ARID ZONE JOURNAL OF ENGINEERING, TECHNOLOGY & ENVIRONMENT
AZOJETE September 2026. Vol.22(3):808-821
Published by the Faculty of Engineering, University of Maiduguri, Maiduguri, Nigeria.
Print ISSN: 1596-2490, Electronic ISSN: 2545-5818
https://doi.org/10.63958/AZOJETE/2026/22/03/021
www.azojete.com.ng

ORIGINAL RESEARCH ARTICLE

HARNESSING NEEM SEED OIL FOR SUSTAINABLE BIO-BASED SURFACTANT SYNTHESIS: A PROMISING AGENT FOR ENHANCED OIL RECOVERY

S.A. Sa'ad[1], M.M. Ahmed[1], Y.M. Oseni[1*], F.A. Muhammad[1], J.I. Tahir[1], U.A. Isah[2]

[1]*Department of Chemical and Petroleum Engineering, Bayero University, Kano.*
[2]*Department of Chemical Engineering, University of Maiduguri.*
[*]*Corresponding author's email: yunusamohammedoseni@gmail.com*

ARTICLE INFORMATION

Received: 26th June 2026
Revised: 29th July 2026
Accepted: 30th August 2026

Keywords:
Agro-based surfactant
Azadirachta indica (neem)
Seed oil
Bio-based surfactant
Critical micelle concentration
Enhanced oil recovery

ABSTRACT

The declining productivity of mature oil reservoirs and the environmental concerns associated with synthetic surfactants have created a pressing need for sustainable chemical solutions in enhanced oil recovery (EOR) in reservoir-like conditions of elevated temperature, high-salinity brine, and crude oil interactions. This study aimed to develop and characterize a bio-based surfactant derived from the non-edible seed oil of Azadirachta indica (neem) for potential EOR application. The bio-based surfactant was synthesized through alkaline saponification using sodium hydroxide (NaOH) at 70–80°C for 3hours, yielding an anionic mixture of fatty acid sodium salts. The product exhibited an inherent alkaline pH of 9.0 and was evaluated for key performance properties using synthetic seawater as the aqueous medium. Surface tension measurements revealed a critical micelle concentration (CMC) of 1.0% w/v, at which the bio-based surfactant lowered the surface tension from 73.0 mN/m to an ultra-low value of 14.8 mN/m. A substantial foam half-life of 14.8 minutes was achieved from the foam stability test, while an emulsification index of 92% was produced from the emulsification studies, and thus, a stable emulsion that remained intact for over 24 hours as seen from the emulsification index. Following the results achieved, in particular, the 80% reduction in surface tension, foam stabilities and robust emulsion, showed a convincing interfacial activity and this was favorably compared with many bio-based surfactants described in the literature. These findings suggested that the neem-derived bio-based surfactants possesses the surface activity, stability, and micellization efficiency required to mobilizing trapped residual oil in reservoir rock. This study established neem seed oil as technically feasible, non-toxic and renewable feedstock for EOR bio-based surfactants, and subsequently recommended core-flooding experiments and techno-economic analysis for field-scale validation.

1.0 Introduction

The petroleum industry has been the main source of the global energy supply, yet critical confrontation comes from the diminution of productivity of crude oil from mature petroleum reservoirs (Okoro *et al.*, 2020). Though, primary and secondary recovery methods are used to extract a portion of the original oil in place (OOIP) or petroleum crude oil, but a significant portion of the crude oil still remains trapped within the complicated pore networks of the petroleum reservoir rocks (Alvarado & Manrique, 2010). This untapped crude oil spurred the growth of enhanced oil recovery (EOR) techniques, which are designed to mobilizing and produce this residual crude oil.

Primary recovery, which relies solely on the reservoir's natural drive mechanisms such as solution gas drive, water influx from an aquifer, and expansion of reservoir fluids and rocks, typically recoups only 5-15% of the original oil (Green & Willhite, 2018). As these natural drive mechanisms deplete (generally termed as reservoir's natural pressure), secondary methods one of which is water flooding, are employed to maintain reservoir pressure and displace additional oil, bringing the total recovery factor to between 15-40% (Sheng, 2014). However, capillary forces trapping the oil within the pores, and the high interfacial tension (IFT) between oil and the displacing fluids, limit the effectiveness of these conventional methods.

To overcome this physical limitation, EOR techniques target the fundamental properties (such as IFT, viscosity and mobility ratio, wettability, density) of the trapped oil. Among the EORs, the chemical approach, particularly surfactant flooding, has proven highly effective for EOR (Hesam & Elnaz, 2025). Surfactants work by drastically lowering the IFT between oil and the injected fluid, thereby emulsifying and mobilizing the previously trapped residual oil (Pal & Mandal, 2019). Despite their technical efficacy, most conventional surfactants are derived from synthetic, petroleum-based precursors. These compounds are often expensive, non-biodegradable, and pose potential ecological risks if released into the environment (Kłosowska-Chomiczewska *et al.*, 2017). Growing regulatory and public scrutiny of EOR operations, the environmental footprints such as carbon and greenhouse gas (GHG) emission, CO_2 – EOR, fugitive methane leak, water consumption and depletion, chemical pollution, and produced water contamination etc. have created an urgent need for more sustainable EOR alternatives.

This has spurred significant research into bio-based surfactants stemmed from renewable resources such as plant oils. These "green" surfactants offer the dual advantage of being environmentally benign – readily biodegradable and non-toxic, while also being potentially cost-effective due to the wide availability of agricultural feedstocks (Mnif & Ghribi, 2016). Neem (*Azadirachta indica*), a tree that is indigenous to the Indian subcontinent and now widespread in tropical regions, presents a promising candidate for this purpose. Neem seeds are rich in oil, comprising a complex of triglycerides and limonoids, which possess inherent amphiphilic structures suitable for surfactant synthesis (Okoro *et al.,* 2020). The utilization of non-edible neem seed oil aligns with sustainability principles, avoiding competition with food resources.

Therefore, this study aimed to formulate and characterize an agro-based surfactant (bio-based surfactant) from neem seed oil for EOR applications, testing it specifically under high-salinity natural seawater for EOR. The study evaluated critical performance parameters, including its capability to lower surface and interfacial tension (IFT), form stable emulsions, and maintain efficacy under reservoir-like conditions. By investigating neem oil's viability, this work sought to contribute a sustainable, economically attractive, and technically sound alternative to conventional synthetic surfactants, supporting the industry's transition towards more environmentally responsible extraction practices.

2. Materials and Method

2.1 Materials

Neem seed oil (for oil extraction, referred to: Muhammad *et. al.,* 2026; Okoro *et al.,* 2020) was used as the primary triglyceride feedstock for the saponification reaction. Sodium hydroxide (NaOH) pellets of the analytical grade, and distilled water, were used to prepare the lye solution. The synthesized bio-based surfactant was subsequently diluted to a series of percentage weight per volume (% w/v) concentrations using deionized water followed by subsequent dilution with natural seawater as the brine medium for all performance evaluations, among which is the solubility test (Osama & Ahmad, 2020; Osama & Ahmad, 2023).

2.2 Method

The bio-based surfactant was synthesized through the alkaline saponification of neem (*Azadirachta indica*) seed oil. This process involved the hydrolysis of triglyceride esters present in the oil, catalyzed by a strong base (NaOH), to yield the corresponding sodium salts of fatty acids (soap – the bio-based surfactant) and glycerol (Figure 1). The bio-based surfactant produced through the alkaline saponification of neem oil is an anionic surfactant, constituting a mixture of sodium carboxylate salts ($R\text{-}COO^{-}\ Na^{+}$). The primary components of this mixture were sodium salts of the major fatty acids found in neem oil, namely sodium oleate (IUPAC: Sodium (9Z)-octadec-9-enoate), sodium linoleate (IUPAC: Sodium (9Z,12Z)-octadeca-9,12-dienoate), and sodium palmitate (IUPAC: Sodium hexadecanoate).

A concentrated lye solution was prepared by dissolving 150 g of NaOH pellets in 500 mL of distilled water. The solution was cooled to ambient temperature (33°C) before use (Galindo-Luna *et al.,* 2019; Mebrat, 2021). A measured volume of 250 mL of neem seed oil was transferred into a 1000 mL round-bottom flask (Muhammad *et al.*, 2026). The flask was placed on a heating magnetic stirrer equipped with a temperature control probe. The prepared 30% (w/v) NaOH solution was gradually added to the 250 mL neem oil under constant stirring at a moderate speed (250 rpm) to ensure efficient mixing. The reaction mixture was heated gently and maintained at a temperature range of 70–80°C. The saponification reaction was allowed to proceed for 3 h under continuous stirring to ensure complete conversion of triglycerides. Upon completion of the reaction, the crude bio-based surfactant mixture was brought down from the heat source and cooled to room temperature (27 °C). Following the saponification reaction, a similar method employed by Kelechi *et. al.* (2024), the crude bio-based surfactant mixture was subjected to a purification process to isolate the surfactant molecules from the glycerol by-product and other impurities. To precipitate the bio-based surfactant and extract glycerol, a volume of 150 mL of anhydrous methanol was added to the mixture under constant stirring for 30 min with gentle warming (40°C). The resulting suspension was then filtered under vacuum using a Buchner funnel lined with Whatman No. 1 filter paper. The filtrate, containing glycerol and methanol, was discarded. The solid residue retained on the filter paper was collected as the crude biosurfactant cake. The process was repeated by redissolving the biosurfactant cake with 100 mL ethanol, the resulting suspension filtered, the filtrate discarded and the solid residue retained on the filter paper collected as the purified crude bio-based surfactant (anionic) with accordance to the method employed by Okoro *et al.,* (2023) (Figure 1). The pH of the bio-based surfactant was measured and recorded using pH meter from Mettler Toledo. The bio-based surfactant was stored in a sealed, airtight container at ambient conditions for subsequent analysis and application. The aqueous solubility of the bio-based surfactant was evaluated at 1% and 5% (w/v) concentrations dilution with sea-water. The foaming characteristics of the 5% (w/v) bio-based surfactant solution were evaluated to assess its potential for foam-based enhanced oil recovery (EOR). The surface activity of the bio-based surfactant was quantitatively assessed by measuring the surface tension of its aqueous solutions across a concentration range (0.1, 0.5, 1.0, and 5.0% w/v).

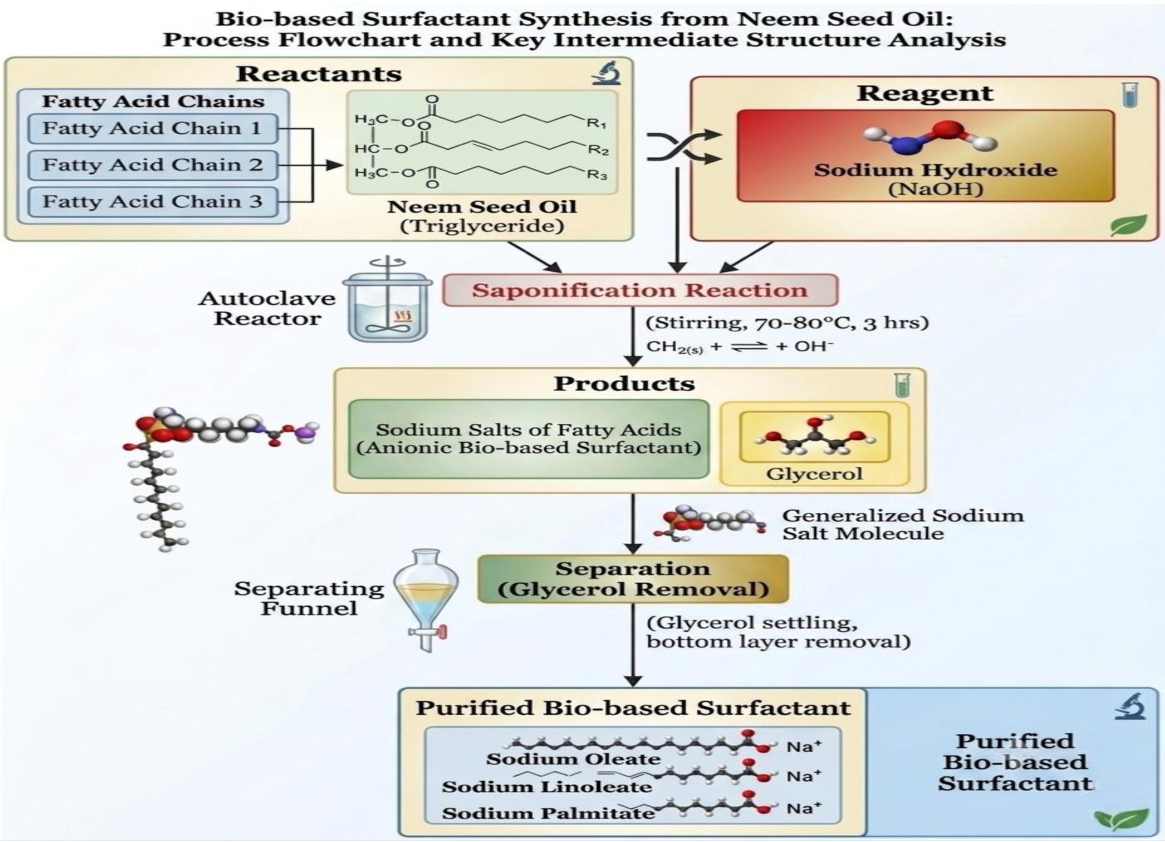

Figure 1: Process Flow Diagram for Bio-based Surfactant Synthesis from Neem Seed Oil through Saponification Reaction

2.2.1 Bio-based surfactant characterization

The fundamental properties of the bio-based surfactant were determined to assess its suitability for enhanced oil recovery (EOR). The following analyses were performed:

i. Physical Appearance and Visual Solubility

The physical state and colour of the bio-based surfactant were visually inspected at ambient temperature (Okoro *et. al.,* 2023). Its odour was noted using the standard wafting technique (Sani & Mohammed, 2024). The visual solubility of the bio-based surfactant was tested on sea-water at concentrations of 1% and 5% (w/v) bio-based surfactant (Osama & Ahmad, 2023). First, stock solutions of the bio-based surfactant were prepared at two concentrations, 1% and 5% (w/v), by dissolving 0.2g and 0.5g of the bio-based surfactant in deionized water, respectively, making a final volume of 20 mL for each stock solution (Osama & Ahmad, 2023). To perform the solubility test, 10 mL of each stock solution was transferred into separate graduated cylinders and mixed with 10 mL of natural seawater (33 510.01 mg/L) (1:1 v/v ratio). This produced final test concentrations of 0.5% and 2.5% (w/v) of the surfactant in the seawater mixture. Each cylinder was then shaken vigorously for 1 minute and allowed to stand undisturbed for 5 minutes. After the standing period, the mixtures were visually inspected, and the results were recorded based on whether a clear, homogeneous solution formed or whether any visible phase separation (e.g., precipitation, cloudiness, or layering) occurred (Osama & Ahmad, 2023).

ii. pH Measurement

The pH of a 1% (w/v) aqueous bio-based surfactant solution was measured at room temperature (27 °C) using a calibrated pH meter (Mettler Toledo FE28, Switzerland). The electrode was immersed directly into the solution, and the reading was recorded after stabilization (Atolani *et. al.,* 2016).

iii. Foaming Capacity

The foaming properties of the purified neem-derived anionic bio-based surfactant were evaluated using a modified shaking method. A 100 mL volume of a 5% (w/v) bio-based surfactant solution was poured in a 250 mL graduated cylinder with an internal diameter of 2.5 cm. The cylinder sealed, and inverted 10 times vigorously. Immediately after shaking was over, the total height of the liquid and the foam column (height) was recorded. The initial foam height was computed by subtracting the known height of the liquid phase. The foam height was subsequently measured at 5-, 10-, and 30- minute intervals of standing undisturbed. The foam half-life ($t_{1/2}$), described as the time taken for the foam to collapse to half of its initial height, was also recorded (Atolani *et al.,* 2016; Rossen, 1996).

iv. Emulsification Capacity

The emulsification capacity of the purified neem-derived anionic bio-based surfactant was evaluated against crude oil. Bio-based surfactant solutions were prepared at dilution concentrations of 0.1%, 0.5%, 1.0%, and 5.0% (w/v) with seawater. For each test, equal volumes (2.5 mL) of the crude oil and bio-based surfactant solution were combined in a 10 mL graduated test tube. The mixture was vortexed or shaken vigorously for 2 minutes to form a primary emulsion. The emulsions were subsequently centrifuged at 5000 rpm for 15 minutes to accelerate phase separation. After centrifugation, the total height of the mixture and the height of the stable emulsion layer were measured directly from the test tube (Osama & Ahmad, 2023).

The emulsification index (EI) was computed as follows:

$$EI\ (\%) = (\text{Height of Emulsion Layer} / \text{Total Height of Mixture}) \times 100 \qquad 1$$

v. Surface Tension and Critical Micelle Concentration (CMC)

The surface tension of aqueous bio-based surfactant solutions at concentrations of 0.1%, 0.5%, 1.0%, and 5.0% (w/v) using seawater for the dilution was ascertained at ambient temperature using the capillary rise method. The internal radius of each capillary tube was pre-measured using a travelling microscope (Muhammad *et al.,* 2026).

For each measurement, a glass beaker was filled with the diluted biosurfactant solution and placed on a retort stand. An optical pin was adjusted to touch just the surface of the solution, and its position was recorded from the vernier scale of a travelling microscope. A pre-wetted capillary tube was then immersed in the solution. To enhance the visibility of the meniscus, a minute amount of red dye was added. The microscope was focused on the meniscus inside the capillary, and its position was recorded when the bottom of the meniscus was tangential to the cross-wire. The height of the capillary rise (**h**) was calculated as the difference between the meniscus position and the pin position. This procedure was performed in triplicate for each concentration using three separate capillary tubes (Muhammad *et. al.,* 2026).

The surface tension (γ) was calculated from the capillary rise data using the following fundamental equation:

$$\gamma = (\rho \cdot g \cdot h \cdot r) / 2 \qquad 2$$

where **ρ** is the density of the solution (g/mL) and taken as (950 kg/m³), '**g**' is the acceleration due to gravity (9.81 m/s²), '**h**' is the capillary rise height (m), and '**r**' is the internal radius of the capillary tube (m). The measured heights (**h**) and radii (**r**) in millimetres were converted to meters (by multiplying by 10^{-3}) prior to calculation to maintain consistent SI units. The final results in N/m were multiplied by 1000 to report them in the standard unit of mN/m.

The critical micelle concentration (CMC) was ascertained by plotting the surface tension (γ) as a function of the logarithm of biosurfactant concentration ($\log_{10}$ C) (Osama & Ahmad, 2023).

vi. Fourier-Transform Infrared (FTIR) Spectroscopy

FTIR analysis was carried out to establish characteristic functional groups of the bio-based surfactant produced. A small quantity, 10 µL of the pure bio-based surfactant was directly placed on the diamond crystal of the infrared spectroscopy machine. The spectrum was recorded over a wavelength range of 4000 to 650 cm^{-1} with a resolution of 8 cm^{-1} (Muhammad *et. al.,* 2026).

3. Results and Discussion

The purified neem-derived anionic biosurfactant was characterized, analyzed, and discussed in the following subsequent sections.

3.1 Bio-based Surfactant Characterization

3.1.1 Physical appearance; solubility and implication for enhanced oil recovery (EOR)

i. Physical Characterization

The synthesized bio-based surfactant is a yellowish-brown, gel-like substance, as shown in Plate 1. This observed colour and physical state are characteristic of many bio-based surfactants derived from natural oils or extracts, where the presence of carotenoids, tocopherols, or other natural pigments from the source material can impart a yellow to brown hue (Soy *et al.,* 2020; Ezgi *et al.,* 2020). More significantly, the observed high viscous nature and gel-like structure of the bio-based surfactant are highly desirable traits for Enhanced Oil Recovery (EOR) (Muhammad *et. al.,* 2026). In porous reservoir rock, a viscous surfactant solution can improve the macroscopic sweep efficiency by mitigating viscous fingering, thereby displacing a larger volume of crude oil toward the production well (Kumika *et al.,* 2021).

Furthermore, the surfactant possessed a strong, distinctive odour described as nutty (Sani & Mohammed, 2024). This specific aromatic profile strongly indicated the nature of the precursor compounds used in the synthesis. A nutty scent is often associated with the degradation products of triglycerides or the presence of specific carbonyl compounds and pyrazines formed during the saponification or heating process (Umesh *et al.,* 2007). While the odour confirms the organic origin of the product, it is a parameter that may require refinement, such as purification or deodorization, for applications in cosmetics or food industries where a neutral scent is preferred even though the applications mentioned are not the focal point for the bio-based surfactant synthesis (Sani and Mohammed, 2024).

Plate 1: Physical Appearance of the Synthesized Bio-based surfactant, Showing its Characteristic Gel Like and Yellowish-Brown Coloration

ii. Visual Solubility and Micellization Behaviour

As summarized in Table 1, both solutions formed optically clear, homogeneous mixtures upon agitation, with no observable precipitation or phase separation.

Table 1: Visual Aqueous Solubility of the Biosurfactant and pH

Concentration (% w/v)	Visual Observation	pH
1%	Clear Solution	9.0
5%	Clear Solution	-

The formation of clear solutions across this concentration range was a critical indicator of the bio-based surfactant's high solubility and effective self-assembly into micellar structures in water (Osama & Ahmad 2023). The absence of turbidity or precipitation at 5% w/v concentration confirmed a high critical micelle concentration (CMC) or, more importantly, excellent solubility of the micelles themselves post-formation (Osama & Ahmad, 2023). This suggested the bio-based surfactant molecules possessed a sufficiently hydrophilic head group that prevented crystallization or liquid crystal formation, which could have caused cloudiness and reduced performance (Birnbach *et al.,* 2023).

For Enhanced Oil Recovery (EOR), this high aqueous solubility was a fundamental prerequisite. It ensured that the bio-based surfactant could be effectively injected and transported through the reservoir's aqueous phase without the risk of pore-throat blockage due to precipitation (Satyajit *et al.,* 2022). The ability to form stable, clear solutions at 5% (w/v) concentration indicated a robust potential for formulating high-concentration injection slugs, which are often required to maintain an effective concentration deep within the petroleum reservoir, overcoming adsorption onto rock surfaces (Narendra *et al.,* 2020). Furthermore, the formation of proper micelles is directly linked to the primary mechanism of oil mobilization. This meant the ultra-low interfacial tension between the bio-based surfactant solution and trapped crude oil was typically achieved at or above the CMC (Ryan & Dorthe, 2012). The observed solubility behaviour strongly suggested that the formulated biosurfactant was capable of achieving this state, making it a promising candidate for chemical EOR applications.

3.1.2 pH measurement (alkalinity and formulation stability)

The pH of a 1% (w/v) aqueous solution of the bio-based surfactant was determined to be 9.0 (Table 1). This alkaline nature was a crucial property that provided important insights into the surfactant's ionic class and its subsequent performance (Neha *et al.,* 2022).

The measured pH was a confirmation of the anionic character of the bio-based surfactant as this was consistent with the presence of carboxylate head groups ($-COO^{-}$) resulting from the saponification of natural oil feedstocks (Banat *et al.,* 2010). This alkalinity was not just an incidental property but a key factor in the formulation's stability (Neha *et al.,* 2022). A pH of 9.0 guaranteed the long-term chemical stability of the bio-based surfactant by preventing the hydrolysis of ester linkages or the protonation of the carboxylate groups back into free fatty acids, which were less soluble and could lead to precipitation (Sheng, 2016).

From an Enhanced Oil Recovery (EOR) perspective, this alkaline property was highly advantageous. When introduced into a petroleum reservoir, an alkaline surfactant can synergistically interact with acidic components of the crude oil to produce *in-situ* soaps, effectively reducing the total amount of synthetic surfactant required and further lowering the interfacial tension (IFT) between the displacing fluid and the trapped oil (Sheng *et al.,* 2015). Furthermore, the elevated pH promoted electrostatic repulsion between the anionic surfactant head groups and the negatively charged sandstone surfaces, which are predominant in oil-bearing formations (Osama & Ahmad, 2020). This repulsion can significantly lower surfactant adsorption onto the reservoir rock, thereby improving the efficiency and economic viability of the chemical flood by ensuring a sufficient surfactant concentration reaches the target oil zone (Xinyu *et al.,* 2025).

3.1.3 Foaming capacity

The results, detailed in Table 2, revealed a dynamic and highly favorable foaming profile characterized by progressive stabilization (Atolani *et al.,* 2016).

Table 2 is a depiction of a crucial "post-generation foam growth". The foam height was seen to increase by over 840% from its original value and braked at a stable maximum foam height of 3.87 cm. A situation like this where foam volume expands instead of decaying instantly after formation is confirmation of slow adsorption kinetics (Atolani *et al.,* 2016). This gave an insight that the bio-based surfactant molecules and molecular aggregates have shown a continuous migration from the bulk solution to the air-water interface and thus strengthening positively the foam lamellae of the bio-based surfactant as time passed (Wei & Mazen, 2022). The 'foam half-life ($t_1/_2$) of 14 minutes and 49 seconds' achieved from measurement of the foam height, signified further, a robust stability of the bio-based surfactant and also affirming that the foam effectively resisted bubble merging and liquid runoff (Atolani *et al.,* 2016).

Table 2: Foam Stability Profile of the 5% (w/v) Bio-based Surfactant Solution

Time (minute)	Foam Volume (mL)	Foam Height (cm)	Percentage Increase in Foam Height (%)	Observations
0 (Initial)	2	0.41	-	Initial Foam Formation Upon Shaking
5	12	2.44	495.12	Pronounced Foam Growth
10	16	3.26	695.12	Continued Expansion and Stabilization
30	19	3.87	843.90	Peak Foam Volume Achieved, Indicating High Stability
14.81 Half-Life ($t_{1/2}$)	9.501	1.935	371.95	Robust Stability

Form flooding behaviour was exceptionally advantageous for enhanced oil recovery (EOR), as the initial growth of the foam and its subsequent growth were of paramount importance for *in-situ* performance. Foam generation not only began immediately when injected into the petroleum reservoir but also continued to generate and stabilize deep within the rock formations. Such properties of the foam can selectively block high-permeability "thief zones" thereby diverting displaced fluids into un-swept oil-rich areas and thus, improved macroscopic sweep efficiency (Arne *et al.,* 2020; Gandomkar *et al.,* 2010). The synergistical effect between the foam substantial growth and a substantial half-life pointed out the fact that this bio-based surfactant can propagate a durable foam capable of enduring the challenging conditions of a reservoir long enough to effectively mobilize and displace trapped crude oil for EOR (Lake *et al.,* 2014). This positioned the neem seed oil-based surfactant as a highly promising and sustainable agent for mobility control in EOR operations.

Foam Height (H_{foam}) and Half-Life ($t_{1/2}$) Calculation

The foam heights presented in Table 2 were derived from the measured foam volumes (V_{foam}). The internal diameter of the graduated cylinder was 2.5 cm (radius, r = 1.25), giving a cross-sectional area (A_c) of:

$$A_c = \pi r^2 = \pi\,(1.25\text{ cm})^2 = 4.91\text{ cm}^2 \quad (3)$$

The foam height (H_{foam}) at each time point was computed using the formula:

$$H_{foam}\,(\text{cm}) = V_{foam}\,(\text{mL}) / A_c\,(\text{cm}^2) \quad (4)$$

where mL is equivalent to cm^3.

The foam half-life ($t_{1/2}$) is described as the time taken for the foam height to collapse to half of its *maximum* value (Atolani *et al.,* 2016). The maximum foam height observed was 3.87 cm. Therefore, the half-life height is:

Foam Half-life$_{(t1/2)}$ Height = 3.87 cm / 2 = 1.935 cm

Foam Half-life$_{(t1/2)}$ Volume = A_c x Foam Half-life$_{(t1/2)}$ Height = 4.91 cm^2 x 1.935 = 9.501 cm^3

Thus, Foam Half-life$_{(t1/2)}$ Volume = 9.501 mL

The experimentally measured time for the foam height to decay to 1.935 cm was 14 minutes and 49 seconds (14.81 minute). This value represents the foam half-life, a key metric of its (foam) stability. Thus, a definitive measure of the bio-based surfactant actual stability and performance over time (Atolani *et al.,* 2016).

3.1.4 Emulsification capacity

The emulsification capacity of the bio-based surfactant against crude oil is a critical indicator of its interfacial activity and this was quantitatively assessed. The calculated Emulsification Index (EI) and the observed stability over 24 hours are presented in Table 3.

Table 3: Emulsification Index (EI) and Stability of Bio-based surfactant Solutions at Varying Concentrations against Crude Oil

Biosurfactant Concentration (% w/v)	Emulsion Layer Height (cm)	Total Height (cm)	Emulsion Index (%)	Emulsion Stability (24 hrs)
0.1	1.5	5.0	30.0	Unstable (Separated)
0.5	3.2	5.0	64.0	Partially stable
1.0	4.3	5.0	86.0	Stable
5.0	4.6	5.0	92.0	Very stable

Table 3 indicated a positively strong relationship between the emulsification performance and the bio-based surfactant concentration. A dramatical increase of the emulsification index (EI) was observed from 30% at 0.1% (w/v) concentration to 92% at 5.0% (w/v) concentration. This pattern was a behaviour exhibited by bio-based surfactant as it is known that higher concentrations resulted to a greater number of molecules to be adsorbed onto the reservoir rock at the oil-water interface and thus brought about more obvious reduction in interfacial tension and large volumes of stable emulsion droplets formed (McClements, 2015).

The stability data is clearly seen from Table 3 as the emulsion formed with the 0.1% (w/v) solution was unstable, an indication that there was an insufficient bio-based surfactant concentration to form a closely-packed and stabilized film around the oil droplets, thus resulting to a very swift coalescence. Partial stability was observed to be reached at 0.5% (w/v) of the bio-based surfactant, pointing to the fact that the concentration was close to the critical threshold for effective droplet coating. The 1.0% (w/v) and 5.0% (w/v) concentrations was observed to form stable, to a very stable emulsions that could withstand phase separation for over 24 hours. This high stability proved the formation of a robust, viscoelastic film at the oil-water interface, a property often associated with biosurfactants that can form complex multilayers or have molecular structures that provide strong steric and electrostatic hindrance against droplet coalescence (Santos *et al.,* 2016

These findings were highly crucial for recouping trapped residual oil in petroleum reservoir rock for enhanced oil recovery (EOR). At a concentration of 1.0% (w/v), the bio-based surfactant formed a stable emulsion with the crude oil and thus the potency to be able to emulsify the trapped residual crude oil within the petroleum reservoir rock (Atolani *et al.,* 2016). This emulsification characteristic was a paramount parameter for mobilizing residual crude oil, since droplets from emulsification could be transported through pore throats more easily compared to a continuous oil phase (Lake *et al.,* 2014). An injection slug of the bio-based surfactant at a concentration of or above 1.0% (w/v) would be crucial for sustainable emulsification and for viable oil displacement in the petroleum oil reservoir as showcased by the concentration-dependent stability profile (a critical operational parameter) of the bio-based surfactant (Rossen, 1996).

3.1.5 Surface tension and critical micelle concentration (CMC)

The results, summarized in Table 4, demonstrated a concentration-dependent reduction in surface tension, a hallmark of surfactant efficacy (Muhammad *et al.,* 2026).

Surface Tension Calculation for 0.1% Concentration:

- $\rho = 950\ kg/m^3$
- $g = 9.81\ m/s^2$
- h = 14.83 mm = **0.01483 m**
- r = 0.93 mm = **0.00093 m**

$\gamma = (950 \times 9.81 \times 0.01483 \times 0.00093) / 2$

$= 0.001207 / 2$

= **0.00006077 kN/m** (or **60.8 mN/m**)

Thus, the demonstration above was applied consistently across all tested concentrations (Table 4).

Table 4: Surface Tension Measurements of Neem Seed Oil-Based Surfactant Solutions at Different Concentrations

Surfactant Concentration (% w/v)	Capillary Rise in Height (h) (mm)	Surface Tension (mN/m)
0.1	14.83	60.8
0.5	10.42	45.2
1.0	6.03	26.1
5.0	3.42	14.8

The bio-based surfactant has the potential to lower the surface tension of seawater from 73.0 mN/m to 60.8 mN/m and also effective in lowering the surface tension even at a low concentration of 0.1% (w/v). As observed from Table 4, increase in concentration of the bio-based surfactant resulted to progressive decrease in surface tension that reached a value of 14.8 mN/m at a 5.0% (w/v) concentration. This ultra-low surface tension achieved, proved a good performance indicator of the bio-based surfactant potency and also reflected the surfactant's efficiency in adsorption onto reservoir rock at the air-water interface and thus reduction of interfacial energy (Rosen & Kunjappu, 2012).

The variation of surface tension with surfactant concentration (Figure 2) revealed a distinct break in the curve (inflection point or CMC point). The surface tension decreased sharply up to a concentration of 1.0% w/v, beyond which the rate of decrease diminished significantly, forming a plateau. The concentration at this inflection point was identified as the Critical Micelle Concentration (CMC), approximately 1.0% w/v for this bio-based surfactant with a surface tension of 26.1 mN/m (Figure 2) (Osama & Ahmad, 2023). Below the CMC, surfactant molecules predominantly migrate to the interface. At and above the CMC, the interface became saturated, and additional molecules clustered in the bulk to form micelles (Myers, 2005).

For Enhanced Oil Recovery, these findings are profoundly significant. The ability to achieve an ultra-low surface tension (14.8 mN/m) was directly correlated with the ability to achieve an ultra-low oil-water interfacial tension in a reservoir (Muhammad *et al.,* 2026). This drastic reduction in interfacial tension was the primary mechanism for mobilizing trapped residual oil by overcoming capillary forces that held the oil in place within the pore throats of the rock (Lake *et al.,* 2014). Furthermore, the identification of a clear CMC around 1.0% w/v provided a crucial parameter for designing EOR injection slugs. Formulating the injection fluid at or slightly above the CMC ensured maximum interfacial activity and optimal use of the biosurfactant, thereby improving the economic viability of the EOR process (Hana *et al.,* 2012). The high efficiency of this neem-based bio-based surfactant positioned it as a highly promising, sustainable alternative for chemical enhanced oil recovery.

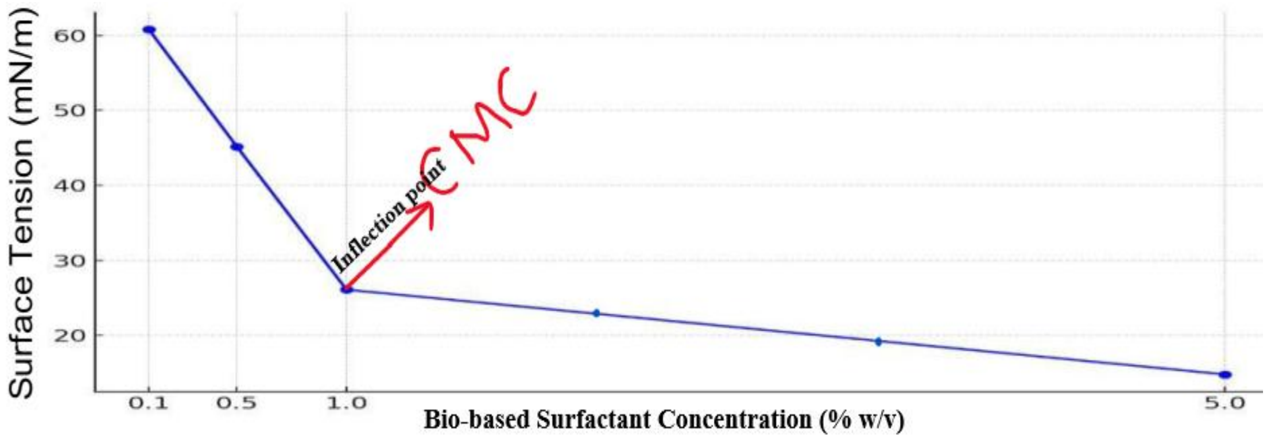

Figure 2: Surface Tension against Bio-based surfactant Concentration Indicating the Point of CMC

3.1.6 Fourier-transform infrared (FTIR) spectroscopy

Fourier-Transform Infrared (FT-IR) spectroscopy was employed to shed light on the molecular structure and identify key functional groups of the synthesized bio-based surfactant. The analysis of the undiluted sample provides definitive evidence of a successful synthesis from a neem seed oil precursor (Muhammad *et al.,* 2026).

The spectrum and corresponding peak assignments are summarized in Table 5, which confirmed the presence of critical moieties characteristic of a glycolipid. While Figure 3 shows a pictorial view of the FT-IR spectrum of the biosurfactant from Agilent Technologies from which Table 5 was summarized.

Table 5: Major FT-IR Absorption Bands and Their Assignments for the Undiluted Bio-based Surfactant

Wavenumber (cm⁻¹)	Transmittance (%)	Assignment	Molecular Origin & Significance
~3588	99.387	O-H Stretching	Hydroxyl groups from alcohols, carboxylic acids, or residual water. A broad feature here is typical of biosurfactants and indicates hydrogen bonding (Satpute *et al.,* 2010).
~2925	51.436	C-H Asymmetric Stretch	Methylene (-CH_2-) groups in long aliphatic chains.
~2854	62.012	C-H Symmetric Stretch	Methylene (-CH_2-) and methyl (-CH_3) groups in long aliphatic chains. The presence of these strong aliphatic peaks confirms the lipid-derived "tail" of the surfactant (Coates, 2000).
~1748	50.087	C=O Stretching	Ester carbonyl group. This is a definitive peak indicating the presence of triglyceride esters from the neem oil or ester linkages in a synthesized surfactant (Silverstein *et al.,* 2014).
~1465	79.852	C-H Bending	Scissoring vibration of methylene (-CH_2-) groups, further confirming the aliphatic hydrocarbon tail.
~1100	76.793	C-O Stretching	Ether or alcohol C-O bonds. This can be associated with the polar head group of a glycolipid-type biosurfactant (Satpute *et al.,* 2010).

The FT-IR spectrum was consistent with the structure of a bio-based surfactant derived from a natural oil. The simultaneous presence of a strong aliphatic chain signature (2925, 2854 cm⁻¹) and polar functional groups (C=O, C-O, O-H) confirmed the amphiphilic nature of the bio-based surfactant produced, and as such, this aligned with the basic need for all surfactant. The triglyceride structure of the neem oil had been modified as depicted and strongly suggested from the prominent ester peak at ~1748 cm⁻¹. This modification, formed part of the bio-surfactant final structure that is usual for many bio-based surfactants (Muhammad *et al.,* 2026).

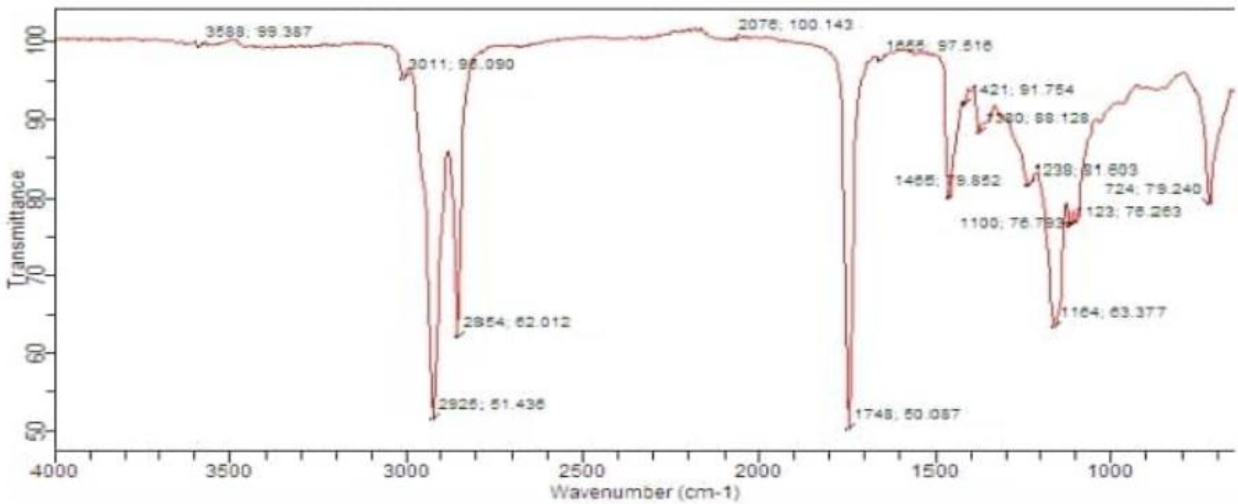

Figure 3: FTIR Spectrum for the Undiluted Synthesized Bio-based Surfactant

For Enhanced Oil Recovery, this molecular structure is highly advantageous. The long aliphatic chains (the "tail") provided high hydrophobicity, enabling strong interaction and penetration into crude oil. Simultaneously, the polar head group (comprising O-H and C-O bonds) brought about certainty, an adequate aqueous solubility and also facilitated the adsorption of the bio-based surfactant onto reservoir rock at the oil-water interface. This amphiphilicity was the direct molecular-level driver for the potent surface activity and stable emulsion formation observed (Osama & Ahmad, 2020). While the direct measurement of interfacial tension (IFT) was not possible due to equipment constraints, the ultra-low surface tension achieved was a well-established proxy for predicting a corresponding reduction in IFT, which is the key mechanism for mobilizing trapped oil in reservoir rock pores (Hana *et al.,* 2012; Xinran *et al.,* 2024). The identified structure confirmed the synthesis of a molecule with the inherent potential to effectively lower capillary forces and enhanced oil displacement efficiency.

4. Conclusion

This study successfully showed conspicuously the synthesis and characterization of a high-performance bio-based surfactant derived from non-edible neem seed oil (*Azadirachta indica*), that exhibited a strong potential for enhanced oil recovery (EOR). The bio-based surfactant, primarily composed of sodium oleate, linoleate, and palmitate, that showed an uncommon, satisfying surface-active properties, including a low critical micelle concentration (CMC) of 1.0% (w/v), an ultra-low surface tension of 14.8 mN/m, and outstanding foam capacity and stability with a half-life of 14.8 minutes. The ability to form stable emulsion (92% emulsification index) and its intrinsic alkalinity (pH 9.0) made it suitable for displacing residual oil in petroleum reservoir rock. By successfully converting an agricultural by-product into an effective EOR agent, this research work provided a sustainable alternative route to synthetic surfactants. The findings suggested that neem seed oil is a feasible, economically attractive and eco-friendly feedstock that aligns with petroleum industry's operational needs, and the principles of green chemistry and circular economy.

To advance toward future deployment needs critical future work like validation of core-flooding under reservoir conditions such as temperature, pressure and salinity, and this must result to incremental oil recovery. Formulation optimization as part of the advancement, should investigate systematically the combine effect between Alkaline-Surfactant-Polymer (ASP) slugs and blending with other bio-based surfactants. Thorough techno-economic analysis (TEA) and life cycle assessment (LCA) are critical for economic and environmental concern. At pilot scale, process intensification and various sources of synthesis (e.g enzymatic catalysis) should be the focal point to avoid the buildup of scale. Oilfield chemical compatibility, biodegradability, and standardized ecotoxicological tests ought to be carried out so as to be certain of operational readiness and environmental safety.

Acknowledgment
The authors gratefully acknowledge the support and resources provided by the Department of Chemical and Petroleum Engineering, Bayero University, Kano. Sa'ad Sa'ad Abubakar specifically wishes to thank his project supervisor, Murtala M. Ahmed, for his major guidance, and Yunusa M. Oseni for his invaluable mentorship in manuscript preparation and writing. Gratitude is also extended to the entire departmental staff for their unwavering support.

References

Alvarado, V., and Manrique, E. 2010. Enhanced Oil Recovery: An Update Review. *Energies*, 3(9): 1529–1575. https://doi.org/10.3390/en3091529.

Arne, S., Jonas, S., Per, A. O., and Morten G. A. 2020. Foam Generation, Propagation and Stability in Porous Medium. Transport in Porous Media (2020) 131: 5-21. https://doi.org/10.1007/s11242-019-01250-w.

Atolani, O., Olabiyi, E. T., Issa, A. A., Azeez, H. T., Onoja, E. G., Ibrahim, S. O., Zubair, M. F., Oguntoye, O. S., and Olatunji, G. A. 2016. *Green synthesis and characterisation of natural antiseptic soaps from the oils of underutilised tropical seed. Sustainable Chemistry and Pharmacy, 4(2016): 32–39.* doi:10.1016/j.scp.2016.07.006

Banat, I. M., Andrea, F., Isabella, G., Giuseppina B., Maria, G. M., Letizia, F., Thomas, J. S., and Roger, M. 2010. Microbial biosurfactants production, applications and future potential. *Applied Microbiology and Biotechnology*, 87(2): 427-444. https://doi.org/10.1007/s00253-010-2589-0

Birnbach, J., Schmiedel, P., and Karg, M. 2023. Multi-component phase behaviour of biosurfactants. *Current Opinion in Colloid & Interface Science, 66(1)*, 101765. https://doi.10.1016/j.cocis.2023.101765

Coates, J. 2000. Interpretation of Infrared Spectra, A Practical Approach. In *Encyclopedia of Analytical Chemistry*. John Wiley & Sons. https://doi.org/10.1002/9780470027318.a5606.

Ezgi, B. A., Oltan, C., Yanis, C., Laurent, D., and Nadir, D. 2020. Production of Bio-Based Pigment from Food Processing Industry By-Products (Apple, Pomegranate, Black carrot, Red Beet Pulps) Using Aspergillus carbonarius. Journal of Fungi, 6: 240; doi:10.3390/jof6040240.

Galindo-Luna, Y. R., Gómez-Arias, E., Ibarra-Bahena, J., Morales-Flores, A., and Romero, R. J. 2019. *New Concentration Correlations of NaOH Aqueous Solutions for a Thermodynamic Process. Journal of Chemical & Engineering Data, acs.jced.9b00536 –*. doi:10.1021/acs.jced.9b00536.

Gandomkar, A., Kharrat, R., Motealleh, M., Khanamiri, H. H., Nematzadeh M., and Ghanzanfari M. H. 2010. Petroleum Science and Technology (Taylor and Francis), 30(10): 976-985. https://doi.org/10.1080/10916466.2010.493905.

Green, D. W., and Willhite, G. P. 2018. *Enhanced Oil Recovery* (2nd Edition). Society of Petroleum Engineers.

Hana, Al-S., Yahaya, M. Al-W., Saif, Al-B., Abdulkadir E., Ali, Al-B., Sanket J., and Shahab, A. 2012. Residual Oil Recovery via Injection of Biosurfactant, Chemical Surfactant and Mixtures of both under Reservoir Condition: Induced Wettability and Interfacial Tension Effects. SPE Reservoir valuation & Engineering.

Hesam, G. and Elnaz, K. 2025. Comprehensive review of hybrid chemical enhanced oil recovery methods: synergistic mechanisms, applications, and insights into chemical-based water alternating gas techniques. Journal of Petroleum Exploration and Production Technology (Springer) 15:98. https://doi.org/10.1007/s13202-025-01999-5.

Kelechi, I. K., Dodo, S. I., Bello, A. M., and Hassan, U. 2024. Production and Characterization of New Surfactant Formulated from Thevetia Peruviana Seed Oil for Use in Enhanced Oil Recovery. Petroleum Science and Engineering, 8(1): 1-6. https://doi.org/10.11648/j.pse.20240801.11

Kłosowska-Chomiczewska, I. E., Mędrzycka, K., and Hallmann, E. 2017. Biosurfactants–biodegradability, toxicity, efficiency in comparison with synthetic surfactants. In *Biosufactants: Sources, Properties, and Applications* (pp. 1-24). Springer, Cham. https://doi.org/10.1007/978-3-319-62482-4_1.

Kumika, I., Tadashi, S., Masaki, A., Kenichi, S. and Hideki, S. 2021. Rheologhy of α-Gel Formed. Langmuir 2021, 37: 7032-7038.

Lake, L. W., Johns, R. T., Rossen, W. R., and Pope, G. A. 2014. Fundamentals of Enhanced Oil Recovery. Society of Petroleum Engineers.

McClements, D. J. (2015). *Food Emulsions: Principles, Practices, and Techniques*. CRC Press.

Mebrat, Y. 2021. A Laboratory Manual for the Preparation of Chemical Reagents, Solutions and Special Indicators. Cognizance Journal of Multidisciplinary Studies, 1(2): 1-62.

Mnif, I., and Ghribi, D. 2016. Glycolipid biosurfactants: main properties and potential applications in agriculture and food industry. *Journal of the Science of Food and Agriculture*, 96(13): 4310-4320. https://doi.org/10.1002/jsfa.7759.

Muhammad, F. A. Ibrahim, A. A., Adefeso, I. B., Isah, B. A., Mohammed, U., and Oseni, Y. M. 2026. Development of Anionic Surfactant from Neem Seed Oil for Enhanced Oil Recovery (EOR) Application. Bayero Journal of Engineering and Technology (BJET),21(1): 38-45.

Myers, D. (2005). *Surfactant Science and Technology* (3rd ed.). Wiley-VCH.

Narendra, K., Saif, A., Amit, K., and Ajay, M. 2020. Design and Formulation of Surfactant Stabilized O/W Emulsion for Application in Enhanced Oil Recovery: effect of pH, salinity and temperature. Oil and Gas Science and Technology – Rev. IFP Energies Nouvelles 75, 72 (2020). https://doi.org/10.2516/ogst/2020066.

Neha, R., Aman, K. B., Jayati, T., Raj, K. S., and Neeraj, A. 2022. Synthesis and Characterization of Biosurfactants from Non-edible Feedstocks: Comparative Assessment and Their Applications in Biodiesel. Chemistry Europe, 7 (36): e202202031. https://doi.org/10.1002/slct.202202031.

Okoro, O. E., Omeru, C., and Dosunmu, A. 2020. Experimental evaluation of neem seed oil as a sustanable source for eco-friendly surfactant in enhanced oil recovery. *Journal of Petroleum Exploration and Production Technology*, 10(8): 3543–3554. https://doi.org/10.1007/s13202-020-00917-1.

Okoro, O. V., Adewale, A. A., Nwankwo, I. U., Hassan, M. A., Eze, C. N., and Oladipo, M. O. 2023. Sustainable Extraction of Bio-Oils from Non-Edible Seeds for Green Surfactant Synthesis. Journal of Cleaner Production, 423, 139840.

Osama, M. and Ahmad, S. A. 2020. The use of surfactants in enhanced oil recovery: A review of recent advances. Elsevier, Energy Reports 6 (2020): 3150-3178. https://doi.org/10.1016/j.egyr.2020.11.009

Osama, M. and Ahmad, S. A. 2023. Application of surfactants in enhancing oil recovery from tight carbonats: Physicochemical properties and core flooding experiments. Elsevier, Geoenergy Science and Engineering, 221 (2023): 211400. https//doi.org/10.1016/j.geoen.2022.211400

Pal, N., and Mandal, A. 2019. Enhanced oil recovery performance of gemini surfactant-stabilized nanoemulsions functionalized with partially hydrolyzed polymer/silica nanoparticles. *Energy & Fuels*, 4(1): 746760. https://doi.org/10.1021/acs.energyfuels.9b03529.

Rosen, M. J., and Kunjappu, J. T. 2012. *Surfactants and Interfacial Phenomena* (4th ed.). John Wiley & Sons.

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.

Ryan, T. A., and Dorthe W. 2012. Investigating the pore-scale mechanisms of microbial enhanced oil recovery. Elsevier, Journal of Petroleum Science and Engineering, 94-95 (2012): 155-164.

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).

Santos, D. K. F., Rufino, R. D., Luna, M. J., Santos, A. V., and Sarubbo, A. L. 2016. Biosurfactants: Multifunctional Biomolecules of the 21st Century. *International Journal of Molecular Sciences*, 17(3): 401. https://doi.org/10.3390/ijms17030401.

Satpute, S. K., Banpurkar, A. G., Dhakephalkar, P. K., Banat, I. M., and Chopade, B. A. 2010. Methods for investigating biosurfactants and bioemulsifiers: a review. *Critical Reviews in Biotechnology*, 30(2): 127-144.

Satyajit, C., Saket, S., and Abhijit K. 2022. Comprehensive Review on the Role of Surfactants in the Chemical Enhanced Oil Recovery Process. Industrial & Engineering Chemistry Research, 2022, 61, 1: 21-64. https://doi.org/10.1021/acs.iecr.1c03301.

Sheng, J. J. 2016. Critical review of alkaline-polymer flooding. Journal of Exploration Production Technology. https://doi.org/10.1007/s13202-016-0239-5.

Sheng, J. J. 2014. A comprehensive review of alkaline–surfactant–polymer (ASP) flooding. *Asia-Pacific Journal of Chemical Engineering*, 12(2): 179-197. https://doi.org/10.1002/apj.1824

Sheng, J. J., Bernd, L., and Nasser, A. 2015. Status of polymer-flooding technology. Journal of Canadian Petroleum Technology. 54(02): 116-126. https://doi.org/10.2118/174541-PA.

Silverstein, R. M., Webster, F. X., Kiemle, D. J., and Bryce, D. L. 2014. *Spectrometric Identification of Organic Compounds*. John Wiley & Sons, 8th Edition.

Soy R. C., Pius K. K., and Kiplangat R. 2020. Synthesis, Characterization, and Evaluation of Solution Properties of Sesame Fatty Methyl Ester Sulfonate Surfactant. ACS Omega 2020, 5: 28643 – 28655.

Umesh, N. C., John, F. S., John, H. O., and Jeffrey H. H. 2007. Removal of VolatileOrganic Compounds from Surfactant Solutions by Flash Vacuum Stripping in a Packed Column. Groundwater Monitoring and Evaluation. 18(4): 157-165. https://doi.org/10.1111/j.1745-6592.1998.tb00175.x.

Wei, Y., and Mazen, Y. K. 2022. Review of foam stability in porous media: The effect of coarsening. Journal of Petroleum Science and Engineering, 208 (2022): 109698. https://doi.org/10.1016/j.petrol.2021.109698.

Xinran, C., Bo, C., Di, W., and Shuangchun, Y. 2024. A Review on Approach for Biosurfactants Enhance Oil Recovery Technology. Geomicrobiology Journal, 41(9): 910-920. https://doi.org/10.1080/01490451.2024.2401881.

Xinyu, T., Yaoyao, T., Yuhui Z., Pujiang, Y., Chuangye W., and Jinhe L. 2025. Study on Mechanism of Surfactant Adsorption at Oil-Water Interface and Wettability Alteration on Oil-Wet Rock Surface. Molecules 2025, 30: 2541. https://doi.org/10.3390/molecules30122541.