

Performance of Lignosulfonate Derived from Coffee Husk as A Natural Emulsifier in Enhanced Oil Recovery: A Phase Behavior Study

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ABSTRACT - The oil industry faces challenges in enhancing oil recovery while reducing environmental impact. This study investigates the utilization of lignin extracted from coffee husk, an agricultural waste, as a natural emulsifier in Enhanced Oil Recovery (EOR). The research identifies lignin's functional groups via Fourier Transform Infrared (FTIR) spectroscopy before and after sulfonation, determining compatible lignosulfonate concentrations through aqueous stability tests, and optimizing salinity for effective emulsification. Lignin was extracted via soda pulping and modified through sulfonation with sodium bisulfite (NaHSO_3) to produce lignosulfonate. FTIR analysis confirmed successful sulfonation, evidenced by new peaks at 636 cm^{-1} (S–O) and 1101 cm^{-1} (SO_3^-). Aqueous stability tests at $60\text{ }^\circ\text{C}$ demonstrated that a 0.8% (w/v) lignosulfonate concentration remained stable in brine with 20,000 ppm salinity. Salinity scans revealed optimal conditions at 25,000 ppm, yielding solubilization ratios of $V_o/V_s = 65.97$ and $V_w/V_s = 69.44$, with a balanced $(V_o/V_s)/(V_w/V_s)$ ratio of 0.95, indicative of low interfacial tension (IFT). These findings highlight the potential of sulfonated coffee husk lignin as a sustainable emulsifier for EOR applications. Optimal emulsion stability was achieved at a concentration of 0.8% and a salinity of 25,000 ppm. This study supports circular economy principles by valorizing agricultural waste and providing a promising alternative to synthetic chemicals in the oil industry.

Keywords: lignin, coffee husk, natural emulsifier, enhanced oil recovery, sulfonation.

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INTRODUCTION

The global energy demand continues to rely heavily on petroleum; however, production from mature oilfields is steadily declining. To address this challenge, the oil and gas industry employs

Enhanced Oil Recovery (EOR) methods, which can recover an additional 20–40% of the original oil in place once primary and secondary recovery stages become ineffective (Putra & Kiono 2021). Among various EOR techniques, chemical injection

is widely applied, often involving surfactants or emulsifiers to reduce interfacial tension (IFT) and stabilize the oil–water interface within the reservoir (Seker et al., 2020). Nevertheless, many conventional emulsifiers are synthetic and pose environmental risks due to their poor biodegradability (Busu et al., 2019). This concern has stimulated growing interest in developing sustainable, bio-based alternatives derived from renewable resources. Lignin, a complex organic polymer abundant in plant cell walls, has emerged as a promising candidate (Czaikoski et al., 2020).

In Indonesia, one of the world's largest coffee producers, the processing of coffee beans generates a substantial amount of coffee husk waste, representing 55–60% of the total bean weight. This agricultural by-product remains underutilized, despite being a potential lignin source (Ibnu & Rosanti 2022; Romadhona et al., 2022). Previous studies have demonstrated the potential of lignosulfonates derived from other biomass feedstocks, such as oil palm empty fruit bunches and sugarcane bagasse, as effective surfactants for EOR applications (Aziz et al., 2017; Setiati et al., 2020). This research direction aligns with broader efforts in Indonesia to develop sustainable EOR chemicals from abundant local feedstocks, including significant progress in synthesizing high-performance surfactants from palm oil (Eni et al., 2018). These studies successfully produced surfactants capable of achieving low IFT, a critical requirement for mobilizing trapped oil.

Despite these advancements, a specific research gap remains concerning the utilization of lignin from coffee husk. To the best of our knowledge, this study represents the first investigation into lignin extraction from coffee husk, its subsequent modification into an emulsifier for EOR, and the evaluation of its performance through phase behavior analysis. Accordingly, this study aims to explore the potential of coffee husk-derived lignin as a novel natural emulsifier for EOR applications, focusing on its formulation and stability under reservoir-relevant conditions.

A multi-step approach was employed to achieve this objective. First, lignin was extracted from coffee husk using the soda pulping method, chosen for its cost-effectiveness, high yield, and proven efficiency in processing non-wood

biomass (Bajpai 2018; Cheremisinoff & Rosenfeld 2010). Subsequently, the extracted soda lignin was chemically modified via sulfonation to produce sodium lignosulfonate. This modification introduced hydrophilic sulfonate groups into the lignin structure, enhancing its amphiphilic character and solubility in water key properties for an effective emulsifier (Dilling 1984). The success of the extraction and sulfonation processes was verified through Fourier Transform Infrared (FTIR) spectroscopy, which enabled characterization of the functional groups in the resulting products (Gargulak et al., 2015). Finally, the performance of the lignosulfonate emulsifier was systematically evaluated through phase behavior tests, including aqueous stability assessments and salinity scans, to determine its compatibility and optimal formulation for generating stable microemulsions.

This study is expected to yield several key outcomes: 1). Identification of the specific functional groups in coffee husk-derived lignosulfonate that contribute to emulsion stability; 2). Determination of its maximum compatible concentration in brine representative of Indonesian oilfields, and; 3). Establishment of the optimal salinity required to achieve a stable and effective microemulsion system. The findings aim to demonstrate the feasibility of coffee husk lignin as an eco-friendly emulsifier for the oil industry, thereby supporting the principles of the circular economy through agricultural waste valorization and reducing reliance on synthetic chemicals in EOR operations.

METHODOLOGY

This section outlines the experimental approach used to extract, modify, and evaluate lignin from coffee husk as a potential emulsifier for EOR. The methodology consists of four main stages: lignin extraction via soda pulping, chemical modification through sulfonation, structural characterization using FTIR, and performance evaluation through phase behavior tests. Figure 1 illustrates the overall research workflow.

Materials

The primary raw material was locally sourced coffee husk. Crude oil with an American petroleum institute gravity of 42.2° and a viscosity of 4.8 cP was used in the emulsification tests. The chemicals

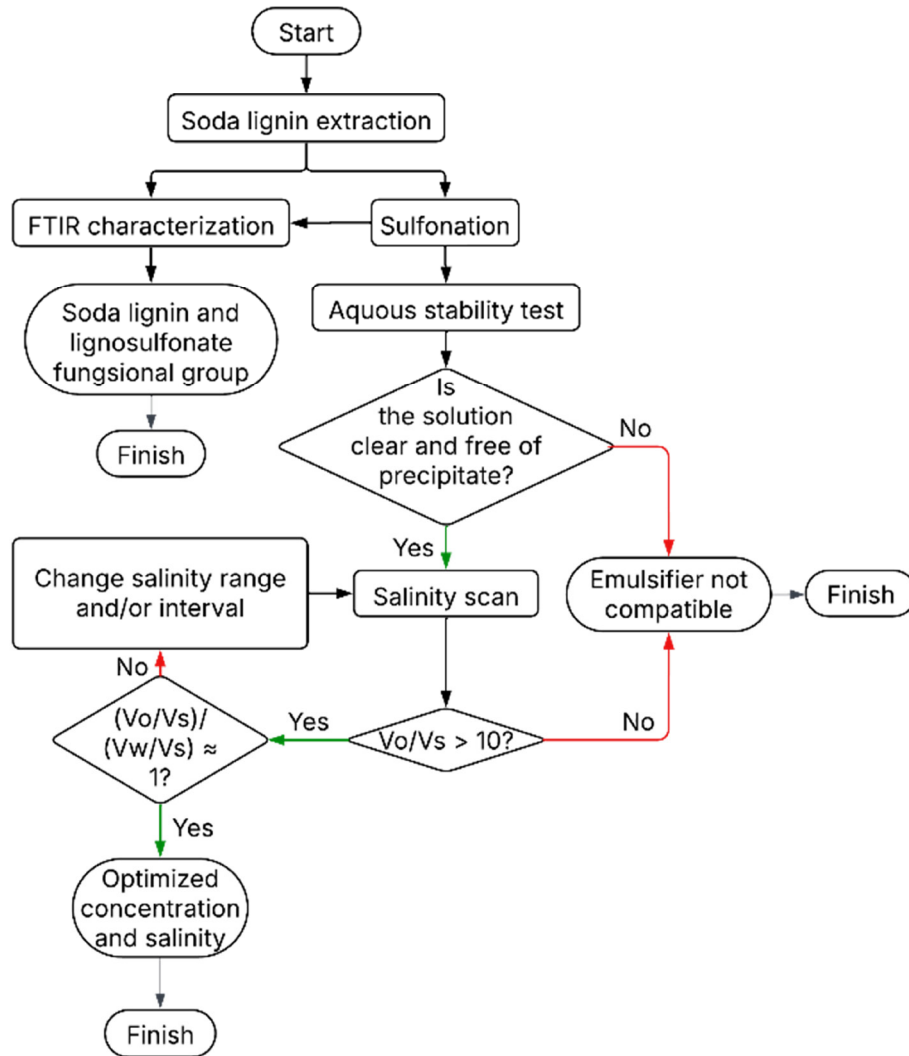


Figure 1. Research workflow

included sodium hydroxide (NaOH, 1 M and 30% w/v), sulfuric acid (H₂SO₄, 20% v/v), sodium bisulfite (NaHSO₃, 0.1 M), and aquadest. Key laboratory equipment included a magnetic stirrer with hot plate, an electric oven, a centrifuge, a pH meter, and an FTIR spectrometer.

Lignin extraction via soda pulping

The soda pulping method was chosen for lignin extraction due to its cost-effectiveness and efficiency in processing non-wood biomass, yielding lignin with high purity. The procedure began by grinding and drying the coffee husks at 50 °C. The dried husks were subjected to delignification in a 30% (w/v) NaOH solution at a solid-to-liquid ratio of 1:8 (w/v). The mixture was heated to 70 °C and continuously stirred for 3 hours, producing a high-alkali black liquor (pH ≈ 12.8) containing dissolved lignin.

Following the principles of acid precipitation from black liquor as described by Bajpai (2018), the lignin was recovered by acidifying the filtered black liquor with 20% (v/v) H₂SO₄ until the pH reached 2. The resulting precipitate, soda lignin, was separated by centrifugation at 3,500 rpm for 10 minutes, washed with acidified deionized water to remove impurities, and dried in an oven at 70 °C to obtain a purified solid powder.

Lignin Modification via Sulfonation

The extracted soda lignin was chemically modified to enhance its functionality as an emulsifier. Soda lignin is naturally insoluble in water at neutral pH; therefore, sulfonation was performed to introduce hydrophilic sulfonate groups (–SO₃[–]), rendering the molecule amphiphilic and water-soluble (Dilling 1984; Ruwoldt et al., 2022). The

sulfonation reaction was conducted by mixing 5 g of soda lignin with 150 mL of 0.1 M sodium bisulfite (NaHSO_3) solution. The mixture was refluxed at 100 °C for 5 hours with continuous stirring. These conditions are consistent with previously established methods for liginosulfonate synthesis (Aziz et al., 2017; Setiati et al., 2020). The final solution was filtered and dried at 60 °C to yield the solid sodium liginosulfonate product.

Structural characterization

FTIR spectroscopy was used to identify key functional groups in the soda lignin and to confirm the successful incorporation of sulfonate groups after modification (Gargulak et al., 2015). Samples were prepared as KBr pellets with a sample-to-KBr mass ratio of 1:10. Spectra were recorded over a frequency range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1} .

Emulsifier performance evaluation: phase behavior tests

The effectiveness of the synthesized liginosulfonate as an emulsifier was evaluated using a

standard two-step phase behavior screening protocol (Sheng, 2011), consistent with recent assessments of green surfactants in the Indonesian context (Maulida et al., 2024). The objective was to identify a stable formulation and determine the optimal salinity to minimize the interfacial tension between oil and water.

Aqueous stability test

This preliminary test was conducted to determine the maximum concentration of liginosulfonate that remained completely soluble in brine, ensuring that the injected fluid existed as a single, stable phase. Liginosulfonate solutions with concentrations of 0.2%, 0.4%, 0.6%, 0.8%, and 1% (w/v) were prepared in synthetic brine with 20,000 ppm salinity, representative of average formation water in Indonesia (Massie et al., 2021). The samples were heated to 60 °C and visually monitored for clarity and precipitation over three days.

Salinity scan

The salinity scan was conducted to identify the optimal salinity for forming a stable Winsor type III microemulsion, indicative of ultra-low IFT. The

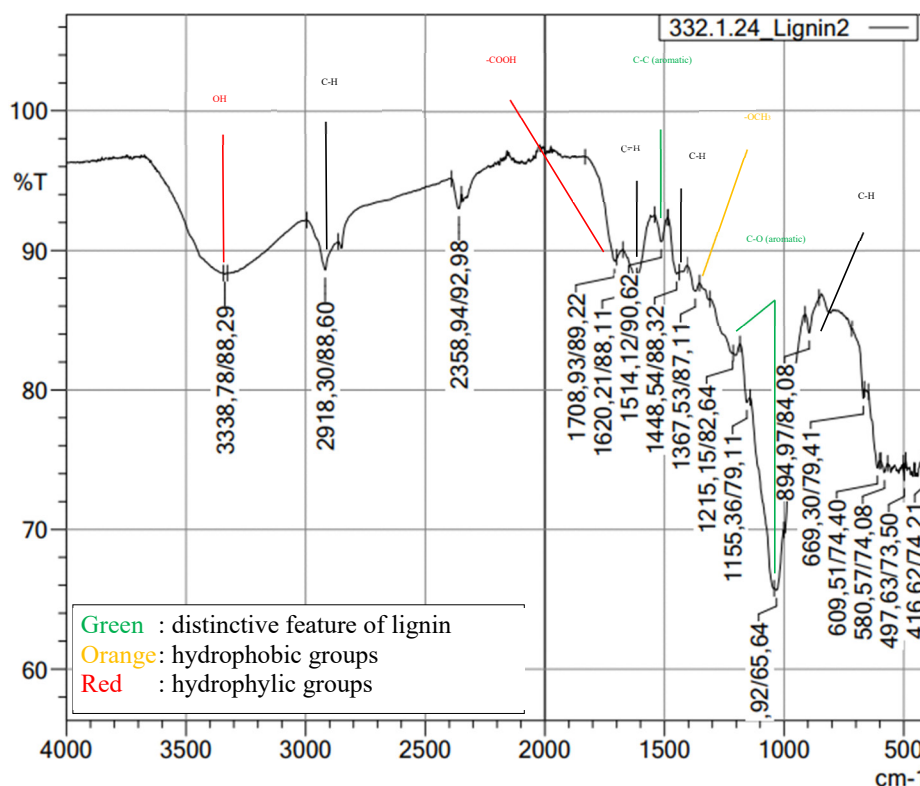


Figure 2. FTIR spectrum of soda lignin extracted from coffee husk, confirming the presence of characteristic lignin functional groups, including hydroxyl ($-\text{OH}$), aromatic ($\text{C}-\text{C}$), methoxy ($-\text{OCH}_3$), and ether ($\text{C}-\text{O}$) groups.

highest stable lignosulfonate concentration identified from the aqueous stability test was mixed with crude oil at a water-to-oil ratio (WOR) 1. The salinity of the aqueous phase was varied from 5,000 to 30,000 ppm. After vigorous mixing, the samples were equilibrated at 60 °C for three days. The volumes of excess oil, excess water, and the microemulsion phases were measured to calculate the oil (Vo/Vs) and water (Vw/Vs) solubilization ratios, key parameters in determining formulation efficiency.

RESULT AND DISCUSSION

Structural analysis via FTIR spectroscopy

FTIR analysis confirmed the successful extraction of lignin from coffee husk and the subsequent incorporation of sulfonate groups through chemical modification. The spectrum for the extracted soda lignin is presented in Figure 2. A broad absorption band at 3338 cm^{-1} , characteristic of O–H stretching vibrations in phenolic and aliphatic hydroxyl groups, is evident and fundamental to lignin's structure (Sarkanen 1971). The peak at 1514 cm^{-1} corresponds to C–C stretching of the aromatic ring, a distinctive feature of lignin that differentiates it from cellulose and hemicellulose. Additionally, peaks at 1368 cm^{-1} (–OCH₃ methoxy groups) and 1215 cm^{-1} (C–O vibrations in the aromatic ring) confirm the presence of hydrophobic structures essential for emulsification (Hon & Shiraishi 2001). The presence of these characteristic peaks verifies that the soda pulping method successfully isolated lignin from coffee husk biomass. These findings are consistent with FTIR results for soda lignin extracted from coffee husk and other non-wood biomass reported in previous studies (Oliviera et al., 2018; Sekeri et al., 2020).

Following sulfonation, the FTIR spectrum of the resulting lignosulfonate (Figure 3) shows distinct changes, providing strong evidence of successful chemical modification. While most of the original lignin peaks are retained, two new absorption peaks appear: one at 1101 cm^{-1} , attributed to symmetric stretching of the sulfonate group (SO₃[–]), and another at 636 cm^{-1} , corresponding to S–O bond vibration. The appearance of these peaks, absent in the original soda lignin spectrum, confirms the successful attachment of hydrophilic sulfonate groups to the lignin backbone. This modification is critical, as it imparts amphiphilic properties to the molecule,

enabling it to function as an effective emulsifier (Aziz et al., 2017; Coates 2000).

Emulsifier performance evaluation

Aqueous stability test

The first step in evaluating lignosulfonate performance was to determine its solubility and stability in brine. This is a critical screening test, as an EOR surfactant must remain in a single, clear phase upon injection to avoid pore plugging. The test was conducted at 60 °C in brine with 20,000 ppm salinity, representing an assumed average for formation water in Indonesia (Massie et al., 2021). The results are summarized in Table 1.

After three days, the lignosulfonate solutions remained clear and free of precipitate at concentrations up to 0.8% (w/v). A visible precipitate formed at 1.0% (w/v), indicating that the solubility limit had been exceeded. Interestingly, some undissolved lignosulfonate was initially present for the 0.6% and 0.8% concentrations; however, after one day in the oven at 60 °C, these solids fully dissolved. This phenomenon occurs because the solubility of substances such as lignosulfonate increases with sustained temperature, allowing the system more time and energy to break down solute particles and reach equilibrium (Astuti et al., 2022). Thus, the maximum compatible concentration for this

Table 1. Aqueous stability test results for lignosulfonate in 20,000 ppm brine at 60 °C

Concentration (%)	Day 1	Day 2	Day 3
0.2	Clear	Clear	Clear
0.4	Clear	Clear	Clear
0.6	Undissolved	Clear	Clear
0.8	Undissolved	Clear	Clear
1.0	Undissolved	Undissolved	Undissolved

emulsifier is 0.8% (w/v), which was subsequently used for all salinity scan tests.

Salinity scan

The salinity scan was conducted to identify the optimal salinity at which the lignosulfonate emulsifier forms the most stable microemulsion, indicated by balanced solubilization of oil and water. An initial wide-range scan revealed the formation of Winsor type III microemulsions at salinities of 15,000 ppm and above, confirming the emulsifier's ability to lower interfacial tension (IFT). However,

this scan produced some anomalies and did not yield a clear optimum, prompting a second, more focused scan.

The results of the second salinity scan (Fig. 4) show a clear trend consistent with surfactant theory (Green & Willhite, 2018). As salinity increases, the oil solubilization ratio (V_o/V_s) rises, while the water solubilization ratio (V_w/V_s) decreases. The optimal salinity is located at the crossover point where the two ratios are nearly equal. For this system, the optimal condition was identified at 25,000 ppm salinity. At this point, the V_o/V_s was 65.97 and the V_w/V_s was 69.44, yielding a balanced ratio of $(V_o/V_s)/(V_w/V_s) = 0.95$. A balanced ratio close to 1 is highly desirable, as it signifies the formation of an optimal Winsor type III microemulsion (Figure 5), corresponding to the lowest interfacial tension (Sheng, 2011). Moreover, the V_o/V_s value of 65.97 exceeds the minimum requirement of 10 for an effective EOR surfactant, confirming the high performance of the formulation.

An additional observation was the potential for two optimal salinity points, attributable to the inherent polydispersity of lignin. Lignosulfonate is a heterogeneous mixture of polymers with varying molecular weights and degrees of sulfonation. Different molecular fractions may perform optimally at various salinities, resulting in multiple performance peaks (Braaten et al., 2003; Ferraro et al., 2024). Although a convergence was noted near 12,000 ppm, performance at 25,000 ppm was clearly superior, yielding a balanced, high-solubilization state.

This optimal salinity of 25,000 ppm differs from the 10,000 ppm reported for bagasse-derived lignosulfonate (Setiati et al., 2020). The difference is attributed to the unique chemical structure of coffee husk lignin and the specific properties of the oil and brine used in this study. Importantly, this highlights the principle that different bio-surfactants exhibit optimal performance under varying reservoir conditions. For instance, recent studies on Methyl

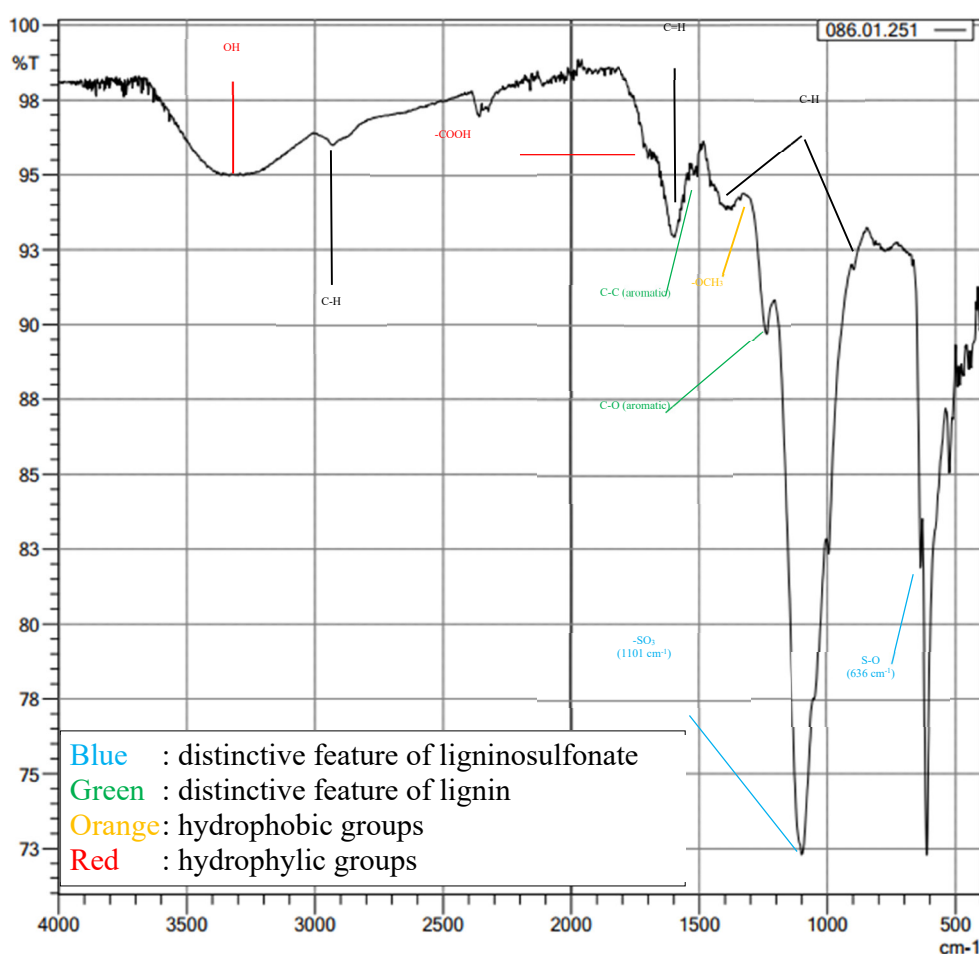


Figure 3. FTIR spectrum of sulfonated lignin (lignosulfonate), showing new peaks at 1101 cm^{-1} ($-\text{SO}_3^-$) and 636 cm^{-1} (S-O), which confirm the successful sulfonation of the lignin structure.

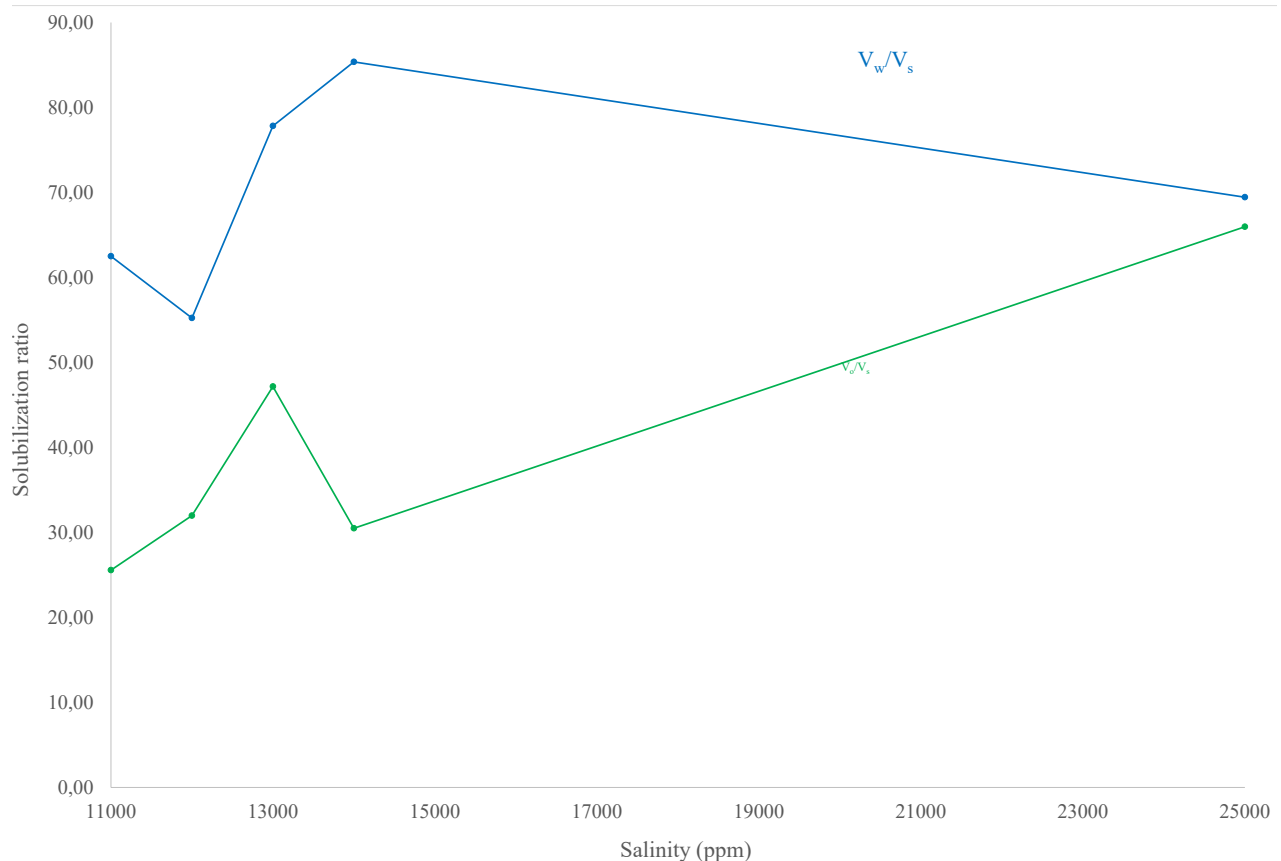


Figure 4. Solubilization ratio as a function of salinity for the 0.8% lignosulfonate emulsifier system at 60 °C. The crossover of the oil solubilization (V_o/V_s) and water solubilization (V_w/V_s) curves at ~25,000 ppm indicates the optimal salinity for microemulsion formation.

Ester Sulfonate (MES) have reported excellent oil recovery factors at higher salinities of 30,000–40,000 ppm and elevated temperatures (Al Fikri et al., 2025). Such variation underscores that the effectiveness of an EOR surfactant is highly specific to its molecular structure and the target reservoir environment, positioning coffee husk-derived lignosulfonate as a tailored solution for moderate- to high-salinity fields.

Implications of findings

The results of this study successfully achieve the research objectives.

Figure 5. Photograph of the Winsor type III microemulsion formed at the optimal salinity of 25,000 ppm after three days of equilibration. The distinct middle phase (ME) between the excess oil and water phases is clearly visible.

Structural analysis confirmed the extraction of lignin and its modification into functional lignosulfonate. Performance testing established an optimal formulation: a 0.8% (w/v) lignosulfonate emulsifier in 25,000 ppm salinity brine. This formulation produced a stable, high-performance

Winsor type III microemulsion, indicating strong potential for mobilizing residual oil in EOR operations. These findings carry significant implications. They demonstrate that coffee husk, a widely available and low-cost agricultural residue, can be effectively valorized into a value-added chemical for enhanced oil recovery (EOR). This research contributes to the principles of circular economy and green chemistry by offering a sustainable, bio-based alternative to synthetic surfactants. The successful development of this emulsifier provides the oil and gas industry with a pathway to reduce its environmental footprint without compromising technical performance, directly addressing the challenges outlined in the introduction.

CONCLUSION

This study demonstrates the potential of coffee husk lignin as a sustainable emulsifier for enhanced oil recovery (EOR). First, the research successfully validated the chemical pathway for converting raw coffee husk biomass into a functional, amphiphilic

lignosulfonate. Structural analysis via FTIR confirmed that the soda pulping method effectively extracted lignin and that the subsequent sulfonation process successfully incorporated hydrophilic sulfonate groups into the lignin backbone. These findings establish coffee husk as a viable feedstock for producing functional surfactants for EOR applications. Second, a critical operational parameter for field application was identified. The maximum compatible concentration of the emulsifier was determined to be 0.8% (w/v), at which the synthesized lignosulfonate remains fully soluble and stable in brine with 20,000 ppm salinity at a reservoir-relevant temperature of 60 °C. This parameter provides an essential guideline for designing injection fluids to prevent surfactant precipitation and potential formation damage. Finally, the optimal conditions for high-performance emulsification were determined. At 25,000 ppm salinity, the lignosulfonate emulsifier formed a stable Winsor type III microemulsion, characterized by a high oil solubilization ratio and a nearly balanced solubilization of oil and water. This state corresponds to the ultra-low interfacial tension required for mobilizing residual oil. These findings demonstrate that coffee husk-derived lignosulfonate can create the ideal physicochemical environment for EOR, positioning it as a promising, sustainable, high-

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performance alternative to conventional synthetic surfactants.

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GLOSSARY OF TERMS

Symbol	Definition	Unit
API	American Petroleum Institute	
EOR	Enhanced oil recovery	
FTIR	Fourier Transform Infrared spectroscopy	
IFT	Interfacial tension	
ME	Microemulsion	
MES	Methyl ester sulfonate	
NaHSO ₃	Sodium bisulfite	
NaOH	Sodium hydroxide	
O/W	Oil-in-water emulsion	
OOIP	Original oil in place	
ppm	Parts per million	

RPM	Rotations per minute
SLS	Sodium lignosulfonate
σ_{om}	Interfacial tension between microemulsion and oil
σ_{wm}	Interfacial tension between microemulsion and water
v/v	Volume-to-volume ratio
V _o /V _s	Oil solubilization ratio
V _w /V _s	Water solubilization ratio
W/O	Water-in-oil emulsion
w/v	Weight-to-volume ratio
WOR	Water-to-oil ratio

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