEO and used cooking oil (UCO) must thus be efficiently repurposed into valuable recycled products. Recycling is thought to be good for the environment and economic growth because it reduces the need for landfill space and typically involves energy savings [6]. Unfortunately, improper handling of UEOs can cause significant environmental harm. However, they are suitable for upcycling because they are primarily made from paraffinic or naphthenic mineral oils [7][8]. Upcycling, which involves the conversion of waste oil into high-performance grease, provides a more environ- mentally sound and valued solution than the current recycling methods, which frequently yield low-grade lubricants [9][10]. In a previous study proved that the recycled mineral oils which produced from Alexandria Petroleum Company can be used to prepare lithium lubricating grease for the alternative of virgin oil [11]. A previous study revealed that it is possible to formulate grease without the addition of additives when UEOs is utilized. The resulting grease has qualities that are similar to those seen in commercial grease. The goal of this research was to see how eco-friendly additives can affect the

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anti-wear and weld load capabilities of the grease prepared with UCUEO and the improvement that the additives add to the grease properties.

2.MATERIALS AND METHODS

2.1. Materials

Upcycling used engine oil (UCUEO),12-Hydroxy stearic acid Lithium hydroxide are obtained from the local source, Jayant agro organic, India and Lithium hydroxide from SQM, China. Talc and calcium carbonate purchased from HTMC Group, Kunal calcium Ltd respectively.

2.2. Preparation of grease samples

In 2L glass flask and under mechanical stirring, UCUEO (225g) and 12-hydroxystearic acid (48 g) were mixed and heated to 95°C. A solution of lithium hydroxide (7g of $LiOH \cdot H2O$ in 35g of H2O) was added to the mixture. The saponification reaction between lithium hydroxide and 12-hydroxystearic acid took place at 120°C for 1 h. After the mentioned reaction, the mixture was heated to 200°C for 10 min. After that, cool started by addition of 225g of the UCUEO.

Table-1

#	Test parameter	Test method	Unit	Results
1	Appearance	Visual determination		
2	Colour Value	ASTM D1500	Value	3.5
3	Density at 25^{0}C	ASTM D7042	g/cc^3	0.807
4	Kinematic viscosity at 40^{0}C	ASTM D7042	mm^2/s	35.62
5	Kinematic viscosity at 100^{0}C	ASTM D7042	mm^2/s	4.81
6	Flash point	ASTM D92	^{0}C	226

Then the sample was cooled down to room temperature. Subsequently, mixing and homogenization procedures were conducted to ensure the uniform dispersion of the thickening agent. The composition of lithium base grease (ULBG) and seven grease samples (ULBGCC-1, ULBGCC-2, ULBGCC-3, ULBGTC-1, ULBGTC-2 and ULBGTC-3 with eco-friendly calcium carbonate and Talc additives are listed in Table -2. The basic parameters of base oil are given in Table -1. The entire samples preparation was systematically organized into five distinct phases as per in Fig. 1.

Table-2: The composition of the grease samples

#	Test parameter	ULBG	ULBGCC-1	ULBGCC-2	ULBGCC-3	ULBGTC-1	ULBGTC-2	ULBGTC-3
1	Base grease (ULBG)	100.00	99.50	99.00	98.50	99.50	99.00	98.50
2	% of calcium carbonate	0.00	0.50	1.00	1.50	0.00	0.00	0.00
3	% of Talc	0.00	0.00	0.00	0.00	0.50	1.00	1.50

2.3 Grease Analysis

To determine the properties of the grease itself, the prepared grease will be subjected to several physical tests, including tribology, oil bleeding and separation, dropping point, and consistency tests.

2.4 Consistency Test

The consistency of a lubricating grease is commonly regarded as its most essential rheological property. It generally determines the appropriateness of a grease for a specific application. Consistency refers to a grease's ability to resist leaking and produce stable lubrication channels in bearings [12]. These channels are critical to a bearing's performance because they act as reservoirs from which moving parts draw lubricating fluid throughout the machine's operating lifetime [13]. Thus, consistency acts as a crucial criterion for choosing a grease, and the ability to accurately measure this property is very significant. The primary method for evaluating grease

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consistency is the cone penetration test defined by ASTM D217 [14]. In this procedure, a cone is allowed to fall into a grease sample for 5 seconds, and the depth of penetration is utilized as an indicator of consistency.

2.5 Oil Separation Test

This test evaluates how effectively upcycled used engine oil (UCUEO) separates from a particular grease over time when stored. It follows the ASTM D-1742 standard, which measures oil separation in stored greases. The ASTM D-1742 test predicts how much oil a grease will separate during storage at room temperature. The results from this test are directly related to oil separation that occurs in containers of different sizes, including 35lb (16kg) containers, when stored at room temperature. The ASTM D-1472 test is an accelerated method in which a grease sample is placed on a 75-micron (200 mesh) sieve and subjected to a pressure of 0.25 psi (1.72 kPa) for 24 hours at 77°F (25°C). Any oil that seeps out of the grease is collected, weighed, and reported as a percentage of the total weight of the oil separated. This test is not suitable for greases that are softer than NLGI No.1 consistency, as softer greases tend to pass through the sieve. Additionally, this test method should not be used to predict oil separation behaviour under dynamic service conditions.

2.7 Dropping Point Test

The dropping point of grease refers to the temperature at which the grease's thickener can no longer keep the grease from becoming liquid. This temperature is measured using ASTM D2265.In this test, a thin layer of grease is spread on the inside walls of a small container that has a hole at the bottom. As the temperature increases during the test, the thickener in the grease starts to break down, and the grease begins to flow through the hole. The dropping point is noted as the temperature at which the first drop of grease falls from the container. This temperature indicates the point at which the grease will no longer retain its shape and structure.

2.8 Tribology Test

In the current study, Eco friendly solids are mixed in lithium grease and tested on a four-ball tester. The wear and friction tests were executed by using the parameters listed in Table 3. The materials are set up in the four-ball tester, and then the internal heater heats the sample up to 75 °C via thermocouple. The testing procedure is carried out as per the ASTM D2266 standard test procedure while varying concentration of the Eco-friendly solids.

Table 3- ASTM D2266 Test Standard of the Four-Ball wear test

	Duration (min)	Load (kg)	Speed (rpm)	Temperature (^{0}C)
ASTM D2266	60	40	1200 ± 60	75 ± 2

Proper cleaning of every component, including all four balls, collet, ball pot, lock nut, and splash guard, must be ensured through the help of cleaning agents such as toluene, acetone, etc. before and after conducting the test to remove any contamination on the surfaces. The upper ball was secured firmly in the collet, which is attached to the rotating ball chuck, and the lower three balls are placed in the stationary ball pot. The load exerted is uniformly distributed among the points of contact of three balls with the upper ball, and it was realized that the configuration of ball-to-ball assembly has point contact. The test setup (ball pot assembly) consists of four AISI E-51200 grade 12.7 mm diameter chrome alloy steel balls. After an hour, the load is released, and the upper ball from the rotor shaft and the lower balls with the ball pot are removed from the four-ball tester [15,16] An inverted microscope GX53 provided by Olympus was used to analyse the worn surfaces of the lower steel balls. The tests were performed three times for individual samples, and the error was within the limit of ±5%. The data such as the WSD and wear images as well as the graphical data for coefficient of friction (COF) for every sample were recorded.

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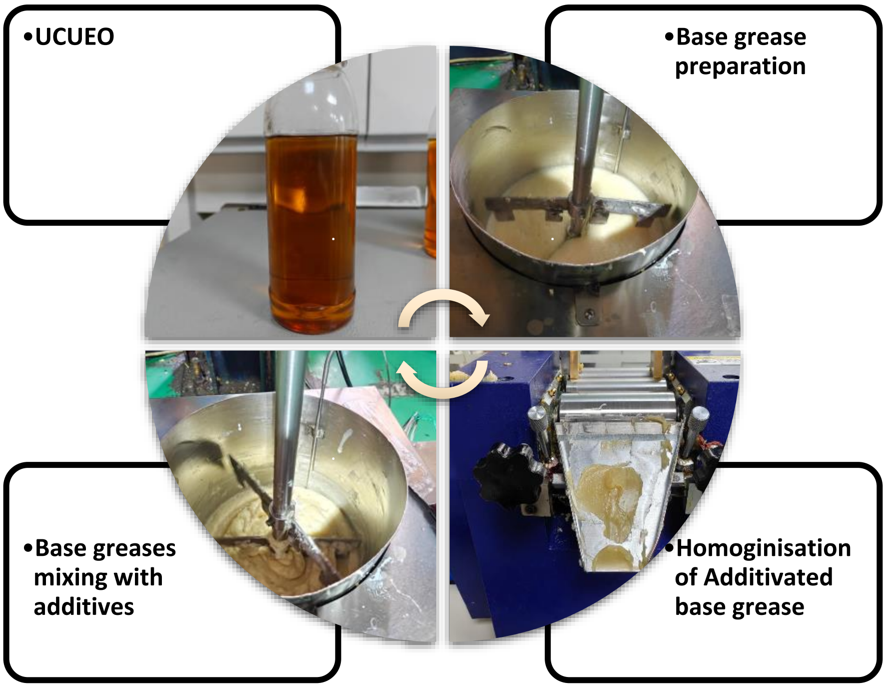

Fig. 1-The entire systematically grease samples preparation phases

3.RESULTS AND DISCUSSION

3.1 Appearance and consistency

The performance characteristics of grease are not influenced by its appearance or colour since this is determined by the manufacturer. Manufacturers use colorants or dyes mainly to aid in identifying the grease and to improve its visual appeal, rather than relying solely on brown or black shades [17]. A specific colour of grease can assist users in recognizing an unsuitable product, which can be a useful indicator. However, colour alone should not be used to identify a grease unless a particular colour is specifically used in a facility. The colour of the prepared grease samples with eco-friendly additives was given in Fig-2. All the grease samples are in light brown colour, with addition of eco-friendly additive colour change was not notice due to low additive concentration.

The NLGI number is a measure of a grease's consistency, determined by its worked penetration value. The penetration test involves measuring how deep a standard cone can penetrate into a sample of grease, expressed in tenths of a millimetre. Each NLGI grade corresponds to a different range of worked penetration values [18]. As the grade number increases, the viscosity of the grease also increases, reaching a solid state at grade 6. NLGI 2 is the most commonly used industrial grade. NLGI 2 grease demonstrates excellent lubricating properties when used in plain and antifriction bearings under moderate load and medium-speed conditions. In general, NLGI 2 is considered the most suitable and frequently used lubricating grease grade in the industry. According to Fig-3, all the grease samples are fall under NLGI 2. Addition of ecofriendly additive to base grease not much penetration change was observed. Thus, considering both appearance and consistency, it can be stated that greases produced from upcycled used engine oil (UCUEO) and eco-friendly additives are equally good [19][20][21]. All seven greases share a common standard property found in industrial greases.

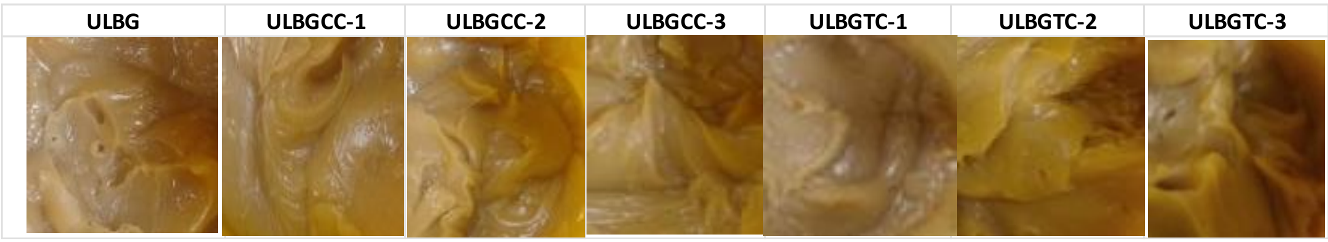

Fig-2-The colour of the prepared grease samples with eco-friendly additives

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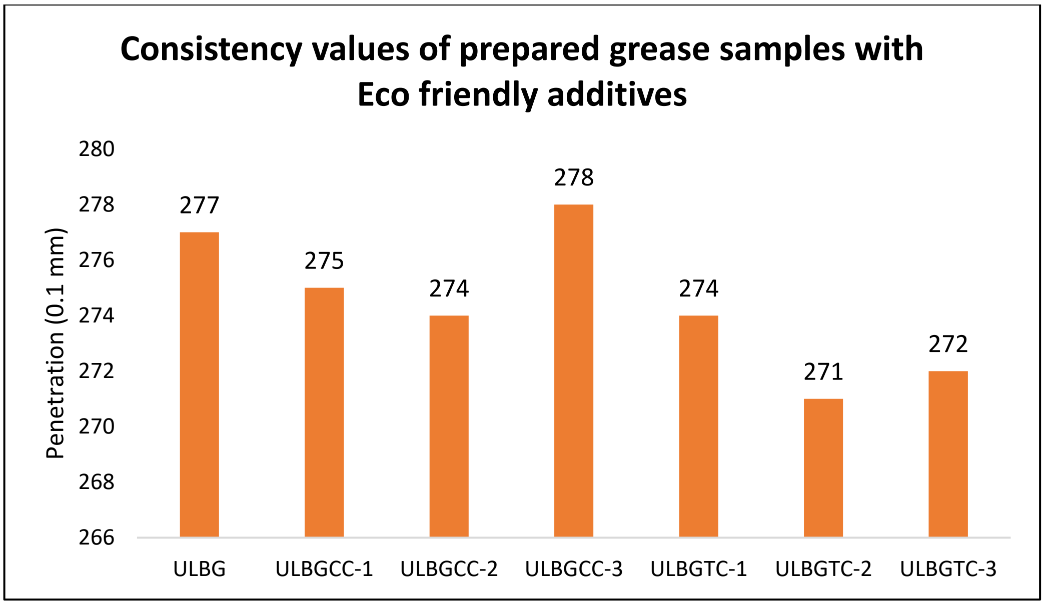

Fig-3-Penetration values of the prepared grease samples with eco-friendly additives

3.2 Oil separation

Oil separation is important because it provides lubrication and helps reduce wear on the equipment [22][23]. Grease is typically made up of oil, a thickener, and sometimes additional components. The thickener acts as a carrier for the oil, sometimes referred to as a sponge. When pressure is applied to the thickener, oil is released, thus starting the lubrication process. All seven grease samples exhibited oil bleed within an acceptable range at 25 °C. From Figure 4 the oil separation values of calcium carbonate blended greases ULBGCC-1, ULBGCC-2 and ULBGCC-3 showed 27.61%,32.52% and 47.15% less compared to ULBG at 25degC. The oil separation values of talc mixed greases ULBGTC-1, ULBGTC-2 and ULBGTC-3 showed 26.01%,29.26.52% and 39.837% less compared to ULBG at 25degC The controlled release of oil is closely related to the microstructure of the thickener network. Grease bleeding or oil separation refers to the oil being released from the grease under static (storage) or regular operating conditions. In static conditions, oil bleeding is identified by small oil pools, particularly when the grease surface is uneven. the desired oil separation percentage should not go beyond 4% to be considered acceptable. The data for oil separation of all seven grease samples were successfully below 4%, meaning all are suitable in this aspect [24]. However, the best performance was recorded by the greases with ecofriendly additive calcium carbonate.

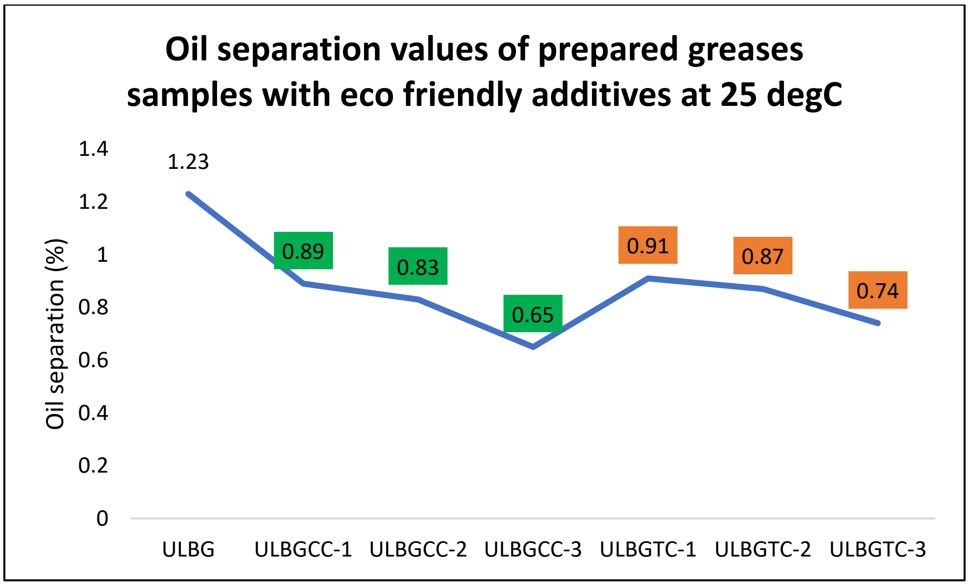

Fig-4-Oil separation values of the prepared grease samples with eco-friendly additives at 25degC

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3.3. Dropping point

The dropping point of a lubricant refers to the temperature at which it transitions into a liquid state. For grease to function effectively, its dropping point should be higher than the maximum operating temperature it will encounter, to avoid issues like bleeding and bearing failure. In high-speed applications, the operating temperature tends to be higher, which can reduce the lifespan of the grease. Therefore, the grease must have properties that enable it to perform well under such conditions. As a result, different dropping points are necessary for various uses [25]. For instance, engine lubrication requires greases with higher dropping points because the internal temperature of an engine can become quite high during operation.

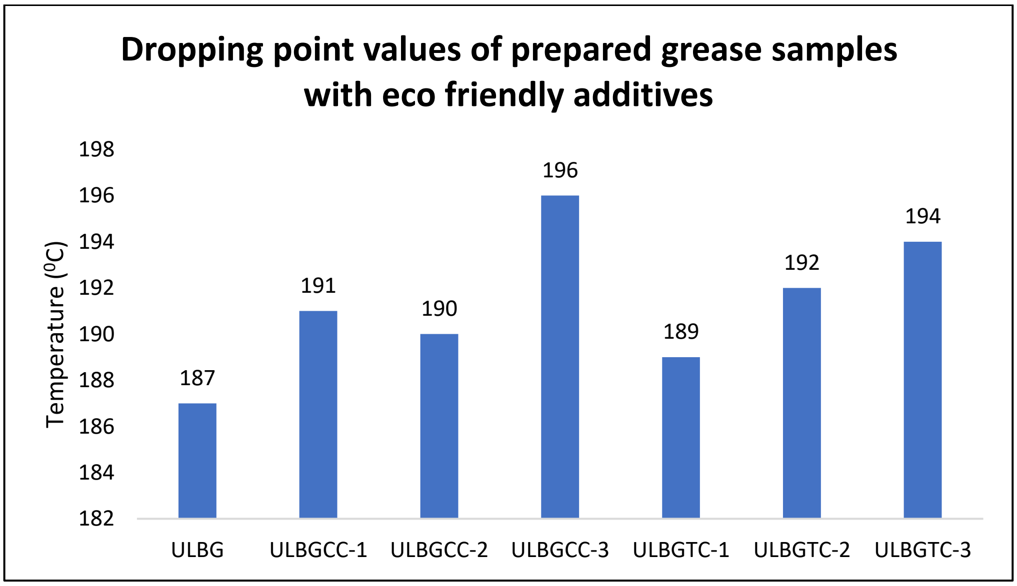

Fig-5-Dropping point values of the prepared grease samples with eco-friendly additives

From a structural and thermal stability standpoint, greases with higher dropping points generally contain thickeners that create strong three-dimensional networks, which remain stable even when exposed to high temperatures [26]. Results of dropping point for ecofriendly additive-greases with different contents are shown in Figure 5. These results were obtained from the average values of conducted experiments with three times repeatability. Raising the weight percentages of ecofriendly additives leaded to the increase of the dropping point for the prepared ecofriendly-greases. This could be a merit for ecofriendly-greases because increasing the dropping point can cause them to be separated from the oil at higher temperatures and as a result, kept their structures at high temperatures.

3.4 Tribology tests

3.4.1 The coefficient of friction (COF)

The coefficient of friction (COF) is a quantitative indicator that characterizes the lubricating characteristics or qualities of lubricants. The lower the coefficient of friction, the less energy is necessary to shear the lubricating film and the less power is transmitted through the fluid. The coefficient of friction is a characteristic, primarily influenced by the specific base oil and VI improver utilized, particularly in the context of mixed and fluid friction, particularly when accompanied by sliding contact [27][28].

In tribological terms, a low COF often correlates with effective boundary or elasto hydrodynamic lubrication films formed between contacting surfaces [29]. These films reduce direct metal-to-metal contact, thereby minimizing shear forces and localized heating. The presence of solid additives like graphite, as in the WEO samples, enhances this performance by forming protective tribo films that further reduce frictional losses via layer-by-layer shearing mechanisms [30].

Typically, a low coefficient of friction (COF) is considered advantageous since it suggests reduced resistance for motion or surface roughness, as shown in Table 6. The recognition of friction as a significant contributor to wear, resulting in material fatigue, necessitates the careful consideration of lubricant selection to enhance the reliability of equipment.

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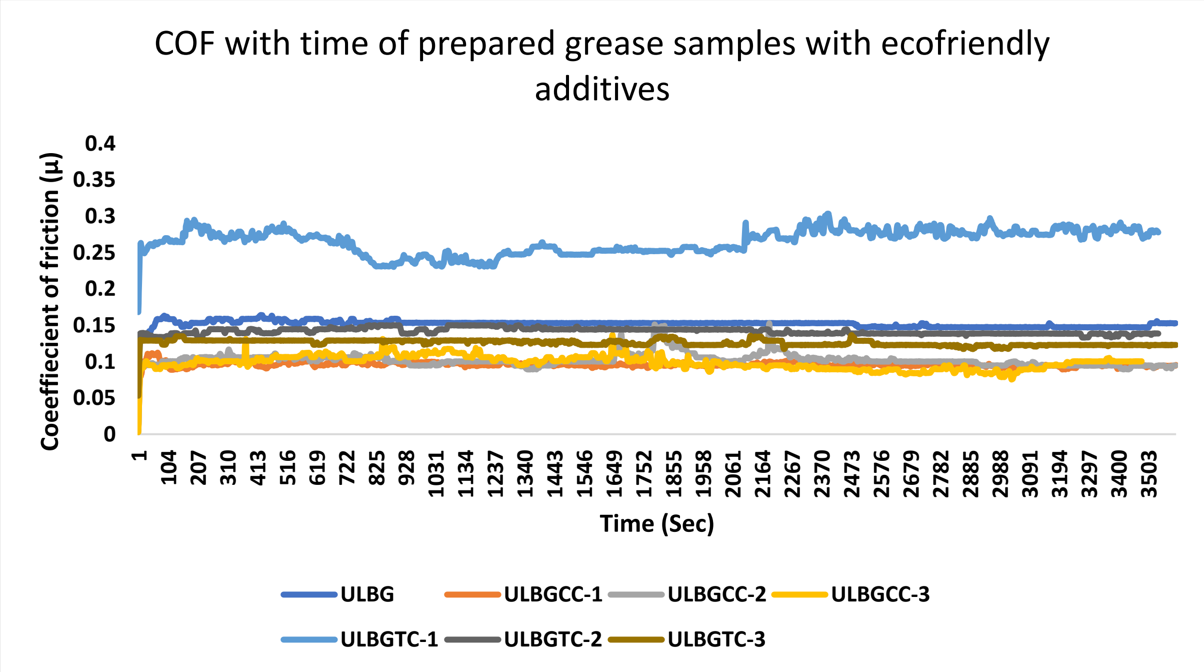

Fig-6- coefficient of friction values with respect to time of the prepared grease samples with eco-friendly additives

Figure 6 and Figure 7 shows the amount of coefficient of friction (COF) values for the base grease and modified greases with ecofriendly additives with different concentrations. Results indicated that for the base grease at the of 0.265 modified greases with 0.5%,1.0,1.5%of calcium carbonate have 0.102,0.098 and 0.095 respectively other words, this test clearly shows that reduction in COF by addition of calcium carbonate to base greases. Similar trend was observed in case of talc modified greases with 0.5%,1.0,1.5%of talc has 0.151,0.141 and 0.125 respectively. It is obvious that raising the contents of ecofriendly additives increased this characteristic and the best performance was obtained for the cases in which Calcium carbonate were used. While for the All Six greases samples exhibited a successful result, with each sample recording a COF that had wear that zero, as all values in the range of 0.005–0.151. This indicates that all values had meet the desired criteria in terms of friction. However, grease from ULBGCC-3 is the lowest COF with 0.095 compared to the other finalized grease sample, meaning that it is the best grease compared to others in this aspect.

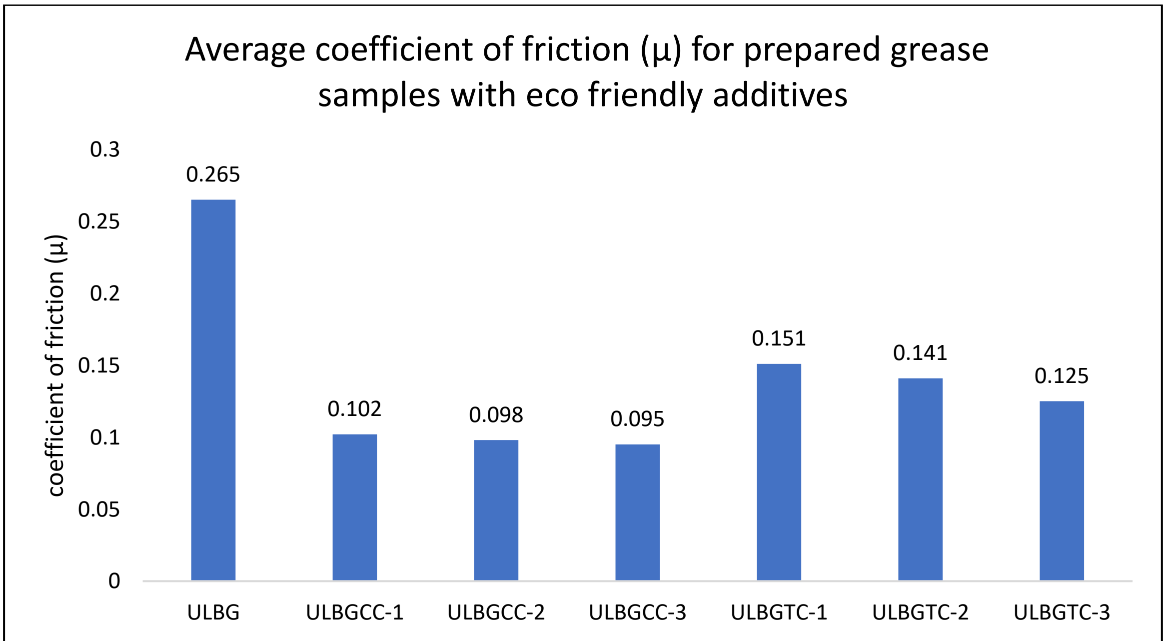

Fig-7-Average coefficient of friction values of the prepared grease samples with eco-friendly additives

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3.4.2 Wear scar diameter (WSD)

The main concept behind Wear Scar Diameter (WSD) is that a smaller scar means less wear, which suggests better lubricity of the fuel sample. The stability of the lubricating layer plays a key role in reducing the WSD on equipment when lubricating grease is used. Applying lubricating grease is expected to lower the WSD by reducing friction between equipment surfaces. Based on the data in Figure 8, ULBGCC-3, which had the lowest WSD of 0.42 mm, showed the best performance in minimizing wear and surface damage, indicating its superior ability to protect against wear compared to the other greases ULBGTC-2, with a moderate WSD of 0.53 mm, offers a balance between wear resistance and performance, making it a good choice for situations where a balance between durability and cost is needed. On the other hand, UGLBG, with a WSD of 0.78 mm, showed the most wear and surface damage, indicating poor wear resistance and making it the least effective of the six ecofriendly additive greases tested.

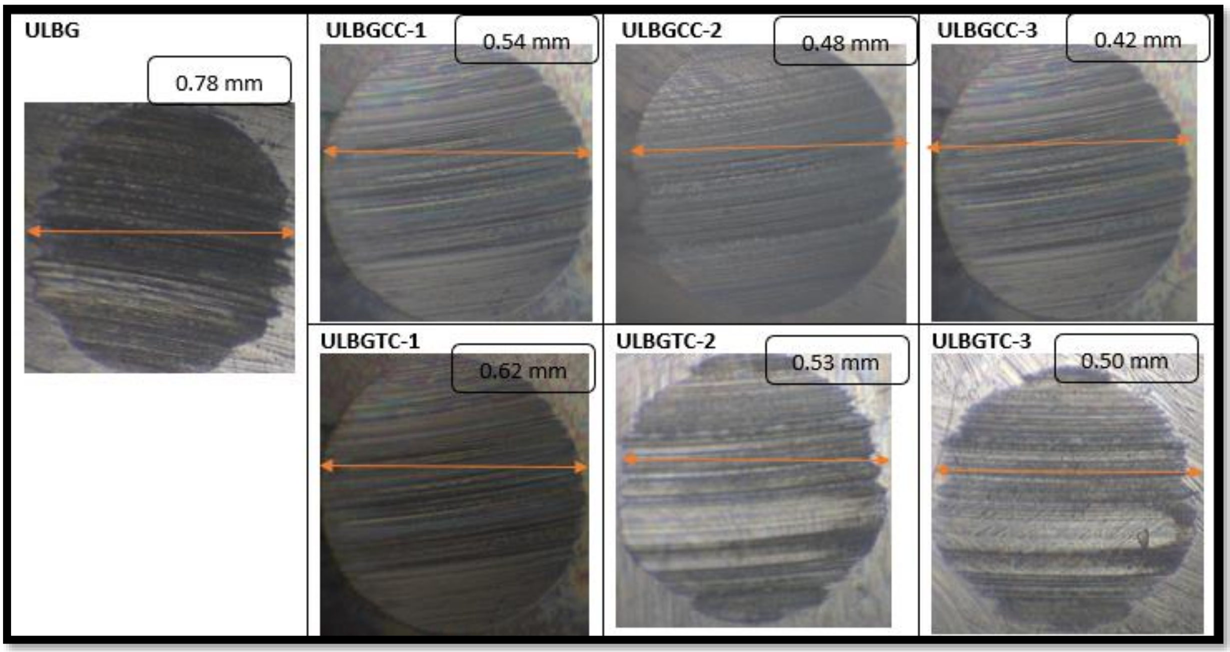

Fig-7-WSD values of the prepared grease samples with eco-friendly additives

Among the Six samples tested, the base grease with calcium carbonate combination showed the least noticeable and most consistent wear scar, clearly visible in the top-left image. The smallest in WSD, indicates strong wear resistance and excellent surface protection under stress, proving its effectiveness as the best grease formulation tested.

4. CONCLUSION

In this study seven lithium-based greases were prepared with ecofriendly calcium carbonate, talc with o.5%,1.0%,1.5% wt with upcycling used engine oil (UCUEO). All the grease samples are in light brown colour, with addition of eco-friendly additive colour change was not notice due to low additive concentration. Addition of ecofriendly additive to base grease not much penetration change was observed. The oil separation values of calcium carbonate blended greases ULBGCC-1, ULBGCC-2 and ULBGCC-3 showed 27.61%,32.52% and 47.15% less compared to ULBG at 25degC. The oil separation values of talc mixed greases ULBGTC-1, ULBGTC-2 and ULBGTC-3 showed 26.01%,29.26.52% and 39.837% less compared to ULBG at 25degC. Raising the weight percentages of ecofriendly additives leaded to the increase of the dropping point for the prepared ecofriendly-greases. The amount of coefficient of friction (COF) values for the base grease and modified greases with ecofriendly additives with different concentrations. Results indicated that for the base grease at the of 0.265 modified greases with 0.5%,1.0,1.5%of calcium carbonate have 0.102,0.098 and 0.095 respectively other words, this test clearly shows that reduction in COF by addition of calcium carbonate to base greases. Similar trend was observed in case of talc modified greases with 0.5%,1.0,1.5%of talc has 0.151,0.141 and 0.125 respectively. It is obvious that raising the contents of ecofriendly additives increased this characteristic and the best performance was obtained for the cases in which Calcium carbonate were used. Through all the results,

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Calcium carbonate, Talc by the weight percentage of (0.5–1.5) % could be the best additive to promote the properties of lithium-based greases made with upcycling used engine oil (UCUEO).

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