COMPARATIVE STUDY OF NATURAL PLANT-BASED DEMULSIFIERS FOR CRUDE OIL EMULSIONS

BY

EDOKPOLOR NOSAZENA IYESOGIE
ENG2106272

DEPARTMENT OF PETROLEUM ENGINEERING
FACULTY OF ENGINEERING
UNIVERSITY OF BENIN
BENIN CITY

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A PROJECT SUBMITTED TO THE DEPARTMENT OF PETROLEUM ENGINEERING IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF BACHELOR OF ENGINEERING (B.ENG) DEGREE IN PETROLEUM ENGINEERING

DEPARTMENT OF PETROLEUM ENGINEERING
FACULTY OF ENGINEERING
UNIVERSITY OF BENIN
BENIN CITY.

CERTIFICATION

This is to certify that this project was carried out by **EDOKPOLOR NOSAZENA IYESOGIE** of the Department of Petroleum Engineering with matriculation number ENG2106272 in partial fulfillment of the requirements for the Award of the Degree, Bachelor of Engineering (B.ENG)

PROF. KELANI BELLO **DATE**
(PROJECT SUPERVISOR)

DR. SEUN TAIWO **DATE**
(PROJECT COORDINATOR)

DR. IKPONMWOSA OHENHEN **DATE**
(HEAD OF DEPARTMENT)

DEDICATION

This thesis is dedicated to God Almighty, who made it possible for me to complete the study successfully. This work is dedicated to my father, mother, siblings, alongside my professors and lecturers who have taught me that the best kind of knowledge to have is that which is learned for its own sake and have been a major source of motivation in this academic journey

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ABSTRACT

The study focuses on the formulation and comparing efficiency of plant-based demulsifiers as sustainable substitutes of disintegrating water-in-oil emulsions during crude oil processing. The objective of the study is to determine the efficiency of the selected natural materials in the demulsification process like clove extract, coconut oil and orange and banana peels combined, besides evaluating the effect of external forces on the work of the demulsifier like diesel dilution and magnetic fields. Fourier Transform Infrared Spectroscopy (FTIR) was used to determine chemical composition of each bio-extract and functional groups to develop a relationship between the structure and activity in terms of emulsion destabilization. The quantitative information about the performance trends was gained with the help of experimental data during a 70-minute period of treatment, which was analyzed using the standard deviation statistics (which are tabulated in the appendix). Findings showed that all the natural demulsifiers were highly interfacially active due to the presence of surface-active compounds, including phenolics, flavonoids, terpenes, and fatty acids, which influence the interfacial activity of the natural demulsifiers through their ability to destabilize the interfacial films and induce droplet coalescence. The clove extract recorded the greatest demulsification efficiency among all samples that can be explained by its high phenolic concentration and good amphiphilicity. It was found that diesel dilution and magnetic treatment can affect, but not change much of the demulsification behavior, which proves that intrinsic chemical composition is a stronger factor, compared to extrinsic factors. All in all, the research proves that the natural plant demulsifiers have a promising potential to substitute the traditional chemical demulsifiers in the process of crude oil treatment and to provide similar or better results at less environmental and economic expenses. The results highlight the promise of green demulsification technologies as an important measure to achieve sustainable production and processing of petroleum.

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF STUDY

As crude oil is extracted out of the reservoirs, it usually contains large quantities of water and other impurities in the form of a stable emulsion that is extremely difficult to manage during production, transportation, and refining. Water and oil cannot combine to get a real solution. Nonetheless, drops of oil can break in water and small drops of water can break in oil. These are referred to as emulsions; oil-in-water (o/w) and water-in oil(w/o), respectively. Naturally occurring surface-active agents like asphaltenes, resins, fine solids, and organic acids in crude oil are the stabilizers of these oil-water emulsions. They raise the viscosity, lead to corrosion and fouling as well as diminishing the efficiency of processing units.

Demulsification is thus an important procedure in petroleum production which entails the separation of emulsion with the view of breaking these emulsions and hence resulting in the effective separation of oil and water phases. Most of the conventional demulsifiers are synthetic and although effective, they tend to be toxic, non-biodegradable and harmful to the environment. The growing interest in sustainability and green chemistry across the world has led to seeking eco-friendly and biodegradable natural options.

Nature plant extracts and oils have presented good prospects as green demulsifiers due to their abundant surface active compounds, in the form of terpenes, phenols, fatty acids and esters. These substances may adsorb at the interface between the oil and water and replace stabilizing agents and induce coalescence of water droplets resulting in the effective phase separation. They can be clove extract (eugenol, a potent surfactant), neem oil (containing fatty acids and triterpenoids) and banana and orange peel extracts (containing natural oils and polar compounds) and coconut oil (lauric acid content, amphiphilic properties). These natural extracts have varying degrees of obtaining success as demulsifiers, which is determined by the chemical compositions, concentration and the properties of the crude oil. Therefore, a comparative analysis is required to understand what natural extract can perform the best in the breaking of crude oil emulsions in the same condition.

To examine more on the mechanism of interaction between the natural demulsifiers and the crude oil emulsion, Fourier Transform Infrared Spectroscopy (FTIR) can be adopted. FTIR analysis informs on the functional groups found in the extracts and chemical transformation of the oil phase at pre-treatment and after treatment. This assists in understanding the nature of interaction of each extract with the emulsion interface in the demulsification process. Thus, the given work is devoted to the comparative analysis of the chosen natural plant extracts clove, neem oil, banana peel, orange peel, and coconut oil as green demulsifiers of crude oil emulsions and their investment in the characterization of the selected products in the FTIR, in order to interpret the interactions between these products and their performance. One of the operational issues that are practiced in the upstream and middle petroleum operations, as the crude oil naturally contains surface-active species that stabilize fine water droplets and slow down phase separation, thus raising viscosity and the risk of corrosion and processes cost (Online Scientific Research, 2025). Due to the non-biodegradability and potential toxicity of traditional synthetic demulsifiers, which are usually petroleum-derived, non-biodegradable, it became of interest to seek a biodegradable and low-toxicity alternative based on plants and other renewable feedstock (Online Scientific Research, 2025). A combination of functional groups phenols, terpenes, fatty acids and esters found in plant oils and extracts (clove, neem, coconut, banana and citrus peels) can adsorb at oil-water interfaces, replace stabilizing film ingredients, and coalesce water droplets making them promising green demulsifiers

1.2 AIM

This study will focus on assessing and comparing the performance of the plant-based demulsifiers chosen in the breaking of water in oil emulsion.

1.3 OBJECTIVES

This research will have the following objectives;

i. To Isolate and obtain green materials that can be used as a demulsifying agent.
ii. To examine the base chemical properties and the functional group.
iii. In order to develop and implement natural vegetable based demulsifiers on crude oil emulsion sample.
iv. Experimentally, prove that dilution of diesel has no effect on demulsifier efficiency
v. Experimentally prove that magnetism at the bottom of the cylinder doesn't have an effect on demulsifier efficiency

1.4 SCOPE OF STUDY

The study area of this paper is the use of local plant demulsifiers based on clove, neem, coconut and orange and banana peel on emulsions of three crude oil sample to identify optimal concentration in the separation of emulsions through the use of a full factorial design method, and the local demulsifer is compared with a commercial one to identify which was more efficient and effective in removing the emulsions.

1.5 PROBLEM STATEMENT

The problem of crude oil emulsion is a longstanding concern in the process of petroleum production and processing because stable and continuous water-in-oil emulsions lower the efficiency of production, raise the cost of water treatment, and may also lead to corrosion and flow assurance issues of the equipment, no matter how much the emulsifier processes the water anyway, the emulsifier has to separate the emulsified water. Presently, imported demulsifers are costly, which brings about the necessity of coming up with a demulsifier derived out of chosen plant, which is cost effective and environmentally friendly. The research is based on this necessity.

1.6 RELEVANCE OF STUDY

The applicability of this research is to come up with natural plant-based demulsifiers, which propagate greener practices during the treatment of crude oil. This study would help promote the use of cost effective solutions that would exploit natural resources by employing locally available plant based materials and minimize the use of full reliance on chemical demulsifiers. The comparison of analysis aids in determining the best plant-based demulsifier.

CHAPTER TWO

LITERATURE REVIEW

2.1 CRUDE OIL

Crude oil or petroleum is a nonrenewable fossil fuel that is made out of the remnants of plants and animals that lived long ago (Britannica, 2022). It is a compound blend of hydrocarbons, which are organic elements made up of carbon and hydrogen (Britannica, 2022).

The crude oil deposits of Nigeria are mainly found at Niger Delta region and offshore fields, and this helps to make it the largest producer of oil in Africa and one of the major exporters of oil around the world. Nigerian crude oils are classified in order to be refined, valued economically and environments.

The crude oils are normally classified according to the API gravity and the sulfur content. The API gravity is a density of oil; high density is associated with light oils; these oils are more valuable as they can be refined more easily and yield more quality products such as gasoline and diesel. Oils whose API movement exceeds 38 are categorized as light, those with a range of 22 to 38 are medium and those that have lower values of less than 22 are heavy. The sulfur content characterizes the sweet and the sour crude; the sweet crude contains less than 0.5% sulfur and is therefore more preferred in refining because it causes less corrosion and less environmental impacts.

In Nigeria, the majority of the crude oils are light and sweet and as such they have high demand in the global markets. These classifications are made on crude oil assay analysis which determines different properties including API gravity, sulfur content and recovery volumes at a particular temperature. As an example, (Adeloye et al. 2022) identified 20 types of crude oil in Nigeria, with 17 of them fitting in the classification of light and sweet, with a sulfur content of less than 0.5 percent and API gravity of higher than 38. It is important to understand how the Nigerian crude oils are classified as a way of refining them in the best way possible, economic planning and environmental management.

These crudes are light and sweet and thus they are very important to the global market, which determines the position of Nigeria in the oil industry. The production of crude oil is hardly made as a single product since it is mixed with water and other contaminants during the production. This mix usually develops an emulsion of crude oil, a steady or semi-steady suspension of water particles in oil (water-in-oil) or oil particles in water (oil-in-water). The emulsion of such is because of non-intended changes in agitation and pressure levels that occur during the production process, as well as because some surface-active compounds are present in the crude, such as resins, asphaltenes and organic acids, which stabilize the interface between oil and water.

In petroleum production processes, the concentration of the basic sediment and water (BS&W) and salt content are important factors in defining the commercial quality and what crude oil needs in terms of handling and processing. Large BS&W values show that there is a high amount of water and solid impurities, which in addition to raising the viscosity of the fluid, also elevate the risks of corrosion, scaling, and fouling in pipelines and processing equipment. Therefore, producers and refiners place strict specification that normally keeps the BS&W at the range of 0.2 to 3.0% in order to make certain that the oil satisfies contractual and processing requirements. The low levels of BS&W and salt would decrease transportation and disposal expenses, chemical and thermal treatment, and deterioration of equipment during transportation and refining. Essentially, crude oil that contains low levels of BS&W and salt is more preferable as it is easier to process, does not present many operating difficulties and results in superior products upon treatment and refining. Therefore, proper demulsification and dehydration are crucial processes in crude oil processing, where they must meet the industry requirements and at the same time enhance economic performance and reliability of the equipment used.

2.2 EMULSIONS

Emulsions represents a liquid heterogeneous system composed of two immiscible liquids (oil and water), in which one of the phases is dispersed in the other, as droplets. An emulsion is formed when two immiscible liquids (like oil and water) are subjected to a shear stress and the presence of a material that is surface active. Due to the provision of water to substitute the fluids in the reservoir at different stages of crude oil production, emulsion occurs.

Crude-oil emulsions are liquid-liquid dispersions, where one of the immiscible phases (usually water) is present as droplets suspended in the other (usually oil), and despite being thermodynamically unstable can also be kinetically extremely stable over time due to the presence of surface-active components like asphaltenes, resins, waxes and fine solids forming viscoelastic films at the oil-water interface (Raya et al., 2020). Such interfacial films reduce interfacial tension and provide a mechanical barrier against droplet coalescence, such that even low concentrations of natural surfactants or solids will convert a rapidly settling problem into an emulsion that forms a mechanical barrier with a tight and thus resists separation and increases viscosity, corrosion risk and processing cost (Raya et al., 2020; MDPI, 2024).

The type of emulsion and stability are determined by numerous factors such as water cut, distribution of droplet sizes, shear history, salinity, pH, crude composition (SARA: saturates, aromatics, resins, asphaltenes), temperature and the existence of solids, and so the same demulsifier may be successful on one crude and unsuccessful on another (Raya et al., 2020; MDPI, 2024). Demulsification of water-in-oil emulsions (e.g., breaking or breaking down of the stabilizing interfacial film) thus is therefore a goal to accelerate the separation process by weakening or eliminating the stabilizing interfacial film with physical treatment (heat, electrostatic coalesces, centrifugation) or chemical treatment (surfactants, polymers) alone or in combination (Raya et al., 2020). Traditional synthetic demulsifiers are usually very effective but toxic, persistent and costly, efficient operation is sensitive to crude properties, leading to the pursuit of greener and biodegradable options based on plants, waste oils and a simple chemical modification of natural oils (Abed et al., 2024; AJAST, 2018).

Recent reviews and experimental research indicate that vegetable oil, plant extracts can be used as bio-demulsifiers since they have amphiphilic molecules (fatty acids, esters, phenolic, terpenes) that adsorb at the interface and can displace or disrupt asphaltene/resin films or alter film elasticity to allow droplets to coalesce (Abed et al., 2024; Research Gate studies; AJAST, 2018)

2.1.1 TYPES OF EMULSIONS

Oilfield emulsions can be categorized into three groups:

- Water-in-oil
- Oil-in-water
- Multiple or complex emulsions

Water-in-oil emulsions contains the droplets of water in a continuous oil phase. Such emulsions are more prevalent in produced Oil fields, where water is the dispersed or internal phase, and oil is the continuous phase

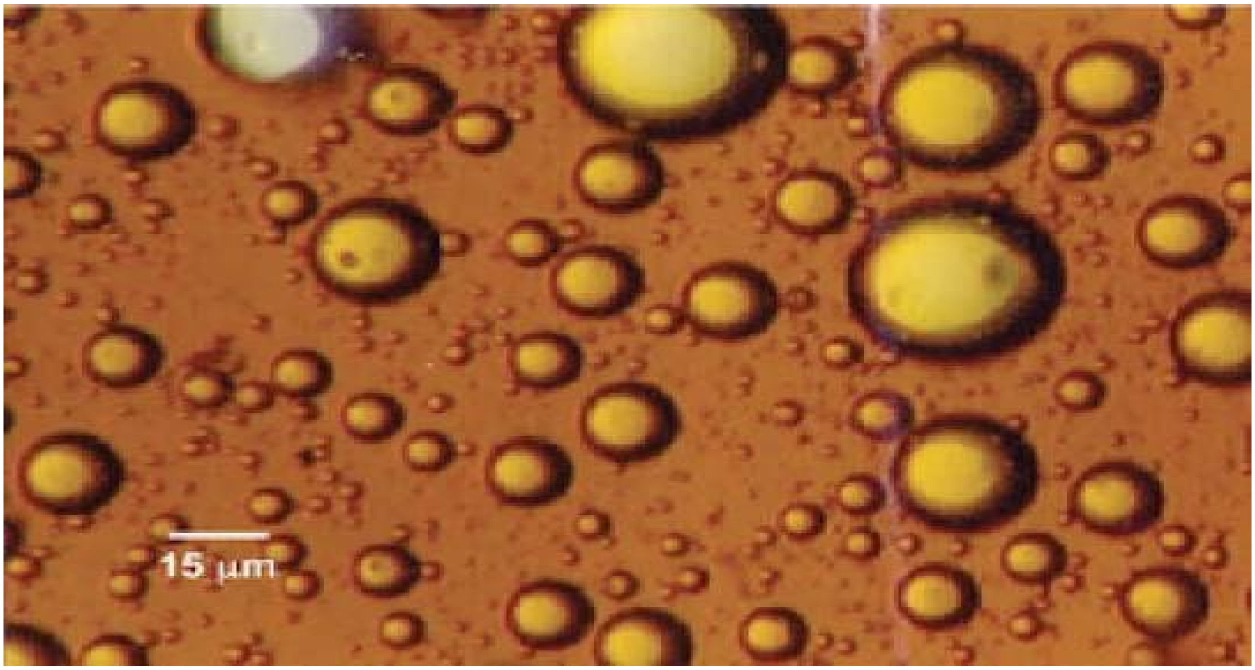


Figure 2. 1 photomicrograph of water in oil emulsion (kimray inc,2025)

Oil- in- water emulsions consists of oil droplets at a water-continuous stage. with oil now being the dispersed or internal stage.

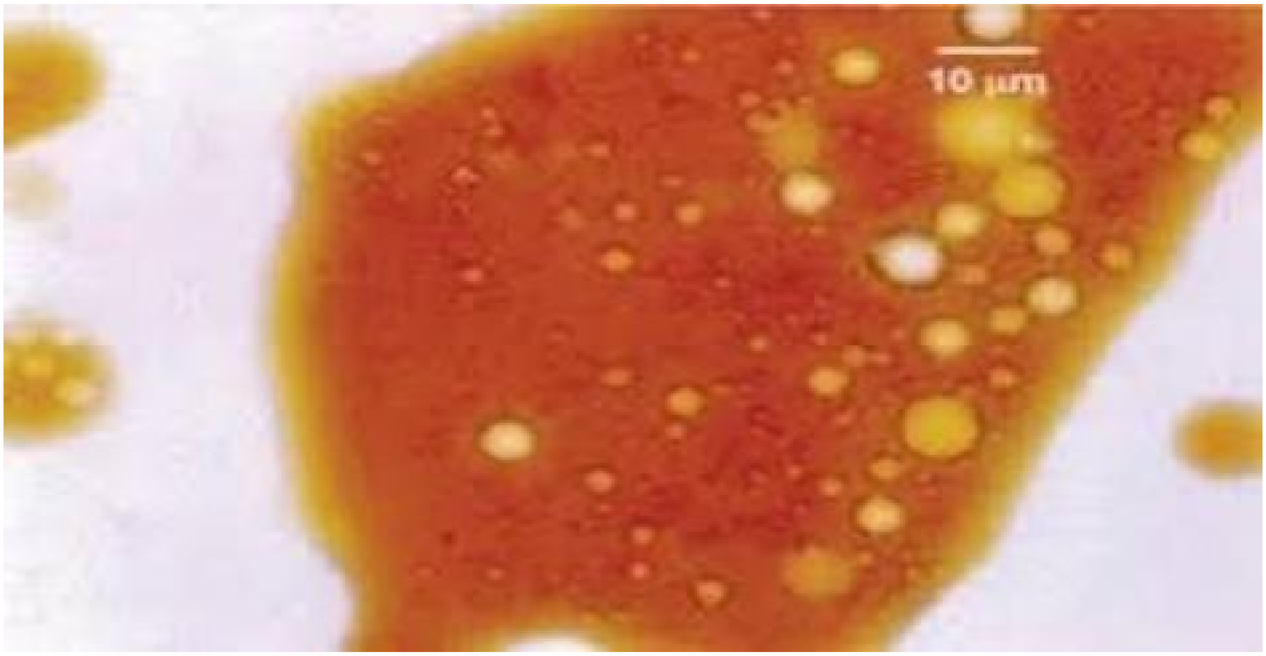


Figure 2. 2photomicrograph of oil in water emulsion (kimray inc,2025)

Combination of water - in - oil and oil - in - water are multiple emulsions. These are small droplets that are suspended by larger droplets suspended in a continuous phase. Giving the case of water-in-oil-in-water emulsion, in which the droplets are made up of water, enclosed in droplets of oil, which in turn are enclosed in a continuous water phase

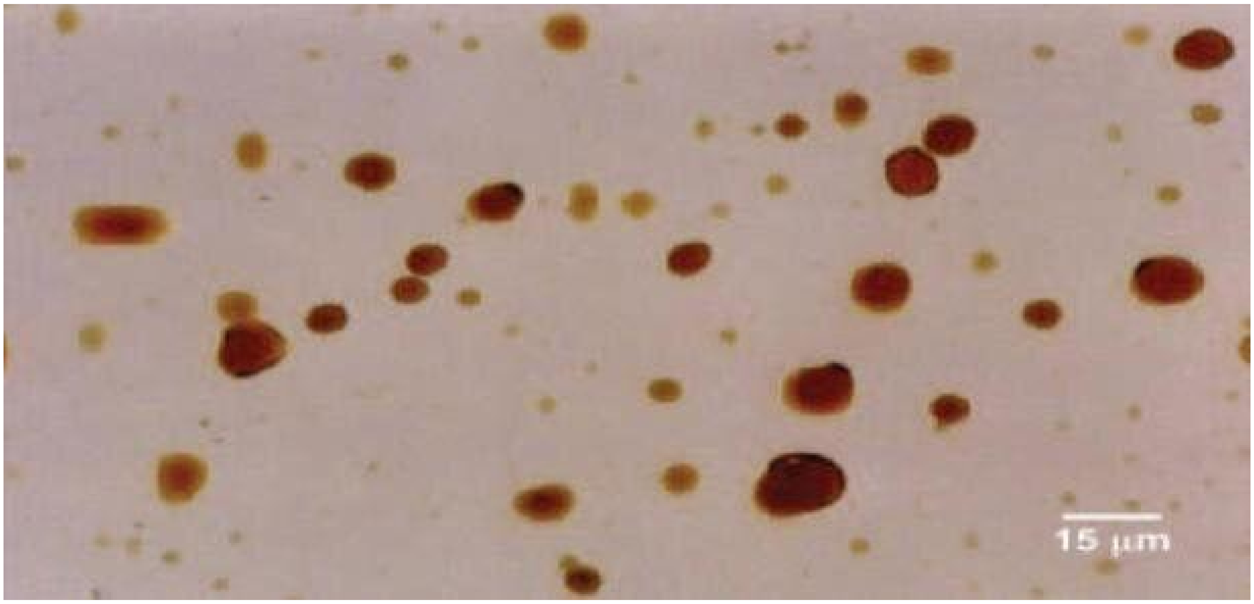


Figure 2. 3 photomicrograph of an Oil -water emulsion (kimray inc,2025)

2.2 FORMATION OF EMULSIONS

The process of creating emulsions is based on the separation of a liquid phase into another one, which also often needs mechanical energy to overcome the interfacial tension between the immiscible phases. This is enabled by the availability of surface-active agents, emulsifiers that adsorb on the oil-water interface, decreasing interfacial tension and holding the droplets together.

The following are the three requirements to emulsion formation;

i. The liquids that make up the emulsion have to be immiscible.
ii. Agitation is required to make a droplet of one liquid in another.
iii. It has to have an emulsifying agent.

When oil and water (or brine) come in contact with each other, as long as they are mixed sufficiently and the presence of an emulsifying agent or surfactant exists, crude oil emulsions are produced. The extent of mixing and accessibility of emulsifiers are also important in the process of the formation of emulsions.

In the course of production of crude oil, mixing occurs through a number of processes, which are often referred to as shear and they may include;

- Flow through reservoir rock
- Flow across bottom hole perforations.
- Flow through pumps
- Flow through tubing
- Flow lines

- Production headers, valves, fittings and chokes.
- Surface equipment

2.3 EMULSIFIERS

These define to a great degree the nature and strictness of an emulsion. Resident in the heavy fractions are the natural emulsifiers in crude. Emulsifiers are those materials that are used when combining two liquids that cannot interact normally, i.e. oil and water.

They are based on the principle that both liquids get low in surface tension, which permits one to be mixed uniformly in the other (Dammak et al., 2020). The substances are commonly referred to as surface-active agents or surfactants. Emulsifiers may either be ionic, nonionic, or natural depending on their structure (McClements, 2021).

Asphaltenes and resins are natural emulsifiers, which are important in stabilizing water-in-oil emulsions in the crude oil systems. These large molecules stick to the oil-water interface and create strong films that stop the drop of water from coming together (Abdulredha, 2018). This renders the emulsion highly stable and hard to part. This stability is determined by the composition of crude oil, specifically the quantity of asphaltenes, resins, aromatics, and saturates (Mohammadpour et al., 2023; Peng et al., 2025).

There are several methods of how emulsifiers can stabilize an emulsion: by reducing the interfacial tension, by creating strong films on droplets, or by providing droplets with an electrical charge stopping their repulsion (Faisal et al., 2022). These activities prevent the emulsions, but they also complicate the process of crude oil treatment, and on this account, demulsifiers are exploited to destroy them.

Over the last few years, the nature of emulsifiers and demulsifiers has changed with a move towards natural or plant-based extracts like clove, orange peel, neem and coconut oil. They are favored since they are more environmentally safe, bio-degradable, and renewable (Dammak et al., 2020; Research Gate Review, 2025). Nevertheless, some of them can be more effective depending on the crude oil type and the test-specific conditions, so additional research is required to enhance their performance and stability (Faisal et al., 2022; Peng et al., 2025)

These emulsifying Agents may be grouped as;

- Surface-active agents
- Finely divided solids

2.3.1 SURFACE-ACTIVE AGENTS

Also referred to as surfactants, surface-active agents are substances that have both hydrophilic and hydrophobic segments, rendering them partly soluble in water and oil. Due to this duality, they will concentrate at the oil-water interface, and thus will form thin interfacial films. Interfacial(IFT) is minimized in these types of films and droplets are easily dispersed, which facilitates the creation of stable emulsions. In the crude oil systems, some of the naturally produced high boiling components serve as emulsifiers.

These includes;

- Asphaltenes and resins
- organic acids
- Bases

These compounds have been found in most oilfield emulsions to be the main components of interfacial films that surround dispersed water droplets. The stabilizing effect that asphaltenes have on these interfacial layers has been widely covered in the literature on stability of crude oil emulsions. Moreover, other surfactant components might also be present in the chemicals brought to the reservoir or wellbore in the production operation, and they may further promote the formation of emulsion and maintenance of emulsions, some examples are;

- Drilling fluids
- stimulation chemicals
- corrosion inhibitors
- wax
- Asphaltene control agents.

2.3.2 FINELY DIVIDED SOLIDS

The fine solid particles are commonly used as mechanical stabilizers. These particles are normally very small compared to the droplets of the emulsion (often in the submicron size range) and accumulate at the oil-water interface, and are partially wetted by both phases. Their stabilizing capacity of emulsions is based on various aspects such as particle sizes, particle-particle and particle-wettability properties of their particles.

Typical finely divided solids that are used in oil production are;

- Asphaltenes and waxes
- Drilling mud
- Corrosion products

- Clay particles
- Sands
- Silt

2.4 OILFIELD EMULSIONS CHARACTERIZATION

Oilfield emulsions particularly the water-in-oil (W/O) type mainly used in crude production are multiphase complex systems the properties of which have a very strong impact on their stability, treatability as well as flow behavior (Kokal 2005).

Oilfield emulsions have various features which include; appearance and color, BSW, droplet size, and rheology.

i. **Appearance and Color** The qualitative characteristics of the oilfield emulsions are frequently considered to be the visual appearance and the color of the oilfield emulsions. Emulsions can have dark reddish-brown, grey, or blackish-brown color (and the choice of oils to use, and the ratio of water dissolved) make a significant difference (e.g., in an emulsified water and suspended solids). The appearance may be lightened or dulled by higher water content or relatively more suspended solids. It may also cause phase separation or color change through oxidation or deposition of solids as a result of changes with time (aging).

ii. **Basic Sediment and Water (BSW)** One of the most important diagnostic indicators of the tightness of an emulsion or the ability to remove the non-oil phase is the amount of the so-called basic sediment and water (BSW) also known as bottom solids and water. Practically, the slightest overdose of a demulsifier can be dosed, and the system can be centrifuged, after which the aqueous and solid layers have been measured in special centrifuge tubes (i.e. graduated tubes or glassware designed to do so) (Sunil & Al-Juraid 1999). The BSW is an indication of the degree of solids and free water that can be removed under treatment and is therefore a measure of the stability of an emulsion or how tight an emulsion is (i.e., tighter emulsions produce less BSW).

iii. **Droplet Size and Droplet Size Distribution**. Droplet size (or diameter) and distribution throughout the dispersed (water) phase is one of the most significant microstructural measures of emulsions. Droplet sizes in oilfield emulsions can be as small as 0.1 um (or smaller) to many tens of um (or larger), depending on the conditions in which the oilfield formation occurs and the stabilizers used. Distribution is determined by interfacial tension (IFT), type and concentration of emulsifying agents, and solids (clays, asphaltenes, scales), and bulk fluid characteristics (viscosities of oil and brine). Examples include smaller droplets (less diameter) which are usually due to increased shear, emulsifier coverage, and interfacial films that are stronger resisting coalescence (Review on fundamental aspects). The size of droplets and their distribution is commonly determined by microscopic and image analysis (optical microscopy, occasionally with image processing) or methods that rely upon electrical conductivity, dielectric response or light scattering (e.g. dynamic light scattering, laser diffraction). Crude droplets are sometimes separated by physical means (e.g. fractionation).

As an example, the smaller droplets (reduced diameter) tend to be formed by increased shear, greater coverage of the emulsifiers, and increased resistance of interfacial films to coalesce (Review on fundamental aspects). To measure the size and distribution of droplets, microscopic and image analysis (optical microscopy, with or without image processing) or methods with regards to electrical conductivity, dielectric response or light scattering (e.g. dynamic light scattering, laser diffraction) are commonly utilized.

Crude droplets are sometimes separated by physical means (e.g. fractionation). Droplet size distribution impacts the stability considerably and rheological behavior: the tighter the distribution (i.e. less deviated droplet sizes) the more stable the emulsions and the more predictable the rheology tend to be (L'Estime et al. 2023). In very concentrated emulsions, the scale effect on elastic modulus and viscosity depends on droplet size variations (Slatter 2004).

iv. **Rheology** The study of flow and deformation of the emulsion to the applied stresses or shear is known as rheology in the context of emulsions. The correlation between shear stress and shear rate is in fact quantified, which assists in determining flow regimes (Newtonian, non-Newtonian, yield stress behavior) (e.g. in colloid/emulsion theory, and in oilfield emulsion literature). With low dispersal or low concentration, emulsions can be approximately Newtonian (viscosity seems to be independent of shear rate). Non-Newtonian behavior is prevalent as the concentration of dispersed droplets rises or the strength of interfacial films is high (viscosity is dependent on shear rate). The viscosity of the bulk emulsion is determined by the viscosity of the continuous (oil) phase and the water volume fraction, the droplet interactions and interfacial resistance (shells, films) (Sousa et al. 2020). The overall behavior is further altered by interfacial viscosity or film viscosity (i.e. the viscosity or elasticity of the interfacial film surrounding droplets) particularly in stiff films that are stabilized by asphaltenes or solid particles (Jones et al.

1978). In high droplet concentration emulsions (i.e. tight emulsions), elastic and yield stress behavior can occur: at low applied stress levels the emulsion will act more like a viscoelastic solid; at high applied stress levels the emulsion will flow. It has been noted that in concentrated W/O emulsions, scaling of the elastic modulus with reciprocal square of droplet size occurs (Slatter 2004). Furthermore, the emulsions can also exhibit shear-thinning (reduced viscosity with increased shear) or even rheopectic behavior (increased viscosity with increased shear) in particular concentrated systems in rheological experiments (Slatter 2004).

2.5 STABILITY OF EMULSIONS

Droplet size, the viscosity of the continuous phase and the existence of stabilizing agents are some of the factors that determine the stability of emulsions. Excessively, emulsions can be subject to destabilization mechanisms such as coalescence, flocculation, and creaming and this results in phase separation (Xiao, 2025).

Emulsions are a priori thermodynamically unstable systems, as the free energy of a system always prefers to assume a phase (i.e. a reduction in the interfacial area) instead of being dispersed (Xiao et al., 2025; Badruddoza et al., 2023). That is, in the absence of stabilizing forces, an emulsion will be more likely to return to distinct oil and water layers so that the total energy of the emulsion is reduced. Although this is their natural tendency to disintegrate, most emulsions exhibit kinetic stability: they remain in a dispersed state over a useful time and only become visibly segregated after a certain period (Xiao et al., 2025).

In reality, the kinetic stability of an emulsion relies on the slowness of the destabilizing processes in case the rates of coalescence and flocculation, Ostwald ripening, creaming, or sedimentation are low enough, the emulsion would seem stable under the regular storage conditions (Badruddoza et al., 2023; Akbari and Nour, 2018).

In that context, emulsions are occasionally categorized on the basis of how hard they are to disintegrate: loose emulsions, medium emulsions, and tight emulsions.

i. Loose emulsions are relatively weakly bonded and can exhibit early separation (e.g. droplets increasing in size, mild creaming).
ii. Medium emulsions are more resistant to change with time, but slower structural change can occur ultimately.
iii. The tight emulsions are the emulsions where destabilization is inhibited successfully during the long periods usually by strong interfacial films, viscosity, or steric/electrostatic repulsion on the droplet surfaces.

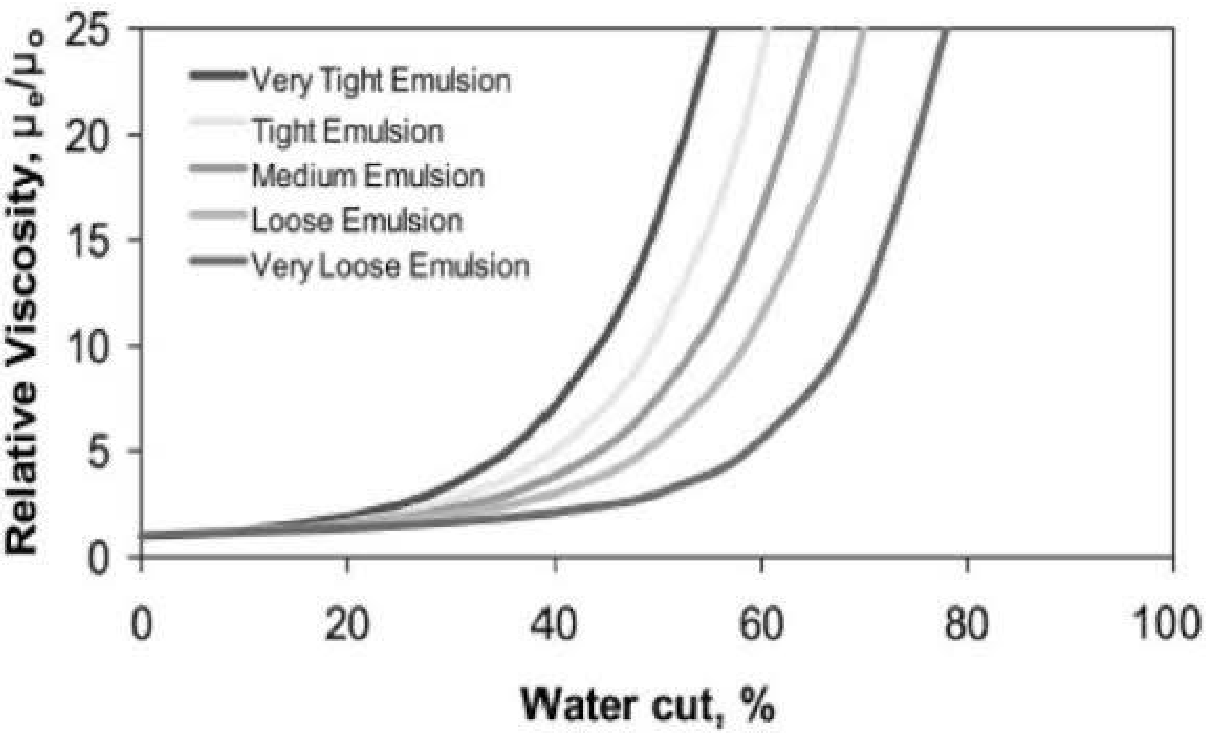


Figure 2. 4stability chart (springer nature link,2020)

The plot shows the correlation between relative viscosity **(μ_e/μ_o)** and water cut (%) of emulsions of different stability- very loose to very tight. The x-axis (water cut) is a ratio of the quantity of the water in the mixture and the y-axis (relative viscosity) is the degree to which emulsion becomes heavier than the base oil. The greater the water cut, the greater the amount of water droplets dispersed in the oil phase, resulting in a greater number of interactions between droplets and causing the viscosity to increase.

The various curves are the different degrees of droplet stability and interaction:

- Very loose emulsions exhibit a slow increase in viscosity, which means loosely held droplets, which easily coalesce and split apart easily.
- Loose and medium emulsions show moderate increases in viscosity indicating that there are more enduring interactions between droplets, yet there is poor structural stability.
- Strong interfacial films and dense droplet packing are indicated by sharp increases in the viscosity of tight and very tight emulsions even when cut with water (50-60%).

2.6 FACTORS AFFECTING THE STABILITY OF CRUDE OIL EMULSIONS

Various interacting physicals, chemical, and environmental factors determine the stability of crude oil emulsions by determining the length of time the dispersed droplets are able to resist the coalescence and separation of the droplets.

i. **Crude oil is composed of heavy polar fractions**. The surface-active, heavy polar, compounds that are naturally found in crude oil, including asphaltenes, resins, and waxes, significantly contribute to the stabilization of emulsions. Asphaltenes are high-molecular-weight, heteroatomic aromatic structures (containing heteroatoms, such as oxygen, nitrogen, and sulfur) of very high complexity. They are apt to be accumulated at the oil-water interface, to form strong, rigid films which inhibit coalescence of droplets. Another factor that has contributed to the high-molecular-weight polar compounds that contain oxygen, nitrogen and sulfur atoms is the resins. They are used together with asphaltenes to stabilize emulsions by enhancing the flexibility and viscosity of the films. The alkanes that are of a high molecular-weight are further stabilizing emulsions, as they increase the viscosity of the emulsions and in certain cases create crystalline networks that entrap water droplets.

ii. **Solids** The emulsions can also be stabilized by solid particles like clays, sand, corrosion products and mineral scales (e.g. CaCO3 and CaSO4). These solids travel towards the oil-water interface, and there they create hard impenetrable films around the droplets and stop them from consolidating. The wettability of their interaction with the two phases depends on their wettability: Solids greased with oil (such as asphaltenes and waxes) prefer the creation of water-in-oil emulsions. Solvents that are water-wet solids (e.g. CaCO3, CaSO4 and clays) facilitate the formation of oil-in-water emulsions.

iii. **Temperature** The level of temperature affects the physical properties of both liquid phases and the characteristics of surfactants in the interface so strongly. Rise in temperature tends to decrease the viscosity of the emulsion and interfacial films, facilitating coalescence and easy separation. Waxes may also be dissolved by heating or softened asphaltene films can further be reduced. Nevertheless, emulsions are more stable at low temperature due to the increase of the viscosity and interfacial strength.

iv. **Droplet-Size Distribution and Droplet Size**. One of the most important issues that determine the emulsion stability is the size of the dispersed droplets. Smaller droplet emulsions are more stable due to the fact that small droplets are less buoyant and are slower to coalesce. A small droplet size distribution also enhances viscosity and uniformity, which contributes to the more resistance of the emulsion to phase separation.

v. **pH of the Brine** The charge and structure of interfacial films is hugely dependent on the pH of the aqueous phase. Water-in-oil emulsions are usually formed in low pH (acidic) which causes the formation of oil wet interfaces. Basic conditions (high PH) are appropriate to oil in water emulsions, as alkaline conditions encourage water-wet interfaces.

vi. **Brine Composition** The formation water is also composed of different dissolved ions, including sodium, calcium and chloride which have a great effect on the emulsion behavior. These ions are able to react with the polar functional groups of the oil-water interface to create insoluble salts or insoluble, non-relaxing, films around the water droplets. The effect of such films is coalescence inhibition of droplets leading to high stability of emulsions.

2.7 EMULSION TREATMENT

Well drilled crude oil may have water, salts and other impurities in the form of stable emulsions. All these emulsions have to be treated and disintegrated in order to transfer, refine, or sell oil. Emulsion treatment is a process of extracting water and solid as well as crude oil in order to come up with clean, dehydrated as well as desalted oil, which meets the pipeline and refinery requirements.

The treatment process entails the destabilization and fracture of the interfacial films containing the droplets of water dispersed in oil. This can be achieved through;

- Chemical method
- Thermal method
- Electrical method
- Mechanical method

Chemical Method

This is by using demulsifiers which are agents that destabilize or displace the natural emulsifiers like asphaltenes and resins of the oil-water interface. Demulsifiers reduce interfacial tension and cause the small water droplets to coalesce into larger ones thus, making the separation process more efficient. Demulsifier selection and dosage is also determined by the crude composition, salinity of water and conditions of operations.

Thermal Method

Demonstrating the heat is the most frequent and efficient method to help demulsification. Increasing temperature decreases the viscosity of crude oil, increases the mobility of the droplets and dissolves the waxes and asphaltenes which stabilize the emulsion. Heat also reduces the strength of interfacial film wherein water droplets merge more readily and are separated by gravity.

Electrical Method

The electric field is used in electrostatic dehydrators to apply electric field on the emulsion causing the water droplets to be polarized. Core shell of oppositely charged droplets occurs due to the attraction of oppositely charged droplets resulting in quick separation. Treatment Electrical treatment is usually employed alongside chemical and thermal techniques in refineries and production plants.

Mechanical Method

In mechanical techniques, gravity settling, centrifugal separation, or filtration is applied to oil and water separation. In a normal separator or De-salters, the heavier water body will be at the bottom whereas the lighter oil will be on the top. This is enhanced with centrifuges and hydro cyclones that exert greater forces of separation.

2.8 DEMULSIFICATION

Demulsification refers to breaking of the crude oil emulsions into oil and water phases. In order to successfully break up the emulsion into oil and water, the interfacial films need to be torn or broken, and then the droplets in the dispersion would come together so that they can be separated out by gravity or other forces. This process can be done by revealing the interfacial barriers with chemical, thermal, electrical or mechanical methods all with the aim of increasing the rate of water removal.

2.8.1 OPERATING FACTORS THAT EFFECT DEMULSIFICATION PROCESS.

There are various parameters that affect the efficiency of demulsification in crude oil treatment. These are temperature, agitation or shear, retention time, removal of solids, control of emulsifying agents and retrofitting of equipment. All these parameters are significant in facilitating the process of oil-water separation and enhancing the overall efficiency of the process of treating.

One of the most important factors which contributes towards improving demulsification is temperature. Heating oil will facilitate the oil-water separation process by decreasing the viscosity of the crude oil and subsequently the water droplets will move freely and converge. The distinction in the densities between the oil and water layers is also enhanced by higher temperatures and makes the water droplets to settle more quickly and accelerates the separation process. Nevertheless, overheating may cause loss of light hydrocarbons and result in an increase in energy and operating costs and thus temperature should be optimized.

Demulsification efficiency is also subject to agitation or shear. Though mild agitation assists in distributing the demulsifying chemicals equally in the emulsion, over agitation may have the reverse effect in forming smaller droplets which are more stable and difficult to separate. The presence of high turbulence caused by chokes, valves or flow restrictions should be kept to a minimum since these conditions enhance an emulsifying process and also make it harder to separate the phases.

Retention Time Residency or treating time is the period of time that the emulsion stays in the treating piece of equipment at the desired conditions. The longer the retention time, the greater is the coalescence and settling time of the water droplets and the more efficient is the separation process and less water left behind in the treated crude. Most crude oils are usually retained in a period of 10 to 30 minutes, although more difficult emulsions might take longer. There is however, a trade-off associated with increasing retention time, which is increased equipment and operational cost.

Another factor that is critical to improve demulsification is solids removal. Dissolved substances like asphaltenes, waxes, clays and corrosion products are solid materials, which stabilize an emulsion by creating hard films on the oil-water interface to avoid a merging of droplets. Water-in-oil emulsions are stabilized by oil-wet solids and water-wet solids can be oil-wet through the addition of the heavy hydrocarbons which further stabilize the emulsion. The management of solids, whether by dispersion or by transforming solids into water-wet solids that can be easily eliminated with the aqueous phase, is an excellent way to increase the efficiency of separation.

Emulsifying agents also control is another very crucial factor in the demulsification process. Chemical additives may neutralize or replace natural emulsifiers like resins, asphaltenes and organic acids. Demulsifiers, dispersants or pour-point depressants are used to assist in weakening the interfacial film that surrounds the water droplets to coalesce. Also, it is possible to avoid mixing the incompatible crude oils, which may result in the creation of especially stable emulsions.

Modernizing the current treatment systems could lead to the improvement of the separation of water and the performance of the plant altogether. This can involve installation of other equipment like free-water knockout drums, heater treaters or coalescers sections which allows droplet coalescence and settling. These modifications will help facilities to attain better dehydration and preserve crude oil quality within reasonable specification. Demulsification is a phenomenon occurring in the presence of water.

2.8.2 MECHANISMS OF DEMULSIFICATION

A number of underlying phenomena lead to the change of state between a dispersed droplet state (in the breaking of crude-oil emulsions) and phase separation. These destabilization mechanisms act alone or in synergy to destabilize the interfacial films covering droplets and facilitate the coalescence, droplet movement, and, finally, oil-water separation (Abed et al., 2019; Alara, Abdurahman & Tade, 2022).

These are the major processes which are flocculation, coalescence, sedimentation, Ostwald ripening, creaming, and phase inversion.

Flocculation The weakly bound aggregates (flocs) of droplets formed under the influence of attractive forces by overcoming repulsive barriers. In crude emulsions this occurs when the electrostatic repulsion decreases due to altered ionic strength, or pH, or as a result of solids/bridging agents which promote droplet adhesion; flocculation causes the droplets to approach one another and thereby raise the probability of rupture and coalescence of the film. Flocculation does not instantly form large droplets but the distance and time to coalesce is shortened, therefore it is commonly an initial stage in the breakdown sequence.

Case study (petroleum): Field tests showed very rapid flocculation of high-salinity, fines of fine mineral crude oil, and when demulsifiers were added, the speed of downstream coalescence increased, enhancing water recovery but giving rise to turbid sludge that needed solids management (Abed et al., 2019).

Coalescence The irrevocable fusion of two drops into a larger drop is known as Coalescence which occurs when the thin film of liquid between droplets breaks. Weakened interfacial films (e.g. following the adsorption of demulsifier), higher temperature (reducing viscosity and film strength) and high collision rates promote it. Coalescence decreases total interfacial area, it is the most crucial process that transforms a dispersed emulsion into separable bulk phases, the faster the coalescence process, the faster the separation process.

Case study (petroleum): It has been found that laboratory and plant experiments indicate that carefully chosen polymeric demulsifiers significantly enhance the rate of coalescence in heavy crudes by displacing asphaltene-rich movies; several industrial experiments are described in which the time of coalescence was reduced by more than half after an optimum dosing of the demulsifier and moderate heating (Alara et al., 2022).

Sedimentation defines the process of settling down of heavier dispersed elements on the bottom of a vessel due to gravity. Water droplets (which are denser than oil) can sink in most oilfield emulsions, which facilitates the separation; however, lighter droplets of oil or gas can also rise. The rate of sedimentation is determined by the size, the difference in density and the continuous phase viscosity (Kind of Stokes-like behaviour in small droplets). Very fine droplets can be very slow to settle by sedimentation, so coalescence or some other improvement is normally required.

Case study (petroleum): When performing low-temperature pipeline pigging activities, operators observed that it was more effective to rotate the flow without increasing flow velocity temporarily to enable sedimentation in the separator tanks, but because of their tiny size, sedimentation would not be possible without chemical coalescers that produced these droplets (Abdulredha et al., 2018).

Ostwald ripening The slow accumulation of large droplets at the cost of small droplets due to the larger Laplace pressure and solubility is known as the Ostwald ripening where the small droplets are more chemically active and the higher chemical potential forces the material to diffuse and redeposit on bigger droplets. This process slowly extends the droplet-size distribution and may destabilize long-term emulsion even with coalescence inhibited. Ostwald ripening occurs particularly in the case of systems where the continuous phase is in which the dispersed phase is partially soluble.

Case study (petroleum): Experiments with the light oil constituents of the produced water demonstrate that compositional ripening (a form of Ostwald ripening) can play a major role in condensate-rich oils; the addition of low oil solubility oil fractions or suitable substances to the water increased the rate of ripening and enhanced the long-term separation (see Processes/MDPI review, 2023).

Creaming refers to the vertical movement of droplets by buoyancy: droplets that are less dense than the continuous phase ascends (cream) whereas those that are heavier descend. Creaming brings the dispersed phase into a layer that makes it easier to coalesce since the droplets are brought closer and the film will be drained between the droplets more rapidly. Creaming is not a separating process but it forms an environment in which coalescence and bulk separation is more effective.

Case study (petroleum): In waxy crude storage tanks, seasonal cooling of the oil raised its viscosity and allowed oil-rich layers to rise to the surface, operators applied gentle heating and coalesce cartridges to break the cream layer and enable the uniform separation of the oil (Abed et al., 2019; Abdulredha et al., 2018).

Phase inversion is a drastic conversion of the structures, in which the dispersive and continuous phases exchange (W/O = O/W). Changes in water cut, surfactant concentration, temperature, shear may cause this; inversion is frequently accompanied by sudden viscosity and separation behavior changes. The presence of phase inversion may facilitate or suppress demulsification based on downstream equipment and specification needs, and therefore predicting and avoiding inversion is significant in the operations.

Case study (petroleum): Transient phase inversion events occurred on Deepwater commingled streams, with rapid rises in the cut of water, which overwhelmed the separators, adjusting demulsifier strategies adaptively and using staged separation units to keep the process steady (Alara et al., 2022).

2.8.3 WHY PETROLEUM INDUSTRY NEEDS TO BE DEMULSIFIED?

Demulsification is a very critical process in the petroleum operations as it aids.

i. Do not overload surface separation equipment.
ii. Reduce pressure buildup.
iii. Reduce corrosion and catalyst poisoning.
iv. Reduce the levels of basic sediment, water and salt.
v. Cut operational costs

2.9 DEMULSIFIERS

These are chemical substances that serve to increase the rate at which water and crude-oil emulsions are separated by destabilizing, displacing or otherwise disturbing the interfacial films that are the natural stabilizers of dispersed water droplets in oil.

As a matter of fact, the selection and design of demulsifiers are determined by the makeup of crude (particularly the existence of asphaltenes, resins and fine solids), water chemistry (salinity and divalent ions), temperature as well as the kind of emulsion (water-in-oil is most prevalent in produced crude) (Abed, Kamil, and Abdurahman, 2019).

The following is the classification of Demulsifiers: Demulsifiers are widely categorized according to chemical structure, ionic nature and mechanism of action. They are designed to destabilize crude oil emulsions by replacing natural stabilizers at the interface of the oil-water boundary, destabilizing interfacial films, and coalescing water droplets.

2.9.2 CHEMICAL DEMULSIFIERS

Chemical demulsifiers are synthetic surfactants or polymeric substances that are specially designed to rupture crude oil emulsions by lowering the interfacial tension and interfering with the stabilizing film created by asphaltenes, resins and other species that are surface-active. They are the most useful type of demulsifier used in the petroleum industry because they are efficient, flexible and can be blended to suit particular types of crude.

The ionic nature and mode of action allow classifying chemical demulsifiers into nonionic, anionic, cationic, and amphoteric (Abed et al., 2019; Alara et al., 2022). The most popular demulsifiers in crude oil treatment are nonionic since they are free of charge, and can be employed on a broad variety of crude systems regardless of pH and salinity. Examples of the typical ones are polyethylene oxide-propylene oxide (PEO-PPO) copolymers, polyether's, and alkyl phenol ethoxylates.

The mechanism of action of these compounds is mainly a reduction in interfacial tension and removal of stiff asphaltenes or resin film which holds water droplets together. They usually work better at elevated temperatures so that they would be useful in separation processes at high temperatures (Efendy et al., 2006; Wang et al., 2024).

Anionic demulsifiers include the sulfonates, carboxylates and phosphates as negatively charged functional groups. They are particularly effective in the dispersement of oil-in-water emulsions which contains mostly negatively charged droplets. They are not as stable in highly saline or acidic environments as they are under neutral conditions because their performance is decreased by the formation of ion-pairs, which restricts their efficiency (Alara et al., 2022).

Cationic demulsifiers are those which have positively charged functional groups, such as amines, and quaternary ammonia salts. They work especially well in acidic media, in which they neutralize the negative-charged water droplets and facilitate coalescence. They should however be employed with care since they will lead to corrosion and can react with other anionic surfactants within the system. They are commonly used in certain crude oils, which have acidic polar matter or where electrostatic stabilization prevails (Abed et al., 2019).

Zwitterionic demulsifiers or amphoteric demulsifiers contain both negative and positive functional groups on the same molecule. The dual charge enables them to accommodate changes in pH and salinity that enable them to operate over a broader range than the purely ionic demulsifiers. They tend to be eco-friendlier and flexible but are less widespread in the market because of the increased cost of production (MDPI, 2023).

2.9.3 NATURAL AND BIO-BASED DEMULSIFIERS

Natural demulsifiers/ bio-based demulsifiers refer to surfactant-free and surfactant-based systems that employ organic matter to create a water-oil separation, or oil-to-water separation. The increasing environmental issues have resulted in studies on green demulsifiers which can be obtained through renewable resources like plant extracts, bio surfactants and modified polysaccharides. Compared to synthetic formulations, these materials are biodegradable and not as toxic, as well as possess competitive demulsification capabilities, and thus can be used as an alternative to synthetic ones (Alara et al., 2022; Abed et al., 2019).

Natural and plant-based substances have received increasing attention as green demulsifiers of crude oil emulsions due to their biodegradability and low toxicity and possible close sourcing advantage over traditional synthetic surfactants. Recent reviews and experimental works indicate that bio based demulsifiers obtained with vegetable oils, essential oils and agro-waste extracts are capable of destabilizing water-in-oil emulsions by adsorbing on the oil-water interface, replacing interactive species in the interface (e.g., asphaltenes and resins) and accelerating the coalescence of water droplets (Al-Humaidi et al., 2024). In these studies, a technique is often applied as Fourier Transform Infrared Spectroscopy (FTIR) to determine the functional groups present in the natural extracts (e.g. -OH, C=O, and aromatic groups) and to measure chemical changes in the oil phase following treatment, which can then be linked to changes in the characteristics of the demulsification behavior

2.9.3.1 CLOVE AS A DEMULSIFIER

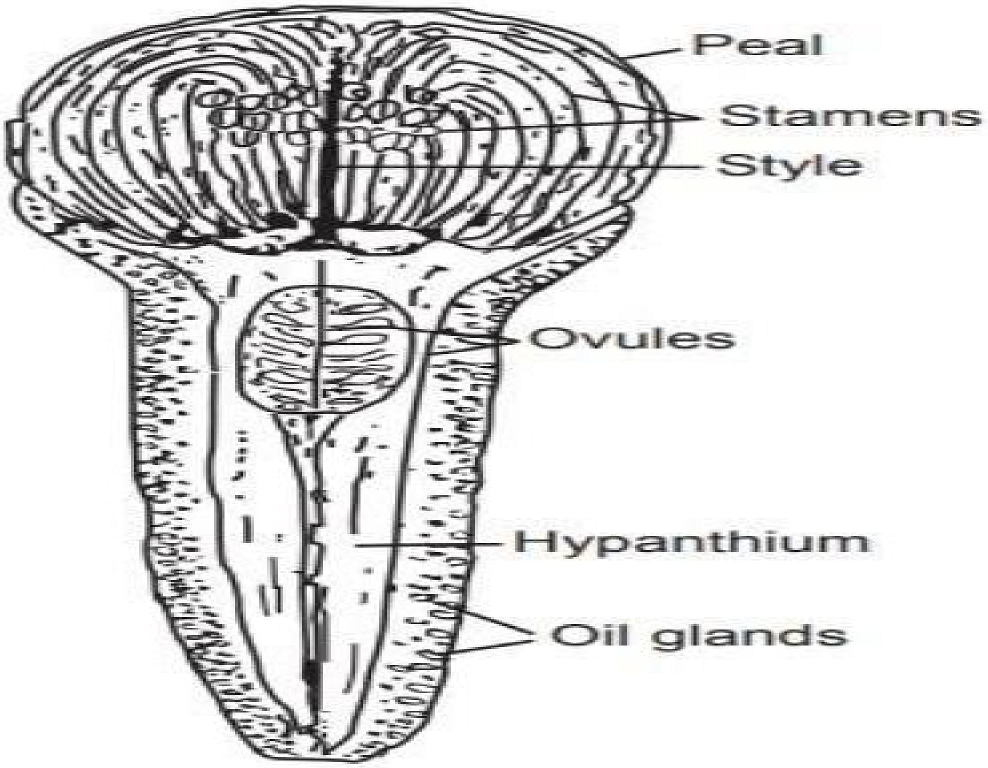


Figure 2. 5clove

Clove or Syzygium aromaticum also known as clove, is an aromatic spice belonging to the Myrtaceae family that has been long-known in culinary and medicinal applications. As the native of the Maluku (Spice) Islands of Indonesia, clove has the essential oils, which make it have its unique smell and bioactive attributes.

In recent research it has also been pointed out that it can be used as a natural demulsifier of crude oil emulsions. Clove oil contains phenolic compounds the most significant of which are Eugenol and other substances such as Acetyl Eugenol b-Caryophyllene a-Humulene Gallic Acid which constitute a considerable portion of the composition (Sharma et al., 2022). These phenolic and aromatic components indicate a possibility of how clove oil may help in the demulsification of water-in-oil emulsions including those in the crude oil industry. When added to a crude oil emulsion, the clove oil phenolic molecules can adsorb at the oil-water interface and compete or destabilize rigid interfacial films of asphaltenes, resins or fine solids.

These adsorbed phenolic have the potential to permit dispersed water droplets to approach and drain their film in between droplets, coalesce and be separated to form a critical pathway in demulsification by changing the interfacial tension and destabilizing the droplet coating films. In addition, since most of the clove oil extracts are moderately lipophilic and phenolic this can enable them to be located at the interface between the non-polar phase of the oil and the aqueous droplet surface.

2.9.3.3 COCONUT OIL AS A DEMULSIFIER

Figure 2. 6coconut(google amazon)

Coconut oil or Cocos nucifera is a potential green demulsifier of the crude oil emulsions. Its special chemical structure and surfactant-like properties. Coconut oil is composed mainly of triglycerides; which contain fatty acids of medium-chain length such as lauric, capric and captrylic acids that give it its amphiphilic properties: hydrophobic hydrocarbon tails and hydrophilic ester/carboxyl groups. Such characteristics of molecules allow coconut oil to align at the oil-water interface, lower the interfacial tension, and destabilize the stabilizing films surrounding the water droplets in crude emulsions. Indicatively, in a Nigerian crude study, coconut oil was found to be better than the triethanolamine in breaking a 40 per cent water-in-oil emulsion at a dosage of 10 per cent (Akinyemi et al., 2023). The results indicate that the surfactancy-like fatty acid molecules in coconut oil in combination with its functional alterations (betaine/amine groups) is what makes the coconut oil demulsifiable by replacing natural crude stabilizers (asphaltenes, resins), enhancing droplet coalescence, and facilitating phase separation. Another benefit of using coconut oil is that it is biodegradable and can be locally available in the tropical regions where oils are produced and this can save on costs and ensure that it is in line with environmental policy, which requires the use of green chemistry. Although additional data on a larger scale is required, there is an increasing amount of literature that validates the promise of coconut oil as a natural demulsifier (when it comes to treating crude oil) (Wang et al., 2024; Xu et al., 2023).

2.9.3.3 ORANGE PEEL AND BANANA PEEL

Figure 2. 7 orange & banana peel (alamy.com)

Orange (citrus) and banana peels contain bioactive substances (e.g., fatty acids, natural surfactants (e.g., saponins, pectins), phenolics and other amphiphilic molecules) that can interact at oil-water interfaces. An example here is the exploration of orange peel extracts as bio based emulsion breakers in the oilfield setting: it was demonstrated that orange mesocarp extract surpassed conventional separants by a significant margin due to its surface-active functional groups when used on simulated oil-water emulsions (Victor-Oji et al., 2025).

The oil-water interface mechanism in this instance is adsorption of amphiphilic molecules in the extract to the interface to decrease interfacial tension and consequently cause phase separation and coalescence of water drops. Also, the banana peel has been observed to work as an oil sorbent in produced water: cellulose, hemicellulose, pectin, as well as functional groups (e.g., hydroxyl, carboxyl) found in the banana peel can give the peel affinity to oil droplets; one study reported nearly perfect oil removability at a specific dose (El-Nafaty et al., 2023). Although that is not so much demulsification as sorption as such, the presence of amphiphilic and functional-group rich materials would imply that the banana peel may play a role in weakening interfaces or adsorbing the stabilizing species (e.g., asphaltenes/resins) within a crude emulsion.

Should a combination of orange peel extract and banana peel be used as a demulsifier, the thinking would be that the combination will exhibit complementary properties: the citrus peel extract would offer classic surfactant-like behavior (through limonene, pectin, flavonoids) of reducing interfacial tension, whereas the banana peel would offer solid-phase functionalized components (cellulose, pectin, lignin) that will adsorb stabilizing solids or particles which strengthens the interfacial film. They could act together on several stabilization mechanisms: the amphiphiles are pushed off or weakened by the asphaltene/resin films; the peel-derived solids or functional groups could be binding with fines or precipitating ions; the combined mixture could allow more tenability of the hydrophile-lipophile balance (HLB) in the crude emulsion environment.

Moreover, since the two peel extracts are renewable, inexpensive and biodegradable, they are also favored by the current trend in green demulsifiers that have minimal environmental impact than their synthetic petroleum based chemical counterparts. This especially applies to other areas such as Nigeria where banana and orange peels (among other agricultural wastes) are in plenty and readily available locally.

2.9.4 DIESEL AS A MIIXING FLUID

In laboratory tests, diesel is commonly used as the continuously present oil phase or mixing fluid, since it is a convenient commercial hydrocarbon whose physical characteristics (density, viscosity, hydrocarbon chain length) can be used to reproducibly form emulsions and compare the performance of demulsifiers (Abdulredha, 2020). Doing an experiment on emulsion stability, mixing dynamics, and the efficacy of treatments, researchers tend to use diesel or other diluent oils, as such systems can be used to approximate the behaviour of a lighter crude oil in a controlled environment (Faisal, 2022). Moreover, diesel-based model emulsions can be used in place of field-carried demulsifier systems, because commercial preparations may have an oil-soluble carrier which can be injected into the crude pipeline. With this said, lots of authors warn that data collected using diesel do not contain the heavier polar components (resins, asphaltenes) of many actual crude oils-and thus might be indicative of early screening conditions, but not complete field behaviour (Abdulredha, 2020; Faisal, 2022). Therefore, consideration of limitation should be made when screening natural plant-based demulsifiers with diesel mixing fluid and validation in the real crude oil system should be taken into consideration to guarantee its field irrelevance.

2.9.5 MAGNET EFFECT ON DEMULSIFIER

The effect of natural and magnetic strategies on the performance of demulsifiers of water-in-oil emulsions is that both magnetic fields and magnetic demulsifiers modify the behaviors of droplets at the oil-water interface and enable a quick phase separation, not to mention that magnetic demulsifiers can be easily removed by a magnet (Romanova et al., 2019; Yau et al., 2020). Recent researches indicate that engineered magnetic demulsifiers - such as magnetite-based composites, or fluorinated magnetic nanoparticles that can be recycled under an external magnetic field, and enhance demulsification efficiency and reuse than some conventional chemicals (Yau et al., 2020; Liu et al., 2024). Individually, metal cations (Fe3+, Al3+, Ca2+, Mg2+ etc.) may destabilize or stabilize emulsions based on ion type, ion concentration and pH with multivalent metal ions, such as Fe3+ and Al3+, commonly increasing droplet aggregation or acting synergistically with coagulants to facilitate separation (Zhang et al., 2021). The hybrids consisting of magnetic material and specific chemistries can confer both magnetic carrier benefits (administer interfacial active groups to droplet surface) and field benefits (speed up collection) and recent papers have shown high water-removal efficacies and high recycleability of hybrids (Liu et al., 2024; Guo et al., 2022). To recap it all, demulsifier performance is affected by both magnetic methods and targeted heavy-metal ions to change interfacial chemistry, droplet coalescence kinetics, and the feasibility of recovery, hence these parameters are significant when comparing natural plant-based demulsifiers with magnetic or metal-assisted methods (Romanova et al., 2019; Yau et al., 2020; Zhang et al., 2021).

CHAPTER THREE

MATERIALS AND METHODS

3.1 MATERIALS AND APPARATUS USED

3.1.1 CHEMICAL AND BIOLOGICAL MATERIALS

The Crude oil sample used for this experiment was collected from a flow station in Delta State.

Tween 20 was gotten from chemical shop at the university of Benin which was used as an emulsifying agent to prepare stable water-in-oil emulsion.

Biological materials which were plant derived materials includes; neem leaf, clove seeds, coconut, banana and orange was gotten from Ekosodin market in Benin City, Edo State

3.1.2 MATERIALS AND EQUIPMENT USED IN THIS STUDY

Table 3. 1Materials used

S/N	MATERIALS	USE
1	Crude Oil sample	To test formulated demulsifier
2	Cloves, neem leaf, coconut, banana and orange peel	Serves as natural demulsifier precursor
3	Ethanol	Solvent for extractor
4	Deionized water	For preparing standard solutions

5	Detergent	emulsifying agent to prepare stable water-in-oil emulsion.
6	pH buffer solutions	To adjust the pH of demulsifier
7	Diesel	Used as a mixing fluid

Table 3. 2Equipments used

S/N	EQUIPMENTS	SPECIFICATION	USE
1	Beakers and Flasks	50ml, 100ml, 250ml, 500ml and 1000ml Pyrex glass	For mixing and holding of samples
2	Hot plate magnetic Stirrer	MS300 Jinotec Scientific, Guandong, China	To heat and mix solution thoroughly
3	Centrifuge	4000 RPM	To accelerate the separation of emulsified layers
4	Micro-Pipette	Volume Range: 0.1µL to 1000µL	To transfer small volumes of liquids accurately
5	Graduated	10ml, 50ml, 100ml	To measure the volume of

	Cylinders	Pyrex glass	separated water and oil
6	Stop watch	Digital 1/100 sec accuracy	To keep track of time during testing process
7	Soxhlet extractor	Borosilicate glass, 1000ml capacity	Used for extraction of plant oil
8	Magnet		Used for testing demulsifer efficiency

3.2 METHODS

3.2.1 COLLECTION AND PREPARATION OF CRUDE OIL SAMPLE AND PLANT DERIVED MATERIALS

3.2.1.1 COLLECTION OF CRUDE OIL SAMPLES

The steps involved in the collection of crude oil sample were the following:

Container Selection: The container used was a clean 1.5-liter container so that there would be no contamination of the sample during the process.

Sampling Procedure: The crude oil was sampled at the well head so that the sample could be representative of the crude oil.

Transportation: The samples that were collected were transported in a secure and safe way to the Petroleum Engineering Laboratory at the University of Benin to be analyzed further.

crude oil samples

3.3 The Crude Oil Characterization

The crude oil emulsions were characterized to find their main physical and chemical properties which are necessary in the study of their behavior during the process of emulsions and demulsifications.

Measured parameters were as the following using standard laboratory procedures:

Specific Gravity (@ 35°C)

Procedure: The particular gravity of the samples of crude oil was measured with the help of a hydrometer. The samples of the crude oils were gradually poured in clean graduated cylinders and the hydrometer inserted into the sample. The reading was made at 35°C the specific gravity being directly read off the hydrometer scale.

Flash Point (°C)

Procedure: The flash point was decided by the method of using a pensky-martens closed-cup tester. The sample of crude oil was carefully poured in the test cup and heated slowly. The flash point was recorded as the temperature at which the sample produced vapors which briefly ignited when exposed to a test flame.

Basic Sediment and Water (BS&W) (mg/L)

Procedure: The centrifuge method was used to establish the BS&W content. Each sample of the crude oil was put in centrifuge tubes with known volume and centrifuged at very high speed over a given period. The two layers of water and sediment were then measured and the BS&W content computed and entered in mg/L.

Cloud Point (°C)

Procedure: A cloud and pour point apparatus were used to find the cloud point. The samples of the crude oil were cooled down gradually under controlled conditions and the temperature at which the first indications of cloudiness were observed was recorded as the cloud point.

Pour Point (°C)

Procedure: A pour point tester was used to measure the pour point. The crude oil was allowed to cool down slowly and the minimum temperature at which the crude oil was found to flow was marked as the pour point.

Viscosity (@ 35°C, mm^2/s)

Procedure: Kinematic viscometer was used to determine the viscosity of each sample of the crude oil. The sample was heated to 35degC and a time which the oil took to pass through the viscometer capillary tube was noted. The instrument calibration constant was used to calculate the viscosity and that was in mm 2/s.

Salinity (wt.%)

Procedure: A salinity meter was used to find the salinity of individual samples of the crude oil. A little part of the crude oil emulsion was tested and the content of dissolved salts in the sample was determined and taken in weight percent (wt.%).

3.3.1 CRUDE OIL ANALYSIS FOURIER TRANSFORM INFRARED SPECTROSCOPY(FTIR).

Materials:

Crude oil samples (A, B, and C)

FTIR spectrometer (4000-400 cm-1 range)

ATR (Attenuated Total Reflectance) pellet crystal or KBr.

Micro pipette/glass rod (of sample application)

Procedure:

Sample Preparation: A small portion of each crude oil sample (A, B and C) was laid on the ATR crystal of the FTIR spectrometer with the help of clean micropipette or glass rod. Instrument Set up: FTIR spectrometer was calibrated and a background scan was carried out using a clean ATR crystal or blank KBr pellets to provide correct baseline correction. The scanning range was fixed at 4000-400 cm-1.

Data Acquisition: A series of scans were done on each sample of the crude oil to ensure the sample gave a clear spectrum. The tool was used to measure the percent transmittance verses wavenumber (cm -1).

Spectral Analysis: FTIR spectra of the crude oil samples were measured and used to determine the presence of functional groups present in the crude oil namely: alkanes (C-H stretching), aromatics (C=C), carbonyls (C=O), and sulfur-containing compounds. The existence of these peaks and their strength were used to give data about the chemical composition and the structure of the molecular information of the crude oil.

3.4 PREPARATION OF PLANT DERIVED SAMPLES

Sampling and preparation of the plant samples to undergo extraction was done in an orderly manner to avoid compromise of the bioactive compounds that are in the samples. Plant materials used in this study were clove seeds, neem leaves, coconut, banana and orange peels. All the samples were freshly collected at a local market at Benin City, Edo State, Nigeria. Samples were selected on the basis of freshness, aroma and physical quality. Only undamaged materials that had no apparent defect, decay or fungal growth were used.

The samples chosen were put into clean dry containers to avoid the absorption of the moisture during the process of transportation to the laboratory. On reaching the laboratory all the samples were vigorously washed with clean water to eliminate dust and impurities i.e. clove seeds, neem leaves, coconut, banana and orange peels.

The wet samples were then dried by air while some oven so as to remove any surface moisture and then prepared.

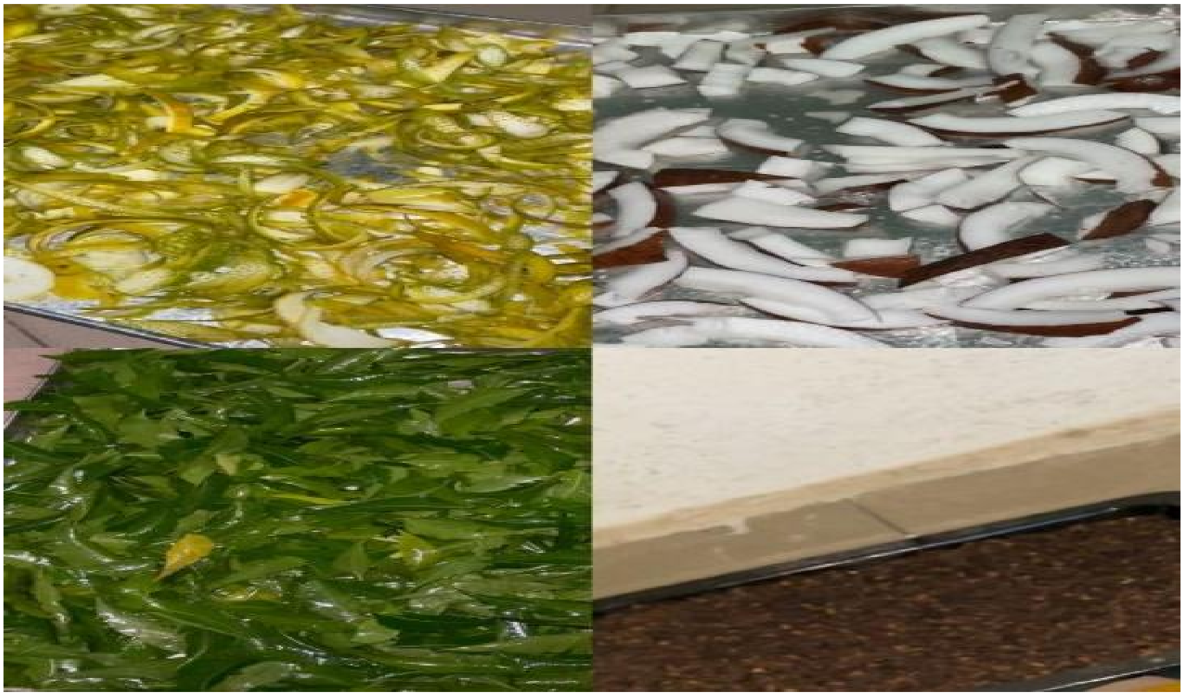


Figure 3. 1: dry plant samples

In case of the banana-orange peel mixture, the two materials were mixed together to increase the overall efficiency of the extract because the two materials have bioactive compounds that are known to demulsify.

A mixture of banana (80 g) to orange peel (260 g) was used to prepare the mixture. The rest of the samples were measured separately in the following manner: clove (240 g), coconut (320 g), and neem leaves (280 g). The individual plant samples were then ground into fine particles to ease the solvent penetration in the extraction process, so as to obtain optimum essential oil and active constituents.

Preparation was done in the following steps:

Drying: the fresh clove seeds, neem leaf, coconut, banana and orange were washed and then piled on clean trays and dried in a shaded place with adequate air flow. This is necessary because it decreases the amount of moisture, which is imperative in extraction of bioactive compounds.

Grinding: Fresh clove seeds, the neem leaf, coconut, banana and orange were completely dried and then crushed into a fine powder using a mechanical grinder. This enhances the surface area and this makes extraction of essential oils and other bioactive materials easier.

Storage: this powdered clove seed, neem leaf, coconut, banana peel, orange peel was stored in an airtight container and stored in a dry, cool environment such that the aromatic qualities of the substance were not destroyed prior to extraction.

3.4.1 EXTRACTION OF CLOVE, NEEM, COCONUT, BANANA AND ORANGE PEEL OIL USING SOXHLET APPARATUS

The oils that were extracted were clove, neem, coconut, banana and orange peel and were performed one by one in the same process with a Soxhlet apparatus of 1000 ml.

The reason why the Soxhlet setup was used is that it can be used to continue extraction of compounds in solid substances using an appropriate solvent. Ethanol was chosen as the solvent used in the extraction process because it is a good solvent of many organic compounds. The equipment used was a distillation flask, a thimble, a reflux condenser, heater mantle and a retort stand.

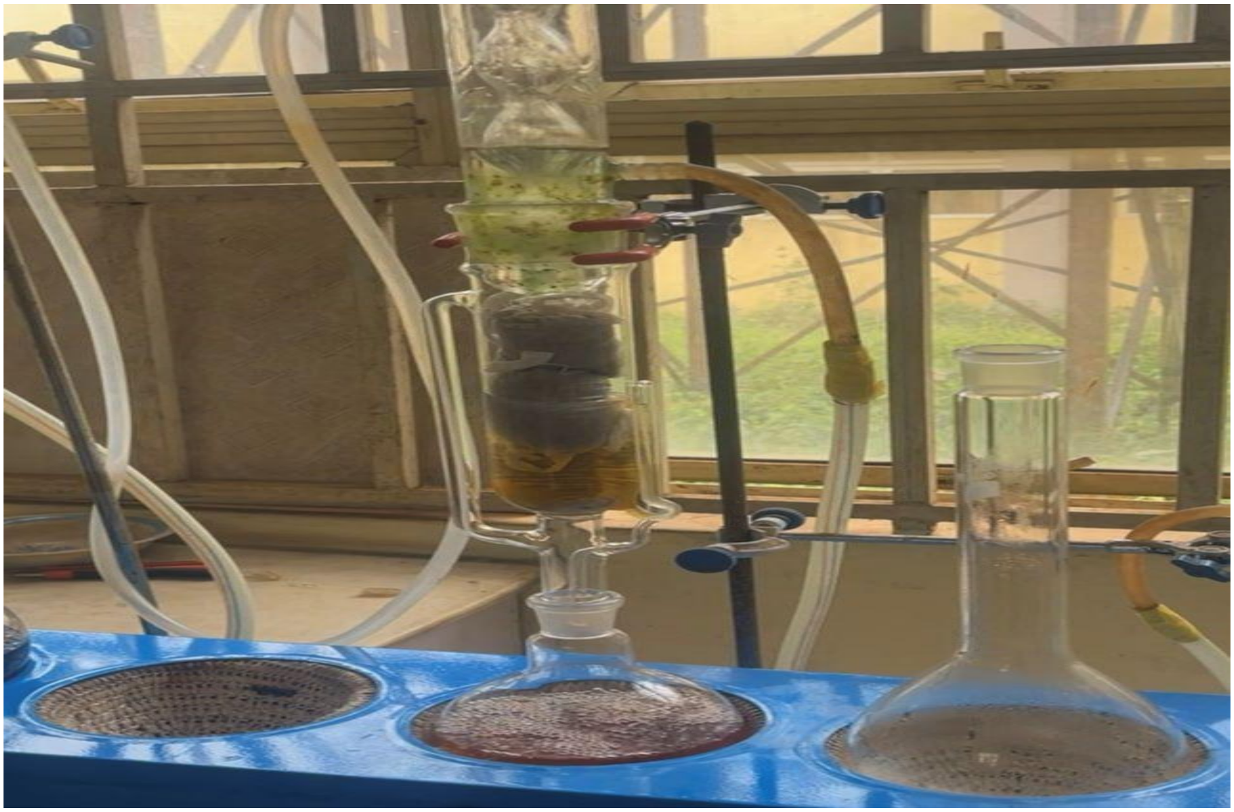


Figure 3. 2: soxhlet extractor

In every extraction, the sample of the plant (clove, neem, coconut, or banana & orange powder) was put into the thimble and was tied in a porous sack. Ethanol was added to the flask of distillation and its heating was performed with the heating mantle. The ethanol was vaporized and ascended up into the distillation arm and into the thimble chamber with the sample. The reflux condenser cooled the vapor of the ethanol and the ethanol condensed and dripped back into the thimble chamber. The solvent was the condensed solvent and was used to dissolve the oil and other soluble compounds of the plant material gradually. Once all of the chamber was filled, a siphoning effect was caused to pump the liquid back into the distillation flask, leaving the undissolved material. This heating, vaporization, condensation and extraction process was repeated several times over several hours so that efficient oil extraction would take place.

Once the extraction of each sample had been done, the mixture contained in the distillation flask was put in an oven at 60°C to dry up any remaining ethanol in the mixture, which took three hours. The dried extract was put in a clean labeled bottle to be analyzed further. This process was also carried out individually on clove, neem, coconut, banana and orange peel in order to produce their respective oils under the same conditions, to make all the extractions consistent.

Figure 3. 3: extracted oil

3.5 DEMULSIFIER CHARACTERIZATION

3.5.1 PLANT-BASED OIL EXTRACTION FTIR ANALYSIS OF FOURIER TRANSFORM INFRARED SPECTROSCOPY

Materials:

Clove oil extract

Neem oil extract

Coconut oil extract

Banana-orange extract

FTIR spectrometer (4000-400 cm-1 range)

ATR (Attenuated Total Reflectance) pellet crystal or KBr.

Micropipette (to apply to the sample)

Procedure:

Sample Preparation: A small amount of each oil extract (drop) was put directly on the ATR crystal of the FTIR spectrometer. Instrument Set up: The FTIR spectrometer was prepared by running a background scan using a clean ATR crystal or blank KBr pellet. The scanning was adjusted to 4000400 cm-1 in order to scan the entire range of infrared. Data Acquisition: Every sample was repeatedly scanned to get clear and stable spectra. Analysis was done on the system by recording the percent transmittance versus wavenumber (cm-1). Spectral Analysis: The FTIR spectra obtained were analyzed to determine specific absorption peaks that are typical of functional groups (O-H, C=O, C-H, C-O) that are found in the plant oil extracts, which point to the bioactive compounds that do the demulsification.

Table 3. 3Infrared Spectroscopy Absorption Table

Wavenumber (cm^{-1})	**Intensity**	**Shape**	**Bond**	**Vibration Type**	**Compound / Functional Group**	**Remarks**
3700–3584	Medium	Sharp	O–H	Stretching	Alcohol	Free (non-H-bonded)
3550–3200	Strong	Broad	O–H	Stretching	Alcohol	Intermolecular H-bonded
3500–3400	Medium	–	N–H	Stretching	Primary amine	Two bands
3400–3300	Medium	–	N–H	Stretching	Aliphatic primary amine	–
3350–3310	Medium	–	N–H	Stretching	Secondary amine	One band
3300–2500	Strong	Broad	O–H	Stretching	Carboxylic acid	Centred ~3000 cm^{-1}
3200–2700	Weak	Broad	O–H	Stretching	Alcohol	Intramolecular bonded
3000–2800	Strong	Broad	N–H	Stretching	Amine salt	–
3333–3267	Strong	Sharp	≡C–H	Stretching	Alkyne	–
3100–3000	Medium	–	=C–H	Stretching	Alkene	–

3000–2840	Medium	–	–C–H	Stretching	Alkane	–
2830–2695	Medium	–	–C–H	Stretching	Aldehyde	Doublet (two weak bands)
2600–2550	Weak	–	S–H	Stretching	Thiol	–
2349	Strong	–	O=C=O	Stretching	Carbon dioxide	–
2275–2250	Strong	Broad	N=C=O	Stretching	Isocyanate	–
2260–2222	Weak	–	C≡N	Stretching	Nitrile	Sharp
2260–2190	Weak	–	C≡C	Stretching	Alkyne	Disubstituted
2175–2140	Strong	–	S–C≡N	Stretching	Thiocyanate	–
2160–2120	Strong	–	N=N=N	Stretching	Azide	–
2150	–	–	C=C=O	Stretching	Ketene	–
2145–2120	Strong	–	N=C=N	Stretching	Carbodiimide	–
2140–2100	Weak	–	C≡C	Stretching	Alkyne	Monosubstituted
2140–1990	Strong	–	N=C=S	Stretching	Isothiocyanate	–
2000–1900	Medium	–	C=C=C	Stretching	Allene	–
2000	–	–	C=C=N	Stretching	Ketenimine	–
2000–1650	Weak	–	C–H	Bending	Aromatic compound	Overtone region

1760–1690	Strong	Sharp	C=O	Stretching	Carbonyl compounds	Depends on type
1680–1620	Medium	–	C=C	Stretching	Alkene	–
1600–1475	Medium	–	C=C	Stretching	Aromatic ring	–
1550–1350	Strong	–	N–O	Stretching	Nitro compounds	Two strong bands
1450–1375	Medium	–	C–H	Bending	Alkane	CH_2, CH_3 deformation
1360–1290	Medium	–	O–H	Bending	Carboxylic acid	–
1300–1000	Strong	–	C–O	Stretching	Alcohol, ester, acid	–
1250–1020	Medium	–	C–N	Stretching	Amine, amide	–
900–675	Medium	Sharp	=C–H	Out-of-plane bend	Aromatic/alkene	Diagnostic for substitution pattern
700–600	Strong	–	C–Cl	Stretching	Alkyl halide	–
600–500	Strong	–	C–Br	Stretching	Alkyl halide	–
500–400	Strong	–	C–I	Stretching	Alkyl halide	–

Source: Adapted from LibreTextu Chemistry- Infrared Spectroscopy Absorption Table (2023) and Complementary IR Reference Data.

The peaks of each from each sample were compared with this table to know the functional groups present in them.

3.6 PREPARATION OF STABLE CRUDE OIL EMULSION

The samples of crude oil were taken out of three different wells which were labeled as Sample A, Sample B and Sample C. The 500 mL of crude oil was measured and in each well, a separate emulsion was made. All the samples were treated in the same emulsification process as well as mixing ratios to enable reasonable comparison of the emulsion stability. detergent was first dissolved in distilled water to prepare the produced water. The total volume of produced water was 500 mL that was prepared by using a spatula to measure the surfactant and mixing it with 495mL of distilled water in a mL beaker. The mixture was mixed with the help of magnetic stirrer over a period of 10 minutes in order to fully mix and evenly distribute the surfactant.

Preparation of Crude Oil Emulsion Each crude oil sample contained 70mL of crude oil and 30mL of produced water that was prepared and measured into 250 mL conical flask. To begin with the dispersion, the mixture was stir manually with the help of a glass rod during 2-3 minutes. Then it was mixed with a mechanical stirrer at 2000 revolutions per minute (rpm) to get a similar and stable emulsion after 10 minutes. The emulsification reaction was done at room temperature (25 ± 2 °C) As the mixture was shaken, surfactant molecules were adsorbed at the oil-water interface and decreased the interfacial tension and preserved the water droplets in the continuous oil phase, creating a stable water-in-oil (W/O) emulsion. Following homogenization, the emulsion (Samples A, B and C) was then transferred into independent labeled glass bottles and left to sit without any stirring after 1 hour. Visual observation of each emulsion was carried out to determine the stability of the emulsion to identify any indications of phase separation, creaming or coalescence during the observation time.

Figure 3. 4: crude oil emulsion

3.7 BANANA-ORANGE PEEL OIL EXTRACT USABLE VISCOSITY PREPARATION

It was noted that the banana and orange peel oil extract (80 g :260 g) was very thick and therefore, it was hard to work with and mix with the crude oil. A dilution mixture of diesel was added to the mixture to achieve a lighter more workable consistency. Diesel was used because of its low viscosity, compatibility with the hydrocarbon system and able to make the extract thin without interfering with the chemical activity of the extract. The dilution was done slowly by adding diesel in small amounts as the mixture was stirred in between to result in homogenous and pourable oil phase. The mixture that was obtained was kept in a closed container in a room temperature to perform later demulsification experiments.

3.8 DEMULSIFICATION EXPERIMENT

After the preparation of the crude oil emulsions, 10 mL of these emulsions was measured into four separate and well labeled graduated cylinders, which acted as a representative of the various experimental conditions. In the first cylinder, there was just the emulsion and plant-based demulsifier (control treatment). The demulsifier was diluted in diesel and put in the second cylinder to decrease the viscosity. The third cylinder was filled with the demulsifier but it was mounted on the magnet that was placed on the bottom to check whether it was affected by magnetism or not. The fourth cylinder was a combination of both conditions, i.e. diesel-diluted demulsifier and magnetic placement. In all therapies, the demulsifier was added at three dosage concentrations (50 uL, 100 uL, and 150 uL) and transferred with the help of a micropipette. Once added, the timer was activated and all the cylinders were stirred in gently to create a homogenous mixture of the demulsifier in the emulsion. The cylinders were then left to rest and observed at 10 minutes' intervals over a total of 70 minutes' period when phase separation was noted and recorded in terms of visual clearness and volume of water recovered.

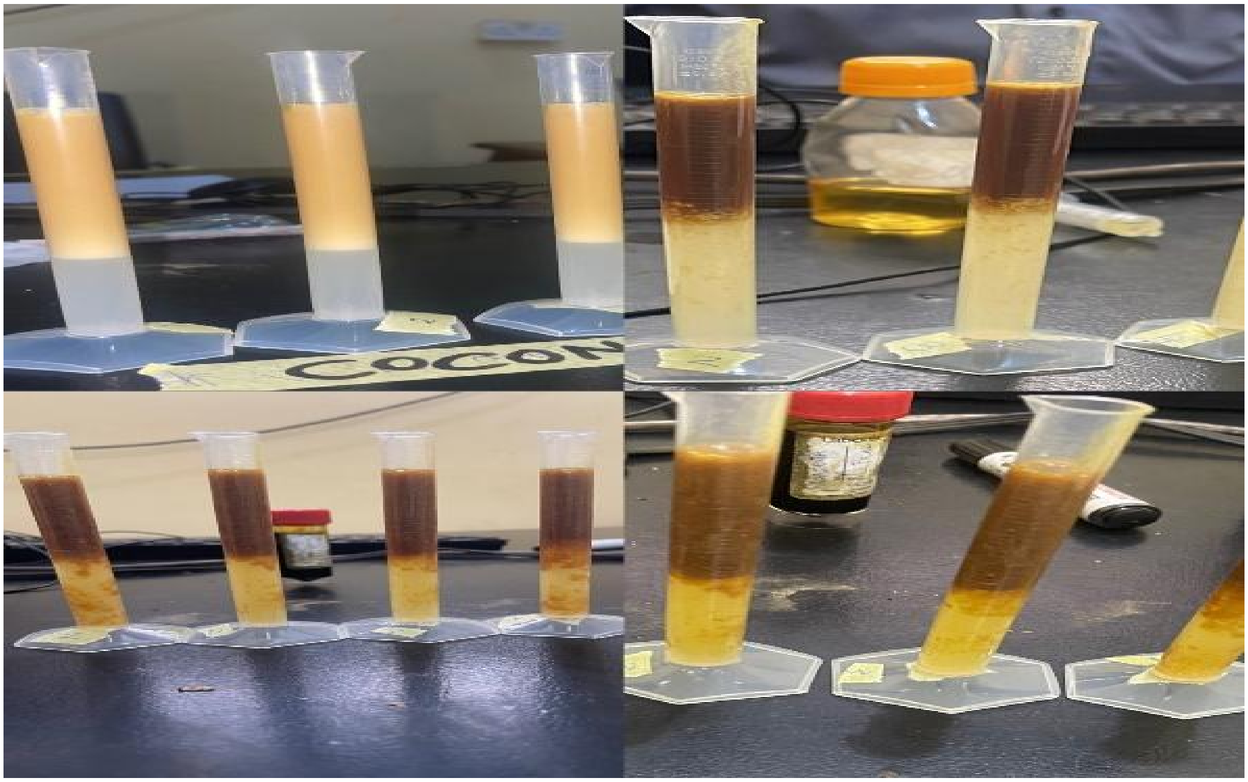


Figure 3. 5: bottle test

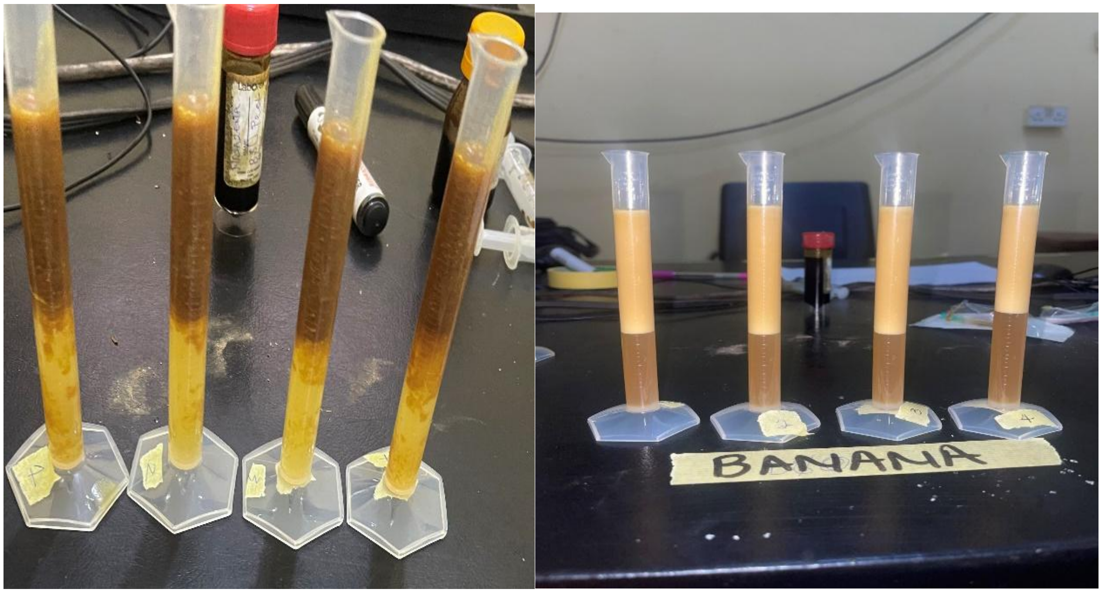


Figure 3. 6: bottle text experiment

CHAPTER FOUR

RESULTS AND DISCUSSIONS

Table 4. 1THE CRUDE OIL CHARACTERIZATION

Parameter	**Value well A**	**Well B**	**Well C**
Specific Gravity @ 35°C	0.8612	0.9205	0.7904
Flash Point (°C)	44	65	32
Basic Sediment and Water (Mg/l)	0.2	0.4	0.1
Cloud Point (°C)	0.6	6.2	-2.8
Pour Point (°C)	-3	3	-9
Viscosity @ 35°C (mm/s^2)	**3.4**	**11.8**	**2.1**
Salinity (wt.%)	**0.26**	**0.39**	**0.18**

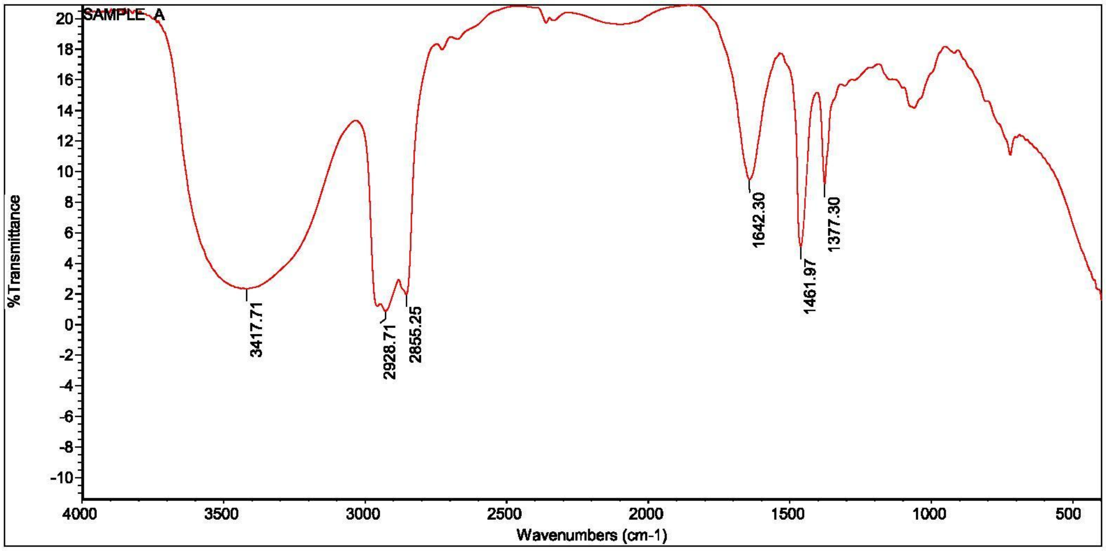
The crude oil properties were assessed to understand the stability of the emulsion, flow behavior, and how easily it can be separated. Each parameter influences the effectiveness of demulsification and the role of natural demulsifiers.

Table 4. 2Crude oil properties

Well	API (approx.)	Type of Crude	Flow Behavior	Wax Tendency
A	~33	Medium, Light,	Low viscosity	Moderate
B	~22	Heavy	High viscosity	High
C	~47	Very light / condensate,	Very low viscosity	Low

Based on the determined physical characteristics, the three crude oil samples of Wells A, B, and C can be characterized based on specific gravity, viscosity, pour point, and other parameter-indicating values. Well A that has a specific gravity of 0.8612 is estimated to have API gravity of about 33° making it fall in the classification of a light crude oil. Its reasonably low viscosity (3.4 mm2/s) and a pour point of -3°C indicates that it is not very waxy and is easily transported at fairly low temperatures and indicates a fairly paraffinic, but still mobile crude. On the other hand, the Well B has a significantly higher specific gravity of 0.9205, which corresponds to API gravity of about 22° hence is classified as a heavy crude oil. The very high viscosity (11.8 mm2/s) and a positive cloud point (6.2°C) and pour point (3°C) imply a large amount of wax, and low flow ability at low temperatures, and it is a heavy and waxy paraffinic crude that would either need heating or additives to process and to transport. Well C having specific gravity of 0.7904, gives an estimated API gravity of approximately 47°, which is on the margin of very light crude oil, which borders on condensate. Its viscosity is very low (2.1 mm2/s) and the lowest pour point of the samples (-9°C) indicates good flow properties and small formation of wax. This causes Well C to be more naphthenic, and has low paraffin content and high mobility. Thus, Well A, Well B and Well C are light crude, heavy oil and very light or condensate oil respectively.

4.2.1 FTIR RESULT FOR CRUDE OIL SAMPLE A



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FIND PEAKS:
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     Sensitivity:       50
     Peak list:
                         Position:   1377.30   Intensity:    9.185
                         Position:   1461.97   Intensity:    5.132
                         Position:   1642.30   Intensity:    9.489
                         Position:   2855.25   Intensity:    1.948
                         Position:   2928.71   Intensity:    0.876
                         Position:   3417.71   Intensity:    2.333
```

Figure 4. 1crude oil sample A

*Table 4. 3*Parameter analysis

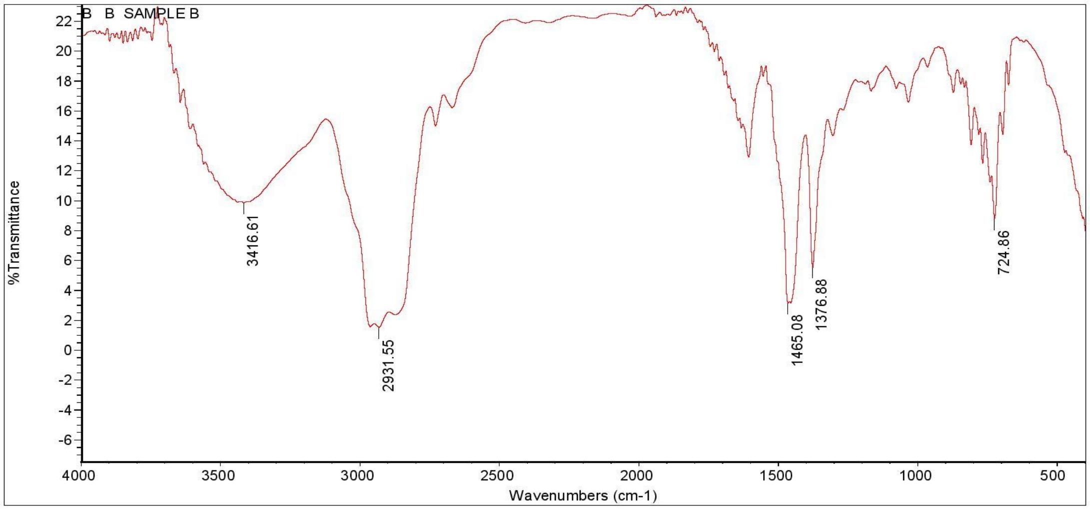
Parameter	**Value**	**Meaning**
Spectrum	SAMPLE A	The name given to the specific FTIR measurement/sample.
Region	4000.00 - 400.00	The range of wavenumbers (cm^{-1}) scanned for peaks. This is the standard mid-infrared range.
Absolute threshold	10.880	The minimum absorption intensity (often percentage transmittance or absorbance) required for a point to be flagged as a peak by the

		software.
Sensitivity	50	A parameter controlling how strictly the software looks for peaks (e.g., how narrow or isolated a feature must be to be counted).

*Table 4. 4*Peak List Analysis

Position	Intensity (likely)	Shape (likely)	Bond	Vibration	Functional group / compound
1377.30	Strong	-	N-O	Stretching	Nitro compounds
1461.97	Strong	-	N-O	Stretching	Nitro compounds
1642.30	Medium	-	C=C	Stretching	Alkene
2855.25	Medium	-	-C-H	Stretching	Alkane
2928.71	Medium	-	-C-H	Stretching	Alkane
3417.71	Medium	-	N-H	Stretching	Primary amine

4.2.2 FTIR RESULT FOR CRUDE OIL SAMPLE B



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     Peak list:
                              Position:     724.86     Intensity:     8.787
                              Position:    1376.88     Intensity:     5.511
                              Position:    1465.08     Intensity:     3.139
                              Position:    2931.55     Intensity:     1.508
                              Position:    3416.61     Intensity:     9.857
```

Figure 4. 2crude oil sample B

Table 4. 5 Parameter Analysis

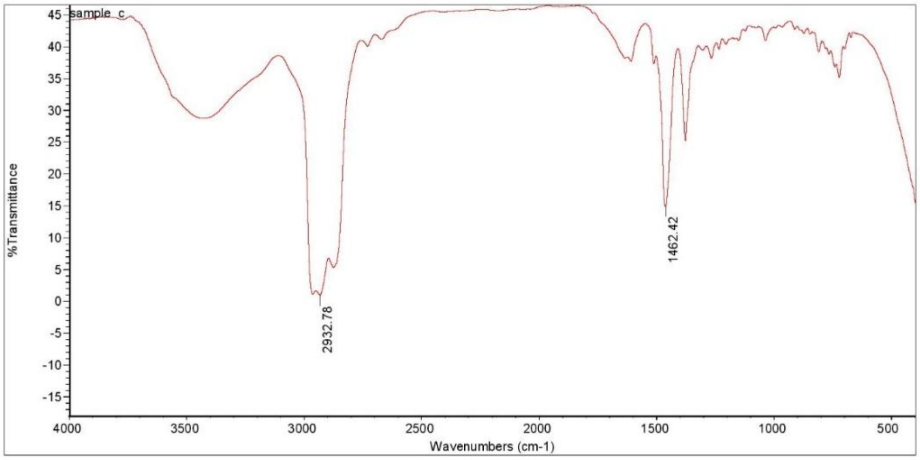
Parameter	**Value**	**Meaning**
Spectrum	SAMPLE B	The name given to the specific FTIR measurement/sample.
Region	4000.00 - 400.00	The range of wavenumbers (cm^{-1}) scanned for peaks. This is the standard mid-infrared range.
Absolute	12.298	The minimum absorption intensity (often percentage transmittance or absorbance) required for a point to be flagged as a peak by the

threshold		software.
Sensitivity	50	A parameter controlling how strictly the software looks for peaks (e.g., how narrow or isolated a feature must be to be counted).

*Table 4. 6*Peak List Analysis

Position	Intensity (likely)	Shape (likely)	Bond	Vibration	Functional group / compound
724.86	Medium	sharp	=C-H	Out of plane bend	Aromatic / alkene
1376.88	Medium	-	C-H	Bending	Alkane
1465.08	Strong	-	N-O	Stretching	Nitro compounds
2931.55	Medium	-	-C-H	Stretching	Alkane
3416.61	Medium	-	N-H	Stretching	Primary amine

4.2.3 FTIR RESULT FOR CRUDE OIL SAMPLE C



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    Region:             4000.00      400.00
    Absolute threshold: 23.711
    Sensitivity:        50
    Peak list:
                        Position:   1462.42   Intensity:   14.881
                        Position:   2932.78   Intensity:    0.926
```

Figure 4. 3crude oil sample C

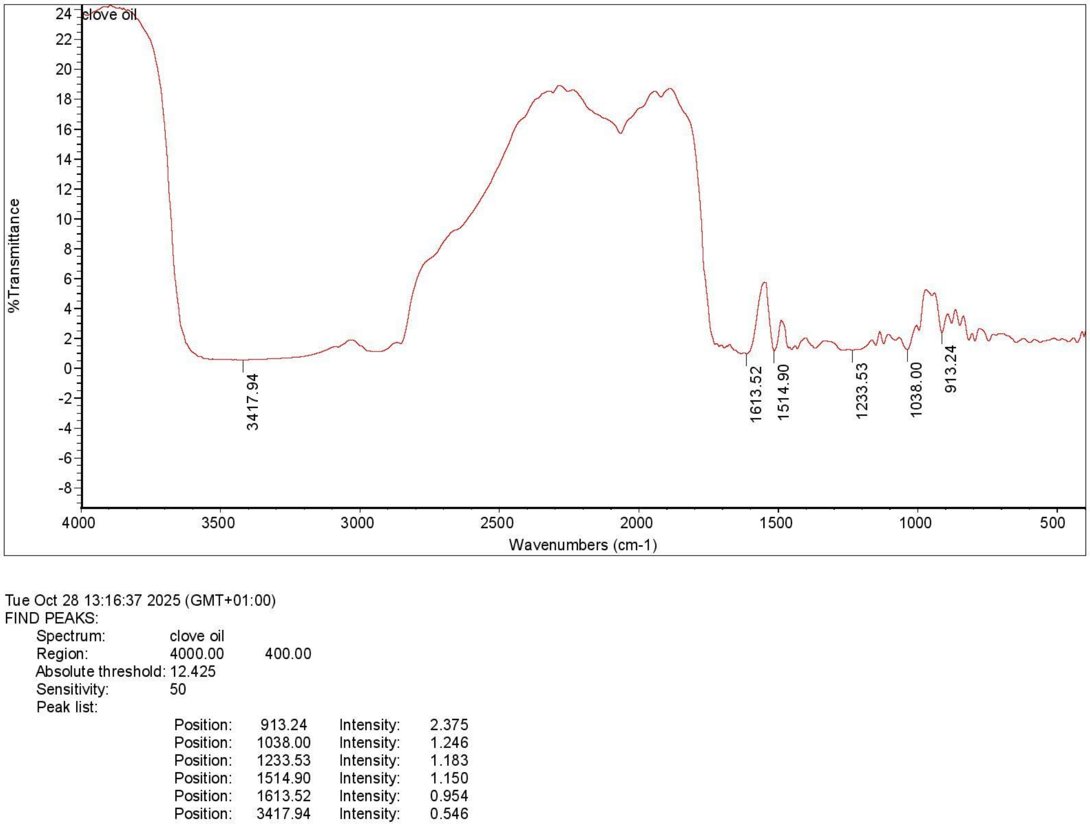
*Table 4. 7*Parameter Analysis

Parameter	**Value**	**Meaning**
Spectrum	SAMPLE C	The name given to the specific FTIR measurement/sample.
Region	4000.00 - 400.00	The range of wavenumbers (cm^{-1}) scanned for peaks. This is the standard mid-infrared range.
Absolute threshold	23.711	The minimum absorption intensity (often percentage transmittance or absorbance) required for a point to be flagged as a peak by the software.
Sensitivity	50	A parameter controlling how strictly the software looks for peaks (e.g., how narrow or isolated a feature must be to be counted).

*Table 4. 8*Peak List Analysis

Position	Intensity (likely)	Shape (likely)	Bond	Vibration	Functional group / compound
1462.42	Medium	-	C=C	Stretching	Aromatic ring
2932.78	Medium	-	-C-H	Stretching	Alkane

4.3 CLOVE ANALYSIS



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      Absolute threshold: 12.425
      Sensitivity:        50
      Peak list:
                           Position:    913.24    Intensity:    2.375
                           Position:   1038.00    Intensity:    1.246
                           Position:   1233.53    Intensity:    1.183
                           Position:   1514.90    Intensity:    1.150
                           Position:   1613.52    Intensity:    0.954
                           Position:   3417.94    Intensity:    0.546
```

Figure 4. 4clove FTIR

*Table 4. 9***Parameter Analysis**

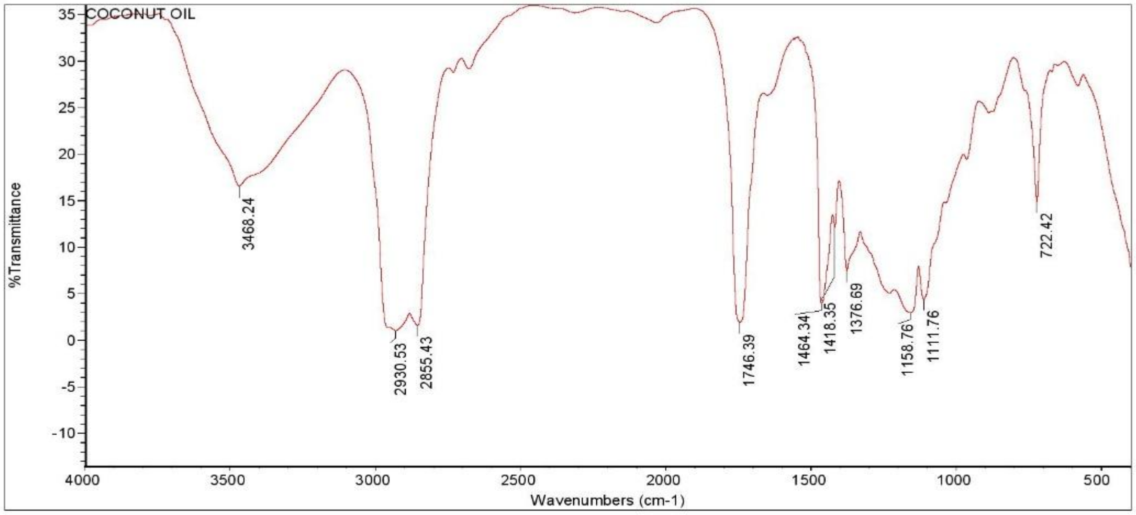
Parameter	**Value**	**Meaning**
Spectrum	CLOVE	The name given to the specific FTIR measurement/sample.
Region	4000.00 - 400.00	The range of wavenumbers (cm^{-1}) scanned for peaks. This is the standard mid-infrared range.
Absolute	12.425	The minimum absorption intensity (often percentage transmittance or

threshold		absorbance) required for a point to be flagged as a peak by the software.
Sensitivity	50	A parameter controlling how strictly the software looks for peaks (e.g., how narrow or isolated a feature must be to be counted).

Table 4. 10 Peak List Analysis

Position	Intensity (likely)	Shape (likely)	Bond	Vibration	Functional group / compound
913.24	Medium	Sharp	=C-H	Out of plane bend	Aromatic /alkene
1038.00	Medium	-	C-N	Stretching	Amine,amide
1233.53	Medium	-	C-N	Stretching	Amine
1514.90	Strong	-	N-O	Stretching	Nitro compounds
1613.52	Medium	-	C=C	Stretching	Aromatic ring
3417.71	Medium	-	N-H	Stretching	Primary amine

4.4 COCONUT ANALYSIS



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     Peak list:
                        Position:    722.42   Intensity:    14.880
                        Position:   1111.76   Intensity:     4.381
                        Position:   1158.76   Intensity:     2.933
                        Position:   1376.69   Intensity:     7.466
                        Position:   1418.35   Intensity:    12.127
                        Position:   1464.34   Intensity:     3.934
                        Position:   1746.39   Intensity:     1.903
```

Figure 4. 5 coconut FTIR

*Table 4. 11*Analysis Parameter

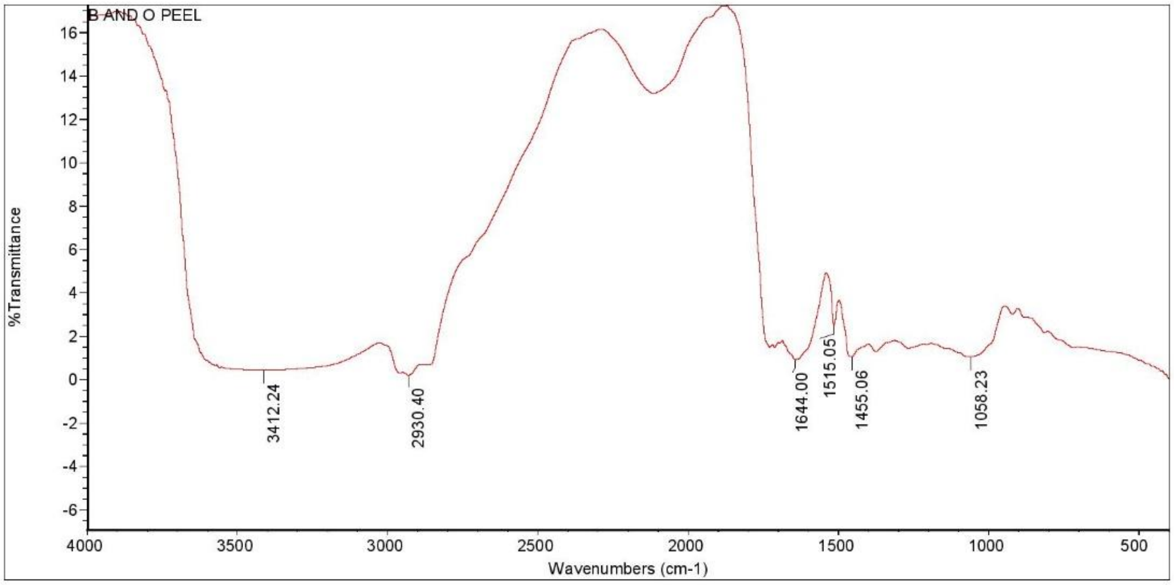
Parameter	**Value**	**Meaning**
Spectrum	COCONUT	The name given to the specific FTIR measurement/sample.
Region	4000.00 - 400.00	The range of wavenumbers (cm^{-1}) scanned for peaks. This is the standard mid-infrared range.
Absolute threshold	18.502	The minimum absorption intensity (often percentage transmittance or absorbance) required for a point to be flagged as a peak by the software.

Sensitivity	50	A parameter controlling how strictly the software looks for peaks (e.g., how narrow or isolated a feature must be to be counted).

*Table 4. 12*Peak List Analysis

Position	Intensity (likely)	Shape (likely)	Bond	Vibration	Functional group / compound
722.42	Medium	sharp	=C-H	Out of plane bend	Aromatic / alkene
1111.76	Strong	-	C-O	Stretching	Alcohol, ester acid
1158.76	Strong	-	C-O	Stretching	Alcohol, ester acid
1376.69	Medium	-	C-H	Bending	Alkane
1418.35	Medium	-	C-H	Bending	Alkane
1464.34	Strong	-	N-O	Stretching	Nitro compounds
1746.39	Strong	Sharp	C=O	Stretching	Carbonyl compounds

4.5 BANANA AND ORANGE PEEL



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                          Position:   1455.06    Intensity:    1.045
                          Position:   1515.05    Intensity:    2.503
                          Position:   1644.00    Intensity:    0.899
                          Position:   2930.40    Intensity:    0.195
                          Position:   3412.24    Intensity:    0.430
```

Figure 4. 6 *B&O FTIR*

*Table 4. 13*Analysis Parameters

Parameter	**Value**	**Meaning**
Spectrum	B & O	The name given to the specific FTIR measurement/sample.
Region	4000.00 - 400.00	The range of wavenumbers (cm^{-1}) scanned for peaks. This is the standard mid-infrared range.
Absolute	8.637	The minimum absorption intensity (often percentage transmittance or absorbance) required for a point to be flagged as a peak by the

threshold		software.
Sensitivity	50	A parameter controlling how strictly the software looks for peaks (e.g., how narrow or isolated a feature must be to be counted).

*Table 4. 14*Peak List Analysis

Position	**Intensity (likely)**	**Shape (likely)**	**Bond**	**Vibration**	**Functional group / compound**
1058.23	Medium	-	C-N	Stretching	Amine, amide
1455.06	Medium	-	C=C	Stretching	Aromatic ring
1515.05	Strong	-	N-O	Stretching	Nitro compounds
1644.00	Medium	-	C=C	Stretching	Alkene
2930.40	Medium	-	C-H	Stretching	Alkane
3412.24	Medium	-	N-H	Stretching	Aliphatic Primary amine

4.6 EFFECT OF NATURAL DEMULSIFER ON CRUDE SAMPLE A SEPARATION

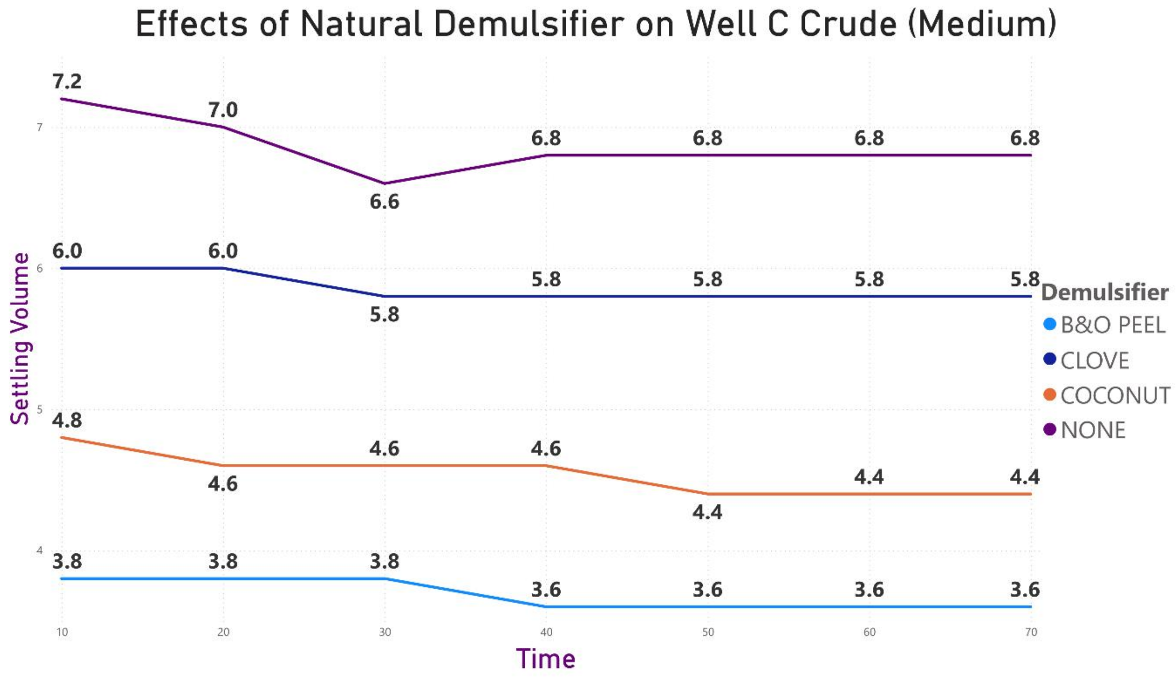

Figure 4. 7 Sample A result

In the case of the medium crude (Well A), the trend of settling volume was found to be irregular as compared to the expectations. The preliminary reading indicated that the settling volume was high, with 7.2 mL but with the slight decrease to 6.6 mL in the untreated sample. The clove extract had an average of 5.8 mL, coconut had about 4.4 mL, and the lowest was the B&O peel which had an average of about 3.6 ml. Nevertheless, this trend is not indicative of a typical demulsification behavior, in which a gradual rise in the settling volume is typically anticipated with time. These values have not been consistent which might have been as a result of droplet coagulation or laboratory measurement errors at the beginning of the test. The rapid aggregation of the water droplets without adequate separation of the phases was possible because of the coagulation and this provided an overestimated initial volume. Consequently, the medium crude could be not a full reflection of the actual demulsification efficiency of the extracts supplied by nature.

4.7 EFFECT OF NATURAL DEMULSIFIER ON CRUDE SAMPLE B SEPARATION

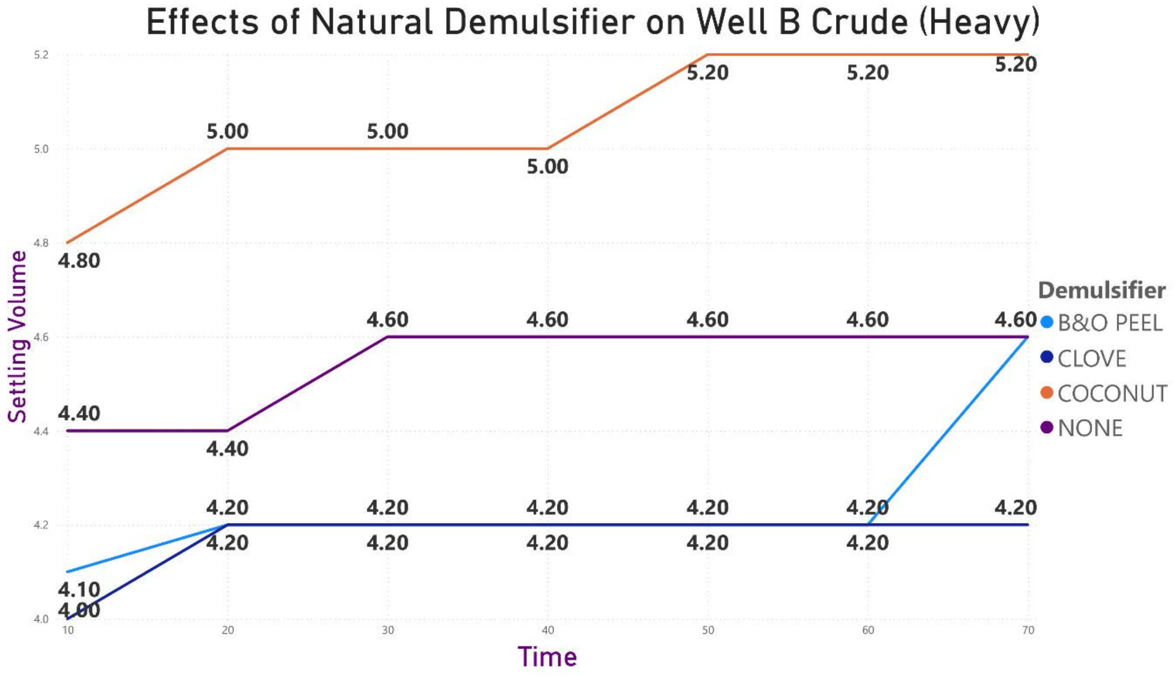

Figure 4. 8 Sample B result

In the heavy crude (Well B), the volumes that settled were typically less, as with the heavy crude, the viscosity is greater and the stability of the emulsion is even tighter. The greatest increment in settling volume was recorded in coconut extract and was approximately 5.2 mL, and this was considered to be a good separation. A constant 4.6 mL of clove was added followed by a constant 4.2mL of the B&O peel. Sample (none) remained close to 4.4 mL without treatment. This implies that coconut extract was the best demulsifier of the heavy crude.

4.8 EFFECT OF NATURAL DEMULSIFIER ON CRUDE SAMPLE C SEPARATION

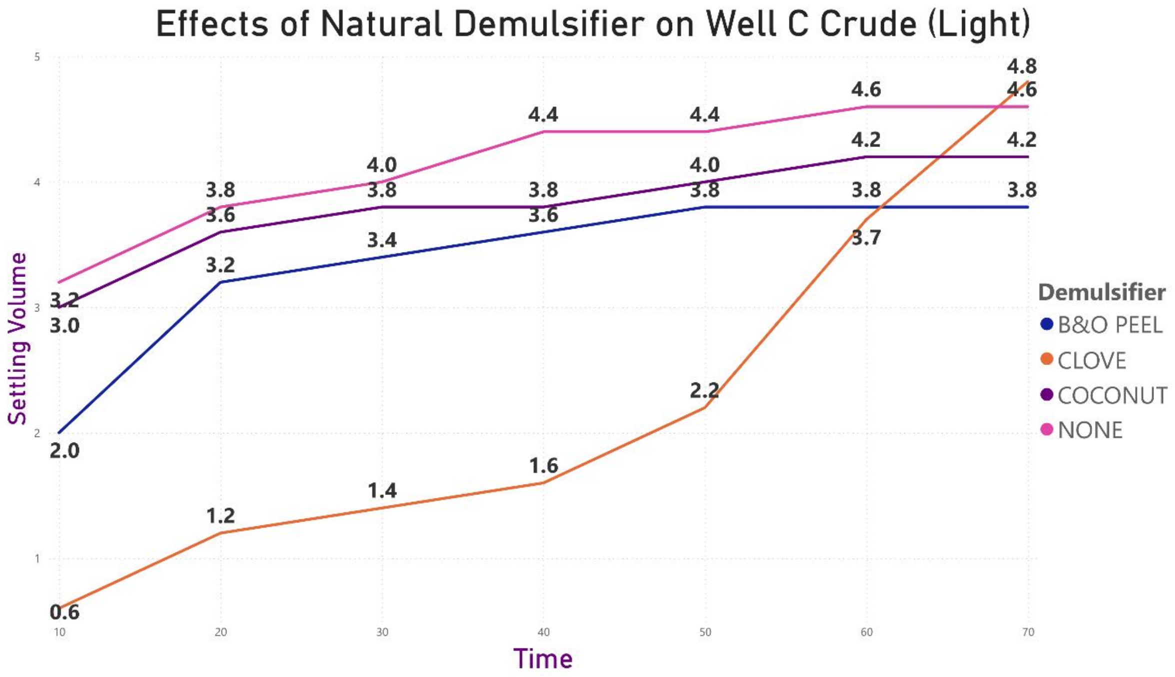

Figure 4. 9 Sample C result

In the case of the light crude (Well C), another trend was observed. Initial separation in the untreated sample was constant but leveled off at 4.4 mL. B&O peel and clove extracts depicted moderate growths in settling volumes to approximately 4.2-4.6 mL. Nevertheless, clove extract showed high progressive increase between 0.6 mL to 4.8 mL meaning that there was a high degree of separation with time. This demonstrates that the clove extract was the most effective in demulsification of the light crude.

4.9 SD OF DEMULSIFIER PERFORMANCE

The experimental data collected during the laboratory study was used to plot the graphs used to represent the effect of each demulsifier on crude oil emulsions. The mean of the replication measurements is taken as the data point and variability is estimated by standard deviation (SD) to ascertain reliability of data. The statistical formula to compute the standard deviation was:

$$SD= \sqrt{\frac{\sum(xi \quad -\bar{x})2}{n-1}}$$

where:

- xi= Sample
- $\bar{x}$ = mean of sample
- n = number of sample.

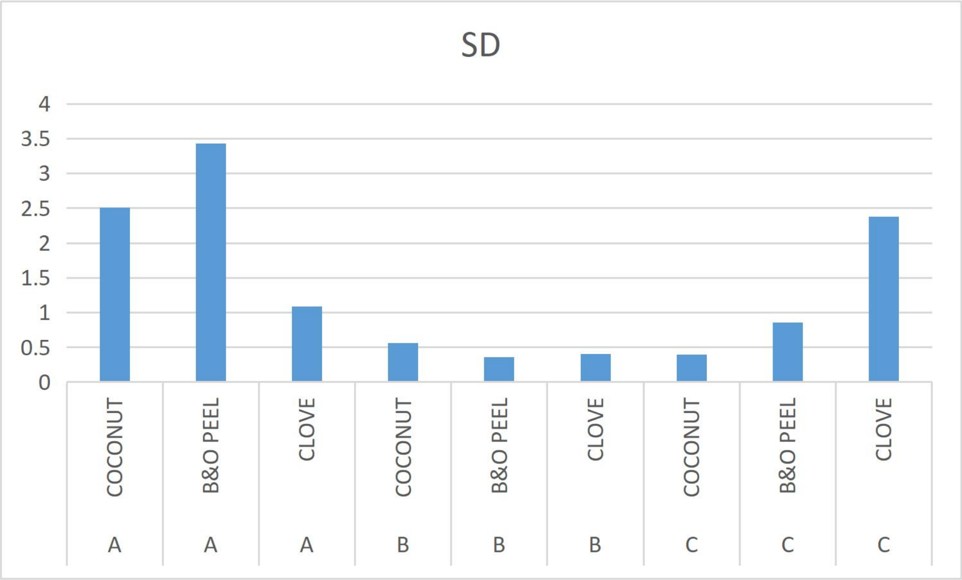

Figure 4. 10 SD of demulsifier performance

The standard deviation (SD) chart in Figure 4.10 shows the difference in the performance of the natural demulsifiers of coconut, banana and orange peel (B&O peel), and clove, in all crude samples (A, B and C).

Standard deviation is used to determine the level of variation of the demulsifier performance over the course of the experiment. An increase in SD value implies that there is inconsistency in performance whereas a decrease in SD implies that there is less variation and predictable separation behavior. In the chart, the SD of B&O peel in sample A was the greatest (nearly 3.5), which meant that its performance was very variable and not favorable. The same sample also revealed a moderate variance in coconuts (approximately 2.5), whereas clove contained much less SD (approximately 1.0), meaning that it was more predictable. The SD values were low in general in sample B and C meaning the demulsifier action in sample B and C was more homogenous. Nevertheless, there were still slight higher SD in sample C of clove and coconut indicating that although they separated better, their results varied slightly across time intervals. The general observation is that the clove extract had the most consistent behavior with all the wells whereas B&O peel demonstrated the greatest variability especially in sample A. This also confirms previous findings that clove exhibited more consistent separation performance, whereas B&O peel was less predictable and perhaps influenced by such factors as emulsifier interaction or experimental error.

The table of the calculated values of the standard deviation of each experimental condition will be provided in the Appendix of this study. The data were charted against the experimental time of 70 minutes and the graphical data demonstrated the change in the separation efficiency and rate of water removal in accordance with time of each of the demulsifiers tested. Graph Trends Interpretation. The graphical trend of the experiment gives an important understanding about the trend of the effectiveness and efficiency of the tested demulsifiers with time. In most cases, when the percentage of water separation increases over time the emulsion of crude oil is breaking efficiently and water droplets are coalescing rapidly whereas a slow or flat trend implies that the demulsification rate is slow or the interfacial films are more strong. The activity and stability of each of the demulsifiers in the same experimental conditions could be assessed by comparing the slopes of the drawn curves. The standard deviation of the variation is used to determine the consistency and reproducibility of the results to ascertain that the observed trends are statistically valid and not because of experimental error. Therefore, the plotted graphs are a clear illustration of the rate and amount of breaking of the emulsion over the 70 minutes of treatment period.

4.10 DIESEL EFFECT ON DEMULSIFIER

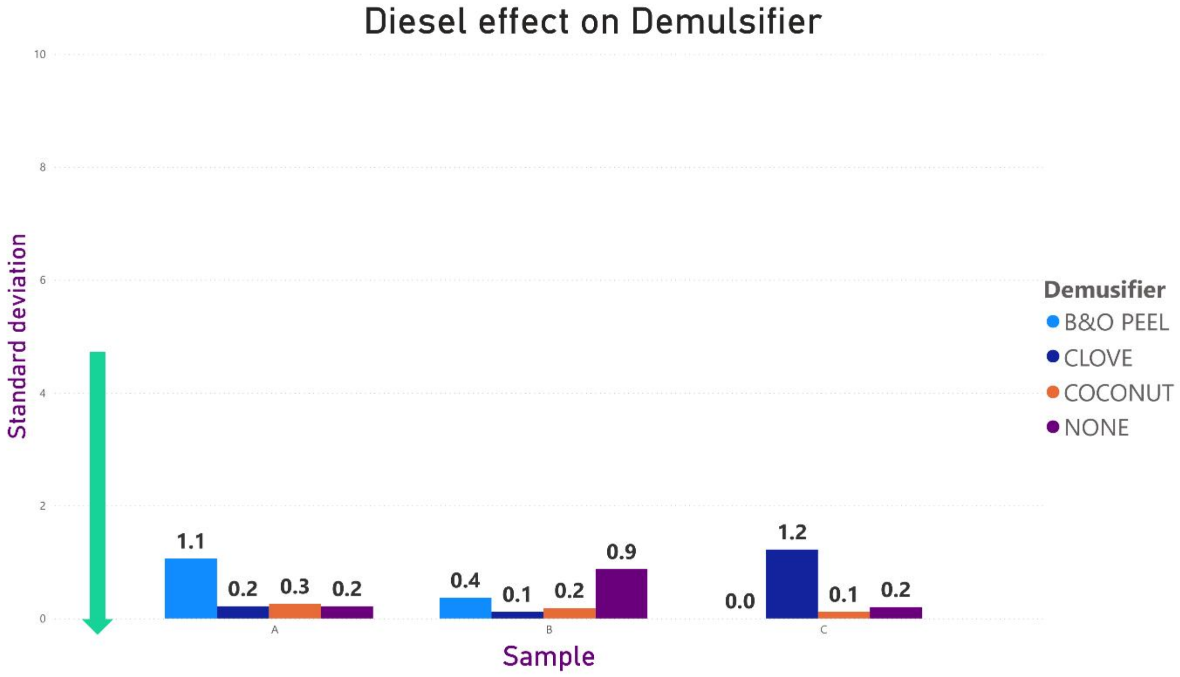

Figure 4. 11 Diesel result

In the chart of diesel effect, sample A had the most variation with B&O peel (1.1) when compared with other samples of clove, coconut and control with relatively low and stable deviation between 0.2 and 0.3 indicating that diesel reacted more with the B&O peel demulsifier. In sample B, the highest deviation (0.9) was observed with the control (none), indicating that when no demulsifier was used, the breakdown of the emulsion was uneven with diesel, but when clove, coconut, or B&O peel was used, the results were more consistent. In sample C, there was the greatest variation of clove (1.2), and B&O peel was far more consistent with a deviation of 0.0 which demonstrates that the active compounds of cloves could have reacted differently with diesel. In general, the diesel chart indicates that diesel exerts greater effect on phenolic and acidic rich demulsifiers like clove and B&O peel hence higher variability in their demulsification is observed compared to coconut that was more stable.

4.11 MAGNET EFFECT ON DEMULSIFIER

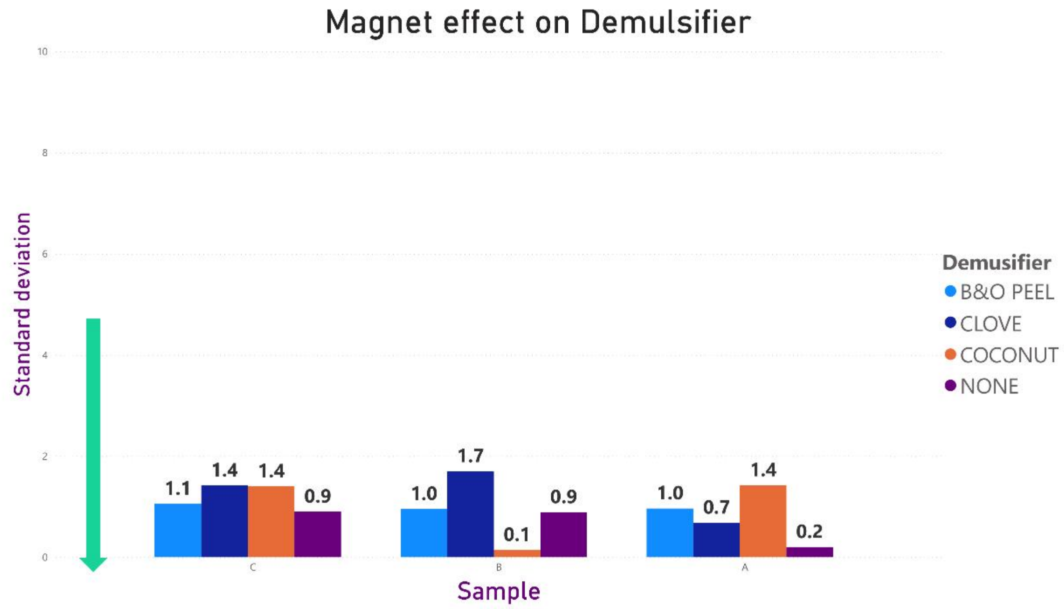

Figure 4. 12magnet result

The magnetic field in the chart shows that depicts magnet effect had a more overall effect on the demulsifier performance as compared to diesel. In sample C, both clove and coconut recorded the highest deviations (1.4), thus, the magnetic field induced inconsistent behavior in both clove and coconut and B&O peel and control recorded slightly lower deviations (1.1 and 0.9

respectively). Sample B showed the most deviation once again of 1.7, next was B&O peel with a deviation of 1.0 and control with a deviation of 0.9, coconut with a deviation of 0.1, which means it had a constant magnetic response. Coconut was the most deviated (1.4) in sample A and B&O peel (1.0), clove (0.7) and the control (0.2) were more stable. In general, the chart of the magnet effect indicates that the exposure to magnet is likely to raise the variability in demulsification efficiency, particularly of natural demulsifiers with aromatic or polar molecules such as clove and coconut, whereas B&O peel expressed intermediate consistency and the control samples were rather stable.

4.12 DIESEL + MAGNET EFFECT ON DEMULSIFIER

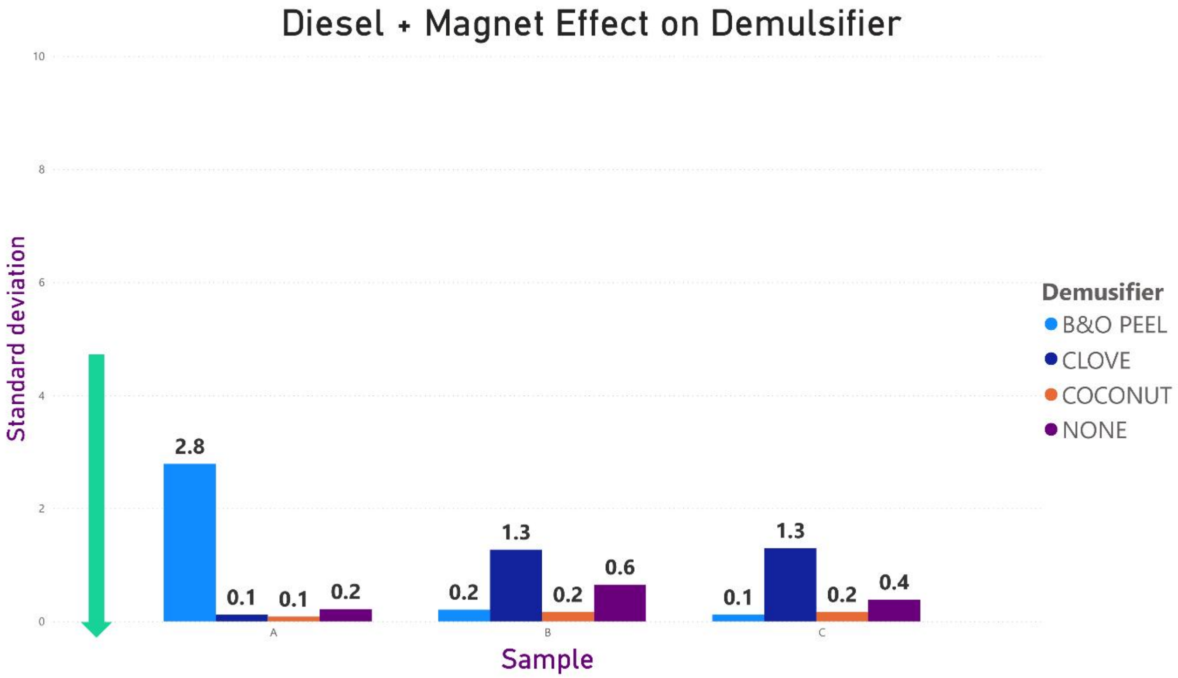

Figure 4. 13diesel +magnet result

The graphs show the influence of diesel and magnetic treatment on the variability of performance of various natural demulsifiers banana and orange (B&O) peel, clove, and coconut in comparison with a control sample which is devoid of demulsifier. It is possible to notice that the value of the standard deviation of B&O peel is the highest in all samples and is equal to 2.8, 1.3, and 1.3, which means that the behavior that B&O peel shows under the impact of a magnet and diesel is extremely variable. By comparison, the remaining demulsifiers clove, coconut and none all show significantly lower and more consistent values of standard deviation, with most falling between 0.1 and 0.6 indicating more predictable and consistent effects. In general, the evidence indicates that the use of a magnet poses a huge change in the variability of the B&O peel demulsifier, whereas clove and coconut show constant behavior with limited fluctuations, which means that the two are more reliable and less subject to magnetic forces in the process of diesel treatment.

CHAPTER FIVE

CONCLUSION, LIMITATION AND RECOMMENDATION

5.1 CONCLUSION

This study represents an important step towards developing sustainable demulsification of crude oil emulsions with an accent on the application of natural and plant-based demulsifiers as a greener alternative to traditional chemical methods.

The study offers a complete insight into the processes that occur during the breaking of crude oil emulsions, and it is acknowledged that the process of demulsification is a multifactor that is affected by various parameters, including temperature, agitation, retention time, solids content, and the availability of emulsifying agents. Using extracts of clove, coconut oil and the banana and orange peels, this study illustrates that bio-based demulsifiers have a great potential to substitute synthetic ones. The comparative analysis showed that these natural materials have active surface compounds, including phenolics, flavonoids, terpenes, and fatty acids, which can be successfully used to destabilize the oil-water interfaces by disrupting the interfacial films and coalescing droplets and modifying the surface tension.

Clove extract was the best behaved and most effective in terms of its demulsification performance with various samples of crude oil which is due to its high phenolic content coupled with its high interfacial activity. The effects of diesel treatment and magnetic treatment on the demulsifier performance were also examined in the study. Findings indicated that these extrinsic factors had a great influence on the demulsification behavior wherein certain demulsifiers were found to have a greater variability at diesel and magnetic exposure.

This result explains why process optimization is significant in actual field use, where the environmental and operational conditions are diverse. The FTIR description of the samples of crude oils and the extracts of plants also gave important information on the functional groups that cause the demulsification. The chemical diversity of the plant extracts and the possibility that it served as an effective surface-active agent was confirmed by the presence of hydroxyl, carboxyl, and aromatic groups. These findings support the scientific value of using natural plant-based materials in order to achieve practical and sustainable demulsification. Environmentally speaking, the study is convincing in terms of the shift in synthetic chemical demulsifiers, which in most cases are ecologically and toxicologically hazardous, to biodegradable, renewable, cost effective and regionally sourced substances.

In addition to similar or better performance, natural demulsifiers enable sustainable development goals by promoting low environmental impact and increased economic feasibility. Eventually, this study is a stepping stone effort in the research of green technologies within the petroleum processing and offers a bright future of environmentally friendly methods of crude oil treatment and a further evolution of natural demulsifier formulations and their use.

5.2 RECOMMENDATIONS

i. Enhancement of Surfactant Quality: Future studies would involve attempting to refine or concentrate the active surface compounds in natural extracts, or make blends with weak synthetic surfactants to enhance their interfacial activity and stability.

ii. Increase Sample Diversity: It is advisable to test more varieties of crude oils which have different densities, salinities, and asphaltene contents in order to determine the versatility of the natural demulsifiers.

iii. Optimize External Conditions: The level of diesel and intensity of magnetic treatment are supposed to be standardized or optimized such that the variation of the experiment is reduced and the effect they have on the experiment is precisely determined.

iv. Adequate Chemical Analysis: Supplement FTIR analysis with GC-MS or HPLC methods to determine and analyze the precise chemical components that cause demulsification which will enable a more efficient structure-activity correlation.

v. Pilot or Field Testing: Hold large-scale or field experiments to test the efficiency of natural demulsifiers in the real-time, its economic feasibility and stability of operation at production scale.

vi. Formulation of Blends: Investigate the option of mixing two or more natural demulsifiers (e.g. clove and coconut, or banana-orange peel and coconut) to find out what synergistic effects they may have.

vii. Comparison to Synthetic Demulsifiers: Add a standard industrial demulsifier to the future study as a control to test the performance comparison and certify natural alternatives.

viii. Environmental and Economic Analysis: Research the biodegradability, cost-effectiveness and sustainability of plant-based demulsifiers to facilitate the use of these products as the green alternative in the oil industry.

5.3 CONTRIBUTION TO KNOWLEDGE

This study makes several important contributions to existing knowledge on crude oil demulsification, especially in the emerging field of green and plant-based demulsifiers:

i. Development of Locally Sourced, Eco-Friendly Demulsifiers:
   The research demonstrates that clove extract, coconut oil, and combined orange–banana peel extracts can effectively destabilize water-in-oil emulsions. This provides scientific evidence that locally available agricultural materials in Nigeria can serve as low-cost, biodegradable alternatives to synthetic chemical demulsifiers.
ii. Comparative Performance Ranking of Natural Demulsifiers:
   The study establishes that clove extract has the highest demulsification efficiency among all natural demulsifiers tested, largely due to its high phenolic content and strong amphiphilic behavior. This finding contributes new knowledge on the structure–performance relationship of natural extracts in crude oil treatment.
iii. FTIR-Based Mechanistic Understanding:
   By using FTIR analysis, the research links the functional groups present in each natural extract (phenolics, terpenes, fatty acids, esters) to their interfacial activity. This provides a scientific explanation for how these plant-based compounds weaken asphaltene and resin films at the oil–water interface.
iv. Evidence that Diesel Dilution and Magnetic Fields Have Minimal Impact:
   The study experimentally shows that diesel dilution and weak magnetic fields have little to no significant effect on demulsification efficiency for the selected natural demulsifiers. This highlights that intrinsic chemical composition is more influential than external operational factors—an insight not widely documented in existing literature.

v. Performance Variability Across Multiple Crude Samples:

By testing three different crude oil samples (A, B, and C), the research contributes practical knowledge on how natural demulsifiers behave across varying crude compositions, emphasizing the importance of crude-specific demulsifier selection.

vi. Promotion of Sustainable Crude Oil Treatment:

The study strengthens the growing body of research supporting green demulsification technologies. It shows that plant-based demulsifiers can achieve comparable, and in some cases better, performance than commercial synthetic ones, reducing environmental impact, cost, and chemical exposure during crude oil processing.

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APPENDIX

CRUDE OIL SAMPLE A- HEAVY CRUDE D- COCONUT				
TIME(MINS)	MC (CONTROL)	MC (DIESEL)	MC (DIESEL&MAGNET	MC (MAGNET)
10	4.8	4.6	4.8	6
20	4.6	4.4	4.6	6
30	4.6	4.4	4.6	5.8
40	4.6	4.2	4.6	5.8
50	4.4	4.2	4.6	5.8
60	4.4	4.2	4.4	5.8
70	4.4	4.2	4.4	5.8
D- BANANA AND ORANGE PEEL				
10	3.8	5	6.8	4.6
20	3.8	4.8	6.4	4.6
30	3.8	4.6	6.2	4.6
40	3.6	4.6	6	4.6

50	3.6	4.6	6.2	4.6
60	3.6	4.6	6.2	4.6
70	3.6	4.4	6	4.4

D- CLOVE

10	6	5.8	6.2	6.6
20	6	5.8	6	6.6
30	5.8	5.6	6	6.6
40	5.8	5.6	5.8	6.4
50	5.8	5.6	5.8	6.4
60	5.8	5.6	5.8	6.4
70	5.8	5.6	5.8	6.4

D-CONTROL

10	7.2	7	7	7.4
20	7	6.8	6.8	7
30	6.6	6.8	6.8	7

40	6.8	6.6	6.6	7
50	6.8	6.6	6.6	6.8
60	6.8	6.6	6.6	6.8
70	6.8	6.6	6.6	6.8

CRUDE OIL SAMPLE B- MEDIUM CRUDE D- COCONUT

TIME(MINS)	MC (CONTROL)	MC (DIESEL)	MC (DIESEL&MAGNET	MC (MAGNET)
10	4.8	4.6	5	5
20	5	5	5.2	5
30	5	4.8	5.2	5.2
40	5	5	5.2	5.2
50	5.2	5	5.2	5.2
60	5.2	5	5.2	5.2
70	5.2	5	5.2	5.2

D- BANANA AND ORANGE PEEL

10	4.1	4.2	4	3.7

20	4.2	4.6	4.4	3.2
30	4.2	4.6	4.4	5
40	4.2	4.6	4.4	5.2
50	4.2	4.6	4.4	5.2
60	4.2	4.6	4.4	5.2
70	4.6	4.6	4.4	5.4

D- CLOVE

10	4	4	5.2	5.6
20	4.2	4.2	5.2	5.6
30	4.2	4.2	5.4	5.8
40	4.2	4.2	5.4	5.8
50	4.2	4.2	5.4	5.8
60	4.2	4.4	5.4	5.8
70	4.2	4.4	5.4	5.8

D-CONTROL

10	4.4	5	5	4.8

20	4.4	5.4	5	5.4
30	4.6	5.4	5.2	5.4
40	4.6	5.4	5.2	5.6
50	4.6	5.4	5.2	5.4
60	4.6	5.4	5.2	5.4
70	4.6	5.4	5.2	5.4

CRUDE OIL SAMPLE C- LIGHT CRUDE D- COCONUT

TIME(MINS)	MC (CONTROL)	MC (DIESEL)	MC (DIESEL&MAGNET	MC (MAGNET)
10	3	3	2.8	4.4
20	3.6	3.4	3.8	3.6
30	3.8	3.8	3.6	5.2
40	3.8	4	3.8	5.2
50	4	4	4	5.4
60	4.2	4.2	4	5.6
70	4.2	4.2	4.2	5.6

D- B&O PEEL

10	2	2	1.8	2.8
20	3.2	3.2	3	4.2
30	3.4	3.4	3.4	4.4
40	3.6	3.6	3.6	4.6
50	3.8	3.8	3.8	4.8
60	3.8	3.8	3.8	4.8
70	3.8	3.8	3.8	4.8

D- CLOVE

10	0.6	0.6	0.8	0.8
20	1.2	2.6	2.6	2.8
30	1.4	2.8	3	3.2
40	1.6	3	3.2	3.4
50	2.2	3.2	3.4	3.6
60	3.7	3.4	3.6	3.8
70	4.8	3.4	3.6	3.8

D-CONTROL

10	3.2	3.4	3	4
20	3.8	3.6	3.4	4.6
30	4	4	3.8	5
40	4.4	4.2	4	5.2
50	4.4	4.2	4	5.2
60	4.6	4.4	4.2	5.4
70	4.6	4.4	4.2	5.4