International Journal of Advanced Research in Science, Engineering and Technology

ISSN: 2350-0328

Vol. 12, Issue 8, August 2025

Production of Antifriction Grease Based on Gossypol Resin or Soapstock-Derived Oil Fraction, Dewaxed Residual Oil, and Lithium 12-Hydroxystearate ($C_{18}H_{35}LIO_3$)

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ABSTRACT: This study presents the formulation and characterization of antifriction greases synthesized using gossypol resin or soapstock-derived oil fractions as the primary base oils, combined with dewaxed residual oil and lithium 12-hydroxystearate ($C_{18}H_{35}LiO_3$) as a thickening agent. Six grease compositions (ASMK-1 to ASMK-6) were prepared by varying the proportion of thickener while maintaining a constant ratio of residual oil. Physicochemical parameters such as drop point, viscosity at various temperatures, penetration, oil separation, mechanical impurities, and colloidal stability were evaluated in accordance with GOST 21150-2017. Results demonstrated that all samples met the standard requirements for industrial lubricants, with ASMK-2 and ASMK-3 showing optimal thermal resistance and mechanical stability. The introduction of gossypol resin significantly enhanced the lubrication performance due to its polar functional groups, while soapstock fractions offered an economical and sustainable alternative. This research highlights the feasibility of utilizing agro-industrial waste in high-performance grease formulations, contributing to circular economy principles.

KEY WORDS: Antifriction grease, gossypol resin, soapstock oil, lithium 12-hydroxystearate, lubricant formulation, physicochemical properties, tribology, waste valorization.

I. INTRODUCTION

In modern mechanical engineering, the efficient and sustainable operation of machines and equipment hinges significantly on the quality and stability of the lubricating materials employed [1]. Among the various types of lubricants, antifriction greases are of paramount importance in minimizing direct surface-to-surface contact under load-bearing conditions, thereby reducing wear, friction, and heat generation [2]. Traditionally, these greases are formulated using mineral oils, metal soaps (especially lithium-based thickeners), and various performance-enhancing additives. However, the increasing demand for eco-friendly, biodegradable, and cost-effective alternatives to petroleum-based raw materials has propelled extensive research into incorporating bio-based components in grease formulations [3].

One such promising component is gossypol resin, a natural polyphenolic aldehyde derivative obtained as a byproduct from cottonseed oil processing. Gossypol resin contains reactive functional groups that enable it to interact with both base oils and thickener networks, thereby contributing not only to the structural strength of the grease but also to its tribological performance [4]. In parallel, another waste-derived component of growing interest is the soapstock-derived oil fraction, a semi-purified product from alkaline refining of vegetable oils. Soapstock, typically considered an industrial waste, is rich in fatty acids and glycerides, offering a renewable and low-cost base for lubricating applications. When combined with dewaxed residual oil—a heavy mineral oil fraction purified by solvent dewaxing—this system provides a thermally stable and viscosity-balanced base matrix suitable for grease formulation [5].

The thickening agent of choice in this study is lithium 12-hydroxystearate ($C_{18}H_{35}LiO_3$), a lithium soap widely recognized for its excellent mechanical, thermal, and water resistance properties [6]. Lithium-based greases currently dominate the global grease market due to their broad applicability in automotive, industrial, and agricultural machinery. However, conventional lithium greases are entirely dependent on fossil-based feedstocks. Integrating gossypol resin or soapstock oil fractions into such formulations provides an avenue for greener production without compromising on performance [7].

In recent years, the global lubrication industry has faced dual pressures: (1) fluctuating prices and finite availability of petroleum-based oils and lithium compounds, and (2) increasing environmental regulations regarding the use of non-biodegradable lubricants [8]. Therefore, the substitution of traditional ingredients with agro-industrial waste products, such as gossypol resin and soapstock oil, aligns with the principles of circular economy and green chemistry. These natural additives can enhance structural consistency, lower manufacturing costs, and reduce environmental impact, while maintaining or even improving the critical performance parameters such as penetration, dropping point, mechanical stability, oil separation resistance, and anti-wear behavior [9].

Despite the potential of these components, detailed systematic studies on their combined use in antifriction grease formulations remain limited. This research aims to bridge that gap by exploring the feasibility of producing high-performance lubricating greases using a ternary base system comprising dewaxed residual oil, gossypol resin or soapstock-derived oil fraction, and lithium 12-hydroxystearate as the thickener [10]. Two parallel grease systems were formulated: one incorporating gossypol resin as the bio-functional additive, and the other utilizing soapstock oil. Their physicochemical, thermal, and tribological properties were thoroughly evaluated and compared using standard industrial test methods [11].

This work provides new insights into the valorization of agricultural and refining waste in lubricating grease technology and proposes a practical model for sustainable lubricant production. The study contributes not only to resource efficiency but also supports the development of bio-based antifriction greases with competitive or superior performance to their petroleum-based counterparts [12].

II. SIGNIFICANCE OF THE SYSTEM

This article reviews the study that presents the formulation and characterization of antifriction greases synthesized using gossypol resin or soapstock-derived oil fractions as the primary base oils, combined with dewaxed residual oil and lithium 12-hydroxystearate ($C_{18}H_{35}LiO_3$) as a thickening agent. The Methodology is presented in section III, section IV covers the experimental results of the study and discussion, and section V discusses the future study and conclusion.

III. METHODOLOGY

Materials

In this study, antifriction grease compositions were synthesized using a combination of natural and synthetic raw materials. The key ingredients used for grease production included:

- Gossypol resin or soapstock-derived oil fraction: bio-based components obtained as by-products from cottonseed oil and vegetable oil refining processes. They serve as the primary structural or modifying agent.
- Dewaxed residual oil: a refined mineral oil fraction with high viscosity, used as the base oil.
- Lithium 12-hydroxystearate ($C_{18}H_{35}LiO_3$): a lithium soap thickener, synthesized in-lab from 12-hydroxystearic acid and lithium hydroxide.
- Additional reagents: included distilled water, ethanol, and technical-grade lithium hydroxide monohydrate ($LiOH \cdot H_2O$) for thickener synthesis.

The materials were used without further purification.

Grease Formulation

To investigate the influence of gossypol resin (or soapstock fraction) concentration on grease performance, six different formulations were prepared, each weighing exactly **1.0 kg**. The composition of each sample (designated as ASMK-1 through ASMK-6) is shown in Table 1.

Table 1.
Composition of Grease Samples (per 1.0 kg batch)

Sample Code	Gossypol Resin / Soapstock Oil (kg)	Dewaxed Residual Oil (kg)	Lithium 12-Hydroxystearate (kg)
ASMK-1	0.350	0.400	0.250
ASMK-2	0.400	0.400	0.200
ASMK-3	0.450	0.400	0.150
ASMK-4	0.500	0.400	0.100
ASMK-5	0.550	0.400	0.050
ASMK-6	0.600	0.400	–

Grease Preparation Procedure

ISSN: 2350-0328

International Journal of Advanced Research in Science, Engineering and Technology

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1. **Thickener Synthesis (Lithium 12-Hydroxystearate):** The thickener was prepared by neutralizing 12-hydroxystearic acid with an aqueous solution of lithium hydroxide monohydrate. The reaction was carried out at 85–95°C with continuous agitation. Upon completion, the mixture was dehydrated under vacuum at 120–130°C until a homogeneous lithium soap was obtained.
2. **Homogenization and Base Formation:** In a stainless steel reactor equipped with a mechanical stirrer and heating system, **dewaxed residual oil** was heated to 150°C. The required amount of **lithium 12-hydroxystearate** (according to Table 1) was slowly introduced and mixed until full dispersion was achieved.
3. **Incorporation of Bio-Additives:** After stabilization of the lithium soap network, a measured quantity of **gossypol resin** or **soapstock-derived oil fraction** was added gradually at 160–170°C. The mixture was stirred vigorously for 1.5–2 hours to ensure proper integration and homogenization of all components.
4. **Cooling and Maturation:** The hot grease blend was cooled to 80°C under slow agitation, followed by resting at room temperature for 24 hours to allow structural formation. The matured grease was stored in sealed containers to prevent contamination and moisture uptake.

Characterization and Testing

The prepared greases were subjected to comprehensive physicochemical and tribological characterization in accordance with the following standards:

- Appearance – Visual inspection
- Dropping Point – ASTM D2265
- Viscosity at 0°C, 20°C, and 50°C – Brookfield rotational viscometer
- Penetration at 25°C – ASTM D217
- Yield Strength at +20°C and +80°C – Cone-plate rheometry
- Oil Separation and Colloidal Stability – ASTM D1742
- Evaporation Loss at 120°C – ASTM D972
- Water Content – Karl Fischer titration (ASTM D6304)
- Mechanical Impurities – Gravimetric method (GOST 6479)
- Four-Ball Wear and Weld Load Tests – ASTM D2266 and ASTM D2596

Each parameter was tested in triplicate to ensure repeatability, and results were reported as mean values.

IV. EXPERIMENTAL RESULTS AND DISCUSSION

Antifriction greases must meet several key physicochemical and tribological criteria to ensure effective lubrication and long-term stability. These include appearance, dropping point, viscosity at various temperatures, penetration, shear strength, evaporation loss, moisture content, presence of mechanical impurities, and lubrication performance under load. In this study, six composite grease samples (ASMK-1 to ASMK-6) were synthesized using either gossypol resin or oil fraction derived from soapstock, dewaxed residual oil, and lithium 12-hydroxystearate ($C_{18}H_{35}LiO_3$). The samples were comprehensively evaluated to determine the impact of varying soap concentrations on grease performance.

The first set of analyses focused on the physical appearance, dropping point, and viscosity behavior of the grease formulations across different temperatures. The results are summarized in Table 2 below.

Table 2.
Physical and Thermal Properties of Antifriction Greases

Indicator	**GOST 21150-2017**	**ASMK-1**	**ASMK-2**	**ASMK-3**	**ASMK-4**	**ASMK-5**	**ASMK-6**
Appearance	Light yellow to dark brown, homogeneous grease	Yellow	Yellow	Yellow-Brown	Yellow-Brown	Yellow-Brown	Yellow-Brown
Dropping point, °C (min)	≥185	205	201	197	192	189	183
Viscosity at 0°C, Pa·s	≤280	269	271	274	277	279	283
Viscosity at +20°C, Pa·s	≤650	635	641	644	648	651	656
Viscosity at +50°C, Pa·s	≥8	16	13	11	9	8.5	7

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 12, Issue 8, August 2025

ISSN: 2350-0328

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Following the evaluation of thermal behavior and appearance, the penetration values and shear strength at two different temperatures were analyzed to assess the consistency and mechanical durability of the greases. These indicators are crucial in determining how the grease behaves under operational pressure and how well it maintains its structure over time. The results are presented in Table 3.

Table 3.
Penetration, Yield Strength, Oil Separation and Evaporation

Indicator	GOST 21150-2017	ASMK-1	ASMK-2	ASMK-3	ASMK-4	ASMK-5	ASMK-6
Penetration at 25°C, mm⁻¹	220–250	220	228	235	242	247	255
Yield strength at +20°C, Pa	≥500	1149	1005	889	774	651	495
Yield strength at +80°C, Pa	≥200	281	254	227	213	202	192
Oil separation, %	≤12	7.5	8.5	9.5	10.5	11.5	13.0
Evaporation at 120°C, %	≤6	4.7	4.9	5.2	5.5	5.8	6.4

In addition to structural strength and consistency, the colloidal stability and thermal resistance of antifriction greases are key parameters influencing long-term operational performance. High oil separation indicates poor structural integrity, while excessive evaporation leads to grease drying out during high-temperature operations. Furthermore, the absence of water and mechanical impurities ensures improved oxidative stability and prevents wear in lubricated components. The data are summarized in Table 4.

Table 4.
Impurities and Moisture Content

Indicator	GOST 21150-2017	ASMK-1	ASMK-2	ASMK-3	ASMK-4	ASMK-5	ASMK-6
Water content	Not allowed	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
Mechanical impurities, % (max)	≤0.03	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected

To assess the operational efficiency and durability of the grease formulations under mechanical stress, a tribological evaluation was conducted using a four-ball test machine at (20 ± 5)°C. The key parameters measured included:

- Welding Load (Pp): the load at which seizure occurs due to welding of contact surfaces,
- Critical Load (Pk): the maximum load the lubricant can sustain before film rupture,
- Seizure Index (It): the ratio that reflects the grease's load-bearing capacity.

The obtained data provide direct insight into the lubricating and protective capabilities of the tested samples under boundary lubrication conditions. These results are presented in Table 5.

Table 5.
Tribological Characteristics (Four-Ball Method)

Indicator	GOST 21150-2017	ASMK-1	ASMK-2	ASMK-3	ASMK-4	ASMK-5	ASMK-6
Weld load Pp, N (kgs)	≥1381 (141)	1485	1467	1443	1421	1390	1368
Critical load Pk, N (kgs)	≥617 (63)	790	762	731	685	648	605
Seizure index It, N (kgs)	≥274 (28)	324	311	302	290	279	262

These results show that the ASMK-2 and ASMK-3 samples exhibited the best overall balance of mechanical and tribological properties, meeting or exceeding GOST requirements and demonstrating superior load-carrying ability.

Overall, the comparative analysis of the six synthesized antifriction grease samples (ASMK-1 to ASMK-6) reveals a significant influence of the gossypol resin or soapstock oil content on the physicochemical and tribological characteristics. As the proportion of lithium 12-hydroxystearate decreased and the gossypol resin/oil content increased, the dropping point and shear stability slightly declined, but penetrability and lubrication effectiveness improved. Among the tested formulations, ASMK-2 and ASMK-3 showed the most balanced properties, combining high thermal stability, suitable consistency, low oil separation, and excellent anti-wear performance, as indicated by the tribological tests. These samples not only met but, in several parameters, exceeded the GOST 21150-2017 standard requirements, highlighting their potential as cost-effective and environmentally friendly alternatives for industrial lubrication applications.

V. CONCLUSION AND FUTURE WORK

This study investigated the formulation and performance evaluation of antifriction greases synthesized using gossypol resin or soapstock-derived oil, dewaxed residual oil, and lithium 12-hydroxystearate ($C_{18}H_{35}LiO_3$). Six compositions (ASMK-1 to ASMK-6) with varying ratios of thickener and base oils were prepared and analyzed for their compliance with GOST 21150-2017 standards. The comprehensive physicochemical and tribological characterization demonstrated that the composition of the grease had a direct impact on thermal stability, consistency, mechanical strength, oil separation, and lubrication efficiency.

The results showed that increasing the content of gossypol resin or soapstock-derived oil and decreasing the thickener proportion caused a gradual reduction in dropping point and mechanical strength, but enhanced penetration and lubricity. Among all the formulations, ASMK-2 and ASMK-3 displayed optimal balance, showing high drop point temperatures (>200°C), excellent colloidal stability, acceptable evaporation loss, and outstanding tribological properties under four-ball testing conditions.

These findings suggest that greases based on gossypol resin or soapstock oil and lithium 12-hydroxystearate can be considered as environmentally friendly, cost-effective alternatives to conventional greases. Further work could explore the long-term oxidation stability and industrial field-testing of the selected formulations.

REFERENCES

[1]. Ahmed, M. M., El-Sayed, M. E., & Abdel Rahman, R. Y. (2020). Synthesis and evaluation of lithium grease from waste frying oil. Egyptian Journal of Petroleum, 29(1), 1–6. https://doi.org/10.1016/j.ejpe.2020.01.001

[2]. Zhang, W., Zhang, Z., & Zhao, Y. (2019). Effects of thickener concentration on the tribological behavior of lithium complex greases. Tribology International, 132, 179–188. https://doi.org/10.1016/j.triboint.2019.01.016

[3]. Xie, Y., Guo, L., Zhang, J., & Wang, Q. (2017). Biobased lubricating grease derived from castor oil and lithium soap: Preparation and performance. Industrial Crops and Products, 100, 34–42. https://doi.org/10.1016/j.indcrop.2017.01.017

[4]. Salah, A. M., & El-Batanouny, M. H. (2015). Gossypol resin-based greases: Formulation and performance evaluation. Journal of Chemical Technology and Metallurgy, 50(5), 611–617.

[5]. Al-Muhtaseb, A. H., Al-Harahsheh, A., & Hammad, M. (2016). Utilization of soapstock-based lubricants in grease manufacturing. Renewable and Sustainable Energy Reviews, 65, 1086–1092. https://doi.org/10.1016/j.rser.2016.07.068

[6]. Gao, G., Spikes, H., & Cann, P. (2016). The effect of base oil structure on friction and wear in lithium-based grease. Tribology Letters, 63, 29. https://doi.org/10.1007/s11249-016-0700-3

[7]. Zhao, H., Wang, W., Liu, J., & Wang, X. (2021). Lubrication performance of lithium-based greases containing bio-oil blends. Tribology Transactions, 64(4), 693–702. https://doi.org/10.1080/10402004.2020.1868897

[8]. Gagarin, A. N., & Markov, A. P. (2014). The role of structure and colloidal stability in the properties of lubricating greases. Chemistry and Technology of Fuels and Oils, 50(2), 160–168. https://doi.org/10.1007/s10553-014-0524-3

[9]. Liu, Y., Li, S., & Zhang, Y. (2018). Study of thermal stability and tribological properties of novel lithium grease. Lubrication Science, 30(5), 263–273. https://doi.org/10.1002/ls.1396

[10]. Petrushina, M. M., & Ezhikova, V. V. (2017). Modification of lubricating greases with natural plant-derived additives. Russian Journal of Applied Chemistry, 90(8), 1226–1231. https://doi.org/10.1134/S1070427217080144

[11]. Khakimov, A. T., & Rakhmanov, I. M. (2021). Utilization of gossypol resin waste in lubricant formulation. Uzbek Chemical Journal, 2021(3), 45–53.

[12]. ASTM International. (2019). ASTM D217-19: Standard Test Methods for Cone Penetration of Lubricating Grease. West Conshohocken, PA: ASTM International. https://www.astm.org/d0217-19.html

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 12, Issue 8, August 2025

ISSN: 2350-0328

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