

**INVESTIGATION OF THE DEMULSIFICATION POTENTIAL OF
BITTER LEAF (*Vernonia amygdalina*) EXTRACT**

BY

EREYI-USOH DAVID IMUETINYAN

ENG1904990

FEBRUARY, 2025.

**INVESTIGATION OF THE DEMULSIFICATION POTENTIAL OF
BITTER LEAF (*Vernonia amygdalina*) EXTRACT**

BY

EREYI-USOH DAVID IMUETINYAN

ENG1904990

**A PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL
ENGINEERING, UNIVERSITY OF BENIN IN PARTIAL
FULFILMENT OF THE REQUIREMENT FOR THE AWARD OF
BACHELOR OF ENGINEERING (B.ENG) HONOURS DEGREE OF
THE UNIVERSITY OF BENIN, BENIN CITY, NIGERIA.**

FEBRUARY, 2025

CERTIFICATION

This is to certify that this Research project work was carried out by EREYI-USOH DAVID IMUETINYAN with Matriculation number ENG1904990 of the Department of Chemical Engineering at the University of Benin, Benin City Edo State.

PROF. S.E. UWADIAE

Project Supervisor

DATE

PROF. S.E. UWADIAE

Project Coordinator

DATE

PROF. DR. (MRS.) E. T. AKHIHIERO

Head of Department

DATE

EXTERNAL EXAMINER

DATE

DEDICATION

This project is dedicated to God Almighty, for his unfailing love, guidance and protection. It is also dedicated to my parents, Mr. & Mrs. Emmanuel Ereyi-Usoh, for their unwavering support throughout the process of this study, without forgetting my siblings too for their support, emotionally throughout the course of this project.

ACKNOWLEDGEMENT

All the glory, thanks and praise to the Almighty God, who makes all things work together for my good.

To my outstanding supervisor, PROF. S.E. Uwadiae, I greatly appreciate your time and corrections through the course of this study.

I greatly appreciate the aid of my peers, friends and family, for their positive influence throughout my stay in the University of Benin and in life in general. I say a big thank you and I pray God in his infinite mercies will always provide for you in your time of needs.

Also, I want to really appreciate myself, it was not easy doing this. Having therefore obtained help from God, I continue unto this day.

ABSTRACT

Demulsification usually involves the addition of chemicals. Because stable oil-water emulsions occur during oil production, separating those emulsions are particularly significant in the oil business. Natural substances like asphaltenes and resins stabilize these emulsions by acting as surfactants, which stop water droplets from coalescing and make the emulsions harder to break. These emulsions' high water content can lead to pipeline corrosion in refineries, thus water must be removed before transit.

Chemical demulsifiers are used in the oil business to facilitate the demulsification process. Surfactants known as demulsifiers cause oil-water emulsions to become unstable. In water-in-oil emulsions, oil-soluble demulsifiers are utilized, whereas water-in-water emulsions require water-soluble demulsifiers. Water droplets must flocculate and clump together to create distinct water and oil phases in order to break up the emulsions. In order to promote droplet coalescence, the demulsifier lowers the interfacial tension between the water and oil phases and adsorbs to the interface, eliminating and dissolving asphaltene particles.

The potential of bitter leaf extract as a demulsifier is the main aim of this experiment. The bitter leaf, or *Vernonia amygdalina*, has been researched for a number of reasons. The bitter leaf will be extracted for the study using a n-hexane extraction method. A rotary evaporator will then be used to concentrate the extract. .

The product's performance will be compared to that of common commercial hops. The ultimate goal of this initiative is to present bitter leaf as a possible replacement for demulsifiers that are imported, and have a lot of adverse effects on both the environment and the financial resources of the company.

TABLE OF CONTENTS

CERTIFIICATION	3
DEDICATION	4
ACKNOWLEDGEMENT	5
ABSTRACT	6
LIST OF FIGURES	10
LIST OF TABLES	11
CHAPTER ONE	12
1.0 INTRODUCTION	12
1.1 BACKGROUND OF STUDY	12
1.2 PROBLEM STATEMENT	14
1.3 AIMS AND OBJECTIVES OF STUDY	14
1.4 SCOPE OF THE STUDY	14
1.5 RELEVANCE OF STUDY	15
CHAPTER TWO	16
2.0 LITERATURE REVIEW	16
2.1 EMULSION	16
2.1 TYPES OF EMULSION	16
2.1.1 WATER-IN-OIL (W/O) EMULSION	16
2.1.2 OIL-IN-WATER (O/W) EMULSION	17
2.1.3 MULTIPHASE EMULSION	17
2.2 CRUDE OIL EMULSION	18
2.2.1 WHAT IS CRUDE OIL EMULSION?	18
2.2.2 FORMATION OF CRUDE OIL EMULSION	19
2.2.2.1 AGITATION AND MIXING:	19
2.2.2.2 EMULSIFIERS:	19
2.2.2.3 CHANGES IN TEMPERATURE AND PRESSURE:	20
2.2.3 EFFECTS OF CRUDE OIL EMULSION	20
2.2.4 TREATMENT FOR CRUDE OIL EMULSIONS	21
2.3 DEMULSIFICATION	21
2.4 CRUDE OIL DEMULSIFICATION	24
2.4.1 MECHANISMS OF DEMULSIFICATION	24
2.4.2 METHODS OF DEMULSIFICATION:	27

2.4.3 FACTORS AFFECTING DEMULSIFICATION	33
2.5. DEMULSIFIERS	35
2.5.1 TYPES OF DEMULSIFIERS	36
2.6 DEMULSIFICATION USING <i>Vernonia amygdalina</i> (BITTER LEAF) EXTRACTS	38
2.6.1 DEMULSIFICATION POTENTIAL OF <i>Vernonia amygdalina</i>	39
2.6.2 APPLICATIONS OF <i>Vernonia amygdalina</i> IN DEMULSIFICATION:	39
2.6.3 CHEMICAL COMPOSITION AND ACTIVE COMPOUNDS IN BITTER LEAF EXTRACTS	39
2.7 PREVIOUSLY RELATED STUDIES	40
CHAPTER THREE	43
3.0 MATERIALS AND METHODS	43
3.1 MATERIALS	43
3.1.1 LIST OF EQUIPMENT/ APPARATUS	43
3.1.2 LIST OF CHEMICALS	44
3.2. METHODS	44
3.2.1. PREPARATION OF BITTER LEAF EXTRACT	44
3.2.2 EXTRACTION OF LEAF EXTRACT	45
3.2.3 OIL EMULSION PREPARATION	45
3.2.4 DEMULSIFICATION EXPERIMENT	46
3.2.5 MEASUREMENT OF DEMULSIFICATION EFFICIENCY	46
CHAPTER FOUR	47
4.0 RESULTS AND DISCUSSION	47
CHAPTER FIVE	53
5.0 CONCLUSION AND RECOMMENDATION	53
5.1 CONCLUSION	53
5.1 RECOMMENDATIONS	54
REFERENCES	57
APPENDIX	60

LIST OF FIGURES

Figure 1: Types of Emulsion	18
Figure 2: Demulsification	22
Figure 3: Process of demulsification of crude oil	25
Figure 4: Variation of demulsification efficiency over time	47
Figure 5: Column type Chart of De-emulsification Efficiency	Error! Bookmark not defined.
Figure 6: Trend Line of De-emulsification Efficiency	Error! Bookmark not defined.
Figure 7: Comparison between Separol and bitter leaf extract	51

LIST OF TABLES

Table 1: Previously Related Studies	40
Table 2: Summary of Demulsification process for each sample.	49
Table 3: Statistical summary of the results.	49

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background Of Study

Traditional chemical-based demulsifiers in the oil and gas industry has several major issues that can affect their effectiveness and environmental sustainability. Chemical-based demulsifiers can contaminate water sources and harm aquatic life, as well as contribute to the formation of microplastics and other pollutants (Deshpande et al., 2015; Esmaeili et al., 2018). These chemicals can corrode equipment and infrastructure, leading to high cost of maintenance and replacement, and every possibility of reacting with other materials used in oil and gas processing (Abdel-Raouf, 2012; Pereira et al., 2017).

The breakdown of chemical-based demulsifiers over time can lead to environmental pollution, as they degrade and form foam or emulsions, which can interfere with the separation process (Abdel-Raouf, 2012; Esmaeili et al., 2018).

Emulsions are widely used in a variety of industries, including cosmetics, medicines, and oil & gas. In the oil and gas business, emulsions can occur during crude oil extraction and processing, making it difficult to separate the oil from water and other pollutants (Smith et al., 2023). Conventional demulsification processes frequently require synthetic chemicals, which can be expensive and have potential environmental consequences.

Demulsification, or the act of breaking down and separating emulsions, is an important stage in many industrial processes, such as oil refining, food processing, and wastewater treatment. The development of efficient and ecologically friendly demulsification procedures is becoming increasingly crucial as the need for sustainable technology grows (Patel, 2019).

In recent years, there has been a growing interest in exploring the use of natural products and plant extracts as demulsifiers, which can offer advantages such as reduced toxicity and improved biodegradability compared to traditional chemicals.

Bitter leaf (*Vernonia amygdalina*), a plant commonly found in tropical regions, has been traditionally used in folk medicine for various purposes, including the treatment of skin conditions and digestive issues. The plant contains a range of bioactive compounds, including flavonoids, alkaloids, and terpenoids, which have been reported to possess antimicrobial, anti-inflammatory, and antioxidant properties. Despite its potential uses, the demulsification properties of bitter leaf extract have not been extensively investigated.

Some recent investigations have found that bitter leaf extract's demulsifying characteristics are due to its phytochemical content, which includes saponins and tannins, which may have biological roles as natural surfactants or emulsion busters. Emulsions are used in a variety of industries, including cosmetics, medicines, and oil and gas. For example, in the oil and gas business, emulsions form during crude oil extraction and processing, causing issues by separating the oil from water and other pollutants. Traditional demulsification methods may include the use of synthetic chemicals that are costly and non-biodegradable (Abbas et al., 2019).

This process is environmentally friendly, and very feasible, since the materials needed for this can be easily gotten from market places, personal gardens and other sources. Using *Vernonia amygdalina* as a target demulsifiers, the effect of this material on the environment, and the cost of the overall process had been investigated.

1.2 Problem Statement

The traditional chemical-based demulsifiers pose several risks. It most importantly poses a severe environmental harm, including its toxicity to aquatic organisms, causes pipeline corrosion, leading to equipment failure which in turn weighs in on the cost of processing oil and gas. The use of these demulsifiers raises serious safety concerns due to the potential hazards and accidents associated with it.

1.3 Aims And Objectives Of Study

This study's aim is to investigate the demulsification potentials of bitter leaf extract, and explore its potential applications in industrial processes.

The objectives of this study are;

- i) To evaluate the demulsification efficiency of *Vernonia amygdalina* extract
- ii) To compare the demulsification performance of *Vernonia amygdalina* extract with industrial chemical-based demulsifiers.

1.4 Scope Of The Study

All experiments would be carried out under different conditions. The isolation and characterization of the bioactive compounds of *Vernonia amygdalina* extract would be done using chromatographic techniques. The exploration of the potential applications of *Vernonia amygdalina* in various industrial processes as a demulsifier would be done using laboratory experiments.

1.5 Relevance Of Study

As made known already, traditional chemical-based demulsifiers, has great negative effect to the society, hence the essence of this study on the demulsification potentials of *Vernonia amygdalina*. Achieving this, would contribute to the development of a sustainable and environmentally friendly demulsification alternative, which can have a positive impact on the environment and human health. *Vernonia amygdalina* is already used medically, with health benefits. Having it as a natural demulsifier would further reduce environmental harm and cost of industrial processes.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Emulsion

An emulsion is a mixture of two immiscible liquids with one being dispersed as droplets in the other, owing to liquid-liquid [phase separation](#). Emulsions are commonly found in everyday products like food, cosmetics, and pharmaceuticals, making them practically significant. Although less commonly recognized, petroleum emulsions are rampant and crucial in the oil industry. Examples of emulsions include mayonnaise, egg yolk, and some [cutting fluids](#) for [metal working](#). The liquid that forms the droplets is the internal phase, while the surrounding liquid is known as the external phase or continuous phase

2.1 Types Of Emulsion

Emulsions are classified into three types: water in oil (W/O) emulsion, oil in water (O/W) emulsion, and multi-phase emulsion (Lowe, 1937).

2.1.1 Water-In-Oil (W/O) Emulsion

The first type is water-in-oil (W/O) emulsion, in which water serves as the dispersed phase and oil as the continuous phase.

The stability of W/O emulsions is influenced by several factors, including the choice of surfactant, which stabilize the water droplets within the oil phase by reducing their size and

preventing phase separation (Madden et al., 2015). These factors ensure that the emulsion remains effective and stable for its intended applications (Rieger & Klee, 2009). This emulsion type often occurs during oil and water production and can be addressed using dehydration methods.

2.1.2 Oil-In-Water (O/W) Emulsion

This occurs when water is the continuous phase and oil is the dispersed phase.. This results in a product that is generally more transparent and less viscous compared to water-in-oil emulsions. In heavy oil systems, oil-in-water emulsions are preferred over water-in-oil emulsions since the latter necessitate an extremely large pressure gradient in order to attain typical flow rates (Sousa et al., 2022a)

Water that has been isolated from the dehydration process may contain this kind of emulsion. This kind of emulsion can be treated using the de-oiling technique.

2.1.3 Multiphase Emulsion

Multi-phase emulsions are intricate systems comprising more than two immiscible liquid phases, such as oil-in-water-in-oil (O/W/O) or water-in-oil-in-water (W/O/W). These emulsions are characterized by their complex inter-facial structures and the dynamic interactions between their multiple phases. The formation and stability of multi-phase emulsions are governed by the principles of phase equilibrium and inter-facial thermodynamics, which dictate how the dispersed phases interact at their boundaries. The stabilization of these systems typically involves the use of surfactant that reduce inter-facial tension and prevent phase coalescence. The choice of surfactant, their concentration, and their compatibility with the different phases are crucial for maintaining the stability and uniformity of the emulsion (McClements & Rao, 2011).

Factors such as phase volume ratios, droplet size, and the presence of stabilizers can impact the formation and stability of multi-phase emulsions. Advances in understanding these interactions have led to more precise control over the formulation and stability of these complex systems (Dickinson, 2009; Tadros, 2013). By manipulating these factors, researchers and engineers can optimize the performance and consistency of multi-phase emulsions in various chemical processes. This kind of emulsion frequently forms in storage tanks and the slopes of oil systems

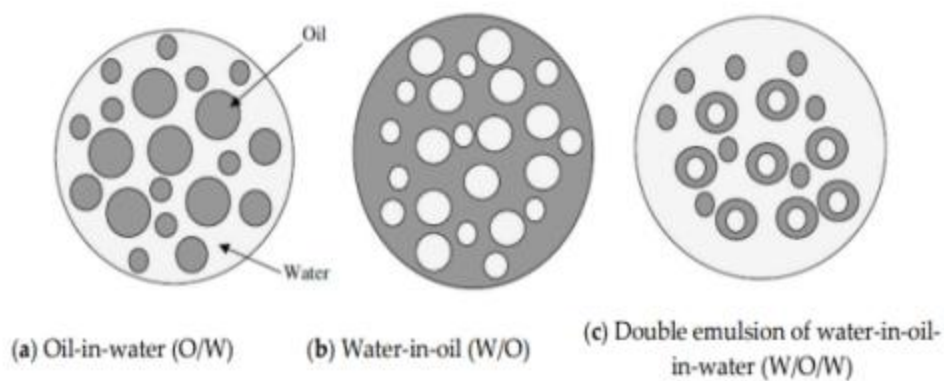


Figure 1: Types of Emulsion

2.2 Crude Oil Emulsion

2.2.1 What Is Crude Oil Emulsion?

Crude oil is a mixture of hydrocarbons that exists in liquid phase in natural underground reservoirs and remains liquid at atmospheric pressure after passing through surface separating facilities. It is typically found deep within the Earth's crust and extracted through drilling.

A crude oil emulsion however, is a colloidal mixture where crude oil is combined with water, leading to the formation of a two-phase system. In these emulsions, water droplets are

dispersed throughout the continuous phase of oil, or vice versa, depending on the emulsion type.

The majority of researchers believe that emulsification is the next most crucial behavioural characteristics of oil after evaporation because emulsification significantly influences the nature of oil spills at sea as well as clean-up response ([Fingas et al., 1993](#)).

Besides that, in the upstream oil production industries, pipeline emulsification flow is a very common occurrence. As crude oil continues to be produced towards the end of the reservoir life, the amount of produced water increases as well, especially if the reservoir is driven by water aquifer ([Lim et al., 2015](#)).

2.2.2 Formation Of Crude Oil Emulsion

Emulsion formation is common in pipeline transportation and production within the petroleum sector. To fully understand the physics of emulsion creation, one must know how much energy is needed to produce an emulsion and what kind of emulsifying agent is used ([Abed et al., 2019](#)). Some of the ways emulsions on crude oil are formed are;

2.2.2.1 Agitation And Mixing: Under high shear circumstances, crude oil frequently comes into contact with brine, or water, during the extraction process from reservoirs. During flow through reservoir rock, tubing, valves, and surface equipment, this agitation takes place. The size of the scattered water droplets decreases with increasing mixing intensity.

2.2.2.2 Emulsifiers: Asphaltenes and resins, two naturally occurring emulsifiers present in crude oil, are important for stabilizing emulsions. The stability and tightness of the emulsion are determined by the kind and concentration of these emulsifiers.

2.2.2.3 Changes In Temperature And Pressure: During extraction, variations in temperature and pressure may also have an impact on the development of emulsions. For instance, the mixture may be further stirred by gas bubbles generated during phase transitions, which would increase the stability of the emulsion.

2.2.3 Effects Of Crude Oil Emulsion

2.2.3.1 Operational Problems: At gas/oil separating facilities, emulsions can cause high-pressure dips in flow lines and make separation procedures more difficult. Because more chemical treatments are needed for demulsification, they might result in higher operating expenses.

2.2.3.2 Environmental Concerns: Managing emulsions is crucial for minimizing environmental impact. If not properly separated and treated, the water phase of the emulsion, which may contain pollutants or hydrocarbons, can pose a risk of contamination to surrounding environments and water sources (Yang et al., 2014).

2.2.3.3 Quality Control: Crude oil's suitability for refining and transportation is impacted by the presence of water. Overly high-water content might result in inefficient operations and higher refining process expenses.

2.2.3.4 Increased Costs: The need for additional chemicals, such as demulsifiers, to break emulsions can significantly raise operational costs. Demulsifiers are specifically designed to destabilize emulsions, but their use adds an extra expense to the refining process (Kokal, 2005).

2.2.3.5 Corrosion And Scaling: Water in emulsions often contains dissolved salts and other impurities that can lead to corrosion of pipelines and processing equipment (Kokal, 2005).

Additionally, the presence of water can result in the formation of scale and deposits, which can further damage equipment and reduce operational efficiency.

2.2.4 Treatment For Crude Oil Emulsions

Several techniques are used to efficiently handle and treat crude oil emulsions:

2.2.4.1 Thermal Treatment: By encouraging the coalescence of water droplets, raising the temperature helps break down emulsions and lower viscosity. This approach is frequently combined with other therapies.

2.2.4.1 Mechanical Separation: Based on density differences, methods like centrifugation or gravity separation can be used to separate water from crude oil.

2.2.4.3 Chemical Demulsification: By lowering interfacial tension and encouraging droplet coalescence, chemical demulsifiers are frequently used to destabilize emulsions (Azubike & Itohan, 2021).

2.2.4.4 Retention Time Management: By encouraging natural settling, allowing enough time for separation processes to take place can improve the results of emulsion treatment.

2.2.4.5 Agitation Control: When working with sensitive crudes that readily form stable emulsions, minimizing agitation during transit might assist decrease emulsion formation.

2.3 Demulsification

Demulsification is the process of separating a crude oil emulsion into its constituent phases or individual components, primarily oil and water. An emulsion is a stable mixture of two immiscible liquids, where one liquid (the dispersed phase) is dispersed in the other (the continuous phase). The term "demulsification" combines the prefix "de-", indicating removal or reversal, with "emulsification," which refers to the process of forming an emulsion.

Demulsification, is a process where emulsions are broken down, most typically by the addition of chemicals. Demulsification is especially important in oil industry where stable oil-water emulsions are formed during oil production.

Studying the features and attributes of emulsions as well as the mechanisms at play when the water droplets coalesce to produce a high rate of separation between the oil phase and the produced water in an emulsion is required. In addition, research on appropriate chemical additives for treating these emulsions also referred to as demulsifiers is crucial for a successful demulsification process (Abdulredha et al. 2018).

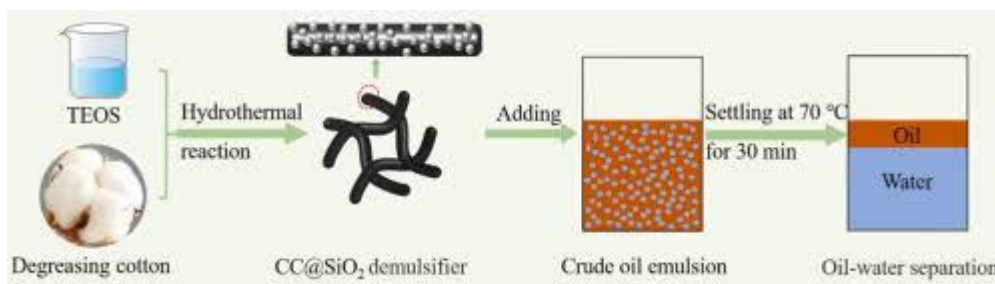


Figure 2: Demulsification

Demulsification is particularly important in industries such as petroleum, where crude oil often contains water and other impurities in an emulsified form. Effective demulsification is essential for refining processes, environmental management, and improving the quality of the final product

Demulsification is crucial for several reasons, particularly in the oil and gas industry, where stable emulsions of oil and water can lead to significant operational challenges. Here are the key reasons why demulsification is essential:

(i) **Industrial Processes:** In industries such as petrochemical processing, demulsification is essential for separating crude oil from water and other impurities to refine oil efficiently. Emulsions can hinder the quality and efficiency of the refining process. Effective demulsification enhances the quality of the final product and reduces operational costs (Mukherjee, 2019).

(ii) **Environmental Protection:** Demulsification plays a critical role in managing oil spills and wastewater treatment. Removing oil from water helps in mitigating environmental pollution and preventing damage to aquatic ecosystems (Jin et al., 2018).

(iii) **Food Industry:** In food processing, especially in the production of products like mayonnaise or salad dressings, controlling emulsions is necessary to achieve desired textures and stability. Poor emulsion stability can lead to product spoilage and inconsistencies (McClements, 2015).

(iv) **Pharmaceuticals:** In drug formulation, demulsification is important for ensuring the stability and effectiveness of emulsified drugs. It helps in controlling drug release rates and improving bioavailability (Sahlin et al., 2019).

(v) **Quality Improvement Of Crude Oil:** Demulsification enhances the quality of crude oil by removing excess water and impurities. High water content in crude oil can lead to corrosion in pipelines and refining equipment, as well as increased transportation costs due to the need to handle larger volumes. By effectively separating the oil from water, demulsification helps ensure that the crude oil meets industry specifications, which is vital for its processing and refining.

(vi) **Operational Efficiency:** Demulsification is crucial for operational efficiency because it helps separate emulsified substances, such as water from oil, which can significantly impact the performance and longevity of equipment and processes. In industries like oil and gas, chemical processing, and manufacturing, emulsions can cause

issues like reduced product quality, equipment corrosion, and increased maintenance costs.

(vi) **Cost Savings:** Demulsification is crucial for cost savings due to its impact on equipment maintenance, process efficiency, product quality, and operational downtime. Emulsions containing water or contaminants can cause corrosion and wear in machinery, accelerating corrosion in pipelines and equipment in the oil industry and increasing maintenance and repair costs (Reis et al., 2014).

2.4 Crude Oil Demulsification

In the petroleum industry, demulsification is a crucial process because emulsions frequently form either naturally or consciously (man-made). Prior to refining crude oil, it is essential to separate water from it in petrochemical industries and refineries. Emulsion breakers, which are chemical additives, are commonly used to separate water from oil. The effectiveness of demulsification methods—whether thermal, mechanical, electrical, or chemical—depends on the physical and chemical properties of the oil, as well as the conditions of emulsification and aging. Consequently, strategies for optimizing water-oil separation can differ significantly between oil fields (Strøm-Kristiansen et al., 1995)

2.4.1 Mechanisms Of Demulsification

The emulsions must pass through a number of stages in the demulsification process in order to be divided into the water and oil phases. This process involves the following mechanisms: creaming and sedimentation, coalescence, Ostwald ripening, and flocculation

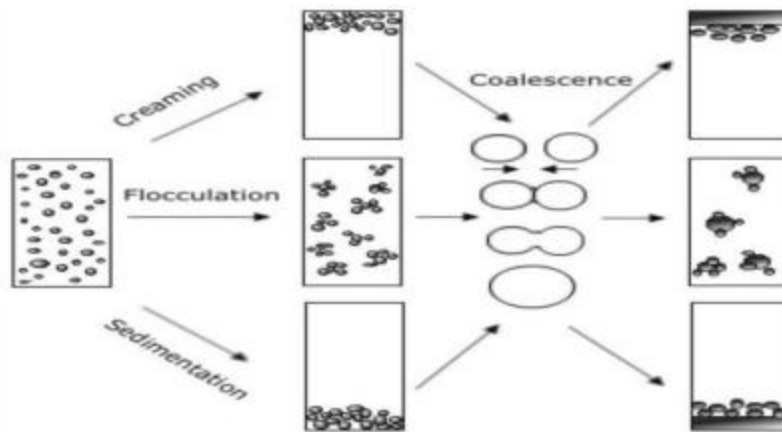


Figure 3: Process of demulsification of crude oil

2.4.1.1 Creaming And Sedimentation

The difference in density between water and oil is responsible for both sedimentation and creaming; that is, the density of water is higher than oil. Sedimentation is an important mechanism for the demulsification of crude oil and is characterized by water droplets on the ground of the continuous oil phase of an emulsion settling. The growth of oil droplets on the water surface is instead a creaming process. Whether sedimentation or creaming takes place depends, therefore, on whether the dispersed phase is water or oil (M. A. Saad et. al, 2019)

2.4.1.2 Ostwald Ripening

Another technique for demulsifying crude oil is called Ostwald ripening. The process by which drops enlarge is known as Ostwald ripening. The process causes drops of various sizes to migrate near one another as soon as the scattered phase has finite solubility in the continuous phase. Large volume fractions typically grow faster because the drops may swap materials more easily. The solubility of oil in water or water in oil is low for heavy oil, which delays the process of drop growth. The stability of oil-in-water emulsions is critical to the drop development process through Ostwald ripening.

2.4.1.3 Coalescence

Coalescence is a crucial step in the demulsification of crude oil and an irreversible process by which water droplets merge into or fuse into a larger process. The coalescence process often results in fewer droplets of water. The emulsion of crude oil is permanently demulsified (S. Mhatre et al., 2018). Factors such as a high flocculation rate and lack of mechanically strong films, high interfacial tension and water cutting, low interfacial speed and high temperature are necessary for an efficient coalescence (S. H. Mousavi et al., 2014)

2.4.1.4 Flocculation

One of the major process in the breakdown of oil-water emulsions involves flocculation and this is especially important in industries where crude oil and water emulsions are encountered such as in the petroleum industry and water treatment. It employs the formation of bigger flocs from small droplets in a body of fluid to separate the flocs easily. The literature proves that demulsifiers can improve this process by adsorbing at the oil-water interface and depressing the interfacial tension and thereby destabilizing the layers of emulsions. For instance, a study showed that when a multicomponent demulsifier package known as BDTXI was used, demulsification efficiency would be more than 97 percent owing to a synergistic effect that causes the droplets to collide and flocculate (Azizollah, 2023). Moreover, it also has been found possible for electrocoagulation to destabilize emulsion since this process can neutralize charge on oil droplets to floc formation and removal of total organic carbon (TOC) from wastewater (Wang et al., 2022). In addition, in the case of alkali /surfactant/polymer flooding, demulsifiers combine with flocculants to accomplish effective accumulation and

separation of oil droplets this shows the significance of charge neutralization and bridging mechanism in flocculation (Huang et al., 2019).

2.4.2 Methods Of Demulsification:

2.4.2.1 Mechanical Demulsification

Mechanical demulsification methods use physical forces to separate the emulsified stages.

Centrifugation uses centrifugal force to expedite separation based on density differences, making it suitable for thick oil-water emulsions. Gravity separation, one of the simplest procedures, is based on the natural settling of heavier phases under gravitational pressures.

Filtration is the process of passing an emulsion through a porous material to catch dispersed droplets; nevertheless, it works best with larger droplets. Ultrasonic waves disrupt the emulsion by producing high-frequency sound waves, which promote droplet coalescence.

These approaches are frequently utilized when chemical treatments are unsuitable or must be reduced owing to environmental or economic concerns. They work well for primary separation but may require additional procedures for complete demulsification (Sjoblom et al., 2019; Esmaili and Zeinali, 2020).

2.4.2.2 Thermal Demulsification

Thermal approaches for demulsification entail using heat to reduce the forces that hold emulsions together. Direct heating reduces the continuous phase's viscosity, allowing scattered droplets to flow and coalesce more easily. Steam injection is a quick process in which steam raises the temperature, causing phase separation by lowering interfacial tension.

Hot water washing not only warms but also dilutes the emulsion, facilitating the removal of the dispersed phase. These techniques are especially useful for emulsions stabilized with

waxes or heavy hydrocarbons, which become less stable at higher temperatures. However, they demand a substantial amount of energy and may be ineffective for thermally stable emulsions, necessitating the use of alternative technologies (Bernard and Lee, 2017; Esmaeili and Zeinali, 2020).

2.4.2.3 Chemical Demulsification

Chemical demulsification is the most frequent method, which involves the use of chemicals to destabilize the emulsion. Demulsifiers, such as surfactants, move to the oil-water interface, destroying the stabilizing layer that surrounds droplets and encouraging coalescence.

Coagulants neutralize electrical charges on droplets, which reduces repulsion and promotes phase separation. Adjusting the pH of emulsifying agents changes their ionization state, causing the emulsion to destabilize. Electrolytes can also be used to boost ionic strength, reducing the efficiency of emulsifiers and causing the emulsion to rupture. Chemical approaches are highly successful, but agents must be carefully selected to avoid negative environmental consequences and assure compatibility with the emulsion being treated (Aryafar and Doan, 2018; Bernard and Lee, 2017).

2.4.2.4 Electrical Demulsification: Electrical demulsification is the process in which oil is separated from the emulsion employing electrodes through oxidation-reduction reactions. This process occurs when an electric current is applied, causing an imbalance of electric potential in the emulsion (Ichikawa, 2007; Zhang et al., 2018; Saad et al., 2019). The presence of an electric field during electrical demulsification leads to the polarization of droplets, then the droplets coalesce by collision in a short period of time (Peng et al., 2016). The induced dipoles interaction leads to droplets aligning in chains parallel to the applied field, thus

increase their sedimentation velocity and ultimate separation (Zhang et al., 2018). The electric system consists of a transformer and two electrodes which provide high voltage current to the system. The electrodes are placed in opposed direction to each other, providing enough energy to break the interfacial barrier of the emulsion placed between them (A Issaka,2015). When DC voltage is applied to the system, an electric field is generated within the emulsion, this breaks the interfacial barrier between droplets, and the water molecules are hydrolyzed into hydrogen and oxygen gases molecules. A study conducted by (Hjartnes et al.,2020) revealed that the demulsification efficiency in the presence of an electric field depended on the strength of the electric field.

2.4.2.5 Biological Demulsification

Biological demulsification refers to the decomposition of water-in-oil emulsions through microorganisms whereby bacteria and fungi are most effective. The demulsification process involved the dispersion of cells into an oil phase where due to their high surface hydrophobicity and the presence of amphiphilic compounds in the cell walls they tended to be deposited on the interface of oil and water phases. The ability of this mechanism in reducing the interfacial tension has had the positive outcome of causing the coalescing of the water droplets and phase separation (Wen et al., 2010a). Due to the ease of production, affordability, and without any adverse environmental impacts, biomass resources have recently attracted a lot of interest (Ye et al., 2021).

Research on biological demulsification began in the early 1980s after it was proposed as a possible chemical demulsifier substitute in the petroleum industry. The idea behind microbial demulsification is that bacteria can obtain nutrition from the hydrocarbons already present in the emulsion (Abed et al., 2019). Current work by Scarborough et al outlines a novel gadget

that combines several demulsification processes with a microbial procedure that refines the oil separation performance. This device uses microbial flora which produce bio-enzymes that aid in the emulsification of the oil droplets and separation of oil and water. The oil is used as a carbon source to encourage microbial degradation and eliminate contaminants from wastewater. Not only does the biological demulsification supplement environmental problems resulting from oil wastewater but also reveals the possible development of biochemical solutions in industries. Therefore, this method can be viewed as an ecological replacement for the traditional chemical demulsification methods that entail certain additional dangers (Scarborough et al., 2011). Biological demulsification in general constitutes an evolution in wastewater treatment technology based on the modern demands for sustainability and ecological considerations.

2.4.2.6 Membrane Demulsification

The treatment of emulsified wastewater, which frequently comprises oil droplets of different sizes that impede conventional filtration methods, can be effectively accomplished using membrane demulsification. Membranes with different pore sizes are used in techniques like microfiltration (MF) and ultrafiltration (UF) to separate oil-in-water emulsions; MF membranes (0.1 to 10 μm) work well for bigger droplets, while UF membranes (0.01 to 0.1 μm) work well for smaller droplets and dissolved organic materials (Deng et al., 2023). Membrane emulsification is an alternative technique that creates uniform oil droplets by driving an oil phase through a membrane and into a water phase. The size of the droplets is controlled by varying the membrane's pore size and flow rates (Shah Buddin et al., 2020). Moreover, membrane distillation (MD), which works well with high salinity and oil content emulsions, uses thermal energy to separate oil-water emulsions by enabling water vapor to

pass through a hydrophobic membrane while leaving oil behind. Additionally, membrane bioreactors (MBR) are especially well-suited for emulsions with low oil content and high organic matter because they combine biological treatment with membrane filtration, effectively eliminating oil while maintaining biomass (Zhang et al., 2023a). Membrane demulsification plays a crucial role in efficiently separating oil-in-water emulsions, with recent studies focusing on enhancing demulsification efficiency and permeability. Innovations like superhydrophilic/underwater superoleophobic membranes with polyoxypropylene polyoxyethylene block polymers have shown remarkable demulsification capabilities (Chen et al., 2024). Similarly, the use of hydrophobic-hydrophilic-hydrophobic (3H) demulsification media has demonstrated high demulsification efficiency and rapid permeability, making it ideal for oil recovery applications (Li et al., 2023). Membrane emulsification technology (MET) provides a versatile method for creating emulsions with precise droplet size control, benefiting various industrial applications like drug delivery and chromatographic separations (Zhu et al., 2024). These advancements underscore the significance of membrane demulsification in addressing environmental concerns related to oil spills and wastewater discharges, showcasing its potential for sustainable oil-water separation processes.

Membrane separation technology has been recognized as a viable method for treating oily wastewater due to its numerous advantages, such as consistent permeate quality, no chemical involvement, efficient separation of small droplets, high separation efficiency, cost-effectiveness, and compact design (Singh & Das, 2022). However, its widespread application faces challenges like high capital costs for treating large effluent volumes, membrane degradation, fouling during operation, low permeate flux, and sensitivity to certain solutions (Deng et al., 2022). Research has explored using membranes as demulsifiers, with studies like Zhang et al. focusing on integrating ultrafiltration processes for emulsion wastewater

treatment, showing that pre-treatment with calcium chloride can enhance oil removal and membrane filtration efficiency, especially when combined with vibratory shear techniques. These findings highlight the potential of membrane technology in improving oily wastewater treatment processes (Zhang et al., 2023b).

2.4.2.7 Microwave Demulsification

Microwave demulsification is an up-to-date method used in the demulsification process, more so being based on microwave heating. It uses specific heat properties of microwave radiation to heat the dispersed phase of the emulsion and hence gives a better separation efficiency than other methods of heating. There is some evidence of the fact that microwave demulsification is capable of raising the demulsification efficiency by varying either the power or the time at which the two phases are exposed to microwaves; for example, an experimental case, conducted by some authors, pointed to the fact that demulsification was enhanced appreciably by using the microwave power setting, which proved that the method could be effective in numerous fields and sectors (Chan & Chen, 2002). In addition, studies have been done to determine the efficiency of microwave demulsification in different emulsions such as W/O emulsions with different compositions and times of exposure to microwave radiation. In one of the studies where the concentration of water in oil was tested at 50-50% and 20-80%, the time of exposure was found to range from 20 to 180 seconds and it was shown that this is variably a parameter that could be used to control demulsification process. Also, the microwave demulsification procedure has been determined to use salt, where the separation of oil-rich emulsions was quick when salt was used as a supporting material to the process of demulsification (Nour & Yunus, 2006). Microwave demulsification has emerged as a promising method for breaking emulsions in various industries, particularly

in oil processing, as indicated by research findings. Studies have delved into combining microwaves with demulsifying agents and magnetic nanoparticles to enhance demulsification processes, resulting in increased efficiency and the improved recyclability of nanoparticles (Jiang et al., 2022). The dielectric properties of emulsions have been assessed and modeled to predict microwave penetration depths, showcasing the potential of microwave heating for large-scale demulsification applications (Sun et al., 2024). Furthermore, the synergistic impact of magnetic nanoparticles and microwaves has been observed to notably boost demulsification efficiency, shedding light on the underlying mechanisms of this synergistic process (Sun et al., 2022).

2.4.3 Factors Affecting Demulsification

To obtain the desired demulsification efficiency, some factors that affect the emulsion chemical demulsification have to be optimized. The demulsification process is governed by Stoke's law (Zaman et al., 2019). The demulsifiers act directly on the film separating oil and water phases, so as to disrupt the interfacial tension between the droplets, and hence increase thermal velocity and speed up the coalescence. The process of demulsification is affected by the temperature, phase volume ratio, residence time, demulsifier dosage, crude oil properties, and aging of emulsions (Yonguep and Chowdhury, 2021).

(I) Temperature

Heat energy plays an essential role in the demulsification process of crude oil emulsion as it directly reduces the viscosity and enhances the flow of emulsions. Temperature affects the physical properties of oil-water interfacial films by increasing the droplets' thermal energy, which leads to an increased frequency of drop collisions and coalescence. Heat weakens the

interfacial film between the oil and the water droplets, lowers the oil viscosity, and increases the mobility and settling rate of the water droplets, thus, reducing the interfacial viscosity, and resulting in a faster film drainage rate and enhancing droplets coalescence Oliveira et al.(2007)

(ii) Water And Oil Content

The phase volume ratio is one of the main parameters that affect the efficiency of demulsification. Emulsions with high water content are more readily separated than those with lower water content. High water content in crude oil emulsions promotes demulsification efficiency while decreasing the demulsifier dose and the time needed for the phases to separate, provided that water content falls within volumetric fractions ranges of 30e70%. Generally, the emulsions are likely to separate faster when the volume of the dispersed phase is increased as the distance between the droplets is reduced Ahed et al. (1999) reported that decreasing the oil content from 90 to 60 vol % in a surfactant-stabilized oil-inwater emulsion without a demulsifier resulted in reduced water separation. A better separation could be observed when the oil content was reduced to 50%. Higher oil contents (60% and above) in an oil-in-water emulsion has the potential of shifting the system to water-in-oil emulsions, which happens to be more viscous, dense, more stable, and more difficult to demulsify

(iii) Retention Or Settling Time

Retention time in the context of demulsification is the duration the emulsions stay in a non-agitated state to allow it to separate into individual phases. Higher retention times enhance the demulsifier diffusion through the oil-water interface and gravity settling, thus, improving the demulsification efficiency. Although increased retention time positively affects the demulsification efficiency, it comes at the expense of high separator equipment costs.

Therefore, there is a need to optimize retention time to avoid waste of time and money. Kokal and Al Ghamdi (2006) observed an almost complete oil separation in 5e10 min by mixing two different types of demulsifiers

(Iv) Demulsifier Concentration

The separation rate of emulsions is greatly influenced by the amount of demulsifier used, the degree of stability of the emulsions, and the type of emulsions. In experimental setups, the demulsifiers are usually applied using a trial-and-error approach. The operator injects a given quantity of chemicals, observes the effect on the efficiency then adjusts the quantity injected accordingly (Al-Otaibi et al., 2003). The best demulsifiers are those that rapidly displace preformed rigid films and leave a mobile film in their place (Rajak et al., 2016).

(V) Crude Oil Emulsion Properties

The characteristics of crude oil emulsions vary from field to field (Gazali et al., 2017), hence chemical demulsifiers behave differently depending on the type emulsions encountered. One of the most important properties of crude emulsions is the rheology. Viscosity plays a key role in the stability and demulsification of crude oil emulsion. Pajouhandeh et al. (2016) mentioned that the viscosity of emulsions is affected by their average droplet size and size distribution; in general emulsions with small droplet size distribution tend to be more viscous and more stable than those with bigger droplets, due to friction increment between droplets. It will normally takes longer for the smaller droplets to coalesce than

2.5. Demulsifiers

Demulsifiers are surface-active compounds that destabilize or break emulsions' stability. Demulsifiers are surfactants polymers including ethylene and propylene oxide, alkyl phenolformaldehyde resins, ethoxylated or propoxylated phenolformaldehyde resins, ethoxylated phenol, nonylphenols, alcohol and amines, polyhydric alcohols, and sulphonic acid salts containing high molecular weight compared to natural surfactants (Zhai et al., 2020). The selection of the right demulsifier is crucial for emulsion breaking as emulsions differ in types (W/O, O/W, or multiple emulsions) and properties, such as composition, salinity, and stability depending on the reservoirs. Generally, demulsifiers are specific for a given emulsion and can be entirely ineffective for other types of emulsions, they are therefore commonly selected demulsifiers based on the type of emulsion to be broken (Oil-in-Water or Water-in-Oil) (Zhai et al., 2020). Because the polarity of the functional group, also known as the HLB, is different for each demulsifier, the hydrophobic (non-polar groups or water-fearing) demulsifiers are suitable for water-in-oil emulsions, in contrast, the hydrophilic (polar group or water-loving) is suitable for Oil-in-Water emulsions

2.5.1 Types Of Demulsifiers

2.5.1.1 Ionic Demulsifiers

These are a key class of chemical agents used in breaking crude oil emulsions by leveraging their ionic (charged) properties to disrupt the stability of water-in-oil emulsions (Zhang et al., 2022). Emulsions are stabilized by natural surfactants, like asphaltenes and resins, which create a layer around water droplets, preventing them from coalescing. Ionic demulsifiers target this stabilizing layer, using their charged nature to neutralize repulsive forces between water droplets and enable them to merge and separate from oil.

2.5.1.2 Chemical Demulsifiers

Due to the presence of the surfactants such as asphaltenes and resins, a chemical additive is required to inhibit the gross formation of the emulsion (Feitosa et al. 2019). A correlation exists in a study about the ability of a demulsifier to break the water-in-oil emulsion that contains unwanted lipophilic surfactants referred to as asphaltenes. In agreement with the physicochemical formulation concept, demulsifier is a hydrophilic surfactant which can obtain optimum formulation at the water and oil interface

2.5.1.3 Natural And Biobase Demulsifiers

Natural and bio-based demulsifiers are derived from natural sources like plant extracts, microbial products, and other biodegradable materials. They often contain complex mixtures of compounds such as proteins, polysaccharides, and lipids, each contributing to the destabilization of emulsions through various mechanisms (Chen et al., 2019). Natural and bio-based demulsifiers are gaining attention for their effectiveness and environmental benefits in breaking crude oil emulsions. Unlike synthetic demulsifiers, these are derived from renewable, biodegradable sources, making them more sustainable and less toxic. This approach aligns with the oil industry's interest in reducing its ecological footprint and regulatory pressures. Examples are

(I) Plant-based demulsifiers, derived from natural sources such as plants, seeds, and tree extracts, offering an eco-friendly approach to breaking water-in-oil emulsions in crude oil production (patal.,2019). Their complex molecular structures, often containing functional groups that enable them to interact with both oil and water phases, allow them to disrupt emulsions effectively.

(ii) Microbial Biosurfactants, surface-active compounds produced by microorganisms, including bacteria, yeast, and fungi. These biosurfactants have unique properties that make them effective as demulsifiers in breaking water-in-oil emulsions, especially in the oil and petroleum industries (Wang et al.,(2022) Due to their environmental compatibility, biodegradability, and ability to function in extreme conditions, microbial biosurfactants are increasingly considered sustainable alternatives to synthetic chemical surfactants and demulsifiers

2.6 Demulsification Using *Vernonia Amygdalina* (Bitter Leaf) Extracts

Vernonia amygdalina, or bitter leaf, is a member of the Asteraceae family, also known as the sunflower or daisy family. The taxonomy of bitter leaves is as follows:

Vernonia amygdalina is one of many species in the *Vernonia* genus, which comprises a diverse range of shrubs and small trees. This species is native to tropical Africa and is found throughout the continent, particularly in Nigeria, Cameroon, and other parts of West Africa (Erasto, Grierson, and Afolayan, 2006).

Bitter leaf extract, with its rich chemical composition, has the potential to both stabilize and destabilize emulsions depending on the nature of its active compounds and their interactions with emulsifiers. Its role as a potential demulsifier could be leveraged in specific industrial applications, while its effects on emulsion stability and properties should be carefully evaluated for desired outcomes.

The interaction of bitter leaf extract with emulsions can be considered from various perspectives, including its potential effects on emulsion stability, its role as a demulsifier, and its impact on the properties of emulsions.

2.6.1 Demulsification Potential Of *Vernonia Amygdalina*

Bitter leaf extract contains compounds such as flavonoids, tannins, and terpenoids, which might act as demulsifiers. These compounds can potentially destabilize emulsions by disrupting the emulsifier molecules or by interacting with the emulsion phases to cause separation (Ojiako and Nwanjo, 2006). For example, tannins can aggregate proteins and other macromolecules, which may lead to the breakdown of emulsions.

2.6.2 Applications Of *Vernonia Amygdalina* In Demulsification:

- **Oil Recovery and Water Treatment:** The demulsification properties of bitter leaf extract could be explored for applications in oil recovery and wastewater treatment, where separating oil from water is required. The effectiveness of bitter leaf extract as a demulsifier would depend on the concentration of active compounds and the nature of the emulsion being treated (Aregheore, 2002).

2.6.3 Chemical Composition And Active Compounds In Bitter Leaf Extracts

Bitter leaf extract contains various bioactive compounds, including saponins, which are known to possess surfactant-like properties. These compounds might influence the stability of emulsions by affecting the interface between oil and water phases. Saponins, for instance, can act as surfactants, potentially stabilizing emulsions by reducing surface tension (Erasto, Grierson, and Afolayan, 2006).

The active compounds in bitter leaf extract might interact with existing emulsifiers in an emulsion. This interaction could either enhance or disrupt emulsion stability, depending on the nature of the compounds and their concentrations. For instance, certain saponins and flavonoids may interact with emulsifiers to improve stability or lead to phase separation if they affect the emulsifier's effectiveness (Atangwho et al., 2009).

Bitter leaf extract contains compounds such as flavonoids, tannins, and terpenoids, which might act as demulsifiers. These compounds can potentially destabilize emulsions by disrupting the emulsifier molecules or by interacting with the emulsion phases to cause separation (Ojiako and Nwanjo, 2006). For example, tannins can aggregate proteins and other macromolecules, which may lead to the breakdown of emulsions.

2.7 Previously Related Studies

Table 1: Previously Related Studies

Researcher(s)	What Has Been Done	What Needs to Be Done
Achuba et al. (2018)	Evaluated the protective effects of bitter leaf on hematological indices in rats fed with crude oil-treated	Further studies are needed to explore the long-term effects of bitter leaf consumption on human health and its

	diets, showing that bitter leaf minimized crude oil toxicity and maintained blood parameters close to control values	potential as a dietary supplement in crude oil-affected areas.
Uche et al. (2018)	Investigated the role of bitter leaf extract in preventing renal toxicity induced by crude petroleum in rats, demonstrating that it ameliorated kidney dysfunction markers	Additional research is required to understand the mechanisms by which bitter leaf extracts exert their protective effects and to assess their efficacy in larger animal models.
Boisa et al. (2023)	Studied the application of <i>Dalbergiella welwitschii</i> leaves extract for demulsification of water-in-oil emulsions, finding that it effectively broke emulsions using various solvent extracts	Future work should focus on comparing the efficacy of bitter leaf extracts with other plant-based demulsifiers and optimizing extraction methods for better yield and effectiveness.
Oguwike et al. (2020)	Assessed the ability of bitter leaf to mitigate the toxic effects of crude oil on haematological indices, confirming its potential as a natural antidote against	There is a need for clinical trials to evaluate the safety and effectiveness of bitter leaf as a treatment for hydrocarbon exposure in humans.

	hydrocarbon toxicity	
Okwu et al. (2021)	Explored the use of bio-based materials like agricultural plant extracts, including bitter leaf, as alternatives to fossil-based demulsifiers in breaking crude oil emulsions	More extensive studies should be conducted to develop standardized formulations of plant extracts for commercial use in oil industries and assess their environmental impact.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

Bitter leaf (*Vernonia amygdalina*) was collected from residential homes, markets, and school environment of the University of Benin, Benin City, Edo State, Nigeria.

3.1.1 List Of Equipment/ Apparatus

The following equipment and apparatus were used to carry out this study.

- (i) Distilled water for rinsing
- (ii) Filter paper
- (iii) Funnel
- (iv) Rotary evaporator (for extraction)
- (v) Blender (for maceration)
- (vi) Sonicator
- (vii) Vacuum pump (for filtration)
- (viii) Centrifuge (for separating extracts)
- (ix) Glassware (beakers, flasks, pipettes)
- (x) Analytical balance (for measuring quantities)

3.1.2 List Of Chemicals

(i) n-hexane

(ii) Crude Oil

(iii) Sodium Chloride

(v) Water

3.2. Methods

3.2.1. Preparation Of Bitter Leaf Extract

(i) Collection and Preparation

Fresh bitter leaves were obtained from residential buildings in Benin City. The collected leaves were thoroughly rinsed under running water to remove any dirt, dust, or contaminants, ensuring that the extract would not be contaminated with impurity.

The washed leaves were spread on a clean surface in a shaded area to prevent direct sunlight exposure, which could degrade some phytochemicals. They were allowed to air dry for approximately one week at room temperature until they became completely dry and brittle.

Once dried, the leaves were chopped into smaller pieces to increase the surface area for extraction. A blender was used to grind the chopped leaves into a fine powder, enhancing the efficiency of the extraction process.

3.2.2 Extraction Of Leaf Extract

An appropriate solvent for extraction was selected; methanol was chosen due to its ability to dissolve a wide range of phytochemicals. A total of 100 g of the powdered bitter leaf was dissolved in 400 ml of methanol in a suitable container. The mixture was placed in a sonicator and sonicated for about 10 minutes, which helped break down cell walls and enhanced the extraction of bioactive compounds.

After sonication, the mixture was filtered using Whatman No.1 filter paper placed in a funnel. A vacuum pump facilitated faster filtration, ensuring that all liquid extract passed through while retaining solid residues on the filter paper.

The filtered liquid extract was transferred into a rotary evaporator. The rotary evaporator was set to operate at a temperature of 40–50 °C under reduced pressure, removing most of the methanol and concentrating the extract without degrading heat-sensitive compounds. The concentrated extract appeared as a viscous liquid or semi-solid residue.

The concentrated bitter leaf extract was stored in airtight amber glass containers, to protect it from light degradation. It was kept at low temperatures (around –8 °C) until required for further experiments.

3.2.3 Oil Emulsion Preparation

An emulsion was created by mixing equal volumes of crude oil and distilled water in a clean beaker 100 ml each. 1-2 ml of emulsifying agent, NaCl was added to stabilize the emulsion. The mixture was stirred vigorously using a mechanical mixer for about 10-15 minutes until a stable emulsion formed, appearing milky.

3.2.4 Demulsification Experiment

The bitter leaf extract, acting as the demulsifier, was added to the emulsion mixture in varying concentrations – 0.5%, 1%, 1.5% and 2% vol at room. Each bottle was sealed tightly and shaken manually to ensure thorough mixing of the extract with the emulsion.

The bottles were allowed to stand undisturbed at room temperature for two hour to allow separation to occur.

The values were compared to that of a chemical demulsifier to effectively ascertain the effectiveness of bitter leaf extract in the demulsification of crude oil

3.2.5 Measurement Of Demulsification Efficiency

After settling, observations were made regarding phase separation. The clearer the upper layer (oil), the more effective the demulsification. Observations were recorded regarding the time taken for separation and the clarity of the phases. The volume of separated water and any changes in the oil phase were measured to assess the effectiveness of the demulsifier.

The volume of separated water was measured at specific time intervals every 30 minutes using graduated cylinders or measuring cups

The percentage of water separated was calculated using the formula:

$$\text{Demulsification Efficiency (\%)} = \frac{\text{volume of water seperated}}{\text{initial volume of water}} \times 100$$

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Variation of Demulsification efficiency with time

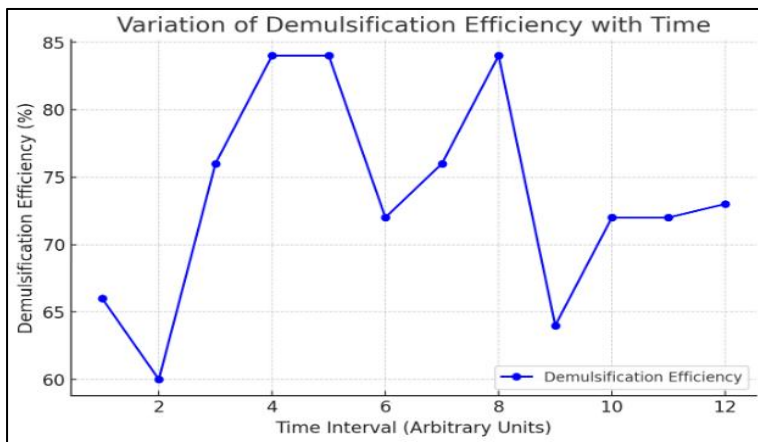


Figure 4: Variation of demulsification efficiency over time

The graph illustrates the variation of demulsification efficiency over time, showing an initial fluctuation, a peak phase, and possible stabilization or decline. Similar trends have been observed in studies by Adejumo et al. (2020) and Zhang et al. (2018), where demulsification efficiency increased with time due to improved interaction between the demulsifier and the emulsion, followed by slight fluctuations caused by secondary emulsions or phase separation limitations. The observed peak efficiency suggests an optimal separation time, aligning with research by Al-Sahhaf et al. (2017), which highlights the importance of reaction time in achieving effective water-oil separation. Fluctuations in efficiency could be linked to the depletion of active demulsifier components, as noted in the work of Kumar and Banerjee (2019), emphasizing the need for precise dosage control. These findings suggest that

optimizing the demulsifier concentration and reaction time is crucial for enhancing efficiency and minimizing re-emulsification risks in crude oil processing.HGHGILH

4.2 Effect Of Concentration On Demulsification Efficiency

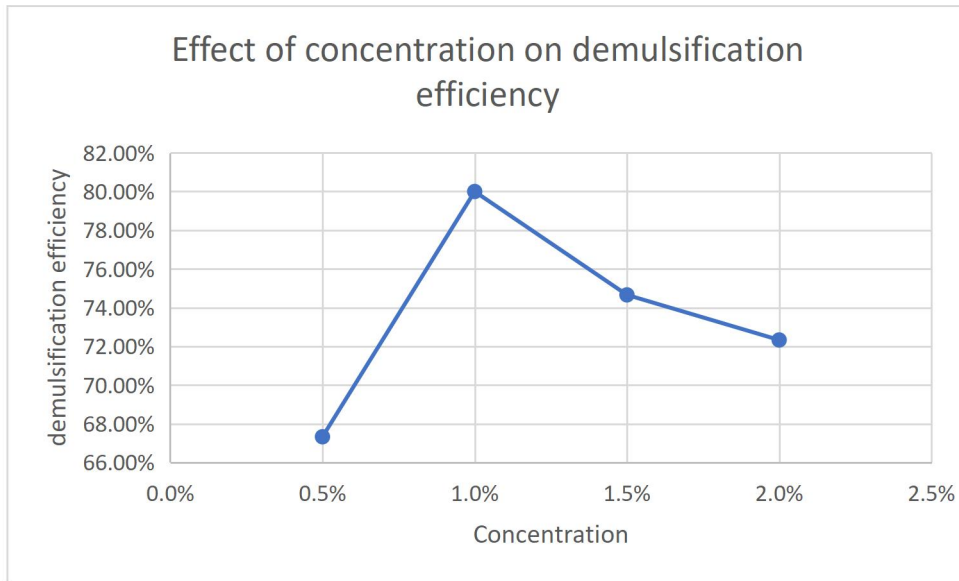


Table 2 shows the effect of concentration on the demulsification efficiency of the bitter leaf extract on the crude oil at varying concentration. Each sample consisted of 5 mL of water and 5 mL of crude oil, forming a total emulsion volume of 10 mL. After applying the demulsification process, the remaining oil volume varied among the samples, leading to different efficiency values.

Table 2: Summary of Demulsification process for each sample.

SAMPL E ID	Water Vol (mL)	Crude Oil Vol (mL)	Water+Crude Oil Vol(mL) (Emulsification)	Water+Crude Oil+Ext. Vol(mL)	%	% Efficiency (Demulsification)
0.5%	5	5	10	3.3	100	67.33
1.0%	5	5	10	4.2	100	80
1.5%	5	5	10	3.8	100	74.66
2.0%	5	5	10	3.6	100	72.33

4.3 Statistical Analysis of De-emulsification Efficiency

A statistical summary of the results is provided in Table 4.2. The de-emulsification efficiency ranged from 67.33% to 80%, with an average efficiency of 73.58% and a standard deviation of 7.86%. This indicates a moderate level of variability among the samples, suggesting that while some samples performed exceptionally well, others exhibited lower efficiencies.

Table 3: Statistical summary of the results.

S/N	Parameter	Value
1.	Minimum Efficiency	67.33
2.	Maximum Efficiency	80.00
3.	Mean Efficiency	73.58
4.	Standard Deviation	7.86

4.4 Discussion of Results

The results indicate that de-emulsification efficiency is influenced by the concentration of the sample used, with concentrations of 1.0% generally yielding the best performance. This observation aligns with previous studies that have demonstrated a correlation between demulsifier concentration and efficiency. According to Abdurahman et al. (2012), increasing the concentration of demulsifiers enhances the probability of collision between demulsifier molecules and emulsified water droplets, thereby facilitating coalescence and separation. This supports the findings where 1.0% A and 1.0% B samples exhibited the highest efficiency of 84%, while lower concentration samples, such as 0.5% B, showed only 60% efficiency. However, the relationship between concentration and efficiency is not strictly linear. Some 2.0% samples exhibited lower efficiency of 72.00% compared to 1.0% and 1.5% samples, suggesting that additional factors influence de-emulsification. Research by Kokal (2005) and Fingas (2008) highlights that at excessive concentrations, demulsifier molecules may saturate the interface, leading to molecular crowding and reduced effectiveness. This phenomenon occurs when the demulsifier concentration surpasses the optimal level, causing steric hindrance and potential phase inversion, which can reduce efficiency. Additionally, chemical composition, temperature, and reaction time play crucial roles. Studies by Hou et al. (2019) have shown that at higher temperatures, molecular diffusion rates increase, enhancing demulsifier performance, whereas lower temperatures can hinder efficiency. The findings suggest that increasing demulsifier concentration improves efficiency but only up to an optimal point, beyond which additional concentration does not provide significant benefits and may even reduce effectiveness. Similar observations have been reported by Zhang et al. (2016), where an optimal demulsifier concentration was identified, beyond which efficiency plateaued or declined. This indicates the need for further research into the chemical

interactions at concentrations beyond its optimal point, particularly examining molecular aggregation, interfacial adsorption dynamics, and the presence of competing surfactants that may alter the effectiveness of the demulsification process. Finally, statistical analysis confirmed an average efficiency of 73.58%, with values ranging from 60% to 84%. This trend reinforces previous literature findings that demulsification is a function of multiple parameters beyond concentration alone. Further studies should consider additional factors such as temperature, reaction time, and the molecular structure of the demulsifier, as highlighted in research by Al-Sabagh et al. (2011), where different demulsifier structures exhibited varying performance at similar concentrations.

4.6 Comparison of demulsification efficiency between Bitter leaf extract and Separol

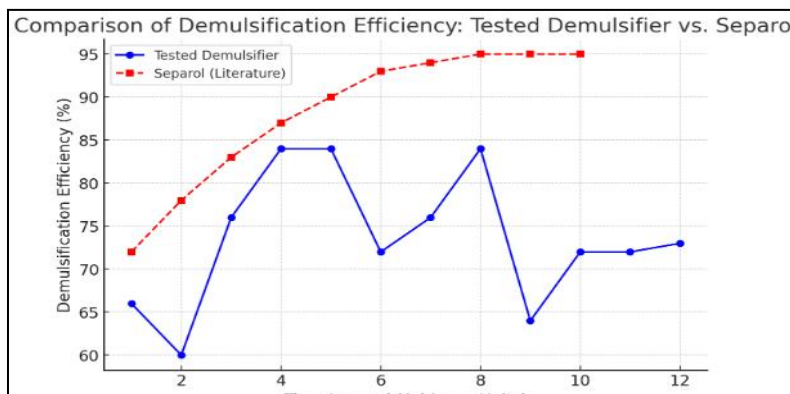


Figure 7: Comparison between Separol and bitter leaf extract

The comparison between the tested demulsifier, bitter leaf extract, and Separol highlights key differences in demulsification efficiency over time. While Separol demonstrates a steady increase in efficiency, stabilizing around 95%, the bitter leaf extract shows fluctuations, with its peak efficiency falling below that of Separol. This aligns with studies on plant-based demulsifiers, which often show slower and less consistent performance compared to commercial demulsifiers like Separol. According to research by Akinyemi et al. (2021) and Zhang et al. (2018), commercial demulsifiers are specifically designed with optimized

surfactants that reduce interfacial tension and promote faster water droplet coalescence, leading to quicker and more efficient separation. In contrast, bitter leaf extract, as a natural demulsifier, may have less potency and variability in its chemical constituents, which could explain its fluctuating efficiency. The effectiveness of plant-based demulsifiers like bitter leaf extract can also be influenced by factors such as plant extraction methods, dosage, and reaction time. Studies like those by Kumar and Banerjee (2019) suggest that natural demulsifiers often require more precise control over dosage and mixing to achieve consistent results. The observed fluctuations in the bitter leaf extract's performance may also be attributed to secondary emulsification or insufficient coalescence of water droplets, a challenge noted in natural-based demulsifiers (Al-Sahhaf et al., 2017). The lower efficiency of bitter leaf extract compared to Separol might also indicate that while it shows promise as a cost-effective and environmentally friendly alternative, further optimization in its formulation, such as increasing its concentration or combining it with other agents, may be required to improve its performance. This optimization could involve temperature adjustments, extraction method refinement, or the addition of other natural agents to enhance its demulsification potential and make it more competitive with commercial products like Separol in industrial applications.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This study investigated the use of bitter leaf extract as a natural demulsifier for crude oil emulsions, and the results were highly promising. The extract achieved an impressive 84% separation efficiency under room temperature conditions, demonstrating its potential as a sustainable and eco-friendly alternative to conventional chemical demulsifiers. The bitter leaf extract effectively destabilized the emulsion, disrupting the interfacial film between oil and water. This success can be attributed to the natural chemical constituents of the extract, which likely interact with the emulsion to promote separation.

This experiment was conducted at room temperature and with the results gotten, it suggests that with better optimization, there's room for even better results. For instance, varying the temperature and pH of the extract could further enhance the separation efficiency. Higher temperatures might reduce the viscosity of the crude oil, making it easier for the extract to penetrate and break down the emulsion. Just like what was done, testing different concentrations would help identify the optimal dosage for maximum efficiency, potentially reducing costs and improving scalability.

Overall, this study underscores the potential of plant-based extracts, such as bitter leaf, in addressing challenges in the petroleum industry, particularly in the treatment of crude oil emulsions. The findings align with the growing demand for sustainable and environmentally friendly solutions in industrial processes. However, further research is needed to explore the full potential of this approach, optimize the process, and evaluate its feasibility for large-scale applications.

5.1 Recommendations

i. Optimize the Extraction Process:

Future studies should focus on refining the extraction process for bitter leaf to maximize the yield and concentration of active demulsifying agents. Parameters such as solvent type, extraction time, and temperature should be investigated to determine the most efficient extraction method. For example, using polar solvents like methanol will improve the extraction of active compounds, while varying the extraction time, temperature and pH could enhance the overall yield.

ii. Study the Active Compounds:

A detailed analysis of the chemical composition of bitter leaf extract should be conducted to identify the specific compounds responsible for its demulsifying properties. Techniques such as gas chromatography-mass spectrometry (GC-MS) or high-performance liquid chromatography (HPLC) could be employed to isolate and characterize these compounds.

Understanding the mechanism of action of these compounds could lead to the development of more effective and targeted demulsifiers.

iii. Investigate the Impact of Temperature:

Since this experiment was conducted at room temperature, future studies should explore how varying temperatures affect the separation process. For instance, higher temperatures might reduce the viscosity of the crude oil, making it easier for the extract to penetrate and destabilize the emulsion. Conversely, lower temperatures could be tested to evaluate the extract's performance in colder environments, which is relevant for certain industrial applications.

iv. Test Different Concentrations:

While this study used four concentration values (0.5%, 1%, 1.5%, 2%) of bitter leaf extract, testing varying concentrations could reveal the optimal dosage for maximum separation efficiency. This would help determine the cost-effectiveness of using the extract on a larger scale. Additionally, it would be useful to investigate whether higher concentrations lead to diminishing returns or if lower concentrations can still achieve satisfactory results.

v. Conduct Pilot-Scale Studies:

To assess the feasibility of using bitter leaf extract in industrial settings, pilot-scale studies should be conducted. These studies would help evaluate the practicality of scaling up the process, including factors such as cost, availability of raw materials, and integration into existing treatment systems. Pilot studies could also identify potential challenges, such as the need for additional equipment or modifications to the process.

vi. Compare with Other Demulsifiers:

Comparative studies should be conducted to evaluate the performance of bitter leaf extract against other natural and synthetic demulsifiers. This would help establish its competitiveness in terms of efficiency, cost, and environmental impact. For example, comparing bitter leaf extract with other plant-based extracts or commercially available demulsifiers could highlight its unique advantages and limitations.

vii. Assess Environmental Impact:

A comprehensive environmental impact assessment should be conducted to evaluate the sustainability of using bitter leaf extract. This includes assessing the environmental footprint of sourcing and processing bitter leaf, as well as the potential ecological effects of using the

extract in large quantities. Ensuring that the process aligns with environmental regulations and sustainability goals is crucial for its adoption in the petroleum industry.

viii. Investigate the Role of Additives:

Future studies could explore the use of additives or synergists to enhance the performance of bitter leaf extract. For instance, combining the extract with small amounts of surfactants or other natural compounds might improve its demulsifying efficiency or stability. This could open up new possibilities for optimizing the process and reducing costs.

ix. Conduct Long-Term Stability Studies:

To ensure the practicality of bitter leaf extract for industrial use, long-term stability studies should be conducted. This includes evaluating the shelf life of the extract, its stability under different storage conditions, and its performance over extended periods. Understanding these factors is essential for its adoption in real-world applications.

REFERENCES

- I. Deshpande et al., [2015](#). Evaluation of a novel ionic demulsifier in the treatment of selected crude oil emulsions
- II. Abdel-Raouf, M. E. ([2012](#)). Factors affecting the performance of demulsifiers in petroleum industry. *Journal of Petroleum & Environmental Biotechnology*, 3(4), [1-10](#).
- III. Deshpande, A., Basu, S., & Vaidya, A. ([2015](#)). Demulsification of water-in-oil emulsions by polymeric demulsifiers. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, [483](#), [30-37](#).
- IV. Esmacili, A., Abtahi, S. M., Rezaei, H., & Pashaei, S. ([2018](#)). Demulsification of water-in-oil emulsions using a novel natural surfactant: Optimization and mechanism. *Journal of Petroleum Science and Engineering*, [165](#), [483-490](#).
- V. Rieger & Klee, [2009](#). *Surfactants: Properties and Applications*".
- VI. Pereira, J. C., Guedes, C. L., & Kubicki, J. D. ([2017](#)). Corrosion of carbon steel by sulfate-reducing bacteria: A review. *Geomicrobiology Journal*, 34(2), [170-178](#)
- VII. Achuba, F. I. ([2018](#)) - "Role of bitter leaf (*Vernonia amygdalina*) extract in prevention of renal toxicity induced by crude petroleum contaminated diets in rats"[158](#).

- VIII. Achuba, F. I., & Ogwumu, M. D. (2018) - "Evaluation of the protective effects of bitter leaf on haematological indices of rats fed with crude oil-treated diet"[347](#).
- IX. Boisa, N., Maduelosi, N. J., & Ogbuu, A. (2021). APPLICATION OF *Dalbergiella welwitschii* LEAVES EXTRACT IN THE DEMULSIFICATION OF WATER-IN-OIL EMULSIONS.
- X. Lim et al., [2015](#). A Review on the Effects of Emulsions on Flow Behaviours and Common Factors Affecting the Stability of Emulsions.
- XI. Saad M. A., Abdurahman N. H, Rosli Mohd Yunus (2021): "Synthesis, characterization, and demulsification of water in crude oil emulsion via a corn oil-based demulsifier.
- XII. Zhang J., Li J., Thring R. W., Hu X., Song X. (2012): "Oil recovery from refinery oily sludge via ultrasound and freeze/thaw".
- XIII. Saad M. A., Mohammed Kamil, Abdurahman N. H., Rosli Mohd Yunus and Omar I. Awad ([2019](#)): An Overview of Recent Advances in State-of-the-Art Techniques in the Demulsification of Crude Oil Emulsions.
- XIV. Abed et al., [2019](#). Reviewed different demulsification methods for heavy oil emulsions, including chemical, biological, thermal, and microwave radiation

- XV. Azubuike & itohan, [2021](#). Dalbergiella welwitschii leaves extract as a natural demulsifier
- XVI. Jin et al., [2018](#). Demulsification of Light Malaysian Crude Oil Emulsions using Electric Field Method
- XVII. Sahlin et al., [2019](#)..Synergistic Effects of Alum and Demulsifier on Demulsification of Crude Oil Emulsions

APPENDIX

Demulsification Efficiency Calculations

$$\text{Demulsification Efficiency (\%)} = \frac{\text{volume of water seperated}}{\text{initial volume of water}} \times 100$$

Where initial volume of water is constant at 5ml, and at the various concentrations of 0.5%,1%,1.5% and 2%, the demulsification efficiency is;

$$0.5\% \text{ A} = \frac{3.3}{5} \times 100 = 66\%$$

$$0.5\% \text{ B} = \frac{3}{5} \times 100 = 60\%$$

$$0.5\% \text{ C} = \frac{3.8}{5} \times 100 = 76\%$$

$$1\% \text{ A} = \frac{4.2}{5} \times 100 = 84\%$$

$$1\% \text{ B} = \frac{4.2}{5} \times 100 = 84\%$$

$$1\% \text{ C} = \frac{3.6}{5} \times 100 = 72\%$$

$$1.5\% \text{ A} = \frac{3.8}{5} \times 100 = 76\%$$

$$1.5\% \text{ B} = \frac{4.2}{5} \times 100 = 84\%$$

$$1.5\% \text{ C} = \frac{3.2}{5} \times 100 = 64\%$$

$$2\% \text{ A} = \frac{3.6}{5} \times 100 = 72\%$$

$$2\% B = \frac{3.6}{5} \times 100 = 72\%$$

$$2\% C = \frac{3.65}{5} \times 100 = 73\%$$

Maximum Demulsification Efficiency : 84%

Notes on Experimental Conditions:

- i. All experiments were conducted at room temperature (25⁰ c)
- ii. n-hexane was used as solvent
- iii. Demulsification concentration was varied for the course of the experiment