



DEVELOPMENT OF ANIONIC SURFACTANT FROM NEEM SEED OIL FOR ENHANCED OIL RECOVERY (EOR) APPLICATIONS

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Received: 21-07-2025

Revised: 28-07-2025

Accepted: 24-12-2025

Published: 26-12-2025

Abstract: The demand for sustainable and eco-friendly alternatives in the oil and gas industry has intensified interest in bio-based surfactants for enhanced oil recovery (EOR). This study investigates the synthesis and characterization of an anionic surfactant derived from *Azadirachta indica* (neem) seed oil, offering a biodegradable and cost-effective substitute for conventional synthetic surfactants used in sandstone reservoirs. Neem seeds were cleaned, air-dried, milled, and subjected to solvent extraction using Soxhlet apparatus with *n*-hexane. The extracted oil underwent proximate and physicochemical analyses to assess its surfactant-producing potential. Proximate analysis revealed a moisture content of 28.76%, oil content of 14.44%, protein content of 16.67%, and ash content of 5.10%. Physicochemical parameters such as acid value (1.88 mg KOH/g), saponification value (184.80 mg KOH/g), oil yield (23.00%), viscosity (32.45 mPa·s), and surface tension (31.55 mN/m) supported its suitability for surfactant production. An anionic surfactant was synthesized via saponification followed by sulfonation with concentrated sulfuric acid under controlled temperature and agitation. The pale-yellow final product exhibited foam formation, indicating surface activity. The results of FTIR analysis confirmed the chemical transformation, with characteristic peaks at 3290 cm⁻¹ (O-H), 1640 cm⁻¹ (C=C), and 1104 cm⁻¹ (S=O), validating the presence of alcohol, alkene, and sulfonate functional groups essential for amphiphilic behavior. SEM analysis of the synthesized surfactant at varying magnifications (500x, 1000x, and 1500x) revealed granular and vesicle-like structures. These micelle-forming features are indicative of self-assembly, crucial for interfacial tension reduction, emulsion stability, and wettability alteration in EOR applications. The results demonstrate that neem seed oil is a promising renewable feedstock for the production of effective anionic surfactants. Its favorable chemical profile, successful structural modification, and supportive morphology confirm its potential in green chemical applications such as EOR, detergents, and emulsifiers. Future work should include performance testing under simulated reservoir conditions to validate field applicability and scale-up potential, especially in regions rich in neem resources.

Key words: Renewable feedstock; neem seed oil; surfactant synthesis; anionic surfactant; amphiphilic.

1 Introduction

The oil and gas industry plays a pivotal role in global economic development, particularly in hydrocarbon-rich nations such as Nigeria, where the discovery of crude oil in 1958 significantly shaped its economic trajectory (Akinlo, 2012). Conventional oil recovery is divided into three phases: primary, secondary, and tertiary. During the primary recovery stage, natural

reservoir forces—such as gas drive and water drive—are used to extract oil, but this method typically recovers only 20–30% of the original oil in place. (OOIP) (Liberto, 2024; Sani et al., 2024). Secondary methods, predominantly water flooding, can increase recovery to around 40%, yet approximately 60% of the oil remains trapped due to capillary forces and reservoir heterogeneity (Tunio et al., 2011; Thomas, 2008). Enhanced oil recovery (EOR) techniques aim

to extract an additional 20-30% of OOIP, thereby increasing total recovery to 60–65% or more (Yao et al., 2022; Nagy et al., 2025). EOR approaches include thermal, gas injection, microbial, and chemical methods. Among these, CO₂ injection is widely adopted due to its dual benefits of viscosity reduction and carbon sequestration (Nwidee et al., 2016). The Water Alternating Gas (WAG) method, initially applied by ExxonMobil in 1959, alternates water and gas injection to improve sweep efficiency (Adawy et al., 2011). Further advancements, such as Foam-Assisted WAG (FAWAG), use surfactant-stabilized foams to reduce gas channeling and enhance displacement efficiency (Rossen, 1996; Martijn et al., 2020). Chemical EOR, particularly surfactant flooding, is widely utilized due to its ability to reduce interfacial tension (IFT), alter rock wettability, form stable emulsions, and mobilize trapped oil (Farias et al., 2021). Surfactants, composed of hydrophilic and hydrophobic moieties, are classified as anionic, cationic, nonionic, or zwitterionic based on the charge of the hydrophilic head group (Massarweh et al., 2020). Anionic surfactants are particularly effective in sandstone reservoirs, while cationic and nonionic types perform better in carbonate formations (Negin et al., 2017).

Despite their performance advantages, synthetic surfactants suffer from drawbacks such as high production costs, environmental toxicity, and poor biodegradability. Consequently, regulatory bodies in Europe and North America are phasing out hazardous variants such as Alkylphenol Ethoxylates and Linear Alkylbenzene Sulfonates (Siirola, 2014; Berihun et al., 2021). Moreover, inappropriate surfactant selection can lead to wettability changes, mineral dissolution, and pore blockage, undermining oil recovery (Negin et al., 2017).

The search for eco-friendly alternatives has turned attention to bio-based surfactants produced from renewable resources. Initially derived from edible oils such as palm, soybean, and coconut, biosurfactants have raised ethical concerns related to food-versus-fuel conflicts, particularly in food-insecure regions (Aitani, 2004; Romero Vega & Gallo Stampino, 2025). This has prompted a shift toward non-edible oil sources like jatropha, castor, and neem seed (*Azadirachta indica*). Neem seed oil is especially promising due to its non-edible nature, widespread availability in tropical regions, high triglyceride content, low toxicity, biodegradability, and chemical suitability for surfactant synthesis.

This study addresses the dual challenge of environmental sustainability and EOR efficiency by investigating the synthesis and application of an anionic surfactant derived from neem seed oil. The aim is to provide a green, cost-effective alternative to synthetic surfactants for use in sandstone reservoir

environments. The objectives of this study are to extract neem seed oil using optimized solvent extraction techniques; synthesize an anionic surfactant through appropriate chemical modification processes; and conduct physicochemical and structural characterization of the synthesized surfactant using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), surface tension measurements, and viscosity analysis, with comparative evaluation against reported data in existing literature.

2 Materials and Method

2.1 Selection, Collection and Preparation of Sample

Fresh neem fruits (*Azadirachta indica*) were sourced from neem trees located within the New Campus of Bayero University, Kano. The seeds were manually cleaned to remove surface impurities and subsequently air-dried at ambient room temperature for six weeks to reduce moisture content while preserving their chemical integrity, following the method described by Okoro et al. (2023). The dried seeds were then milled using a Preethi TAURUS crusher and sieved to obtain a uniform particle size of 300 µm. The resulting seed powder was subjected to characterization through Fourier Transform Infrared Spectroscopy (FTIR) and proximate analysis to determine its chemical composition.

2.2 Neem Seed Oil Extraction

Neem seed oil was extracted using a Soxhlet apparatus with n-hexane as the solvent. A 50.69 g portion of neem seed powder was placed in a thimble and inserted into the extractor. Approximately 800 mL of n-hexane was added to a 1000 mL round-bottom flask positioned on a heating mantle maintained at 65 °C. Continuous condensation was ensured by circulating water through the condenser. As the solvent vaporized and condensed, it repeatedly contacted the sample, effectively extracting the oil. After 1.2 hours, the extraction was terminated upon observing a colorless condensate, indicating oil exhaustion. The oil was cooled and purified using a rotary evaporator, as illustrated in Figure 1 and following Obasi et al. (2023).

2.3 Neem Seed Oil Extraction

To determine the acid value of neem seed oil, 5 g of the sample was dissolved in equal parts ethanol and petroleum ether, followed by the addition of five drops of phenolphthalein. The mixture was heated for 10 mins and titrated with 0.5 N KOH to a pink endpoint. A blank was also prepared and titrated for reference. For saponification value, 2 g of the oil was dissolved

in 25 mL of 1:1 ethanol and KOH, heated under reflux, and titrated with 0.5 N HCl after phenolphthalein addition. The procedure was repeated with a blank, following methods reported by Obasi et al. (2023).



Figure 1: Soxhlet Extraction and Purification of Neem Seed Oil

2.4 Synthesis of Anionic Surfactant

According to the methodology employed by Sani et al. (2024); 10 ml of neem seed oil was weighed and simmered for 15 mins at 85 °C. 5 g of NaOH was added and heated continuously for 2.5 h at 80 °C while stirring at 230 rpm. A total of 50 ml of distilled water was added during the synthesis period until a solid product was formed. The product was observed as pale-yellow solid agglomerates. A large quantity of foam was formed when 2 % (w/v) was prepared in distilled water and after being manually agitated, a foam height of 1.5 cm was observed after 30 s. To reduce the foam height, the anionic surfactant solution was further heated to 80 °C, using a surfactant to sulfuric acid molar ratio of 1:0.5, with acid added dropwise under continuous magnetic stirring for 2 h. The temperature was then lowered to 50 °C, and 0.5 M NaOH was added for 1 h until neutralization (pH 7) was achieved. The final surfactant was manually agitated, producing a foam height of 0.8 cm after 30 s as reported by Sampora et al. (2017).

2.5 Characterization of Anionic Surfactant from Neem Seed Oil

The Surfactant produced was characterized using Fourier Transform Infrared Spectroscopy (FTIR) to study the wavelengths present that represent presence of certain organic groups which would validate the result of the obtained anionic surfactant.

3 Results and Discussion

3.1 Proximate and FTIR analysis on Neem Seeds

Proximate analysis was conducted to determine the biomass composition, including oil content, of the neem seed sample prior to synthesis, as shown in Table 1. Fourier Transform Infrared Spectroscopy

(FTIR) further confirmed the presence of key functional groups, validating the sample's chemical composition and suitability for surfactant production, as illustrated in Figure 1.

Table 1: Proximate analysis result of neem seed sample

Parameter	This Study (%)	Obasi et al. (2023) (%)	Okoro et al. (2023) (%)
Moisture	28.76	24.80	22.50
Oil (Fat)	14.44	15.20	13.50
Protein	16.67	17.80	17.20
Ash	5.10	4.80	5.30

The proximate analysis of the neem seed sample as presented in the Table 1, revealed a moisture content of 28.76%, oil content of 14.44%, protein content of 16.67%, and ash content of 5.10%. These values reflect the biomass composition and are influenced by factors such as seed variety, harvest time, storage, and drying conditions. The relatively high moisture content may be attributed to extended air-drying under ambient conditions, which is less efficient than oven-drying methods used in other studies. For instance, Obasi et al. (2023) recorded 24.80%, and Okoro et al. (2023) reported 22.50%.

The oil content observed aligns well with values reported by Obasi et al. (2023) at 15.20% and Okoro et al. (2023) at 13.50%, supporting neem seed's viability as a lipid source for bio-based surfactant synthesis. Protein content (16.67%) is comparable to 17.80% and 17.20% reported by Obasi et al. (2023) and Okoro et al. (2023) respectively. Ash content also falls within expected limits. These findings confirm the presence of saponifiable lipids and essential biomolecules suitable for surfactant production. Variations across studies are attributed to environmental and genetic factors, while the consistent oil yield underscores neem seed's potential in green chemical applications.

The FTIR spectrum of the neem seed sample, shown in Figure 1, complements the proximate analysis by confirming key functional groups typical of fatty acids. Notable peaks include O–H stretching (hydroxyl/carboxylic acids), C–H stretching (aliphatic hydrocarbons), and C≡N or C≡C stretching (nitriles or alkynes), all indicating the presence of saponifiable lipids suitable for surfactant production.

The FTIR spectrum of the oil shows a broad band between 3754-3291 cm⁻¹, attributed to O–H stretching vibrations from residual moisture or minor hydroxylated components. The strong band at 2962 cm⁻¹ corresponds to aliphatic C–H stretching of –CH₃ and –CH₂ groups, confirming the presence of long hydrocarbon chains that later serve as the hydrophobic tail of the surfactant. The band observed at 2259 cm⁻¹ is likely due to combination or overtone vibrations and

does not contribute directly to surfactant activity. The peak at 1160 cm^{-1} is assigned to C–O stretching vibrations of ester groups, characteristic of triglycerides in the oil, while the band at $\sim 870\text{ cm}^{-1}$ corresponds to C–H out-of-plane bending vibrations of alkyl chains.

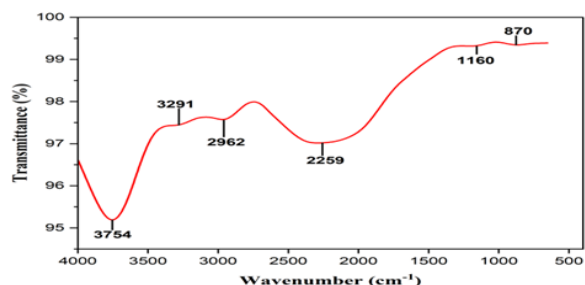


Figure 1. FTIR Spectrum of Neem Seeds

These findings are consistent with literature reports; Agu et al. (2025) identified 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), confirming the presence of esters, carboxylic acids, and nitrogen-containing groups in neem oil. Similarly, Ismaila et al. (2022) reported bands at $2851\text{--}2920\text{ cm}^{-1}$ corresponding to symmetric and asymmetric C–H stretching and at 1743 cm^{-1} assigned to C=O stretching of triglycerides. Collectively, these results affirm FTIR spectroscopy as a reliable technique for verifying the chemical integrity of neem seed oil prior to its conversion into surfactants and other industrially relevant products.

3.2 Physicochemical Analysis of Neem Seed Oil Sample

Physicochemical analysis was conducted to assess the physical and chemical properties of neem seed oil, with a focus on its potential for surfactant production. The results, presented in Table 2, provide insight into key parameters relevant to its suitability for use in green surfactant synthesis.

Table 2: Physicochemical result of neem seed oil sample

Parameter	This Study	Sani & Mohammed (2024)	Obasi et al. (2023)	Okoro et al. (2023)
Acid Value (mg KOH/g)	1.88	1.70	1.65	1.92
Saponification Value (mg KOH/g)	184.80	190.30	189.50	180.75
Free Fatty Acid (mg KOH/g)	0.94	—	—	—
Moisture content (%)	2.60	—	—	—
Oil Yield (%)	23.00	—	24.50	21.80
Viscosity (mPa·s)	32.45	35.20	33.60	31.85
Surface Tension (mN/m)	31.55	—	30.80	32.00

Parameter	This Study	Sani & Mohammed (2024)	Obasi et al. (2023)	Okoro et al. (2023)
Colour	Deep yellow	—	—	—
Weight (10ml) g	9.04	—	—	—

The physicochemical properties of neem seed oil, as presented in Table 2, indicate its strong potential for use in surfactant synthesis and other industrial applications. The acid value recorded in this study (1.88 mg KOH/g) closely aligns with values reported by Sani and Mohammed (2024) at 1.70 , Obasi et al. (2023) at 1.65 , and Okoro et al. (2023) at 1.92 . A low acid value implies a low free fatty acid (FFA) content, which is advantageous for surfactant synthesis by minimizing undesirable side reactions during chemical processing. The measured FFA value of 0.94 mg KOH/g further supports the oil's quality, although similar data were not provided in the compared studies. The saponification value of 184.80 mg KOH/g falls within the range reported by Obasi (189.50 mg KOH/g) and Sani and Mohammed (2024) (190.30 mg KOH/g), and is slightly higher than that of Okoro (180.75 mg KOH/g). This value reflects the presence of saponifiable lipids, essential for converting triglycerides into surfactants.

The oil yield obtained (23.00%) is consistent with values reported by Obasi (24.50%) and Okoro (21.80%), with variations attributable to factors such as seed variety, maturity, and extraction methodology. Okoro's slightly lower yield may result from differences in drying methods or geographical origin of the seeds. Viscosity was measured at $32.45\text{ mPa}\cdot\text{s}$, closely comparable to the values of Obasi et al. (2023) ($33.60\text{ mPa}\cdot\text{s}$), Okoro ($31.85\text{ mPa}\cdot\text{s}$), and Sani and Mohammed (2024) ($35.20\text{ mPa}\cdot\text{s}$). This moderate viscosity is suitable for enhanced oil recovery (EOR) applications, as it ensures favorable flow characteristics during injection processes. The surface tension of neem oil in this study was 31.55 mN/m , within the range of 30.80 mN/m and 32.00 mN/m reported by Obasi and Okoro, respectively. This property is particularly important in evaluating the oil's capacity to reduce interfacial tension - a critical factor in surfactant performance.

Other physical attributes such as the deep yellow color and the weight of 9.04 g per 10 mL were not available in comparative literature but are in line with typical neem oil profiles, suggesting effective extraction and minimal impurities. However, the results from this study are in good agreement with existing literature, with minor deviations attributed to environmental factors, processing conditions, and seed variability. The observed low acid and FFA values, combined with favorable saponification, viscosity, and surface tension, confirm neem seed oil's suitability as a

sustainable and effective feedstock for bio-based surfactant production.

3.3 Characterization of Anionic Surfactant from Neem Seed Oil

The synthesized anionic surfactant from neem seed oil appeared pale yellow and weighed 15.15 g. Functional group analysis was conducted using Fourier Transform Infrared Spectroscopy (FTIR), as shown in Figure 2 and Table 3. The FTIR results confirmed the presence of characteristic functional groups associated with anionic surfactants, validating the success of the chemical modification process.

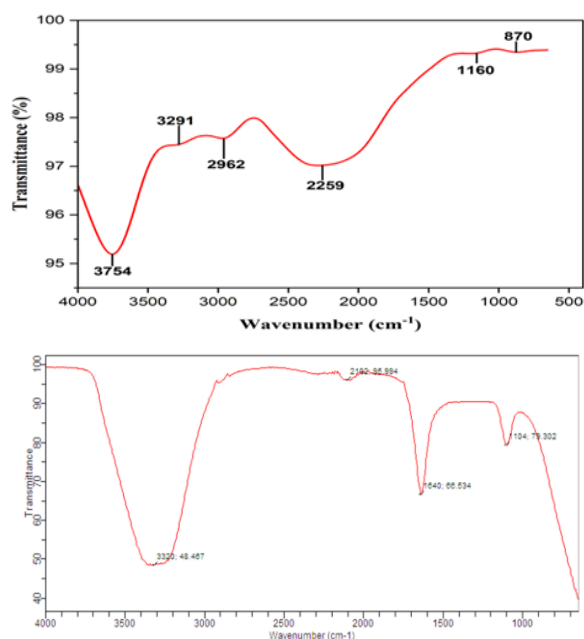


Figure 2: FTIR Peak of Anionic surfactant from neem oil (ASNO) and FTIR Spectrum of Neem Seeds

Table 3: Peak Interpretation of Anionic Surfactant from Neem Oil (ASNO)

Functional Group	Vibration Type	This Study Wavenumber (cm ⁻¹)	Obasi et al. (2023)	Farias et al. (2021)
O–H	Stretching	3290	~3285 (O–H) broad	~3300 (O–H) broad
C=C (Alkene)	Stretching	1640	1644 (C=C/C=O overlap)	1638 (C=C)
S=O (Sulfonate)	Stretching	1104	1100 (S=O)	1108–1110 (SO ₃ ⁻ , S=O)

The FTIR spectrum of the synthesized anionic surfactant (Figure 2 and Table 3) reveals characteristic transmittance bands confirming the successful sulfonation of neem seed oil. The broad and intense band observed in the ~3336–3290 cm⁻¹ region is assigned to O–H stretching vibrations associated with

alcohol or phenolic groups and increased hydrogen bonding, indicating enhanced hydrophilicity of the product. These polar functional groups contribute to the amphiphilic nature of the surfactant, which is essential for emulsification and interfacial activity. Similar O–H stretching bands have been reported for neem-based surfactants by Obasi et al. (2023) and Farias et al. (2021).

The band at ~1640 cm⁻¹ is attributed to C=C stretching vibrations, suggesting partial retention of unsaturated bonds from the parent triglyceride structure, consistent with reports that sulfonation does not fully saturate the carbon chains. Notably, new bands appearing in the ~1640–1550 cm⁻¹ region are associated with asymmetric stretching vibrations of –SO₃⁻ / –OSO₃⁻ groups, confirming the introduction of the anionic head group. The strong transmittance band observed at ~1104–1047 cm⁻¹ corresponds to S=O/S–O stretching vibrations and serves as a diagnostic marker for sulfonate or sulfate functionalities, directly confirming anionic surfactant formation. This sulfonate-specific peak is absent in the nonionic surfactant spectrum, demonstrating the effectiveness of the chemical modification. Comparable sulfonate bands at 1170 and 1068 cm⁻¹ were reported by Sampora et al. (2017) during the sulfonation of polyethylene glycol oleate.

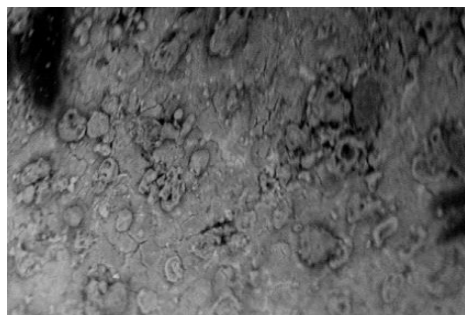
The retention of aliphatic C–H stretching bands further indicates that the long hydrocarbon chains of the oil remain intact, preserving the hydrophobic tail necessary for surface activity. The coexistence of hydrophilic (O–H, S=O) and hydrophobic (C=C, C–H) functional groups confirms the successful conversion of neem seed oil into a functional anionic surfactant, highlighting its potential as a sustainable alternative for enhanced oil recovery, detergent formulations, and emulsification applications. Figures 1 and 2 clearly demonstrate the structural transformation of neem seed oil into an anionic surfactant. Overall, the appearance of sulfonate functional groups alongside intact alkyl chains confirms the effective conversion of neem oil into a functional anionic surfactant.

3.4 SEM of Anionic Surfactant from Neem Seed Oil

Scanning Electron Microscopy (SEM) was employed to examine the surface morphology of the synthesized anionic surfactant, as shown in Figure 3. The analysis provided detailed insights into the structural features and surface texture relevant to its performance in surfactant applications.

The SEM micrographs of the synthesized anionic surfactant, captured at magnifications of 500x, 1000x, and 1500x, reveal a complex granular morphology

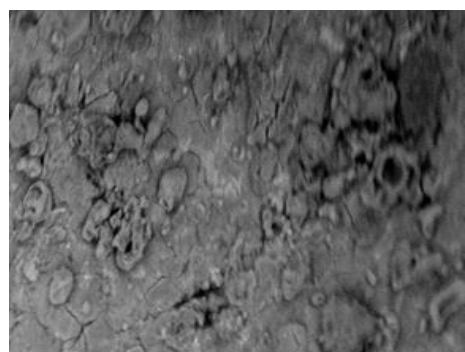
with vesicle-like structures distributed across the surface. These spherical and semi-spherical aggregates suggest the formation of self-assembled micellar or vesicular structures, a common trait of amphiphilic compounds such as sulfonated surfactants.



(a)



(b)



(c)

Figure 3: SEM images of anionic surfactant magnification at (a) 500x (b) 1000x (c) 1500x

The presence of these formations can be attributed to successful sulfonation, which introduces sulfonate groups that enhance hydrophilicity and drive micellization behaviour.

Such micellar structures are critical in applications like enhanced oil recovery (EOR), where they contribute to interfacial tension (IFT) reduction, emulsion stability, and wettability alteration the key mechanisms for mobilizing trapped oil in porous

media. These morphological findings are consistent with Farias et al. (2021), who observed similarly granular and vesicle-like surfaces in SEM analysis of biosurfactants derived from castor oil. Their study highlighted the role of these structures in improving dispersion and emulsification capacity, especially under high salinity and temperature conditions, typical of reservoir environments. Likewise, Adebayo et al. (2023) reported micelle-dense microstructures in sulfonated jatropha oil surfactants. Their SEM analysis confirmed that these surface features correlated strongly with low surface tension values and improved oil displacement efficiency during core flooding tests. In line with these studies, the granular and vesicular morphology observed in the present work underscores the effective molecular organization of the neem-derived anionic surfactant, reinforcing its potential as a green and functional alternative to synthetic surfactants in oilfield and industrial applications. The structural integrity and amphiphilic behavior inferred from SEM analysis complement the FTIR findings and physicochemical data, collectively validating the surfactant's suitability for EOR deployment.

4 Conclusion

This study successfully demonstrated the synthesis and characterization of an anionic surfactant derived from neem seed (*Azadirachta indica*), offering a green and sustainable alternative to conventional synthetic surfactants for enhanced oil recovery (EOR) and other industrial applications. The proximate analysis confirmed the presence of saponifiable lipids, while physicochemical properties such as low acid value, moderate viscosity, and favorable saponification value further established the oil's suitability for surfactant synthesis.

The chemical transformation of neem oil into an anionic surfactant was validated using FTIR spectroscopy, which identified key functional groups including sulfonate (S=O), hydroxyl (O-H), and alkene (C=C), confirming successful sulfonation. SEM imaging revealed a granular surface with vesicle-like structures typical of amphiphilic molecules, supporting the surfactant's micellization potential essential for reducing interfacial tension, emulsification, and wettability alteration in EOR operations. When compared with reported studies, the neem-based surfactant exhibited comparable structural and functional characteristics, emphasizing its viability as a low-cost, biodegradable alternative. Given its abundant availability and minimal competition with food resources, neem seed oil presents a promising bio-resource for environmentally friendly surfactant development. The outcomes of this research provide a foundation for future scaling and

application of neem-derived surfactants in petroleum and allied industries.

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