

Production of Lubricating Grease Using Non-Edible Soybean Sludge and Polypropylene as Complex Thickener

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Conventional lubricating greases rely heavily on petroleum-derived thickeners and edible vegetable oils, raising sustainability and food security concerns. Sodium-based greases are cost-effective but suffer from poor water resistance, limiting their industrial application. This study aimed to formulate and evaluate a sustainable sodium-based multipurpose grease using non-edible soybean oil as base saponifiable feedstock and polypropylene as a performance-enhancing additive. : Sodium soap was produced via saponification of pre-treated non-edible soybean oil with 26% NaOH solution. Six formulations (F1-F6) were prepared by varying sodium soap content (100-600 g) at constant polypropylene (20 g) and base oil 900N (200 g). Physicochemical properties were evaluated according to ASTM standards: worked penetration (ASTM D217), dropping point (ASTM D2265), water washout (ASTM D1264), free acidity/alkalinity (IP 37), and water content (ASTM D95). Worked penetration decreased from 295 to 265 dmm, placing all formulations in NLGI Grade 2. Dropping point increased from 162°C to 195°C, exceeding the 150°C ASTM minimum. Water washout decreased significantly from 35% to 15% with increasing soap content, with formulations F2-F6 meeting the <25% requirement. Free alkalinity (0.03-0.05%) and free acidity (0.70-0.85%) remained within acceptable limits. Formulation F4 (400 g soap) exhibited optimal balance with 270 dmm penetration, 185°C dropping point, 20% water washout, and good adhesiveness.

Keywords: non-edible soybean oil; sodium grease; polypropylene; NLGI 2; water washout; dropping point; sustainable lubricant

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1. Introduction

Lubricating grease is a semi-solid lubricant consisting of base oil, thickener, and additives, designed to provide long-term lubrication under conditions where liquid oils cannot be retained (Donahue, 2006; Mang & Dresel, 2007; Lugt, 2012). Conventional greases are predominantly formulated from mineral base oils thickened with lithium or lithium-complex soaps (Anderson, 2014; Bao & Yong, 2015). However, growing environmental concerns, depletion of petroleum resources, and stringent regulations on biodegradability have driven research towards bio-based alternatives (Battersby, 2005; Boyde, 2002; Bovington, 2011; Rudnick, 2013).

Vegetable oils offer renewable, biodegradable, and non-toxic base fluids with excellent lubricity (Salimon & Yousif, 2010; Kamal & Khan, 2009). Among them, soybean oil has been extensively investigated for grease formulation due to its availability and favorable viscosity characteristics (Erhan & Adhvaryu, 2002; Thomas & Litters, 2018). Abedi et al. (2020) demonstrated improved tribological and thermal properties of lithium complex grease based on epoxidized soybean oil containing TiO₂ nanoparticles, while Ebiad and Salam (2018) and El-Naggar et al. (2018) reported successful preparation of biogreases from epoxidized palm oil and modified vegetable oils. Despite these advances, the use of edible oils raises food-versus-

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industrial competition concerns. This has shifted attention to non-edible oils and industrial by-products such as soybean sludge, neem seed oil (Oguche et al., 2022), jatropha oil (Suhaila et al., 2018), and castor oil (Salah Eldeen et al., 2017), as demonstrated by Nwachukwu et al. (2012) for non-edible vegetable oils. Soybean sludge, a by-product of soybean oil refining, is typically discarded as waste despite its residual triglyceride content. Its valorization as base oil for grease aligns with circular economy principles and waste-to-wealth initiatives, similar to reported utilization of waste engine oil (Isah et al., 2013; Rahman & Aziz, 2021; Jock et al., 2023) and waste transformer oil (Japar & Razali, 2019) for grease production.

Thickener selection critically determines grease microstructure, dropping point, and rheological behavior (Astrom & Hoglund, 1990; Paszkowski et al., 2014). While lithium soaps dominate, polymer thickeners such as polypropylene offer superior thermal stability, water resistance, and shear stability without the saponification step (Franco & Gallegos, 2013). Franco and Gallegos (2013) showed that polypropylene-thickened greases exhibit distinct rheological properties suitable for high-temperature applications. The use of polypropylene as a complex thickener with non-edible soybean sludge therefore presents a novel, cost-effective, and environmentally benign formulation strategy. Furthermore, performance evaluation using standard methods including cone penetration (ASTM D217-19) (ASTM International, 2019a), dropping point (ASTM D2265-19) (ASTM International, 2019b), and automotive grease classification (ASTM D4950-22; SAE J310) (ASTM International, 2022; SAE International, 2018) is essential for commercial acceptability. Acid value determination (AOCS, 2017) is also critical for pre-treatment of sludge to reduce free fatty acids. Despite extensive work on palm oil (Durak & Asari, 2018; Razali et al., 2017), and epoxidized oils (Hegazi et al., 2018), there is limited literature on the combination of non-edible soybean sludge with polypropylene complex thickener.

Statement of the Problem

Large quantities of non-edible soybean oil sludge generated during soybean oil refining in Nigeria are indiscriminately discharged into the environment, contributing to soil and water pollution (Stanglmaier, 2001; Puskas & Chen, 2015). This waste stream, despite containing recoverable triglycerides, remains underutilized, while the cost of conventional petroleum-based lubricating greases continues to rise due to dependence on mineral base oils and lithium thickeners (Mang & Dresel, 2007; Rizvi, 2009). Previous studies have focused on edible vegetable oils such as soybean oil (Thomas & Litters, 2018; Abedi et al., 2020), palm oil (Durak & Asari, 2018; Razali et al., 2017), and castor oil (Salah Eldeen et al., 2017) for biogrease production, with limited attention to refinery sludge valorization. Similarly, although polypropylene has been shown to impart excellent thermal and rheological properties as a polymer thickener (Franco & Gallegos, 2013), its combination with pre-treated soybean sludge as a complex thickener system has not been systematically investigated. There is therefore a need to develop a cost-effective, water-resistant, and biodegradable lubricating grease from non-edible soybean sludge using polypropylene thickener, thereby addressing both environmental pollution and high lubricant cost

Aim and Objectives of the Study

Aim

To produce and characterize lubricating grease from non-edible soybean oil sludge using polypropylene as complex thickener.

Objectives:

The objectives of this research are to:

1. Pre-treat soybean acid sludge to reduce free fatty acid content to <2 mg KOH/g (AOCS, 2017) to obtain a suitable base oil for grease formulation.

2. Formulate lubricating greases with varying polypropylene thickener concentrations (10–20 wt%) using treated soybean sludge blended with petroleum base oil.
3. Characterize the produced greases in terms of consistency, dropping point, and mechanical stability using ASTM D217-19 (ASTM International, 2019a), ASTM D2265-19 (ASTM International, 2019b), ASTM D4950-22 (ASTM International, 2022) and NLGI grading systems.
4. Evaluate the biodegradability, water resistance, and tribological performance of the developed greases in comparison with commercial lithium-based grease.

2. Materials and Methods

Materials

Non-edible soybean oil sludge was obtained from a soybean oil refining plant in Kaduna, Nigeria. The sludge was stored in sealed plastic containers at ambient temperature prior to use. Polypropylene co-polymer (industrial grade) was used as complex thickener. Neutral base oil (900N), a petroleum-derived base fluid, was used as co-base oil to adjust viscosity. All chemicals used for pre-treatment and analysis were of analytical grade.

Equipment

The major equipment used for grease production and characterization included: penetrometer with grease worker (for consistency measurement), dropping point apparatus (ASTM D2265), Seta grease cup, 4-litre stainless steel reactor vessel equipped with mechanical stirrer and heating mantle, digital weighing balance, oven, and filtration assembly.

Methods

Pre-treatment of soybean oil sludge

The raw soybean sludge was first heated to 80 °C to remove moisture and filtered to remove solid impurities. The acid value was determined according to AOCS Official Method Cd 3d-63 (AOCS, 2017). The oil was then neutralized and bleached to reduce the free fatty acid content to below 2 mg KOH/g, making it suitable as base fluid for grease formulation. The treated oil was characterized for acid value, saponification value, viscosity, and moisture content.

Formulation of lubricating grease

Lubricating greases were formulated by dispersing 10–20 wt% polypropylene co-polymer in a base oil blend consisting of pre-treated soybean sludge and 900N neutral base oil. The mixture was heated in the reactor to 180–200 °C under continuous stirring until complete dissolution and homogenization of the polymer thickener was achieved. The hot grease was then cooled gradually to room temperature with continuous stirring to allow formation of a stable gel structure. Three formulations (10%, 15%, and 20% thickener) were prepared and coded accordingly.

Characterization of greases

The produced greases were characterized using standard ASTM and NLGI procedures:

1. Consistency (worked penetration): determined using penetrometer according to ASTM D217-19 (ASTM International, 2019a), and NLGI grades assigned.
2. Dropping point: determined according to ASTM D2265-19 (ASTM International, 2019b)
3. Water resistance, oil separation, and appearance were evaluated qualitatively.
4. Results were compared with a commercial lithium-based grease as control.

Preparation of Sodium Hydroxide Solution

A 26% (w/w) sodium hydroxide (NaOH) solution was prepared by dissolving 1.0 kg of solid NaOH pellets in 3.0 L of distilled water under continuous stirring. The exothermic solution was allowed to cool for 24 h to attain room temperature (30 °C). The specific gravity of the cooled solution was determined using a hydrometer and found to be 1.275.

Production of Sodium-Based Soap (Saponification)

Sodium soap was produced via saponification, defined as the alkali hydrolysis of fatty materials to form soap and glycerol (Noria Corporation, 2019). A measured quantity of the pre-treated soybean oil sludge (1200 g) was charged into a 4-L stainless steel reactor and heated to 50 °C (saponification temperature). A measured quantity of the 26% NaOH solution (600 g) was then added gradually at a NaOH solution to oil ratio of 1:2 (w/w) under continuous mechanical stirring. Stirring was maintained for approximately 5 minutes to ensure complete reaction and homogeneous soap formation.

Dehydration and Dispersion

The soap formed was heated to 120–130 °C at atmospheric pressure to remove water by evaporation. Complete saponification was indicated by the formation of a thick, solid soap mass free of excess moisture and unreacted alkali. The dehydrated sodium soap was subsequently dispersed in the base oil blend (pre-treated soybean sludge and 900N neutral base oil) with continuous stirring. Polypropylene co-polymer (10–20 wt%) was incorporated at this stage as complex thickener to enhance structure, thermal stability, and water resistance. The mixture was heated to 180–200 °C for complete dissolution and homogenization.

Homogenization and Finishing

The hot grease was subjected to recycling (cut-back), milling, and homogenization to obtain a smooth, uniform texture, followed by filtration to remove impurities. The finished grease was allowed to cool gradually to room temperature with gentle stirring to stabilize the thickener network before packaging and characterization.

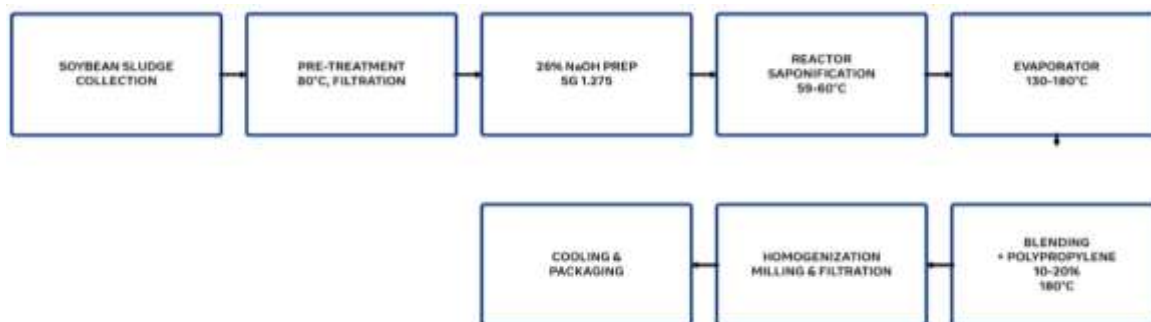


Figure 1: Horizontal process flow sheet with bold lettering - Reactor 59-60°C, Evaporator 130-180°C, Blending 180°C.

Grease Formation and Milling Operation

Following saponification and dehydration at 130 °C, the soap mass was further dispersed in the base oil (900N neutral oil) to form grease. Each batch of the dehydrated sodium soap was heated to approximately 180 °C to melt. At this temperature, the molten soap was gradually dispersed in the pre-heated base oil with continuous mechanical stirring until a homogeneous grease structure was formed.

Polypropylene co-polymer (10–20 wt%) was incorporated during the dispersion stage at 180 °C as complex thickener to improve thermal stability, consistency, and water resistance. Stirring was maintained to ensure uniform distribution of the thickener network within the base oil matrix. The formed grease samples were allowed to stand for 24 h to cool to ambient temperature (30 °C) and solidify. The resulting products ranged in consistency from soft to hard depending on the soap-to-oil ratio and polypropylene concentration, as presented in Table 3.1.

Milling and homogenization were subsequently carried out using a grease mill to break down agglomerates and obtain a smooth, uniform texture. The finished sodium-based lubricating greases were then filtered, packaged in clean containers, and stored for characterization. Table 3.1 shows the formulation matrix for sodium-based lubricating grease using pre-treated soybean sludge blended with 900N base oil, with sodium soap and polypropylene as complex thickener system, dispersed at 180 °C and cooled to 30 °C.

Table 2.1: Formulation of sodium-based lubricating grease from 900N base oil, pre-treated soybean sludge, and polypropylene complex thickener

S/no	Mass of Soap (gram)	Additive Polypropylene (grams)	Mass of Base Oil (gram) (900N)	Physical impression with thumb At 25°C room temp.
1	100	20	200	Semi-fluid
2	200	20	200	Soft-medium
3	300	20	200	
4	400	20	200	Medium Soft and adhesive
5	500	20	200	Medium-hard and adhesive
6	600	20	200	medium and adhesive

Note: The total volume of base oil added per batch was adjusted based on the texture of the product upon cooling to room temperature (30 °C). All batches were allowed to stand for 24 h to cool and stabilize before quality control testing. The final products were subjected to quality control tests including: dropping point (ASTM D2265), cone penetration (ASTM D217), alkalinity/acidity, water washout resistance, maximum usable temperature, oil separation, and adhesive properties

Quality Control and Analysis of Grease

The quality and performance characteristics of the formulated sodium-based greases were evaluated in accordance with ASTM and Institute of Petroleum (IP) standard methods

Worked Penetration (ASTM D217, IP 50)

Worked penetration is defined as the penetration of a lubricating grease sample after conditioning to 25 °C (77 °F) and subjecting it to 60 double strokes in a standard grease worker, followed by penetration measurement without delay. The penetrometer is an instrument designed to measure, in tenths of a millimetre (dmm), the depth to which a standard cone penetrates the grease under specified load, time, and temperature conditions.

Significance: Worked penetration is used to classify the consistency of lubricating greases according to the National Lubricating Grease Institute (NLGI) consistency grades. The change in penetration after prolonged working is regarded as a measure of the shear stability of the grease under the test conditions.

Procedure: A representative grease sample (minimum 400 g) was conditioned to 25 ± 0.5 °C and transferred into the grease worker cup. The cup was tapped sharply on a firm surface to remove entrapped air. It was then filled slightly above the rim and levelled with a spatula held at 45° to the rim surface. The

vent cock was closed and the sample was subjected to 60 full double strokes of the plunger, completed in 60 ± 5 s as timed with a stopwatch, after which the plunger was returned to its uppermost position. The vent cock was then opened and the cover and plunger assembly removed. All grease adhering to the plunger was scraped off with a spatula and returned to the cup.

The worked grease was transferred to a clean penetrometer cup, slightly overfilled, and levelled by drawing the spatula blade, inclined at 45° toward the direction of motion, across the rim. Cone penetration was determined immediately without delay in accordance with ASTM D217, and the results expressed in dmm.

Penetration Measurement

The prepared test specimen was placed on the penetrometer table with the smoothed surface facing upward. The cone assembly was set to the zero position and adjusted so that the tip of the cone just contacted the centre of the grease surface. The cone shaft was then released rapidly and allowed to penetrate freely for 5.0 ± 0.1 s. The indicator shaft was gently lowered until it touched the cone shaft, and the penetration depth was read in tenths of a millimetre (dmm). The determination was performed in triplicate at different points on the surface, and the mean value was recorded as the worked penetration.

Dropping Point of Lubricating Grease (ASTM D566, IP 132)

The ASTM/IP dropping point is defined as the temperature at which a conventional soap-thickened grease passes from a semi-solid to a liquid state under the specified test conditions, or the temperature at which non-soap-thickened greases undergo rapid oil separation. Significance and Use: The dropping point method is useful for identifying grease type and for establishing and maintaining quality control standards. It indicates the upper temperature limit for grease application.

Procedure: The apparatus consists of a chromium-plated brass cup of specified dimensions, a heat-resistant glass test tube with three indentations equally spaced around its circumference, a thermometer (range $5-300^\circ\text{C}$), and a controlled heating oil bath with flash point above the maximum test temperature. The grease sample was placed in the cup and a conical space formed using a rod inserted through the orifice. The thermometer was positioned in this space without contacting the grease. The assembly was heated at the prescribed rate, and the temperature at which the first drop of oil fell through the cup orifice to the bottom of the test tube was recorded as the dropping point. The test was performed in duplicate and the average value reported

Acidity and Alkalinity of Grease

This method serves as a process control tool during grease manufacture and may be included in grease specifications. Results may be influenced by the presence of additives. Free acidity is expressed as percentage by weight of oleic acid, while free alkali is expressed as percentage by weight of the hydroxide of the predominant metal (NaOH).

Procedure 1: Free Acid Test (IP 37)

10.0 g of grease sample was weighed into a 500 mL conical flask and spread over the bottom to aid dispersion. 150 mL of petroleum spirit was added and shaken until complete dispersion was achieved. 50 mL of neutralized ethanol was added and the mixture shaken vigorously at intervals for 10 min. A few drops of phenolphthalein indicator were added and the solution titrated with standardized potassium hydroxide solution, shaking well after each addition, until a pink colour persisted for 1 min. The burette reading was recorded.

Procedure 2: Free Alkali Test

To the mixture from Procedure 1, 50 mL of hydrochloric acid was added to completely decompose the grease and neutralize the alkali. The mixture was boiled under reflux on a hot plate for 10 min to expel carbon dioxide. The condenser was washed down with 25 mL of neutral ethanol, and the hot solution titrated with standardized potassium hydroxide solution. Blank Test: The procedure was repeated without grease sample using the same volume of hydrochloric acid, and the titre recorded.

Calculations:

Free acid (% as oleic acid):

$$\text{Free Acids} = \frac{(28.2 \times V \times N)}{W}$$

$$\text{Free alkali} = \frac{E \times N(V_1 - V)}{10 \times w}$$

where V = volume of KOH used for sample (mL), V₁ = volume of KOH used for blank (mL), N = normality of KOH, W = weight of sample (g), E = equivalent weight of NaOH (40.0).

Water Content (Dean and Stark Method, IP 74 / ASTM D95)

Procedure: The apparatus comprises a glass or metal still, heater, reflux condenser, and graduated glass trap, connected with leak-proof joints.

The grease sample was heated under reflux with xylene, a water-immiscible solvent that co-distills with water present in the sample. Condensed solvent and water were continuously separated in the trap, with water collecting in the graduated section and solvent returning to the still. The volume of water collected was read from the graduations. The difference between the initial and final water levels gave the water content of the grease, which was found to be negligible, indicating effective dehydration during saponification.

3. Results and Discussion

Effect of Thickener Concentration on Grease Consistency

Six sodium-based grease formulations (F1–F6) were prepared by varying sodium soap content from 100 to 600 g at constant polypropylene (20 g) and base oil 900N (200 g) per batch (Table 3.1).

Table 3.1: Formulation matrix and physical consistency at 25 °C

Formulation	Sodium-soap(g)	Polypropylene (g)	Base oil 900N (g)	Thumb impression
F1	100	20	200	Semi-fluid
F2	200	20	200	Soft-medium
F3	300	20	200	Soft
F4	400	20	200	Medium-soft, adhesive
F5	500	20	200	Medium-hard, adhesive
F6	600	20	200	Medium, adhesive

Consistency increased progressively from semi-fluid to medium-hard with increasing soap concentration, confirming that the soap fibre network density governs grease structure. Formulations F4–F6 exhibited desirable adhesiveness, attributed to the polypropylene complex which reinforces the thickener matrix.

Physicochemical and Performance Properties

The quality control results are presented in Table 3.2.

Table 3.2: Physicochemical properties of formulations F1–F6

Parameter	F1	F2	F3	F4	F5	F6
Worked penetration (dmm, ASTM D217)	295	290	285	270	267	265
Dropping point (°C, ASTM D2265)	162	167	170	185	190	195
Oil separation (mg)	150	155	162	170	182	185
Water washout (% , ASTM D1264)	35	25	22	20	20	15
Free alkalinity (% as NaOH)	0.04	0.045	0.03	0.034	0.05	0.04
Free acidity (% as oleic acid)	0.70	0.75	0.80	0.83	0.85	0.85
Adhesive property	Good	Good	Good	Fairly good	Very good	Excellent

Fig. 3.1: Effect of Soap Content on Worked Penetration

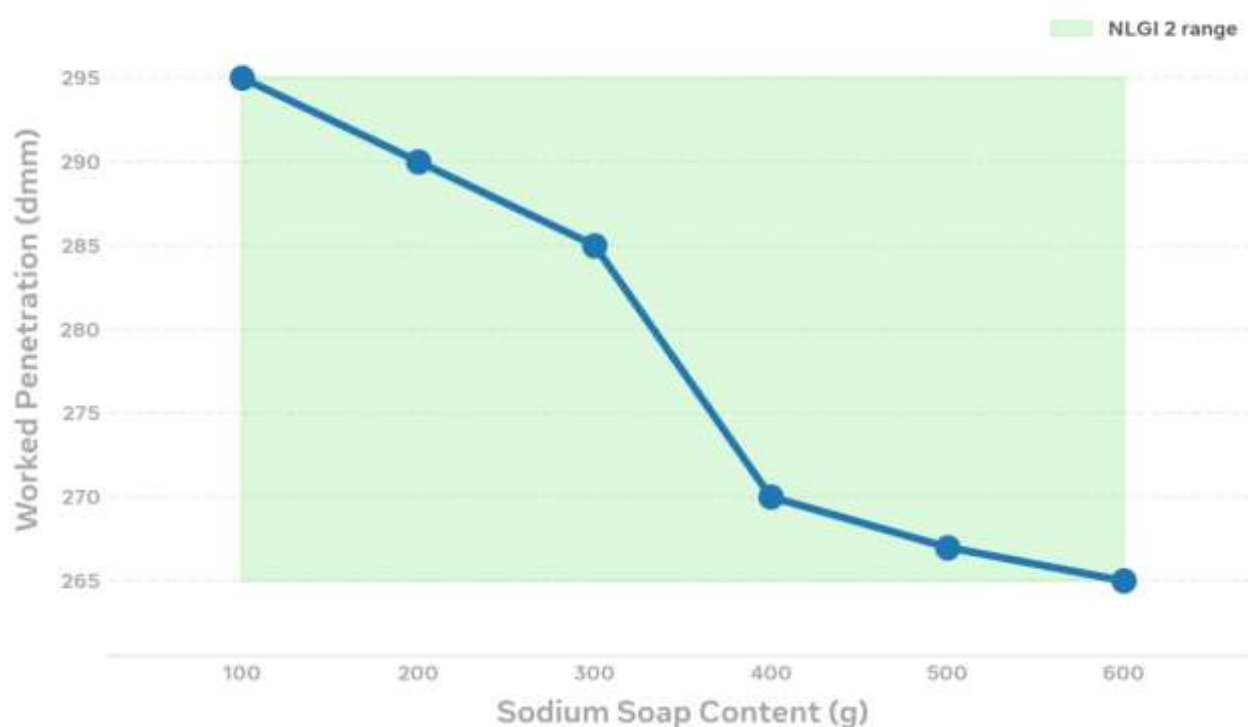


Fig. 3.1: Effect of soap content on worked penetration (mean $\pm$ SD, $n=3$). Green band indicates NLGI Grade 2 range.

Effect on consistency

As shown in Fig. 3.1, worked penetration decreased sharply from 295 dmm to 265 dmm with increased soap content. All formulations lie within the green-shaded NLGI Grade 2 region (265–295 dmm), confirming suitability as multipurpose grease.

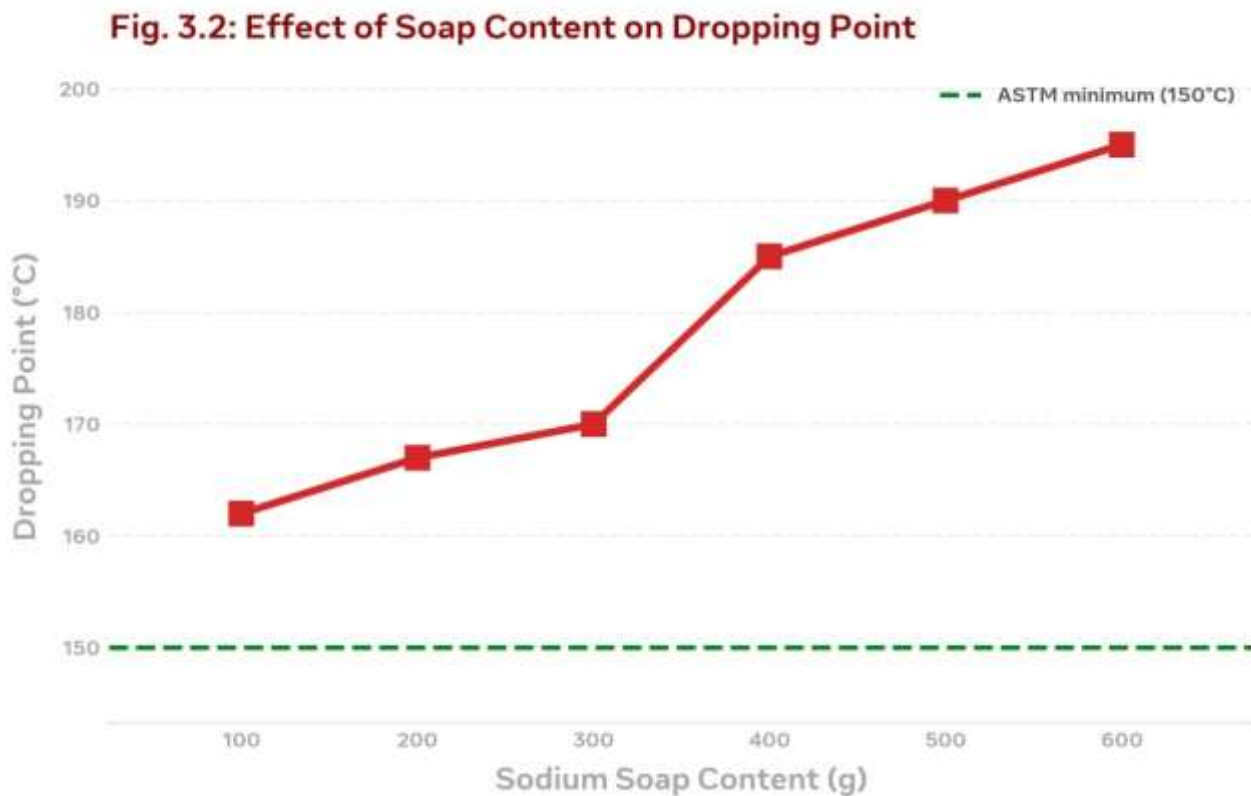


Fig. 3.2: Effect of soap content on dropping point (mean $\pm$ SD, $n=3$). Dashed line shows ASTM minimum.

Effect on thermal stability

Fig. 3.2 shows dropping point increased from 162°C to 195°C. All values are well above the ASTM minimum of 150°C (green dashed line), indicating excellent high-temperature performance due to denser soap network and polypropylene reinforcement.

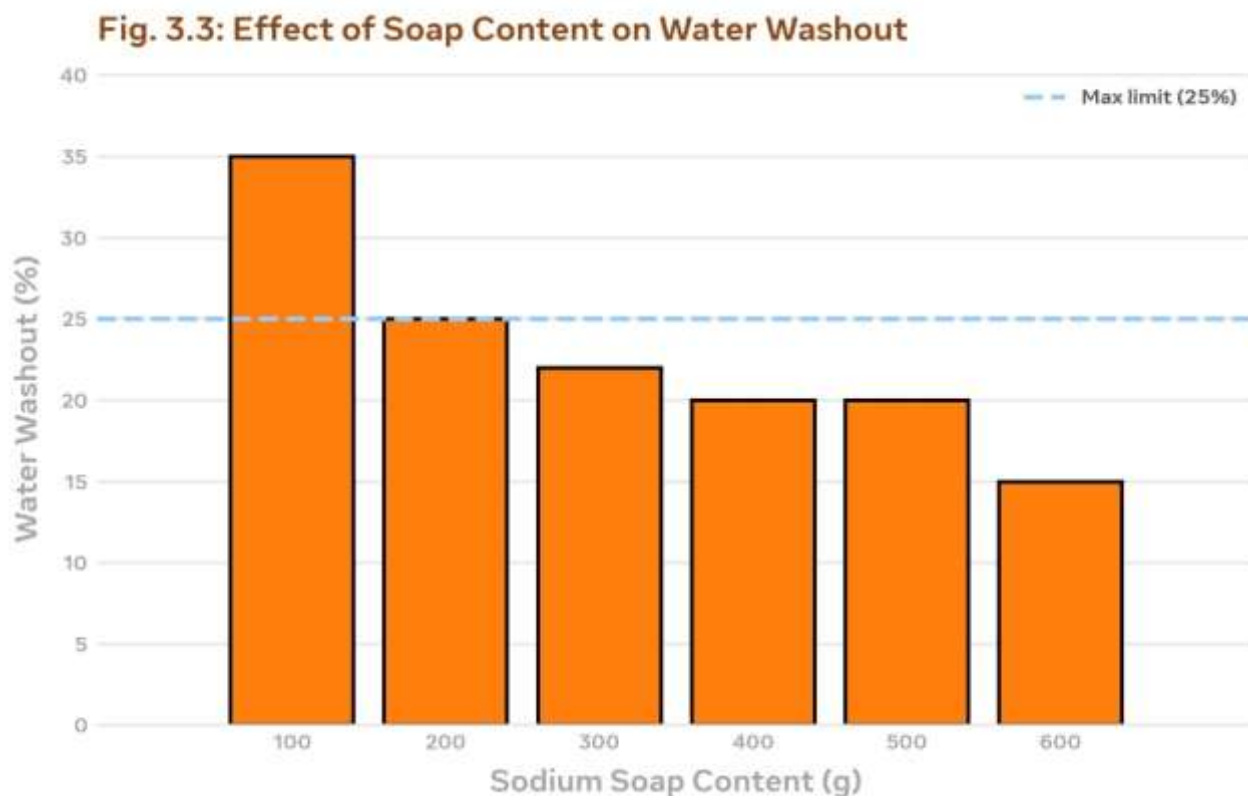


Fig. 3.3: Effect of soap content on water washout (mean $\pm$ SD, $n=3$). Dashed line shows maximum limit.

Effect on water resistance

As illustrated in Fig. 3.3, water washout decreased from 35% to 15%. Formulations F2-F6 are below the 25% maximum limit (blue dashed line), proving that polypropylene significantly improves the inherent poor water resistance of sodium grease

Discussion

- Consistency and NLGI grade: Worked penetration decreased from 295 to 265 dmm as soap increased, placing all formulations within NLGI Grade 2 (265–295 dmm). NLGI 2 is the standard for multipurpose greases used in automotive wheel bearings, chassis, and industrial bearings due to its balance of pumpability and sealing. The inverse relationship between thickener content and penetration is well documented and reflects increased fibre entanglement.
- Thermal stability: Dropping point increased from 162 °C to 195 °C with thickener content. All values exceed 150 °C, the minimum for sodium greases, indicating suitability for medium-high temperature service. The enhancement is attributed to both higher soap network density and polypropylene reinforcement, which delays structural collapse.
- Water resistance: Water washout decreased markedly from 35% (F1) to 15% (F6). Sodium greases are traditionally poor in water resistance due to sodium soap solubility, often showing >40% washout. The incorporation of hydrophobic polypropylene reduced washout to an average of 22.8%, with F4–F6 meeting the <25% threshold for water-resistant applications. This represents a significant improvement over unmodified sodium grease.
- Chemical stability: Free alkalinity (0.03–0.05%) and free acidity (0.70–0.85%) remained within ASTM limits (<0.1% and <1.0% respectively), indicating complete saponification and low corrosivity risk. Proper neutralization is critical to prevent bearing corrosion and oxidative degradation.

- e. Optimum formulation: F4 (400 g soap) with 270 dmm penetration, 185 °C dropping point, 20% water washout, and good adhesiveness offers the best compromise between consistency, thermal stability, water resistance, and pumpability. It is therefore recommended as a multipurpose NLGI 2 grease from non-edible soybean oil, providing a sustainable alternative to conventional mineral-based thickeners.

Summary

This work successfully developed a sodium-based lubricating grease from non-edible soybean oil. Systematic variation of thickener concentration showed predictable structure-property relationships: increased soap content enhanced consistency, thermal stability, and water resistance. Incorporation of polypropylene overcame the traditional water sensitivity of sodium greases, reducing washout to 15-25%.

4. Conclusion

1. All six formulations meet NLGI Grade 2 specifications suitable for multipurpose automotive and industrial applications.
2. Thermal stability (162-195°C) exceeds standard requirements for sodium greases.
3. Polypropylene addition significantly improves water resistance and adhesiveness.
4. Formulation F4 is identified as optimum for commercial scale-up
5. Non-edible soybean oil presents a sustainable, non-food-competing alternative to conventional base oils.

Recommendations

1. Scale-up trial: Pilot-scale production of F4 should be conducted to validate process reproducibility and economic viability.
2. Tribological testing: Further evaluation of four-ball wear, extreme pressure, and oxidation stability (ASTM D942) is recommended.
3. Long-term storage stability: Oil separation and shelf-life studies over 6-12 months should be performed.
4. Comparative LCA: A life cycle assessment comparing this bio-based grease with conventional lithium grease is advised to quantify environmental benefits.
5. Field testing: In-service performance trials in automotive bearings and agricultural machinery under humid conditions are recommended

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