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Study of Graphene- Nanomaterials as an Extreme Pressure Additive for Sustainable Rerefined BASE Oil Lithium Greases

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Abstract: The tribological performance of graphene-enhanced Sustainable Rerefined Base Oil (SRRBO) based lithium greases is examined in this work with an emphasis on wear resistance, water wash out stability, and friction reduction. The effects of adding different weight percentages of graphene (0.25 wt%, 0.50 wt%, and 0.75 wt%) to the grease matrix were assessed using a variety of experimental procedures, such as water resistance tests, thermal stability evaluations, and the four-ball wear test. The findings showed that the optimum performance balance was provided by lower graphene concentrations, especially 0.25 weight percent, which significantly reduced wear and friction while enhancing thermal stability and water resistance. Due to agglomeration, higher concentrations showed diminishing returns in tribological performance even if they improved thermal stability. This study demonstrates graphene's potential as an industrial lubricant additive, particularly in settings that demand great mechanical stability and heat resistance.

1.INTRODUCTION:

Graphene, a new type of material, is being studied for a wide range of uses, including electronic devices and as an additive in polymers, paints, and concrete, among other things [1–3]. It also shows promise as an additive in applications related to friction and lubrication [4]. Its natural low friction, high strength, and ability to conduct heat can offer extra benefits in reducing friction.

Graphene's strong thermal and electrical conductivity can also help lower the operating temperatures of machinery parts like rolling bearings and gears. This temperature reduction can extend the lifespan of both the mechanical components and the lubricant grease. According to predictive maintenance data, the lifespan of lubricant grease can double for every 10 ◦C drop in temperature during use [5–6].

The most common grease additives are oxidation and rust inhibitors. In situations involving extreme pressure contacts, sulphur-based and other extreme pressure additives are used. The grease thickener can create tribofilms and has a role when contact occurs [7]. Solid particulate additives like molybdenum disulfide or graphite have become more common, with a focus on improving energy efficiency, extending the time between maintenance, and lowering the cost of ownership. Most lubricant additives include heavy metals such as zinc, copper, lead, and others, or involve sulphur atoms [8-11], which can cause environmental concerns. In addition to its minimal environmental impact, graphene's two-dimensional structure and its properties make it a possible replacement for other materials used as additives in the grease lubricant industry. Studies cited in Liu's review mention that oxidation and rust inhibitors are commonly used. In applications involving extreme pressure contacts, sulphur-based and other extreme pressure additives are used. The grease thickener can form tribofilms and play a role during contact. Moreover, graphene's high thermal conductivity helps dissipate heat generated during sliding motion, as reported in Liu's review [12].

Graphene, as an additive, provides improved thermal stability along with reduced friction, lower wear, and better load-bearing capacity compared to graphite. When used as a grease additive, it was found that graphene platelets (2 to 15 nm) significantly reduced wear on bearings during oscillating tests and achieved nearly a 50% decrease in friction when the test sliding speed was less than 1×10^{-6} m·s^{-1}.Fan studied multilayer graphene (MLG) as a solid additive and also when mixed into bentone grease. The addition of graphene increased the dropping point and improved resistance to cone penetration [13]. Singh added reduced graphene oxide concentrations of 0.2, 0.4, and 0.6 wt% to commercial lithium grease. Friction was reduced by up to 30% in rolling contacts and 20% in sliding-induced rolling contacts. Vibration and noise were also lowered [14]. Wang examined graphene and graphite in concentrations ranging from 0.2 to 2 wt% in lithium greases. They noted an increase in dropping point and resistance to penetration. Friction and wear reduction were observed using a four-ball tribometer [15]. Fu tested concentrations of 1–4 wt% and found the best tribological performance at 2 wt. As the concentration increased, thermal conductivity also improved, reaching a 56% increase at 4 wt% graphene [16]. Pape and Poll tested multilayer graphene platelets of different thicknesses and observed reductions in wear and friction during oscillating and sliding tests. The authors noted that

International Journal of Engineering Research & Technology (IJERT)
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Vol. 15 Issue 09 , September - 2026

the graphene nanoplatelets covered the rough spots on rubbing surfaces and prevented direct metal-to-metal contact [17]. Wang studied 0.5–2.0 wt% of few-layer graphene (FLG) in polyurea grease. Higher FLG content led to increased storage and loss moduli in rheological tests. Lower wear and friction were achieved at low loads in both mild and high-temperature conditions during reciprocating tests. Initially, FLGs were deposited on the metal surfaces, reducing roughness. Later, the FLGs in the grease increased the stiffness of the tribofilm formed. Finally, the thermal conductivity of graphene helped prevent tribofilm degradation and supported the formation of protective oxide tribofilms. However, beyond a certain load, the benefits in terms of tribology became less significant [18].

In the current work, commercially available graphene-based material was investigated as additives to a simple lithium soap based on sustainable rerefined oil. In the current work, the graphene powder was mixed with base oil to improve the dispersion and performance. They were then transformed into additives that were finally mixed with the baseline lithium-based grease.

2.MATERIALS AND METHODS

2.1. Materials

Sustainable rerefined base oil (SRRBO),12-Hydroxy stearic acid Lithium hydroxide are obtained from the local source, Jayant agro organic, India and Lithium hydroxide from SQM, China. Graphene from Adnano Technologies Pvt Ltd.

2.2. Preparation of grease samples

In a 2-liter glass flask under mechanical stirring, SRRBO (225 grams) and 12-hydroxystearic acid (48 grams) were combined and heated to 95°C.A solution of lithium hydroxide (7 grams of $LiOH·H_2O$ in 35 grams of water) was then added to the mixture. The saponification reaction between lithium hydroxide and 12-hydroxystearic acid occurred at 120°C for one hour. Following this reaction, the mixture was heated to 200°C for 10 minutes. Cooling then began by adding 225 grams of SRRBO.For each sample, the required amount of graphene was carefully measured and placed into a container with the appropriate amount of oil. The two components were mixed using a vortex mixer at maximum speed for 5 minutes to start the dispersion process. After the initial mixing, the container was placed in an ultrasonic cleaner and subjected to sonication at 40 kHz for 30 minutes to break up any agglomerated graphene and ensure a uniform dispersion. Following sonication, the lithium complex grease was quickly added to the graphene-oil mixture to maintain the graphene in suspension. The mixture was then homogenized using a shear mixer at 2000 rpm for 10 minutes, resulting in a consistent texture. The composition of the lithium base grease (LBG) and three grease samples (LBGGN-1, LBGGN-2, and ULBGGN-3, which contain graphene oxide) are listed in Table 2. The basic parameters of the base oil are provided in Table 1.

Table-1

#	Test parameter	Test method	Unit	Results
1	Appearance	Visual determination		Yellow
2	Colour Value	ASTM D1500	Value	3.1
3	Density at 25^0C	ASTM D7042	g/cc3	0.829
4	Kinematic viscosity at 40^0C	ASTM D7042	mm2/s	37.42
5	Kinematic viscosity at 100^0C	ASTM D7042	mm2/s	5.01
6	Flash point	ASTM D92	^{0}C	231

2.3. Four ball wear test

To assess the wear preventative characteristics of the samples, the four- ball wear test (ASTM D2266) was employed which consists of four steel balls, three of which are coated with the grease sample and placed in a triangular position, with the fourth ball placed on top. The fourth ball was rotated at 1200 revolutions per minute under a controlled temperature of 40^0C. A time versus coefficient of friction graph was generated during the test at specified intervals.

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Table-2: The composition of the grease samples

#	Test parameter	LBG	LBGGN-1	LBGGN-2	LBGGN-3
1	Base grease (ULBG)	100.00	99.50	99.00	98.50
2	% of Graphene oxide	0.00	0.250	0.500	0.750

2.4. Dropping point test

The dropping point test, according to ASTM D2265, required heating the grease samples from their semi-solid form until a liquid dripped from them. The dropping point, identified as the transition temperature, was measured and the outcomes were extrapolated for additional comparison.

2.5. Penetration test

The cone penetration test, performed according to ASTM D217, was utilized to assess the consistency and structural integrity of the grease samples. A standardized cone was permitted to enter the grease for a designated duration, and the penetration depth was measured in tenths of a millimetre. This examination identifies the NLGI consistency grade of the grease and evaluates any alterations in texture resulting from the inclusion of graphene.

2.6. Water washout test

The water washout test (according to ASTM D1264) evaluated the lubricating grease's resistance to water exposure, specifically its capacity to stick to surfaces. The grease sample was placed on a rotating bearing and exposed to a continuous flow of water. The amount of grease lost during the test was noted as a percentage.

Table-3 The worked penetration values of grease samples

#	Test parameter	LBG	LBGGN-1	LBGGN-2	LBGGN-3
1	Worked penetration	276	275	278	274

3.RESULTS AND DISCUSSION

3.1. Appearance and Penetration test

After homogenization, the lubricant grease samples were tested using an Anton Paar Penetrometer. Penetration tests were conducted to assess the consistency and shear stability of the petroleum lubricant greases; the appearance of the grease samples shown in Figure 1. A standard cone was released from the penetrometer and allowed to drop freely into the sample for five seconds at a constant temperature. The depth of the cone's penetration was measured in tenths of a millimetre using the penetrometer. The testing followed the ASTMD1403 procedures with half-scale penetration cone equipment. Subsequently, the samples underwent further testing with a Roll Stability Tester to evaluate shear stability by measuring the change in worked penetration. This test was performed in accordance with ASTMD1831. No significant effect on grease consistency was noted as shown in Table-3

LBG **LBGGN-1** **LBGGN-2** **LBGGN-3**

Figure-1. Physical appearance of prepared grease samples

3.2. Dropping point

The dropping point of grease denotes the temperature at which the initial droplet of fluid, usually the base oil, fully detaches from the grease mixture. This measurement indicates the heat resistance of the grease and is affected by the type of thickener employed and its cohesiveness within the oil and thickener matrix. While the dropping point temperature indicates the threshold at which the grease retains its structure, it does not necessarily represent the highest temperature for grease application. In instances where graphene samples were added, marginally elevated temperatures were observed at the dropping point, as demonstrated in Figure 2.

Energy conversion at the atomic level in solid materials takes place through the natural diffusion of electrons and vibrations within the lattice structure. The incorporation of graphene into a base lubricant typically improves the thermal conductivity and heat transfer efficiency of the lubricant. Various factors, including Brownian motion, radiative heat transport, the liquid oil layer enveloping nanoparticles, and the aggregation of nanoparticles, play a significant role in determining the thermal conductivity of lubricants that contain graphene or other nanoparticles. The thermal conductivity in this context is primarily influenced by the Brownian motion of nanoparticles within the oil. This motion enhances thermal conductivity through multiple mechanisms, such as collisions between nanoparticles that promote heat transfer via both solid–solid conduction and convection.

Graphene is distinguished from other carbon-based materials by its exceptional thermal conductivity. When properly incorporated into a grease matrix, it improves heat dissipation and reinforces intermolecular bonds, resulting in a higher dropping point.

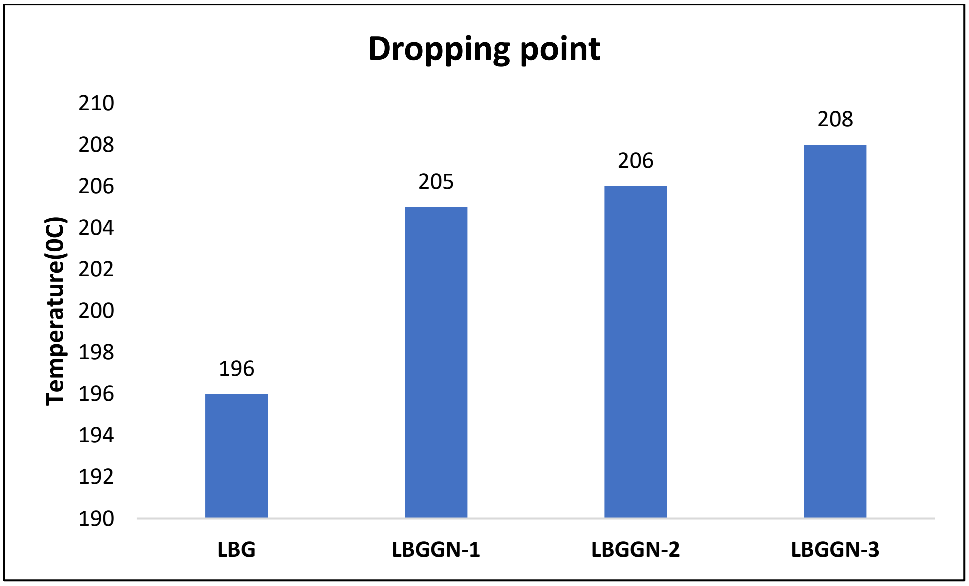

Figure-2. Dropping points of the tested greases

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3.3. Water washout test

With only 1.62% of the grease being washed out, the 0.25 weight percent sample fared the best in the water washout test, indicating higher water resistance. The 0.75 weight percent sample had the lowest performance at 2.35 percent, whereas the other three samples (0.50weight percent and 0.75 weight percent) all performed similarly. These findings imply that water resistance in lithium grease formulations is enhanced by lower graphene concentrations, especially 0.25 weight percent. These formulations are more effective for usage in damp or humid conditions because of the improved dispersion of graphene at these lower concentrations, which probably helps to improve protection against water exposure Figure 3 shows the produced grease samples' water wash out details.

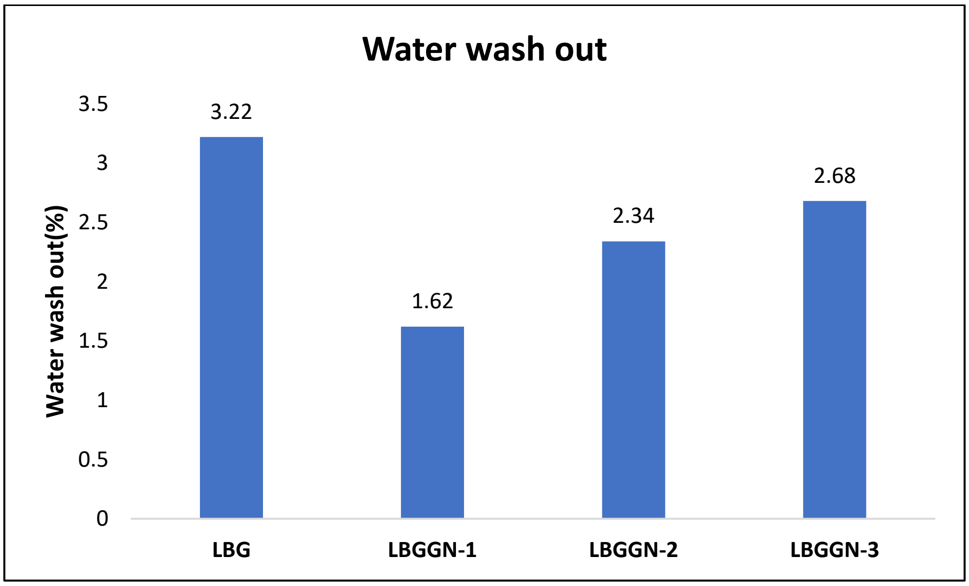

Figure-3. The water wash out of the tested greases

3.4. Four ball wear test

The wear and friction test findings shed light on the graphene-enhanced grease's tribological performance. In terms of wear resistance, two samples—0.25 weight percent and 0.50 weight percent of graphene—stand out because they significantly reduce the size and depth of wear scars in comparison to the LBG sample. The worn scar's surface area decreased by 51% for the 0.5 weight percent sample and by 45.4% for the 0.50 weight percent sample. By sheltering the surface during the testing, this performance implies the production of a tribofilm that might either remain as a protective layer or serve as a sacrificial medium, minimizing material loss. The surface area of the wear scar features in the generated grease samples is displayed in Figure 4

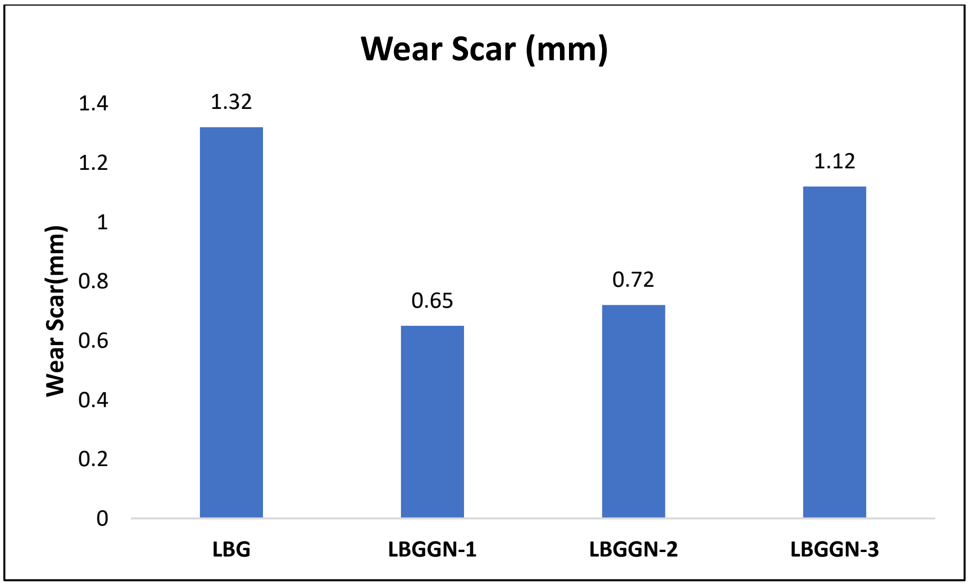

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Figure-4. The surface area of the wear scar of the tested greases

These conclusions were further corroborated by profilometry data, which showed that the 0.5 weight percent sample had the shallowest wear scar depth of 8 µm, closely followed by the 0.50 weight percent sample at 10 µm. Contrary of these findings, greater graphene contractions (0.75 weight percent) demonstrated wear scar depths of 15 µm, which were additional improvements over the control sample's wear scar depth of 18 µm. This suggests that agglomeration may occur at these concentrations. The wear scar depth details of the generated grease samples are displayed in Figure 5.

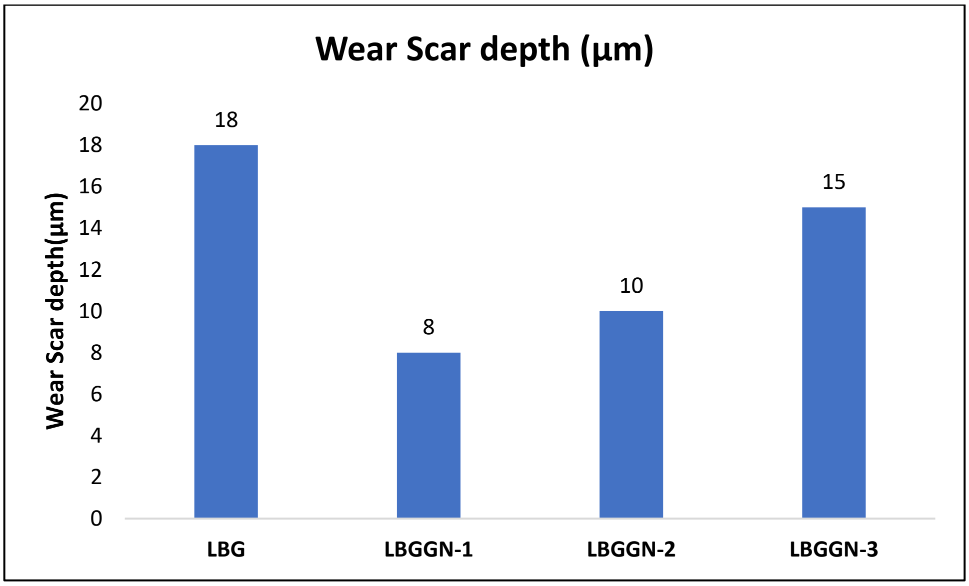

Figure-5. The wear scar depth of the tested greases

Since the 0.25 weight percent sample showed the most stable COF over the course of the test, the COF results were in line with the wear scar analysis. The 0.50 weight percent sample did better at first, but over time, its COF slightly increased. The idea that agglomeration at greater concentrations can impair lubrication performance is further supported by the higher COF values of the 0.75wt% samples. These findings imply that the best friction and wear reduction qualities are provided by lower graphene concentrations, especially 0.25 weight percent.

4.CONCLUSION

The effects of adding graphene to lithium greases based on Sustainable Rerefined Base Oil (SRRBO) were investigated in this study, with particular attention paid to wear, water resistance, friction, and thermal stability. The findings showed that lower graphene concentrations, especially around 0.25 weight percent, provided the most reliable and excellent outcomes in a variety of tests. The 0.50 weight percent sample outperformed the control sample and greater graphene concentrations in terms of wear resistance, water washout, and friction reduction. Graphene improved the thermal robustness of the grease formulations, according to thermal stability evaluations, including the dropping point.

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