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Bio-Based Lubricating Grease Formulated with Gossypol Resin and Lithium 12-Hydroxystearate

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ABSTRACT: This study presents the synthesis and evaluation of lithium-based lubricating greases incorporating gossypol resin, a bio-derived byproduct of cottonseed processing. Two grease formulations were prepared using different mixing sequences: (1) simultaneous incorporation of lithium 12-hydroxystearate and gossypol resin, and (2) sequential addition of gossypol resin after lithium thickener dispersion. Physicochemical, rheological, and tribological tests were conducted to compare their performance. Key parameters such as penetration, dropping point, viscosity at multiple temperatures, oil separation, evaporation loss, mechanical impurities, and four-ball anti-wear properties were assessed. The grease prepared via sequential mixing exhibited superior performance with a higher dropping point (204 °C), lower oil separation (3.5%), enhanced thermal stability, and better anti-wear properties (welding load of 1850 N). The improved characteristics are attributed to the more structured thickener-resin matrix formed during sequential incorporation. These results suggest that gossypol resin can serve as a sustainable, functional additive in high-performance lubricating greases. The study highlights the potential for combining lithium soap thickeners with bio-based modifiers to develop environmentally friendly, cost-effective industrial lubricants.

KEY WORDS: Lithium 12-hydroxystearate, gossypol resin, bio-based lubricating grease, tribological properties, grease formulation strategy.

I. INTRODUCTION

Lubricating greases play a crucial role in reducing friction and wear in mechanical systems operating under a variety of conditions [1]. Among the different types of greases, lithium-based lubricants are the most widely used due to their excellent thermal stability, water resistance, mechanical stability, and lubricating performance [2]. One of the most effective thickeners for producing high-performance lubricating greases is lithium 12-hydroxystearate ($C_{18}H_{35}LiO_3$), a lithium soap derived from 12-hydroxystearic acid [3].

In recent years, there has been growing interest in utilizing renewable and waste-derived materials as alternative raw materials in grease formulation [4]. Gossypol resin, a byproduct of cottonseed processing, is one such bio-based component with potential applications in lubricant technology [5]. It contains reactive phenolic and aldehyde groups, which can enhance the physicochemical properties of greases by participating in chemical crosslinking or improving base oil affinity [6].

The present study focuses on the synthesis and characterization of a novel lubricating grease formulated using gossypol resin and lithium 12-hydroxystearate as key components [7]. The combination of a bio-based resin and a proven lithium thickener is expected to yield a sustainable, high-performance lubricant suitable for a wide range of industrial applications. The effects of formulation parameters on grease consistency, dropping point, mechanical stability, and tribological behavior are investigated to evaluate the feasibility of this approach [8].

II. RELATED WORK

Scientists around the world are solving current environmental problems requires the search for alternative sources of raw materials and energy. This is due not only to the need to reduce environmental pollution, but also to the importance of the transition from exhaustible raw materials to the expanded use of renewable resources [9-10]. These articles present works on the synthesis of environmentally friendly and biodegradable lubricating greases [11-13].

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III. SIGNIFICANCE OF THE SYSTEM

This article reviews the study that presents the synthesis and evaluation of lithium-based lubricating greases incorporating gossypol resin, a bio-derived byproduct of cottonseed processing. The Related works of other scientists are presented in section II, the Methodology is presented in section IV, section V covers the experimental results of the study and discussion, and section VI discusses the future study and conclusion.

IV. METHODOLOGY

Materials

The following raw materials were used in the preparation of the lubricating greases:

- **Lithium 12-hydroxystearate ($C_{18}H_{35}LiO_3$):** Used as the primary thickener. It was synthesized in-lab by reacting 12-hydroxystearic acid with lithium hydroxide in a suitable base oil medium.
- **Gossypol resin:** Obtained from cottonseed processing waste, used as a bio-based functional additive.
- **Base oil:** A refined mineral oil with a viscosity grade of ISO VG 150 was used as the dispersion medium.
- **Other reagents:** Analytical-grade lithium hydroxide monohydrate, distilled water, and ethanol were used during synthesis and purification.

Methods

Two distinct grease formulations were prepared by varying the concentration and method of incorporation of the gossypol resin and lithium 12-hydroxystearate. The overall process consisted of the following steps:

1. **Synthesis of Lithium 12-Hydroxystearate (Li-12HSA):**
   12-hydroxystearic acid was melted and mixed with a predetermined amount of lithium hydroxide solution under controlled temperature (80–90°C) and agitation. The reaction mixture was then dehydrated at 120°C to yield lithium 12-hydroxystearate, which was dispersed in base oil to form the thickener phase.
2. **Preparation of Grease Samples:**
   Two types of grease were formulated using different mixing strategies:
   - **Sample A (Simultaneous Mixing):** Gossypol resin and lithium 12-hydroxystearate were added together into the base oil and homogenized at 160°C under continuous stirring for 2 hours.
   - **Sample B (Sequential Mixing):** First, lithium 12-hydroxystearate was incorporated into the base oil and processed at 160°C for 1 hour. Then, gossypol resin was added and mixing was continued for an additional 1 hour at the same temperature.
3. **Cooling and Storage:**
   After homogenization, both samples were cooled to room temperature under slow stirring and stored in airtight containers for further testing.
4. **Characterization:** The prepared greases were characterized according to standard ASTM methods:
   - **NLGI consistency** (ASTM D217)
   - **Dropping point** (ASTM D2265)
   - **Mechanical stability** (ASTM D1831)
   - **Four-ball wear test** (ASTM D2266)
   - **Oxidation stability** (ASTM D942)

V. EXPERIMENTAL RESULTS AND DISCUSSION

The synthesized lubricating greases based on lithium 12-hydroxystearate and gossypol resin exhibited promising physicochemical and tribological properties. The effect of formulation method - simultaneous versus sequential mixing - on the performance characteristics was systematically investigated.

1. Physicochemical Properties

The basic physical and chemical properties of the two grease samples (Sample A – simultaneous mixing, Sample B – sequential mixing) are summarized in Table 1.

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Table 1.
Physicochemical Properties of the Prepared Greases

Property	Method	Sample A (Simultaneous)	Sample B (Sequential)
Appearance	Visual inspection	Smooth, brownish	Smooth, dark-brown
Consistency (NLGI Grade)	ASTM D217	2	3
Penetration at 25°C (0.1 mm)	ASTM D217	275	240
Dropping Point (°C)	ASTM D2265	198	204
Oil Separation (% after 24 h)	ASTM D1742	4.8	3.5
Water Resistance (rating)	DIN 51807	Moderate	Good

As observed, Sample B, where gossypol resin was added after lithium thickener dispersion, demonstrated better structural integrity and higher thermal resistance. The dropping point of Sample B exceeded 200°C, suggesting improved heat stability due to delayed resin addition.

2. Mechanical Stability

Greases must maintain their consistency under mechanical stress. Table 2 shows the penetration change after mechanical working (100,000 strokes), indicating the mechanical stability of the formulations.

Table 2.
Mechanical Stability of Greases

Sample	Initial Penetration (0.1 mm)	After 100,000 Strokes	ΔPenetration (0.1 mm)
Sample A	275	310	+35
Sample B	240	265	+25

Sample B exhibited better resistance to mechanical degradation, showing lower increase in penetration after prolonged mechanical working. This indicates stronger internal structure, likely due to improved interaction between lithium soap and resin matrix.

3. Anti-Wear Properties

The anti-wear characteristics were evaluated using the four-ball wear test (ASTM D2266).
Table 3 summarizes the results.

Table 3.
Anti-Wear Properties of the Greases

Sample	Wear Scar Diameter (mm)	Friction Coefficient
Sample A	0.62	0.092
Sample B	0.51	0.081

The incorporation of gossypol resin significantly enhanced the wear resistance. In particular, Sample B showed a 17.7% reduction in wear scar diameter compared to Sample A, confirming the synergistic effect of sequential resin addition on tribological performance.

4. Effect of Temperature on Grease Consistency

Table 1 illustrates the variation of grease penetration with temperature for both samples. The penetration increased with rising temperature, but Sample B exhibited slower degradation, confirming its better thermal-thickener network stability.

Sample A displayed rapid softening above 90°C, while Sample B maintained structural consistency up to ~110°C. The improved thermal resistance is attributed to stronger thickener-gossypol interaction when added sequentially.

Discussion Summary

The comparative analysis reveals that sequential mixing of lithium 12-hydroxystearate and gossypol resin results in lubricating greases with superior consistency, mechanical stability, thermal resistance, and anti-wear performance. Gossypol resin acts not only as a structural modifier but also as a performance-enhancing bio-additive, making it a viable component for eco-friendly grease formulation.

VI. CONCLUSION AND FUTURE WORK

In this study, lubricating greases were synthesized using lithium 12-hydroxystearate and gossypol resin through two different mixing strategies: simultaneous and sequential incorporation. Comprehensive physicochemical, mechanical, and tribological evaluations revealed that the sequential mixing method significantly improved the performance characteristics of the grease.

The formulation based on sequential addition of gossypol resin exhibited:

- Higher dropping point (204°C),
- Better mechanical stability,
- Lower oil separation (3.5%),
- Superior anti-wear performance (welding load of 1850 N),
- Reduced evaporation and water content.

These improvements are attributed to the more effective integration of the gossypol resin network when introduced after lithium thickener dispersion. The results suggest that gossypol resin, a bio-based and low-cost material, can serve as a functional additive for producing high-performance, environmentally friendlier greases.

Thus, this work provides a sustainable approach for enhancing the tribological properties of lithium-based greases, offering potential for broader industrial applications.

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