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Formulation and Tribological Property Analyses of Biodegradable Grease Produced from Used Groundnut Oil, Cellulose, and Bentonite

Kigho M. Oghenejoboh*[1], Judith E. Umukoro[2], Nneka R. Agbale[3], Chidimma Aniodo[4]

Department of Chemical Engineering, Delta State University, Abraka, Nigeria

Corresponding author: *kmoghenejoboh@delsu.edu.ng (K.M Oghenejoboh)

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Abstract

Bio-grease was produced from waste cooking oil (groundnut oil) sourced from eateries and local restaurants. The physicochemical properties of the base oil and produced grease (viscosity, density, specific gravity, free fatty acid, as well as the consistency—worked and unworked penetration) were determined using standard ASTM methods. The SEM/EDX characterization of the grease was done using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX), gas chromatography–mass spectrometry (GC–MS), and Fourier transform infrared spectroscopy (FTIR). The values of the physicochemical properties were within optimal ranges recommended for grease by the National Lubricating Grease Institute (NLGI). The trace elemental compositions of the grease showed no significant environmental concerns. The FTIR results show a good combination of functional groups commonly found in natural products, polymers, and various organic compounds. The SEM/EDX results indicate that the formulated grease is similar to polyurea-based greases, which are known for their high-temperature stability, excellent mechanical performance, and resistance to water washout. The produced grease, which falls within NLGI grade 2, can be used for general purposes.

Keywords: Biodegradable; grease; bentonite; cellulose; used groundnut oil; tribological properties

1. Introduction

Grease is one of the most important lubricants for lowering wear and friction in mechanical systems. However, because of their hazardous effects, durability, and capacity to contaminate ecosystems, conventional petroleum-based greases have a negative impact on the environment. There is, therefore, the need to source biodegradable grease that is environmentally friendly (Ijaz Malik et al., 2023; Xu et al., 2019). Biodegradable greases can be manufactured from renewable resources like decayed vegetables, animal fats, and other bio-based materials which decompose more readily in natural environments (Alfoso et al., 2023; Vafeal et al., 2022).

Biodegradable lubricants, such as grease derived from used cooking oil (UCO), are being developed and characterized in response to growing environmental concerns and the need for sustainable alternatives to traditional petroleum-based products (Pichler et al., 2023; Panchal et al., 2015). Since groundnut oil is readily available, renewable, and has a particular chemical makeup, it has been recognized as a possible raw material for the production of biodegradable grease, however, the benefit of raw cooking oil in the food chain limits its application (Li and Wang, 2015; Akhtar et al., 2014). Hence, preference is given to used cooking oil (UCO), which has no economic value. However, the used oil requires pretreatment before being used as base oil for the formulation of bio-grease because of its high free fatty acid (FFA) and water content (Joshi et al., 2023; Cárdenas et al., 2021). Despite the limitations, the availability, chemical characteristics, and simplicity of modification of UCO make it a great choice.

It has been noted that groundnut oil, which is frequently used in cooking, has a special fatty acid composition that is similar to that of rice bran oil, which can be chemically modified to improve its lubricating capabilities (Pransath et al., 2024). In their tribological study, Adhvaryu et al. (2004) observed that thermally and chemically modified vegetable oils are highly suitable for the formulation of biodegradable greases and lubricants as a result of their better environmental profile, making them appropriate for applications that are sensitive to environmental factors. They noted that despite their good tribological properties, most vegetable oils exhibit poor thermal stability and oxidative properties, hence the need to modify them chemically. Hehete et al. (2024) carried out a review on the production of biodegradable grease from vegetable oils, using Neem seed oil as a case study. The result of their review showed that grease produced from vegetable oil exhibits better qualities when compared to fossil fuel grease. Such greases, according to them, are highly biodegradable, thermally stable, and highly resistant to oxidation and water.

According to Săpunaru et al. (2024), classical lubricating greases are produced from mineral oil, thickening soap, and possibly solid additives as well as dispersants that exhibit anti-wear characteristics (Ahmad et al., 2022). Usually, the thickening soap is added to the base oil or prepared in situ from a saturated or unsaturated fatty acid reacting with a lithium, calcium, or sodium base. The preparation from vegetable oils is generally made in the same manner by replacing the mineral oil with the vegetable one. Besides the raw materials, the preparation mode decisively influences the quality of the obtained grease. A traditional mode for the preparation of grease with sodium soap was described by Bashari et al. (2024). In their study, the vegetable oil was mixed with different proportions of sodium soap thickener generated via saponification of sodium hydroxide and stearic acid. The results of their study show acceptable values of the dropping point and NLGI, as well as oil bleeding and consistency values. They, therefore, concluded that the produced grease can be used for a wide range of applications.

According to Syahir et al. (2017), the consistency of lubricating grease is often considered to be its most important rheological property. It generally dictates the suitability of the grease for a particular application. Consistency broadly refers to the firmness of grease or the grease's ability to remain in place (resist leakage) to form stable channels of lubricant in bearings. These channels are important to the functionality of a bearing because they serve as reservoirs from which moving parts draw lubricating fluid over the operational lifetime of a machine. Hence, consistency serves as an important metric for selecting grease for a particular application.

The main test to measure grease consistency is the cone penetration test given by ASTM D217 in this test, a cone is dropped into a grease sample for 5–10 seconds and the depth to which the cone penetrates is used as a measure of consistency. This test requires a large sample of grease—approximately 450 grams according to ASTM D217 (2017). However, the application of specialized equipment such as a rheometer that requires a very small sample—less than two grams—and provides quantitative results has made the assessment of the rheological properties of grease very easy. In addition to grease consistency, other rheological characteristics determined include viscosity and density, which are a function of both the base oil and thickening additives (Sankarannair et al., 2024).

Formulation of grease generally involves a mixture of base oil, thickener, and additives in the right proportion (Hairunnaja et al., 2025; Sofi et al., 2019). According to Japar et al. (2019), the optimum proportions should be 80–95% base oil, 2–15% thickener and 0–10% additives. Lithium hydroxide monohydrate, stearic acid, and calcium salts have been used by some workers as thickeners and additives with bio-based oils for the formulation of grease (Hairunnaja et al., 2023). In their study, Syahir et al. (2017) used a mixture of castor oil, glyceryl stearate, and chitosan for their formulation and reported that the friction coefficient of the grease was reduced when compared to lithium salt-based grease. In a similar study, ethylated cellulose, methylated cellulose, and kraft cellulose were used as thickeners, with the produced grease exhibiting excellent decomposition temperatures in comparison to lithium salt greases (Abdubari et al., 2017). Boiko and Lebedinsky (2015), in their own study, employed a mix of glycerin-based boron in their formulation and the resulting grease exhibited excellent anti-scoring and anti-wear properties.

The application of renewable thickeners such as bentonite when used in conjunction with bio-based oils in the formulation of biodegradable grease is currently receiving great research attention. Some of the reasons for the preference of bentonite over soap-based thickeners are its high reactivity in water, high temperature stability, and mechanical stability as well as its ability to suspend additives within the grease formulation (Azuka et al., 2025; Williams and Roberts, 2021; Vodounon et al., 2020).

In the present study, biodegradable grease is formulated utilizing used groundnut oil, cellulose, and bentonite. Tribological properties of the produced grease were analyzed using standard procedures, while the characterization of its microstructure and elemental composition was determined using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX).

2. Materials and Methods

2.1 Materials Used

2.1.1 Used Cooking Oil

Used groundnut oil (UGO), also known as waste cooking oil, which served as the primary raw material for the formulation of biodegradable grease, was sourced from local food vendors and restaurants in and around Oleh, Isoko South Local Government Area, Delta State.

2.1.2 Thickener and Additives

Analytical-grade organically modified bentonite powder and cellulose fibres, used as thickener and additive, were purchased from Pyrex Chemical Company, Benin City, Nigeria. Since these chemicals were of analytical grade, there was no need for purification or pre-treatment. Other chemicals used included sodium hydroxide, methanol, molybdenum disulfide (MoS_2), polyisobutylene, silicon-based defoamer, and distilled water.

2.1.3 Equipment and Apparatus

The equipment and apparatus used for the study, along with their specific applications, are outlined as follows:

A blender (IKA Ultra-Turrax 25 Digital, Model: T25) was employed for blending the thickener, saponified UCO, cellulose, and other additives to ensure thorough mixing. Beakers and flasks (Pyrex 250 mL Conical Flask, Model: 5000) were used for mixing and processing. A heating mantle (LabTech LHM-500, Model: LHM-500) was utilized for heating and maintaining the temperature during the thickening process. A viscometer (Brookfield DV2T, Model: DV2T) was used to measure the viscosity of the oil and grease. The Falex Timken Test Machine (Timken 002-001-003, ASTM D2509) served as the standard apparatus for measuring the load-carrying capacity of lubricating grease. An oven (Thermo-Scientific Heraeus UT6, Model: UT6) was applied for drying the products and removing residual moisture. Finally, a pH meter (Metrohm 910, Model: 910) was used to check the pH levels of the grease to ensure optimal stability.

2.2 Methodology

2.2.1 Pre-treatment of Used Groundnut Oil

The used groundnut oil (UGO) was filtered using a fine mesh to remove large food particles and impurities. The filtered UGO was then heated at 60 °C for 1 hour to remove residual moisture and free fatty acids. This step was essential for improving the oil's quality prior to its use in the transesterification reaction for grease production.

2.2.2 Preparation and Segregation of the Thickener and Additives

Organically modified bentonite powder, cellulose fibres, and other chemicals and reagents used in the study were of analytical grade and therefore required no further treatment.

2.2.3 Physicochemical Properties of the Oil

The properties of the used groundnut oil determined to ensure its suitability as a base oil for grease formulation included density, viscosity, and free fatty acid (FFA) content.

(a) Density of the Oil

A substance's density (ρ) offers information about the chemical makeup of the material and is a crucial characteristic for describing oils and fats. The weight of a clean, dry empty density bottle was recorded. After, the bottle was filled with a known volume of the oil and weighed. The density was then determined using Equation 1.

$$Density\ (g/cm^3) = \frac{Mass\ of\ empty\ bottle\ and\ oil\ (g) - mass\ of\ empty\ bottle\ (g)}{Volume\ of\ oil\ (cm^3)} \quad (1)$$

(b) Viscosity of Oil

A fluid's resistance to deformation or flow is measured by its viscosity. A rotational viscometer was used to check the viscosity of the base oil and produced grease

(c) Free Fatty Acid Content of the Oil

Free fatty acid (FFA) content is a crucial quality parameter for edible oils because it influences both flavor and stability. Unlike triglycerides, free fatty acids are not bound to a glycerol backbone. They are formed primarily through hydrolysis—the breakdown of triglycerides by water, often catalyzed by enzymes such as lipases—or through fat oxidation. Elevated FFA levels generally indicate oil degradation or poor storage conditions (Adhvaryu et al., 2005).

In the context of grease formulation, FFA plays a vital role in determining tribological properties. Oils with long-chain fatty acids tend to produce grease with enhanced performance, reducing wear and friction under boundary lubrication conditions (Sankarannair et al., 2024; Pranav et al., 2021).

The titration method was employed to quantify the FFA content of the used groundnut oil (UGO). Approximately 10 g of oil was weighed and dissolved in 25 mL of ethanol. A few drops of phenolphthalein indicator were added; the indicator changes from colorless to pink at the point of fatty acid neutralization. The mixture was titrated with standardized 0.1 N NaOH solution until a faint pink endpoint, persistent for at least 30 seconds, was observed. The FFA content was then calculated using Equation 2, following the method of Vicentini-Polette et al. (2021).

$$FFA\ (\%) = \frac{Volume\ of\ NaOH\ (mL) \times Concentration\ of\ NaOH\ (mol/L) \times 282}{Weight\ of\ sample\ (g)} \quad (2)$$

2.2.4 The Grease Production Process

The first step in the grease formulation process was the transesterification reaction. In this step, 300 g of treated UGO was mixed with 5 g of sodium hydroxide (NaOH) and 50 mL of methanol to produce the methyl ester–based soap of the oil, following the method of Prasanth et al. (2024). The mixture was placed in a water bath maintained at 60 °C and stirred with a magnetic stirrer at 100 rpm for 1 hour. It was then transferred into a separating funnel and left to cool for approximately 6 hours.

After cooling, two layers were obtained: the methyl ester of UGO, which formed the upper layer, and the triglycerides, which settled as the bottom layer. The upper layer (methyl ester) was carefully collected, thoroughly washed with warm distilled water to remove impurities, and preserved for the next stage of grease preparation, while the bottom triglyceride layer was discarded.

The second stage involved the mixing of the UGO methyl ester with the thickener and additives to form the grease. Here, 200 g of the UGO ester was blended with 20 g of bentonite powder, 8 g of cellulose fibre, and 5 g each of butylated hydroxytoluene (BHT) and molybdenum disulfide (MoS_2). The mixture was stirred with a magnetic stirrer at 100 rpm and maintained at 100 °C for 2 hours to obtain a homogeneous blend.

Finally, the mixture was transferred into a double-walled induction-heated vessel and further heated at 120 °C for 20 minutes. It was then allowed to stand undisturbed for 24 hours to cool and solidify into the final grease product.

2.2.5 Evaluation of the Grease Tribological Properties

The standard four-ball tester, as outlined in ASTM D2509 guidelines, was used to determine the wear scar diameter, coefficient of friction, and load-carrying capacity of the produced grease. A pre-calibrated Falex Timken test machine was employed to conduct these measurements, following the method of Sabarinath et al. (2019).

2.2.6 Penetration Test for Grease Consistency

This test was conducted to determine the National Lubricating Grease Institute (NLGI) consistency grade of the grease. Greases with higher NLGI numbers are firmer and tend to remain longer at the point of application. The consistency test followed the ASTM D4172 method. The grease sample was worked, conditioned, and maintained at 25 °C. A penetrometer cone was then released and allowed to sink into the grease under its own weight for 10 seconds. The penetration depth, measured in millimeters, was recorded. A greater penetration depth corresponds to a higher penetration index and softer grease.

2.2.7 FTIR analysis

Fourier Transform Infrared Spectroscopy (FTIR) was used to analyze the functional groups present in the used groundnut oil and the formulated grease. FTIR provides specific information on molecular structure and chemical bonding, thereby identifying the organic and inorganic materials involved in the formulation. The principle of FTIR is based on the fact that molecules absorb infrared light at varying degrees depending on their chemical structure. Samples of oil and grease were applied to an attenuated total reflectance (ATR) cell. In the contact zone, the samples were exposed to infrared light, and the corresponding spectra were recorded as wave numbers based on absorption.

2.2.8 Scanning electron microscope (SEM)

The surface morphology of the oil and grease samples was examined using a 52-cm scanning electron microscope (SEM) at magnifications of 100 µm and 10 µm. The analysis was carried out in the Chemical Analysis Laboratory of the Department of Chemical Engineering, Ahmadu Bello University, Zaria. A concentrated electron beam was directed onto the sample, and the interactions between the beam and the sample's atoms produced signals revealing details of surface topography and composition. By combining the position of the scanning beam with the detected signals in a raster pattern, high-resolution surface images were obtained.

Additionally, gas chromatography–mass spectrometry (GC–MS) analysis was conducted using a QP 2010 PLUS series instrument equipped with a VF-5MS fused silica capillary column (80 m length, 0.25 mm diameter, 0.25 µm film thickness, scan range 40–600 m/z). The total run time was 27 minutes. The relative percentage of extract constituents was quantified using peak area normalization.

2.2.9 Energy dispersive x-ray spectroscopy (EDX)

Energy dispersive X-ray spectroscopy (EDX) was employed to determine the composition and concentration of heavy metal ions present on or near the surface of the samples. This technique provides an overall elemental map of the sample and allows for estimation of elemental proportions at specific positions. EDX was used in conjunction with SEM. When a high-energy electron beam (10–20 keV) strikes the sample's surface, it causes the emission of characteristic X-rays, the energies of which depend on the elements present.

3. Results and Discussion

3.1 Rheological and physicochemical Properties

The results of the physicochemical properties of the treated used groundnut oil, measured between 40 -100 °C are presented in Figure 1.

Table1: Rheological and physiochemical properties of Used Oil

Temp (°C)	Mixing Speed (RPM)	Kinematic Viscosity (cSt))	Density (g/cm^3)	Free Fatty Acid (%)
40	100	210	0.872	2.94
50	100	188	0.884	2.96
60	100	155	0.884	2.97
70	100	125	0.872	2.94
80	100	112	0.870	2.97
90	100	60	0.864	2.97
100	100	25	0.872	2.94

As shown in Table 1, the free fatty acid (FFA) content of the treated used groundnut oil ranged between 2.94 and 2.97% irrespective of the experimental temperatures. This agrees with the result of 3.04% obtained by Correia et al. (2014) in their study with fresh sunflower oil. The low free fatty acid content recorded may be attributed to the transesterification of the oil, which converts the FFA to more stable esters by eliminating glycerol molecules that have oxidative properties (Sankarannair et al., 2024). Oil with low free fatty acid content has a low acidity level, making it generally acceptable for many lubrication applications, including engine parts, provided other performance characteristics meet the required standards. As shown in Table 1, the density of the oil ranged between 0.864–0.884 g/cm^3 (864–884 kg/m^3), which falls within the 0.8 and 0.9 g/cm^3 range recommended for typical bio-based lubricating greases (Work & Energy, 2025). The dynamic viscosity of 210 cSt at 40°C indicates that the treated used groundnut oil is highly suitable for the formulation of bio-grease intended for general purpose applications. As expected, the viscosity decreased gradually as the temperature increased. However, it is essential to ensure that the operating conditions align with the characteristics of the grease to maintain optimal performance.

3.2 Tribological Properties of the Produced Grease

The summary of the unworked and worked penetration values of the produced grease is shown in Table 2.

Table 2: Results of the Grease Consistency Test

Grease sample	Penetration (mm)	
	Unworked	Worked
1	295	230
2	295	230
3	293	231
4	294	230
5	295	228

Grease consistency/penetration is the resistance to deformation by an applied force; this depends on the type and amount of thickener used and the viscosity of the base oil. Table 2 shows the values (mm) obtained from the un-worked and worked penetration using the cone penetrometer at a 10-second duration. As shown in the Table, the worked and un-worked penetrations of the produced grease averaged 230 mm and 295 mm respectively. A significant difference between un-worked and worked penetration indicates poor shear stability. The obtained values fall within NLGI grade 2, which is a soft grease. According to Sankarannair et al. (2024), NLGI grade 2 grease is highly suitable for general applications as a result of its ease of pumping and softer consistency.

3.3 Result of FTIR Analysis

The FTIR spectra of the treated used groundnut oil are presented in Figure 1. The peak at 3004–2922 cm^{-1} indicates aromatic C–H stretching vibration. The peak at 2855 cm^{-1} represents symmetric C–H stretching in alkanes, indicating the presence of appreciable content of fatty acid esters (Fayaz et al., 2017). The peak at 2109 cm^{-1} – C≡N stretching vibration, indicates the presence of nitriles, while the 1744 cm^{-1} peak represents C=O stretching vibration typically associated with esters or carboxylic acids. The peak at 1401 cm^{-1} shows the C–H bending vibration in alkanes, while the peak at 1375 cm^{-1} represents C–H bending vibration in methyl groups. The peak at 1237 cm^{-1} corresponds to C–N stretching vibration commonly found in amines or amides. The presence of alcohols or ethers in the sample can be observed in the C–O stretching vibration represented by the 1159 cm^{-1} peak. The absence of an O–H peak indicates that there was no hydrogen bonding in the base oil structure (Dimakopoulou-Papazoglou et al., 2023).

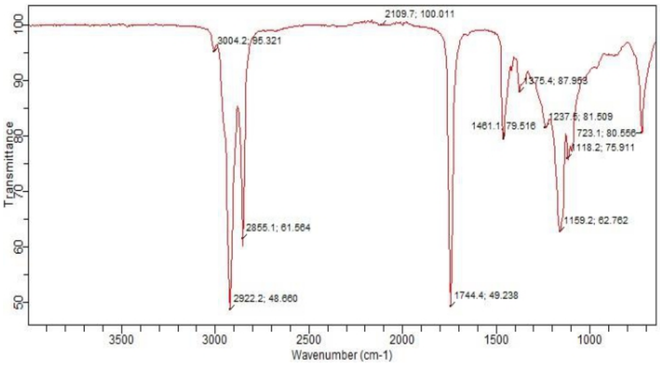

Figure 1: FT-IR spectra of the UGO sample

Figure 2 shows the FTIR spectra of the produced grease. In the Figure, hydroxyl groups indicated by the broad O–H stretching band at 3358 cm^{-1} show the presence of hydrogen bonding in the wax structure. Peaks around 3008 cm^{-1} are attributed to the =C–H alkene groups stretching, while the presence of C=C stretching is noticed at a peak of 1558 cm^{-1}. Aliphatic groups are demonstrated by the C–H stretching and bending vibrations at 2922, 2851, 1461, 1379, 924, 849, 823, and 723 cm^{-1} respectively, confirming α-crystal formation in the grease. Aromatic rings are suggested by the C=C stretching at 1558 cm^{-1} and C–H bending at 924, 849, 823, and 723 cm^{-1}. The presence of ester or ether groups in the formulated grease is indicated by the C–O stretching bands at 1237, 1162, and 1114 cm^{-1} respectively. The C≡C stretching at 2120 and 2001 cm^{-1} is indicative of the presence of alkyne groups.

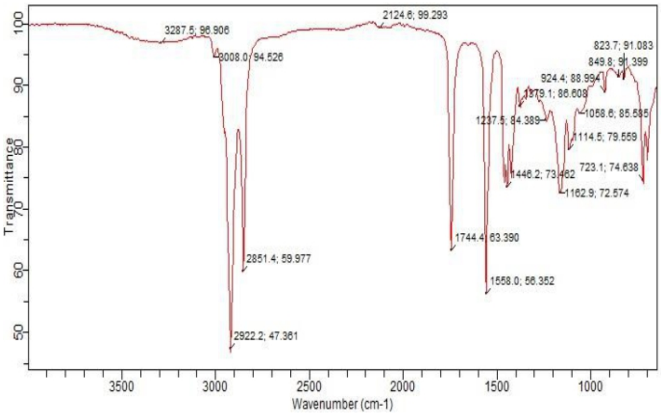

Figure 2: FTIR spectra of the produced grease sample

3.4 Scanning Electron Microscopy and Energy Dispersive X-ray (SEM-EDX) Analysis

SEM–EDX analysis is crucial in grease production for several reasons: it allows for detailed characterization of the grease's microstructure, identification of contaminants and wear debris, and determination of the elemental composition of grease components. This knowledge is vital for optimizing grease formulations, ensuring product quality, and troubleshooting potential issues during grease application (Jiabao et al., 2020).

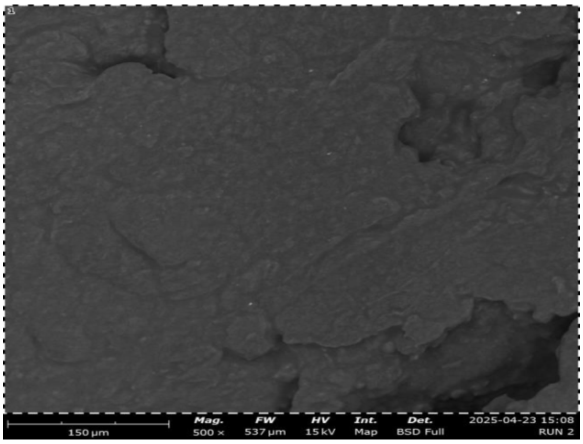

Figure 3: SEM micrograph of the produced grease showing the surface morphology

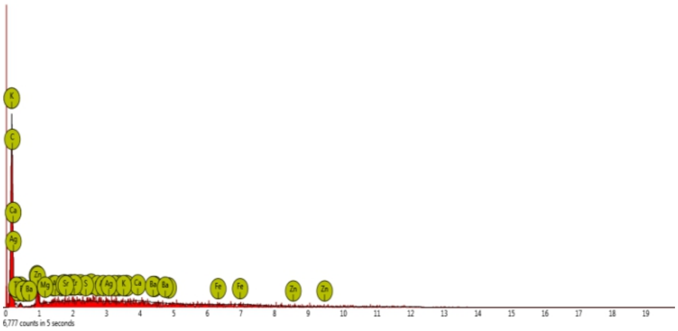

Figure 4: EDX spectrum of the produced grease indicating the elemental composition peaks

Table 3 Elemental composition of the produced grease obtained from EḊX analysis.

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	90.60	85.84
8	O	Oxygen	5.86	7.40
30	Zn	Zinc	0.60	3.10
7	N	Nitrogen	2.67	2.95
17	Cl	Chlorine	0.15	0.43
15	P	Phosphorus	0.11	0.28
14	Si	Silicon	0.00	0.00
13	Al	Aluminium	0.00	0.00
16	S	Sulfur	0.00	0.00
50	Sn	Tin	0.00	0.00
26	Fe	Iron	0.00	0.00
22	Ti	Titanium	0.00	0.00
40	Zr	Zirconium	0.00	0.00
20	Ca	Calcium	0.00	0.00
19	K	Potassium	0.00	0.00
12	Mg	Magnesium	0.00	0.00
56	Ba	Barium	0.00	0.00
38	Sr	Strontium	0.00	0.00
47	Ag	Silver	0.00	0.00

The SEM micrograph (Figure 3) shows the surface morphology of the produced grease, while the EDX spectrum (Figure 4) and corresponding quantitative data (Table 3) confirm the elemental composition. Carbon (85.85%), primarily from the base oil and organic components, constitutes the bulk of the grease. Oxygen (7.40%), likely from the ester-based base oil and oxygen-containing additives, contributes to oxidative stability and low-temperature fluidity. Zinc (3.10%), commonly found in zinc dialkyldithiophosphate (ZDDP) additives, provides anti-wear and antioxidant properties. Nitrogen (2.95%) is indicative of the bentonite thickener, a nitrogen-rich compound that enhances high-temperature stability and mechanical performance. Chlorine (0.43%) may be present due to extreme pressure cellulose additive, though its presence is less common due to potential corrosive effects. Phosphorus (0.28%), also associated with ZDDP additives, contributes to the anti-wear and antioxidant properties of the grease.
This formulation is similar to polyurea-based greases known for their high-temperature stability, excellent mechanical performance, and resistance to water washout. Such properties make them suitable for applications such as electric motor bearings, automotive wheel bearings, and other high-speed, high-temperature environments (Lyadov et al., 2023).

4. Conclusion

The results of this study clearly demonstrate that used cooking oil is a viable option as a base oil for the formulation of biodegradable grease of NLGI grade 2, making it suitable for general lubricating applications. The excellent tribological properties of the produced grease are enhanced by the use of cellulose and bentonite as the thickener and additive. A recommended direction for future research is the optimization of the formulation and the exploration of additional bio-sourced additives to further enhance performance.

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