PRODUCTION OF NATURAL SURFACTANT USING BITTERLEAF EXTRACT WITH BASIC ALKALINE SOURCE FROM CORN COB ASH FOR ENCHANCEMENT OIL RECOVERY

By

TRACY ETUSI

ENG2002032

A PROJECT SUMMITED TO THE DEPARTMENT OF CHEMICAL ENGINEERING,

UNIVERSITY OF BENIN ,BENIN CITY, NIGERIA.

OCTOBER, 2025.

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IN PARTIAL FUFILLMENT OF THE REQUIREMENT FOR THE AWARD OF BACHELOR OF

ENGINEERING IN CHEMICAL ENGINNEERING

OCTOBER, 2025.

CERTIFICATION

This **is** to certify that this project work was carried out by **ETUSI TRACY** with matric number **ENG202032** of the Department of Chemical Engineering**, University** of Benin,Benin City, Edo State.

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DEDICATION

This work is dedicated to God Almighty who has keep me to this stage and also to my parents, friends and well-wishers for their support , care and love throughout my years in this University.

ACKNOWLEDGEMENT

All thanks to God Almighty who created the whole universe for these great opportunity to be alive and healthy throughout my stay in the university ,Special thanks goes to Mr. and Mrs. Etusi ,my sibling ,friends and well-wishers and also my generous project supervisor Prof Aluyor and my coordinator Dr .F. O Oshomogho for their support throughout my project work.

Special thanks to the Head of Department and other Academic and Non Academic staff of the Department of Chemical Engineering for their voluntary efforts, my fellow project mate for their support when carrying out my project work **.**

Special thanks goes to Luco chemical laboratory limited for all the available resources, tools and equipment made available to carry my project work.

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NOMENCLATURE

CSS- Cyclic Steam Stimulation

EI – Emulsification Index EOR – Enhanced Oil Recovery

FTIR – Fourier Transform Infrared Spectroscopy

GCMS-Gas Chromatography- Mass Spectroscopy

IFT – Interfacial Tension ISC- In-Situ Combustion

V_e – Volume of emulsion layer (mL)

Vt – Total volume of the liquid mixture (Ml

ABSTRACT

This study investigates a plant-based surfactant alternative as a result of the growing need for surfactants that are both economical and ecologically friendly for enhanced oil recovery. These research project comprises the study of enhance oil recovery with it crucial application of Natural surfactant from bitter leaf extract , while using alkaline from calcined corn cob ash to form basic medium . Corn cobs were calcined for 3 hours at 750°C to produce the alkali, which was then extracted using distilled water to produce the alkaline for the medium, the extraction process for the bitter leaf to extract the saponin content to produce the surfactant The extraction process was carried out according to the experimental design with 19 runs with independent variables of extraction time (30-300 minutes) ,mass of bitterleaf (1-10grams) and temperature (50-90 degree census),and a constant volume of 100 ml of methanol with a single response yield (%), Alkaline surfactant was also produced using alkaline hydrolysis of the saponin was carried out to form the surfactant and75ml of the saponin was mixed with 25ml of the ash solution.

Various Physical characteristics was carried out in the process such as forth test where 2.0 gms portion of the powdered sample was boiled into 20ml of distilled water in a test tube in boiling water bath and filtered , 10ml of the filtrate was mixed with 5ml of distilled water and shaken vigorously to foam, Total Saponin Content Quantitative Analysis where a quantity ,1.0 gms of the powdered sample was weighed using electric weighing balance into 25ml beaker and soaked with 100 ml of 20% Methanol for 3 minutes and heated for 3 hours at 55 degree census for proper extraction then filtered and lastly . The volume and stability of the emulsion was observed and the emulsion index was calculated.

Chemical analysis such as FTIR (Fourier _ transform infrared spectroscopy) these analysis was done to get information about the different functional groups in the dried weighted surfactant produced **,** GCMS of a chat which shows the x –axis which represent the time range and the y –axis as the intensity each peaks correspond with the various compound in the dried weighted sample produced from the bitter leaf using a GC column and for the MS it shows the mass to charge ratio with relative abundances of the bitter leaf plant extract.

CHAPTER ONE

INTRODUCTION

1.1. BACKGROUND OF STUDY;

The beauty of these research project is to produce a natural surfactant from Bitter leaf botanically known as Vernonia Amygdalina as an environmentally sustainable and cost-effective alternative to synthetic surfactants used in Enhanced Oil Recovery (EOR). Vernonia Amygdalina (VA) is a plant found in Africa and Asia region and often refer to as bitter leaf because of its bitter taste (Adenutsi, 2023) . Some of its constituent are used in making pharmaceutical products and cosmetics. The phytochemical break down of its extracts shows the presence of saponin based natural surfactant (SBNS). Saponins are natural plant glycosides with powerful foaming nature in aqueous solution. it foaming is due to the presence of non-polar sapogenin and water-soluble side chains, which has an extreme look alike in structure to synthetic surfactants (S. Najimi , 2019) . Saponin extraction method has high solvent consumption, and also long extraction period (H. Imuetinyan, 2021). More recently, the use of ultrasonic extraction has more economic, effective and a clear extraction method for green plants (M. Pordel, 2012).

The extraction process enhances the extraction effectively and detach structural destruction and degradation of the complex starch (O.M. Haghighi, 2020). Surfactant are chemical constituent which decrease the surface tension or interfacial tension between two fluid (P .T. jaf, 2023). The word ***SURFACTANT*** is a mixture of "surface-active agent" designed in 1950 as they contain a water-repellent and a water-attracting part, they enhance water and oil to mix, they can form foam and facilitate the detachment of dirt (B S.Hisgel, 2023) .Surfactants are also the most widely and commercially relevant chemicals (S. Naijimi, 2019). Household as well as several industries use them in large extend as detergent and cleaning agents, and also as emulsifiers, wetting agent ,foaming agent ,antistatic additives (Nowrouzi, 2019).

Surfactant are organic chemicals arranged by hydrocarbon linkage chain (hydrophobic makeup – the tail) and a hydrophilic makeup (the head), it is grouped by the nature of the head as anionic, cationic, nonionic, and zwitterionic (Zhang et al.., 2020). Nonionic surfactants has no presences of charge; anionic surfactants has a negative charge on its polar head, w cationic surfactants has a positive charge. Also, zwitterionic surfactants has both positive and negative charge on its polar head. Based on chemical arrangement and placement, surfactants does adsorb into solid or liquid interface and at the solid to liquid and liquid to liquid surfaces, mostly at lower concentrations (Joshi et al .., 2015). This specially changes the physicochemical constituent of the systems, such as slowly degrading the interfacial tension of water to oil interfaces and altering the wettability of reservoir rocks. These effects changes above and below the critical micelle concentration (CMC). Above CMC, surfactants in solution generate wider particles known as micelles. In the study of EOR bases , it is important to input surfactants above the CMC to accomplish low IFT values, enhance foam stamina , and reduce adsorption on the reservoir rock interface) (Pogaku et al .., 2018).

EOR methods helps to get more oil than primary and secondary recovery procedures, consisting of natural production and also of water injection (Pogaku et al., 2018).

These procedures are known as conventional, and tertiary recovery been coined as EOR. The three procedures are recognized, to enhance the recovery of oil. EOR methodology assist with the aid of more oil recovery by enhancing displacement and sweeping effectively . One of the most effective EOR procedures are the use of surfactants, polymers and alkalis.

Recently, research worldwide on the need of oil and gas increases rapidly , with current changes in substitute energy channels such as solar, wind , geothermal and hydroelectric energy (Grassia et al.., 2016). There is a pressing need for oil as an energy medium because of renewable energy are not able to meet this achievable demand for global consumption (Shamsijazeyi et al., 2014). The globe improvement in industrialization is the basic reason for the high need for oil factor . Getting embedded hydro carbons and enhancing the making of oil results to the major issues in petroleum and chemical engineers due to needs of pressure (Abbas et al., 2020).

Manufacturing of oil and gas starts with extraction of hydrocarbons from well depose as a cause of reduction in pressure within the well (Karatayev et al.., 2019) . After the use

of primary and secondary recovery procedures are used , a reasonable amount of oil still remains in the as bypassed / residual oil enhanced Oil Recovery (EOR) skills has been used recently (Gyan et al.., 2019).

Surfactant flooding is a recognized or unique way of chemical enhance oil recovery. This research has used to enhance oil recovery by practicing similar mechanisms such as;

1. interfacial tension (IFT) reduction
2. Wettability alteration
3. foam creation
4. emulsification

Surfactant flooding is affected by problems such as instability under harsh (or normal) well conditions and over adsorption (He et al., 2019). These obstacles that disturbs the actual oil recovery and causes reduction in the economics of EOR projects. Surfactants can also be selected in accordance to the wells conditions or rock type. This is mostly carried out by using surfactant screening procedures , that impose small relationship with the interfacial tension , surfactant adsorption and some other causes on some chemicals variables such as temperature and salinity conditions (Rezaei et al .., 2020). The organized perspective of surfactant characterization and also the phase behavior, the duties of surfactants in oil recovery, surfactant adsorption in reservoir rock, and also the use of surfactants in EOR both in labs and field measures (Tayari et al .., 2018) .

1.2. AIMS AND OBJECTIVES;

The aim is to extract natural surfactant from bitterleaf for use in crude oil extraction for Enhance oil Recovery .

The objectives are as follows;

1. Alkaline preparation from corn cob ash.
2. To prepare surfactant from bitter leaf as a natural surfactant for EOR.
3. Perform a foam test, emulsion test and a saponin test to know the quality of the surfactant to be produced.
4. Experimental study by considering Interfacial reduction.

1.3 .SCOPE OF THE STUDY:

This research project is a laboratory conducted study carried out at Luco chemical laboratory located at 200 Uselu Lagos road Benin city Edo state. The preparation of the natural surfactants is to be made from Bitter leaf for enhanced oil recovery (EOR). Preparation of a suitable understanding of the naturally made surfactant and a specific known synthetic surfactant.

The Uniqueness of bitter leaf as a natural surfactant medium that can lower Interfacial Tension (IFT) and enhance the effectiveness of oil recovery has become very productive . In well or reservoirs, bitter leaf is used as a natural surfactant that enhances oil displacement and lower ITF with high saponin content and surface active agent . The extraction the surfactants from bitter leaf , with respect to the extraction procedures and breakdown of how the surfactants performs when carrying out EOR.

1.4. RELEVANCES OF THE STUDY;

Plant-based alternatives are becoming more and more popular as a result of the rising need for biodegradable and ecologically friendly surfactants. Bitter leaf has been recognized as a possible source of bio surfactants due to its extensive phytochemical content. Furthermore, using alkali made from agricultural waste—like corn cobs—offers a sustainable and environmentally beneficial method of producing surfactants. Though a lot of study has been done on microbial and synthetic surfactants, little has been established about how to produce surfactants generated from bitter leaf utilizing alkali from corn cobs for enhanced oil recovery. Research on bio surfactants has mostly examined microbial sources such as sophorolipids and rhamnolipids (Negin et al., 2017b), with little investigation into plantbased substitutes. Although bioactive substances like saponins and glycosides, which have surfactant-like qualities, are found in bitter leaf, little has been established about how to systematically extract and purify these substances using alkali made from corn cobs. Instead of investigating bio-based alternatives like alkali obtained from corn cob ash, the majority of studies 34 concentrate on traditional alkali sources (such as sodium hydroxide and potassium hydroxide (Sahare et al., 2012).

Surfactants produced from Bitter leaf need to meet specified physicochemical qualities, such as surface tension reduction, emulsification efficiency, and stability under reservoir conditions for Enhanced Oil Recovery (EOR) applications. Few research have evaluated the efficacy of bitterleaf surfactants to reduce interfacial tension (IFT) in oil-water systems (Chatterjee & Muralidhar, 1995b). In comparison to conventional synthetic and microbial surfactants, very few research examine the foaming capacity, wettability alteration, and emulsification efficiency of bitter leaf surfactants. Comparative research on surfactants obtained from bitter leaf is limited, despite the extensive study of bio surfactants originating from microorganisms (Navaei & Esmaeilnezhad, 2020). Research on using waste biomass, like maize cobs, to produce surfactants is still in its development; the majority of studies concentrate on producing bioethanol .

1.5. METHODOLOGY;

Alkali made from corn cobs was used in the study to produce a natural surfactant from bitter leaf. The procedure was broken down into several key stages:

1**. Alkali production from maize cobs;** was accomplished by crushing, drying, and washing the corn cobs. After that, they were calcined for 3 hours at 750°C to produce ash corn cob ash and distilled water were combined to create a solution, which was then heated, agitated, and allowed to soak for few hours before being filtered to extract the alkali solution.

1. **Preparation / Extraction of bitter leaf extract**: The extraction process was carried out according to the experimental design with 19 runs with independent variables of extraction time (30-300 minutes) ,mass of bitterleaf (1-10grams) and temperature (50-90 degree census),and a constant volume of 100 ml of methanol with a single response yield (%) .Each

runs was subjected to a reflux condenser and their respective extract were dried in an oven at 90 degree census and placed in a sealed plastic container until it was used for analysis.

2. **Akaline solution preparation:** A solution was created by mixing 20g of the corn cob ash with 500ml of distilled water. The solution was agitated using a magnetic stirrer. It was then heated using a heating mantle and allowed to cool and soak for 48 hours. Then, the soaked ash was then filtered from the solution using a filter paper and a funnel to get the liquid which is the alkali. The pH of the liquid was determined using pH paper to know if it was alkaline.
3. **Alkaline surfactant preparation:** Alkaline hydrolysis of the saponin was carried out to form the surfactant. 75ml of the saponin was mixed with 25ml of the ash solution. The mixture was agitated in a magnetic stirrer for one hour that also supplied heat. The resulting solution was the alkaline surfactant.

4. **Physical characterization of the surfactant produced;**

Froth Test (Foam test Qualitative analysis): 2.0 gms portion of the powdered sample was boiled into 20ml of distilled water in a test tube in boiling water bath and filtered , 10ml of the filtrate was mixed with 5ml of distilled water and shaken vigorously to foam a persistent froth for the formation of emulsion characteristic of Saponins (Obadoni et al..., 2001) which lasted for up to 5 minutes.

Total Saponin Content Quantitative Analysis: A quantity ,1.0 gms of the powdered sample was weighed using electric weighing balance into 25ml beaker and soaked with 100 ml of 20% Methanol for 3 minutes and heated for 3 hours at 55 degree census for proper extraction then filtered. The residue was re –extracted with another 100ml 0f 20% methanol .The two extracts were combined and heated to 40ml at 90 degree census on a water bath .the concentrate was transferred into a 250ml separating funnel and 20ml of n-hexane was added and shaken vigorously , the upper layer was discarded .The purification process was repeated and 60ml of n-butanol was added, the tower layer was discarded while the upper layer was collected .The combined n- butanol extract was washed with 10ml of 5% aqueous NaCl brine solution and the lower layer was discarded and the upper layer was collected in a weighed beaker and heated to dryness .the breaker was allowed to cool and was reweighed.

Emulsification test: The mixture was done in a volume ratio of 1:1 (Zhang et al., 2022). 2 ml of oil sample and 2m of distilled water were accurately measured with a syringe into a test tube. The process was repeated into two other test tubes. 2 ml of just the saponin extract was added to one of the test tubes, 2 ml of the alkaline surfactant was also added to one of the test tubes and nothing was added to the last test tube. The volume and stability of the emulsion was observed and the emulsion index was calculated .

6. Chemical Characterization of the produced Surfactant:

Fourier Transform Infrared Spectroscopy (FTIR): This analysis was done to get information about the different functional groups in the dried weighted surfactant produced, the saponin extract and the alkaline surfactant produced**.**

GCMS (Gas chromatography – Mass spectroscopy): A chat which shows the x –axis which represent the time range and the y –axis as the intensity each peaks correspond with the various compound in the dried weighted sample produced from the bitter leaf using a GC column and for the MS it shows the mass to charge ratio with relative abundances of the dried surfactant content.

1.6. STATEMENT OF THE PROBLEM ;

Enhancing oil recovery from existing reservoirs is a challenge facing the petroleum industry, especially when conventional procedures become less effective and environmental concerns about synthetic surfactants increase. The limitations of synthetic surfactants, which are mostly expensive, hazardous, and sourced from non-renewable resources, have been identified by the need for efficient and ecologically friendly Enhanced Oil Recovery (EOR) techniques. Natural surfactants, which come from renewable resources, offer a viable substitute because of their lower toxicity and less detrimental effects on the environment. Although natural surfactants present an alternative, their application in EOR is underexplored.

CHAPTER TWO

LITERATURE REVIEW

Surfactant plays a very crucial role in Enhanced oil recovery (EOR) methods help retrieve extra crude oil from oil field and use to maximize output from reservoir beyond what is recoverable by primary and secondary recovery methods. In order to improve oil mobilization, particular surfactants—are important for lowering the interfacial tension (IFT) between water and oil. (Elrufai, A.2020) Furthermore, surfactants plays a crucial role in enhanced oil recovery by reducing interfacial tension and altering rock wettability , allowing more oil to be extracted which improve more effective oil displacement and improves recovery efficiency overall. Bio-based surfactants offers several advantages such as environmental benefits, effectiveness and sustainability. It covers all modes of oil-recovery processes (drive, push-pull, and well treatments and most oil-recovery agents. EOR improvement are also being used for in-situ extraction of organic pollutants from permeable media (Rita, N.1972). In these context , the extraction is referred to as remediation and the pollutant as the gain. The reference of these technologies still refer to carbon dioxide (CO2) storage. It does not only apply EOR to a specified phase (primary, secondary, and tertiary) in the crucial life of a reservoir. Primary recovery are oil recovery with natural medium : solution gas, water influx, gas-cap drive, or gravity drainage, Secondary recovery refers to ways , such as gas or water injection, that have the important purpose of encouraging or maintaining reservoir pressure (Raheem, A. A. 2009) Tertiary recovery is a technique used after secondary recovery. Almost all EOR medium have been at least tested as secondary method. Many thermal process are ready in both primary and secondary mode(Adesanya,2009). Interest has made based on tertiary EOR, but the definition does not relate any of such delay n explanation is to improved EOR, which is EOR plus a lot of innovation that are also made to increase apex oil recovery . they include hydraulic fracturing, horizontal and multilateral wells, infill drilling, well stimulation, and enhancing the manufacturing or inserting rates of personal wells(Almahfood, M., & Bai, B.2018). This review is an approach in better use of bitter leave biologically known as Vernonia Amygdalina with saponin content of about 60mg/g to 801.33mg/g (Oyefolu, P. K. 2021).

Horizontal reservoir, wear Artificial lift Pressure maintenance Pump, gas lift, etc. Water/gas reinjection are Secondary recovery, Tertiary recovery Enhanced recovery Conventional recovery and Unconventional recovery Displacement Thermal Chemical Solvent Other Natural flow — Oil- recovery classes (adapted from Oil & Gas Journal) . Benefit of enhanced oil recovery (EOR) requires comprehension of equal side of chemistry and engineering. Each of these relate our understanding through one flowing phase which may compound some components. moreover, because of varying temperature, pressure, and composition, these part may mix completely in some site of the flow domain, causing the removal of a phase in those site(K.O Azike,2021). Atmospheric pollution, ground water flow, and chemical and nuclear waste storage causes problems.

This essay gives the equations that relate multiphase, multicomponent fluid flow permeable media to this conservation such as laws for mass, energy, entropy, and linear constitutive theory (Manrique, E. 2010).At first, we work for the most generality by allowing the transport of each part in each phase. Also, we obtain specified cases from the main equations by making more assumptions. The goal in arriving at the unique equations is as valid as the equations themselves because it will help to comprehend the specific hypothesis—and the constrains—that are being brought about for a specific application, (M. A., & Ferreira, I. C. F. R.

2019). The result for conservation of mass initially consist of two basic different manner for the overall equations: overall compositional stability and phase-conservation equations. The general compositional balance s are help full for stimulate the manner in which components in rural thermodynamic equilibrium are moved through permeable means (Pinela Jbarros,2019).

The phase-conservation equations are help full for stimulating multiple mass transfer along phases. Our formulation difference medium for multiphase, multicomponent flows. For example, it contains As specified cases for the multicomponent, single-phase flow equations which are three phase, multi component equations (Crichlow 1977; Peaceman 1977; Coats et al. 1980; Skjaveland 1991). (Toddand Chase 1979; Fleming et al. 1981; Larson 1982) having presented multicomponent, multiphase formulations for flow in permeable medium, except with assumptions such as proper mixing or incompressible fluids ,A lot of these prediction must be ready before the equations are analysis but we are encourage to keep the formulation as the overall possibility for as long as possible (Todand Chase 1979; Fleming et al. 1981; Larson 1982).

2.1. BACK GROUND STUDY OF ENHANCEMENT OIL RECOVERY;

Enhanced Oil Recovery (EOR), also known as tertiary recovery, is the extraction of crude oil from an oil field that could not be extracted with other method(S..,etal, 2012). EOR is a medium of extracting oil from a reservoir that has already attained primary and secondary process of oil recovery (John, 2021). Primary production mostly recovers less than 20% of the real oil (Bülow, 2012). Secondary recovery procedures, like gas or water injection, are meant to sustain reservoir pressure (Pope & Morsi, 1987). EOR techniques are used when primary and secondary methods have been used up (John, 2021). When EOR is use ,it account for 30% to 60% or more of the wells oil can be extracted, compared to 20% to 40% involving only primary and secondary recovery. EOR ability of altering the chemical make up of the oil to make it more easier to extract, instead of relying on the pressure measures between the surface and the underground reservoir (John, 2021). EOR techniques includes injecting materials not present in petroleum well (Arriola & Morsi, 1987). These method can be difficult and costly, making them optional only when primary and secondary recovery techniques are no longer useful or when the price of oil is high enough to fulfill the investment (John, 2021). Petroleum companies put hopes on EOR to consistently expand the life of reservoir in sustainable oil fields. There are four classes of EOR techniques: carbon dioxide (CO2) injection, other gas injection, thermal EOR, and chemical EOR. Gases, like carbon dioxide, are mainly injected into the reservoir, to reduce the oil's viscosity and push it to The surface (Bülow, 2012). The Co2 can be pumped into the reservoir to heat the oil and make it less viscous, or a polymer chemical structures can also be injected into the reservoir(RossenW.E, 2001). especially when conventional procedures become less effective and environmental concerns about synthetic surfactants increase. The limitations of synthetic surfactants, which are mostly expensive, hazardous, and sourced from non-renewable resources, have been identified by the need for efficient and ecologically friendly Enhanced Oil Recovery (EOR) techniques. Natural surfactants, which come from renewable resources, offer a viable substitute because of their lower toxicity and less detrimental effects on the environment. Although natural surfactants present an alternative, their application in EOR is underexplored. The potential of bitter leaf as a natural surfactant source that can successfully lower Interfacial Tension (IFT) and increase the efficiency of oil recovery has gained attention. In reservoirs, bitter leafmay be used as a natural surfactant that increase oil displacement and lower interfacial tension because of its high saponin concentration and surface-active qualities. Nevertheless, little is known about the production procedures used to extract these surfactants from bitter leaf, especially with regard to improving extraction techniques and analysing how well the surfactants work in enhanced oil recovery (EOR) applications.

2.1.1. TYPES OF ENHANCED OIL RECOVERY ENHANCED OIL RECOVERY (EOR);

Advanced techniques aimed at extracting extra oil from reservoirs that surpass the capabilities of primary and secondary recovery methods. A range of EOR techniques has been created to enhance recovery efficiency and facilitate oil mobility. The main types of EOR are thermal EOR, chemical EOR, and gas injection EOR, each possessing distinct mechanisms and uses (Owen & Floyd, 1981).

2.1.1.1 Thermal EOR: Thermal Enhanced Oil Recovery (EOR) uses heat to lower the viscosity of oil, which increases its flow and eases extraction. This technique is especially successful when dealing with heavy crude oil and tar sands (Muzzafaruddin, 2019). Different types of thermal EOR includes;

I**. Steam Injection**; Steam injection is the most commonly utilized thermal enhanced oil recovery (EOR) method. It entails introducing steam into the reservoir to raise the temperature of the oil, thereby decreasing its viscosity and enhancing its flow. There are two primary categories:

a**. Cyclic Steam Stimulation**: Cyclic Steam Stimulation (CSS), commonly referred to as the "huff and puff" technique, involves injecting steam into a well, permitting it to soak for a period, and then utilizing the same well for production.

b. **Steam Flooding**: This technique entails the ongoing injection of steam through specific injection wells, driving the heated oil towards the production wells (Green & Willhite, 2018).

II. **In-Situ Combustion (ISC):** This technique involves burning a segment of the oil within the reservoir to produce heat, which subsequently lowers the viscosity of the leftover oil. Although ISC is more intricate than steam injection, it can potentially recover as much as 80% of the oil available in the reservoir (Thomas, 2008).

III**. Hot Water Flooding**: Hot water flooding is a method that involves injecting heated water into the reservoir to decrease oil viscosity, serving as an alternative to steam flooding. Although it is not as efficient as steam injection, it demands less energy and can be used in certain reservoir conditions (J. Sheng, 2020).

2.2. WHAT ARE SURFACTANT ?

Surfactants are known as surface active agents use to treat surface tension and reduce wettability(S..,etal ,2012). Surfactants are also known as amphiphiles because they have polar heads and no polar tails. The polar heads are called hydrophilic heads and they are attracted to polar solvents, The non-polar tails are called the hydrophobic tails (Embid, S.M. 1999]. Surface tension is reduced as a result of these molecules' structural dimensions, which reduces the cohesive forces that brings water molecules together (lakeL .W 2009). Aside from reducing surface tension, they are used as emulsifiers, foaming agents, corrosion inhibitors and antistatic agents (Nagtode et al., 2023).

Surfactants are good emulsifying agent, dispersing and foaming agents because of their surface activeness. Surfactants polar compounds in dissolving their organic solvents. They are use for production of soaps and detergents (Anderson .G, 2006), food production, agriculture and pharmaceuticals.

Surfactant are organic chemicals arranged by hydrocarbon linkage chain (hydrophobic makeup – the tail) and a hydrophilic makeup (the head), it is grouped by the nature of the head as anionic, cationic, nonionic, and zwitterionic (Zhang et al 2020). Nonionic surfactants has no presences of charge; anionic surfactants has a negative charge on its polar head, w cationic surfactants has a positive charge.

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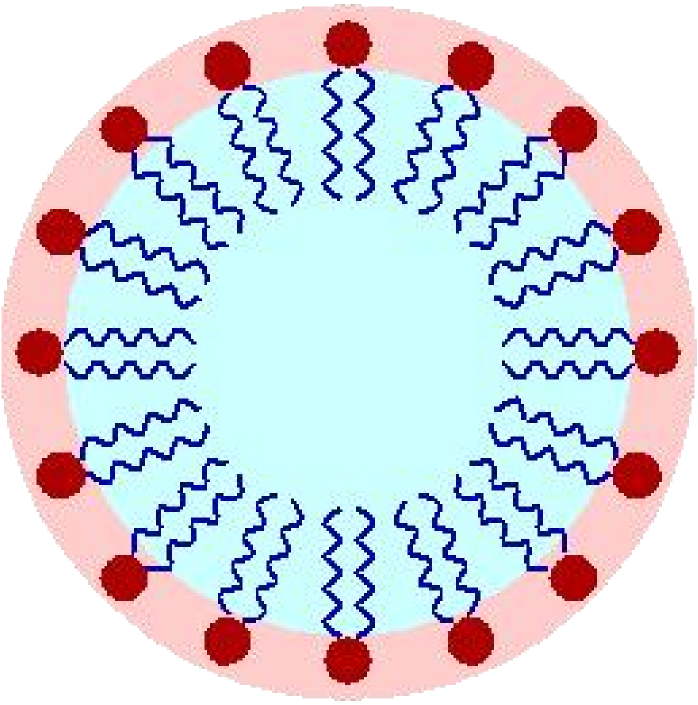

Fig 2.2. Schematic illustration of a surfactant (Saad et al… 2020).

2.3. TYPES OF SURFACTANT;

2.3.1. ANIONIC SURFACTANT; Anionic surfactants are known by a negatively charged hydrophilic head (Tiwari et al., 2018).when dissolved in water they generate negatively charged surface group, whose aqueous solution is neutral or alkaline (Yuan et al., 2014). This negative charge comes from functional groups such as sulfate, sulfonate, phosphate, or carboxylates (Yuan et al., 2014). The negative charge head helps react with positively charged or neutral surfaces making them effective in removing oily and particulate soils from surfaces. Anionic surfactants are commonly found in laundry detergents, dishwashing liquids, shampoos, and hand soaps (Yuan et al., 2014).

2.3.2. CATIONIC SURFACTANTS; Cationic surfactants have positively charged hydrophilic head group (Tiwari et al., 2018). Cationic surfactants are dissolved in water for surface activity of positive ions (Yuan et al., 2014). They are active when subjected to acid medium and reduces when subjected to alkaline medium. (Yuanet al., 2014). The positive charge is typically related to ammonium ion (Phil et al., 2000). Cationic surfactants are not commonly used due to their low detergency and higher cost. However, they show special characteristics that make them useful in special applications. Cationic surfactants are used as fabric softeners and antistatic additives. Cationic surfactants are spiltted into open-chain cationic surfactants, heterocyclic group cationic surfactants and bonded. (K.O Azike, 2021).

2.3.3. NONIONIC SURFACTANTS; Nonionic surfactact has hydrophilic head group that has no charge (Tiwari et al., 2018). Their hydrophilic head is derived from the presence of polar groups, such as polyethylene glycol (PEG) chains or polyol groups (© EOSCA EOSCA European Oilfield Speciality Chemicals Association). Nonionic surfactants are milder and less irritating than ionic surfactants. Nonionic surfactants are used in detergents, emulsifiers, and wetting agents. They get their polarity from an oxygen–rich shares of the molecules at one end. Examples are alcohol ethoxylates, alkyl polyglucosides (APGs) and sorbitan esters (Phil et al., 2000).

2.3.4. AMPHOTERIC SURFACTANTS .Amphoteric surfactants, known as zwitterionic surfactants, has both positive and a negative charge hydrophilic head group charge can change Due to the pH concentration in the solution (Phil et al., 2000). Examples of amphoteric surfactants are betaines and sultaines. . They are good foaming and cleaning properties and they conforms both with both anionic and cationic.

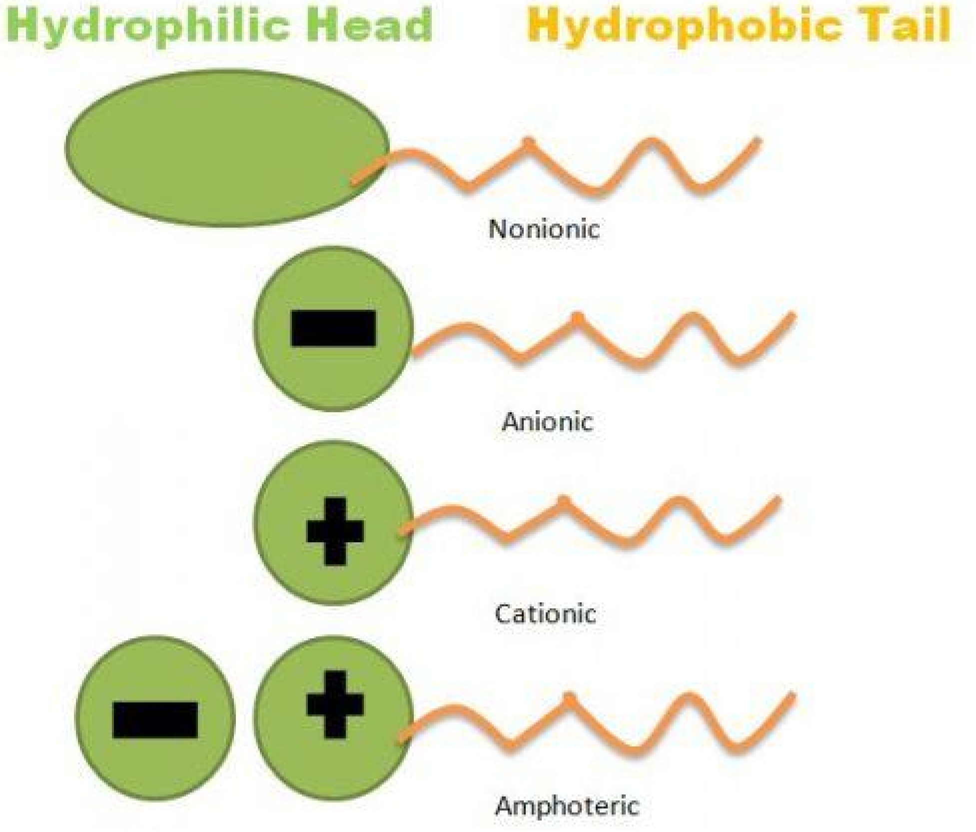

Fig 2.3. Schematic illustration of the types of surfactant (phil et al., 2002)

2.4. MECHANISM OF NATURAL SURFACTANTS IN ENHANCEMENT OIL RECOVERY

The role of a surfactant in EOR is to reduce the interfacial tension between the oil in the reservoir and the injected water(Han et al., 2022). surfactants facilitate the removal of oil droplets from the rock surface, helping their movement towards production wells. Surfactants can also facilitate the wettability of reservoir rocks, Changing them from oil-wet to water-wet, which changes the oil by water, leading to more recovery rates. It create microemulsion systems together with the crude oil at the interfacial between the oil and water thus lowering their interfacial tension (IFT) to ultra-low (0.001 mN/m), which will constantly mobilize the remaining oil and will help improve oil recovery (Bülow, n.d.). Enhanced oil recovery (EOR) methods are employed to extract additional crude oil from reservoirs beyond what is recoverable by primary and secondary recovery methods In order to improve oil mobilization, chemical EOR techniques—in particular, surfactants—are essential for lowering the interfacial tension (IFT) between water and oil. Furthermore, surfactants have the capacity to alter the wettability of reservoirs, which encourages more effective oil displacement and improves recovery efficiency overall. Bio-based surfactants have received great interest for their environmental benefit and cost-effectiveness.

2.5. ADVANTAGES OF NATURAL SURFACTANT IN ENHANCEMENT OIL ROCOVERY

Surfactant formation should be valid in reservoir medium before being used in oil recovery medium this is carried out using surfactant screening, which is a problem of process that needs time and material bases (Jin et al .., 2017,). There are two reasons that make surfactant screening difficult . Firstly, the types of surfactants used largely affected the conditions of the oil reservoir in addition to the the kind of the reservoir rock (Ivanova et al.. 2020).

Secondly, there is a wide variety of surfactants that has proper useage for EOR (Miller et al ..,2020); starting with a specific group of surfactants to be used is also challenging. Because of the importance of this issue, the parameters and process that are usually considered during surfactant screening. also presently a variety of the most commonly used surfactants in chemical EOR.Surfactant adsorption is an special parameter that affects the resourcefulness of surfactant flooding and the economic importances of EOR projects (Amirmoshiri et al .., 2020), surfactant adsorption is disturbed by the temperature, salinity, pH and other reservoir parameters (Puntervold et al .., 2018). important research has put theirs on improving the procedures used to maintain surfactant adsorption, thereby lowering its negative involvement on oil recovery. examples include changing the salinity and ionic composition of the injection water to alkali addition and nanoparticle (NP) (Cheraghain 2017,). Aside lessing surfactant adsorption, specific types of NPs help to improve the properties of surfactant solutions oil recovery is enhanced. It is important to note that NPs improves other chemical EOR processes (Cheraghian et al .., 2020,), which are listed in other parts of the context .The benefit of applying surfactant in EOR are;

1. Reduction of interfacial tension; Surfactant reduces the surface tension between oil and water reducing capillary force that traps oil in rock pores.
2. Mobilization of trapped oil; reducing IFT helps for the mobilization of trapped oil that could be recovered using common method.
3. Change in wettability; it helps change rock properties from oil-wet to water- wet which helps for easy movement of the oil.
4. Environmental benefit; Surfactant reduces the total amount of chemicals required in reducing pollution risk for enhancing better soil properties.
5. improved performance and stability of oil; it increases oil recovery process and prevent emulsion from forming or breaking them apart.
6. Synergistic effect on nanoparticles; Increases stability by improving the performances of dispersion ,increases form ability and stability and reducing interfacial tension.

2.6. BITTER LEAF *Vernonia amygdalina*, AS A NATURAL SURFACTANT

The importance of bitter leaf found worldwide exhibit a wide variety of health importance has capture the interest of researchers (Farombi & Owoeye, 2011). Bioactive compounds involved in these plants has physiological involvement that effect the human health that make it an interest for consumption .however, it is important that more 80 % of people use bitter leaf as either their dietary usage .Bioactive make of these bitterleave account for antioxidant antibacterial, antidiabetic ,anticancer, antiasth ma, antiparastic (Tungmunnithum et al., 2018). In tropical regions of Africa, the plant Vernonia amygdalina; known as bitter leaf because of the bitter taste of the leaves. The plan tcomes from the Asteraceae family and are present in trace amount of about 2.5–3 m height (Achuba, 2018). Some tall V. amygdalina tree has 7 m of highet (Farombi & Owoeye, 2011). V. amygdalina consist of green color leaves that have a features odor (Akpoghelie et al., 2022). V. amygdalina has African names such as; ewuro (Yoruba), etidot (Efik), ityuna (Tiv), Congo Bololo (D. R. Congo), oriwo (Edo), onugbu (Igbo), grawa (Amharic), shuwaka or chusar-doki (Hausa),

2.6.1.CHEMICAL CONTENT OF BITTER LEAF ;

.The V. amygdalina leaf happens to proven to contain some quantities like

1. lipid
2. carbohydrates
3. protein and essential amino acid (Edo et al., 2022),
4. Fiber
5. iron
6. phosphorous
7. copper
8. calcium
9. potassium

10. cobalt
11. manganese.

Biological active compounds like ascorbic acid, saponins, alkaloids, steroids terpenes, flavonoids, coumarins, lignans,(Erhonyota et al., 2022).

Extracts from bitter leaf are used to treat a various illness such as, emesis, nausea, diabetes, anorexia, diarrhea, dysentery and other gastrointestinal issues (Adebukola Adeyanju et al., 2022) which explains why many herbalists uses them.it also discovered that the relevance some

compounds in bitter leaves like vernodalin and vernonioside, can lower the activity of inflammatory enzymes like COX-1 and COX-2 (Farombi & Owoeye, 2011), leading to a less in pain and inflammation , bitter leaf as a useful treatment various inflammatory conditions like arthritis, asthma (Sweis & Cressey, 2018). bitter leaf extracts also been found to have antioxidant properties (Iruoghene Edo, 2022). The antioxidants in bitter leaf are used to neutralize harmful free radicals that contribute to tissue damage (Edo et al., 2023).

Also, organic extracts from bitter leaf demonstrated cytotoxic effects to nasopharyngeal cancer cells (Hussain et al., 2022). It makes them a beneficial treatment for conditions like cancer, diabetes and liver sickness .The antimicrobial extracts of bitter leave is also use to treat ailment at a wide range.

Recently , the readily availability of bitter leaves in supplement bases makes it uses for people their daily diets. However, the view aims to get a understandable overview of the bioactive components such as the saponin content whch is about 6 percent can use as surfactant based on their foaming characteristics and for it to have and hydrophic head and hydrophobic tail making it active to recovery more oil from oil rock resevior.(Rossen W.R.,2006)

2.7. ROLE OF ALKALINITY OF CORN COBS ASH IN PRODUCTION OF SURFACTANT

The alkali in alkaline-surfactant-polymer (ASP) helps in oil recovery. it effect based on alkali, surfactant, and polymer can help promote emulsion promotion , reduce surface tension, and enhance bulk viscosity, and enhancing flooding efficiency (Li et al., 2024). The main of Alkalinity in Surfactant is that when added it increase efficiency to help reduce surfactant adsorption in the resevior . it combines with organic acids in crude oil to increase surfactants action (Li et al., 2024). The reaction between alkali and surfactants gives ultra-low interfacial tension (Li et al., 2024). The active substances active in the alkali react with the surfactant to form a more useful interfacial film(Li et al., 2024). Alkalinity crucial roles are:

I. **Reduction of Surfactant Adsorption:** Alkali reduces the adsorption of surfactants the rock face, by increases the efficiency of the surfactant.

II. **In-situ Surfactant Formation:** can react with organic acids present in crude oil to produce surfactants within the reservoir (Li et al., 2024).

III. **Interfacial Tension Reduction:** The combination of alkali and surfactants can achieve ultra-low interfacial tension between oil and water, which is essential for mobilizing trapped oil (Li et al., 2024).

IV. **Emulsification Promotion:** Alkali can also promote the emulsification of crude oil, which aids in the displacement of oil (Li et al., 2024).

V. **Wettability Alteration:** Alkali can change the wettability of rocks (Li et al., 2024).

VI. **Regulation of pH and Salinity:** It helps in regulating pH and salinity (Li et al., 2024).

2.8. COMMON ALKALIS USED IN SURFACTANT PRODUCTION

Comon alkalis role in promoting the performance of surfactant systems, especially in applications such as (EOR). we explore some of the common alkalis commonly used in surfactant production.

2.8.1.Sodium Hydroxide (NaOH); Alkali for ASP is Sodium Hydroxide (NaOH). Caustic soda, another name for sodium hydroxide, used in the manufacturing of surfactants. When reacting with surfactants, it is well known for its ability to efficiently lower interfacial tension (IFT), which enhances the surfactant ' emulsification and wettability process (Kumar & Mandal, 2016). By changing the wettability of reservoir rocks and lowering the IFT between oil and water, NaOH is employed in alkalisurfactant-polymer (ASP) flooding to improve oil recovery (Wang et al., 2019).

2.8.2. Sodium Carbonate (Na2CO3) Alkali for ASP is Sodium Carbonate (Na2CO3). ASP flooding has a strong alkaline type to a poor alkaline type (Na2CO3)(Li et al., 2024). Another popular alkali in the synthesis of surfactants is sodium carbonate, sometimes known as soda ash. It is highly after for its capacity to reduce the promotion of surfactant adsorption on reservoir rocks, which is for preserving the surfactant's effectiveness throughout EOR procedures (Rezaei et al., 2020). Anionic surfactants and sodium carbonate are used to produce ultra-low IFT s, which are necessary for effective oil recovery (Lv et al., 2011).

2.8.3.Ammonium Hydroxide (NH4OH); In oil recovery applications, ammonium hydroxide is

employed as an alkali in surfactant systems to improve surfactant effectiveness. other alkalis like NaOH and Na2CO3, it aids in lowering the IFT and changing the wettability of reservoir rocks (Kumar & Mandal, 2016). In some reservoir situations when other alkalis may not work as well, ammonium hydroxide can be useful.

2.8.4. Sodium Metaborate (SMB); Another alkali used in the synthesis of surfactants, especially in EOR . It is known for its ability to increase the surfactant system's efficiency by reducing the adsorption of surfactants on reservoir rocks (Kumar & Mandal, 2016). To .enhance the improvement of the flooding operation, SMB is frequently combined with additional alkalis and surfactants.

2.8.5. Diethanolamine (DEA); In surfactant systems, diethanolamine used as an alkali to enhance the surfactant's emulsification and wettability properties. It works very well to lower the IFT between water and Oil

2.8.1.1. POTENTIAL ALKALINE REACTION IN FORMATION ARE;

$2NaOH+Ca+2 \rightarrow 2Na++Ca\ (OH)2$

$2NaOH+Mg+2 \rightarrow 2Na++Mg\ (O$

$2\ Na2CO3+Ca+2 \rightarrow 2Na++CaCO3$

2.9. CORN COB ASH AS A SUSTAINABLE ALKALI SOURCE

Corn cob ash (CCA) is obtained by burning corn cob waste to a high temperature (Kumari et al., 2018). The chemical composition of CCA is dependent on factors such as the type of corn, crop timings, climate, and geographical conditions(Kumari et al., 2018). Composition and Properties of Corn Cob Ash CCA contains a combined content of about 70% of SiO2 and CaO (Singh et al., 2018).The chemical composition of CCA includes (Kumari et al., 2018):

1.Silica (SiO2); Corn cob ash contains 52.32% silica (Rofiqoh, 2024) .

2. **Alumina (Al2O3);** corn cob ash contains 5.85% alumina.
3. **Lime (CaO);** corn cob ash contains 3.50 % calcium oxide .
4. **Iron Oxide (Fe2O3);** corn cob ash contains 2.95% iron oxide.
5. **Magnesia (MgO) ;** corn cob ash contain 2.11% magnesia.
6. **Sodium Oxide (Na2O);** corn cob ash contains 1.14% sodium oxide.
7. **Potassium Oxide (K2O);** corn cobs contains 23% of potassium oxide.
8. **Sulphate (SO3);** corn cob contains 1.3% of sulphur trioxide.

The silicon content in CCA is significant (Șerbănoiu et al., 2022). CCA consists of irregular particles with micropores, which are due to the burning of organic components (Kumari et al., 2018).

CHAPTER THREE

MATERIALS AND METHODS

3.1. RAW MATERIALS USED;

The raw materials used to carry out these research work are ;

1. **BITTER LEAVE**: botanically known as **Vernonia Amygdalina** it is the main constituent of materials used to carry out these research work , it is sourced locally from the bush , and dried properly through the means of sun rays which will aid the extraction of the saponin content for the production of the surfactant.

Plate 3.1 ; Pictorial view of dried bitterleaf.

2. **CORN COBS:** Fresh Corn cobs was obtained was waste from local market, properly washed to remove dirt and sun dried to obtain the ash for the production of the alkali .

Plate 3.2; Pictorial view of dried bitterleaf

3.2. CHEMICALS AND REAGENTS

Table 3.1. The Following chemicals were ultilized;

CHEMICALS/ REAGENT	**USES**
Methanol	Used as a solvent for extraction of the bitterleaf.
Hexane	Used for decantation process of the saponin content
n_butanol	Was used during decantation process for saponin content

chloride(NaCl)	A mixture of brine was prepared to separate the saponin from the extract
Distilled water	Used as solvent for blending of the extract and boiling process.
Olive oil	Used to for emulsion during froth process
Crude oil	Used for emulsification process

3.3 .EQUIPMENT/ APPARATUS

Table 3.2 The following equipment and apparatus was ultilized;

APPARATUS	**USES**
1. Soxhlet extractor	Used for the extraction of the bitterleaf.
2. FTIR (Fourier _ transform infrared spectroscopy)	Used to identify the chemical and structural composition of the bitterleaf extract
3. GCMS (Gas chromatography – Mass chromatography)	Used to separate the compounds , identify and quantify the bitterleaf extract.
4.Laboratory furnace	For the ashing process of the corncobs.
5. Glass wears (measuring cylinders, beakers, conical flask).	Used for collecting samples of the extract for purification process.

6.Test tube	Used for the shaking process during froth process.
7.Seperating funnel	Used for the liquid _liquid extraction of the brine and the saponin content.
8.filter paper	Used for the extraction of the saponin rich phase of bitterleaf extract.
9.Round bottom flask	Used for the solvent extraction process of the bitterleaf.
10. Oven drier	Used for solvent evaporation and drying process of the extract.
11.Retort Stand	Used to clip and hold the reflux condenser firmly.
13. Reflux condenser	Used for cooling process during extraction.
14.Magnetic stirrer	Used to set the various temperature and time varying for the heating process during extraction
15. Sensor	Used to regulate the set point temperature during extraction process.
16.Stir bar	Used to keep the extraction process in an agitated motion.
17. Spatula	For measuring the various dried bitter leaf
18. Electrical weigh balance	Used for measuring the weight of varios samples and solvent.

3.4. METHODS

The process carried out are as follows;

1. Preparation/ Extraction of bitter leaf.
2. Preparation of Corn cob Ash.
3. Physical characterization of the Surfactant.
4. Chemical characterizations of the produced Surfactant.

3.4.1. Preparation / Extraction of bitter leaf extract: The extraction process was carried out according to the experimental design with 19 runs with independent variables of extraction time (30-300 minutes) ,mass of bitterleaf (1-10grams) and temperature (50-90 degree census),and a constant volume of 100 ml of methanol with a single response yield (%) .Each runs was subjected to a reflux condenser and their respective extract were dried in an oven at 90 degree census and placed in a sealed plastic container until it was used for analysis.

3.4.2: Preparation of Corn Cob Ash : 500gms of the crushed corn cob was weighed in to a crucible and placed into a muffle furnace at 750 degree census for 3 hours for the ashing process in the absences of air , it was allowed to cool and placed in a tight sealed container

3.4.3: Alkaline solution preparation: A solution was created by mixing 20g of the corn cob ash with 500ml of distilled water. The solution was agitated using a magnetic stirrer. It was then heated using a heating mantle and allowed to cool and soak for 48 hours. Then, the soaked ash was then filtered from the solution using a filter paper and a funnel to get the liquid which is the alkali. The pH of the liquid was determined using pH paper to know if it was alkaline.

Plate 3.3 ; Alkaline solution preparation.

3.4.4: Alkaline surfactant preparation: Alkaline hydrolysis of the saponin was carried out to form the surfactant. 75ml of the saponin was mixed with 25ml of the ash solution. The mixture was agitated in a magnetic stirrer for one hour that also supplied heat. The resulting solution was the alkaline surfactant.

3.5.1. Physical characterization of the surfactant produced;

3.5.1.1: Froth Test (Foam test Qualitative analysis): 2.0 gms portion of the powdered sample was boiled into 20ml of distilled water in a test tube in boiling water bath and filtered , 10ml of the filtrate was mixed with 5ml of distilled water and shaken vigorously to foam a persistent froth for the formation of emulsion characteristic of Saponins (Obadoni et al..., 2001) which lasted for up to 5 minutes.

Plate 3. 4; froth test formation.

3.5.1.2: Total Saponin Content Quantitative Analysis: A quantity ,1.0 gms of the powdered sample was weighed using electric weighing balance into 25ml beaker and soaked with 100 ml of 20% Methanol for 3 minutes and heated for 3 hours at 55 degree census for proper extraction then filtered. The residue was re –extracted with another 100ml 0f 20% methanol .The two extracts were combined and heated to 40ml at 90 degree census on a water bath .the concentrate was transferred into a 250ml separating funnel and 20ml of n-hexane was added and shaken vigorously , the upper layer was discarded .The purification process was repeated and 60ml of n-butanol was added, the tower layer was discarded while the upper layer was collected .The combined n- butanol extract was washed with 10ml of 5% aqueous NaCl brine solution and the lower layer was discarded and the upper layer was collected in a weighed beaker and heated to dryness .the breaker was allowed to cool and was reweighed .The saponin content was determined using the following formula;

Plate 3. 5; total saponin content.

$$ExtractYield(\%) = \frac{W_0}{W_s} \times 100$$ --eqn 1

Where

W_o =mass of extract

W_s= mass of saponin obtained

3.5.2.1. EMULSIFICATION TEST: The mixture was done in a volume ratio of 1:1 (Zhang et al., 2022). 2 ml of oil sample and 2m of distilled water were accurately measured with a syringe into a test tube. The process was repeated into two other test tubes. 2 ml of just the saponin extract was added to one of the test tubes, 2 ml of the alkaline surfactant was also added to one of the test tubes and nothing was added to the last test tube. The volume and stability of the emulsion was observed and the emulsion index was calculated using the formula below.

$$EI\% = \frac{Ve}{Vt} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots eqn2$$

Where:

EI% = Emulsification Index (percentage)

V_e = Volume of the emulsified layer (ml)

Vt= Total volume of the liquid mixture (ml)

Plate3. 6; emulsification test of surfactant blend with crude oil and water and alkaline solution.

3.6.1. Chemical Characterization of the produced Surfactant:

3.6.1.1. Fourier Transform Infrared Spectroscopy (FTIR): This analysis was done to get information about the different functional groups in the dried weighted surfactant produced, the saponin extract and the alkaline surfactant produced**.**

3.6.1.2. GCMS (Gas chromatography – Mass spectroscopy): A chat which shows the x –axis which represent the time range and the y –axis as the intensity each peaks correspond with the various compound in the dried weighted sample produced from the bitter leaf using a GC column and for the MS it shows the mass to charge ratio with relative abundances of the dried surfactant content.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Alkali from Corn Cob Ash;

The chemical analysis of corn cob reported by (Adesanya & Raheem, 2009) indicated the presence of significant amounts of potassium oxide (K_2O), sodiumoxide (Na_2O), and calcium oxide (CaO), which contributes to its alkalinity. These compounds effectively react with water to form bases. The pH of the corn cob ash solution prepared in the lab was 8.5 when measured with a pH meter and this indicated alkalinity.

4.2 Extract result for the dried bitter leaf sample;

The design composite method was employed for the experimental design. The response obtained from the design composite was optimized using response surface methodology. Factors to be optimized were coded at 3 different levels, which gave the range for mass of sample (1-10g), Temperature (50 - 90 °C), and Time (30- 300 minutes). The saponin yield was chosen as the response for process optimization using Response Surface Methodology (RSM). The experimental design consisted of 19 runs. Experimental observations from the extraction process were analyzed and fitted according to a quadratic design model as a second-order polynomial equation. Analysis of variance (ANOVA) and response surface plot were generated using Design Expert software. The optimized value of the independent variables for optimum response was determined using numerical optimization.

4.2.1.Design Factors; These table shows the minimum and maximum range of the solid mass ,time interval and temperature range of sample extract from the bitterleaf of both low and high range values of the parameter from negative to positive intervals.

Table 4.1. Minimum and Maximum range code for solid mass, time and temperature range.

Name	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A: Solid mass (g)	1.0000	10.00	-1 ↔ 2.82	+1 ↔ 8.18	5.50	2.33
B: Time (minutes)	30.00	300.00	-1↔84.73	+1↔245.27	165.00	69.92
C:Temperature(℃)	50.00	90.00	-1↔ 58.11	+1 ↔ 81.89	70.00	10.36

Table 4.2. Extract yield for bitterleaf extract;

	A: Solid mass (g)	B: Time (minutes)	C: Temperature (℃)	Extract yield (wt%)
1	5.5	165	70	28.35
2	2.82428	245.271	81.8921	14.31
3	5.5	165	70	33.75
4	1	165	70	3.78
5	2.82428	84.7285	58.1079	5.94
6	5.5	165	70	33.75
7	5.5	165	70	33.75

8	8.17572	84.7285	58.1079	14.31
9	8.17572	245.271	58.1079	9.99
10	5.5	300	70	18.09
11	8.17572	245.271	81.8921	12.42
12	5.5	165	90	12.15
13	10	165	70	13.77
14	2.82428	245.271	58.1079	9.72
15	8.17572	84.7285	81.8921	12.42
16	5.5	165	70	33.75
17	5.5	30	70	9.72
18	2.82428	84.7285	81.8921	3.78
19	5.5	165	50	12.42

4.2.3. ANOVA for the Quadratic model; Analysis of variance (ANOVA) and response surface plot were generated using Design Expert software. The optimized value of the independent variables for optimum response was determined using numerical optimization

Table 4.3.Response result of Extract yield

Source	**Sum of Squares**	**D**	**Mean Square**	**F-value**	**p-value**	
Model	1954.70	9	217.19	58.41	< 0.0001	Significant
A-Solid mass	75.88	1	75.88	20.41	0.0015	
B-time	42.41	1	42.41	11.41	0.0082	
C-Temperature	0.4635	1	0.4635	0.1247	0.7322	
AB	43.38	1	43.38	11.67	0.0077	
AC	0.4465	1	0.4465	0.1201	0.7369	
BC	15.32	1	15.32	4.12	0.0730	
A^2	975.17	1	975.17	262.28	< 0.0001	
B^2	601.56	1	601.56	161.79	< 0.0001	
C^2	709.84	1	709.84	190.91	< 0.0001	
Residual	33.46	9	3.72			
Lack of Fit	10.13	5	2.03	0.3476	0.8615	Not significant
Pure Error	23.33	4	5.83			

Cor Total	1988.16	1				

The Model F-value of 58.41 indicates that the model is statistically significant. There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case A, B, AB, A^2, B^2, and C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (excluding those required to support the hierarchy), model reduction may improve your model. The Lack of Fit F-value of 0.35 implies the Lack of Fit is not significant relative to the pure error. There is an 86.15% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

4.2.4. Final Equation in Terms of Coded Factors

$$\begin{aligned} Extract\ yield\ (\%) \\ &= 32.67 + 2.36A + 1.76B + 0.1842C - 2.33AB - 0.2362AC + 1.38BC \\ &\quad - 8.45A^2 - 6.64B^2 - 7.21C^2 \end{aligned}$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1, and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

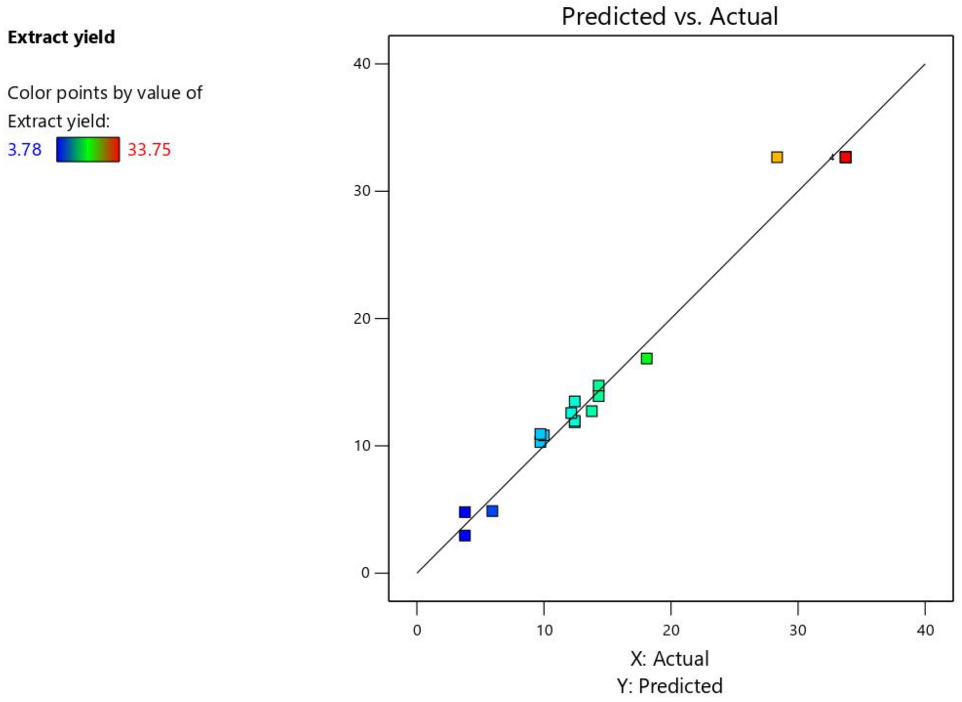

Fig 4.1 graph of actual vs predicted values of extract yield.

These plots are used to assess the performances of a predictive model .the closer the points are to the diagonal line, the better the models prediction . the x axis represent the actual values, and the y axis represents the predicted values. The distribution of point around the diagonal line indicate the models accuracy and potential biases. The color of the points represents the value of the extract yield, allowing for visual assessments of model performances across different yield ranges. The majority of the points are clustered relatively close to the diagonal line, indicating a reasonably good fit. This suggests that the model is generally accurate in predicting extract yield. There are a couple of point , particularly at higher yield values which are red or orange point that deviate slightly from the line .This suggest that the models predictive accuracy might be slightly lower at higher yield levels. The color gradient from blue to red represent increasing extract yield.

The clustering of blue points near the lower end of the line and the presences of red or orange points at the higher yield values suggesting potential for improvement in that range the closer the data point are to the diagonal line, the better the models predictive performance. Deviations from the line indicate areas where the model prediction are less accurate. Color coding provide additional insight into model preformances across different value ranges . This type of plot is a standard tool for evaluating the effectiveness of regression model.

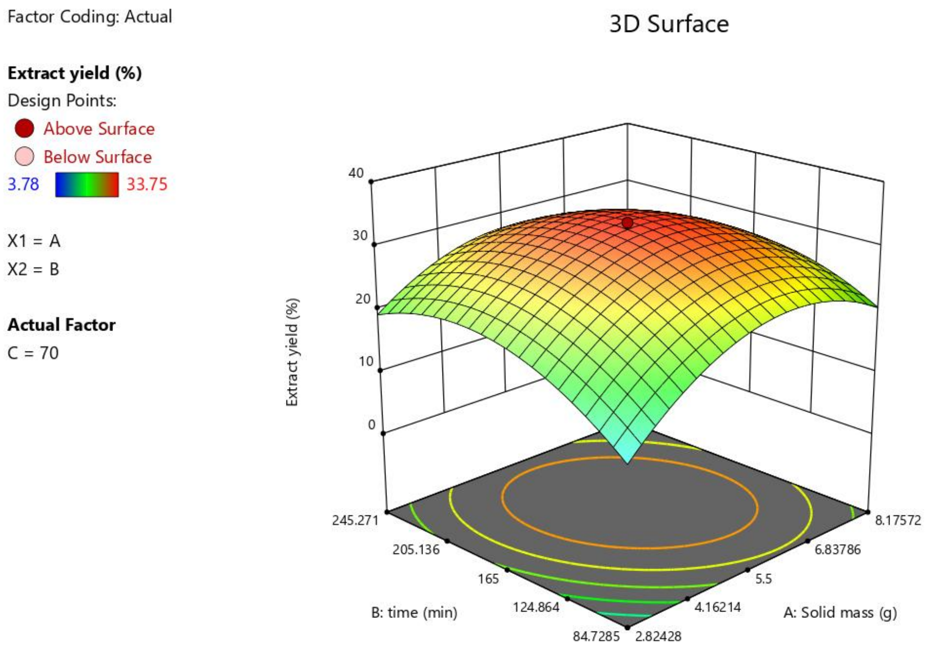

Fig 4.2. surface responses for temperature variances at (℃) .

This image displays a 3D responses surface plot generated using Responses surface methodology .The plot visualizes the relationship between two independent variable A:

solid mass (gms) and B: time (mins) and a dependent responses variable, Extract yield (%). The curved surface shows how the extract yield changes as the solid mass and time factors are varied simultenously. The peak of the surfaces (the red point) indicates the optimal combination of solid mass and time that maximize the extract yield ,which appears to be around 30- 33% .The contour lines are visible on the planes providing a two dimensional view of areas with constant extract yield values .

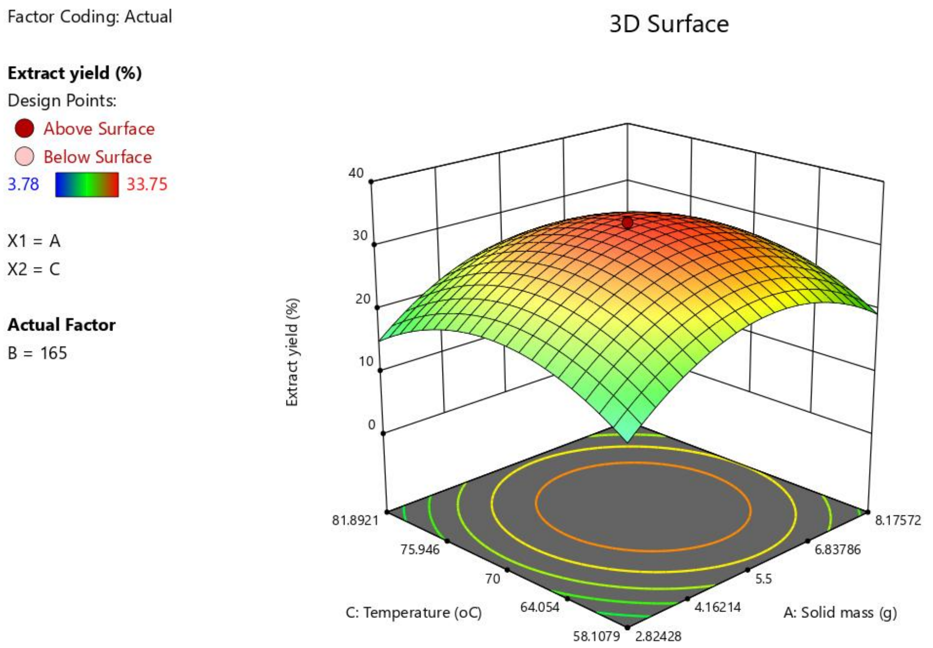

Fig4.3. . surface responses for time variances in (minutes)

This image displays a 3D responses surface plot generated using Responses surface methodology .The plot visualizes the relationship between two independent variable A: solid mass (gms) and B: temperature and a dependent responses variable, Extract yield

(%). The curved surface shows how the extract yield changes as the solid mass and temperature factors interact to affect the extract yield. The peak of the surfaces (the red point) indicates the optimal combination of solid mass and temperature that maximize the extract yield ,which appears to be around 30- 33% .The contour lines are visible on the planes providing a two dimensional view of areas with constant extract yield values .

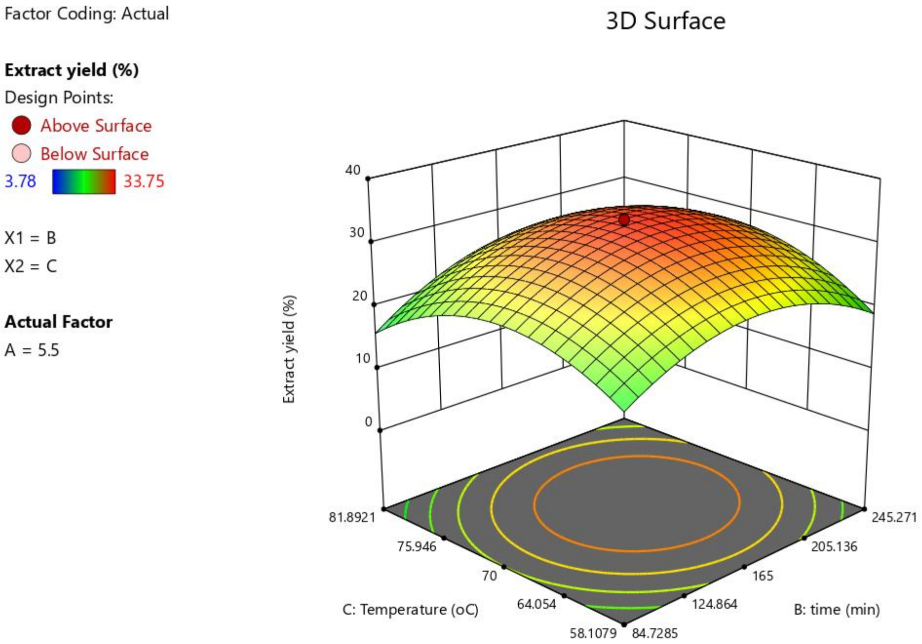

Fig 4.4. Surfaces responses for mass variances (g).

This image displays a 3D responses surface plot generated using Responses surface methodology .The plot visualizes the relationship between two independent variable A: time and B: temperature affect the Extract yield (%) responses. The curved surface indicate a non –linear relationship between the variables suggesting that there is an optimal point for maximum yield. The peak of the surfaces (the red point) represents the maximum predicted extract yield, which appears to be around 30- 33 .75% .The contour lines projected onto the bottom planes show combinations of time and temperature that result in the same yield.

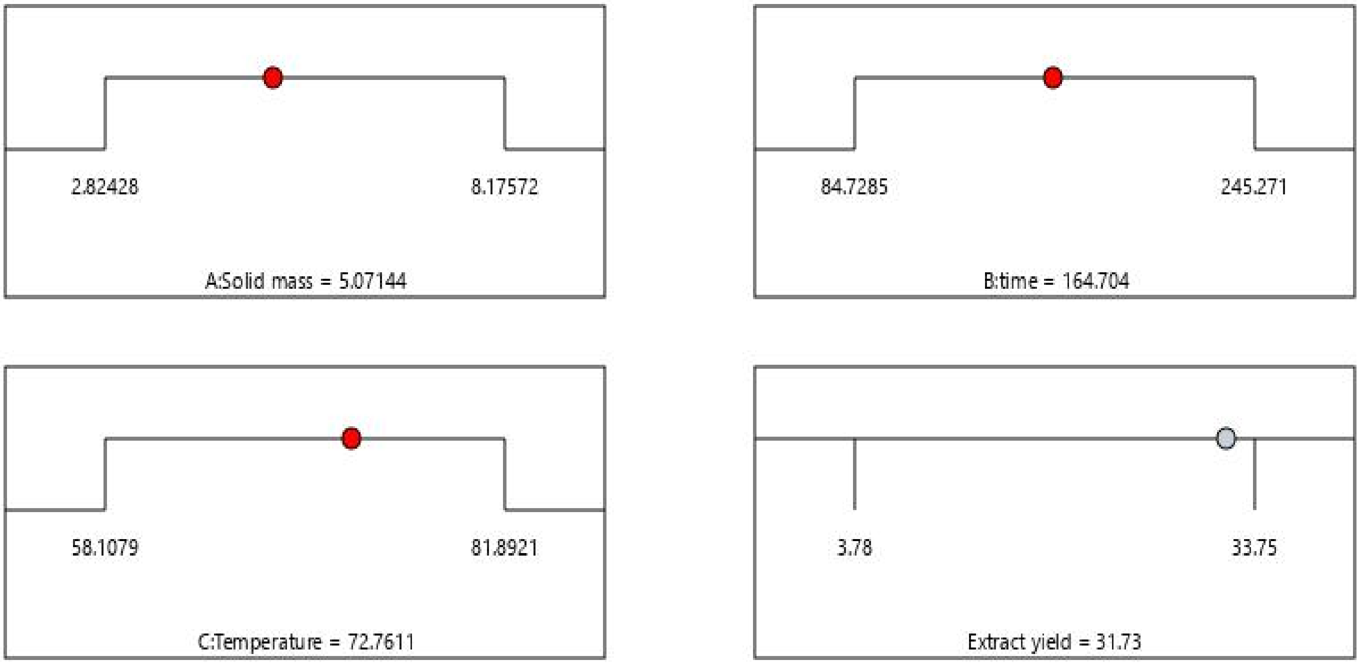

Fig 4.5. Block diagram of solid mass, time ,temperature and extract yield.

The image displays optimization result derived from responses surface methodology .The plots show the relationship between three independent variable solid mass ,time, temperature and dependent variable extract yield . The optimal conditions identified are a solid mass of approximately 5.07g, a time of 164.7 mins and a temperature of 72.76 degree census. These conditions result in a predicted extract yield of 31.73 ,the durability = 1.000 indicates that these parameter achieves a maximum possible desired outcome within the tested experimental spaces.

4.3. Total Saponin Content;

The extraction process was carried out according to the experimental design proposed by the response surface methodology. This optimization design produced runs of 19 with independent variables of extraction time (30-300 minutes), mass of bitterleaf (1-10 grams), and temperature (50-90 °C), and a constant volume of 50ml with a single response yield (%). Each run was subjected to a reflux condenser and their respective extract were dried in an oven at 90 °C and placed in a sealed plastic container until it was used for analysis.

$$ExtractYield(\%) = \frac{W_0}{W_s} \times 100 \text{ --equation 3.1}$$

Where

W_o =mass of extract

W_s= mass of solid used

Table 4.4.Extract Yield (%) of each solid was obtained at various runs are;

Mass of extract (gms)	Mass of solid used (gms)	Extract yield (wt%)
1.55	5.5	28.35
0.53	2.82428	14.31
1.55	5.5	33.75
0.09	1	3.78
0.53	2.82428	5.94
1.55	5.5	33.75
1.55	5.5	33.75
5.1	8.17572	14.31
5.1	8.17572	9.99
1.55	5.5	18.09
5.1	8.17572	12.42
1.55	5.5	12.15
8.2	10	13.77
0.53	2.82428	9.72
5.1	8.17572	12.42
1.55	5.5	13.77
1.55	5.5	9.72
0.53	2.82428	12.42

4.4 Emulsification test result;

The creation of an emulsion through the use of surfactants can lead to a reduction in interfacial tension (IFT). It enhances the compatibility with encapsulated oil, reduces the movement of water, and alters the pore structure and microscopic channels, which can enhance oil recovery, (Imuetinyan et al., 2022). The emulsion index is a measure of the stability of an emulsion, indicating the percentage of the dispersed phase (usually oil) that remains suspended in the continuous phase (usually water) after a specified period. A higher emulsion index signifies better stability. To determine the emulsification index, a standard procedure was followed, where equal volumes of oil and surfactant solution were vigorously mixed and allowed to stand for a specified period. After agitation of the alkaline surfactant in a test tube, emulsion was formed and it was observed to be stable over time.

Plate 4.1. emulsification result test on different sample on a measuring cylinder.

4.4.1. SAMPLE 1 AND 2; Measuring cylinder containing 2gms of water and 2gms of crude to form and emulsion mixture which forms and oil/water interfaces

4.4.2.SAMPLE 3; Measuring cylinder with contains 2gms of water, 2gms of crude oil and 2gms of alkaline surfactant which forms surface active agent and lower the interfacial tension between the oil and water which mobilized trapped oil in the cylinder sample .The alkaline surfactant solution react with the crude oil and water to form a Naphthenic solution (Sodium naphthenate) to form and "in-situ" surfactant which significantly reduces the interfacial tension between the oil and water phase the alkaline surfactant makes it easy for the water to displace and move the trapped oil in the sample and form the basis for alkaline flooding in EOR. The alkaline surfactant changes the wettability of the emulsion from oil wet to more water wet which further displacement can occur.

4.4.3. SAMPLE 4; Measuring cylinder with 2gms of water, 2gms of crude oil and 2gms of saponin or surfactant Which forms and emulsion by reducing the interfacial tension, the surfactant amphiphilic nature causes to move to the oil – water interfaces the polar head and the nonpolar tail at the oil- water interfaces the head migrate or align with the water and the tail to the oil which allow for a stable emulsion or a tiny oil droplet dispersed in water .

The volume and stability of the emulsion was observed and the emulsion index was calculated using the formula below.

$$EI\% = \frac{Ve}{Vt} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots . eqn2$$

Where:

EI% = Emulsification Index (percentage)

V_e = Volume of the emulsified layer (ml)

Vt= Total volume of the liquid mixture (ml)

1. **Top Layer: Oil Phase (Dark, Nearly Black Layer);** This is the layer of pure oil that stays on top because it is less dense than water. It is made up of extra, un-emulsified oil.

2. **Emulsified Phase (Emulsion Layer) - Middle Layer (Brownish Layer):** This is the real emulsion that was created when water, oil, and surfactant were combined. It is made up of scattered droplets stabilized by the surfactants, which is water-in-oil. Its volume serves as a gauge for emulsification efficiency.

3. **Water Phase, Bottom Layer (Clear or Yellowish Layer):** The remaining water that wasn't emulsified is this. Water settles at the bottom of the test tube because it is the densest component in the test tube.

The emulsion index for the alkaline surfactant was calculated and the result was 71% using the formula in equation 2.This moderate emulsification is as a result of the low concentration of 0.5 wt % of the alkaline surfactant in the measuring cylinder. Further observations of the emulsion formed in plate 4.4.3.1 showed that the surfactant maintained its emulsifying capacity. This suggests that the surfactant system has a prolonged effectiveness, making it a viable candidate for field applications in enhanced oil recovery. In this case, an emulsion index of 71% moderate emulsification. The results of this study indicate that the locally produced surfactant is capable of generating stable emulsions, which is a critical factor in its application for enhanced oil recovery. The emulsification index of 35% suggests that the surfactant provides sufficient surface activity to facilitate oil mobilization while maintaining a stable emulsion system.

4.5 FTIR (Fourier _ transform infrared spectroscopy):

This analysis was done to get information about the different functional groups in the dried weighted surfactant produced**.** Among various characterization techniques, Fourier transform infrared (FTIR) spectroscopy is a particularly valuable tool for determining the functional groups (Mohamed et al., 2017). The FTIR is a plot of Transmittance (%) against wavenumber (cm-1) and the signals are generated when the sample absorbs light energy and can be broad or sharp depending on the polarity of the bonds.

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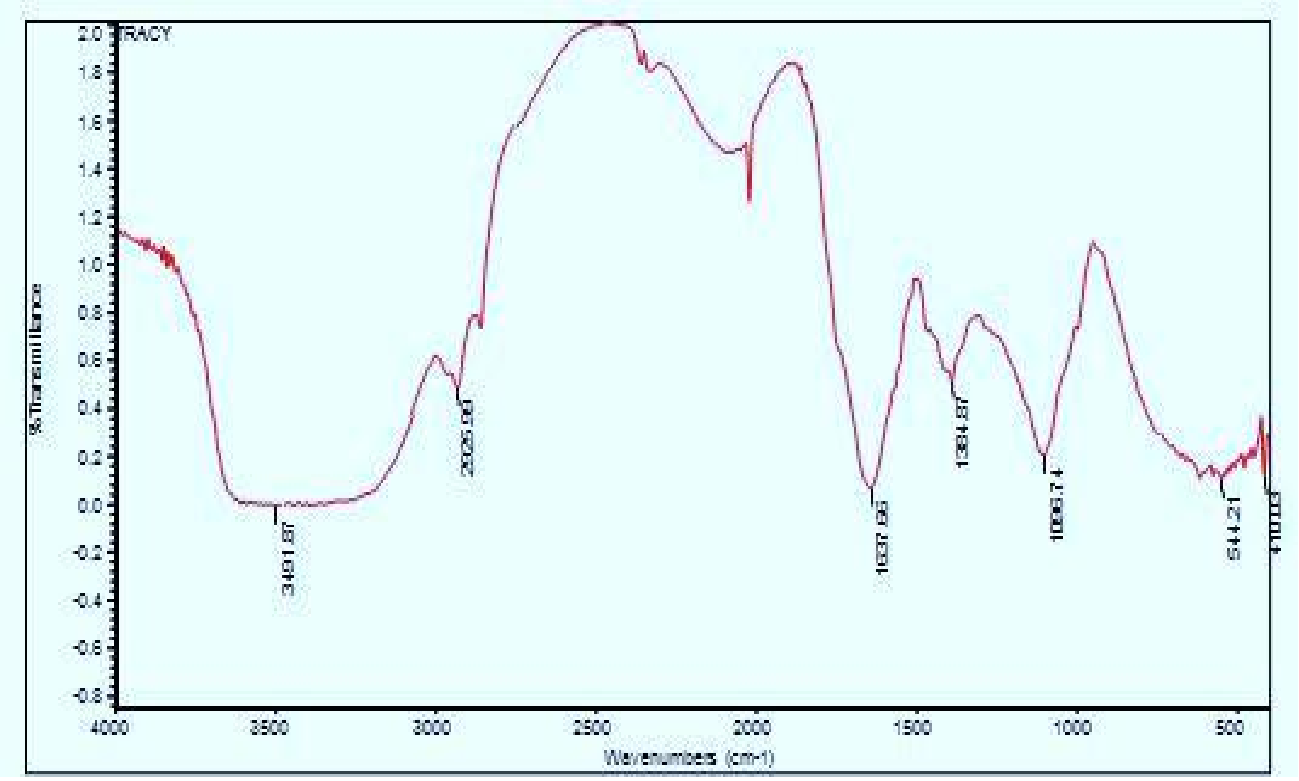

Fig 4.6. FTIR Spectra of the dried weighted surfactant produced

Table 4.5.Peak number, their corresponding wavenumber (cm-1), intensity and functional group for the dried weighted surfactant produced.

Peak Number	**wavenumber (cm-1)**	**Intensity**	**Functional group**
1	3479.87	-0.0070	N-H
2	2925.99	0.487	C-H
3	1637.66	0.0699	C=N
4	1384.87	0.515	S=O
5	1096.74	0.205	C-O
6	544.21	0.100	C-CL
7	410.03	0.112	M-O

Fig 4.6. gives the FTIR result of the dried weighted surfactant produced from bitterleaf with the alkaline gotten from corn cobs .it has been seen that the frequency of 3479.87 (cm-1) despite the presences of a primary amine in bitterleaf (Nitrogen and hydrogen) which function as the hydrophilic (water loving) head that makes the molecule water soluble. Peak at 2925.99(cm-1) indicating the C-H stretching ,suggesting the Alkane which plays the primary role by using the alkanes nonpolar (hydrophobic) hydrocarbon chain to react with the oil and the other end a polar (hydrophilic) and interact with the water for emulsification process. Strong bond of low peak at 1637.66(cm-1) which has the presences of Oximes or Imine which create stimuli responses which create properties like forming or breaking emulsion based on change in pH or oxidation process. The next peak with frequency of 1384.87(cm-1) with the presences of Sulfonyl Chloride builds the surfactant molecules they react with linear aliphatic hydrocarbon to create alkanesulfonate, peak with frequency 1096.74(cm-1) which contains Alphatic ether ,the ether group especially when part of the polyoxyethylene chain interact with the water molecules forms hydrogen bonds that contribute to the surfactant solubility. The penultimate peak with frequency of 544.21(cm-1) contains the halogen which are the salt formers act as anion to balance the positive charge of the cation allow for solubility of the surfactant in water. Lastly the peak number with frequency of 410.03 (cm-1) which is a metal oxygen plays the role of film deposition by floating on the surface which promote more uniform layer by layer development of metal and enable control over the film structure. The various role of the functional groups plays a crucial role in the dried weighted surfactant produced from natural bitter leaf.

4.6. GCMS (Gas Chromatography- Mass Spectroscopy) test of plant extract:

A chat which shows the x –axis which represent the time range and the y –axis as the intensity each peaks correspond with the various compound in the dried weighted sample produced from the bitter leaf using a GC column and for the MS it shows the mass to charge ratio with relative abundances of the bitter leaf plant extract.

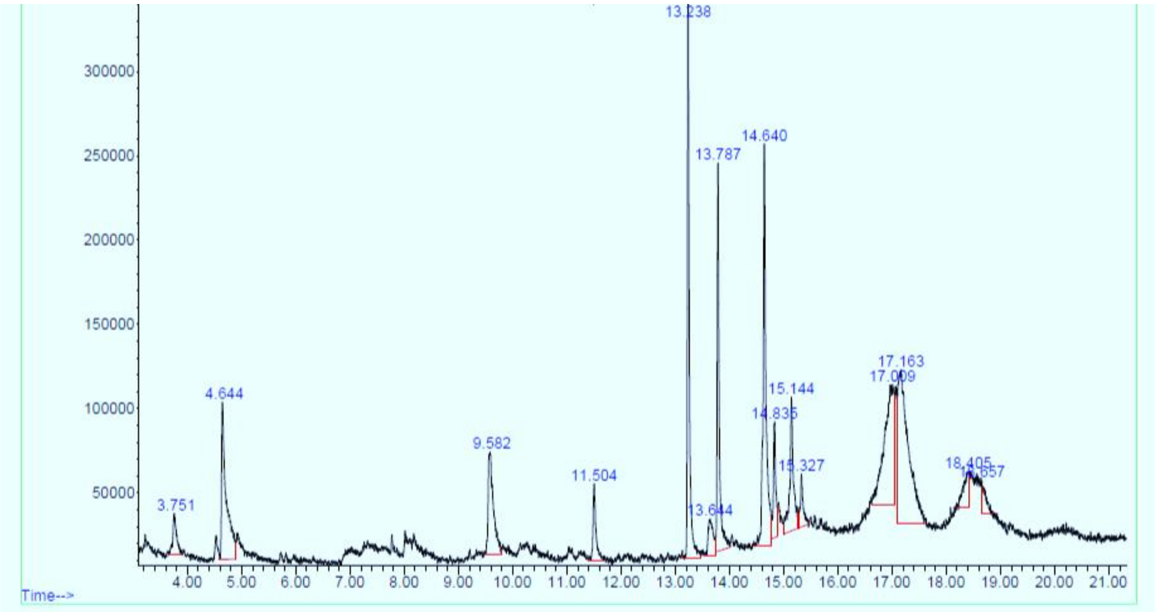

Fig 4.7. GCMS Chromatogram of the bitter leaf extracts plant extract;

Table 4.6. GCMS chromatogram of bitterleaf extract with peak number, compound name and concentration range (%).

Peak Number	Compound Name	Concentration (%).
1	N,N-dimethyl-Ethanethioamide	1.8
2	Methyl stearate	3.2
5	Hexadecanoic acid	20.88
8	8-Octadecanoic acid	13.05
10	9,17-Octadecadienal, (Z)	5.46
11	Ethyl 14-methyl-hexadecanoate	1.64
12	7-Isopropenyl-1,4a-dimethl-4,4a,5,6,7,8-hexahydro-3H-napthalen-2 –one	12.99
13	3,5,6,7,8,8a –Hexahydro-4,8a-dimethyl-6-(1-methylethenyl)-2(IH)Naphthalenone	18.55`
14	3-Methylindole-2-carboxylic acid,4,5,6,7-tetrahydro-,ethyl ester	2.1

Fig 4.7 shows the GCMS Chromatogram of the bitter leaf extract plant extract it shows the presences of some unique compounds as identified from the GCMS graph like N-N-dimethyl-Ethanethioamide with 1.8 % concentration which shows the presence of acetic acid and dimethylamide compound, the acetic acid act as a pH adjuster and emulsifier in the produced surfactant there by protonating it, From the graph Methyl stearate is present with 3.2% in the extract which is a fatty acid methyl ester it plays a crucial role in the produced surfactant act by helping to dissolve and stabilize mixture of oil and water. Hexadecanoic acid is known as palmatic acid with 20.88% concentration it plays the role of the surfactant preparation by lowering surface tension, 8-Octadecanoic acid Which is present in the plant extract also known as stearic acid with 13.05% concentration which plays a role in surfactant by binding with metal cation and always solubility in organic solvent, 9,17-Octadecadienal, (Z) which is a long –chain fatty aldehyde with 5.46 % concentration which helps the surfactant undergo reaction like hydroxyalkylation to form surfactant structure , Ethyl 14-methyl-hexadecanoate which is a fatty acid ester which contain 1.64 % concentration helps to stabilize the mixture of oil and water by reducing interfacial tension and thereby creating a stable emulsion. 7-Isopropenyl-1,4a-dimethl-4,4a,5,6,7,8-hexahydro-3H-napthalen-2 –one is also known as Eucarvone with 12.99 % concentration which is cyclic ketone with an isopropenyl group which role in surfactant is removing hydrophobic contaminants from water. The pernultimate compound found in the chart which is 3,5,6,7,8,8a –Hexahydro-4,8a-dimethyl-6-(1-methylethenyl)-2(IH)Naphthalenone which is known as known as alpha-isohumulone contains 18.55% concentration plays a role in surfactant which constitute to it high foaming and strong emulsification properties such as surface activity , hard water resilience's and good dispersion process, Lastly the presences of 3-Methylindole-2-carboxylic acid,4,5,6,7-tetrahydro-,ethyl ester also known as ethyl ester which contain 2.1% concentration helps increase wettability and impact in surface behavior of the surfactant with the ability to form multilayers at the interface between oil and water. From the GCMS result is should that the extract contains more of Hexadecanoic acid which is known as palmatic acid with 20.88% concentration it plays the role of the surfactant preparation by lowering surface tension which is the purpose of producing the natural surfactant from the bitter leaf extract.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

This study effectively evaluated the potential of Bitter leaf as a natural surfactant for enhanced oil recovery (EOR) using alkali made from corn cobs. In line with the growing need for ecologically suitable alternatives for synthetic surfactants, the study showed a sustainable and economical method of surfactant synthesis. The extraction process was carried out according to the experimental design with 19 runs with independent variables of extraction time (30-300 minutes) ,mass of bitterleaf (1-10grams) and temperature (50-90 degree census). The surfactant's surface-active qualities were subsequently confirmed by several types of tests, such as forth test, total saponin test and emulsion formation test. The result from the emulsification test revealed that the Alkaline surfactant with crude oil and water has and emulsion index of 71% had an emulsifying index and that of surfactant and crude with water yield 35% of the emulsion index from calculations.

From the chemical characterization for FTIR analysis with different peak number and various wave length from 3479.87 to 410.3(cm-1) with the presences of compound like the N-H, C-H, C=H, S=O,C-O,C-CL and the M-O functional group present in the produced dried weight surfactant ,GCMS analysis was also carried out to shows the GCMS Chromatogram of the bitter leaf extract plant extract to shows the presences of some unique compounds as identified from the GCMS result is shows that the extract contains more of Hexadecanoic acid which is known as palmatic acid with 20.88% concentration it plays the role of the surfactant preparation by lowering surface tension which is the purpose of producing the natural surfactant from the bitter leaf extract.

5.2 RECOMMENDATION

1. The need to optimize the extraction of bitter leaf as a natural surfactant to increase productivity and efficiency for EOR process.

2. Further research is should be made available to compare the effectiveness of bitterleaf surfactants to other natural, synthetic and biosurfactants.

3. Long-term stability and biodegradability of bittererleaf surfactants should be tested in different environmental circumstances to ensure their sustainability.

4.Proper understanding of the extraction properties such as temperature should be taken into consideration.

5. Method like maceration, can also be used to preserve the acive compound present in the bitter leaf extract.

6. Waste food product such as calcined banana peels, avocado seed which as the presence of basicity can also be used in place of corn cob.

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APPENDICES

The volume and stability of the emulsion was observed and the emulsion index was calculated using the formula below.

$$\boldsymbol{EI\% = Ve/Vt \times 100} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots . eqn2$$

Where:

EI% = Emulsification Index (percentage)

V_e = Volume of the emulsified layer (ml)

Vt= Total volume of the liquid mixture (ml)

1. ALKALINE SURFACTANT + CRUDE OIL+ WATER;

Volume of emulsified layer = 4.3ml

Total volume of the liquid = 6.0ml

EI%= $\frac{4.3}{6.0} \times 100\% = 71\%$

2. SURFACTANT + CRUDE OIL+ WATER;

Volume of emulsified layer=2.1ml

Total volume of the liquid = 6.0ml

EI%= $\frac{2.1}{6.0} \times 100\% = 35\%$