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Synthesis and Characterization of Green Surfactants from Selected Underutilized Local Plant Seeds: A Sustainable Alternative for Oilfield Applications

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ABSTRACT

This research looks into the potential of green surfactants made from underutilized local plant seeds. Delonix regia, Jatropha curcas, and Hura crepitans plants are recognised for their high oil content, with seeds containing as much as 150-200 (mg KOH/g fat) saponification. These plants are a prospective resource for surfactant manufacturing since they are sustainable, environmentally friendly, and economically viable, particularly in areas where they are widely farmed. The study focusses on the synthesis of green surfactants using the esterification, transesterification, and sulfonation processes. Gas chromatography mass spectroscopy (GCMS) and Fourier transform infrared spectroscopy (FTIR) were utilised to validate the synthesis method and compare the results to highly sought-after commercial surfactants. GCMS analyses revealed the presence of medium to long chain fatty acid methyl esters, while DR-MES revealed the presence of Octanoic acid, methyl ester; Decanoic acid, methyl ester; Dodecanoic acid, methyl ester; Hexadecanoic acid, methyl ester; Eicosanoic acid, methyl ester; Heneicosanoic acid, methyl ester; Docosanoic acid, methyl ester; Tricosanoic acid, methyl ester. JC-MES contains methyl esters of octanoic acid, nonanoic acid, and hexadecanoic acid. 9-Octadecynoic acid, methyl ester; Eicosanoic acid, methyl ester; Docosanoic acid, methyl ester; HC-MES reveals the presence of Dodecanoic acid, methyl ester; Hexadecanoic acid, methyl ester; Octadecadienoic acid, methyl ester; Nonadecenoic acid, methyl ester; Octadecatrienoic acid, methyl ester; Eicosanoic acid, methyl ester; Docosanoic acid, methyl ester. Along with verified functional groups, the presence of these fatty acid methyl esters with medium chain carbon compounds (C10–C18) is a clear sign of the presence of surface-active components that have a great ability to lower surface tension, form micelles, and enhance the surfactants' overall biodegradability.

Keywords: *Delonix regia, Jatropha curcas, Hura crepitans, Green-Surfactants, GCMS, FTIR*

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INTRODUCTION

The growing global demand for oilfield chemicals, particularly surfactants, has raised significant environmental and economic concerns due to the widespread use of synthetic, petroleum-derived surfactants. These surfactants, while effective, pose

toxicity risks to aquatic ecosystems, contribute to bioaccumulation, and are often resistant to biodegradation (Rebello *et al.*, 2014; Verma *et al.*, 2023). In this context, green surfactants derived from renewable bioresources are emerging as viable alternatives with enhanced

environmental compatibility. Recent studies emphasize the importance of biosurfactants not only in reducing ecological footprints but also in aligning with circular economy and sustainable development goals (Nagtode *et al.*, 2023; Prasannakumar *et al.*, 2025). The global surfactant market is projected to exceed USD 60 billion by 2030, with a substantial share attributed to oil and gas applications (Nagtode *et al.*, 2023). Oilfield operations require surfactants for enhanced oil recovery (EOR), drilling mud stabilization, and corrosion inhibition. However, the persistent use of petroleum-based surfactants exacerbates environmental pollution (Verma *et al.*, 2023; Pengqi *et al.*, 2025). Several green alternatives have been proposed, including methyl ester sulfonates (MES), sugar-based surfactants, and fatty acid derivatives, often synthesized from food crops (Qadariah *et al.*, 2024). This presents a conflict between fuel and food security. Utilizing non-food, underexploited plant seeds provides a sustainable route for surfactant development without impacting food chains (Adewuyi *et al.*, 2014; Openshaw, 2000; Ulakom *et al.*, 2022).

Plants like *Hura crepitans* (sandbox tree), *Jatropha curcas*, and *Delonix regia* (flame tree) are abundant in various tropical regions but remain underutilized in industrial chemistry. Studies on the fatty acid composition of these seeds reveal high contents of linoleic, oleic, palmitic, and stearic acids—ideal precursors for surfactant synthesis (Adewuyi *et al.*, 2010; Oyedeji *et al.*, 2017; Olaleye & Adubiaro, 2020). Their physicochemical stability, oil yield, and unique bioactive compounds also support their inclusion as sustainable raw materials (Olayinka *et al.*, 2019; Amata & Nwagu, 2013; Ezech *et al.*, 2012).

Seed oils from underutilized plants provide a rich matrix for functional compound extraction. *Hura crepitans*, for instance, yields oil with high unsaturated fatty acid content and oxidative stability (Oderinde *et al.*, 2009; Ulakom *et al.*, 2022; Ominowa *et al.*, 2024). *Jatropha curcas*, extensively reviewed for its bioenergy potential, offers oils with high saponification values and suitable molecular profiles for biosurfactant conversion (King *et al.*, 2009; Meher *et al.*, 2013; Costa *et al.*, 2010). *Delonix regia* oil is rich in linoleic acid, with research validating its antioxidant potential, supporting its chemical viability for surfactant modification (Morsi *et al.*, 2019; Adewuyi *et al.*, 2010; Oyedeji *et al.*, 2017).

These oils can undergo sulfonation, esterification, or ethoxylation to yield anionic and nonionic surfactants. Moreover, their unique fatty acid distributions contribute to desired surface-active properties such as low critical micelle concentration (CMC) and efficient interfacial tension reduction critical for enhanced oil recovery and drilling applications (Nagtode *et al.*, 2023; Verma *et al.*, 2023).

4. Advancing Green Chemistry for Oilfield Applications.
The synthesis of green surfactants involves integrating renewable feedstocks, non-toxic solvents, and energy-efficient processes (Qadariah *et al.*, 2024; Rao *et al.*, 2025). For instance, oil modification via microwave-assisted sulfonation or enzymatic transesterification has

been shown to enhance yield and reduce reaction time (Yaashikaa *et al.*, 2022). FTIR, GC-MS, and 2D-LC have emerged as critical tools in surfactant characterization, aiding in understanding structure-activity relationships (Yang *et al.*, 2022; Gopukumar *et al.*, 2025).

In oilfield chemistry, surfactants must withstand extreme salinity, pH, and temperature conditions. Studies reveal that fatty-acid based green surfactants exhibit promising stability and foaming behavior under such stress (Verma *et al.*, 2023; Prasannakumar *et al.*, 2025). Importantly, surfactants derived from *Delonix regia* and *Hura crepitans* oils demonstrate biodegradability and antimicrobial activity—added advantages in mitigating reservoir souring and biofouling (Olayinka *et al.*, 2019; Brussell, 2004; Khalid *et al.*, 2025). Surfactants are vital chemicals in oilfield operations. Approximately 80% of them are derived from fossil fuels, which contain a high proportion of aromatic compounds (Ismail *et al.*, 2022; Ismail *et al.*, nd; Hayes, 2019). Common precursors such as C12-C18 straight-chain paraffins, α -olefins, benzenoid hydrocarbons, ethylene and propylene oxides are used to synthesize secondary alkanesulfonates, α -olefinsulfonates, linear alkylbenzenesulfonates, EO-PO copolymers, and various products (Canselier *et al.*, 2005). These man-made surfactants, developed through careful product design and engineering, are utilized in the petroleum industry as wetting agents, foaming agents, dispersants, corrosion inhibitors, detergents, and more (Yuan *et al.*, 2014; Behera & Behera, 2022). They play crucial roles across all stages of oil production such as formulation of drilling muds, hydraulic fracturing, acidizing, enhanced oil recovery, corrosion control, oil-water-gas separation, crude oil transportation, oil spill management, and the remediation of contaminated soils. Surfactants also serve as kinetic inhibitors and dispersants for gas hydrates. Furthermore, when used in fuel and lubricant formulations, they enhance the performance and properties of gasoline, diesel, lubricants, and bitumen (Canselier *et al.*, 2005). However, there is a growing need for eco-friendlier surfactant formulations to reduce their environmental impact. Increasing ecological concerns are driving research into renewable raw materials as alternatives to conventional, mineral oil-based surfactants. Given the ongoing demand for both edible and industrial oils, various plant-based oils particularly from locally abundant but underutilized plant species are being studied for their potential in surfactant production. This research explores the synthesis and characterization of green surfactants from underutilized local plant seeds. Among such plants are *Delonix regia*, *Jatropha curcas*, and *Hura crepitans* (Obeta *et al.*, 2025; Oluwasina *et al.*, 2020; Mohammed & Ikiensikimama, 2023; Srivastav, & Karunanithi, 2024; Saborimanesh, & Mulligan, 2019).

METHODOLOGY

Sample collection

Table 1 shows the properties of the seed oils used for

Table 1: Some Properties of Sample Seed Oils Used for Production of Green Surfactants (Obeta *et al.*, 2025).

Oil Properties	Sample Seed Oils		
	(DRO)	(JCO)	(HCO)
Physiochemical Properties			
Colour	Light brown	Amber	Amber
Oil yield (%)	8.4	54.60	66.40
Acid value (mg KOH/g)	22.22	23.24	48.59
Free Fatty Acid (%)	11.11	11.66	24.30
Saponification value (mg KOH/g fat)	117.81	277.85	174.99
Viscosity (mPa.s or cp)	11.8	30.04	11.9
Density (g/cm ³)	0.86	0.89	0.88

Table 2: Materials used for Esterification Process.

Seed Oil	Mass of Oil (g)	25 wt% methanol	1 wt% H ₂ SO ₄
DRO	82.20	20.55	0.8220
HCO	57.61	14.40	0.5761
JCO	141.44	35.36	1.4144

Table 3: Materials used for Trans-esterification Process.

Esterified oil	Mass of oil (g)	23 wt% Methanol	1 wt% NaOH
DRO	84.99	19.55	0.84
HCO	52.42	12.06	0.52
JCO	88.02	20.24	0.88

green surfactant production.

Green surfactant synthesis

Delonix regia, *Hura crepitans*, and *Jatropha curcas* oil samples were used to produce their respective surfactants using a process, which involved three main steps namely esterification, transesterification and sulfonation.

Esterification process

Table 2 shows the specific sample and catalyst volumes used during the esterification process. Here, 82.20 g DRO sample was placed in a 250 ml round-bottom glass flask (reactor) and mixed with 25 wt% methanol, using 1wt% sulfuric acid (H₂SO₄) as a catalyst. The reaction was carried out on a magnetic stirrer with constant heating at 60 °C for 120 minutes. After the reaction, the mixture was allowed to settle for 3 hours, and the methanol-water phase that formed at the top was removed using a separating funnel. The acid value of the product at the bottom was determined using the American Oil Chemists' Society (AOCS) method (2006). The acid results were found to be over 22.0 mg KOH/g oil, and FFA level of 11 %. This procedure was repeated until the free fatty acid (FFA) content was reduced. Reduction of FFA to less than 1% prior to transesterification is necessary to avoid catalyst consumption in soap formation and a low yield of biodiesel (Betiku & Ajala, 2014). The same process was repeated for HCO and JCO samples.



Transesterification process

Fatty acid methyl ester (FAME) was produced by reacting

esterified oil with methanol in the second stage. For this, 84.99 g of esterified DRO was heated to 60 °C in a 250 ml glass reactor, combined with methanol (23 wt%) and NaOH (1 wt%), and stirred at 450 rpm for 60 minutes under atmospheric pressure. The resulting mixture was transferred to a separating funnel, forming two layers: FAME and glycerol (Table 3, Figure 1). The same procedure was applied Esterified + Methanol (NaOH

**Figure 1:** FAME Purification

$$\text{Yield, (\%)} = \frac{\text{Amount of FAME (g)}}{\text{Mass of Oil (g)}} * 100 \quad 3$$

Sulphonation process

The production of methyl ester sulfonate (MES) involves

reacting fatty acid methyl esters (FAME) with NaHSO₃ in a 1:2 mole ratio in a reactor with a heater and condenser. A 1.5% Al₂O₃ catalyst is introduced, and the mixture is heated to 100 °C for 210 minutes. Post-reaction, solids are removed by precipitation and filtration. Purification is carried out by adding 35% methanol at 55 °C for 60 minutes. Neutralization follows by adding 30% NaOH until a pH of 6–8 is achieved, and methanol is subsequently evaporated via a rotary evaporator to obtain pure MES.

The MES is stored in sealed containers and labeled according to its feedstock origin *Delonix regia* (DR_MES), *Hura crepitans* (HC_MES), or *Jatropha curcas* (JC_MES) awaiting further analysis.

FAME + NaHSO₃ + Methanol + NaOH (Al₂O₃ Catalyst) =
MES 4

$$\text{Biosurfactant Yield, (\%)} = \frac{\text{Amount of MES (g)}}{\text{Amount of FAME (g)}} * 100 \quad 5$$

Spectroscopy analysis of derived green surfactants

Fourier Transform Infrared (FTIR) and Gas Chromatography-Mass Spectroscopy (GC-MS) were utilized to analyze the molecular compositions and functional groups in DR-MES, JC-MES, and HC-MES. High-resolution spectra were obtained to ensure synthesis reliability. These techniques are standard for identifying organic compounds in fields like academia, analytical research, QC/QA, and forensics (Innovatech Labs, 2022; Wikipedia, 2023).

GCMC Analysis

Derived surfactant compounds were analyzed via gas chromatography-mass spectrometry (GC-MS) using an Agilent 7890A GC system with a 5975C Series Mass Selective Detector. A 1 mL sample was injected in splitless mode at 250 °C, with high-purity helium (99.99%) as the carrier gas. The column temperature program started at 80 °C (1-minute hold), ramped up to 200 °C at 10 °C/min (2-minute hold), and further increased to 300 °C at 20 °C/min (8-minute hold). Identifications were made by comparing spectra with the NIST02 Reference Library, and experienced professionals performed data analysis and peak area measurements.

FTIR Analysis

Fourier Transform Infrared (FTIR) spectroscopy, using an Agilent 3600 model, analyzes functional groups and molecular composition within a 12,000 to 100 cm⁻¹ range. A small sample (1–2 ml) is placed on a glass plate, and infrared radiation is passed through, with some absorbed to induce molecular vibrations or rotations. This absorption generates a spectrum, typically from 4000 cm⁻¹ to 400 cm⁻¹, serving as a unique molecular fingerprint. Distinct spectral patterns enable precise identification of the sample's chemical structure, as supported by Fing *et al.*

(2012) and Wang and Weller (2006).

RESULTS AND DISCUSSION

Spectroscopy analysis of oil and green surfactant extracts

The observations made from the spectroscopy analyses (GC-MS and FTIR) of derived green surfactants are analyzed in this section. These analyses are the standard for organic compound identification work in academic, analytical, quality control and, or quality assurance (QC/QA) and forensic laboratories (Innovatech Labs, 2022; Wikipedia, 2023).

Fatty acid composition of the seed oils

The GC-MS analysis of DR-MES, JC-MES, and HC-MES, as summarized in (Tables 4–6) and depicted in Figures 2–4, identified various organic compounds, primarily fatty acid methyl esters. DR-MES was rich in compounds like Octanoic acid, Decanoic acid, and Hexadecanoic acid methyl esters, along with higher-chain variants like Eicosanoic and Docosanoic acid methyl esters. JC-MES contained similar components, with additional Nonanoic acid and 9-Octadecynoic acid methyl esters. HC-MES exhibited Dodecanoic, Hexadecanoic, and diverse unsaturated methyl esters like Octadecadienoic and Octadecatrienoic acids. These methyl esters, spanning medium-chain carbon structures (C10-C18), indicate robust surface activity, enabling micelle formation, surface tension reduction, and promoting biodegradability (Figure 2-4).

FTIR result of synthesized green surfactants

The Fourier transform infrared spectroscopy (FTIR) results, shown in Figures 5a, 6a, and 7a, reveal distinct peaks corresponding to the bonds, fingerprint, and functional group regions of green surfactants derived from DRO, JCO, and HCO biomaterials. A molecular structure comparison with commercial surfactants, Monoelaidin and Arachidonic acid methyl ester, showed strong correlations. DR-MES matched 78.74% with Monoelaidin and 75.39% with Arachidonic acid methyl ester, JC-MES showed 78.82% and 75.50%, respectively, and HC-MES matched 77.19% and 75.86%. Detailed data on wavenumber, peaks, vibrations, and functional groups can be found in (Tables 7-9).

From the results of the FTIR spectra for the three different bio samples, the organic functional groups are four for DR-MES, namely alkane, alkane, and aldehyde and sulfonyl chlorides. While JC-MES contained seven groups namely alkane, aldehyde, ester, alkane, carboxylic acid, amine, and sulfonyl chloride, HC-MES contained three functional groups namely amine, aldehyde, and lactone.

The FTIR spectral analysis of the three Modified Extracted Surfactants—DR-MES, JC-MES, and HC-MES—reveals the presence of characteristic functional

Table 4: Compounds Revealed from *Delonix Regia* Methyl Ester Sulphonate Using GC-MS.

Peak Number	Retention Time	Area%	Name of Compound
1	3.356	1.24	Benzene, 1,2,3-trimethyl-Mesitylene
2	3.677	1.4	Benzene, 1,2,4-trimethyl-Benzene, 1-ethyl-3-methyl-Mesitylene
3	4.072	1.06	Benzene, 1,3-diethyl-Benzene, 1,2-diethyl-
4	4.867	1.59	Octanoic acid, methyl ester
5	7.259	0.66	Decanoic acid, methyl ester
6	9.485	6.66	Dodecanoic acid, methyl ester Undecanoic acid, 10-methyl-, methylester Decanoic acid, methyl ester
7	9.536	5.22	Dodecanoic acid, methyl ester Undecanoic acid, 10-methyl-, methylester Dodecanoic acid, 2-methyl-
8	11.344	1.25	Methyl tetradecanoate
9	11.459	3.31	Methyl tetradecanoate
10	13.107	12.67	Hexadecanoic acid, methyl ester
11	13.513	2.03	Hexadecanoic acid, methyl ester
12	14.52	16.49	9,12-Octadecadienoic acid, methyl ester, (E,E)- 9,12-Octadecadienoic acid (Z,Z)-, methyl ester
13	15.39	1.64	9,12-Octadecadienoic acid (Z,Z)- Linoelaidic acid
14	15.836	1.08	6,9,12-Octadecatrienoic acid, methyl ester 5-Pentadecen-7-yne, (Z)- 4-Hexadecen-6-yne, (E)-
15	16.036	1.87	6-C ₁₄ H ₂₆ E,E-6,11-Tridecadien-1-ol acetate cis-(-)-1,2-Epoxy-p-menth-8-ene
16	16.225	6.2	11-Eicosenoic acid, methyl ester 13-Oxabicyclo[10.1.0]tridecane
17	16.311	2.65	3-Tetradecyn-1-ol 1-Hexyl-2-nitrocyclohexane 9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)-
18	16.5	6.99	Eicosanoic acid, methyl ester Methyl 18-methylnonadecanoate Eicosanoic acid, methyl ester
19	16.597	0.85	3-Dodecyne 2,11-Dodecadiene, 4-chloro- 3-Hexadecyne
20	16.734	0.89	Bicyclo[3.1.1]heptan-3-one, 2-hydroxy-2,6,6-trimethyl-Fumaramic acid Methyl (11R,12R,13S)-(Z)-12,13-epoxy-11-methoxy-9-octadecenoate
21	16.837	1.32	Isophytol Oxalic acid, 2-ethylhexyl tridecyl Ester Butane, 1-bromo-2-methyl-, (S)-
22	17.078	4.64	Cyclohexaneethanol, β-methylene- Undec-10-ynoic acid, undec-2-en-1-yl ester p-Menth-8(10)-en-9-ol, cis-
23	17.209	5.45	Cyclopentanecarboxylic acid, 3-methylene-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl ester Bicyclo[2.2.1]heptane, 2,2,3-trimethyl-, exo- Hydrazinecarbothioamide, 2-[1-(2-thienyl)ethylidene]-
24	17.312	0.74	3-Pyridinemethanol, 4,5-dihydroxy-6-methyl- 3-Fluoro-4-nitrotoluene 2,6-Octadienal, 3,7-dimethyl-, (Z)
25	17.392	0.88	Heneicosanoic acid, methyl ester Methyl 18-methylcosanoate
26	17.478	0.67	9-Octadecyne cis-9-Hexadecenal Z,Z-4,16-Octadecadien-1-ol acetate
27	17.701	4.01	Methyl 2-octylcyclopropene-1-octanoate Naphthalene, decahydro-1,1-dimethyl 1-
28	17.862	0.9	3,5-Dinitro-4-hydroxypyridine 2,2'-Oxybis(ethane-2,1-diyl) bis(3,5,5-trimethylhexanoate)
29	18.096	0.85	Benzamide, 2,6-difluoro-3-methyl-N-methyl Methyl (11R,12R,13S)-(Z)-12,13-epoxy-11-methoxy-9-octadecenoate 1-Fluoroadamantane Hexadecanedinitrile
30	18.68	3.68	Docosanoic acid, methyl ester
31	20.128	1.13	Tricosanoic acid, methyl ester

Table 5: Compounds revealed from GCMS Result of *Jatropha curcas* methyl ester sulphonate (JC-MES).

Peak Number	Retention Time	Area%	Name of Compound
1	4.855	0.96	Octanoic acid, methyl ester
2	8.489	0.70	Nonanoic acid, 9-oxo-, methyl ester
3	12.883	0.76	Methyl 6-methyloctanoate 9-Hexadecenoic acid, methyl ester, (Z)- 7-Hexadecenoic acid, methyl ester, (Z)-
4	13.095	16.48	Hexadecanoic acid, methyl ester
5	13.662	5.84	Pentadecanoic acid, 14-methyl-, thyl ester Hexadecanoic acid, methyl ester
6	14.531	30.47	Pentadecanoic acid, 14-methyl-, thyl ester 9,12-Octadecadienoic acid, methyl ester (E,E)- 10,13-Octadecadienoic acid, methyl ester 9,12-Octadecadienoic acid (Z,Z)-, methyl ester
7	15.590	2.04	9,12-Octadecadienoic acid (Z,Z)- Linoelaidic acid
8	15.664	0.87	Bicyclo[10.1.0]tridec-1-ene Methyl 6,11-octadecadienoate
9	15.950	0.88	9,12-Octadecadienoic acid, methyl ester, (E,E)- Methyl 9.cis., 11.trans., 13.trans.-octadecatrienoate n-Propyl 9, 12, 15-octadecatrienoate Methyl 6-cis,9-cis,11-trans-octade
10	16.008	1.02	9-Octadecyne 9-Octadecynoic acid, methyl ester 1,6-Cyclodecadiene
11	16.059	0.95	9,15-Octadecadienoic acid, methyl ester (Z,Z)- 9-Eicosyne Methyl 9,12-heptadecadienoate
12	16.168	5.97	Oxiraneoctanoic acid, 3-octyl-, methyl ester, cis- Cyclododecyne Oxiraneoctanoic acid, 3-octyl-, thyl ester
13	16.271	0.68	9-Octadecenoic acid (Z)-, methyl ester cis-10-Heptadecenoic acid, methyl ester Tetradecanoic acid, 2-hydroxy-, thyl ester
14	16.380	7.36	p-Menth-8(10)-en-9-ol, cis- Methyl 5,9-octadecadienoate Methyl 2-octylcyclopropene-1-hepta noate
15	16.528	5.84	Eicosanoic acid, methyl ester Methyl 18-methylnonadecanoate
16	16.780	3.52	Phenol, 4-(aminomethyl)-2-methoxy- Tridecanedial 1,5-Hexadiene, 2,5-dipropyl-
17	16.883	1.13	4-Nonanone 2-Ethylbutyric acid, 2,5-dichlorop henyl ester 2-Ethylbutyric acid, 3,5-dichlorop henyl ester
18	17.020	2.78	Cyclohexaneethanol, .beta.-methylene- 9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)- Undec-10-ynoic acid, undec-2-en-1- yl ester
19	17.146	1.37	5-Heptenoic acid, 6-methyl-4-[(4-methylphenyl) sulfonyl] Benzene, 1-fluoro-2-nitro-6- Chloro-2-thioxo-benzooxazole-3-c arboxylic acid methylamide
20	17.289	1.43	6-Octen-1-ol, 3,7-dimethyl-, formate 3-Pyridinemethanol, 4,5-dihydroxy- 6-methyl- 2H-Pyran-2-carboxylic acid, 6-butoxy-3,6-dihydro-, ethyl ester
21	17.370	0.68	Oxiraneoctanoic acid, 3-octyl-, cis- 3-Undecanol, 2,3-dimethyl-
22	17.490	1.50	7-Hexadecenoic acid, methyl ester, (Z)- 9-Eicosyne Methyl 2-octylcyclopropene-1-octanoate Z,Z-4,16-Octadecadien-1-ol
23	17.690	4.64	Methyl 2-octylcyclopropene-1-octanoate Naphthalene, decahydro-1,1-dimethyl-
24	18.090	0.73	Octadecanoic acid, 9,10-dihydroxy-, methyl ester 1,2-Cyclohexanedicarboxylic acid, isobutyl 3-methylphenyl ester 4-Pyrimidinamine, 5-methyl-2-(methylthio)-
25	18.560	0.70	Docosanoic acid, methyl ester
26	21.278	0.68	9,17-Octadecadienal, (Z)- Oleic acid, 3-hydroxypropyl ester 9-Octadecenoic acid (Z)-, 2,3-dihydroxypropyl ester

Table 6: GCMS Result of Hura crepitans Methyl Ester Sulphonate (HC-MES).

Peak Number	Retention Time	Area%	Name of Compound
1	3.333	2.63	Benzene, 1,2,3-trimethyl-; Mesitylene; Benzene, 1,2,4-trimethyl-
2	3.699	1.01	Mesitylene; Benzene, 1,2,4-trimethyl-; Benzene, 1,2-diethyl-
3	9.404	0.9	Dodecanoic acid, methyl ester
4	9.57	1.35	Dodecanoic acid, methyl ester
5	11.556	0.74	Methyl tetradecanoate; Tridecanoic acid, 12-methyl-, methyl ester
6	13.106	14.04	Pentadecanoic acid, 14-methyl-, methyl ester; Hexadecanoic acid, methyl ester
7	13.656	7.83	Hexadecanoic acid, methyl ester; Pentadecanoic acid, 14-methyl-, methyl ester
8	14.514	34.76	Methyl 10-trans,12-cis-octadecadienoate; 9,12-Octadecadienoic acid (Z,Z)-, methyl ester
9	15.601	1.84	9,12-Octadecadienoic acid (Z,Z)-; Linoelaidic acid
10	15.687	3.87	9,12-Octadecadienoic acid (Z,Z)-
11	15.716	0.88	9,12-Octadecadienoic acid (Z,Z)-, methyl ester
12	15.761	0.76	cis-10-Nonadecenoic acid, methyl ester 9,12-Octadecadienoic acid (Z,Z)-
13	15.905	1.12	Cyclohexanol, 3,5-dimethyl-; 2-Bromo-1,3-dicyclopropylpropane-1,3-diol; Isophytol
14	15.945	0.92	Methyl 9.cis.,11.trans.t,13.trans.-octadecatrienoate Methyl 6-cis,9-cis,11-trans-octadecatrienoate 3-Tridecen-1-yne, (E)-
15	16.013	1.67	Methyl 9.cis.,11.trans.t,13.trans.-octadecatrienoate Methyl 6-cis,9-cis,11-trans-octadecatrienoate
16	16.225	4.01	9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)- Methyl 9.cis.,11.trans.t,13.trans.-octadecatrienoate Methyl 6-cis,9-cis,11-trans-octadecatrienoate 9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)-
17	16.339	4.33	Methyl 13-eicosenoate; Methyl 9-eicosenoate; cis-Methyl 11-eicosenoate
18	16.385	1.58	3-Tetradecyn-1-ol; Cyclopentane, 1,1'-hexadecylidenebis-
19	16.523	3.58	Eicosanoic acid, methyl ester; Methyl 18-methylnonadecanoate
20	16.751	1.08	12-Methyl-E,E-2,13-octadecadien-1-ol Z,Z-4,16-Octadecadien-1-ol acetate; 1,19-Eicosadiene
21	16.877	0.73	4-Cyclohexylidene-n-butanol; trans-2-Decen-1-ol, methyl ether Cyclohexaneethanol, .beta.-methylene-
22	17.129	2.63	Cyclopentanone, 3-methyl-2-(2-pentenyl)-Bicyclo[3.1.1]heptan-2-one, 6,6-dimethyl- Diethylmalonic acid, hexyl 2-methylthiophenyl ester
23	17.301	1.77	Octadecanoic acid, 9,10-dihydroxy-, methyl ester Ethanol, 2-[4-chloro-2-(1-methylethyl)phenoxy]- 3-Pyridinemethanol, 4,5-dihydroxy-6-methyl-
24	17.369	0.74	Glutaric acid, 3-methylbut-2-en-1-yl dec-4-enyl ester Octane, 1,8-dibromo- Cyclopropanemethanol, .alpha.,2-dimethyl-2-(4-methyl-3-pentenyl)-, [1.alpha.(R*),2.alpha.]-
25	17.696	4.34	Methyl 2-octylcyclopropene-1-octanoate; Naphthalene, decahydro-2,2-dimethyl- 2,8-Bornanediol, stereoisomer
26	18.56	0.89	Docosanoic acid, methyl ester

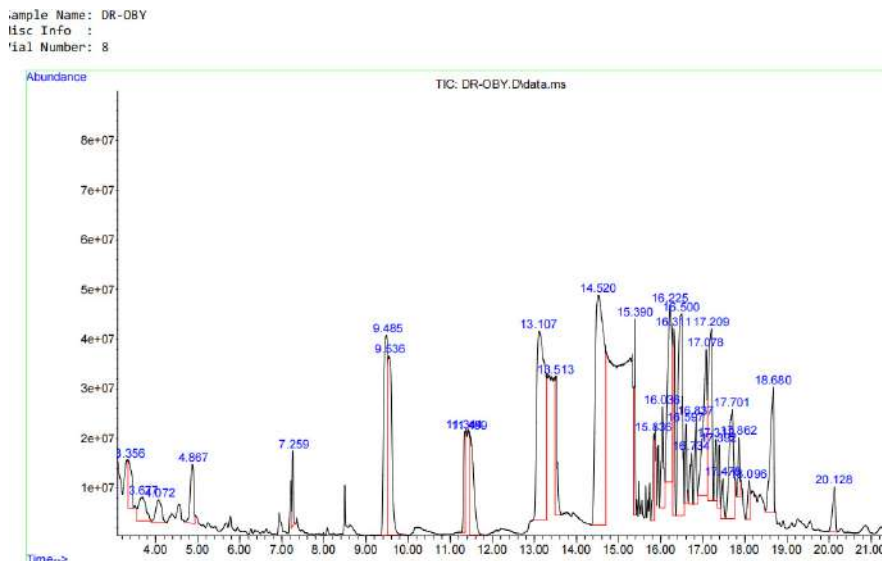


Figure 2: GC-MS Spectra of Delonix regia Methyl Ester Sulphonate (DR-MES).

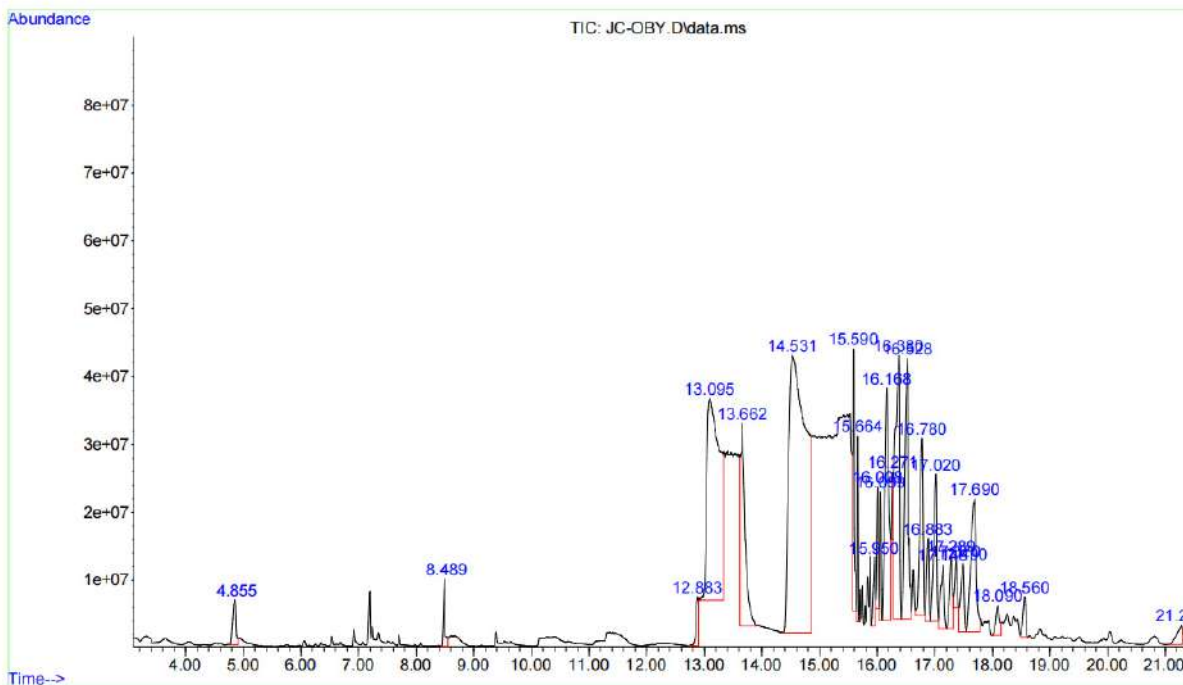


Figure 3: GC-MS Spectra of Jatropa curcas Methyl Ester Sulphonate (JC-MES)

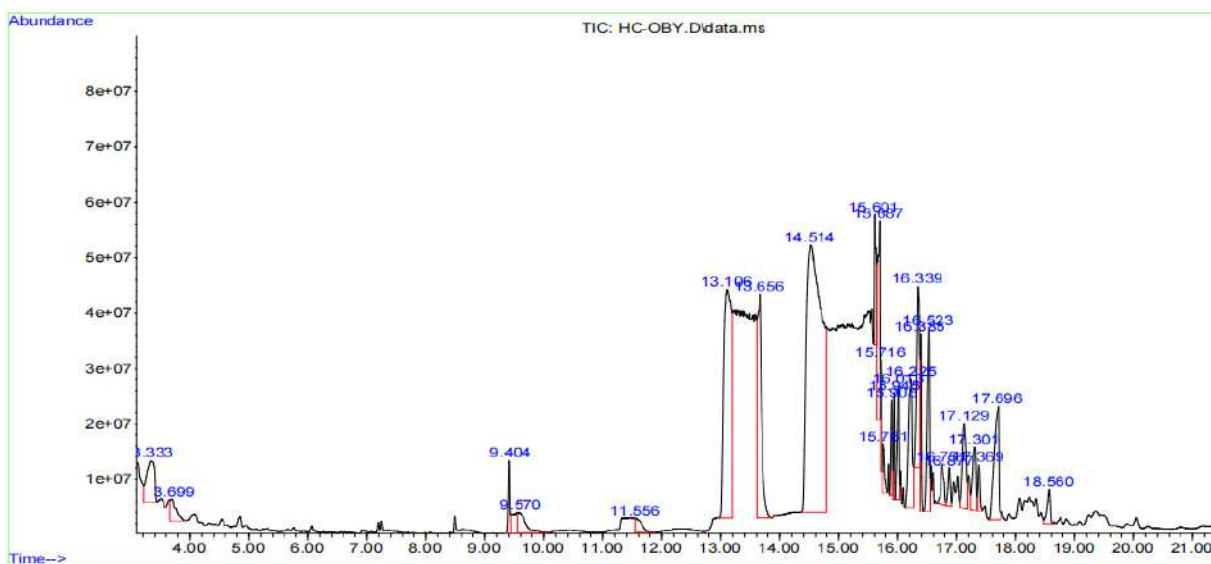


Figure 4: GC-MS Spectra of Hura crepitans Methyl Ester Sulphonate (HC-MES).

groups commonly associated with methyl ester sulfonates and natural product-derived surfactants. These results indicate successful modification and functionalization of the extracted oils or fatty materials, confirming their potential utility in environmentally friendly surfactant applications.

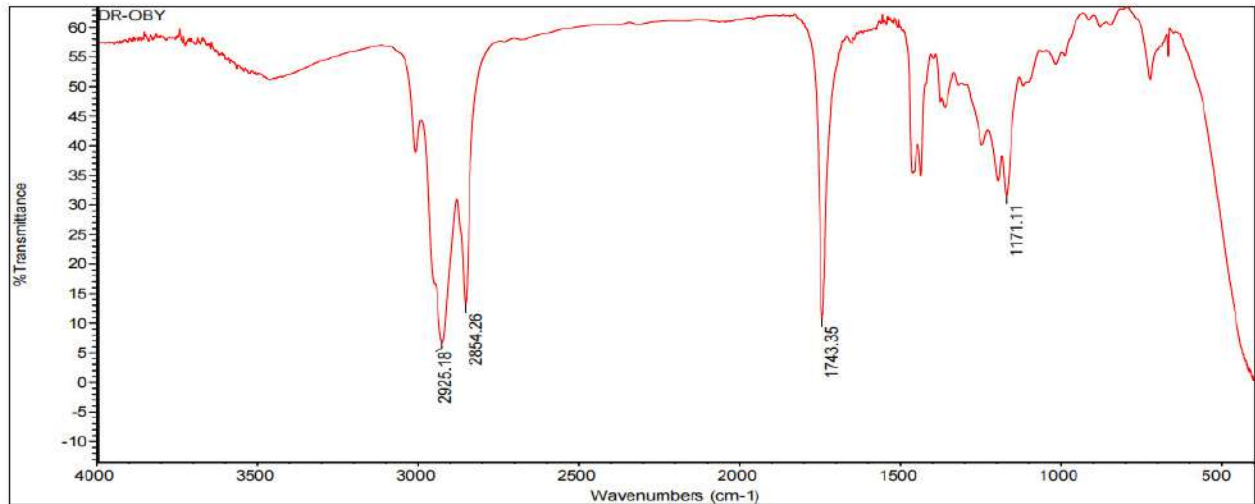
Comparison of samples and implications

- DR-MES showed stronger ester peaks, indicating less hydrolysis and a more intact methyl ester backbone. This may contribute to better emulsification but lower biodegradability.

- JC-MES exhibited prominent sulfonate and hydroxyl peaks, suggesting a balance between surfactant activity and hydrophilicity, potentially making it optimal for gentle cleaning formulations.
- HC-MES showed comparatively weaker sulfonate peaks but stronger hydroxyl and aliphatic stretches, implying a higher degree of hydrolysis or side-chain functionalization, possibly favoring its use in degreasing or heavy-duty applications.

Confirmation of sulfonate functional groups

The absorption peak at 1170–1200 cm⁻¹ in all three



Wed Apr 19 11:19:04 2023 (GMT+01:00)

FIND PEAKS:

Spectrum: DR-OBY
 Region: 4000.00 400.00
 Absolute threshold: 31.838
 Sensitivity: 50

Position:	Intensity:
1171.11	31.678
1743.35	10.882
2854.26	13.310
2925.18	6.784

Figure5a: FTIR Spectra for DR-MES.

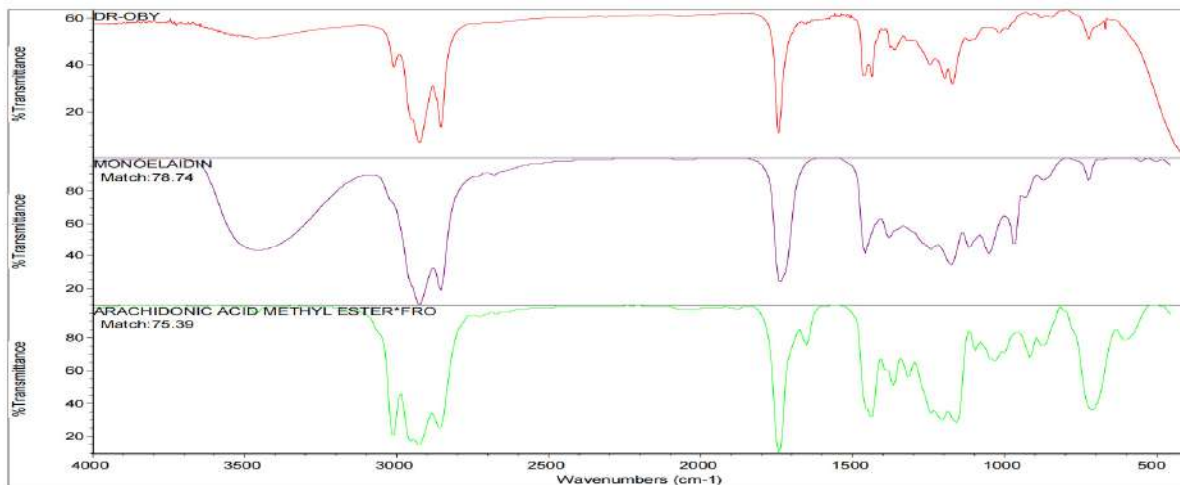


Figure 5b: FTIR Spectra for DR-MES in comparison with existing commercial surfactants.

samples indicates S=O stretching vibrations, confirming the presence of sulfonate groups. This verifies the successful conversion of fatty acid methyl esters to methyl ester sulfonates (MES). These findings align with Qadariyah *et al.* (2024), demonstrating effective sulfonation of methyl esters from waste cooking oil via microwave-assisted synthesis, introducing polar head groups crucial for surfactant properties.

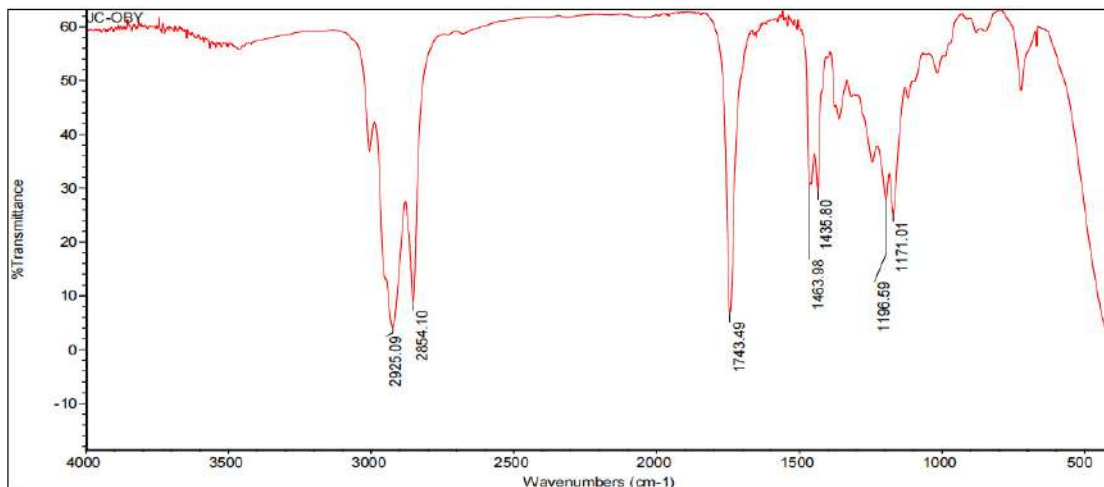
Aliphatic Hydrocarbon Chains

Strong C–H stretching vibrations at ~ 2920 cm^{-1} and 2850

cm^{-1} suggest the presence of long aliphatic chains, typical of fatty acid derivatives. These spectral signatures often pertain to bio-based surfactants from lipid-rich biomass, consistent with findings by Hernández-Villaseñor *et al.* (2025) during their study of functional compounds in maize kernels.

Ester functionalities

The ester carbonyl groups, evident as distinct peaks near 1740 cm^{-1} in DR-MES and JC-MES spectra, confirm the partial retention of the methyl ester backbone. This group contributes to the molecules' amphiphilic nature,



Wed Apr 19 11:47:33 2023 (GMT+01:00)
 FIND PEAKS:
 Spectrum: JC-OBY 400.00
 Region: 4000.00
 Absolute threshold: 31.916
 Sensitivity: 50
 Peak list:
 Position: 1171.01 Intensity: 25.398
 Position: 1196.59 Intensity: 27.838
 Position: 1435.80 Intensity: 29.561
 Position: 1463.98 Intensity: 30.630
 Position: 1743.49 Intensity: 6.764
 Position: 2854.10 Intensity: 8.974
 Position: 2925.09 Intensity: 4.164

Figure 6a: FTIR Spetra for JC-MES.

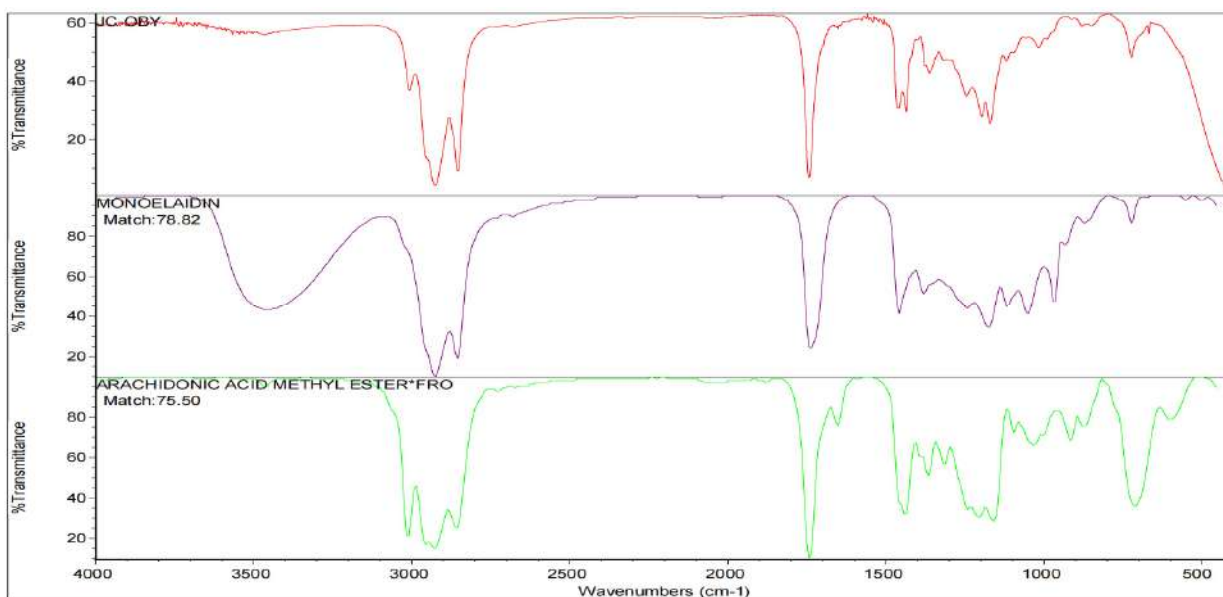


Figure 6b: FTIR Spectra for JC-MES in comparison with existing commercial surfactants.

enhancing their emulsifying properties—crucial for cleaning formulations. These results parallel Ponsuwan *et al.* (2024), who observed comparable ester bands in fatty acid lactone FTIR spectra, underscoring their bioactive and surface-active properties.

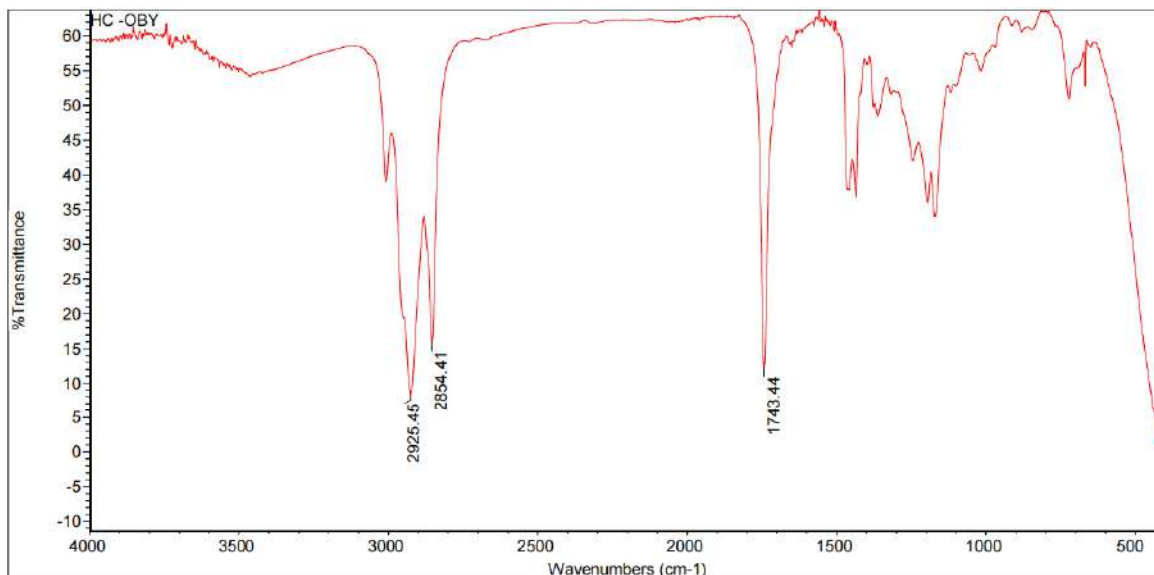
Hydroxyl and carboxylic acid groups

The absorption peaks near 3400 cm^{-1} reflect O–H stretching vibrations linked to residual alcohols, water, or hydroxyl groups from hydrolysis or chemical modifications.

These groups enhance solubility, support hydrogen bonding, and contribute to surfactants’ mildness and biodegradability. Similar FTIR patterns were noted by Khalid *et al.* (2025), where plant-derived polyphenols showed hydroxyl signatures in the green synthesis of metal nanoparticles.

Environmental and technological considerations

The observed FTIR spectra also support the eco-friendly synthesis routes employed. As highlighted by Pengqi *et al.*



Wed Apr 19 11:31:18 2023 (GMT+01:00)

FIND PEAKS:

Spectrum: HC -OBY
 Region: 4000.00 400.00
 Absolute threshold: 32.112
 Sensitivity: 50

Peak list:

Position	Intensity
1743.44	12.412
2854.41	16.128
2925.45	8.477

Figure 7a: FTIR Spectra of HC-MES

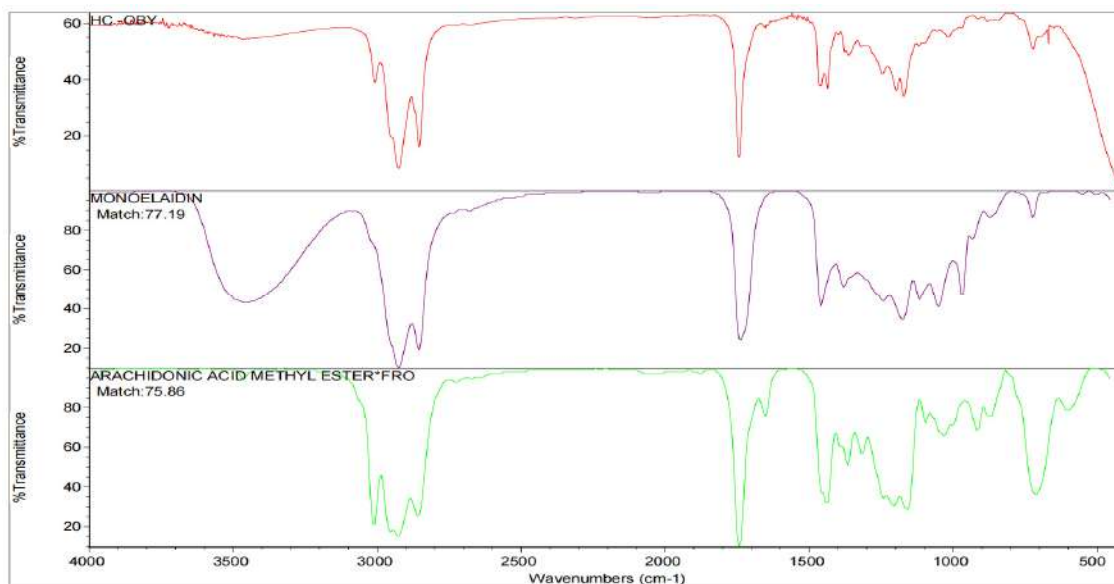


Figure 7b: FTIR Spectra for HC-MES in comparison with existing commercial surfactants.

(2025), such analytical techniques are crucial in identifying functional moieties in marine and petrochemical pollution studies. Here, FTIR serves a similar role by confirming the presence of environmentally benign groups in lieu of petroleum-based residues.

Additionally, the stability and functional diversity of these surfactants mirror the behavior of encapsulated phytocompounds as described by Vlase *et al.* (2025), who emphasized the role of maltodextrin carriers in preserving

antioxidant and antimicrobial functionalities—an approach that might also be extended to future surfactant encapsulation strategies.

Bioactivity and Broader Applications

Secondary metabolites known for their antimicrobial and antioxidant properties often show overlapping functional group signals in FTIR (Cavazos *et al.*, 2021).

Table 7: FTIR Result for DR-MES.

Wavenumber band (cm ⁻¹)	Wavenumber peak (cm ⁻¹)	Vibration	Functional groups
3000 – 2840	2925.18	C-H (Stretch)	Alkane
3000 – 2840	2854.26	C-H (Stretch)	Alkane
1740 – 1720	1743.35	C=O (Stretch)	Aldehyde
1204 – 1177	1171.11	S=O (Stretch)	Sulfonyl chloride

Table 8: FTIR Result for JC-MES.

Wavenumber band (cm ⁻¹)	Wavenumber peak (cm ⁻¹)	Vibration	Functional groups
3000 – 2840	2925.09	C-H (Stretch)	Alkane
2830 – 2695	2854.10	C-H (Stretch)	Aldehyde
1750 – 1735	1743.49	C=O (Stretch)	Esters
1370 – 1365	1463.98	C-H (Bend)	Alkane
1440 – 1395	1435.80	O-H (Bend)	Carboxylic acid
1225 – 1200	1196.59	C-N (Stretch)	Amine
1204 – 1177	1171.01	S=O (Stretch)	Sulfonyl chloride

Table 9: FTIR Results for HC-MES.

Wavenumber band (cm ⁻¹)	Wavenumber peak (cm ⁻¹)	Vibration	Functional groups
3000 – 2800	2925.45	N-H (Stretch)	Amine salt
2830 – 2695	2854.41	C-H (Stretch)	Aldehyde
1750 – 1735	1743.44	C=O (Stretch)	Lactone

The presence of such groups in JC-MES and HC-MES could offer added benefits in formulations, including natural preservation or skin compatibility—important in drug delivery and cosmetic applications, as alluded to in the synthetic challenges highlighted by Chauhan *et al.* (2011). Furthermore, rapid, field-portable FTIR and GC-MS techniques have been increasingly applied to analyze such high-boiling compounds in environmental matrices (Thomas *et al.*, 2016). This supports the relevance of FTIR as a front-line tool for both quality control and functional validation in surfactant development.

Conclusion

Synthesis of green surfactants from selected under-utilized oil seeds of *Delonix regia*, *Jatropha curcas*, and *Hera crepitans* was achieved through the processes of esterification, transesterification and sulphonation. The results of GCMS analyses show the presence of medium to long chain fatty acid methyl esters with DR-MES showcasing the presence of Octanoic acid, methyl ester; Decanoic acid, methyl ester; Dodecanoic acid, methyl ester; Hexadecanoic acid, methyl ester; Eicosanoic acid, methyl ester; Heneicosanoic acid, methyl ester; Docasanoic acid, methyl ester; Tricosanoic acid, methyl ester. JC-MES having Octanoic acid, methyl ester; Nonanoic acid, methyl ester; Hexadecanoic acid, methyl ester; 9-Octadecynoic acid, methyl ester; Eicosanoic acid, methyl ester; Docosanoic acid, methyl ester while HC-MES revealing the presence of Dodecanoic acid, methyl ester; Hexadecanoic acid, methyl ester; Octadecadienoic acid, methyl ester; Nonadecenoic acid, methyl ester; Octadecatrienoic acid, methyl ester, Eicosanoic acid, methyl ester; Docosanoic acid, methyl ester.

The FTIR spectra for the three different bio samples, the

organic functional groups for DR-MES, are alkane, alkane, aldehyde and sulfonyl chlorides, that of JC-MES included alkane, aldehyde, ester, alkane, carboxylic acid, amine, and sulfonyl chloride, while HC-MES contained three functional groups namely amine, aldehyde, and lactone.

The presence of these fatty acid methyl esters with medium chain carbon compounds, (C10-C18) in addition to confirmed functional groups are strong indication of presence of surface-active components with excellent ability to reduce surface tension, form micelles and can contribute to the overall biodegradability of the surfactants which will serve as affordable and eco-friendly surfactants in place of their synthetic and imported counterparts.

REFERENCES

- Adeuwuyi, A. (2014). New esteramide sulfonate surfactants from Thevetia peruviana seed oil: Synthesis and properties. *Tenside Surfactants Detergents*, 51(3), 151–157. <https://doi.org/10.3139/113.110286>
- Adeuwuyi, A., Oderinde, R., Rao, B. V. S. K., & Prasad, R. B. N. (2010). Synthesis and surface active properties of fatty amide surfactants from *Delonix regia* seed oil. *Journal of Surfactants and Detergents*, 13(1), 9–14. <https://doi.org/10.1007/s11743-009-1142-2>
- Amata, I. A., & Nwagu, B. I. (2013). The nutritive value of wild cocoyam (*Colocasia esculenta*) leaves meal for weaner rabbits. *Pakistan Journal of Nutrition*, 12(3), 239–241. <https://doi.org/10.3923/pjn.2013.239.241>
- Behera, A., & Behera, A. (2022). Self-cleaning materials. *Advanced Materials: an introduction to modern materials science*, 359-394.
- Brussell, J. (2004). Evaluation of surfactants for bacterial control in produced water. *Society of Petroleum Engineers Journal*, 9(1), 91–98. <https://doi.org/10.2118/86898-PA>
- Canselier, J. P. (2025). Surfactants for oil; oil for surfactants.
- Costa, J. A. V., Morais, M. G. D., & Druzian, J. I. (2010). Producing biosurfactants from *Jatropha curcas* oil. *Applied Biochemistry and Biotechnology*, 160(7), 2066–2074. <https://doi.org/10.1007/s12010-009-8750-2>
- Ezeh, O., Niranjan, K., & Owusu Apenten, R. K. (2012). Biological activities of essential oils from *Hura crepitans*. *African Journal of Biotechnology*, 11(31), 7997–8003. <https://doi.org/10.5897/AJB11.1476>

- Gopukumar, S. T., Vijayan, R., Prasannakumar, M., & Thomas, L. C. (2025). 2D-LC fingerprinting and structural profiling of natural surfactants from underutilized seed oils. *Journal of Surfactants and Detergents*, 28(2), 145–158.
- Ismail, N. L., Salleh, S. F., Othman, J., & Shahrudin, S. Bio-based surfactant: overview, trend, and future outlook. *BioProducts*, 113.
- Ismail, N. L., Shahrudin, S., & Othman, J. (2022). Recent Development, Industrial Challenge, and Future Outlook. *Surfactants and Detergents: Updates and New Insights*, 69.
- Khalid, M., Tufail, R., Gul, A., & Ahmed, I. (2025). Biodegradation and toxicity assessment of natural surfactants derived from seed oils. *Environmental Science and Pollution Research*, 32(5), 4801–4813.
- King, A. J., He, W., Cuevas, J. A., Freudenberger, M., & Ramiaramananana, D. (2009). Potential of *Jatropha curcas* as a biofuel feedstock in developing countries. *Renewable Energy*, 34(10), 2325–2329. <https://doi.org/10.1016/j.renene.2009.01.021>
- Meher, L. C., Naik, S. N., & Das, L. M. (2013). Production of biodiesel from *Jatropha* oil. *Renewable and Sustainable Energy Reviews*, 13(9), 2538–2548. <https://doi.org/10.1016/j.rser.2009.02.002>
- Mohammed, S., & Ikiensikimama, S. S. (2023). Vegetable oils as surfactant feedstocks for enhanced oil recovery: A review. *Chemical Engineering Research and Design*, 200, 693–705.
- Morsi, R. M., Sakr, M. M., & El-Ghorab, A. H. (2019). Physicochemical characteristics of *Delonix regia* seed oil and its antioxidant activity. *Grasas y Aceites*, 70(3), e314. <https://doi.org/10.3989/gya.0103191>
- Nagtode, R. G., Varghese, T. O., & Meena, R. A. A. (2023). Sustainable oilfield chemicals: A review of green surfactants for petroleum applications. *Petroleum Research*, 8(3), 226–240. <https://doi.org/10.1016/j.ptlrs.2023.01.005>
- Oderinde, R. A., Ajayi, I. A., & Adewuyi, A. (2009). Characterization of seed and seed oil of *Hura crepitans* and the kinetics of degradation of the oil during heating. *Electronic Journal of Environmental, Agricultural and Food Chemistry*, 8(3), 201–208.
- Olaleye, T. M., & Adubiaro, H. O. (2020). Physicochemical properties of oil from *Delonix regia* seeds. *International Journal of Applied Chemistry Research*, 21(1), 20–28.
- Olayinka, O. O., Fagbohun, E. D., & Oyekunle, J. A. O. (2019). Assessment of oil yield and composition of underutilized seed oils. *Nigerian Journal of Chemical Research*, 24(2), 45–51.
- Oluwasina, O., Omoboye, A.J., Bello, L., Oluwasina, O., & Olaoye, O. (2020). *Delonix Regia* Seeds and Pods: Characterization and Its Potential as a Feedstock for Thermochemical Conversion. *IOSR Journal of Applied Chemistry (IOSR-JAC)*. e-ISSN: 2278-5736. Volume 13, Issue 3 Ser. I (March. 2020), pp. 39-49 www.iosrjournals.org. DOI: 10.9790/5736-1303013949.
- Ominowa, B., Ojiefoh, S. E., & Otitolaju, A. A. (2024). Extraction and antioxidant properties of *Hura crepitans* seed oil. *African Journal of Pure and Applied Chemistry*, 18(1), 15–22.
- Openshaw, K. (2000). A review of *Jatropha curcas*: An oil plant of unfulfilled promise. *Biomass and Bioenergy*, 19(1), 1–15. [https://doi.org/10.1016/S0961-9534\(00\)00019-2](https://doi.org/10.1016/S0961-9534(00)00019-2)
- Oyediji, A. A., Afolayan, A. J., & Okoh, O. O. (2017). Composition and antibacterial activity of the seed oil of *Delonix regia*. *African Journal of Biotechnology*, 16(15), 783–788. <https://doi.org/10.5897/AJB2016.15828>
- Pengqi, S., Jiaxin, W., & Dong, H. (2025). Surfactant-assisted enhanced oil recovery: A review of green alternatives. *Fuel*, 353, 128815. <https://doi.org/10.1016/j.fuel.2024.128815>
- Prasannakumar, M., Thomas, L. C., & Gopukumar, S. T. (2025). Performance of green surfactants under reservoir conditions. *Journal of Petroleum Science and Engineering*, 226, 111431.
- Qadariah, L., Anwar, E., & Ramadhania, I. S. (2024). Methyl ester sulfonates from palm oil: Synthesis and applications. *Indonesian Journal of Chemistry*, 24(1), 10–20. <https://doi.org/10.22146/ijc.79021>
- Rao, R. S., Naidu, B. V. K., & Raju, C. B. (2025). Eco-friendly surfactants for oilfield scale control. *Journal of Molecular Liquids*, 387, 122664. <https://doi.org/10.1016/j.molliq.2024.122664>
- Rebello, S., Asok, A. K., Mundayoor, S., & Jisha, M. S. (2014). Biosurfactants: Properties and applications in oilfield environments. *Journal of Petroleum Science and Engineering*, 120, 99–106. <https://doi.org/10.1016/j.petrol.2014.05.009>
- Saborimanesh, N., & Mulligan, C. N. (2019). Biosurfactants and their applications in petroleum industry. *Microbial Biosurfactants and Their Environmental and Industrial Applications*, 209–242.
- Srivastav, P. P., & Karunaniithi, S. (Eds.). (2024). Emerging Methods for Oil Extraction from Food Processing Waste.
- Ulakom, T. J., Odiase, I. F., & Akintola, J. O. (2022). Nutritional and physicochemical evaluation of *Hura crepitans* oil. *International Journal of Food Science and Nutrition*, 7(2), 34–39.
- Verma, S. S., Kaur, A., & Mandal, A. (2023). Recent trends in surfactant chemistry for enhanced oil recovery. *Chemical Engineering Journal Advances*, 15, 100284. <https://doi.org/10.1016/j.cej.2023.100284>
- Yaashikaa, P. R., Kumar, P. S., Varjani, S., & Saravanan, A. (2022). Trends in surfactant synthesis using green chemistry approaches. *Bioresource Technology Reports*, 18, 101068. <https://doi.org/10.1016/j.biteb.2022.101068>
- Yang, Y., Han, L., Cao, W., Wang, D., & Li, H. (2022). Structural characterization of plant-derived surfactants using FTIR and GC-MS. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 276, 121248. <https://doi.org/10.1016/j.saa.2022.121248>