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Assessment of the Potential of Natural Surfactant in Carbonate Reservoir

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DECLARATION OF ORIGINALITY

We truthfully declare that this submitted work, entitled “Assessment of the Potential of Natural Surfactant in Carbonate Reservoir,” is a product of our original research work, to the best of our knowledge. The only exceptions are used published works and internet sources, presented as quotations and references that have been duly cited. Also, we declare that the report has not been previously published, in whole or in part, in any resources nor submitted for any other degree at Nazarbayev University.

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ABSTRACT

One of the main drawbacks of chemical enhanced oil recovery (CEOR) is to reduce the excessive use of chemicals. Recently, natural surfactants got recognition in CEOR since they are environmentally friendly, less toxic, and cheaper than traditional synthetic surfactants. However, the applicability of natural surfactants has yet to be tested under different conditions; thus, the research focuses on the natural saponin synthesized from the flaxseed oil available in Kazakhstan and its effect on efficiency of the available synthetic oil, octane.

The primary extraction method was conducted by the saponification of the flaxseed oil. The study used the IFT-700 apparatus, which uses the pendant drop method to determine the interfacial tension (IFT) between surfactant and oil. The salinity variation between 0 and 35,000 ppm of NaCl resulted in critical micelle concentration (CMC) values of about 6 wt% for the solution with distilled water (DIW) and 2.5 wt% for the brine surfactant solution. The effect of temperature on the rheological properties of the natural surfactant solution was measured in the range of 25–55 °C. Interfacial tension alteration with oil at the CMC values for DIW and brine surfactant solutions was observed, with 76.36 and 48.54% IFT alteration, respectively. The core flooding with surfactant solutions resulted in an oil recovery of 72% for DIW and 74.6% for the brine solution.

This study presented the potential of the extracted natural surfactant for the CEOR application as a low-cost and more accessible alternative to the traditionally used surfactants. Technically, the natural surfactants need more studies for their performance under salinity and temperature. However, the study is a direction to unmask the potential of Kazakhstan natural resources to fulfil its oil industry chemical needs.

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LIST OF SYMBOLS, ABBREVIATIONS, AND NOMENCLATURE

SYMBOL, ABBREVIATION, OR NOMENCLATURE	FULL MEANING
BP	British Petroleum
EOR	Enhanced Oil Recovery
IFT	Interfacial Tension
CEOR	Chemical Enhanced Oil Recovery
OGJ	Oil & Gas Journal
NCOC	North Caspian Operating Company
CO ₂	Carbon dioxide
FT-IR	Fourier-Transform Infrared Spectroscopy
NaCl	Sodium chloride
API	The American Petroleum Institute number
HPAM	Hydrolyzed Polyacrylamide
SSW	Synthetic seawater
SW	Sulphonated-Smart water
AS-01	Prepared anionic surfactant
ASP	Alkaline-surfactant-polymer flooding
ENORDET	Enhanced Oil Recovery Detergents
HAP	Hydroxyapatite
CMC	Critical micelle concentration
TDS	Total dissolved solids
C16DmCB	N-Hexadecyl-N,N-dimethyl-2-ammonio-1-ethanecarbonate
SDS	Sodium dodecyl sulfate
Duomeen TTM/ DTTM	N.N.N'-trimethyl-N'- tallow-1,3- diaminopropane
FB	Formation brine
Aerosol-OT	Sodium bis(2-ethyl-1-hexyl) sulfosuccinate
CTAB	Cetyltrimethylammonium bromide

TX 100	Triton X-100
BW	Laury dimethylaminoacetic acid betaine
CA	Cocamidopropyl betaine
CS-50	Cocamidopropyl Hydroxysultaine
APG	Alkyl polyglycosides
AAS	Anionic alcohol alkoxy sulfate
HS	High salinity
LS	Low salinity
CAPB	Cocamido propyl betaine
LB	Lauryl Betaine
IOS	Internal olefin sulfonate
TDA	Tridecyl alcohol propoxy sulfate
Na ₂ CO ₃	Sodium carbonate
NX-610	Dodecyl dimethyl amine betain
SiO ₂	Silicon dioxide
HISNP	Hydrophilic silica nanoparticles
NaOH	Sodium hydroxide
DIW	Distilled water
N/A	Not available
CaCl ₂	Calcium chloride

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Chapter One

Introduction to the study

1.1. Introduction

Despite the global tendency leaning towards green energy, oil, and gas remain a leading energy source worldwide. According to BP's statistical review of world energy in 2021, energy consumption remained upward until 2020. Now oil and gas still hold the top place in the world's energy system. As the world experiences primary energy demand, increasing oil production is essential. The major oil and gas companies have different approaches to solving this problem. Among the solutions are finding new discoveries, improving production technology, and better subsurface evaluation and management. Beyond the typical pressure on companies in terms of the profit margins, environmentalists demand rapid change toward a greener oil and gas sector (Temizel et al., 2022).

The life of oil and gas fields goes through discovery, evaluation, development, production, and abandonment stages. After discovering the potential oil reservoir, the reserves are estimated to determine the economic margins of the potential producing field. Then, the field investment decision sets the required number of wells to be drilled and the required designs. Managing the fields and wells requires utilizing the initial energy as a driving force. This is known as primary production. The primary recovery theory is based on the surface and reservoir pressure differences. Following the first stage, the secondary recovery aims to maintain the reservoir pressure with an external fluid, such as water or gas injected. The primary and secondary recovery methods can result in only 20–50% production of the original oil in place, whereas a considerable amount of oil remains in the reservoir (Taber, 1981). Since a substantial amount of oil is trapped, the tertiary recovery process, known as enhanced oil recovery (EOR), enables its production. It is important to highlight that many oil fields are either at the EOR stage or are already undergoing implementation.

EOR targets to increase the mobility of oil by changing physical or chemical properties; it can be achieved by different processes. Generally, the methods include the injection of gas, water, or chemicals in the reservoir. The mainstay of the processes relies on oil displacement and volumetric sweep efficiencies. Injected fluids improve the overall displacement, mobilizing and

producing more trapped hydrocarbons (Alamooti and Malekabadi, 2018). Up until 2020, the EOR processes included only thermal, miscible, chemical, and microbial methods. Thermal oil recovery is based on steam injection into heavy oil formations to reduce the viscosity of heavy crude oil. The miscible recovery method, in turn, is the injection of miscible gases into the reservoir. It improves oil displacement and reservoir pressure maintenance by reducing the oil viscosity. The microbial method affects the reservoir in various ways. For example, microorganisms can release gases and increase the pressure of the reservoir, reduce viscosity by breaking heavy molecules, and alter the interfacial properties between the reservoir rock and oil by producing natural surfactants. The chemical-enhanced oil recovery (CEOR) method leads to reduced interfacial tension between oil and water, alteration of the wettability of reservoir rock, and increased viscosity of the injected water (Alamooti and Malekabadi, 2018). Lately, the EOR has included hybrid applications and nanoparticle applications as a new approaches that improved or optimized the overall performance of tertiary recovery methods.

Chemical flooding uses chemicals such as polymers, surfactants, and alkalis. Chemical EOR increases oil recovery by increasing the effectiveness of water injected into the reservoir. In detail, the injection of polymers increases the viscosity of the aqueous phase. Consequently, oil mobility improves, increasing oil flow to the production well. Besides mobility control, polymer flooding improves sweep efficiency due to disproportionate permeability (Hazarika, Gogoi, and Kumar, 2022). Therefore, polymer flooding decreases the relative permeability of water so that low permeable zones can be reached by injectant water. Surfactant solutions mix with crude oil within the reservoir and solubilize interfacial films causing emulsification. As a result, IFT between water and oil reduces, lowering the capillary forces of trapped and residual oil in the reservoir. Also, due to the wettability alteration, the surfactant desorbs the oil-wet layer and changes it to a water-wet state, increasing oil production. Alkali is used to modify the surfaces by chemical reaction, or it can react with the oil and creates insitu soap. CEOR has several combinations that proved effective and useful. However, the final production or recovery depends on further complicated factors. Primarily alkali is used in a mixture with a surfactant, polymer, or both to improve the sweep efficiency in the reservoir (Wei et al., 2021).

1.2. Research Background

Applying chemical enhanced oil recovery (CEOR) requires a concentrated focus on chemical effectiveness and efficiency. Also, the risk factor associated with reservoir geological and operational conditions needs careful assessment before field application (Sheng, 2013). Bearing these conditions opened long-standing debates regarding their application on the field scale, including the surfactant applications. Surfactants are known for their ability to reduce the IFT to different degrees. A successful surfactant has to achieve an IFT of less than 2-10 mN/m. These values cannot always be achieved. This is because the surfactant's effectiveness depends on the reservoir temperature, salinity, and rock composition. On another upscale level, the initial water saturation can play a role in risk factors, especially in the numerical modeling outcomes (Wang et al., 2020).

The surfactant's foremost challenge is that it adsorbs in the reservoir. Throughout the process of micelle formation, surfactants interact, resulting in an undesirable but inevitable reduction in concentration. Therefore, their effectiveness in terms of IFT reduction is weakened, leading to decreased EOR efficiency and, consequently, not financially efficient outcomes of the process. The adsorption process occurs in both sandstone and carbonate reservoirs. The second challenge of the application is the surfactant's chemical structure and its stability, which level sometimes diminishes at high temperatures above 60°C (Atta et al., 2021). Considering the chemical side, the surfactant's polar head carries charges and an active functional group. This leads to the interactions between free ions in rock or water and salt and, consequently, to the loss of solubility characteristics.

Recently, synthetic surfactants derived from petroleum sources have been reported to have a negative environmental impact, including toxicity that harms the entire ecosystem. Therefore, many industrial applications are encouraged to reduce the harmful chemical impact on the environment. Thus, the extraction and implementation of the natural surfactant can be a possible solution. This is because, in comparison to conventional surfactants, they can be sustainable and cheaper, are less hazardous to the environment, and have excellent potential for IFT reduction.

Natural surfactants are obtained from the renewable sources, such as animals or plants, through the extraction process. Saponin is the primary surfactant source and is a compound that consists of naturally occurring glycoside groups. The complex molecular structure results in the detergent characteristics of the saponin; as a result of shaking it creates the soapy formation in the

water solutions. Therefore, stable foam is produced in the water (Norouzpour et al., 2022; Navaie et al., 2022). The natural surfactant's behavior and its efficiency in the oil recovery process can vary depending on the source of extraction. Therefore, their comparative analysis with conventional surfactants and natural ones has become one of the significant purposes of the studies in the EOR sphere.

1.3. Problem Statement

According to the Oil & Gas Journal (OGJ), Kazakhstan oil reserves amounted to 30 billion barrels of in 2018. It is the 12th largest in the world and the second largest in Eurasia after Russia, with proven oil reserves. Kazakhstan has three giant fields: Tengiz, Kashagan, and Karachaganak, operated by Chevron, Shell, Eni, North Caspian Operating Company (NCOC), and KazMunaiGas. Many of these large producers already applied EOR methods such as the miscible gas injection method, which was frequently implemented to enhance oil production. This method is favored because the raw materials for injectant gases, such as CO_2 , are abundant within the reservoir. Also, steam flooding and hot water injection methods were used in some fields. However, CEOR, particularly surfactant application, has excellent potential as an EOR method in Kazakhstan oil fields (Sagyndikov et al., 2018).

Moreover, besides oil and gas, Kazakhstan is the largest vegetable oil producer in Central Asia and produces flaxseed, sunflower, and rapeseed oils. East Kazakhstan has versatile raw materials that can be a good source of natural chemicals such as fatty acids in the flaxseed oil (Yergaliyeva, 2020). This variety in nature can be utilized for oil production by CEOR. Therefore, seeking new sources of needed sustainable, environmentally friendly, and technically efficient chemicals is crucial. Natural surfactants, overall, are environmentally friendly and less poisonous than synthetic surfactants. Different plants containing saponin can be used to extract natural surfactants. Saponin is a nonionic surfactant with a hydrocarbon ring skeleton attached to sugar. The saponin concentration depends on the plant and the method of its extraction.

Therefore, this study aims to answer the following general questions:

- 1) How can Kazakhstan's natural resources contribute to the synthesis of greener chemicals for the oil and gas industry?
- 2) Is natural surfactant applicable in carbonate reservoir?

1.4. Aims and Objectives

This project aims to investigate the possibility of developing a natural surfactant in Kazakhstan and assess its effectiveness. The objectives are:

1. To synthesize a natural surfactant in Nazarbayev University laboratories.
2. To evaluate the natural surfactant efficiency for Kazakhstan oil and reservoir conditions.

1.5. Study Scope

The focus of the study is a preliminary study. The study presents an experimental early assessment.

1. Produce a natural surfactant with raw materials from Kazakhstan.
2. Characterize the produced natural surfactants using FT-IR.
3. Assess the effective concentrations of natural surfactant.
4. Assess the IFT reduction experimentally.
5. Assess the effect of sea water salinity on the surfactant (35,000) ppm.
6. Evaluate the surfactant performance during core flooding.

1.6. Significance of the Study

Improving oil production is essential for meeting global demand. The use of new techniques shall be carefully investigated before the pilot test in the field. This research is motivated by the promising technical benefits of using natural surfactants in CEOR. The significance of the study

1. The primary concern regarding conventional surfactants is their high price. In contrast, naturally extracted surfactants are cheaper and more accessible due to renewability and the natural occurrence of plants as extraction sources. Therefore, the natural surfactants in the chemical EOR are more cost-effective to be applied on a larger scale.
2. Surfactants can impact the interface between oil and water, as well as the properties of the rock surface itself. As a result, surfactant reduces interfacial tension, consequently improving solubilization, increasing oil production, and enhancing oil recovery.

3. Using natural materials as the sources of natural surfactant extraction does not have a hazardous effect on the environment as compared with petroleum-based and other toxic chemicals applied in conventional EOR methods.
4. As the natural surfactants are synthesized from a vegetable oil, the possible waste of unused and spoilt products can be combated in the agriculture. Such a sustainable action will eliminate the concerns among farmers and their agriculture losses.

1.7. Chapter Organization

Chapter 1 defines the research background regarding EOR, specifically CEOR, and natural surfactants. It includes the set problem statement, aims, and objectives of the study, as well as its scope, which states the main focuses of the research work. The significance of the study was also described based on four aspects: economic, technical, environmental, and social.

Chapter 2 reviews previous studies on EOR, CEOR, and the application of extracted natural surfactants under various conditions. Based on the available literature, it explores the overall effect of concentration, temperature, salinity, pH, adsorption, and oil-type aspects on the result of the surfactant application. Further, the application of natural surfactants was explicitly considered from studies with different extraction sources.

Chapter 3 presents a detailed description of the tools and techniques used during the experiment. All three parts of the experiment were thoroughly considered, starting from the synthesis of the natural surfactant from the flaxseed oil via saponification reaction and followed by the characterization of the extracted surfactant. Furthermore, the effectiveness of the oil with natural surfactant was studied based on the oil recovery results from the core flooding.

Chapter 4 analyzes the results of the conducted experiments. The FTIR spectrum data for both synthesized surfactant and flaxseed oil is presented. The effect of the temperature on the rheological properties is also examined. Moreover, the variation of the salinity presents different IFT measurement, permeability and oil recovery results for distilled water and seawater solutions.

Chapter 5 draws the main conclusions as a result of the conducted study and provides several recommendations on the further development of the research project for more accurate and reliable results.

1.8. Summary

This chapter provides an overview of enhanced oil recovery, chemical EOR, and detailed information on natural surfactants. The oil field's stages and mechanisms for enhanced oil recovery were briefly described. The research background presented the theory behind surfactant effectiveness in the CEOR application. The rising interest in natural surfactants started from a global tendency to green energy and the lower cost of natural surfactants compared with synthetic ones. Kazakhstan's oil and gas fields are assumed to be suitable for natural surfactant flooding. The application of natural surfactants will result in a positive effect on cost-efficiency, effective performance, reduction of environmental harm, and sustainability within society.

Chapter Two

Literature Review

2.1. Overview

This chapter aims to generally cover the main aspects of enhanced oil recovery, chemical enhanced oil recovery, and the factors affecting their performance. The chapter analyzes the surfactant flooding, mechanisms, and field application. A particular focus is given to the recent advances in natural surfactant flooding. The critical review is devoted to reaching the ultimate understanding of the gaps in the current knowledge of the applicability of the natural surfactant in different reservoir conditions.

2.2. Enhanced Oil Recovery (EOR)

The Enhanced Oil Recovery is considered one of the most explored and highly demanded study fields within reservoir engineering in the twenty-first century. Numerous studies have been conducted on method classification, their applicability, and screening criteria throughout a few decades (Babadagli, 2020). Overall, EOR aims to produce oil that can not be produced by the primary and secondary recovery methods or that remains after their application. Recovery enhancement is achieved through the injection of gases or liquid chemicals as well as by applying thermal energy in some cases.

The use of EOR techniques enhances the reservoir's initial energy for oil displacement toward the producing well. In thermal recovery methods water at high temperatures or steam is injected, or oil combustion within reservoir rocks is conducted to reduce the viscosity of the oil. Furthermore, injected chemical fluids within the reservoir interact with the rock-petroleum system, causing changes in conditions such as lower interfacial tension and oil viscosity, oil swelling, modifying wettability, or phase behavior.

As shown in Fig. 2.1, the main oil displacement methods can be further classified into multiple categories.

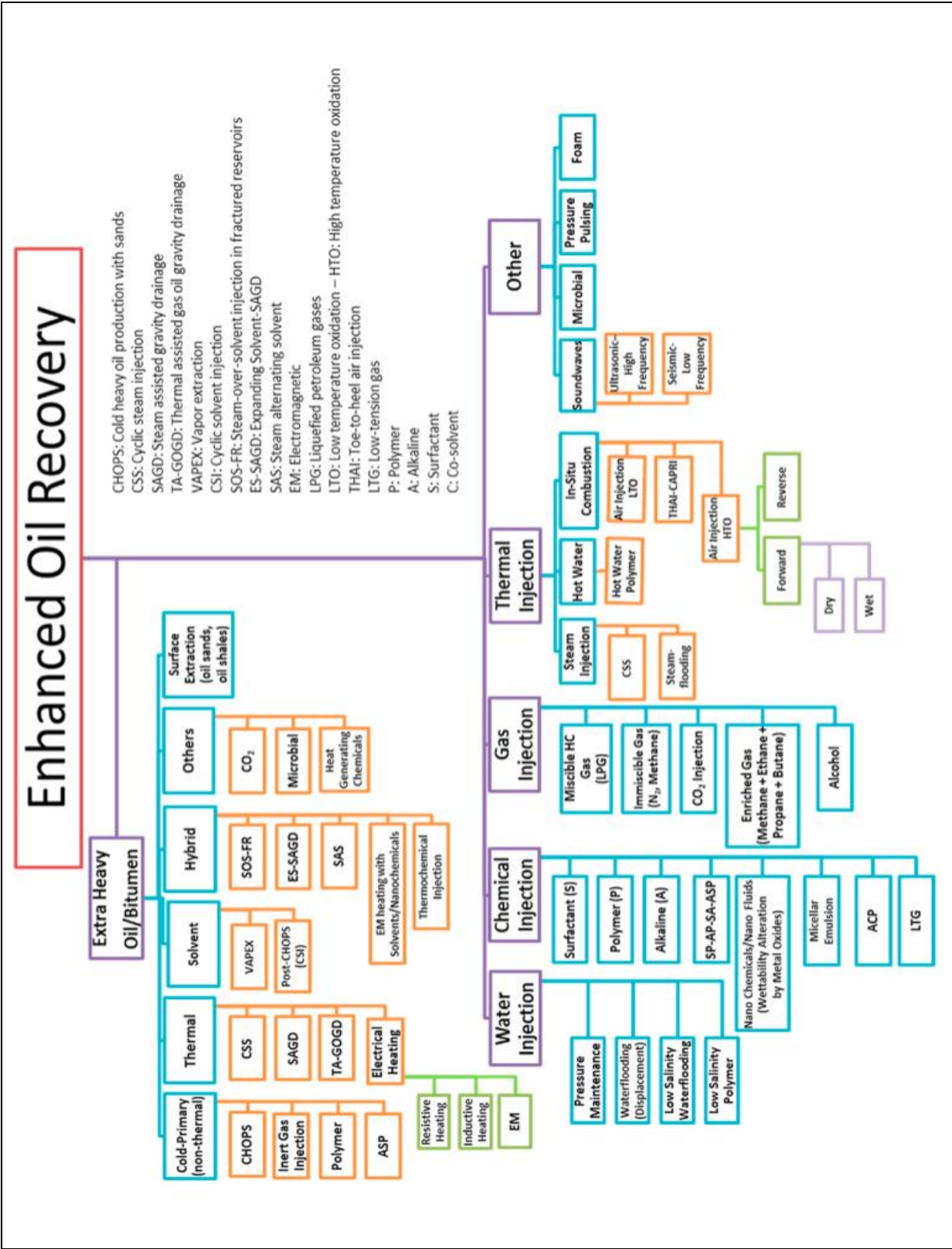


Fig. 2.1. Extended classification of EOR methods. Extra heavy-oil/bitumen can be considered 6–20 API range and viscosities greater than 1000 cP (Babadagli, 2020).

2.3. Chemical Enhanced Oil Recovery (CEOR)

Chemical flooding (CEOR) is a prominent EOR method that focuses on improving recovery efficiency by lowering the mobility ratio or increasing the capillary number. The mobility ratio is often altered by raising the water's viscosity, whereas the capillary number is altered by decreasing the interfacial tension (IFT). Overall, the technique involves injecting chemicals into the system.

Injected fluids usually include alkalies, polymers, or surfactants. Chemical flooding is considered one of the most effective methods for oil recovery from depleted reservoirs under low-pressure conditions. However, it is marginally cost-effective or sometimes not profitable at all (Austad and Milner, 2000).

Alkaline Flooding

Alkaline flooding injects a high pH system of alkaline chemicals, which react with some oil components for surfactant in situ generation. Increased oil displacement has primarily resulted from surfactant formation effects, such as a reduction in IFT. This method is usually applied for relatively low API gravity oil types. Moreover, it can be used in combination with the surfactant in cosurfactant-enhanced alkaline flooding by injecting both the surfactant and the alkaline chemical. Furthermore, recovery efficiency can be further improved by mobility control. Thus, polymers can be injected with alkaline fluid for primary slug displacement as a mobility buffer (Green and Willhite, 1998).

Polymer Flooding

Polymer flooding involves the injection of chemicals for the purpose of mobility control. Primarily, the volumetric sweep efficiency and, in some cases, the microscopic displacement efficiency can be improved as a result of the application of this method. This is done by an increase in the apparent viscosity of the fluid or a reduction in the effective rock permeability. A considerable increase in the water viscosity can be achieved by adding water-soluble polymers with a high molecular weight (Sheng, 2011).

Surfactant Flooding

The injection of surface-active agents, so-called surfactants, is a method of adding chemical substances that adsorb on the surface or the interface between two fluids. As a result, the interfacial properties are considerably altered, reducing the interfacial or surface tension (Green and Willhite, 1998). By decreasing the IFT between oil and the surfactant solution, the capillary trapping is reduced, enabling an easier flow of the oil droplets through the pore throats, forming an oil bank further. Furthermore, the injection of the surfactants leads to a decrease in the residual saturation and, hence, an increase in the relative permeability, resulting in higher sweeping efficiency of the oil (Sheng, 2015).

The search for the best-performing chemical candidates still continues. In the best-case scenario, an effective and successful chemical candidate must enhance the oil recovery. The range of success depends on their performance in actual conditions within the reservoir in real-world practice. Table 2.1 summarizes recent efforts and research in this area.

Table 2.1: Recent studies of chemical application for CEOR.

Type of flooding	Injected chemicals	Results	Additional Rf	Importance	Reference
Polymer	HPAM polymer and SSW and SW	Slight reduction in polymer shear viscosity compared to SSW with polymer	~6%	- Potential of obtaining even higher additional recovery by adding SSW.	(Tahir et al., 2020)
Polymer	HPAM and associative polymer	Pressure stabilization at higher injected volume.	6.52%	- Associative polymer is more stable than the HPAM polymer. - Associative polymer	(Abirov et al., 2019)

				flooding was recommended for pilot testing at South Turgay Basin.	
Surfactant	The ChampionX surfactant formulation : zwitterionic	IFT reduction by 38.0 ± 1.0 mN/m	16%	- Loss reduction by natural clay nanotubes - halloysites which are inexpensive, accessible, thermally stable and environmentally friendly.	(Ojo et al., 2020b)
Surfactant	The novel anionic surfactant AS-01	Ultra-low IFT- 1.0×10^{-2} mN/m was achieved	12.5%	- AS-01 surfactant application in reservoirs with low permeability, and even high temperature and salinity conditions.	(Zhang et al., 2017)
Alkaline-Surfactant-Polymer	NaOH, heavy alkylbenzene sulfonate, and HPAM-polymer	Dynamic IFT reduction	N/A	- The importance of composition of the crude oil and ASP flooding was confirmed.	(Sun, Kang, and Zhang, 2021)
Alkaline-Surfactant-Polymer	Na_2CO_3 , mixture of ENORDEE Internal Olefin	Reduction in the average oil saturation 81%-28%; water cut	Over 30%.	- Innovative and cost-effective, decreasing the chemical cost by ~25%. - Further engineering lead times and project	(Alkindi et al., 2018)

	Sulfonate and Alcohol Propoxy Sulphate, and Flopaam 3630S polymer.	dropped from 85% to 68%.		costs were reduced.	
Alkaline-Surfactant-Polymer	NaOH, heavy alkylbenzen esulfonate, and HPAM and HAP polymers	Very low and highly dispersed residual oil saturation and strong profile control.	10.35%	- New technology and effective way of applying EOR for reservoirs with ultra-high water cut after polymer flooding. - Both economic and technological development.	(Gao et al., 2020)

2.4. Surfactant

Surfactants are amphipathic particles that contain both hydrophilic and hydrophobic components. The surfactant's head tends to combine with water, but the tail tends to reject mixing with water and oil. Because of the compounding property of surfactants, they are frequently used in the industry as detergents, emulsifiers, foaming agents, and wetting agents.

Surfactants are utilized in the oil and gas industry because of their distinctive capability to decrease interfacial tension (IFT) at the oil-water interface. They are classified based on the method of their production or by their charge. The first classification consists of two types: synthetic (by chemical synthesis) and natural surfactants, which are derived biologically (Amanat et al., 2022). The second classification categorizes anionic, cationic, nonionic, and zwitterionic surfactants. The description is affected by the polar head charge. Anionic surfactants are negatively

charged, whereas cationic - positively charged and nonionic surfactants have no charge (Green and Willhite, 1998).

2.4.1. Factors affecting surfactant performance

Numerous parameters can impact surfactant performance and its application. Some of them are the surfactant concentration, temperature, salinity, pH, adsorption, and the oil type. In this section, a general description of the importance of each factor is analyzed. A summary of recent studies on different surfactants is provided in Table 2.2.

Table 2.2: Recent application of different surfactants and relevant factors studied.

Surfactant	Parameters	Key findings	Reference
N-Hexadecyl-N,N-dimethyl-2-ammonio-1-ethanecarboxylate (C16DmCB)	<ul style="list-style-type: none"> -CMC - 50 ppm, -Temperature, 30 °C -Salinity NaCl: 0, 15000, 30000, and 45000 ppm, -pH: 7.1–7.5 for sandstone -7.7–7.9 carbonate samples -Medium: sandstone and carbonate ● -API oil 33.28° 	<ul style="list-style-type: none"> - Reaching a plateau value higher than its CMC. - Increase in salinity has resulted in increasing the adsorption. 	(Kumar and Mandal, 2019)
Gemini cationic surfactant	<ul style="list-style-type: none"> -CMC - 0.1 wt%, ● -Temperature: 25 °C, 40 °C, 70 °C, and 100 °C 	<ul style="list-style-type: none"> - With increasing surfactant concentration, firstly the adsorption increased. - The adsorption of surfactant decreased with an increase in temperature. 	(Mao et al., 2019)

Sodium dodecyl sulfate (SDS) and Tritriplex III	<p>-CMC - 150 ppm,</p> <p>-Temperature - 30-70 °C,</p> <p>-Salinity - 4445 ppm,</p> <ul style="list-style-type: none"> -API oil 27° 	<p>- With an increase in temperature, the adsorption rate decreases because of the reduction in viscosity.</p> <p>- IFT reduction from 16.3 mN/m to 0.22 mN/m.</p>	(Saha, Uppaluri and Tiwari, 2017)
Duomeen TTM (C16-18N(CH3)C3N(CH3)2)	<p>-CMC - 0.04%,</p> <p>-Temperature: 25-120 °C,</p> <p>-Salinity: DI water, FB with divalent ions, and 5.07 M NaCl,</p> <ul style="list-style-type: none"> -pH values: 7.5, 7.0, 5.3, and 4.0 	<p>- High-salinity conditions decreases surfactant adsorption.</p> <p>- With reduced pH values, DTTM adsorption decreases.</p> <p>- Increase in the temperature leads to the adsorption reduction.</p>	(Zhang et al., 2019)
Soap-nut oil surfactant	<p>-CMC - 8000 mg/L,</p> <p>-Temperature - 27 °C.</p> <p>-pH varying between 2.3 and 11.5,</p> <ul style="list-style-type: none"> -API oil 18.9° 	<p>- With the surfactant concentration increase the adsorption increased as well until the CMC value.</p> <p>- Increase in salinity was observed to increase the adsorption.</p>	(Saxena, Kumar and Mandal, 2019)
Aerosol-OT, di-chain anionic surfactant	<p>-CMC - 0.21 wt%,</p> <p>-Temperature: 45 °C and 85 °C,</p> <p>-Brine 35000 ppm.</p>	<p>- Temperature led to intensification of the surfactant CMC, reducing adsorption.</p> <p>- Reduction in temperature has resulted in the increase in adsorption.</p> <p>- Increase in salinity increased the adsorption as well.</p>	(Abbas et al., 2020)

Cetyltrimethylammonium bromide (CTAB), Triton X-100 (TX 100)	<p>-CMC: 0.029 wt% for CTAB, 0.02 wt% for TX100,</p> <p>-Temperature: 30–80 °C,</p> <ul style="list-style-type: none"> -Salinity: 1-3 wt% 	<p>- The adsorption increases with the increasing surfactant concentration until the value of CMC.</p> <p>- Higher salinity resulted in an increase in adsorption.</p> <p>- Increase in the temperature has led to the more effective adsorption reduction.</p>	(Yekeen et al., 2019)
BW, CA, and CS-50 surfactants	<p>-Temperature: 20, 80, and 105 °C,</p> <p>-Salinity: DI and brine (TDS = 289 820 mg/L),</p> <p>-pH = 6.2 ± 0.3,</p> <ul style="list-style-type: none"> -API oil 20° 	<p>- The adsorption increases with increasing surfactant concentration until the specific value.</p> <p>- Higher temperature conditions have led to lower adsorption.</p>	(Zhong et al., 2019)
Sodium dodecyl sulfate (SDS)	<p>-CMC - 0.2 wt%,</p> <p>-Temperature: 26, 50, and 80°C,</p> <p>-Salinity: 0-5 wt%</p>	<p>- Increase in surfactant adsorption with an increase in surfactant concentration.</p> <p>- The adsorption has increased with decreasing temperature and increasing salinity.</p>	(Yekeen et al., 2017)
Alkyl polyglycosides (APG)	<p>-CMC - 0.025 wt%,</p> <p>-Temperature: 20-115°C,</p> <ul style="list-style-type: none"> -Salinity: 10-160 mg/L. 	<p>- Adsorption increases with increasing surfactant concentration because of the surfactant clusters.</p> <p>- Increasing temperature, slightly decreases adsorption.</p> <p>- With increase in salinity the adsorption has dramatically increased.</p>	(Wei et al., 2020)
Anionic surfactant	<p>-CMC - 0.15 wt %,</p> <p>-Temperature 23 ± 0.1</p>	<p>- The AAS adsorption has increased with increasing pH.</p>	(Liu et al., 2019)

(AASJ771)	<p>°C, -pH=9, ● -Salinity varies between HS and LS seawater</p>	- Increasing the temperature from 23 to 65 °C has led to the slight increase in the adsorption.	
Cocamido propyl betaine (CAPB)	<p>-CMC - 0.025 wt%, -Temperature 80 °C, -Salinity: 0.1-19.8 wt%, -pH of 6.6, ● -API oil 21°</p>	<p>- Increase in pH, reduced adsorption. - Applying CAPB surfactant with Na₂CO₃ resulted in additional oil recovery of about 18.2%.</p>	(Rezaei et al., 2020)
Lauryl Betaine (LB), ENORDET O332, i.e., IOS15-18 (IOS), and Marlowet 4538	<p>-CMC: 0.50, 0.50, 0.12 wt%, -Temperature: 60-110°C, -Salinity 80 g/L NaCl, -pH > 11, ● -API oil 21°</p>	- Surfactant formulation was developed for high-salinity and temperature ~100°C conditions.	(Puerto et al., 2018)
Tridecyl alcohol propoxy sulfate (TDA; C13-13PO-SO ₄) and internal olefin sulfonate (IOS; C20-24IOS)	<p>-Temperature: 24-78°C, -Salinity 0.1 wt% NaCl, -pH values 6.5, 8.5, and 10.5</p>	- The adsorption almost linearly decreased with increasing pH above the value of 9.	(Tagavifar et al., 2018)
Sodium	-CMC about 288 ppm,	- Increasing temperature below CMC	(Mahmou

dodecyl sulfate (SDS)	-Temperature: 26-65°C, -Salinity 3.5 wt%, • -API oil 12.5°	value leads to higher oil recovery, whereas the effect is only slight at the values higher than optimum concentration.	di, Jafari and Javadian, 2019)
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2.4.1.1. Concentration

The surfactant's critical micelle concentration (CMC) varies according to the surfactant injected and, hence, its source of extraction. The definition of CMC is the concentration at which the micelle structures are formed, and any extra surfactant amphiphilic molecules go to the micelles. Accordingly, it describes the effective concentration for surfactant flooding (Ramesh and Sakthishobana, 2021). At a concentration of the surfactant below the CMC value, a significant adsorption increase is observed due to surface aggregation. As opposed to it, the values above CMC, increasing concentration does not affect the adsorption behavior of the surfactant (Belhaj et al., 2019).

2.4.1.2. Temperature

Although some surfactants can be applied in reservoir conditions of high temperature, the majority of studies use a specific range of temperature values. According to the review article by Sheng (2015), most researchers propose a limited reservoir temperature of about 93.3° C, with a median value of 25.3 °C. Although some of the studies present temperatures up to 100–150 °C, they were obtained only in laboratory conditions without proper application in the actual reservoir conditions of the field projects. The surfactant should be stable at both low and high temperatures.

2.4.1.3. Salinity

Usually, the reservoirs undergo the water flooding process during some period of time before the application of CEOR, and the injected water salinity may be close to the value of the salinity of the reservoir water itself (Ramesh and Sakthishobana, 2021). The salinity of the injected water should be approximately equal to the optimum salinity; this is the value at which the lowest IFT is observed. The optimum value depends on the type of oil and surfactant applied. In the review study of Sheng (2015), the values of 50,000 ppm of TDS and 100 ppm of divalent were

suggested based on the considered literature sources. The importance of salinity can be explained in terms of anionic surfactants; using water with a high salinity value can result in the interaction of surfactants with salt ions, which can lead to unfavorable precipitation. As opposed, the salinity increase can also lead to the reduction of the repulsive forces between the surface of the rock and surfactant molecules (Belhaj et al., 2019; Kumar and Mandal, 2019).

2.4.1.4. pH

As with varying pH, the charge of the surfaces of the solid changes as well, and the adsorption of the surfactant depends on this parameter. With an increase in the pH value of the solution of surfactant, the number of hydroxyl groups is reduced, affecting hydrogen bonding. As a result, at lower pH values, surfactant adsorption is increased. Moreover, the solution pH also affects the surface rock charge; by increasing the pH, the surfactant stability is enhanced, leading to higher oil recovery (Belhaj et al., 2019).

2.4.1.5. Adsorption

Surfactant adsorption onto the reservoir rocks can be achieved by different methods, such as association or exchange of ions, adsorption by dispersion forces or π electrons polarization, or hydrophobic bonding. It occurs because of the van der Waals and electrostatic interactions between the surface of the solid and the surfactant itself. This parameter is one of the most vital aspects and can negatively affect the efficiency of the application of CEOR. As surfactant adsorption can not be completely neglected, it can be decreased to a certain minimum value. As a result of this reduction to this certain limit and hence, the economic optimization of the method, the oil displacement efficiency can be successfully improved (Sheng, 2011).

2.4.1.6. Oil type

Although the composition of the oil plays an important role in surfactant flooding, affecting the salinity range with low IFT values, the research studies have not suggested any screening criteria in terms of the oil composition. The API gravity of the oil is less important than its viscosity in consideration of these parameters as potential screening criteria (Sheng, 2015). Thus, without

any justified in-depth analysis of the studies, it can not be stated that the type of oil is an essential criterion for surfactant application.

2.5. Application of natural surfactants

Various surfactants are used in the chemical flooding technique in oil reservoirs to reduce water-oil interfacial tension (IFT). Natural surfactants, like plant extracts, have recently received a lot of interest in the enhanced oil recovery (EOR) process. Certain parameters of these surfactants, such as their efficiency in obtaining the required IFT values, tolerance to different salinities, and temperature stability under reservoir conditions, must be suitable. Currently, engineers diligently assess the natural surfactants extracted from renewable sources, as they not only resolve the issue of high cost but also minimize the ecological and environmental impact, reducing the toxicity and harm to the entire ecosystem (Amanat et al., 2022). Table 2.3 covers the recent research done on natural surfactants.

Alfalfa

The natural surfactant was extracted from a perennial flowering plant, Alfalfa (*Medicago sativa*), whose implementation has resulted in a 63.39% IFT optimization with an IFT reduction by 29.29 mN/m at the CMC value of 4 wt%. In addition, the rock wettability has changed from the initial oil-wet state to a more water-wet one with a reduced contact angle of 86.84°. In order to study the synergistic influence of the Alfalfa natural surfactant with synthetic seawater, solutions of ions of calcium, sulfate, and magnesium were prepared. According to the results, with calcium ions used, IFT was reduced from 63.39% to 71.0% for the surfactant and surfactant—hybrid ion solutions, respectively. The hybrid solution with an extracted natural surfactant and a particular concentration of calcium ions has resulted in an increase in oil recovery by 19.2%.

Although this study has provided a comparative analysis with different ions to study the compatibility with the Alfalfa surfactant, the effect of temperature was not evaluated to investigate whether extracted surfactant is sensitive to the changes in temperature values. Furthermore, additional specific data about the extraction procedure is required for future research on the topic (Eslahati et al., 2020).

AlkaSurf X, SDS, Palm kernel oil, Moringa oleifera

Three natural surfactants: AlkaSurf X, Palm kernel oil, and Moringa leaf from the hibiscus plant, *Elaeis guineensis*, and *Moringa oleifera*, respectively, were considered in the study “An experimental approach to low cost, high-performance surfactant flooding.” According to the research, compatibility tests with brine were conducted, as well as an analysis of the pH of the surfactant concentration. It was found that all of the surfactants were compatible with the brine, and the most effective performance of the natural surfactants was observed at a CMC value of 0.4 wt%. AlkaSurf X surfactant has resulted in the highest additional recovery by 22.7%, whereas the oil recovery factor has increased by 18.8% for *Moringa oleifera*.

However, the behavior of the natural surfactants was not studied under the reservoir conditions of temperature and pressure to ensure the high potential in the enhanced oil recovery application (Obuebite et al., 2020). Also, the type of chemical within those surfactants was not mentioned, as well as extraction method details.

Fenugreek, Sugar beet leaves, and Chickpeas

In the study “Comparative study of natural chemical for enhanced oil recovery: Focus on extraction and adsorption at quartz sand surface,” three different raw materials were used for the extraction using the soxhlet: fenugreek, sugar beet leaves, and chickpeas. The CMC value was in the range of 4–5.5 wt% in all of the considered samples, with the highest adsorption found in sugar beet leaves at 192 g/kg, compared to 25% and 37% higher values for fenugreek and chickpeas, respectively. Moreover, the exothermic nature of the process was proven with the repetition of the test under varying temperatures, which resulted in reduced adsorption at higher temperature values. However, the estimated and obtained CMC value was not validated following Beer Lambert’s law, as at a concentration value higher than 5 wt%, the shape of the line has resulted in a noticeable curvature. The investigation was confined to adsorption and did not cover experimental recovery processes that can show the effectiveness of surfactants. Therefore, the increase in oil recovery as well as the wettability change results, have not been presented as a result of this study (Abbas, Abd Alsaheb and Abdullah, 2022).

Avena Sativa plant

The Avena Sativa plant, which is also more commonly known as oat, was used for saponin extraction. Moreover, different salts were tested to check for compatibility with the extracted natural surfactant; Na₂CO₃ salt was found to be the most effective in terms of compatibility with the surfactant, with an IFT reduction of 1.38 mN/m. According to the results of the core flooding, the injected 5 PVs of the extracted surfactant have increased the recovery factor of the oil by 28.89%. Also, DSW2000 was used as the reference case for smart water, the conjunction of which with the natural surfactant was also tested to enhance oil recovery. Based on the results of the measurement of the contact angle, the addition of smart water has changed wettability more effectively than it would have without it (55.12 and 99.13, respectively). However, the total oil recovery with DSW2000 was obtained to be 23.14%, which is lower than the value with Na₂CO₃ salt alone (Sami et al., 2022).

Chuback

In the research "Xanthan gum-added natural surfactant solution of Chuback: A green and clean technique for enhanced oil recovery," Chuback, a new natural surfactant, was used with bio-based polymer xanthan gum to result in the effective wettability alteration from 16.71 to 60.52 in the carbonate samples and from 8.87 to 50.1 in sandstone. As a result, an effective IFT reduction by 74% was observed, as well as an increase in the oil recovery in the carbonate core by 28.6% (Navaie, Esmailnezhad and Jin Choi, 2022). However, aspects such as features, values of pressure, and temperature were not included in the analysis, which could be helpful in determining the natural surfactant's potential.

Quinoa plant

According to the study conducted by Norouzpour et al., (2022), the natural surfactant was extracted from a quinoa plant for enhanced oil recovery operations and has demonstrated thermal stability at reservoir temperature conditions based on the test results. With the considerable contact angle reduction from 146° to 26.3°, whereas IFT has decreased from 33.2 to 8.7 mN/m, the natural surfactant introduction has increased the oil recovery factor by 24.1%. Moreover, the results were compared with some commercial surfactants, and it was found that the quinoa surfactant produced emulsions with higher stability and density, according to the emulsion tests. It is worth stating that

TR-880 and NX-610, which were used as commercial surfactants for comparative analysis in this study, have shown better performance in terms of effective IFT reduction as opposed to the natural quinoa surfactant.

Glycyrrhiza glabra

In the research "Adsorption of Glycyrrhiza glabra natural nonionic surfactant onto the carbonate reservoir rock in the presence of SiO₂ nanoparticles surface: Towards enhanced oil recovery" saponin was extracted from the glabra plant called Glycyrrhiza for the purpose of reducing the interfacial tension and consequently enhancement of the oil recovery. The study was conducted in the presence of hydrophilic silica nanoparticles, also called HISNPs, and although the electrical conductivity methods were conducted, as well as the adsorption data collection, the adsorption process was characterized only as a result of the study. Adsorption starts with a short period of rapid process and is followed by a long period of slower adsorption (Dagbandan, Shahrabadi and Arabiyoun, 2022). The extraction method and properties also weren't addressed. Furthermore, the paper did not explore core flooding under reservoir settings, which can demonstrate the surfactant potential as well as its effectiveness in field conditions.

Aspilia africana, Dialium guineense Willd, Vernonia amygdalina, and Jatropha curcas

Gbonhinbor et al. analyzed the behavior of four different natural surfactants extracted from the following plants: *Aspilia africana*, *Dialium guineense Willd*, *Vernonia amygdalina*, and *Jatropha curcas*. CMC values were obtained to be 0.6%, 0.45%, 0.50%, and 0.50%, respectively. Hydrophobicity was mainly studied in work as well as the molecular weight identification of the natural surfactant based on the freezing point dipping method with sodium dodecyl sulfate as a reference surfactant. As a result, hydrophobicity values were found to be in the range of 0.116 to 0.194, which has proven the ability of the IFT reduction.

Although four different plant surfactants were studied, the main focus of the study was on their hydrophobic properties of them, whereas the IFT reduction and increase in oil recovery were mentioned only as the theoretical consequences and assumptions based on the study results. Therefore, there is no accurate data regarding the IFT reduction, wettability change, or recovery factor increase, according to the experiments in the research. Adsorption and core flooding tests should be conducted in further studies for the development of the suggested hypotheses. In

addition, the paper did not describe the extraction of surfactants and under which conditions experiments were conducted in detail. (2022).

Elaeis Guineensis from Palm Kernel oil

A natural surfactant extracted from the palm kernel oil was analyzed for application in the enhanced oil recovery methods. Foam stability tests and static adsorption tests were performed to study the characteristics of the natural surfactant. The most stable emulsions were obtained to be formed at the CMC value of approximately 5 g/L, with an increase in the emulsion stability with a rise in the surfactant concentration value. The study was limited to the conduction of these tests. As a result, the synthesized surfactant was concluded to have potential in the application in the enhanced oil recovery techniques without any specific data. Therefore, no values in terms of the wettability alteration, IFT reduction, and recovery factor changes were presented throughout the process of the work on this study to consider the effectiveness of the extracted surfactant in terms of the oil recovery enhancement (Ogunkunle et al., 2022). However, since the surfactant application needs flooding and dynamic behavior consideration, it still needs to comprehensively understand the particular solution's performance and effectiveness.

Table 2.3: Summary of natural surfactant studies.

No	Plant	Oil API/ density	Pressure	Temperature	Salinity	CMC	IFT reduction	Wettability alteration	Add. Rf	Reference
1	Alfalfa (Medicago sativa)	0.7998-0.8801 g/cm ³	14.7- 6043 psia	N/A	N/A	4 wt%	63.39%	49.91%	19.2%	(Eslahati et al., 2020)
2	Hibiscus, Moringa oleifera	23.3° API, 0.92g/cm ³ density	N/A	N/A	0.5 wt.% and 3.0 wt.%	0.4wt %	N/A	N/A	Increase by 22.7% for Hibiscus surfactant and 18.8% for Moringa oleifera	(Obuebite et al., 2020)
3	Fenugreek	N/A	N/A	35°C- 55°C	N/A	5.3 wt%	10 ³ -10 ⁴ mN/m reduction	N/A	N/A	(Abbas, Abd Alsaheb and Abdullah, 2022)
4	Avena Sativa	31.56° API	4500 psi	80°C	2000 ppm	At 6.45 mN/m	9.09 mN/m	The contact angle altered from 148.97° to 55.13° after 120 hrs, and 55.12° after 135 hrs	28.89%	(Sami et al., 2022)
5	Chuback.	32.292° API,	N/A	N/A	42000	1.25	74%	From 16.71° to	For surfactant-	(Navaie,

	Acanthophyllum	0.832 g/cm ³			ppm NaCl; 42000 ppm NaCl + 18000 ppm CaCl ₂	wt%		60.52°(carbonate) and from 8.87° to 50.1°(sandstone)	16%and for surfactant-polymer- 19%	Esmailnezhad and Jin Choi, 2022)
6	Quinoa	32.6° API, 0.85 g/cm ³	414 bar	100°C	8000 - 12000 ppm	1500 ppm	24.5 mN/m	Contact angle reduction from 146° to 26.3°	24.1%	(Norouzpour et al., 2022)
7	Glycyrrhiza glabra	27.06° API, 0.8924 g/cm ³	N/A	25°C	100-7000 mg/L (ppm)	0.35 wt%	49.06% to 65.64%	N/A	N/A	(Daghbandan, Shahrabadi and Arabiyoun, 2022)
8	Aspilia africana, Dialium guineense Willd, Vernonia amygdalina, and Jatropha curcas	0.87 g/cm ³	N/A	2.7°C to -3.1°C	0.1 g/100ml brine solution	0.6%, 0.45%, 0.5%, and 0.5% respectively	N/A	N/A	N/A	(Gbonhinbor et al., 2022)
9	Elaeis guineensis	45.40° API, 0.80 g/cm ³	N/A	28 °C	Varied 0-5 wt%	5 g/L	N/A	N/A	N/A	(Ogunkunle et al., 2022)

2.6. Summary

This chapter provided an overview of the role of chemical-enhanced oil recovery in the production enhancement. The main focus was given to surfactant flooding and the project worldwide. An extensive study on natural chemicals in chemical-enhanced oil recovery was presented. The outcome of the finding is that several researchers were interested in environmentally friendly chemicals. The researcher was motivated by the low cost of these chemicals in comparison with synthetic surfactants.

The key findings regarding the natural surfactants:

- The current practical trend focuses on finding cheap, environmentally friendly, and technically feasible alternatives for petroleum-based chemicals.
- Certain parameters of the natural surfactants, such as their efficiency in obtaining the required IFT values, tolerance to different salinities, and temperature stability under reservoir conditions, must be suitable.
- Natural surfactants have been used with oil with API varying from 23° to 46°. For now, the natural surfactant seems to be effective for medium and light oil types.
- The source of the natural surfactant has an effect on the concentration. The surfactant's critical micelle concentration describes the effective concentration for surfactant flooding.

Chapter Three

Methodology

3.1. Chapter Overview

This chapter includes a detailed explanation of the materials and techniques required to achieve the objectives of this project. It discusses the experimental study, materials, and methods that were applied to investigate a new natural surfactant. The experimental study was divided into three parts. In the first part, natural surfactant was synthesized using organic oil and a chemical esterification process. In the second part, characterization and efficiency were measured. In the third part, oil recovery was experimented with by core flooding. All materials, apparatus, and methods involved are discussed in detail in this chapter.

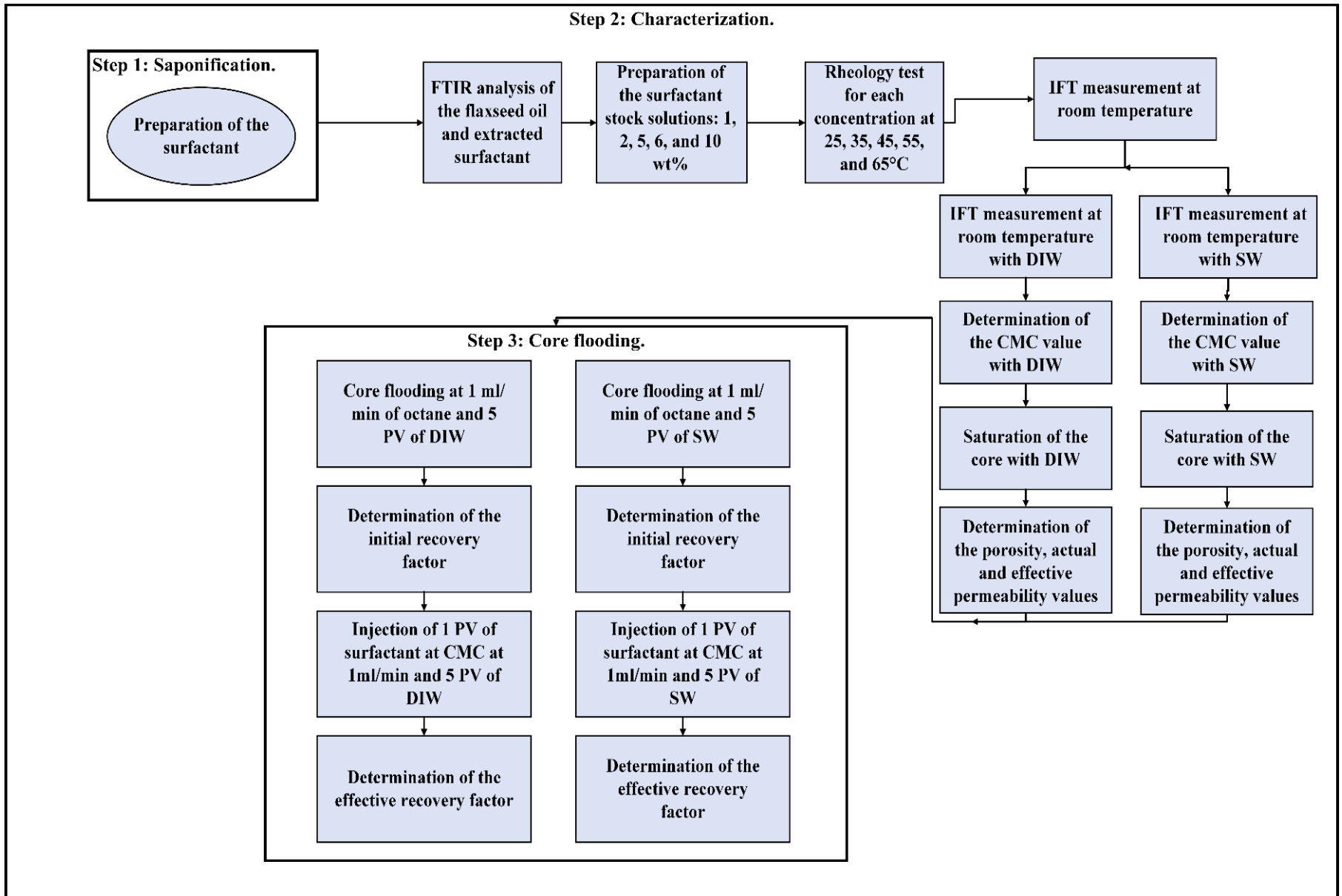


Fig. 3.1. Study workflow diagram.

3.2. Materials

Cold-pressed flaxseed oil, produced by “Lyubimoye” company, was used for this study. It is unrefined, natural, and was supplied at the local store. This type of oil is produced from flax seeds, also called “*Linum usitatissimum*,” and is well-known for its versatility. With a high concentration of omega-3 fatty acids, flaxseed oil is often used in health care, cooking, skincare, etc.

SIGMA-ALDRICH Company supplied sodium hydroxide (NaOH) used in the saponification process with a 97% purity and 40.00 g/mol molecular weight. The synthetic oil, octane, was provided by SIGMA-ALDRICH Company with a 98% purity and molecular weight of 114.23 g/mol. Moreover, the Oil Red O dye with a molecular formula $C_{26}H_{24}N_4O$ from the AlfaAesar company was used to impart a color to the oil for higher accuracy and precision of the results.

The core that was used is carbonate Edwards Brown limestone from Texas, USA. The age of the limestone is Upper Cretaceous, and Unconfirmed Compressive Strength (UCS) ranges between 1500-1700 psi.

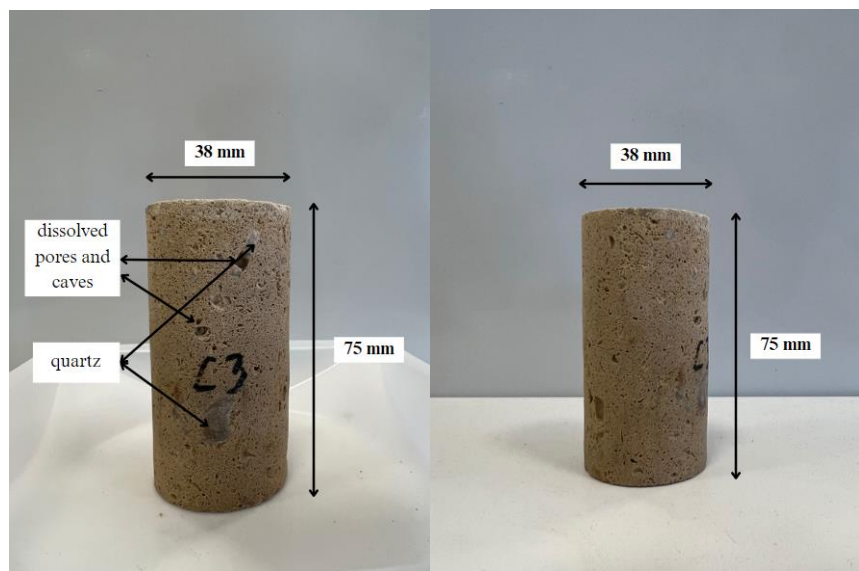


Fig 3.2. Limestone core used in experiment.

Table 3.1: Solid materials used in the experiment.

Solid Materials	Location	Additional Information
Edward Brown Limestone core	Texas, USA	The age of the limestone is Upper Cretaceous and Unconfirmed Compressive Strength(UCS) ranges between 1500-1700 psi.
Sodium chloride	Sigma-Aldrich, USA	Molecular formula NaCl, powder.
Sodium hydroxide	Sigma-Aldrich, USA	Molecular formula NaOH, pellets.
Oil red O dye	AlfaAesar, USA	Molecular formula C ₂₆ H ₂₄ N ₄ O, powder.

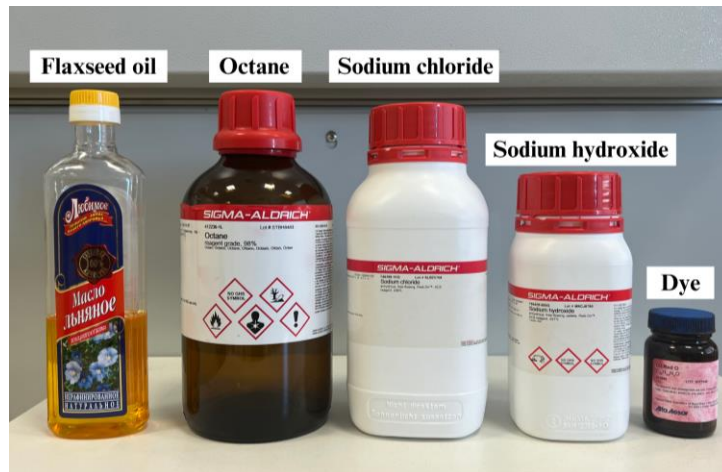


Fig. 3.3. Materials used in the experiment.

Table 3.2: The data for material properties.

Material properties	Density/ g/cm³	Viscosity at 25°C/ cP
Flaxseed Oil	0.929	25
Octane	0.703	0.51
Brine	1.023	0.97
Water	0.997	1.00

3.3. Surfactant Preparation by Saponification

A sodium hydroxide solution was prepared in a standard 250 ml beaker with 40 g of NaOH and 100 ml of distilled water (DIW) to obtain a concentration of 10M. As the reaction is exothermic and vigorous, the NaOH was added gradually by 6-7 pellets until they dissolved. The solution was constantly stirred using the magnetic stirrer IKA RH digital S000 with a minimum speed of 100 rpm.

It was decided to prepare five separate samples for the saponification process for faster and easier solidification. Five 250 ml beakers were used, with 10 ml of flaxseed oil and 10 ml of the obtained NaOH solution in each beaker. All five were placed on the magnetic stirrers at 50°C and 150 rpm stirring speed. After complete homogenization in 30 minutes, all beakers with samples were closed with laboratory wax paper and stored in a dry, dark place for 24 hours. Finally, the excessive oil was removed from the sample for further use of the solidified part.

3.4. Preparation of the surfactant solution

Surfactant stock solutions of 100 ml were prepared for each of the following concentrations: 1 wt%, 2 wt%, 5 wt%, 6 wt%, and 10 wt%. A similar preparation was also conducted for the seawater salinity. All solutions were placed on the magnetic stirrer with a speed of 150-200 rpm until their complete homogenization.

3.5. Chemical Characterization

3.5.1. FTIR analysis

Fourier Transform Infrared Spectroscopy (FT-IR) was used to characterize the group function. This type of examination is known as FT-IR Analysis or FT-IR Spectroscopy, and it's used to detect organic, polymeric, and in some cases, inorganic materials. Fourier Transform Infrared Spectroscopy (FT-IR) was used to characterize the functional chemical groups and structures of both flaxseed oil and flaxseed surfactant samples. FT-IR spectra of samples were collected using the Nicolet iS10 FT-IR Spectrometer equipped with a ZnSe flat crystal. The scan graphs were collected using OMNIC software. The background spectrum was collected initially and compared with the reference background spectra for each sample. Then, 20mg of the smashed and powdered samples were applied to the crystal, and the pressure was adjusted. Spectra were determined by recording 32 transmission scans in the range of (4000–400) cm^{-1} with a resolution

of 4 cm^{-1} . The ratio between the sample spectrum and the background spectrum displays the spectrum of the sample.



Fig. 3.4. FTIR Spectrometer.

3.5.2. IFT determination

Interfacial tension is the force existing at the interface between two immiscible phases. The apparatus used for IFT measurement was the IFT 700-HPHT Interfacial Tension Meter from Vinci Technologies, Nanterre, France. This device uses a pendant drop method to determine IFT using the Laplace equation. The mechanism is the following: through a capillary needle, a droplet (drop fluid) is formed in the chamber containing bulk fluid. Droplets can be captured by state-of-the-art image capture, and the system computes necessary geometric parameters to derive the interfacial tension using the Laplace equation. As a drop in the experiment, we used the synthetic oil octane with a red dye to differentiate it from the surfactant, as both fluids were transparent. The surfactant stock solutions with DIW and the seawater brine have been used as bulk. To set up the apparatus, the density of the fluids was considered. If the density of the drop fluid is lower than the bulk fluid, then the capillary needle needs to be set from the bottom so the drop will rise; if not, it should be the opposite.

1, 2, 5, 6, and 10 wt% concentration solutions have been used with DIW, with the former as a bulk fluid and the latter as a drop fluid. A similar procedure was conducted for the seawater samples.

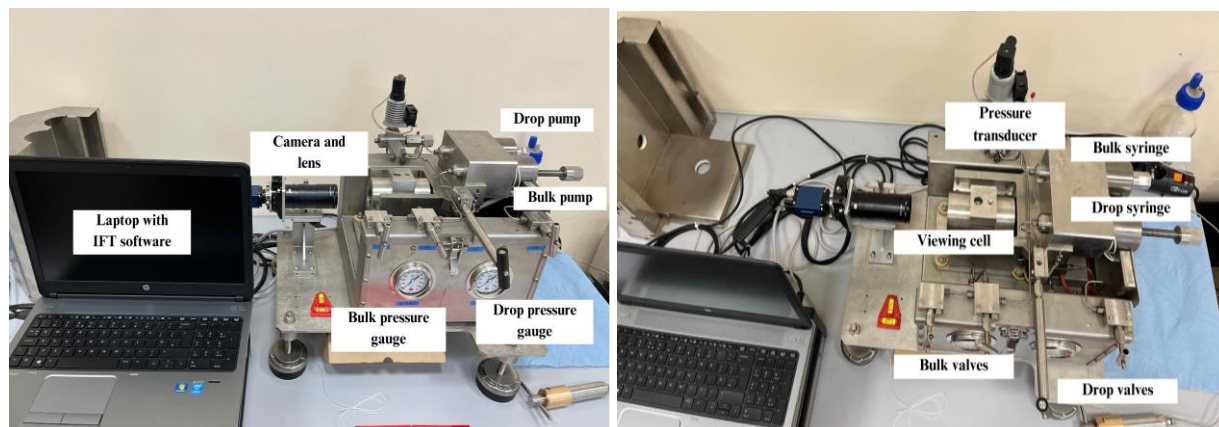


Fig. 3.5. IFT 700-HPHT Interfacial Tension Meter.

3.5.3. Rheology Test

The Rheology test was completed using the Physica MCR-302, the Austrian Anton Paar rheometer. This tool utilizes an electric current motor technique, an optimized normal force sensor, and low friction bearings to operate. As the motor moves with torque, the internal resistance of the flow of the material can be measured. Different rotational speeds are set up to determine the shear rate profile. The rheometer is able to determine the shear stress according to the Viscosity Law.

The stock solutions have low viscosities; thus, the measuring tool for the MCR-302 was changed to a cylindrical type, which requires 50 ml of solution for each run. 50 ml of stock solution was placed in the measuring cylinder, and the viscosity was measured at different temperatures between 25°C and 65°C with increments of 10°C for each concentration. The concentrations used in this experiment are 0.5, 2, and 5 wt.% of the surfactant solution with DIW.

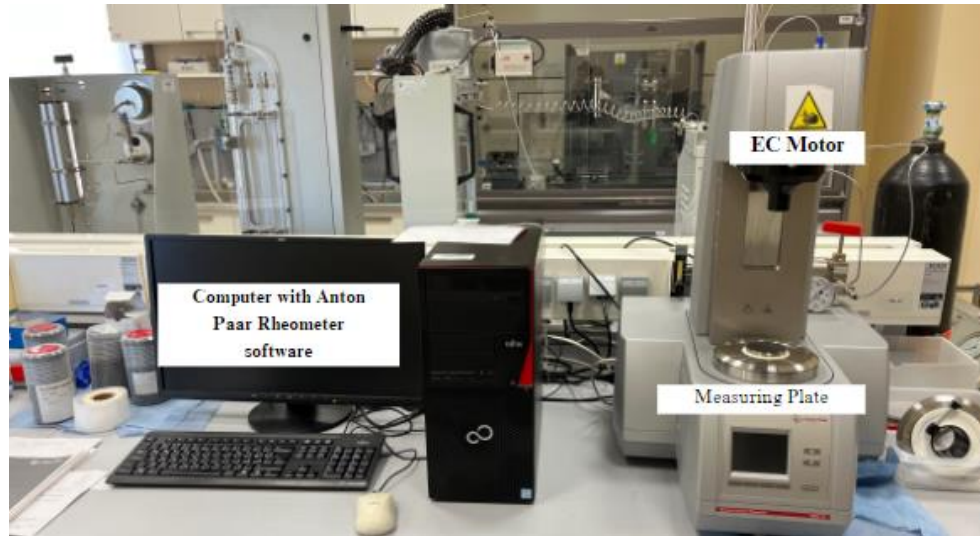


Fig. 3.6. Physica MCR-302.

3.6. Core Flooding

The ACA-700 Aging cell apparatus provided by the French Vinci Technologies was used in the further experiment. Firstly, DIW and octane were injected into the core at 5 different rates: 0.5, 1, 2, 3, and 4 ml/min for the determination of the porosity and actual and effective permeability values by using Darcy's equation. Then the flooding process began with the injection of 1 PV of octane, followed by 5 PV of DIW for the oil displacement to obtain the initial oil recovery factor by the water flooding. Then unit PV of the surfactant solution at the CMC was injected, followed by an injection of 5 PV of DIW. As a result, the total oil recovery factor has been found. The constant rate of 1 ml/min was used for the described steps. Then, the same procedure was repeated for the 35000 ppm seawater with a surfactant solution at the CMC.

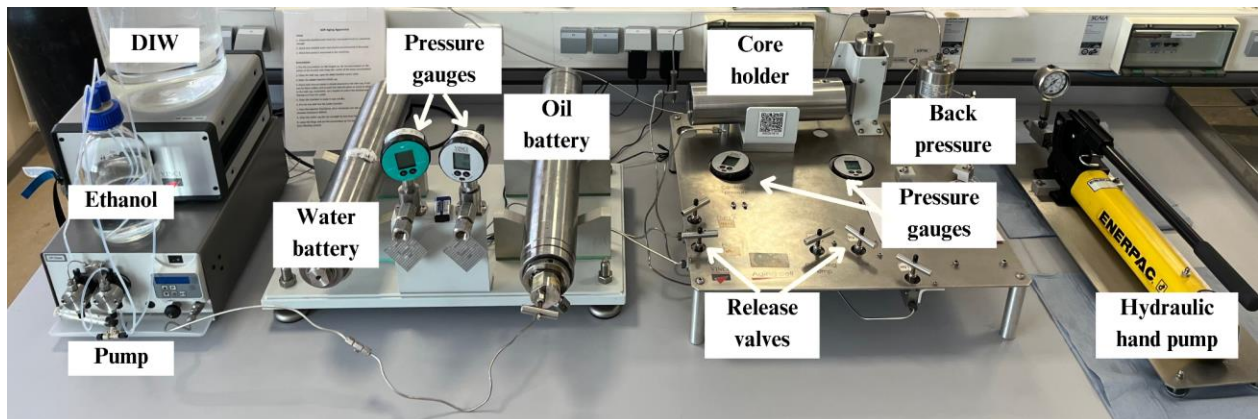


Fig. 3.7. ACA-700 Aging cell apparatus.

Chapter Four

Results and Discussion

4.1. Saponification

After stirring the solution of 10 ml of flaxseed oil and 10 ml of NaOH solution, the obtained sample was air-dried for 24-48 hours. As a result of the saponification, the fatty esters within the flaxseed oil have been broken, and fatty acid salts have been formed. The mass of the obtained surfactant was 18.45 g. The solidification process can be observed in Fig. 4.1.

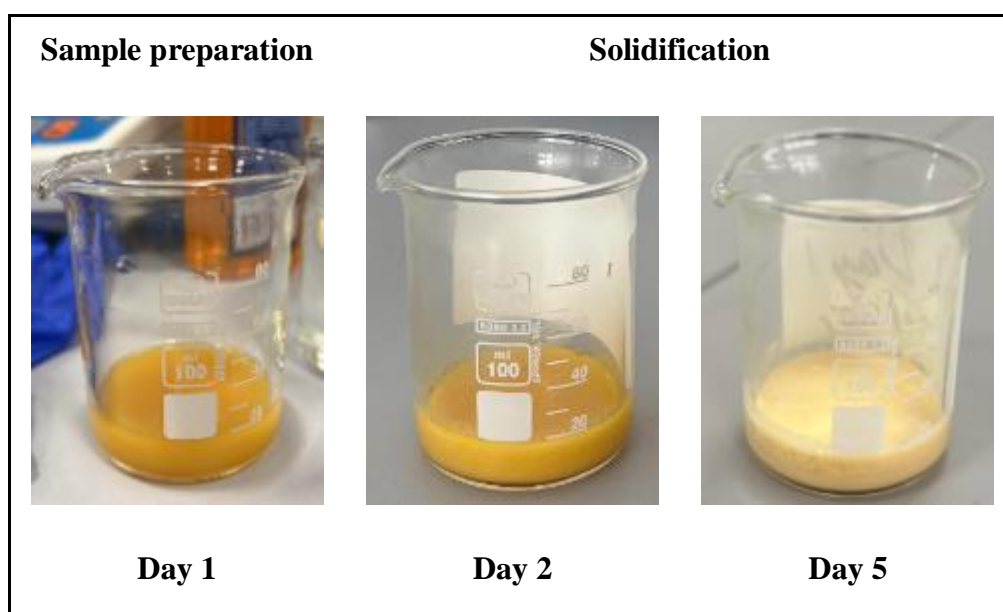


Fig. 4.1. Saponification stages.

The saponification reaction is simply the hydrolysis of an ester with a base, which results in the formation of the salt of the acid, which is soap, and the alcohol. Considering the mechanism of the process, it is considered a nucleophilic carbonyl substitution. Firstly, the ester group is attacked by a nucleophilic hydroxide ion, which forms the intermediate. Then this intermediate is rearranged by a leaving group release, which is then removed to form the carboxylic acid and alkoxide. Finally, a carboxylate ion and alcohol are formed due to deprotonation, where the hydrogen is removed from the carboxylic acid by an alkoxide (Chemistry Learner, 2021).

In this case, the triglyceride in the flaxseed oil reacted with sodium hydroxide, forming salt and glycerol. Therefore, it is suggested that the saponification process has occurred within the experiment, as can be seen from the equation in Fig. 4.2b.

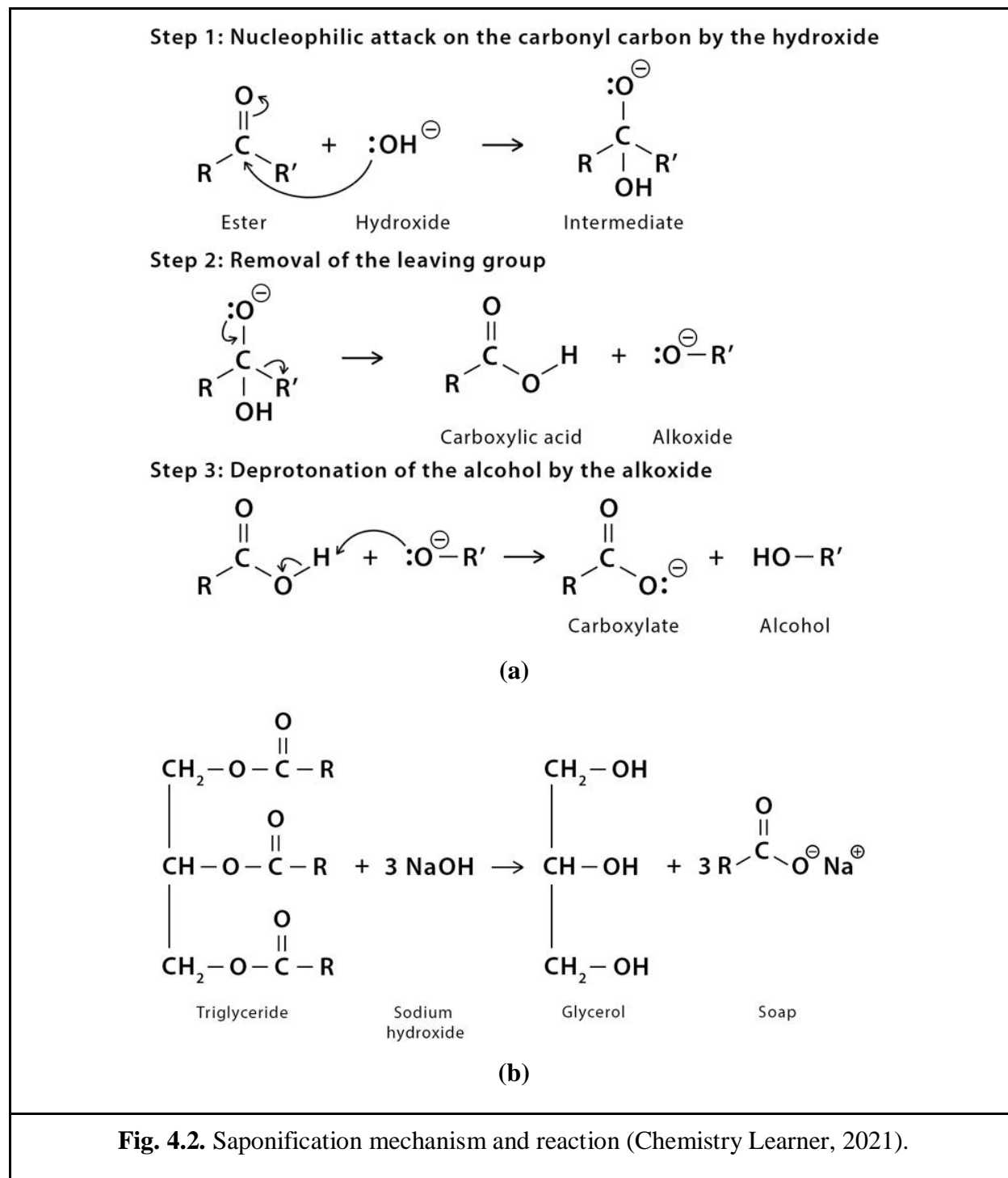


Fig. 4.2. Saponification mechanism and reaction (Chemistry Learner, 2021).

Similarly, the soap has been made in studies from different types of oil. According to Sutheimer, Caster, and Smith (2015), green soap was formed as a result of the saponification of avocado oil. The extracted avocado oil was mixed with aqueous sodium hydroxide for 20–30 minutes, and then the soap bars were cured for the following month for further solidification. Furthermore, *Jatropha curcas* seed oil was used in another experiment conducted by Salimon, Abdullah, and Salih (2012). *J. curcas* seed oil was mixed with potassium hydroxide and ethanol, with the later addition of water to form soap. Generally, the presented reaction is similar to what was reported in the literature.

4.2. Natural Material Characterization

4.2.1. Fourier Transform Infrared Spectroscopy

Fourier transform infrared (FTIR) spectroscopy was conducted to identify the functional groups by measuring the infrared radiation absorbance by the flaxseed oil and saponin samples. Considering the infrared absorption spectrum of the flaxseed oil, the bands at 3007 cm^{-1} and 2921 cm^{-1} are attributed to the vibrations of stretching C=C-H and C-H, respectively. Also, the C=O stretching and C-O stretching vibrations were observed at 1741 cm^{-1} and 1157 cm^{-1} , respectively.

The extracted natural surfactant sample's infrared absorption spectrum was considered compared to the standard saponin spectrum from the available literature (El-Keiy, Radwan and Mohamed, 2019). The characteristic infrared absorbance of the hydroxyl group (OH) at 3292 cm^{-1} was observed, whereas the terpenoid presence can be identified by the presence of the following groups: O-H, C=C, and C=O=C. The C=C absorbance was seen at a peak value of 1634 cm^{-1} , while the C=O=C absorbance was observed at 1457 cm^{-1} . Thus, the presence of saponin in the extracted surfactant was confirmed as a result of the identification of its signature - terpenoid.

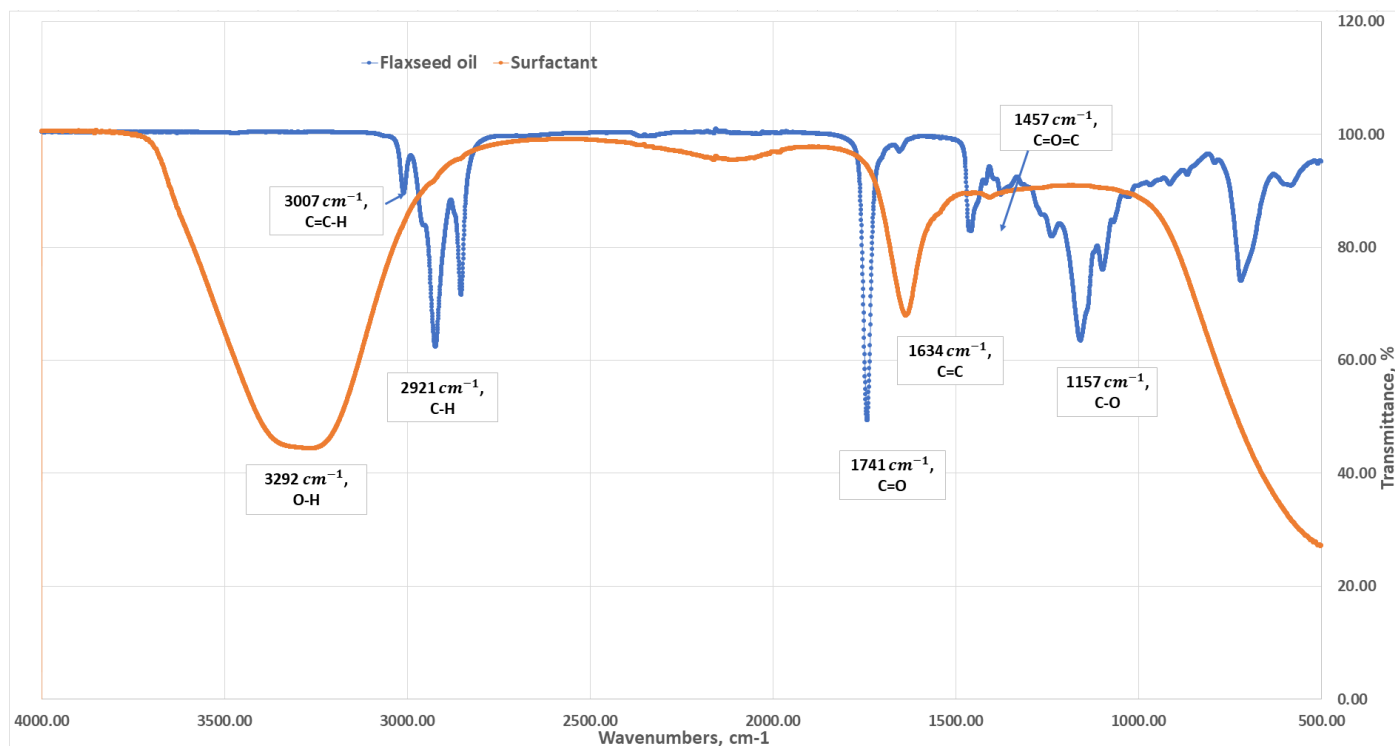


Fig. 4.3. FTIR absorption spectrum data for flaxseed oil and the extracted surfactant.

The spectra for the extracted natural surfactant were found to be similar to earlier published studies. For instance, the FTIR analysis of the surfactant extracted from the seed oil of *Baphia nitida* has identified the presence of the C-H stretching and C=C-H vibrations (Adewuyi, 2019). Moreover, the results obtained from the palm fatty acid-based polymeric surfactant analysis showed the presence of O-H and C=O functional groups (Lee, Ling and Heng, 2019). In general, most of the research papers have presented different results in the FTIR analysis, with a primary similarity by the identified C=C, O-H, and C=O functional groups. As these bonds were shown from the spectrum of the extracted surfactant sample, the presence of saponin within the surfactant can be stated based on the comparative analysis within the published literature.

4.3. Surfactant Properties

4.3.1. Interfacial Tension

The formation of micelle is an aggregation process. Micelles are formed at certain critical micelle concentration (CMC). The micelles formation results in making either the head or the tail group of the surfactant pointed inside. Accordingly, they will not be active to react with both oil

and water. Therefore, it is necessary to identify the CMC. In the current study, the IFT measurement is used as identification of the inflation point. It is usually recorded by the maximum concentration that will result in minimum interfacial tension.

The CMC values for the surfactant solutions with both DIW and at 35000 ppm salinity (sea water salinity) were identified as a result of the IFT measurement. As can be seen in Fig. 4.4a, the stabilization concentration for the surfactant in a distilled water solution was about 6 wt%. At this point, the extracted surfactant was examined for the IFT between distilled water and octane; the IFT was altered from 33 mN/m to 7.8 mN/m which is a 76.36% reduction.

The CMC value is considered important because it reflects the cost of surfactant needed to be used. The low CMC values indicate economic advantage compared to the higher one. It is well known that above the CMC value, aggregates of monomers are formed. This does not allow the interaction of the oil and water, trapping one phase inside the new shaped structure (Curbelo, Garnica and Neto, 2013).

The reduction of water-oil IFT by surfactant is similar to the micelle formation. The surfactant expands and adsorbs on oil and water, forming a film.

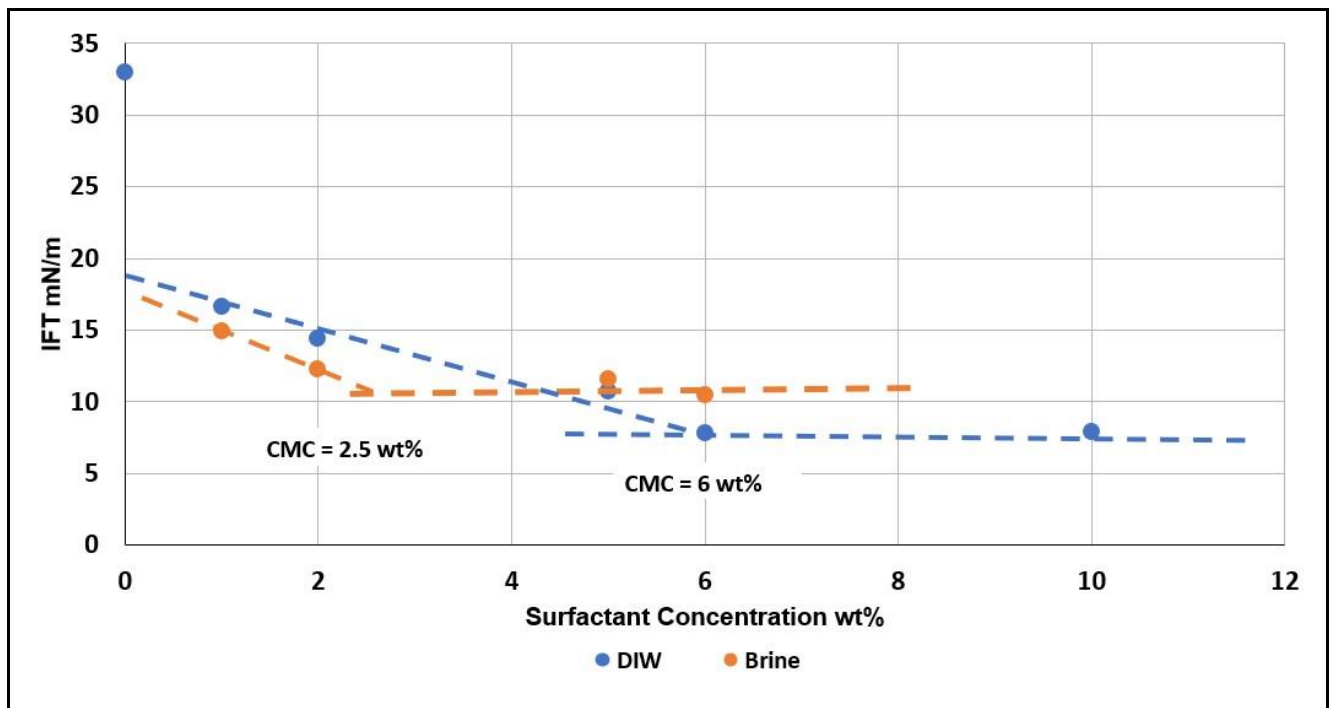


Fig. 4.4. IFT of the surfactant solution a) DIW and b) with brine with octane.

Since Kazakhstan's oil fields are mostly offshore, it is essential to consider that distilled or deionized water will not be used to prepare the aqueous phase. Therefore, the use of saline seawater is critical. In terms of the brine, an increase in the salt concentration leads to the micelles forming more readily, thus, reducing the CMC value (Zhang, Li and Zhao, 2022). The CMC value for the surfactant in the brine solution can be observed in Fig. 4.4a, which was obtained to be approximately 2.5 wt%. The IFT was altered from 23.9 mN/m to 12.3 mN/m, a reduction of 48.54%.

This observation attributes to the many mechanisms associated. The oil-to-brine IFT at high salinity is known to be less than in freshwater. This is because the ions absorb in the oil's polar component. The surfactant produced additional mechanisms for the octane case to strengthen the thin film. It allowed the stretching of the surfactant between the interfaces, reducing the CMC and resulting in less IFT compared to the DIW case in Fig. 4.4a. This phenomenon has been under discussion for all nonionic surfactants. It was reported that any addition of salt causes the CMC of nonionic surfactants to be lowered, but not as much as for ionic surfactants (Miyagishi, Okada and Asakawa, 2001).

Considering the results of published studies, the saponin extracted from the *Avena Sativa* plant has led to a 74.20% IFT alteration (Sami et al., 2022). Moreover, the surfactant extracted from *Glycyrrhiza glabra* has reduced the IFT from 49.06% to 65.64% (Daghsbandan, Shahrabadi and Arabiyoun, 2022). Compared to the different studies' values from the literature, which varied in the range of 50-70% of alteration, the IFT alteration between DIW and octane has presented a similarly high result. In contrast, the alteration between brine solution and octane resulted in a relatively lower value.

4.3.2. Rheology

In this study, the rheology of the natural surfactant was measured by rheometer using DIW solutions of 0.5, 2, and 5 wt%. The surfactant is expected to be a non-Newtonian fluid. Surfactant changes surface and interfacial tension. Also, surfactants can promote emulsification; these factors change the molecular structure of the solution (Nakama, 2017). As a result, the apparent viscosity of the solution varies depending on the concentration of the surfactant.

The rheology results in Fig. 4.5 show that increasing the surfactant concentrations results in higher viscosity, which is common behavior for natural surfactants. Additionally, as the

temperature increases from 25 to 55°C, the viscosity drops by 13%, 15%, and 23% at concentrations of 0.5, 2, and 5 wt%, respectively. The temperature mechanism has been interpreted as the rise in temperature leading to quicker movement of particles that shortens interaction time within particles and reduces internal friction; thus, the viscosity decreases (Reid, 2021).

For every concentration, the recorded viscosity increased by 0.5 cP. The rise in viscosity with concentration is due to a change in the number of molecules. The more molecules of surfactant in the solution, the harder it is to move the solution (Singh et al., 1965).

The viscosity at 10-15 s^{-1} shear rate altered between 1-2 cP. As fluids in the reservoir move at 1 ft/s speed and have a low shear rate of 8–10 s^{-1} , the above-mentioned values were taken (Nilsson and Rothstein, 2014). Also, the general viscosity of the solution is low at high shear rates.

According to Nareh'ei, Shahri, and Zamani (2012), where the characterization of plant-based surfactants was discussed, the saponin surfactant's viscosity and shear stress were similar to this study's results. However, the rheometer recorded the slight shear thickening behavior of the solution as the shear rate increased from 1 to 1000 s^{-1} . Also, some unusual decreases in the viscosity at 500 s^{-1} were shown at 2 and 5 wt%.

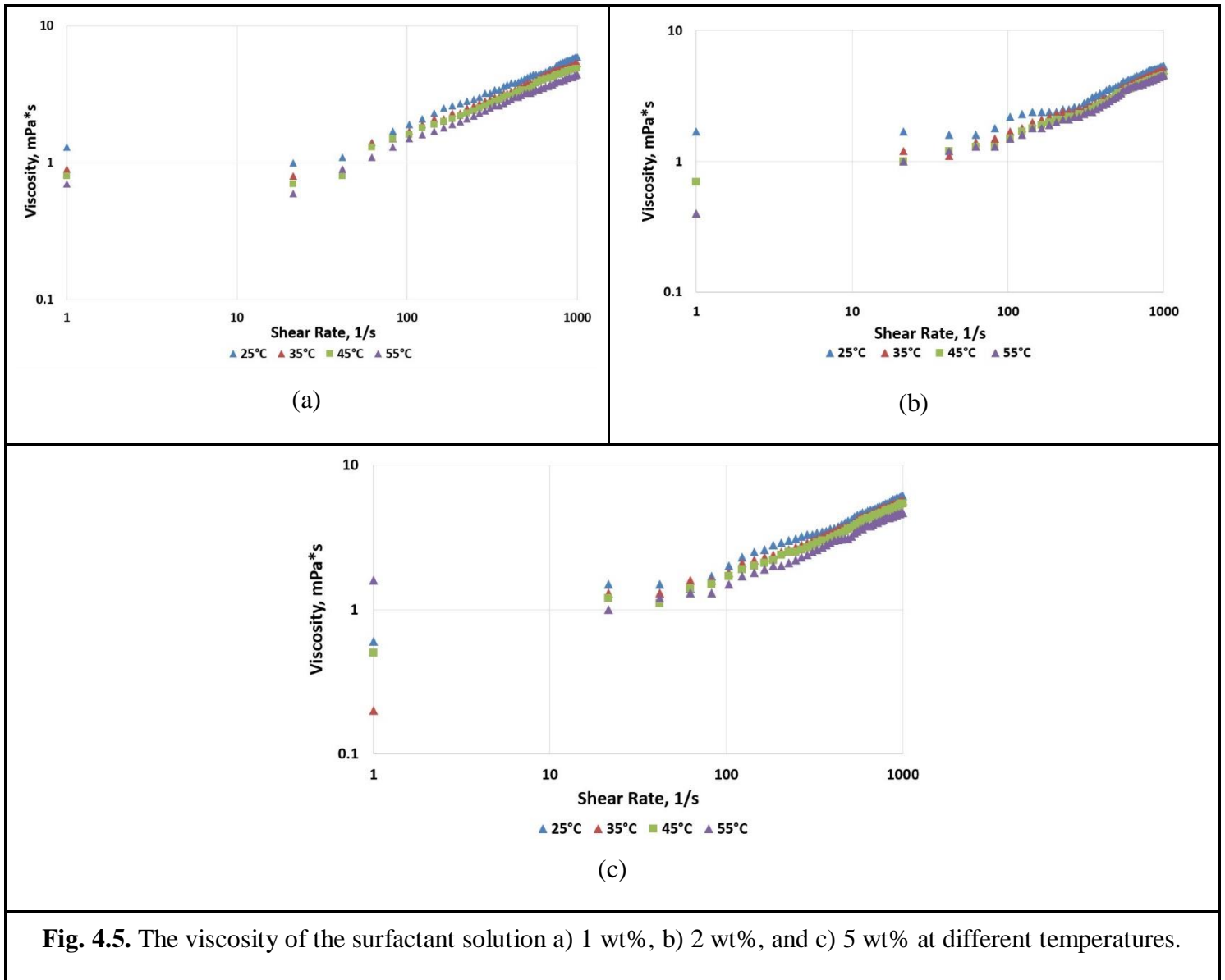


Fig. 4.5. The viscosity of the surfactant solution a) 1 wt%, b) 2 wt%, and c) 5 wt% at different temperatures.

4.4. Core Flooding

4.4.1. Core Characterization

High porosity limestone core was used in the experiment. It is a sedimentary rock with very fine grains and pebbles or quartz bodies within the rock. The core has caves and visible dissolved pores.

4.4.2. Porosity

The porosity of the core was indicated using a simple method of mass subtraction of the dry core from the saturated one. The saturated core was soaked for one day, and the pore volume of the cores was 38.81 and 37.5 ml for DIW and brine, respectively. So the porosity for both cores was calculated to be approximately equal to 38% and 36%, respectively.

4.4.3. Absolute Permeability

Core flooding in this study was conducted to characterize the core and determine further oil recovery using aging cell apparatus. The absolute permeability of core #1 with brine was reported and analyzed to characterize core pressure differences at different rates. Absolute permeability was calculated to be 287 mD and 288mD for DIW and brine, respectively (see Appendix B). According to Kocurek Industries, from which the limestone core was provided, the average permeability is 60–300 mD, which shows that the resulting value is within the range of reference. For absolute permeability calculation, the injection rates were altered; as the reaction to the rate changes, the pressure difference between the inlet and outlet also changed. Darcy's equation was used to define absolute permeability.

4.4.4 Coreflooding

For each core, 11 PVs were injected in total, with 5 PV of DIW followed by 1 PV of the surfactant solution at the CMC of 6 wt% and 5 PVs of DIW for core #1. As the flooding does not result in the recovery of all the oil within the rock, there is a limited oil recovery after the particular injected pore volume (Blunt, Fayers and Orr, 1993). As a result of the oil-water IFT, a portion of the crude oil is usually trapped in small interstices inside the rock matrix formed as oil ganglia enclosed by water. In order to confirm this concept, the injection of water proceeded after the first PV.

Regarding core #2, 5 PV of the brine solution was injected, followed by 1 PV of the surfactant solution at a CMC value of 2.5 wt% and 5 PV of the brine. The oil recovery was observed and monitored after each pore volume injection. The results are presented as a graph of the cumulative oil recovery for both DIW and brine in Fig. 4.6. As can be seen, the injection for core #1 has resulted in a cumulative recovery of 21 cc with the injection of DIW. In contrast, brine injection in core #2 has led to a total cumulative oil recovery of 28 cc of octane.

As can be observed from Fig. 4.6, only DIW injection achieved 36.5% oil recovery, whereas further injection of the surfactant solution and DIW resulted in a total oil recovery of about 72%. Such enhancement is related to the reduction in IFT and the alteration of the wettability as a result of surfactant utilization. Regarding the brine injection in another core, initial water flooding with brine resulted in an oil recovery of approximately 27%. In contrast, surfactant solution injection with brine achieved a total oil recovery of 74.6%. This attributes to the role of salinity in improving natural surfactant performance.

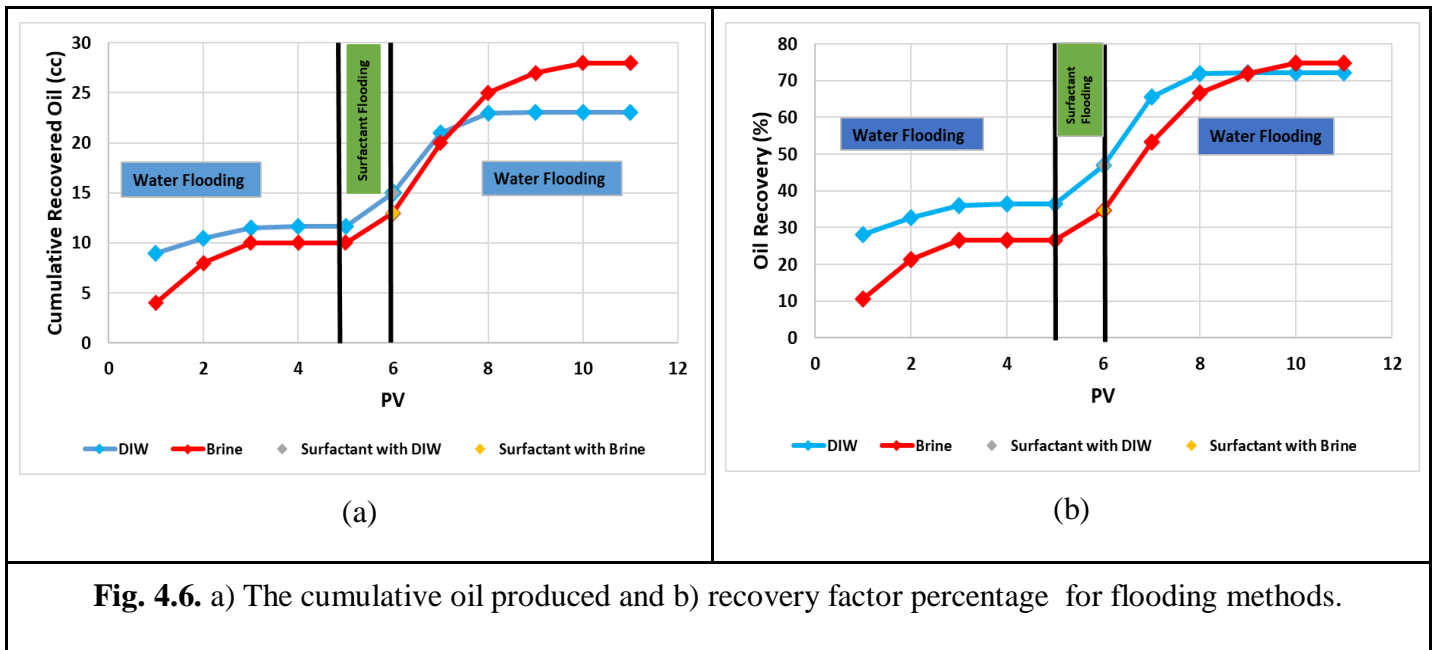


Fig. 4.6. a) The cumulative oil produced and b) recovery factor percentage for flooding methods.

Based on the obtained results, the extracted natural surfactant can be considered a suitable material to be applied in the field in the carbonate reservoirs, even in the presence of some natural fractures and caves. Furthermore, during the core flooding process, a stable increase in the differential pressure values was observed, which indicates the absence of injectivity issues.

4.5. Summary

This chapter presents and analyzes the experimental results of the study. Firstly, the mechanism and reaction of the saponification process were described as an extraction method of the saponin from the flaxseed oil. Then, the spectroscopy results from the FTIR apparatus for the extracted surfactant and flaxseed oil were analyzed separately; the triterpenoid presence indicated the presence of saponin in the surfactant solution. Moreover, IFT results for both surfactant

solutions with DIW and brine solutions were shown to determine the CMC values. The rheology and absolute and effective permeability analyses were further presented in the characterization part, followed by the core flooding data. Finally, the surfactant solutions at CMC values were utilized to obtain the total recovery factors of 72% for the solution with DIW and 74.6% for the brine solution.

Chapter Five

Conclusion and Recommendations

The need to reduce the excessive use of chemicals has drawn attention to the extraction of natural surfactants. Therefore, the potential of the natural surfactant extracted as a result of the saponification of the flaxseed oil was considered in the carbonate reservoir. Thus, this study focused on analyzing the possible application of this natural surfactant in CEOR and the changes in rock properties. Based on the investigation's findings, the following main conclusions were drawn:

- The synthesized raw material contains saponin, identified by the FTIR spectrum analysis and the distinct presence of the triterpenoid.
- The saponification reaction's effectiveness was proved by the interfacial tension reduction.
- The synthesized natural surfactant has reached to the critical micelle concentration was 6 wt% in distilled water and 2.5 wt% with a brine solution of 35000 ppm. This proves the role of seawater in reducing the necessary surfactant concentration.
- The IFT reduction was found to shift from 33 mN/m to 7.8 mN/m with a 76.36% alteration, while at the seawater salinity the IFT has decreased from 23.9 mN/m to 12.3 mN/m with a 48.54% alteration. This generally is a good indicator for the possible application at the offshore using the seawater.
- The total recovery factors for the surfactant solutions with DIW and brine solution were determined to be approximately equal to 72% and 74.6%, respectively.
- Overall, the extracted natural surfactant has demonstrated a good potential in the production enhancement in the carbonate reservoir conditions as more accessible and cost-effective alternative to the conventional surfactants.
- Thus, as Kazakhstan is considered as the main exporter of the flaxseed oil in the world, our country has sufficient resources for surfactant synthesis, which can contribute to a greener chemicals for the oil and gas industry.

On the basis of the obtained results and drawn conclusions, the following recommendations were suggested for the further analysis:

- Future research should deeply focus on the effects of the salinity and temperature on the surfactant performance. Wider range of the salinity and temperature values should be studied for the better sensitivity analysis.
- As only the synthetic oil has been used in the study, the crude oil should be utilized in the further experiment for testing the application of the natural surfactant.
- Furthermore, the actual reservoir conditions should be simulated to more accurately analyze the surfactant performance and effectiveness in Kazakhstan's fields.

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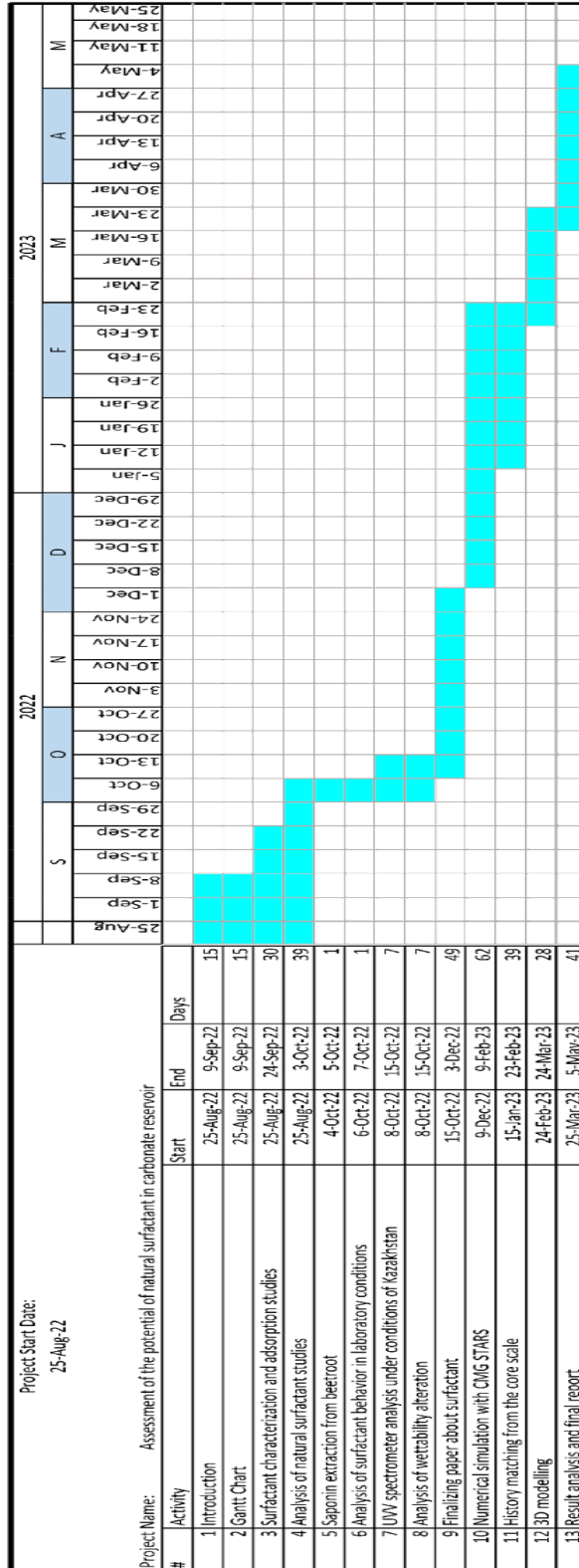
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Appendix A

Gantt Chart



Appendix B

Permeability values of core #2 with DIW: a) absolute, b) effective and core #1 with brine: c) absolute, d) effective

