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Load-Dependent Tribological Performance of a Rice Husk–Derived Lithium Grease without Conventional Extreme Pressure Additives

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Abstract

This study evaluates the load-dependent tribological performance of a lithium grease formulated from rice husk–derived materials, comprising rice husk pyrolysis oil (60 wt%), lithium stearate thickener (35 wt%), and Rice Husk Ash (RHA) additive (5 wt%). Gravimetric wear tests were conducted at 1490 rpm under applied loads of 800 g, 900 g, and 1000 g, and the results were benchmarked against two commercial lithium-based greases. At 800 g, all greases exhibited comparable specific wear rates on the order of $10^{-6} – 10^{-7}$ $g·N^{-1}·s^{-1}$. However, at 1000 g, both commercial greases failed prematurely, entering a severe wear regime with specific wear rates of approximately $3.4–3.6 \times 10^{-4}$ $g·N^{-1}·s^{-1}$. In contrast, the rice husk–derived grease completed the full 300 s test duration with a significantly lower wear rate of 1.70×10^{-6} $g·N^{-1}·s^{-1}$, corresponding to about a 99.5% reduction relative to the commercial formulations. This superior load-bearing performance is attributed to synergistic effects between polar oxygenated compounds in the bio-oil, the mechanically stable lithium stearate network, and load redistribution by finely dispersed RHA particles. Notably, this performance was achieved without the use of conventional sulfur- or phosphorus-based Extreme Pressure (EP) additives, demonstrating the potential of agro-residue-derived greases as a sustainable and high-performance alternative for lubrication applications.

Keywords: Bio-based grease, Extreme pressure additives, Boundary lubrication, Specific wear rate, Rice husk pyrolysis oil, Agricultural waste valorization, Sustainable lubricants.

1 | Introduction

Conventional lubricating greases play a critical role in reducing friction and wear in mechanical systems; however, most commercially available products are derived from petroleum based oils and metallic thickeners

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that present environmental disposal and long term sustainability concerns [1]. The dependence on finite fossil resources and the environmental persistence of conventional petroleum-based lubricants after leakage or disposal have intensified research into renewable and biodegradable alternatives [2].

Bio-based lubricants derived from renewable biomass sources have emerged as promising alternatives to conventional petroleum-derived lubricants, offering the potential for comparable tribological performance with reduced environmental persistence and improved resource renewability [3]. Biomass-derived oils containing polar oxygenated functional groups exhibit strong surface affinity, promoting adsorption-driven boundary film formation that mitigates direct asperity interaction under high load conditions [4]. These intrinsic polar functional groups improve surface adhesion and promote the formation of protective tribofilms during sliding contact. Despite these advantages, pure bio oils often exhibit limitations in oxidative stability and thermal resistance compared to conventional mineral oils, which can restrict their industrial application [2]. To address these shortcomings, recent research has explored chemical modification routes such as esterification and epoxidation, as well as the incorporation of solid additives to enhance load bearing capacity and Anti-Wear (AW) characteristics [5]. The inclusion of reinforcing particles has demonstrated measurable improvements in wear resistance in several bio grease systems, particularly under Extreme Pressure (EP) conditions.

Biomass pyrolysis offers an additional pathway for developing sustainable lubricants. The thermochemical conversion of agricultural residues generates bio oil rich in oxygenated organic compounds, alongside solid carbonaceous residues such as biochar and ash. Rice husk, an abundant agricultural by product rich in silica and carbon, represents a particularly attractive feedstock for such valorization. Although rice husk bio oil has primarily been investigated for fuel applications and diesel blending [6], its intrinsic lubricity and chemical composition suggest potential utility in lubrication formulations. Furthermore, studies indicate that pyrolysis derived residues and silica rich ash can enhance tribological behavior by supporting load and promoting protective film formation at sliding interfaces [7], [8].

While previous studies have examined bio lubricants, vegetable oil greases, and biomass derived additives independently, there remains limited systematic evaluation of grease formulations that simultaneously utilize pyrolyzed bio oil as the base fluid and biomass derived ash as a solid additive in direct comparison with commercial grease products. Addressing this gap, the present study investigates the production and performance evaluation of a lithium based grease formulated from rice husk pyrolyzed oil as the base oil and Rice Husk Ash (RHA) as an AW additive. The tribological performance of the formulated bio grease is compared with two commercial multipurpose greases under varying applied loads using standardized wear testing. By integrating biomass valorization with tribological assessment, this work contributes to the development of high performance and environmentally sustainable lubricant systems.

2 | Material and Method

2.1 | Materials

Rice husk was obtained from a local rice milling facility located in Ifite Ogwari, Ayamelum LGA, Anambra State, Nigeria. Stearic acid (≥98% purity) and lithium hydroxide monohydrate (≥98% purity) were procured from a local chemical supplier at Eke Awka market, Anambra State, Nigeria, and used as received without further purification. These reagents were employed for in situ synthesis of lithium stearate thickener via saponification.

RHA was produced through controlled combustion of rice husk at 450 °C for 4 hours in air using a muffle furnace at the foundry workshop of Nnamdi Azikiwe University. The ash was sieved to particle size below 150 μm prior to incorporation into the grease matrix.

All weighing operations were conducted using a Kolzp high precision digital balance with LCD display.

Two commercial lithium based multipurpose greases were used for comparative analysis:

I. X Grease: allied multipurpose bearing grease

II. Y Grease: atlas grease

The experimentally formulated rice husk grease was designated as Z Grease.

2.2 | Preparation of Rice Husk Feedstock

Raw rice husk was manually sorted to remove extraneous materials and sun dried for four days to reduce inherent moisture content. The dried husk was milled into fine powder using a flour milling machine.

Particle size uniformity was achieved through sequential sieving using 600 μm, 450 μm, and 150 μm mesh sieves. The processed feedstock was stored in airtight polyethylene bags to prevent moisture reabsorption prior to pyrolysis.

Fig. 1. Rice husk feedstock.

2.3 | Production of Rice Husk Ash

RHA was prepared by controlled combustion of rice husk in air using a muffle furnace at 450 °C for 4 hours at foundry workshop in Nnamdi Azikiwe university.

The resulting gray ash was cooled to ambient temperature and sieved through a 150 μm mesh to obtain uniform fine particles. The ash was stored in airtight containers for subsequent use as a solid AW additive.

Fig. 2. RHA.

2.4 | Pyrolysis of Rice Husk for Bio Oil Production

Pyrolysis was conducted in a fixed bed reactor. The prepared rice husk feedstock was loaded into the reactor and the reactor temperature was gradually increased to 600 °C. Thermal decomposition of the biomass produced bio oil, non-condensable gases, and solid bio char. Vapors were condensed and collected as liquid bio oil. The oil was filtered to remove suspended particulates and stored in sealed containers prior to grease formulation.

Fig. 3. Fixed Bed Reactor used for pyrolyzing feedstock.

2.5 | Grease Formulation

A Grade 2 lithium grease formulation was prepared using a base oil to thickener to additive mass ratio of 60:35:5.

For a 200 g batch:

I. 120 g rice husk pyrolyzed oil (base oil)
II. 70 g lithium stearate (thickener)
III. 10 g RHA (additive)

2.5.1 | Preparation of Lithium Stearate Thickener

Lithium stearate was synthesized via saponification of stearic acid with lithium hydroxide according to:

$$C_{17}H_{35}COOH + LiOH \rightarrow C_{17}H_{35}COOLi + H_2O.$$

Based on stoichiometric calculations, 68.6 g stearic acid reacted with 5.8 g lithium hydroxide to yield 70 g lithium stearate, with approximately 4.4 g water removed during heating.

Stearic acid was melted at approximately 50 °C, followed by gradual addition of lithium hydroxide under continuous stirring. The mixture was heated to approximately 110 °C for 60 minutes to ensure complete reaction and water evaporation.

2.5.2 | Grease synthesis

The synthesized lithium stearate was blended with 120 g of pyrolyzed rice husk oil preheated to 140 °C. The mixture temperature was raised to 170 °C and maintained for 30 minutes under continuous stirring.

The system was cooled to 80 °C before incorporation of 10 g RHA additive. Stirring continued for 5 minutes to ensure uniform dispersion. The grease was allowed to cool to room temperature and stored in airtight containers.

Fig. 4. Stirring the grease mixture.

Fig. 5. Grease at room temperature.

2.6 | Wear Resistance Testing

Tribological performance was evaluated using a Ball Wear Tester in accordance with ASTM D2266, DIN 51350, and IP 239 standards. This device showcases a rotating wheel bearing mechanism immersed in lubricating grease. As the wheel rotates within the grease, it emulates the frictional forces encountered in real-world scenarios. This straightforward apparatus offers invaluable insights into a lubricant's capacity to mitigate wear and sustain efficiency over time. Additionally, the applied load brings the bearing into contact with the rotating wheel bearing mechanism, ensuring a comprehensive simulation of operational conditions.

Tests were conducted at:

I. Applied loads: 800 g, 900 g, and 1000 g

II. Rotational speed: 1490 rpm

III. Maximum test duration: 300 seconds

In conducting the test, a total of 9 bearings was utilized. Precise measurements were taken using Kolzp high precision digital balance to ensure accuracy and reliability before and after each simulation.

Failure was identified by visible smoking and direct metal to metal contact.

Specific Wear Rate was calculated as:

$$K = \frac{\Delta m}{F \cdot t}$$

Force was calculated as:

$$F = \text{mass (g)} \times 0.00981$$

(since 1 g ≈ 0.00981 N)

Table 1. Applied load and corresponding force.

Load (g)	F (N)
800	7.848
900	8.829
1000	9.810

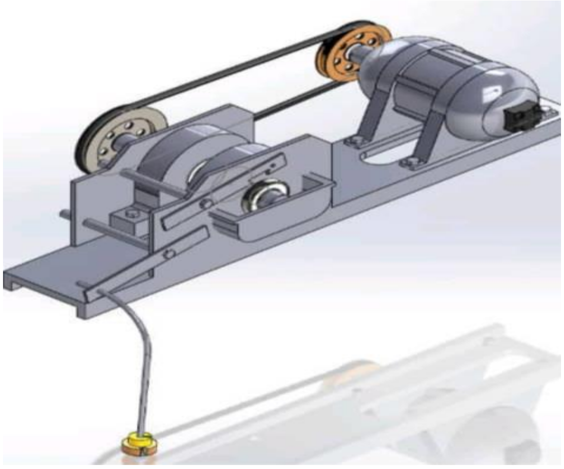

Fig. 6. Grease/bearing test rig apparatus.

Fig. 7. GBTR apparatus during test X.

Fig. 8. GBTR apparatus during test Y.

Fig. 9. GBTR apparatus during test Z.

Fig. 10. Steel bearing.

3 | Results and Discussion

Table 2. Comparative performance of grease X, Y, and Z under identical conditions.

	Applied Load (g)	F (N)	Bearing Int. Mass (g)	Bearing Fin. Mass (g)	Mass Difference (g)	Specific Wear Rate K ($g \cdot N^{-1} \cdot s^{-1}$)	Simulated Time (s^{-1})	Speed (v) (rpm)
X-Grease Tabular Analysis	800	7.848	7.338	7.336	0.002	8.5×10^{-7}	300	1490
	900	8.829	7.366	7.332	0.034	1.28×10^{-5}	300	1490
	1000	9.810	7.349	7.285	0.064	3.43×10^{-4}	19 (failed)	1490
	800	7.848	7.352	7.345	0.007	2.97×10^{-6}	300	1490

Y-Grease Tabular Analysis	900	8.829	7.336	7.295	0.041	1.55×10^{-5}	300	1490
	1000	9.810	7.343	7.322	0.021	3.57×10^{-4}	6 (failed)	1490
Z-Grease Tabular Analysis	800	7.848	7.360	7.357	0.003	1.27×10^{-6}	300	1490
	900	8.829	7.363	7.358	0.005	1.89×10^{-6}	300	1490
	1000	9.810	7.345	7.340	0.005	1.70×10^{-6}	300	1490

3.1 | Load Dependent Wear Behavior and Comparative Performance

The dependence of wear on applied load was evaluated through gravimetric determination of bearing mass loss under controlled tribological conditions at 1490 rpm for three grease formulations: two commercial greases (Allied and Atlas) and the rice husk derived grease (Z grease). Mass loss was normalized by load and time to obtain the specific wear rate (K).

At 800 g (approximately 7.85 N), all formulations exhibited minimal wear, with specific wear rates within the order of 10^{-6} to 10^{-7} $g\cdot N^{-1}\cdot s^{-1}$, indicating stable lubrication under moderate loading. However, as the load increased to 900 g (approximately 8.83 N), divergence in performance became evident. Both commercial greases showed marked increases in mass loss and specific wear rate, whereas Z grease maintained comparatively low wear.

The most significant contrast was observed at 1000 g (approximately 9.81 N). Neither commercial grease sustained the full 300 s test duration, with premature termination at 19 s for Allied and 6 s for Atlas, indicating catastrophic failure under severe loading. The corresponding specific wear rates increased sharply to approximately 3.4×10^{-4} and 3.6×10^{-4} $g\cdot N^{-1}\cdot s^{-1}$, reflecting transition into a severe wear regime. In contrast, the rice husk derived grease completed the full 300 s test with a specific wear rate of 1.70×10^{-6} $g\cdot N^{-1}\cdot s^{-1}$. This represents an approximate 99.5% reduction in wear rate relative to the average value of the commercial greases at the same load, corresponding to more than two orders of magnitude improvement in wear stability.

The divergence observed at elevated load is consistent with boundary lubrication theory, where lubricant performance depends on the ability to maintain protective films as contact pressure increases. Under boundary conditions, failure typically occurs when the structural integrity of the grease matrix collapses or when protective films become insufficient to prevent direct asperity interaction [9], [10]. The abrupt cessation of testing and sharp rise in wear rate for the commercial greases at 1000 g indicate rapid breakdown of load bearing structure and loss of effective surface separation.

3.2 | Failure Mechanism and Structural Stability under High Load

The catastrophic behavior observed for the commercial greases at 1000 g suggests collapse of the lubricating film and transition to extensive surface interaction. In high stress tribological contacts, such failure is often associated with breakdown of the thickener network, excessive oil bleed, or insufficient boundary film formation under increasing contact pressure.

The rice husk-derived grease (Z-grease), comprised of 60 wt % rice husk pyrolysis oil (base oil), 35 wt % lithium stearate thickener, and 5 wt % RHA additive, did not exhibit similar failure. The sustained low wear rate at the highest applied load indicates that the formulation maintained protective separation between bearing surfaces throughout the test duration.

The observed performance is the result of intrinsic formulation synergy, arising from adsorption of polar oxygenated species present in rice husk pyrolysis oil onto metallic surfaces, structural integrity provided by the lithium stearate thickener network, and stress redistribution effects of finely dispersed RHA particles. Polar functional groups common in biomass derived oils enhance adsorption onto metal surfaces and promote boundary film formation that reduces direct asperity contact under stress. Such adsorption driven mechanisms have been shown to contribute to wear reduction in polar lubricant systems [11].

Commercial greases frequently rely on chemically reactive EP and AW additives containing sulfur, phosphorus, or chlorine to form sacrificial tribofilms under severe boundary conditions [12]. In contrast, the Z grease formulation achieved stable load bearing performance without conventional sulfur or phosphorous based EP additives. The combined action of an adsorptive bio based oil, a mechanically stable lithium soap network, and functional biomass derived particulates appears to provide non reactive yet effective protection under boundary stress conditions.

Collectively, these findings demonstrate that the rice husk derived grease not only matches but exceeds the high load stability of the evaluated commercial products under the tested conditions.

3.3 | Sustainability and Resource Valorization Implications

Commercial lubricating greases commonly incorporate chemically reactive AW and EP additives to enhance load carrying capacity under boundary lubrication conditions. These additives, frequently based on sulfur and phosphorus containing chemistries such as Zinc Dialkyldithiophosphate (ZDDP), form protective tribochemical films on metal surfaces during high stress asperity interaction, thereby reducing adhesive wear and surface damage [13]. Such additives, while effective, introduce metal-containing and environmentally persistent species into lubricant systems, raising concerns regarding toxicity and environmental accumulation during disposal or leakage.

In contrast, the Z-grease formulation evaluated in this study contained no conventional sulfur or phosphorus-based EP additives, yet it maintained low specific wear rates and sustained operation at 1000 g without catastrophic failure. The observed performance is the result of intrinsic formulation synergy, arising from the adsorption of polar oxygenated species present in rice husk pyrolysis oil onto metallic surfaces, the structural integrity provided by the lithium stearate thickener network, and the stress redistribution effects of finely dispersed RHA particles. Studies on bio-based lubricants demonstrate that polar functional groups in renewable oils can enhance boundary film formation and reduce wear without reliance on traditional EP chemistries [14].

From a sustainability standpoint, rice husk represents an abundant agricultural residue with significant potential for valorization. When improperly managed, it contributes to waste accumulation and open burning emissions [15]. Pyrolysis transforms this residue into value-added products while supporting circular economy strategies for agricultural waste management [16]. Furthermore, bio-based lubricants derived from renewable feedstocks demonstrate advantages such as improved biodegradability and reduced dependence on fossil resources compared with petroleum-derived systems [3].

Incorporating rice husk pyrolysis oil as the base oil and RHA as a functional additive in the grease formulation therefore constitutes a dual valorization pathway that transforms agricultural waste into high performance tribological materials while reducing dependence on environmentally persistent EP additives. When combined with competitive tribological performance under elevated loads, this approach illustrates a resource efficient route toward more sustainable lubricant systems.

4 | Conclusion

This study evaluated the tribological performance of a rice husk–derived grease formulation under increasing load conditions and compared it with two commercial lithium-based greases. While all formulations exhibited comparable wear behavior at 800 g, significant divergence emerged at elevated loads. At 1000 g, both commercial greases failed prematurely, transitioning into a severe wear regime, whereas the rice husk–derived grease sustained the full 300 s test duration with a specific wear rate more than two orders of magnitude lower than the commercial counterparts.

The superior load-bearing stability of the bio-derived grease is attributed to intrinsic formulation synergy involving adsorption of polar oxygenated species from rice husk pyrolysis oil onto metallic surfaces, structural reinforcement provided by the lithium stearate thickener network, and stress redistribution facilitated by finely dispersed RHA particles. These combined mechanisms enabled sustained boundary lubrication without reliance on conventional sulfur- or phosphorus-based EP additives.

Beyond tribological performance, the integration of rice husk pyrolysis oil and RHA demonstrates a dual resource valorization pathway, converting agricultural residue into functional lubricant components. The ability to achieve competitive high-load stability without environmentally persistent EP additives highlights the potential of agro-waste–derived formulations as viable alternatives within sustainable lubricant development.

Overall, the findings demonstrate that rice husk–based grease formulations can provide effective load-bearing performance while supporting resource efficiency and circular material utilization, offering a technically and environmentally relevant advancement in grease technology.

5 | Recommendations

Future work should examine the long-term thermal and oxidative stability of rice husk–derived grease formulations under extended service conditions to assess durability beyond short-term testing. Further investigation into the influence of ash particle size distribution and surface modification on dispersion stability and wear behavior is recommended. Tribological evaluation under a wider range of operating speeds and contact geometries would help define application-specific performance limits. Finally, scale-up and life-cycle assessments are recommended to support potential industrial adoption

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