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Production and Performance Evaluation of Biodegradable Grease from Palm Kernel Oil

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Abstract

This study presents the production and performance evaluation of biodegradable grease derived from palm kernel oil (PKO), utilizing calcium hydroxide as the thickener and molybdenum disulfide as a performance-enhancing additive. The grease was synthesized via a four-stage process involving saponification, soap dilution, recrystallization, and homogenization. Two grease samples were prepared and compared to conventional mineral-based grease (Oando) as a control. Characterization tests included penetration (ASTM D217), drop point (ASTM D2265), and biodegradability (ASTM D974-04). Results show that the biodegradable greases demonstrated superior thermal stability, with drop points of 320°C and 312°C, compared to 260°C for the conventional grease. Penetration tests yielded values of 231 and 266 (0.1 mm) for Set A and Set B, respectively, placing them within NLGI grade 2, suitable for general-purpose applications. The biodegradability grade based on acid number titration indicated significantly higher environmental performance for Set A (up to 4.21) compared to Set B (2.94) and conventional grease (1.02). These findings affirm the feasibility of producing high-performance, environmentally friendly greases from renewable feedstock such as palm kernel oil.

Keywords: *Biodegradable grease, palm kernel oil, calcium-based grease, drop point, penetration, acid number, ASTM D974, NLGI, molybdenum disulfide.*

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1. Introduction

Lubricating greases are commonly used in both automotive and industrial applications and are essential for reducing wear and friction between the moving parts in relative motion. Consequently, due to the potential toxicity and non-biodegradability, mineral-based greases pose environmental challenges despite their prevalent use. This has required investigating other alternatives that are biodegradable and renewable, that provide equal performance while being eco-friendly.

African oil palm (*Elaesis guineenssis*) fruit contains two types of oils, these include: palm kernel oil (PKO) which is extracted from the kernel, and palm oil, which is extracted from the mesocarp (Aadb *et al.,* 2014; Abd Rashida *et al.,* 2016; Melvia *et al.,* 2022). The main fatty acid composition of crude palm kernel oil is lauric acid. Oleic, palmitic and linoleic acids are major fatty acids present in crude palm oil (Philipp *et al.,* 2021). This shows that the two oils can be used for different industrial applications due to the difference in composition of fatty acids. Crude palm oil is suitable for consumption due to its high saturated fat content (Denis *et al.,* 2021). Tocotrienols, carotenoids and tocopherols are present in a significant amount (Niamketchi *et al.,* 2021). It serves as a richest source of provitamin A (Melvia *et al.,* 2022). Crude palm kernel oil is often used for non-edible applications. It is a less dense product (Denis *et al.,* 2021). It is used in the cosmetics, pharmaceutical, and food processing industries, traditional medicine, biolubricant or biodiesel (Fabienne *et al.,* 2021). Kernel extract acted as an efficient corrosion inhibitor of mild steel in HCl (Afia *et al.,* 2011 and 2013)

Calcium based soaps are commonly used as thickeners in grease production. Their outstanding mechanical stability, water resistance and ease of manufacture are derived particularly from calcium hydroxide. Calcium thickener is formed through the saponification reaction of calcium hydroxide with fatty acid present in the vegetable oil. The calcium soap produced imparts the semi-solid consistency of the grease, and also serves as the fibrous matrix that holds the base oil.

Apart from having base oil that is biodegradable, additive and thickeners should also be eco-friendly; (Rahman et al., 2023; Willrich & Biehl, 2014). Calcium grease is preferred option for railway lubricant aside from aluminium and lithium greases due to its environmentally friendly properties (Razak *et al.,* 2024). Beside availability in large quantities, low cost and easy handling, calcium grease also has good shear stability, water and corrosion resistance (Sterpu *et al.,* 2016). Calcium grease, under the CEC-L-33-T-82 test standard, shows high level degradation rate (Razak *et al.,* 2024). Calcium greases are mostly valued for their high performance under reasonable loads, temperatures, and their rust-inhibiting properties. Furthermore, calcium soap is friendly with biodegradable base oils, further improving the ecological profile of the final product.

Performance additives are widely added to improve the performance of biodegradable lubricants. Molybdenum disulphide (M_oS_2) is among the effective additives used in biodegradable greases. M_oS_2 is versatile solid lubricant that reduces friction in dry environments when in thin layers. It is often added to oils, greases, or materials to boost lubrication. It can also be applied via spray coating, laser ablation, or chemical decomposition, making it versatile option for extreme conditions (Ilie & Cristescu, 2022). M_oS_2 is commonly used as extreme pressure additive in grease for overrunning rail wheels due to its ability to withstand strong shock loads and vibrations (Fryza & Omasta, 2016; Uddin *et al.,* 2014; Willrich & Biehl, 2014). However, this material is sometimes restricted due to environmental concerns, as it is not biodegradable and contains heavy metals, sulphur, making it pollutant, potentially abrasive and corrosive at high temperatures (Gachechiladze *et al.,* 2018; Gorbacheva *et al.,* 2020; Waynic, 1992; Willrich &Biehl, 2014).

In this study, palm kernel oil is utilized as the base oil, calcium hydroxide as the thickener, and molybdenum disulphide as the performance-enhancing additive to formulate biodegradable grease. The final product is then compared with conventional mineral-based grease (Oando) to evaluate its suitability for practical applications.

2. Materials and methods

2.1 Collection and Preservation of Base Oil

Palm kernel oil (crude), used as based oil for the bio-grease, and was locally sourced from a market in Anyigba, Kogi State, Nigeria. The oil sample was kept in a sealed container away from sunlight and heat. All the chemicals used were high-quality and meant for laboratory analysis.

2.2 Grease Production Process for Two Samples

Two calcium-based biodegradable grease samples, labelled Sample A and Sample B were formulated using palm kernel oil (PKO) as the base oil, calcium hydroxide as the thickener, and molybdenum disulphide (MoS_2) as the performance additive. While both samples followed the same production stages—saponification, soap dilution, recrystallization, and homogenization—they differed in thickener, additive concentrations and temperature to evaluate performance variations under different lubrication conditions.

2.2.1 Sample A

A mixture of calcium hydroxide Ca $(OH)_2$ and stearic acid in a 1.2:1 ratio was combined with palm kernel oil, which was used in an amount three times that of the $Ca(OH)_2$ and stearic acid mixture. This mixture was heated to 90°C in a glass reactor and then the temperature was increased to 140°C, where it was maintained for 2 hours with continuous stirring until soap foams formed (Sharma *et al.,* 2016). Subsequently, 70g of palm kernel oil was heated to 90°C and then mixed with 26.4g of the soap that had been produced. The mixture was heated to 190°C, then lowered to 160°C to allow for the addition of an additive. To develop a stable soap microstructure that resists oil bleeding and enhances thermal resistance, controlled cooling was applied to the final product (Sharma *et al.,* 2016). The resulting mixture was then roll-milled to produce the grease. The mixture was initially heated to 190°C to allow the base oil to become trapped within the soap's fibre network (Awoyale *et al.,* 2011).

2.2.2 Sample B

A mixture of calcium hydroxide ($Ca(OH)_2$) and stearic acid in a 1.5:1 ratio was combined with palm kernel oil, which was used in an amount three times that of the $Ca(OH)_2$ and stearic acid mixture. The mixture was heated to 100°C in the reactor. Subsequently, 75g of base oil was mixed with 22g of the produced soap at temperatures of 100°C and 200°C during the cooking process. The mixture was then cooled to 165°C before adding the additive. Finally, the grease was obtained at room temperature (Awoyale *et al.,* 2011).

2.3 Performance Characterization of the Greases

The experiments for the characterization of the performance of the grease were performed on the produced samples and the conventional grease (Oando) which was used as control. The tests were carried out at the Quaternary International Company Limited, Effurun-Delta State of Nigeria.

2.3.1 Biodegradability Test Procedure (ASTM D974-04)

The biodegradability of grease samples was assessed by monitoring the increase in acid number over a 28-day period using the ASTM D974-04 method. Grease samples were incubated in a biodegradation medium (e.g., compost or activated sludge) under aerobic conditions at 25–28 °C. At regular intervals (e.g., days 0, 7, 14, 21, and 28), samples were extracted and titrated with 0.1 N KOH in ethanol using a mixed ethanol–toluene solvent. The increase in acid number (mg KOH/g) was used as an indirect indicator of microbial degradation, with greater acid formation suggesting higher biodegradability (ASTM, 2004):

$$\text{Acid Number (mg KOH/g)} = \frac{(V \times N \times 56.1)}{W}$$

Where:

- V = Volume of KOH used (mL)
- N = Normality of KOH
- W = Weight of grease sample (g)
- 56.1 = Molecular weight of KOH

2.3.2 Drop Point Test Procedure (ASTM D2265)

The drop point test measures the temperature at which grease changes from a semi-solid to a liquid under standardized conditions. The ASTM D2265 method is used for greases with higher drop points and wide temperature ranges. A clean drop point cup is filled with the test grease, avoiding air pockets or overfilling. The surface is levelled for even heating. The filled cup is placed into the test tube, which is then mounted in a drop point apparatus equipped with a heating bath and a thermocouple or thermometer. A metal shaft with an orifice is positioned below the sample to detect the first drop. The bath temperature is increased at a controlled rate of 8–12°C per minute. The sample is observed continuously during heating. The drop point is recorded as the temperature at which the first drop of melted grease falls from the test cup into the shaft below. The result is recorded in °C and compared with reference values. Replicates may be required for accuracy. This method is applicable for a wide range of greases and is particularly useful for products with drop points above 200°C, including calcium-based and lithium-complex greases (ASTM D2265-22, 2022).

2.3.3 Procedure for Measuring Grease Penetration (ASTM D217)

The penetration test was conducted in accordance with the ASTM D217 standard to determine the consistency of the grease samples. This method measures the depth to which a standard cone penetrates the grease surface under specified conditions, providing a quantitative assessment of the grease's hardness or softness (ASTM, 2022). Grease samples were conditioned at 25 ± 0.5 °C for at least 2 hours prior to testing to ensure uniform thermal equilibrium, as recommended by ASTM D217. A penetrometer equipped with a standard cone assembly (total weight: 150 g) was calibrated. The grease was packed into a standard sample cup to avoid air entrapment and ensure a smooth, level surface.

Unworked Penetration Test

For unworked penetration: The sample cup was placed on the penetrometer base. The cone was carefully positioned just above the grease surface. Upon release, the cone was allowed to penetrate freely into the grease for 5.0 seconds. The depth of penetration was recorded in tenths of a millimetre (0.1 mm units) as the unworked penetration value.

Worked Penetration Test

The grease was placed into a standard grease worker and subjected to 60 double strokes. The worked sample was then repacked into the sample cup and tested using the same procedure as above. The resulting measurement was recorded as the worked penetration. The worked penetration was done to simulate mechanical shearing during actual application.

3. Results and discussion

3.1 Grease Preparation Data

Table 1 shows a summary of weights, percentage compositions and reacting temperatures of PKO, calcium hydroxide and stearic acid used in soap production for the grease samples. **Table 2** provides a summary of weights, percentage compositions and reacting temperatures of base oil, thickener and additive used in production of the grease samples.

Table 1: Variation of compositions and temperatures of the produced soap

Soap Composition	Sample A	Sample B
Mass of PKO (g)	26.4	30.0
Mass of calcium hydroxide [$Ca(OH)_2$] (g)	4.8	6.0
Mass of Stearic Acid (g)	4.0	4.0
Room Temperature (^0C)	32.0	32.0
Reacting Temperature (^0C)	78.0	70.0
Maximum Temperature (^0C)	140.0	140.0

Table 2: Variation of compositions and temperatures of the produced grease

Grease Composition	Sample A	Sample B
Mass of Base Oil (PKO) (g)	70.0	75.0
Mass of Thickener (Soap) (g)	26.4	22.0
Mass of Additive (MoS_2) (g)	3.6	3.0
Mixing Temperature (^0C)	90.0	100.0
Cooking Temperature (^0C)	190	200
Temperature of Additive (^0C)	160	165
Room Temperature (^0C)	28.0	28.0

3.2 Biodegradability Test Results

The biodegradability of grease samples was assessed by monitoring the increase in acid number over 28-day period using the ASTM D974-04 method, incorporating both biodegradable samples and control sample. From Table 3 and Figure 1, Biodegradability progression was observed. Sample A shows higher biodegradability, meeting the threshold for readily biodegradable classification, aligning with OECD301B and European Union (EU) eco-label criteria standards (OECD, 1992; ASTM, 2004). Sample B displayed moderate biodegradable grade. Standard grease has the list grade, stressing the poor environmental performance of petroleum-based greases.

Table 3: Biodegradability Grades (ASTM D974-04)

Time (Days)	Acid Number (mg KOH/g)	Biodegradable Grade (Set A)	Biodegradable Grade (Set B)	Conventional Grade (Oando)
0	0	1.14	3.05	0.52
5	72	2.56	3.18	0.68
10	96	2.86	3.22	0.74
15	120	2.81	4.02	0.83
20	144	2.83	4.18	0.91
28	216	2.94	4.21	1.02

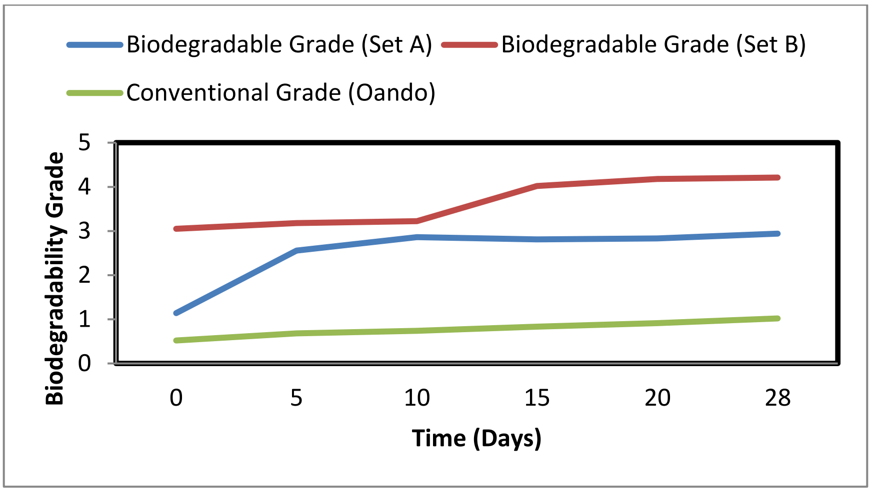

Figure 1: Biodegradability Progression over Time *Line graph comparing biodegradable grades of Sample A, Sample B, and Oando over 28 days.*

3.3 Drop Point Results of the grease samples

Table 4 below shows the summery of the determined drop point in degree Celsius of the two grease samples and the control sample.From Table 4 and Figure 2, grease sample A and B outperformed Oando. Higher drop point values of the sample greases suggest superior thermal stability, likely due to molybdenum disulphide (additive) and calcium-based soap used, which improve thermal and oxidative resistance (Sharma *et al.,* 2018). The results obtained shows that bio greases produced are suitable for high-temperature applications such as in automotive or industrial machinery lubrication systems.

Table 4: Drop Point Results (ASTM D2265)

S/N	Test Samples	Dropping point (^{0}C)
1	Sample A	320
2	Sample B	312
3	Control (Oando)	260

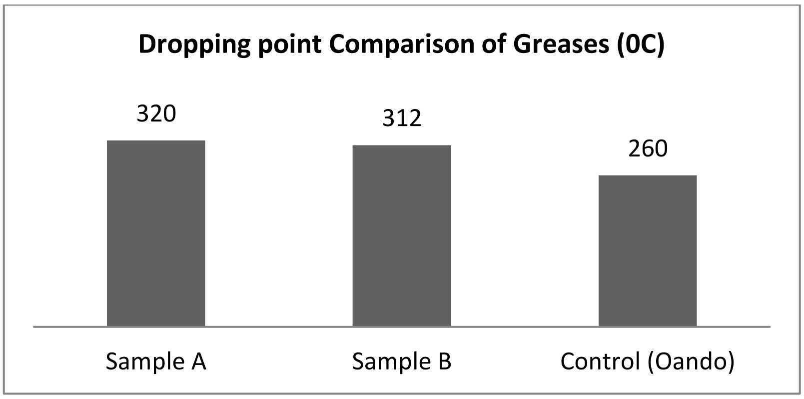

Figure 2: Drop Point Comparison of Greases *Bar chart showing drop point values of Sample A, Sample B, and Oando.*

3.4 Consistency Test (Un-Worked and Worked Penetration)

After the test was carried out, worked and un-worked penetration values of the two grease samples along with the control sample were measured and recorded below.

Table 5: Consistency/Penetration **Test Results (ASTM D217)**

Grease Type	Worked Penetration (0.1 mm)	Un-worked Penetration (0.1 mm)
Sample A	231	223
Sample B	266	252
Control (Oando)	287	275

Table 6: NLGI Grade

NLGI Grade	Worked Penetration (0.1 mm)	Consistency Description	Typical Application
000	445 – 475	Fluid	Centralized lubrication systems
00	400 – 430	Very soft	Gearboxes, enclosed bearings
0	355 – 385	Soft	Cold climates, centralized systems
1	310 – 340	Semi-soft	Chassis components, general machinery
2	265 – 295	Normal or general-purpose grease	Automotive bearings, industrial use
3	220 – 250	Semi-hard	High-temp or vertical shaft bearings
4	175 – 205	Hard	Special applications, sealing greases
5	130 – 160	Very hard	Rare; sealing or high-temp grease
6	85 – 115	Block	Extremely rare; solidified applications

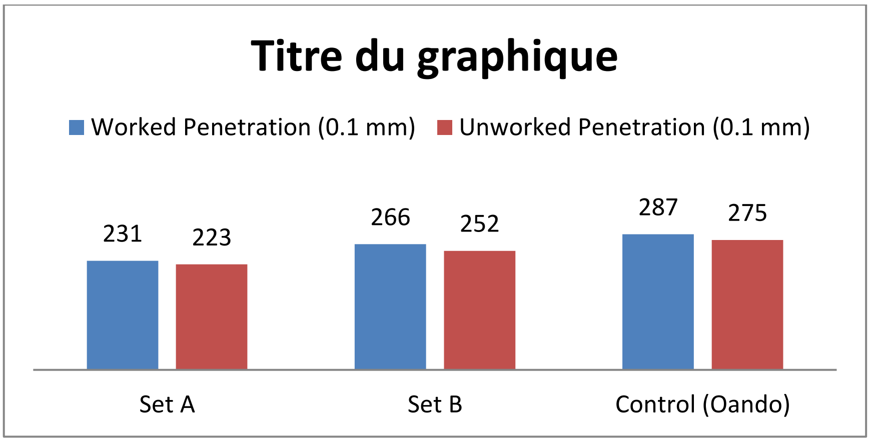

Figure 3: Penetration Comparison of Greases *Bar chart of worked and unworked penetration for Smple A, Sample B, and Oando.*

From Table 5 and Figure 3, the grease samples produced fall within the NLGI grade 2 thereby classified as general purpose greases. Grease sample B is softer than A due to it slightly higher worked penetration. Oando (control) grease showed the highest penetration falls within NLGI grade 1, classified as soft grease which can be used for centralized systems. Sample A has better mechanical stability under shear conditions compared to simple B and control, due to the slight difference between its worked and unworked penetration values. Sample A suggest greater mechanical integrity under shears due it stronger texture, while sample B may be used in dynamic lubricant conditions due to its improved flowabilty.

A holistic evaluation of thermal, mechanical, and environmental performance reveals that Sample A is superior in all three dimensions. Its high drop point and moderate penetration make it an ideal candidate for high-load, high-temperature, and environmentally sensitive applications. Sample B, though slightly less thermally stable and biodegradable, maintains adequate consistency and remains a viable bio-based alternative. The conventional grease, while mechanically workable, underperformed in thermal resistance and biodegradability.

Conclusion

This research validates the environmental and technical capability of producing calcium-based bio grease from palm kernel oil. Adhering to standardize testing procedure (ASTM D974-04, ASTM D2265, ASTM D217), and by mixing with molybdenum disulfide and calcium hydroxide, the produced greases not only matched but exceeded mineral-based products in key performance metrics. The adoption of these formulations in industrial practice could significantly reduce ecological impact while maintaining operational performance.

Conflict of Interest

The author declares that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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