

SYNTHESIS OF NON-IONIC SURFACTANT FROM NEEM SEED OIL AS A POTENTIAL MATERIAL FOR ENHANCED OIL RECOVERY (EOR)

¹Ibrahim A.A., Dibis, A. A., ¹Muhammad F.A., ¹Adefeso I.B. *, Saidu, H. M., ¹Mukwashi, N.,
Ismail, F. S.

¹Department of Chemical and Petroleum Engineering, Faculty of Engineering, Bayero
University, PMB 1033, Kano State, Nigeria

²Centre for Renewable Energy and Sustainability Transitions, Bayero University, PMB 1033,
Kano State, Nigeria

*Corresponding Author: ibadefeso.cpe@buk.edu.ng/aaibrahim.cpe@buk.edu.ng

TEL:+234 803260719

Received: 18 May 2025; Accepted: 29 June 2025; Published: 30 June 2025

doi: 10.35934/seg.v10i1.132

Highlights:

- The research proved that the synthesis of surfactant from bioresource oil from neem seed oil for EOR.
- The extracted neem seed oil displayed a low acid value (AV) and, moderate saponification value (SV).
- The synthesized non-ionic surfactant demonstrated good surface-active properties.

Abstract: Surfactants are amphiphilic compounds that enable the mixing of immiscible substances like oil and water by reducing interfacial tension. However, their application in Enhanced Oil Recovery (EOR) is limited by issues such as toxicity, poor biodegradability, and dependence on edible oils, which can compromise food security. This study focused on synthesizing a non-ionic surfactant from neem (*Azadirachta indica*) seed oil—a non-edible, biodegradable alternative. Neem seeds were collected from Bayero University Kano, dried for six weeks, and subjected to proximate and Fourier Transform Infrared Spectroscopy (FTIR) analyses. Proximate analysis revealed 28.76% moisture, 14.44% oil/fat, 16.67% protein, and 5.1% ash. FTIR analysis identified key functional groups such as O-H, C-H, and C≡N stretching. Oil extraction was carried out using Soxhlet apparatus with 800 ml of n-hexane at 65°C for 1.2 hours, yielding 23% oil, consistent with literature values. The extracted oil showed an acid value of 1.88 mg KOH/g and a saponification value of 184.8 mg KOH/g, indicating suitability for surfactant synthesis. Further FTIR analysis confirmed the presence of functional groups like C-H, C=O, CH₂, CH₃, C-O, and -(CH₂)_n. A non-ionic surfactant was synthesized by reacting 10 ml of neem oil with 5 g NaOH at 80 °C for 2.5 hours, along with 50 ml of distilled water under continuous stirring. FTIR analysis of the product revealed characteristic

O-H, COO⁻, and C-O functional groups, confirming successful surfactant formation. The findings support neem oil as a viable, sustainable surfactant source for EOR applications.

Keywords: Surfactant, Neem Seed Oil, Oil Recovery, Nonionic, Synthesis

1. Introduction

Crude oil, a naturally occurring petroleum resource composed mainly of hydrocarbons, is formed from ancient organic matter buried under sedimentary rock and subjected to heat and pressure over time (Liberto, 2024; Mohammed *et al.*, 2025). It remains a major global energy source, refined into products like gasoline, diesel, LPG, and petrochemicals. Despite growing interest in renewables, crude oil demand continues to rise, accounting for 31.5% of global energy consumption in 2011 (Sirola, 2014), with daily usage reaching 9.05×10^7 barrels (Yao *et al.*, 2022). However, conventional extraction methods are inefficient. Primary recovery, relying on natural reservoir pressure, retrieves only 20–30% of oil due to capillary and viscous forces (Sani *et al.*, 2024), while secondary recovery, such as water flooding, increases recovery to about 40% (Tunio *et al.*, 2011). Alarming, an estimated 2×10^{12} barrels of conventional oil and 5×10^{12} barrels of heavy oil will remain unrecovered globally (Thomas, 2008). This has led to the growing adoption of enhanced oil recovery (EOR) techniques designed to improve extraction efficiency from mature reservoirs.

To address the limitations of conventional oil recovery, enhanced oil recovery (EOR) techniques have been developed, including thermal recovery, gas injection, microbial injection, and chemical injection. Gas injection, especially with carbon dioxide (CO₂), is one of the most widely applied methods. It functions through miscible and immiscible displacement, where CO₂ either dissolves into crude oil to reduce viscosity or increases reservoir pressure to displace oil. Additionally, CO₂ injection serves as a carbon sequestration method (Nwider *et al.*, 2016; Mohammed *et al.*, 2025). To improve efficiency, CO₂ is often combined with water in the Water Alternating Gas (WAG) process. Furthermore, the use of foam stabilized by surfactants in Foam Assisted WAG (FAWAG) enhances sweep efficiency by reducing gas mobility and improving conformance control (Rossen, 1996; Abdurrahman *et al.*, 2023).

Chemical EOR, particularly surfactant flooding, plays a vital role by reducing interfacial tension (IFT), altering wettability, forming emulsions, and stabilizing foams, thereby mobilizing trapped oil (Farias *et al.*, 2021).

Surfactants are amphiphilic compounds with hydrophilic and hydrophobic parts, classified into anionic, cationic, non-ionic, and zwitterionic types based on the charge of the hydrophilic head

(Massarweh & Abushaikha, 2020). Despite their effectiveness, synthetic surfactants derived from petroleum pose serious environmental concerns. They are non-biodegradable, toxic to aquatic life, and contribute to persistent organic pollutants. Consequently, regions like Europe and the United States are phasing out harmful synthetic surfactants such as alkylphenol ethoxylates, linear alkylbenzene sulfonates, and dialkyl quaternary ammonium compounds (Bronzo *et al.*, 2021).

As a sustainable alternative, research has shifted toward bio-based surfactants. Although surfactants from edible oils like coconut, palm, and soybean have shown potential, they raise food security concerns due to competition with food supplies. Non-edible oils such as neem, jatropha, and castor provide a more sustainable option without affecting food chains. Neem seed oil is particularly attractive due to its abundance, underutilization, and high triglyceride content, making it suitable for surfactant production.

The characteristics of non-ionic surfactants such as temperature, concentration, pH, ionic strength, and pressure are essentials for surfactants, particularly in relation to environmental factor. Non-ionic surfactants, composed of hydrophilic polyoxyethylene chains and hydrophobic alkyl groups, are widely used in industries like detergency, pharmaceuticals, and enhanced oil recovery due to their mildness and biodegradability (Duan *et al.*, 2024).

Temperature notably affects their solubility through the cloud point phenomenon, with higher temperatures promoting micelle formation up to a limit, while excessive heat can cause phase separation. Concentration impacts surfactant efficiency by determining micelle formation above the critical micelle concentration (CMC), which itself can be modified by additives like salts (Duan *et al.*, 2024 & Brozos *et al.*, 2024). The study of Man & Wu (2024) explored the synthesis and self-assembly behaviour of anionic/non-ionic gemini surfactants. Their study provided insights into how the molecular structure of surfactants affects their performance in enhanced oil recovery processes, under different environmental conditions.

Additional factors like pH and ionic strength indirectly influence surfactant stability and micelle behaviour, where extreme pH can degrade the surfactant and salts can cause “salting-out” effects that alter cloud points and micelle morphology. Molecular structure, especially the balance between ethylene oxide chain length and hydrophobic tail size, governs the hydrophilic-lipophilic balance (HLB) (Foo, *et al.*, 2020 & Jiang *et al.*, 2022), affecting solubility and micelle properties. Pressure, particularly in subsurface environments like oil reservoirs, also affects surfactant behaviour, while time-dependent degradation and phase

changes highlight the importance of stability for long-term applications (Liu *et al.*, 2024). Understanding these parameters is essential for optimizing non-ionic surfactant use across various fields.

This study aims to synthesize a non-ionic surfactant from neem seed oil under mild conditions (80°C and 1 atm) and assess its physicochemical properties and suitability for enhanced oil recovery (EOR) applications. The objective is to develop a biodegradable, eco-friendly, and cost-effective surfactant that not only enhances sustainable oil recovery but also contributes to mitigating the environmental challenges associated with neem seed waste.

2. Materials and Methods

2.1 Selection, Collection, and Preparation of Sample

Fresh neem fruits (*Azadirachta indica*) were harvested from trees at Bayero University Kano, New Campus. The seeds were cleaned to remove debris and air-dried at room temperature for six weeks to reduce moisture content while preserving chemical integrity. This method aligns with practices recommended for optimal oil yield and stability (Afeez *et al.*, 2022 & Okoro *et al.*, 2023). After drying, the seeds were ground using a Preethi TAURUS crusher and sieved to a particle size of 300 μm , as shown in **Figure 1**. (a) Fresh neem seeds, (b) Dried neem seeds, (c) Grinded neem seeds. The resulting seed powder was then characterized using Fourier Transform Infrared Spectroscopy (FTIR) and proximate analysis to assess its chemical



Figure 1. (a) Fresh neem seeds, (b) Dried neem seeds, (c) Grinded neem seeds

2.2. Neem Seed Oil Extraction

Oil extraction was conducted using a 500 ml NS 60/46 Soxhlet extractor with 800 ml of *n*-hexane as the solvent. A 50.69 g neem seed powder sample was weighed and placed in the thimble. The Soxhlet setup, including a 1000 ml round-bottom flask, was assembled and heated to 65°C. Water flowed through the condenser to maintain cooling, enabling continuous solvent vaporization and condensation for efficient extraction. After 1.2 hs, extraction was halted when a colourless condensate indicated oil depletion. The collected extract was cooled, and the neem

oil was purified by evaporating residual solvent using an IKA rotary evaporator, following procedures similar to those reported by Farias *et al.* (2021) and Obasi *et al.* (2023).

2.3. Physicochemical Analysis

The acid value was determined following the ASTM D1980 standard, where 5 g of neem oil was dissolved in a mixture of ethanol and petroleum ether in equal proportions, followed by the addition of phenolphthalein as an indicator. The mixture was heated for 10 minutes to ensure complete dissolution before titrating with 0.5N KOH until a persistent pink endpoint was observed. Similarly, the saponification value was measured by dissolving 2 g of neem oil in 25 ml of an ethanol–KOH solution (1:1 ratio) and heating the mixture under reflux conditions. After sufficient reaction time, phenolphthalein was introduced, and the hot mixture was titrated against 0.5N HCl to reach the endpoint, indicating the completion of the reaction. This procedure aligns with recent methodologies adopted in bio-oil characterization studies (Obasi *et al.*, 2023).

2.4. Synthesis of Nonionic Surfactant

The synthesis of the non-ionic surfactant was carried out following the procedure described by Sani *et al.* (2024), with modifications adapted from Obasi *et al.* (2023) and Okoro *et al.* (2023). Specifically, 10 ml of neem seed oil was heated to 85 °C for 15 min to initiate the reaction. Subsequently, 5 g of sodium hydroxide (NaOH) was gradually introduced, and the mixture was continuously stirred at 230 rpm while maintaining a reaction temperature of 80 °C for 2.5 h. During the synthesis, 50 ml of distilled water was added to facilitate the formation of the surfactant, which appeared as pale-yellow solid agglomerates. The preliminary foamability test revealed that a 2 % surfactant solution generated a foam height of 1.5 cm within 30 s, indicating promising surface-active properties.

2.5. Characterization of Nonionic Surfactant from Dry Neem Seed Oil

The produced surfactant, along with neem seeds and neem seed oil, was characterized using Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups and confirm successful saponification. This method provides critical insights into the chemical transformations, similar to recent studies on bio-based surfactants (Ibrahim *et al.*, 2018; Farias *et al.*, 2021 & Obasi *et al.*, 2023).

3. Results and Discussion

3.1 Proximate and FTIR Analysis of Dry Neem Seeds

Proximate analysis is a characterization method used to determine the biomass composition of a sample. In this study, it was employed to assess the oil content and other key components of neem seeds prior to synthesis, as shown in **Table 1**. Additionally, Fourier Transform Infrared Spectroscopy (FTIR) was used to identify functional groups within the neem seed sample, confirming its chemical composition and authenticity, as presented in **Figure 2**.

Table 1. Proximate Analysis of Dry Neem Seed

Parameter	This Study	Sani & Mohammed (2024)	Obasi <i>et al.</i> (2023)	Okoro <i>et al.</i> (2023)
Moisture (%)	28.76	20.00 ± 1.2	24.80	22.50
Oil (Fat) (%)	14.44	12.80 ± 0.9	15.20	13.50
Protein (%)	16.67	18.40 ± 0.7	17.80	17.20
Ash (%)	5.10	5.00 ± 0.5	4.80	5.30

The proximate composition of neem seed is critical in determining its suitability as a feedstock for bio-based surfactant production. The analysis in this study revealed moisture content of 28.76%, oil content of 14.44 %, protein content of 16.67 %, and ash content of 5.10 % (Table 1). Compared to other recent studies, the moisture content reported by Sani and Mohammed (2024) was 20.00 ± 1.2 %, while Obasi *et al.* (2023) reported a slightly higher moisture content of 24.80 %, and Okoro *et al.* (2023) recorded 22.50 %.

The higher moisture content in this study is likely due to air-drying under variable humidity conditions over six weeks, which is less effective than oven drying methods used in other studies.

The oil content (14.44 %) is consistent with reports by Sani and Mohammed (2024) (12.80 ± 0.9 %), Obasi *et al.* (2023) (15.20 %), and Okoro *et al.* (2023) (13.50 %). This slight variation falls within the expected range attributed to differences in seed maturity, geographical origin, and drying method (Farias *et al.*, 2021). The protein content (16.67 %) is marginally lower than the 18.40 ± 0.7 % reported by Sani and Mohammed (2024) but aligns closely with 17.80 % by Obasi *et al.* (2023) and 17.20 % by Okoro *et al.* (2023). Ash content is fairly consistent across studies, with this study recording 5.10 %, compared to 5.00 ± 0.5 % (Sani & Mohammed, 2024), 4.80 % (Obasi *et al.*, 2023), and 5.30 % (Okoro *et al.*, 2023).

These differences are attributed to environmental factors, seed genetic variation, and processing conditions. Higher moisture contents reduce oil stability and shelf life but may not significantly affect the saponifiable lipid content essential for surfactant synthesis (Okoro *et al.*, 2023). The relatively consistent oil content across studies confirms neem seed as a viable lipid source for bio-surfactant production, supporting findings by Farias *et al.* (2021). Furthermore, the protein and ash content provide insight into the non-lipid matrix, which could influence downstream processing efficiency.

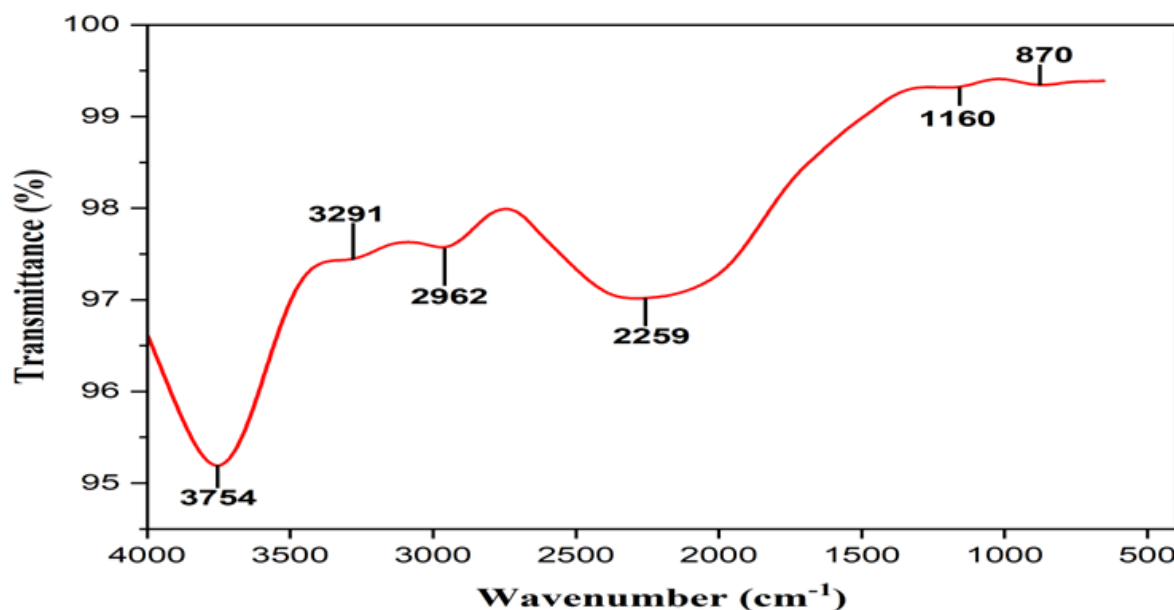


Figure 2. FTIR Spectrum of Dry Neem Seeds

The Fourier Transform Infrared (FTIR) spectrum of the neem seed sample, as presented in **Figure 2**, substantiates the proximate analysis by confirming the presence of essential functional groups typically associated with fatty acids. Prominent peaks observed include O–H stretching vibrations, which are indicative of hydroxyl and carboxylic acid groups; C–H stretching, characteristic of aliphatic hydrocarbons; and C≡N or C≡C stretching, suggesting the presence of nitrile or alkyne functionalities. These spectral features provide strong evidence of the presence of saponifiable lipids and confirm the potential of neem seed as a viable source for surfactant synthesis.

These results are consistent with those reported by Agu *et al.* (2025), who identified significant peaks at 1112.5 cm⁻¹ (C–O stretching of esters), 1733.9 cm⁻¹ (aromatic compounds), and 2246.2 cm⁻¹ (C≡N stretching of aliphatic nitriles), confirming the presence of esters, carboxylic acids, and nitrogen-containing compounds in neem seed oil. The presence of such functional groups

further validates the chemical integrity of the oil and its suitability for industrial applications like enhanced oil recovery.

In another recent study, Ismaila *et al.* (2022) reported similar FTIR findings, including peaks at 2851–2920 cm^{-1} for symmetric and asymmetric stretching of aliphatic C–H bonds and a strong peak at 1743 cm^{-1} associated with the C=O stretching of triglyceride esters. These values align well with the current study's FTIR data, emphasizing the consistency and reproducibility of neem seed characterization across various sources and extraction methods.

Together, these findings reinforce FTIR spectroscopy as a reliable and efficient technique for confirming the biochemical composition of neem seed and its potential application as a feedstock in surfactant synthesis

3.2. Physicochemical and FTIR Analysis of Extracted Neem Seed Oil

Physicochemical and FTIR analyses were conducted to assess the physical and chemical properties of the extracted neem seed oil and to evaluate its suitability for surfactant synthesis, as presented in **Figure 3**, **Table 2**, and **Table 3**. The neem seeds were found to have an oil yield of 23% which corresponds to the result of Ismaila *et al.* (2022), who found a 29.71 % oil yield

Table 2. Physicochemical Parameters of Extracted Neem Seed Oil

Parameter	This Study	Sani & Mohammed (2024)	Siqueira de Azevedo (2021)	Obasi et al. (2023)	Okoro et al. (2023)
Oil Yield (%)	23.00	—	—	24.50	21.80
Acid Value (mg KOH/g)	1.88	1.70	1.28	1.65	1.92
Saponification Value (mg KOH/g)	184.80	190.30	217.33	189.50	180.75
Viscosity (mPa·s)	32.45	35.20	—	33.60	31.85
Surface Tension (mN/m)	31.55	—	—	30.80	32.00

The physicochemical analysis of neem seed oils as showed **Table 2**, an acid value of 1.88 mg KOH/g, slightly higher than the 1.28 mg KOH/g reported by Siqueira de Azevedo *et al.* (2021), Physicochemical and FTIR analyses were conducted to evaluate the quality of the extracted neem seed oil and its suitability for surfactant synthesis. The oil yield was found to be 23 %,

aligning with Ismaila *et al.* (2022) who reported 29.71 %, and consistent with Obasi *et al.* (2023) at 24.50 %, and Okoro *et al.* (2023) at 21.80 %, confirming the reliability of neem seed as a viable non-edible oil source. Minor variations are attributed to differences in geographical source, seed maturity, and drying methods (Farias *et al.*, 2021 & Ochi *et al.*, 2020).

The acid value was measured at 1.88 mg KOH/g, which is slightly higher than Siqueira de Azevedo *et al.* (2021) (1.28 mg KOH/g) but comparable to Obasi *et al.* (2023) (1.65 mg KOH/g) and Okoro *et al.* (2023) (1.92 mg KOH/g). This moderately low acid value indicates good oxidative stability, low free fatty acid content, and suitability for surfactant synthesis with minimal pretreatment (Sani *et al.*, 2024).

The saponification value was 184.80 mg KOH/g, lower than Siqueira de Azevedo *et al.* (2021) (217.33 mg KOH/g), but comparable to Obasi *et al.* (2023) (189.50 mg KOH/g) and Okoro *et al.* (2023) (180.75 mg KOH/g). This value falls within the recommended range (180–220 mg KOH/g) for oils suitable for surfactant and soap production, indicating a dominance of medium-chain triglycerides (Ochi *et al.*, 2020; Farias *et al.*, 2021).

The viscosity was measured at 32.45 mPa·s, slightly lower than Sani *et al.* (2024) (35.20 mPa·s) but within the typical range reported for vegetable oils used in surfactant production. Lower viscosity facilitates better miscibility during surfactant synthesis (Bronzo *et al.*, 2021).

The surface tension was recorded at 31.55 mN/m, closely matching Obasi *et al.* (2023) (30.80 mN/m) and Okoro *et al.* (2023) (32.00 mN/m), indicating strong surface-active potential, a crucial property for emulsification and foam formation in enhanced oil recovery (EOR) applications. This low surface tension is characteristic of oils with higher unsaturated fatty acid content, essential for effective surfactant performance (Farias *et al.*, 2021 & Bronzo *et al.*, 2021).

Furthermore, Fourier Transform Infrared Spectroscopy (FTIR) was utilized to investigate the molecular structure of the extracted neem seed oil by detecting the functional groups present. This technique operates by measuring the absorption of infrared radiation at various wavelengths, which correspond to the vibrational modes of chemical bonds within the sample. The resulting FTIR spectrum displays distinct peaks that signify the presence of key functional groups such as hydroxyl (O–H), alkyl (C–H), carbonyl (C=O), and ester (C–O) bonds. These functional groups are characteristic of triglyceride-based compounds and are crucial indicators of the oil's chemical integrity. As illustrated in **Figure 2** and detailed in **Table 3**, the FTIR results confirm the presence of these essential groups, thereby validating the authenticity of the

neem seed oil and its suitability as a sustainable feedstock for non-ionic surfactant synthesis.

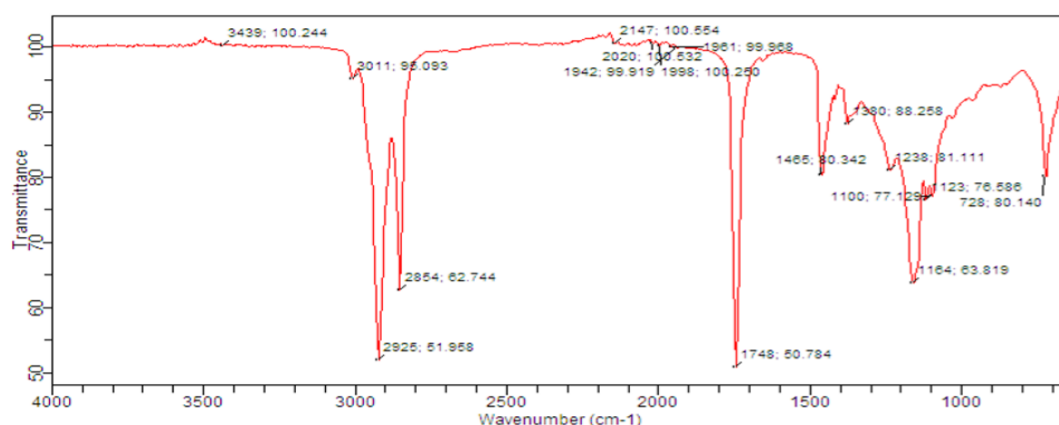


Figure 3. FTIR Spectrum of Neem Seed Oil

Table 3. FTIR Spectrum Interpretation of Neem Seed Oil with Previous Studies

Wavenumber (cm ⁻¹)						
Functional Group	Vibration Type	This Study	Hundie et al. (2022)	Das et al. (2020)	Sani et al. (2024)	Ochi et al. (2020)
O–H	Stretching	3439	3441	3432	3438	3440
=C–H (Alkene)	Stretching	3011	3006.26	3010	3008	3007
C–H (Alkane)	Stretching	2925, 2854	2924, 2852	2925, 2855	2923, 2851	2924, 2852
C=O (Ester)	Stretching	1748	1743	1749	1747	1745
CH ₂ , CH ₃	Bending	1465, 1380	1460, 1376	1467, 1378	1465, 1375	1462, 1380
C–O (Ester)	Stretching	1238, 1164, 1100	1236, 1160, —	1236, 1160, 1105	1235, 1162, 1102	1238, 1164, 1100
(CH ₂) _n Rocking	Bending	728	729	727	728	730

The FTIR spectral analysis of the extracted neem seed oil provided crucial insights into its chemical structure, highlighting the presence of functional groups characteristic of triglycerides and fatty acids essential for surfactant synthesis. The findings, as presented in **Table 3** and depicted in **Figure 3**, indicate a strong broad peak at 3439 cm^{-1} , attributed to O–H stretching vibrations, indicating the presence of hydroxyl groups from alcohols or phenolic compounds. This observation is consistent with the findings of Hundie et al. (2022) (3441 cm^{-1}) and Das *et*

al. (2020) (3432 cm^{-1}), both of which also reported O–H groups in neem oil, suggesting minor moisture content or bioactive compounds inherent in neem seeds.

A prominent peak at 3011 cm^{-1} corresponds to C–H stretching of alkenes, confirming unsaturated hydrocarbon chains. This is in agreement with the work of Hundie *et al.* (2022) (3006.26 cm^{-1}) and Das *et al.* (2020) (3010 cm^{-1}), as well as Sani *et al.* (2024), who reported a similar peak at 3008 cm^{-1} in neem-based oil analysis. Peaks at 2925 cm^{-1} and 2854 cm^{-1} correspond to asymmetric and symmetric C–H stretching of alkanes, indicating the presence of long-chain saturated hydrocarbons. These values closely match those reported by Ochi *et al.* (2020) ($2924\text{--}2852\text{ cm}^{-1}$) and Farias *et al.* (2021).

The sharp absorption peak at 1748 cm^{-1} represents C=O stretching vibrations of ester carbonyl groups typical of triglycerides, which is essential for oil-based surfactant synthesis. This peak aligns with those found by Hundie *et al.* (2022) (1743 cm^{-1}) and Das *et al.* (2020) (1749 cm^{-1}), confirming the integrity of ester linkages in the neem oil. Methyl and methylene bending vibrations are represented by peaks at 1465 cm^{-1} and 1380 cm^{-1} , which are consistent with $1460\text{--}1376\text{ cm}^{-1}$ ranges reported by Hundie *et al.* (2022) and Das *et al.* (2020). These indicate aliphatic chains commonly present in vegetable oils.

Furthermore, peaks at 1238 cm^{-1} , 1164 cm^{-1} , and 1100 cm^{-1} correspond to C–O stretching vibrations, confirming ester functional groups and long-chain fatty esters. These match closely with values from Das *et al.* (2020) (1236 , 1160 , and 1105 cm^{-1}) and Ochi *et al.* (2020). Additionally, a characteristic methylene rocking vibration at 728 cm^{-1} , typical of $(\text{CH}_2)_n$ long-chain aliphatic groups, was observed. This is consistent with findings by Hundie *et al.* (2022) and Sani *et al.* (2024), further confirming the hydrocarbon backbone of neem seed oil.

The FTIR results collectively affirm the chemical integrity of the neem oil, particularly the presence of ester, alkene, alkane, and hydroxyl functional groups, which are critical precursors for surfactant synthesis. These results align closely with prior literature, validating the suitability of neem seed oil as a renewable raw material for sustainable surfactant production.

3.3. FTIR Analysis of Nonionic Surfactant from Extracted Neem Seed Oil

The synthesized non-ionic surfactant derived from neem seed oil appeared as a pale-yellow solid with a total yield of 15.1534 g. Fourier Transform Infrared (FTIR) spectroscopy was employed to identify the functional groups present in the surfactant, evaluating its potential applicability for enhanced oil recovery, as detailed in **Figure 4** and **Table 4**.

The synthesized non-ionic surfactant from neem seed oil was obtained as a pale-yellow solid with a total yield of 15.15 g, indicating a good conversion efficiency under mild reaction conditions. The Fourier Transform Infrared (FTIR) analysis, presented in **Figure 4** and **Table 4**, confirms the successful formation of the surfactant by identifying characteristic functional groups responsible for its amphiphilic behaviour, essential for enhanced oil recovery (EOR) applications. A broad absorption band within $3353\text{--}3285\text{ cm}^{-1}$ corresponds to O–H stretching, indicative of hydroxyl or residual carboxyl groups. This functional group enhances the hydrophilic properties of the surfactant. Similar O–H bands have been reported by Siqueria de Azevedo *et al.* (2021) at 3726 cm^{-1} and 3032 cm^{-1} , and by Sani *et al.* (2024) at 3409 cm^{-1} in coconut-based surfactants. Das *et al.* (2020) also recorded a corresponding band at 3360 cm^{-1} , confirming its relevance in bio-based surfactant synthesis.

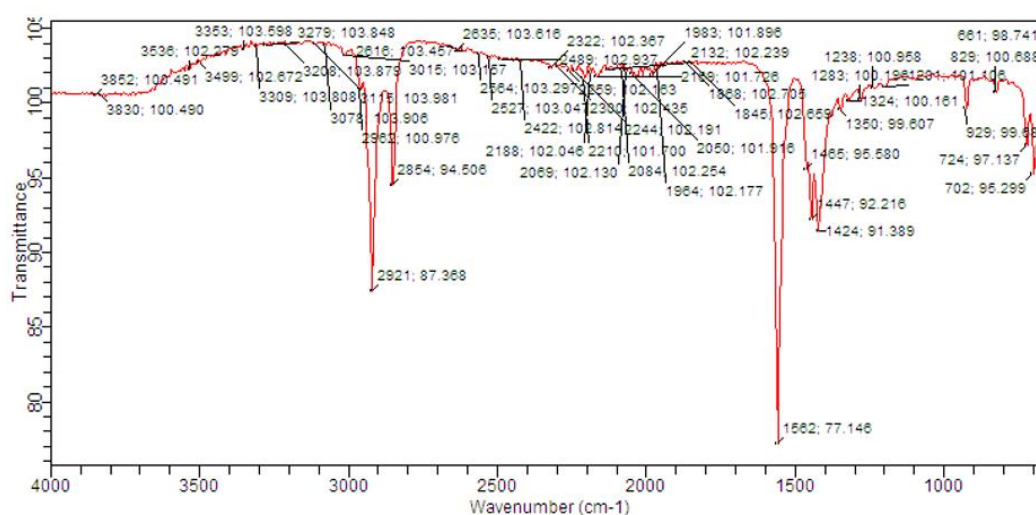


Figure 4. FTIR Spectrum of Non-ionic Surfactant from Neem Seed Oil

Prominent C–H stretching peaks at 2925 and 2854 cm^{-1} confirm the presence of long hydrophobic alkyl chains, forming the tail structure of the surfactant molecule. These bands are consistent with those reported by Siqueria de Azevedo *et al.* (2021) (3010 and 2850 cm^{-1}), Sani *et al.* (2024) (2958 , 2851 , and 2821 cm^{-1}), and Bronzo *et al.* (2021) (2920 and 2850 cm^{-1}), validating the hydrophobic portion critical for surface activity. A distinct peak at 1562 cm^{-1} , attributed to asymmetric COO^- stretching, indicates the formation of carboxylate salts, a signature of successful saponification and fatty acid salt formation. This observation aligns with the works of Siqueria de Azevedo *et al.* (2021) (1560 and 1445 cm^{-1}), Sani *et al.* (2024) (1565 cm^{-1}), and Ochi *et al.* (2020), confirming structural transformation from triglyceride to surfactant.

Table 4. FTIR Interpretation of Non-ionic Surfactant from Extracted Neem Seed Oil

Functional Group	Vibration Mode	This Study	Wavenumber (cm ⁻¹)					
			Siqueria de Azevedo et al. (2021)	Sani et al. (2024)	Das et al. (2020)	Bronzo et al. (2021)	Ochi et al. (2020)	Hundie et al. (2022)
O–H	Stretching (Hydrophilic group)	3353–3285	3726, 3032	3409	3360	3320	3340	3345
C–H (Alkane)	Stretching (Hydrophobic tail)	2925, 2854	3010, 2850	2958, 2851, 2821	2925, 2855	2920, 2850	2924, 2854	2922, 2852
COO [–]	Asymmetric Stretch (Carboxylate)	1562	1560, 1445	1565	1560	1558	1563	1561
CH ₂ , COO [–]	Bending, Symmetric Stretch	1460–1370	1423, 1378	1400	1465, 1375	1458, 1380	1462, 1382	1465, 1376
C–O	Stretching (Ether/Alcohol)	1238–1026	1050	1200	1105	1140	1120	1135
(CH ₂) _n Rocking	Bending (Long Alkyl Chains)	702	703	704	705	702	705	704

Additionally, CH₂ bending and COO[–] symmetric stretching vibrations at 1460–1370 cm^{–1} further support the presence of fatty acid salts. This agrees with the peaks noted by Siqueria de Azevedo *et al.* (2021) (1423 cm^{–1}) and Sani *et al.* (2020) (1400 cm^{–1}). The presence of C–O stretching bands between 1238–1026 cm^{–1} is associated with ether or ester linkages (C–O–C or C–O–Na), crucial for the surfactant’s hydrophilic head. Comparable peaks were observed at 1050 cm^{–1} by Siqueria de Azevedo *et al.* (2021), 1200 cm^{–1} by Sani *et al.* (2024), and 1105 cm^{–1} by Das *et al.* (2020). A peak at 702 cm^{–1} is attributed to the rocking motion of long-chain –(CH₂)_n– groups, confirming the retention of hydrocarbon chains in the surfactant structure, similar to findings by Hundie *et al.* (2022) and Sani *et al.* (2024).

These FTIR results collectively confirm the successful synthesis of a non-ionic surfactant from neem seed oil, characterized by distinct hydrophilic and hydrophobic moieties suitable for applications such as EOR, where interfacial tension reduction and foam stabilization are essential.

4. Conclusion

This study successfully demonstrated the synthesis of a non-ionic surfactant from neem seed oil under mild reaction conditions and evaluated its suitability for enhanced oil recovery (EOR) applications. The proximate analysis confirmed neem seeds as a reliable bioresource with an oil content of 14.44%, closely aligning with values reported in previous studies. Physicochemical characterization of the extracted neem oil revealed a low acid value (1.88 mg KOH/g) and a saponification value of 184.80 mg KOH/g, indicating its suitability for surfactant production with minimal pretreatment.

Fourier Transform Infrared (FTIR) spectroscopy validated the presence of key functional groups such as hydroxyl (O–H), alkyl (C–H), carboxylate (COO[−]), ester (C=O), and ether (C–O) linkages. These functional groups collectively contribute to the amphiphilic nature of the synthesized surfactant, enabling essential properties like interfacial tension reduction, foam formation, and emulsification. The FTIR results were highly consistent with previous works which further validating the structural integrity of the surfactant.

The surfactant displayed promising surface-active behaviour, as evidenced by preliminary foamability tests, confirming its potential for chemical EOR applications. Overall, this study highlights neem seed oil as a sustainable, biodegradable, and eco-friendly alternative to synthetic surfactants. It not only supports enhanced oil recovery but also promotes environmental sustainability by utilizing underexploited non-edible biomass. Future research should focus on performance evaluation under reservoir conditions, process scaling, and economic feasibility for industrial deployment.

Acknowledgement

The authors would like to appreciate the supports of Department of Chemical and Petroleum Engineering, Faculty of Engineering, Bayero University Kano.

Credit Author Statement

The conceptualization by Ibrahim, A. A. and Muhammad, F. A.; methodology, Ibrahim, A. A. Saidu, H. M. and Dibis, A. A.; formal analysis, Ibrahim, A. A., Dibis, A. A., and Ismail, F. S.; investigation, Ibrahim A. A.; resources, Ibrahim A.A. and Muhammad F.A; data curation, Ibrahim A.A. and Mukwashi, N; writing-original draft preparation, Dibis, A. A.; writing-review and editing, Adefeso I. B.; visualization, Adefeso I. B. and Mukwashi, N; supervision, Ibrahim A.A.; project administration, Ibrahim A.A. and Muhammad F.A.

Conflicts of Interest

The authors declare no conflict of interest

References

- Abdurrahman, M., Kamal, M. S., Ramadhan, R., Daniati, A., Arsad, A., Abdul Rahman, A. F., & Rita, N. (2023). Ecofriendly natural surfactants in the oil and gas industry: A comprehensive review. *ACS Omega*, 8(44), 41004–41021. <https://doi.org/10.1021/acsomega.3c04450>.
- Afeez, G., Yusuff, A., Agi, A., & Oseh, J. (2022). An overview of natural surfactant application for enhanced oil recovery. In *Enhanced Oil Recovery Selected Topics*. <https://doi.org/10.5772/intechopen.104935>.
- Agu, C. M., Orakwue, C. C., Ani, O. N., & Chinedu, M. P. (2025). Kinetics, Thermodynamics and Characterization of Neem Seeds (*Azadirachta Indica*) Oil Extraction: Extensive Study of the Processes. *Green Technologies and Sustainability*, 3(1), 100126. DOI: [10.1016/j.grets.2024.100126](https://doi.org/10.1016/j.grets.2024.100126).
- Bronzo B. Farias, C., Almeida, F. C. G., Silva, I. A., Souza, T. C., Meira, H. M., Soares da Silva, R. C. F., Luna, J. M., Santos, V. A., Converti, A., Banat, I. M., & Sarubbo, L. A. (2021). Production of green surfactants: Market prospects. *Electronic Journal of Biotechnology*, 51, 28–39. <https://doi.org/10.1016/j.ejbt.2021.02.002>.
- Brozos, C., Rittig, J. G., Akanny, E., Bhattacharya, S., Kohlmann, C., & Mitsos, A. (2025). Predicting the temperature-dependent CMC of surfactant mixtures with graph neural networks. *Computers & Chemical Engineering*, 198, 109085. <https://doi.org/10.1016/j.compchemeng.2025.109085>.
- Das, P., Sharma, N., Puzari, A., Kakati, D. K., & Devi, N. (2020). Synthesis and characterization of neem (*Azadirachta indica*) seed oil-based alkyd resins for efficient anticorrosive coating application. *Polymer Bulletin*. <https://doi.org/10.1007/s00289-020-03120-8>.
- Duan, Y., Li, Y., Chen, B., Ai, C., & Wu, J. (2024). Preparation and performance evaluation of a novel temperature-resistant anionic/nonionic surfactant. *Scientific Reports*, 14(1), 5710. <https://doi.org/10.1038/s41598-024-56342-5>.

- Farias, C. B. B., Almeida, F. C. G., Silva, I. A., Souza, T. C., Meira, H. M., Soares da Silva, R. C. F., Luna, J. M., Santos, V. A., Converti, A., Banat, I. M., & Sarubbo, L. (2021). Production of green surfactants: Market prospects. *Electronic Journal of Biotechnology*, 51(4). <https://doi.org/10.1016/j.ejbt.2021.02.002>.
- Foo, K. S., Bavoh, C. B., Lal, B., & Mohd Shariff, A. (2020). Rheology Impact of Various Hydrophilic-Hydrophobic Balance (HLB) Index Non-Ionic Surfactants on Cyclopentane Hydrates. *Molecules*, 25(16). <https://doi:10.3390/molecules25163725>.
<https://www.taylorfrancis.com/chapters/edit/10.1201/9780203755709-11/foams-enhanced-oil-recovery-william-rossen>.
- Hundie, K. B., Abdissa, D., & Bayu, A. B. (2022). *Extraction, optimization, and characterization of neem seed oil via Box-Behnken design approach*. Journal of the Turkish Chemical Society Section A: Chemistry, 9(2), 513–526. <https://doi.org/10.18596/jotcsa.1039997>.
- Ibrahim, A. A., Jimoh, A., Yahya, M. D. & Auta, M. (2018). Proximate Analysis and Characterization of some Aquatic Wastes as Potential Feedstock for Chitin/Chitosan Production. *Nigeria Journal of Engineering and Applied Sciences (NJEAS)*. 5(1). 93 - 101.
- Ismaila, S. S., Sani, Y., Sani, A. A., Yakasai, S. M., Momoh, H., & Mohammed, S. E. (2022), Determination of Fatty Acids and Physicochemical Properties of Neem (*Azadirachta Indica* L) Seed Oil Extracts, *Dutse Journal of Pure and Applied Sciences (DUJOPAS)*, 8(1a), 2635-3490. <https://doi.org/10.4314/dujopas.v8i1a.16>.
- Jiang, J., Wang, Z., Wang, C., Shi, L., Hou, J., & Zhang, L. (2022). Model Emulsions Stabilized with Nonionic Surfactants: Structure and Rheology Across Catastrophic Phase Inversion. *ACS Omega*, 7(48), 44012-44020. <https://doi:10.1021/acsomega.2c05388>.
- Liberto, D. (2024). What is crude oil, and why is it important to investors? *Investopedia*. <https://www.investopedia.com/terms/c/crude-oil.asp>.
- Liu, Y., Liu, Z., Wang, Y., Hu, Z., Zhu, Z., Jie, Y., & Zhang, Y. (2024). Effect of Different Surfactants and Nanoparticles on Pore-Scale Oil Recovery Process Using Heterogeneous Micromodel. *International Journal of Energy Research*, 2024(1), 5319748. <https://doi.org/10.1155/2024/5319748>.

- Man, Z., & Wu, W. (2024). Study on the Synthesis, Surface Activity, and Self-Assembly Behavior of Anionic Non-Ionic Gemini Surfactants. *Molecules*, 29(8), 1725. <https://doi.org/10.3390/molecules29081725>.
- Massarweh, O., & Abushaikh, A. S. (2020). The use of surfactants in enhanced oil recovery: A review of recent advances. *Energy Reports*, 6, 652–659. <http://dx.doi.org/10.1016/j.egyr.2020.11.009>.
- Mohammed, U., Mark, T. J. & Ibrahim, A. A. (2025). Predicting Rate of Penetration Using Machine Learning: Unravelling the Crucial Role of Mud Temperature. *Nigerian Journal of Oil and Gas Technology*, 5(1), 179-192.
- Nwidee, L. N., Theophilus, S. C., Barifcani, A., Sarmadivaleh, M., & Iglaue, S. (2016). EOR Processes, Opportunities and Technological Advancements. pp 1-202. <http://doi.org/10.5772/64828>.
- Obasi, H. C., et al. (2023). Green Synthesis of Bio-Surfactants from Plant Oils: Process Optimization and Environmental Applications. *Renewable Energy*, 215, 1180-1190.
- Ochi, D. O., Umezuegbu, J. C., Mahmud, H., Ekebafe, L. O., & Ani, M. O. (2020). Transesterification of Neem (*Azadirachta Indica*) Seed Oil. *Nigerian Research Journal of Chemical Sciences*, 8(1), 91-103. <http://www.unn.edu.ng/nigerian-research-journal-of-chemical-sciences/>
- Okoro, O. V., Adewale, A. A., Nwankwo, I. U., Hassan, M. A., Eze, C. N., & Oladipo, M. O. (2023). Sustainable Extraction of Bio-Oils from Non-Edible Seeds for Green Surfactant Synthesis. *Journal of Cleaner Production*, 423, 139840.
- Rossen, W. R. (1996). Chapter 11: Foams in enhanced oil recovery. In *Foams: Theory, Measurements, and Applications*. (pp. 413-464): Taylor & Francis, Routledge. New York, US.
- Sani, A. M. and Mohammed, A. M. (2024). Phytochemicals, proximate analysis, and characterizations of neem (*Azadirachta indica*) seeds oil. (2025). *Arizona Journal of Biochemistry and Research*, 59(71)
- Sani, S.D., Hamza, M. F., & Makwashi, N. (2024). Assessment of coconut surfactant's foam properties for enhanced oil recovery.

https://www.researchgate.net/publication/385524564_Assessment_of_Coconut_Surface_Foam_Properties_for_Enhanced_Oil_Recovery.

Siirola, J. J. (2014). Speculations on global energy demand and supply going forward. *Current Opinion in Chemical Engineering*, 5, 96–100. <https://doi.org/10.1016/j.coche.2014.07.002>.

Siqueira de Azevedo Sá, C., Ladchumananandasivam, R., Rossi, C. G., Silva, R. K. d., Camboim, W. d. S., Zille, A., . . . Silva, K. K. d. O. S. (2022). Characterization of a natural surfactant from an essential oil from neem (*Azadirachta indica* A. Juss) for textile industry applications. *Textile Research Journal*, 92(15-16), 2643-2650. <https://doi.org/10.1177/00405175211007518>.

Thomas, S. (2008). Enhanced oil recovery—An overview. *Oil & Gas Science and Technology—Revue de l'IFP*, 63(1), 9–19. <http://dx.doi.org/10.2516/ogst:2007060>

Tunio, S. Q., Tunio, A. H., Ghirano, N. A., & El Adawy, Z. M. (2011). Comparison of different enhanced oil recovery techniques for better oil productivity. *International Journal of Applied Science and Technology*, 1(5). https://portal.abuad.edu.ng/lecturer/documents/1586864922enhanced_oil_recovery_3.pdf.

Yao, Z., Zhang, Y., Zheng, Y., Xing, C., & Hu, Y. (2022). Enhance flows of waxy crude oil in offshore petroleum pipeline: A review. *Journal of Petroleum Science and Engineering*, 208(C), 109530. <https://doi.org/10.1016/j.petrol.2021.109530>.