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SYNTHESIS, CHARACTERIZATION, AND EVALUATION OF GEMINI POLYMERIC SURFACTANTS FOR CRUDE OIL TREATMENT AND ANTI-CORROSIO

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
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ALI ABDULLAH NEAMAH ALTALAB

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SYNTHESIS, CHARACTERIZATION, AND EVALUATION OF GEMINI POLYMERIC SURFACTANTS FOR CRUDE OIL TREATMENT AND ANTI-CORROSION

By Ali Abdullah Neamah ALTALAB

July 2023

We certify that we have read this thesis and that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Philosophy.

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ABSTRACT

SYNTHESIS, CHARACTERIZATION, AND EVALUATION OF GEMINI POLYMERIC SURFACTANTS FOR CRUDE OIL TREATMENT AND ANTI-CORROSION

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Master of Science in Chemistry

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Excessive water content in the form of emulsions within crude oil has emerged as a primary challenge within the contemporary oil industry. The formation of these emulsions in oil wells not only diminishes the desirable properties of the crude oil but also escalates the overall costs associated with the refining process. Adding to this complexity is the inadvertent mixing of saline water from nearby reservoirs into the crude oil, which subsequently triggers corrosive reactions in pipelines, insulators, tanks, and vessels through which the crude oil traverses. Consequently, the application of emulsifiers has become an imperative facet of the purification and refining protocols, given their substantial impact on the fundamental characteristics of crude oil. In order to mitigate these issues, the deployment of demulsifiers has emerged as a crucial strategy, as they facilitate the separation of water from crude oil emulsions, thereby mitigating the corrosion rates experienced within oil equipment. The current investigation focuses on the synthesis and evaluation of four novel demulsifiers (designated as F1, F2, F3, and F4). These demulsifiers were synthesized and subsequently applied at varying concentrations to serve the dual roles of demulsification and corrosion inhibition. The design of these synthesized surfactants was inspired by Gemini surfactants and they were produced via a ring-opening polymerization process, utilizing ethylene diamine tetraacetic acid as the foundational precursor material. For comprehensive characterization, techniques such as Fourier-transform infrared (FT-IR) spectroscopy and proton nuclear magnetic resonance (1H-NMR) spectroscopy were employed. Furthermore, key parameters including average molecular weight, viscosity, critical

micelle concentration (CMC), and the Hydrophilic-Lipophilic Balance (HLB) were determined to gauge the fundamental properties of the synthesized demulsifiers. In order to assess their efficacy, the synthesized demulsifiers were subjected to varying concentrations (ranging from 50 ppm to 250 ppm) and evaluated for their capacity to efficiently separate water from oil emulsions. The outcomes of this investigation led to the discernment that the separation efficiency and the effectiveness in mitigating corrosion can be ranked in the following order: DF2 > DF1 > DF4 > DF3. This study sheds light on the paramount importance of demulsification in addressing water-related challenges within the oil industry. The newly synthesized demulsifiers showcase potential in not only enhancing the separation efficiency of water from oil emulsions but also in curbing the corrosive tendencies associated with such mixtures. The detailed analysis of their structural, chemical, and physical attributes contributes to a comprehensive understanding of their potential applications in the field.

2023, 94 pages

Keywords: Synthesis, Surfactant, Gemini polymers, Anti-corrosion, Crude oil treatment.

ÖZET

HAM PETROL İŞLEME VE KOROZYON ÖNLEYİCİ İÇİN GEMİNİ POLİMERİK YÜZEY AKTİF MADDELERİNİN SENTEZİ, KARAKTERİZASYONU VE DEĞERLENDİRİLMESİ

Ali Abdullah Neamah ALTALAB
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Temmuz 2023

Ham petroldeki emülsiyon formundaki aşırı su içeriği, çağdaş petrol endüstrisinde temel bir sorun olarak ortaya çıkmıştır. Petrol kuyularında bu emülsiyonların oluşması, yalnızca ham petrolün arzu edilen özelliklerini azaltmakla kalmaz, aynı zamanda rafinaj işlemiyle ilgili genel maliyetleri de artırır. Bu karmaşıklığa ek olarak yakındaki rezervuarlardan gelen tuzlu suyun ham petrole yanlışlıkla karıştırılması da ekleniyor ve bu durum daha sonra ham petrolün içinden geçtiği boru hatlarında, yalıtkanlarda, tanklarda ve gemilerde aşındırıcı reaksiyonlara neden oluyor. Sonuç olarak, emülgatörlerin uygulanması, ham petrolün temel özellikleri üzerindeki önemli etkileri göz önüne alındığında, saflaştırma ve rafinasyon protokollerinin zorunlu bir yönü haline gelmiştir. Bu sorunları azaltmak için emülsiyon önleyicilerin kullanılması, suyun ham petrol emülsiyonlarından ayrılmasını kolaylaştırdığı ve böylece petrol ekipmanlarında yaşanan korozyon oranlarını azalttığı için çok önemli bir strateji olarak ortaya çıkmıştır. Mevcut araştırma dört yeni emülsifiye edici maddenin (F1, F2, F3 ve F4 olarak adlandırılan) sentezi ve değerlendirilmesine odaklanmaktadır. Bu emülsifiye edici maddeler sentezlendi ve ardından demülsifikasyon ve korozyon önleme gibi ikili rollere hizmet etmek için değişen konsantrasyonlarda uygulandı. Sentezlenen bu yüzey aktif maddelerin tasarımı Gemini yüzey aktif maddelerinden ilham aldı ve temel öncü malzeme olarak etilen diamin tetraasetik asit kullanılarak halka açma polimerizasyon işlemi yoluyla üretildi. Kapsamlı karakterizasyon için Fourier dönüşümü kızılötesi (FT-IR) spektroskopisi ve proton nükleer manyetik rezonans (1H-NMR) spektroskopisi gibi teknikler kullanıldı. Ayrıca, sentezlenen emülsifiye edici maddelerin temel özelliklerini

ölçmek için ortalama moleküler ağırlık, viskozite, kritik misel konsantrasyonu (CMC) ve Hidrofilik-Lipofilik Denge (HLB) gibi temel parametreler belirlendi. Etkinliklerini değerlendirmek için, sentezlenen emülsiyon önleyiciler değişen konsantrasyonlara (50 ppm ila 250 ppm arasında değişen) maruz bırakıldı ve suyu yağ emülsiyonlarından verimli bir şekilde ayırma kapasiteleri açısından değerlendirildi. Bu araştırmanın sonuçları, ayırma verimliliği ve korozyonun azaltılmasındaki etkinliğin şu sırayla sıralanabileceği anlayışına yol açtı: DF2 > DF1 > DF4 > DF3. Bu çalışma, petrol endüstrisinde suyla ilgili zorlukların çözümünde emülsifikasyonun büyük önemine ışık tutuyor. Yeni sentezlenen emülsiyon önleyiciler, yalnızca suyun yağ emülsiyonlarından ayrılma verimliliğini arttırma konusunda değil, aynı zamanda bu tür karışımlarla ilişkili aşındırıcı eğilimleri azaltmada da potansiyel sergiliyor. Yapısal, kimyasal ve fiziksel özelliklerinin ayrıntılı analizi, bu alandaki potansiyel uygulamalarının kapsamlı bir şekilde anlaşılmasına katkıda bulunur.

2023, 94 sayfa

Anahtar Kelimeler: Sentez, Sürfaktan, Gemini polimerleri, Korozyon önleyici, Ham petrol arıtma.

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Ali Abdullah Neamah ALTALAB

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CONTENTS

LIST OF SYMBOLS

Ca	Calsium
Cu	Copper
/	Divide
dL	Deciliter
=	Equal
<	Greater than
≤	Greater or equal to
g	Gram
kg	Kilogram
>	Less than
≥	Less or equal to
L	Liter
µg	Microgram
Mg	Magnesium
mg	Microgram
mIU	Milli-international units
min	Minute
mL	Milliliter
mmol	Millimole
mol	Mole
-	Minus
Ml	Microliter
nm	Nanometer
NS	Non-significant
ng	Nanogram
+	Plus
±	Plus minus
%	Percent
rpm	Revolutions per minute
**	Significant
m^2	Square meter
Se	Selenium
Zn	Zinc

LIST OF ABBREVIATIONS

AE	Auxiliary electrode
BLDH	British drug house
CMC	Critical micelles concentration
DTA	Differential thermo analysis
EI	Electrochemical impedance
EN	Electrochemical noise
HLB	Hydrophilic – Lipophilic balance
IDT	Initial decomposition temperature
IE	Inhibitor efficiency
LPR	Linear polarization resistance

LIST OF FIGURES

LIST OF TABLES

1. INTRODUCTION

Crude oil was extracted from wells accompanied by some impurities such as water, salt, and sediments (Islam 2015, (A M Al-Sabagh *et al*. 2016). Crude oil extraction is the process of removing oil from the ground, which is a complex and multi-stage process. After extracting to the surface, crude oil is then transported to a processing facility, where it is separated from any natural gas and water. Emulsion water is a common component of crude oil, that may have come up with it during extraction, which makes processing crucial to extract crude oil. Chemical engineer use demulsifiers to separate water from crude oil based on an effective surfactant (Kelland 2014, Jiang *et al*. 2013).

These materials are used in a variety of industries due to their high ability to adsorb and separate both surface interfacer and produce building elements as detergents, dispersants, foams, pharmaceuticals, anti-corrosives (liquid crystals and corrosion) for environmental and petroleum treatments, cosmetics, and electronic applications (Martínez-Palou and Aburto 2015, Zarrouk *et al*. 2012 ,Wang *et al*. 2015). The importance of surfactants in the oil industry is demonstrated by the practical aspect of surfactant use of our interest. The petroleum recovery process uses surfactants, which include reservoir injection, oil well production, surface station operations, petroleum emulsion pipelines, and maritime transportation (Schramm 2000).

A type of surfactant known as a Gemini surfactant has two hydrophobic tails and two hydrophilic heads that are covalently connected by a spacer group. Unlike conventional single-tailtail and single-headhead surfactants, Gemini surfactants have special characteristics. Some of these characteristics include excellent aqueous solubility, low critical micelle concentration (CMC), heat resistance, salt resistance, and ultra-low interfacial tension (IFT). Numerous applications in the oil field have been successfully applied cationic Gemini surfactants based on ammonium (Nguele *et al.* 2016).

Gemini surfactant is a surface-active agent, its main objective is to lower the surface tension of emulsion and help the oil and water to mix in a continuous phase. The main steps of isolation and treatment process are summarized in three consecutive steps: heating, chemical treatment and finally electrical treatment. Heating: Heat is still a traditional method for separating water from oil. As it helps to reduce the viscosity of the oil, and there are two basic methods of heating: the first is direct heating by passing the oil through a file that is exposed to hot combustion gases. The second is heating in the indirect way through the heat exchange of oil with another thermal medium such as water. The choice of heating method depends on the amount of hot water in the oil. The effect of heat in reducing viscosity, helps to increase the separation speed by enhancing the merging of droplets that will form large droplets as a result of their collision with each other. Despite the positive effects of heat on the entire process, there is some negative effect on oil containing light compounds, as their evaporation may affect the total volume of oil (Babich and Moulijin 2003). As approximately 1% of the total volume lost with the liberation of these compounds or gases, which may cause turbulence that impedes the gathering of water droplets and prevents their stability. This problem is addressed by taking into account the design of the buffer (Devold 2013).

In the upscaled designs, the oil is kept above bubble pressure by placing small isolators on top of the treatment vessels. As the heat required to heat the water is double then the heat required to heat the oil. Chemical treatment: After heating, the oil enters the water buffer; But after injecting it with a chemical substance called an emulsifier the water remains suspended in the oil and is not subject to sedimentation (Devold 2013). An mulsification is process takes place in the presence of two liquids that do not dissolve in each other, but rather disperse one in the other. One of the most important factors for emulsification is the presence of solids, paraffin materials, and asphalt which dissolve organic acids and other chemicals. Emulsifiers are polymeric substances with large molecular weights that reduce the surface tension of water droplets, thus helping to separate water from oil (Azim *et al.* 2011). In addition to the emulsifier, the oil is treated with wash water,

which leads to a decrease in the concentration of the brine, and then helps to extract the largest amount of salts in the oil and water as the degree of salinity is less than the salinity of the water in the oil. The wash water is prepared through a system called the wash water organization and the emulsifying fractions can be injected into the oil entry line to the separator. Electrical treatment: Inside the oil insulator, the oil rises to the top to enter an electric field. This method has a major role in the merging of small water droplets scattered and dispersed in the oil. These drops become active when a high electric current is applied to them. As the water becomes charged with an electric charge that causes attraction and repulsion with other droplets, and then collides with them and forms large droplets that facilitate the process of separation and sedimentation due to their heavy weight (Martínez-Palou and Aburto 2015, Zarrouk *et al*. 2012, Wang *et al*. 2015).

The electric current is supplied by some electrical transformers installed on top of the insulator, which consists of a network of carbon-steel electrodes. The phenomenon of water droplets uniting with each other as a result of the electric current is explained by one of the following physical phenomena: These droplets may become polarized and tend to align themselves with the electric field lines, as a result of torque. These positive and negative droplets collide with each other, forming larger droplets, which facilitate their collection. The droplet elongates both horizontally and vertically due to the increase in surface tension. The oil insulators contain two or three groups of electrodes connected to an electrical transformer. The oil enters through a tube that contains openings to distribute the oil evenly in the insulator to rise the electric field, prepared by the electrodes (Kelland 2014, Jiang *et al*. 2013).

So, the surface production plants, with the help of petroleum insulators can be summarized in three steps: Breaking the emulsion, droplet merger and sedimentation by gravity. In the event of some operational problems; The oil leaving the buffer will not meet the required specifications in terms of the percentage of water and salts, so another line must be prepared to return the oil to processing again. If the separation process is successful, the treated oil is

transferred to storage tanks to be pumped later for export. As for the gas and water coming out of these buffers, each of them goes to another treatment system for the purpose of benefiting from them (Azim *et al.* 2011, Devold 2013).

1.1 The Aim of Study

The objective of the study is as follows:

A. The preparation of polymeric surfactant materials and evaluating their efficiency using electrochemical measurements to determine critical micelles concentration (CMC) and other chemical analysis techniques.

B. Preparation of laboratory demulsifiers and evaluation of their efficiency in separating water from crude oil for use in industrial treatment of natural oil emulsion problems.

C. The weight loss method was used to assess the effectiveness of emulsifiers as corrosion inhibitors on carbon steel corrosion in hydrochloric acid solution.

2 . LITERATURE REVIEW

2.1 Surfactants and Their Importance

Surfactants are chemical compound that can lower the surface tension between two substances, typically between a liquid and a gas or between two immiscible liquids. They are made up of molecules that have both hydrophobic and hydrophilic properties, which allow them to intract with different type of substance. They are used in a wide range of scientific and industrial applications, commonly in cleaning, detergent, personal care items and as a helping material in emulsification and solubilization of oil and dirt. Moreover, surfactans play variety of other function including, foaming agent, wetting agent, dispersants and viscosity modifier. However, these days synthetic surfactants are preferred over natural surfactant, because of their greater versatility in term of their properties and performance (Guzman *et al.* 2016, Attwood 2012).

Synthetic surfactants are type of surfactants, made up of synthetic or man-made materials as opposed to natural surfactent. Now a days, much of the synthetic surfactant industry is well documented, one common type of synthetic surfactant is sodium lauryl sulfate which manly used in personal care products. Abother type of synthetic surfactant is alkylbenzene sulfonates (ABS), which are commonly used in laundry detrgents and other cleaning products. Beauty of the thing is, synthetic surfactans are versatile as they can be designed with a specific properties and functions to meet the need of a wide range of application. Even, they offer greater versatility than natural surfactants and can be tailored to specific uses and performance requirments (Schramm *et al*., 2000, Soderman *et al*., 1999).

Moreover, theyir property of consistency makes them more unique compare to natural surfactant, as they can be manufactured to consistant specification, ensuring that they can perform predictability and reliability. Moreover, they need lower concentration to achieve the same level of performance as natural surfactant. In addition, synthetic surfactant have higher stability and long shelf life, which makes them more suitable for use in different area. The novelty of synthetic surfactants offers novel properties and

function that are abscent in natural surfactant. Such as gemini surfactants are a type of synthetic surfactant that has unique properties such as lower critical micelle concentration and ability to form smaller micelles then traditional surfactants. In our proposed study we will discus about some method of synthesis of surfactet obtained from crude oil and their applications in different areas (Menger *et al.* 2000, Rosen *et al.* 1998).

2.2 Crude Oil

It is a complex mixture of hydrocarbons that can exist as a solid, liquid, or gas, depending on temperature and pressure. All types of crude oil extracted from the ground are liquid or gaseous. The composition of crude oil varies depending on its source, but typically it contains a mixture of different hydrocarbons with varying molecular weights and structure (Yarranton., 2022). As for the external appearance, it is an oily liquid with a color between yellow and brown, and sometimes it is dark black, lighter than water. The degree of the American Petroleum Institute (API) ranges between (50-60) and viscosity of (5-75000) centipoise in standard conditions. Its smell is acceptable if it is free of sulfur and nitrogen and visversa. In terms of content, crude oil is a complex mixture of hydrocarbon chains with carbon and hydrogen in their chemical composition. Crude oil also contains sulfur, nitrogen, oxygen, metallic elements, and a small percentage of inorganic salts (demirbas *et al*. 2015, Yarranton., 2022, Njoku *et al*., 2009).

Table 2.1 Elements present in crude oil (Panchal *et al*. 2017).

NO	Element Symbol	Percentage
1	C	83-87 %
2	H	11-13 %
3	S	0.1-3 rarely up 7%
4	N_2	0.1-2 %
5	O_2	1.5 max
6	Ni, Na, Pb, As, V	A tiny percentage, expressed in parts per million (ppm)

The preliminary gravimetric analyzes of the various types of oil indicate that the elements present in the hydrocarbon compounds found in crude oil in general are shown in Table 2.1. Crude oil is a valuable resource because it can be refined into a range of products, including gasoline, diesel fuel, lubricants and other chemicals. The refining process invoves seaparating the different hydrocarbons in the crude oil mixture through a process called fractional distillation which further processed to get the desired product (Panchal *et al*. 2017).

2.3 Classification of Surfactants

Commercially, surfactants are frequently categorized into different categories based on their chemical structure, charge and solubility. Though, it is confusing and ineffective to categorize them, as many surfactants have multiple functions. Some common classification scheme for surfactants is based on charge, hydrophilic-lipophilic balance, charge and acccoring to their applications. However, the most popular and widely accepted classification system in science is based on surfactants' capacity to dissociate in water (Salager 2002). In a catalytic mechanism, an anion (a negatively charged ion that moves toward the anode during electrolysis) and a cation (a positively charged ion that moves toward the cathode) are produced when surfactants separate in water. The anion is typically an alkaline metal (Na^+, K^+), or quaternary ammonium ion. (Tadros 2005, Rocha e Silva *et al*. 2017). Based on their polar group, anionic compounds (Figure 2.1) are classified as follows (Farn 2008):

1. Sulphonates Cn H_2n +1SO_3^- M^+
2. Sulphates Cn H_2n +1$OSO_3^-M^+$
3. Phosphates Cn H_2n +1OPO (OH)O^- M^+
4. Carboxylates Cn H_2n +1COO^- M^+

The counter ion M is usually Na with n = 8–16 atoms.

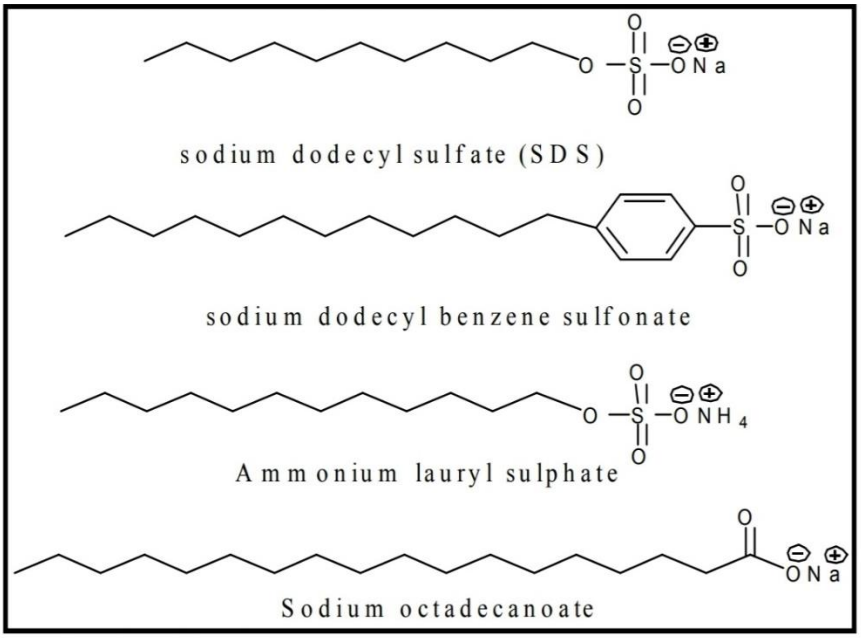

Figure 2.1 Structural formula of anionic surfactants with hydrophilic and hydrophobic characteristics (Farn 2008)

Nonionic surfactants have no charge and are often used in hair and skin care products and accounting for nearly half of all industrial output. Due to the hydrophilic group, they dissolve into alcohol, phenol, ether, ester, or amide. However, they do not ionize in an aqueous solution (Salager 2002, T F Tadros 2005, Rocha e Silva *et al.* 2017) as shown in Figure 2.2.

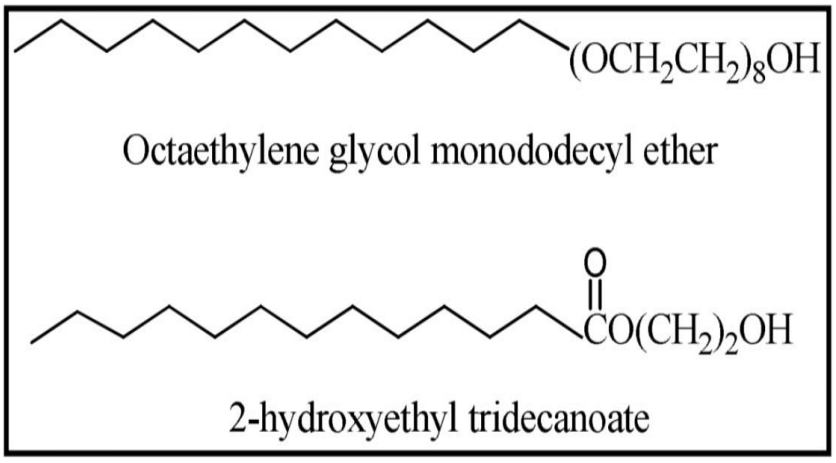

Figure 2.2 Structural formula of nonionic surfactants (Salager 2002)

Moreover, cationic surfactants split into cation and an anion in water, which are both halogens. A sizeable portion of this class is made up of nitrogen compounds that have one or more long alkyl chains, such as fatty amine, salts and quaternary ammoniums. Alkyl trimethyl ammonium chloride is one typical class of cationic surfactant, as shown in Figure 2.3, where R contains (8–18) carbon atoms.

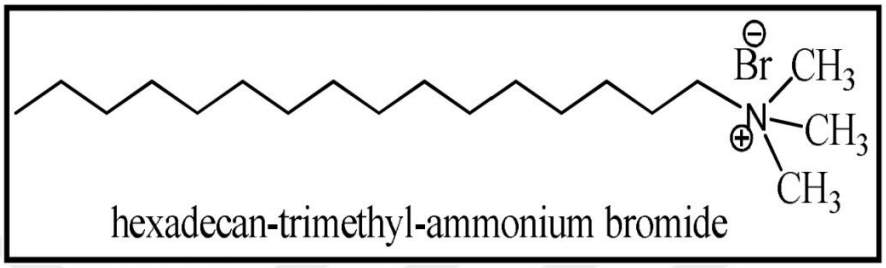

Figure 2.3 Structural formula of cationic hexadecane trimethyl-ammonium bromide (Rocha e Silva *et al.* 2017)

There is another type of ionic surfactants called amphoteric (zwitterionic) surfactants. As cationic compounds have two functional groups, anionic and cationic, while amphoteric surfactants have both negative and positive charge on their structure. They differ from other types of surfactants as they are very sensitive to the pH of the solution in which they are dissolved.

In acidic pH solutions, the molecules have a positive charge and functions as a cationic surfactant. However, in alkaline pH solutions, it contains negative charge and function as an anionic surfactant. The isoelectric point of a molecule depicted in Figures 2.4 and 2.5, is the pH at which both ionic groups exhibit equal ionization potential such as trimethyl glycine's derivatives, called N-alkyl betaines, $(CH_3)3NCH_2COOH$ are the most common amphoteric (described as betaine) (Salager 2002, Tadros 2005, Tadros 2009).

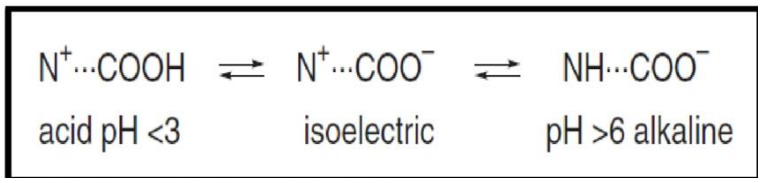

Figure 2.4 Amphoteric surfactants in different acidic environment (Salager 2002)

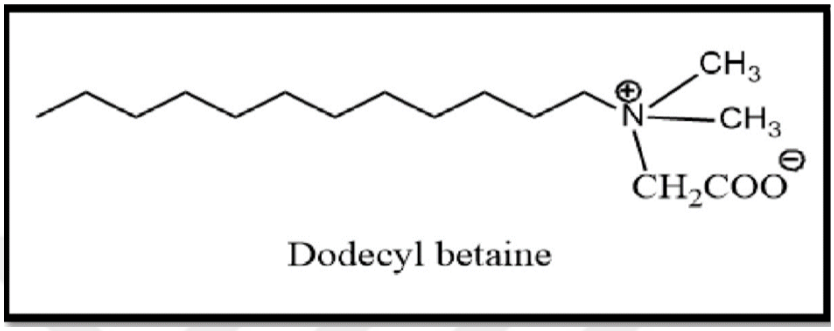

Figure 2.5 Structural formula of dodecan betain an amphoteric surfactant (Tadros 2005).

2.4 Gemini Surfactants and Its Properties

Gemini surfactants (GS) also known as dimeric or gemini-type surfactant (Figure 2.6), which have unique properties compared to traditional surfactants. This novel type of dimeric surfactant has recently catched the interest of people in both industrial and academic worlds. There is a spacer unit, which connects two conventional surfactant units in Gemini surfactants (Chu, Feng, and Seeger 2015). The two terminal hydrocarbon tails may be short, long, flexible, or rigid, and their polar head groups may be cationic, anionic, or nonionic. However, the spacer can also be rigid, flexible, short, or long as shown in Figure 2.6. Moreover, the gemini surfactant do not need to be symmetrically arranged around the spacer's center (Ahmed *et al.* 2011). In terms of characteristics of gemini surfactant, such as, critical micelle concentration (CMC), enhanced wetting properties, and lower limiting surface tensions. Gemini surfactants performs better job than conventional surfactants. Inaddition, there unusual morphologies for aggregation (Menner and Bismarck 2006), resulting in its suitability for catalysis, adsorption applications, analytical separations, solubilization procedures,

nanoscale technology, biotechnology, enhanced oil recovery, and in paint additives (Tharwat 2017).

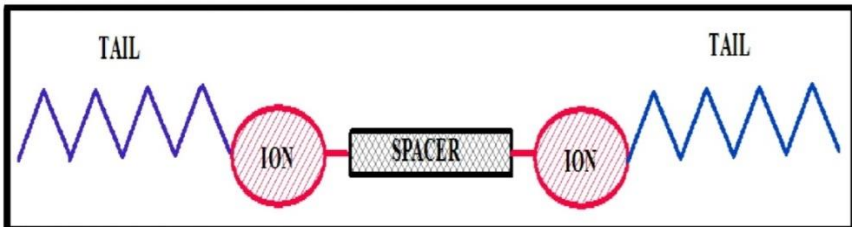

Figure 2.6 Structural representation of gemini surfactant (Tharwat 2017)

Dipti Shukla and Tyagi (Yin *et al.* 2020) synthesized alkanediyl-, bis(alkyl dimethyl ammonium bromide), and bis(alkyl dimethyl ammonium bromide) bis(alkyl dimethyl ammonium bromide) bis(alkyl dimethyl ammonium bromide) bis(alkyl dimethyl ammonium bromide) bis(alkyl dimethyl ammonium bromide) bis (quaternary ammonium bromide). These compound's surface-active characteristics were looked into their solubility, CMC values, surface tension at CMC value, foam ability, and foam stability. Researchers looked into anionic-cationic mixed surfactant systems made up of cationic gemini surfactants from the N, N'-bis-(dimethyl dodecyl), alkane diammonium dibromide series and anionic 30 sodium dodecyl trioxymethylene sulfate (SDES) (Urquidi-Macdonald and Macdonald 2014).

Moreover, it was investigated that gemini surfactant have anti-adsorption and anti-corrosion properties (Heselmans *et al.* 2013). The abbreviation CmC6Cm refers to four quaternary ammonium gemini surfactants from the hexanediol-1,6-bis-(diethyl alkyl ammonium bromide) series. In a one mol/L HCl solution at 25 °C, 2Br (m=10, 12, 14, and 16) was synthesized and tested as an inhibitor of corrosion in aluminum. As the concentrations of surfactant approached critical micelle concentrations by increasing 2Br concentrations, resulting a good inhibition efficiencies (Heselmans *et al.* 2013). Researchers also looked into how chemical demulsifiers affected water and oil separation from water in oil emulsions (Sulka and Jóźwik 2011). However, cationic surfactants from gemini was being investigated (Al-Silawi 2014). Therefore, two different types of demulsifiers (DH_2 and DH_3) were prepared as (4,4'-Di amino-di

phenyl ether). The separation efficiency was 30 % for DH_2 and 50 % for DH_3 (AL-Silawi 2014).

2.5 Emulsions

The mixture of two or more immisible liquids is refer to emulsion, that are stabilized by third component known as emulsifier or surfactant. Emulsions are colloidals suspensions in which disperse liquid is present in small droplets through out the continuous liquid phase. Emulsions can be classified into two broad categories: oil-in-water (O/W) and water-in-oil (W/O). In an O/W emulsion, the dispersed phase is oil, and the continuous phase is water, while in a W/O emulsion, the dispersed phase is water, and the continuous phase is oil (Menner *et al.* 2006, Tharwat 2017).

Emulsions are commonly used in a range of industries, including food, pharmaceuticals, cosmetics, and paints. In the food industry, emulsions are used to create stable mixtures of oil and water-based ingredients, such as mayonnaise, salad dressings, and ice cream. In the pharmaceutical industry, emulsions are used to deliver drugs in liquid form for oral or topical administration. In the cosmetics industry, emulsions are used in lotions, creams, and other personal care products (Wang *et al.* 2015, Tamilvanan and Benita 2004). The stability of an emulsion depends on several factors, including the choice of emulsifier, the concentration of the emulsifier, and the ratio of the dispersed phase to the continuous phase. Emulsions can be unstable and can break over time, resulting in the separation of the two phases. To improve the stability of emulsions, additional ingredients such as stabilizers or thickeners may be added (Menner *et al.* 2006, Tharwat 2017).

2.5.1 Classification of emulsions

Emulsions can be classified based on the nature of the dispersed and continuous phases. There are two main types of emulsions: oil-in-water (O/W) and water-in-oil (W/O).

- **Oil-in-water (O/W) emulsion:** In an O/W emulsion, the dispersed phase is oil and the continuous phase is water. O/W emulsions are the most common type of emulsion and are used in a variety of applications, such as food, personal care products, and pharmaceuticals (Tamilvanan and Benita 2004). Examples of O/W emulsions include milk, mayonnaise, and lotion.
- **Water-in-oil (W/O) emulsion:** In a W/O emulsion, the dispersed phase is water and the continuous phase is oil. W/O emulsions are less common than O/W emulsions, but they are important in certain industries such as the production of certain types of creams and ointments (Wang *et al.* 2015). Examples of W/O emulsions include butter and margarine (Figure 2.7)

Emulsions can also be classified based on the type of emulsifier used to stabilize the mixture. There are three main types of emulsifiers such as anionic, cationic and non-ionic imulsifiers. Furthermore, they can also be classified on their physical properties, such as the droplet size, viscosity, and stability. Emulsions can range from low viscosity liquids to thick gels, and their stability can vary depending on factors such as the type and concentration of emulsifier used, temperature, and pH (Ouyang and Han 2017, Kedzior *et al.* 2021).

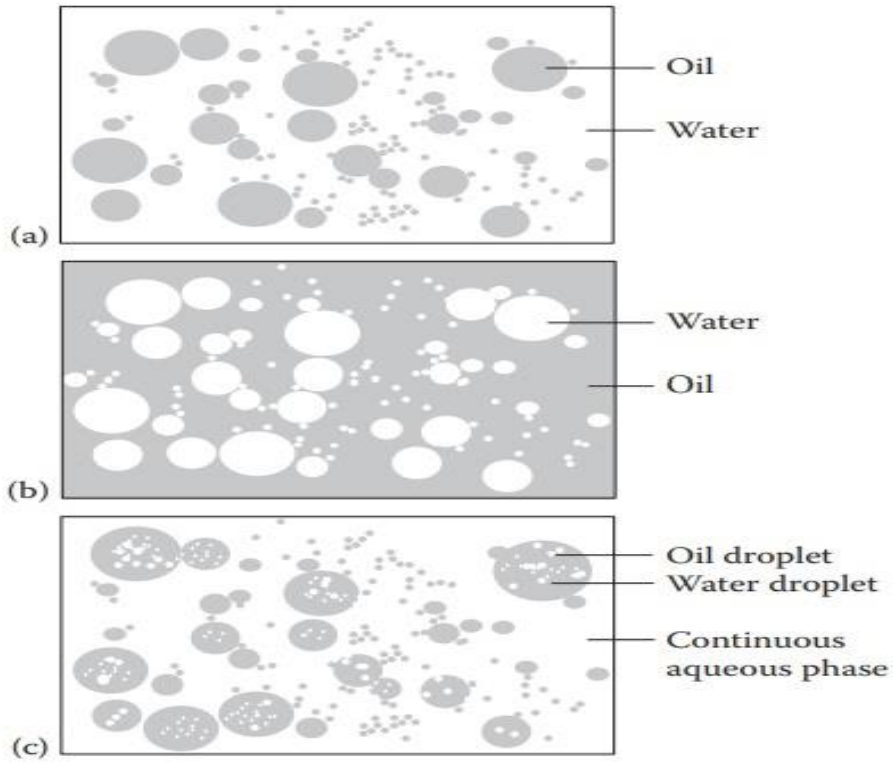

Figure 2.7 Emulsions and its types (Pharmacy180.com)

2.6 Demulsifier and Their Mechanism of Action

A demulsifier is a substance that is used to break down emulsions, which are mixtures of two or more immiscible liquids stabilized by a third component, such as an emulsifier or surfactant. Demulsifiers are also known as emulsion breakers or emulsion destabilizers (Yin *et al.* 2020, Alvarado *et al*. 2011). In emulsions, the water ratio with oil extraction process is up to (10%) by weight or more of the total production. Therefore, it requires additional separation stages, especially when using steam to move heavy oil. The presence of water, damages the purification and filtering equipment, consumes double amount of heat then oil and causes corrosion in pipelines, storage tank and transport basins. Demulsifiers work by disrupting the interfacial film that stabilizes the emulsion, causing the dispersed phase to separate from the continuous phase. This can be achieved through a variety of mechanisms, such as electrostatic destabilization, steric hindrance, or chemical reaction with the emulsifier or the dispersed phase. Demulsifiers are used in a variety of industries, such as oil and gas production, wastewater treatment, and chemical processing (Pensini *et al.* 2014). In the oil and gas industry, for example, water-in-oil emulsions can form during production and transportation, reducing the efficiency of the extraction process and causing corrosion and equipment damage. Demulsifiers are added to break down these emulsions and separate the water from the oil (Yin *et al.* 2020, Alvarado, Wang, and Moradi 2011).

- Aggregation: The collecting of water droplets during a short period leads to the crash of the emulsion combination where the separation process is done in two-phase (Kang *et al.* 2018).
- Adsorption occurs at the emulsion surface and increases the surface area by surfactant materials breaking at the interface point between surfactant material and emulsion (Ramalho, Lechuga, and Lucas 2010), (Yang *et al*. 2010).
- Remove the dense parts like sediments by surfactants.

When treating crude oil emulsions, copolymers based on poly ethyleneimine (PEI demulsifiers as an accepter are being investigated and evaluated (Wang *et al.* 2015)). It

was discovered that the best demulsifying performance was (the P199-mn series). At a concentration of 50 mg, the separation efficiency was 95%. L^{-1} with a 60-minute time limit (Wang *et al.* 2015) .

In a study of demulsification by alkyl benzene sulfonic acid (L9), the best results were obtained (Al-Sabagh *et al.* 2016) as water was making half of the volume. According to the findings, the maximum demulsifier efficiency found at a concentration (800 ppm) was 85 %. Biniaz and his team (Biniaz *et al*. 2016) investigated demulsification using ionic liquid statistical modeling and optimization. However, the water made up 7.07 % of the total volume. Demulsifier was made with tri-octyl methylammonium chloride (TOMAC) and efficiency was found100 % at the concentration (1039 ppm) (Biniaz *et al*. 2016).

Moreover, studying the dipoles in poly (ionic liquids) led researchers to discover the demulsifier 2-acrylamido-2-methylpropane sulfonic acid-co-hydroxyethyl methacrylate. Atta and his coworker, (Atta *et al.* 2016) prepared (AMPS-EOA/HEMA) compound. Water count was 10% of the total volume. The result was 90% when the concentration was 1000ppm (Atta *et al.* 2012). In addition, two other demulsifiers were developed in a study on polymer flooding by (Bi *et al.* 2017) benzyl-G3 and octyl-G3. Demulsifier efficiency calculated was 99.3 % and 99.8 % at 400 $mg.L^{-1}$ concentration with in 40 minutes, respectively (Jiang *et al*. 2013, *Bi et al*. 2017). In the study of demulsification by PEI-L230 (xylene/butyl glycol), ethoxylated lauryl acid was used as a non-ionic surfactant (Souza *et al.* 2017). The container held one-quarter gallon of water. The demulsifier efficiency had reached up to 80% after 25 minutes (Souza *et al.* 2017). Using amphiphilic materials derived from natural products, Atta and his coleagues synthesized a new nonionic cardanol compound (DECA). It had a water content of 10% by volume and the result was 100 % at the concentration of ($100mg.L^{-1}$ (Atta *et al.* 2018).

Researchers synthesized and characterized some new Gemini amphiphilic polymeric surfactants by using ethylene diaminetetraacetic acid (Ahmed and Hussain 2018). Five demulsifiers were prepared (HA, HB, HC, HD, and HE) having water content of the

mixture up to 15%. The outcomes were compared to those obtained with a commercial demulsifier (Rp6000). The separation efficiency was 100 %, 93 %, 53 %, 33 %, and 100 % at 100ppm and 30 minutes, respectively (Al-Sabagh *et al.* 2016).

2.7 Critical Micelles Concentration (CMC)

Critical micelle concentration (CMC) is a term used to describe the concentration of a surfactant in a solution at which the surfactant molecules start to spontaneously form micelles. Micelles are formed when surfactant molecules aggregate in a solution to form a structure in which the hydrophobic tails of the surfactant molecules are shielded from the surrounding solvent, while the hydrophilic heads are exposed to the solvent. Moreover, the CMC is the point at which the concentration of surfactant in solution is sufficient to overcome the energy barrier to micelle formation (Ruckenstein and Nagarajan 1981, Porte *et al.* 1984). At concentrations below the CMC, surfactant molecules exist as monomers, while at concentrations above the CMC, surfactant molecules form micelles. Therefore, CMC is an important parameter for understanding the behavior of surfactants in solution, as it affects properties such as surface tension, solubility, and emulsification. Above the CMC, surfactants can form stable monolayers at interfaces, which can reduce the interfacial tension between two immiscible phases. This property is exploited in a wide range of applications, including detergents, emulsifiers, and foaming agents (Lu *et al.*2018). Surface tension begins to decrease as surfactant concentration rises to a certain level, and micelles gathered the surface to achieve the lowest surface tension (Perinelli *et al.* 2020), as shown in Figure 2.8.

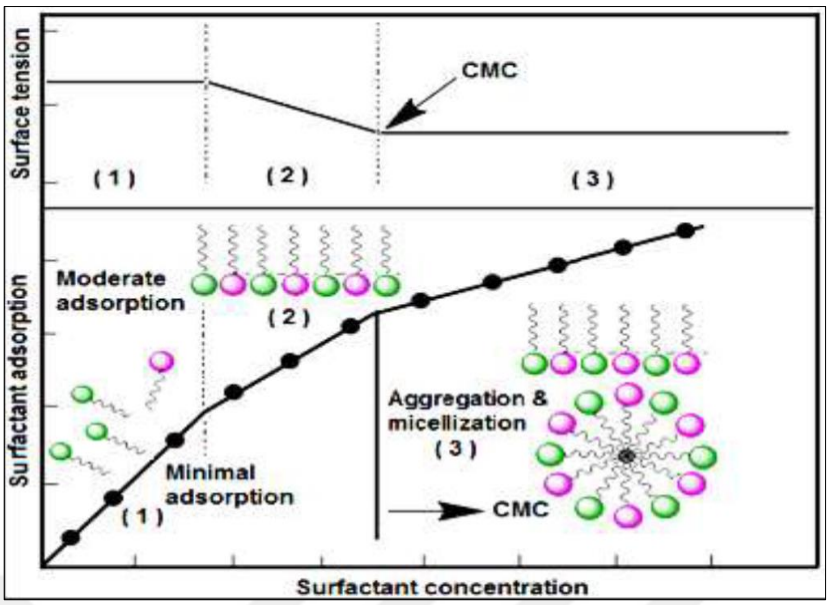

Figure 2.8 Stages of the formation of micelles with surface tension (Perinelli *et al.* 2020)

It is observed that with the addition of surfactant, surface tension increase. In addition, from starting point to the maximum, there was a consistency in increase of amount of surface tensioning force, where at the lowest concentration, no micelles formed. As the concentration of the surfactant is increased, the number of amphiphile gathering at the interface starts to affect the strength of the surface tension. Hence, it attaine a stage where the surface tension become minimal. Which is the point where the micelles are formed. When the surfactant concentration increases, we notice that the surface tensile strength remains constant because the interface becomes filled with amphiphilic molecules and increase in the concentration of surfactant becomes ineffective (Liu *et al.* 2018, Sharma and Mahajan 2014). The critical micelles concentration can be measured by measurement of some characteristic properties by changing many physiological characteristics of liquids with a change of concentration of surfactant such as the conductivity, pressure, viscosity, surface tension or light properties (Serafim *et al.* 2016, Faustino *et al.* 2015).

2.7.1 Formation of critical micelle concerntation

The formation of critical micelle concentration is a result of the balance between the intrmolecular interactions of surfactant molecule and the surrounding solvent molecules. As the concentration of surfactant in solution increase, the hydrophobic groups become more and more shielded by the hydrophilic group, leading to a decrese in free energy of the system. At a certan concentration, the intrmolecular interaction become strong enough to overcome the thermal energy of the system, leading to the spontaneous formation of micelles. On other side, when the temperature of the system exceeds the critical micelle temperature (Krafft temperature) and the concentration of surfactant exceeds the critical micelle concentration (CMC) (Manojlovic., 2012). This concentration is known as the critical micelle concentration. It is possible to comprehend the thermodynamics of micelle formation (Moroi *et al.* 1975). Micelles can form on their own because of a balance between entropy and enthalpy. The hydrophobic effect is what propels the formation of micelles in water, despite the fact that assembly of surfactant molecules is unfavorable for the system's enthalpy and entropy. At very low surfactant concentrations, the solution only contains monomers. An increase in entropy caused the release of the solvation shells around the surfactant tails, offsets the unfavorable entropy contribution from clustering the molecules hydrophobic tails. Above the CMC, the concentration of surfactant in the solution is sufficient to stabilize the micelles and maintain them in solution. The size and shape of the micelles depend on various factors, such as the surfactant structure, temperature, pH, and the presence of other solutes in the solution. Micelles can have a variety of applications, such as in detergents, emulsifiers, solubilizers, and drug delivery (Leibler *et al*., 1983, Lo, Chun-liang *et al*., 2009, Maibaum *et al*., 2004).

2.7.2 Application of critical micelle concerntation

When surfactants are present above the critical micelle concentration, they have the capacity to act as emulsifiers, detergent, stabilizer in nanoparticle synthesis, drug delivery agent and allowing a substance that is typically insoluble (in the solvent being used) to dissolve (CMC). Moreover, the critical micelle concentration (CMC) has a

wide range of applications in various fields, including chemistry, pharmacy, and materials science. CMC is an important parameter in the design and formulation of detergents and cleaning agents. Surfactants with lower CMC values are more effective in cleaning and emulsifying oils and other hydrophobic substances (Perez-Rodriguez *et al.* 1998). The formation of stable emulsions requires the use of surfactants at concentrations above the CMC. By stabilizing the interface between two immiscible liquids, surfactants can prevent the coalescence of droplets and maintain the stability of the emulsion. Moreover, CMC is a crucial factor in the synthesis of nanoparticles by microemulsion techniques. The formation of micelles can serve as nanoreactors, allowing for the controlled growth and stabilization of nanoparticlesIt is an important factor in the solubilization and purification of biomolecules, such as proteins and DNA. By using surfactants above the CMC, the solubility of hydrophobic biomolecules can be enhanced, leading to improved purification and analysis (Blankart *et al.* 2022, Klevens., 1948). The most common example of this phenomenon is detergents, which remove poorly soluble lipophilic material (like oils and waxes) that cannot be removed by water alone. Detergents also lower the surface tension of water to remove dirt from a surface. The emulsifying properties of surfactants support emulsion polymerization as well. Micelles may also be important players in chemical processes. Micelles' interiors are used to house chemical reactions in micellar chemistry, which can sometimes make multi-step chemical synthesis more practical (Paprocki *et al.* 2018, Pasquali *et al*. 2008). By doing this, it is possible to increase reaction yield, create conditions that are better suited for specific reaction products (like hydrophobic molecules), and generally use fewer solvents, side products, and conditions (e.g. extreme pH). Because of these benefits, micellular chemistry is known as a type of green chemistry (Macquarrie 2009). . İn addition, designing of drug delivery systems that also use surfactants to enhance the solubility and bioavailability of drugs. By controlling the concentration of surfactants above the CMC, drug molecules can be encapsulated in micelles or liposomes, leading to improved drug delivery and targeting. Micelle formation, for instance, can also stop chemical reactions, when reacting molecules shield a molecular component from oxidation (Ji *et al.* 2022). For the body to absorb complex lipids and fat-soluble vitamins, micelle formation is required. Bile salts, which are made in the liver and secreted by the gall bladder, allow fatty acid to form micelles. As a result, the micelle's

complex of lipids like lecithin and lipid-soluble vitamins like A, D, E, and K can be absorbed by the small intestine. During the milk clotting process, proteases interact with the soluble casein, -casein, to produce an unstable micellar state that results in clot formation. Micelles can also be used for targeted drug delivery, like gold nanoparticles (Chen *et al*. 2008).

2.8 Balance Between Hydrophilic and Lipophilic Molecules (HLB)

The Hydrophilic-Lipophilic Balance (HLB) is a concept that is used to describe the relative solubility of a surfactant in water (hydrophilic) and oil (lipophilic) phases. The HLB value of a surfactant is a measure of the balance between its hydrophilic and lipophilic properties and is used to predict its emulsifying properties. The HLB value of a surfactant is a numerical value that ranges from 0 to 20, with lower values indicating a more lipophilic (oil-loving) surfactant and higher values indicating a more hydrophilic (water-loving) surfactant. For example, surfactants with an HLB value of 1-6 are considered lipophilic and are better suited for oil-in-water emulsions, while surfactants with an HLB value of 7-14 are considered hydrophilic and are better suited for water-in-oil emulsions. Surfactants with an HLB value of 15-20 are highly hydrophilic and are typically used as emulsifying agents for water-based formulations (Pasquali *et al.* 2008, Ontiveros *et al.* 2014, Lu, shenzhou *et al.* 2015). The HLB value can be calculated based on the chemical structure of the surfactant, particularly the balance between the size and polarity of the hydrophilic and lipophilic groups. A common method for determining the HLB value of a surfactant is the Griffin method, which involves calculating the ratio of hydrophilic groups to lipophilic groups in the surfactant molecule. It's a mathematical method for calculating or valuing surfactants based on their chemical composition, so that the maximum performance for desired application can be achieved (Pasquali *et al.* 2008, Gadhave 2014). A specific ranges for surfactents are given in Table 2.2 to find out the behavioural property of surfactant (Severino *et al.* 2011, McCrary *et al.* 2013).

Table 2.2 HLB values and nature of polymer (Severino *et al*. 2011, McCrary *et al*. 2013).

HLB	**Applications**	
1-3	Antifoaming agents	0 lipophilic ↑ ↓ hydrophilic 20
3-8	(W/O) - emulsion	
7-9	Wetting agents	
8-16	(O/W) - emulsion	
13-16	Detergents	
16-18	Solubilizing agents	

Various mathematical and scientific methods can be used to determine the best surfactant for the application. Some mathematical laws such as Griffin William equation is given below Equation 2.2 to find out the HLB value of nonionic surfactants.

$$HLB=20(1–S\backslash A) \qquad (2.2)$$

Where: S = The ester's saponification number, A = Represents the acid number.

Moreover, the HLB value can be calculated when there are less esters and have good saponification number data from the Griffin William Equation (2.3).

$$HLB=E+P\backslash 5 \qquad (2.3)$$

Where: E= weighted ratio of oxy ethylene series, and P= weighted ratio of alcohol poly hydroxyl.

2.9 Corrosion

Corrosion is the process of metal dissolution brought on by a chemical or electrical reaction between the metal and the medium in which it is present. It typically happens when there is a lot of oxygen and moisture. When electrons are rearranged between the

metal surface and the electrolyte solution, a process known as corrosion takes place (Faustino *et al.* 2015). Anode oxidation reactions and cathode reduction reactions are the two types of electrochemical reactions.

To be aqueous corrosion, the following four components are required:

A. Anode: It is the metal that is exposed to corrosion. The oxidation process is done by losing the metal electrons like iron dissolution, as in the Equation (2.5) (Szczerba 2010).

$$M \longrightarrow M^n + ne^- \qquad (2.5)$$

Where, M= corroded metal, and n= number of lost electrons

B. Cathode: It is a part of the metal that receives the released electrons from the anode. Reduction occurs on the cathode electrode in two ways, and when oxygen is present, the hydroxyl is formed as in the Equation (2.6).

$$O_2 + 4e^- + 2H_2O \longrightarrow 4OH^- \qquad (2.6)$$

In the absence of oxygen, hydrogen gas released as shown in the Equation (2.7).

$$2H^+ + 2e^- \longrightarrow H_2 \qquad (2.7)$$

C. Electrolyte: It is the solution that is put inside the piece of metal that is exposed to corrosion; also, electrons and ions formed are transmitted in it.

D. Connecting wire: It is used to complete the electrical circuit, where the anode is attached to the cathode by a conductive wire, as they are separate such as corrosion of iron in water as shown in the following Figure 2.9. in addition, the Equation (2.8) describes the anodic reaction, Equation (2.9) describes the cathodic reaction, and

Equations (2.10), (2.11), and (2.12) describe the corrosion of iron in a solution of hydrochloric acid.

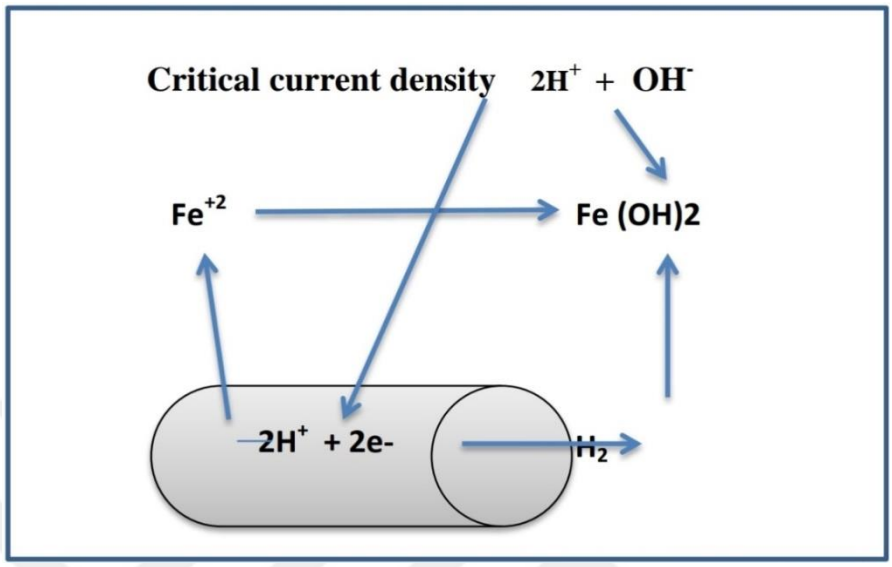

Figure 2.9 The schematical representation of corrosion of iron in water (Faustino *et al.* 2015)

$$Fe \longrightarrow Fe^{+2}+2e^{-} \qquad (2.8)$$

with the oxygen

$$2H+2e^{-} \longrightarrow H_2 \qquad (2.9)$$

without oxygen

$$\tfrac{1}{2}O_2+H_2O \longrightarrow 2OH^{-} \qquad (2.10)$$

$$Fe^{+2}+2Cl \longrightarrow FeCl_2 \text{ (unstable)} \qquad (2.11)$$

$$FeCl_2+2H_2O \longrightarrow Fe(OH)2+2HCl \qquad (2.12)$$

2.9.1 Type of corrosion

Corrosion can be classified according to the appearance of metal that is affected into several groups. Therefore, corrosion has been devided into multiple types on the base of

there affect on the surface of metal. Such as, uniform corrosion, galvanic corrosion, crevice corrosion, pitting corrosion, intergranular corrosion, stress corrosion and erosion-corrosion (Sastri 2014). Uniform corrosion occure when the rate of corrosion on metal surface is uniform, while galvenic corrosion take place when two different metals are in contact in the presence of an electrolyte, such as saltwater. The flow of electric current causes the more active metal to corrode. However, cervice corrosion occure in small cervices or gaps where stagnant solution accumulates and become corrosive. On the other side, pitting corrosion take place when localized area of a metal's surface damaged resulting in small pits. Similarly other types of corrosion occur when metal exposed to a corrosive enviornemt leading to crack or mechanical erosion or abrasion, having impurities or change in composion of metal as shown in the following Figure 2.10 (Sastri 2014).

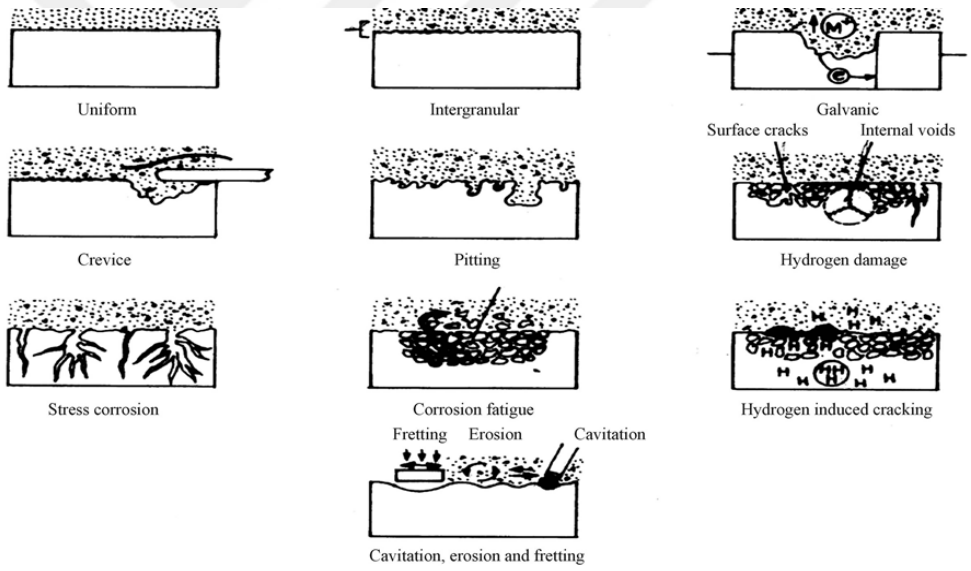

Figure 2.10 Types of corrosion (Surfaceprotech.com).

2.9.2 Methods of corrosion

A. Biologic corrosion

The degradation of metal or non-metal materials brought on by the presence of microorganisms in water, wastewater, crude oil or the interior walls of piping is referred to as biological corrosion. The presence of bacteria in the form of biofilm that adheres to the interior surfaces of piping systems frequently causes this type of corrosion (Edyvean and Videla., 1991). Also known as microbiological corrosion or biological corrosion. The primary cause of biological corrosion is the production of hydrogen sulfide gases by naturally occurring microorganisms found in water, wastewater and crude oil. The sulfide gasses react with air and moisture when they reach the surface to create sulfuric acid, which is extremely corrosive to both metal and non-metal materials. On the surface of inner walls of piping systems, biological corrosion is typically visible as pitting or sulfide stress cracking. The most typical techniques for preventing this kind of corrosion include, regular upkeep or cleaning, using corrosion inhibitors designed to inhibit bacterial growth (such as benzalkonium chloride) and preventing stagnation by ensuring that the system is properly drained (Fontana and Stactile., 1970, stanaszek-Tomal and Fiertak., 2016).

B. Physical corrosion

This happens when the metal reacts with the oxygen in the air or corrodes in it. The end result is a thin oxide film that prevents the metal from continuing to react. Examples of this include the weathering of some sculpture materials and the patina that develops on copper (Bazant and Zdenek., 1979, Schweitzer 2003).

C. Chemical corrosion

Chemical corrosion is the name given to the gradual breakdown of a metal's surface brought on by the surface's interaction with elements in its environment (Cicek and al-

Numan 2011). It is frequently characterized by the formation of oxides, when metal oxidizes in the presence of an acid. When a certain metal is under stress or is deprived of airflow, chemical corrosion starts to occur. Especially in a moist environment, the metal ions consequently start to dissolve, hastening the reaction between the metal ions and the water. When free ions and hydrous oxides react with iron, leads to corrosion known as rust. This method is done by weight of the sample before being exposed to a corrosion medium (Mansfeld 1986, Kondo *et al*. 2000, Voorhies 1972). After that, it's immersed in the corrosion medium for a while. Then the sample is taken, and corrosion products are removed by chemical methods and weighed; the difference in weight represents the lost weight obtained after completion of corrosion process (Jomdecha *et al.* 2007(Xiang *et al*. 2011). The Equation (2.13) can be used to calculate the corrosion speed.

$$R_{corr} = \frac{\Delta W}{S.corr.t} \qquad (2.13)$$

Where: R_{corr} = The corrosion speed ($gm.cm^{-2}.hr^{-1}$), ΔW = Lost weight (g), S_{corr} = Surface area (cm^2) and t = Time of sample stay in corrosion medium (hr).

2.9.3 Factors affecting corrosion

Several factors can affect the rate and extend of corrosion, including environmental condition, properties of metal, pH value, temperature, mechanical stress, galvenic effect and oxygen contents. Presence of gases in the air, such as CO_2, SO_2, SO_3, water especially salt water or when metal exposed to moisture, increases the rate of corrosion. Moreover, it was found that, corrosion accelerate by rising temperatures. Some oxides, like Al_2O_3, create an impenetrable barrier that can halt further corrosion. Moreover, mechanical stress, such as tension, compression or bending can increase the likelihood of stress corrosion cracking (Selwyn 2004, Sereda 1974, Glass 1991).

2.10 Mechanism of Inhibition

Corrosion inhibition is the process of using chemicals to prevent or reduce the rate of corrosion. Corrosion inhibitors work by forming a protective layer on the metal surface, which prevents the metal from coming into contact with corrosive agents. The protective layer can be formed through various mechanisms, such as passivation, adsorption, or film formation. There are two main types of corrosion inhibitors: organic and inorganic. Organic inhibitors are typically organic compounds, such as amines or organic acids, that adsorb onto the metal surface and form a protective layer. Inorganic inhibitors are typically metal ions, such as chromates or phosphates, that react with the metal surface to form a protective film (Sancy *et al.* 2010, Yasakau *et al*. 2006, Neufeld *et al.* 2002). Corrosion inhibitors can be added to the environment surrounding the metal, such as in cooling water systems or in oil and gas pipelines, or they can be applied directly to the metal surface as a coating or paint. Corrosion inhibitors are often used in combination with other corrosion prevention methods, such as protective coatings or cathodic protection, to provide additional protection against corrosion. However, the effectiveness of a corrosion inhibitor depends on several factors, such as the type of metal, the corrosive environment, and the concentration and type of inhibitor used. Proper selection and application of a corrosion inhibitor requires a thorough understanding of the factors affecting corrosion and the mechanisms by which the inhibitor Works (Sancy *et al.* 2010, Yasakau *et al*. 2006, Neufeld *et al.* 2002). The first venue in the method of surfactant work as a corrosion inhibitor is the absorption of amphiphilic molecules on the metal surface, as shown in the Equation (2.14).

$$\text{Nonionic[sol]}+n\text{H}_2\text{O[ads]}\text{Nonionic[ads]}+n\text{H}_2\text{O[sol]} \qquad (2.14)$$

Where (n) is the quantity of water molecules that are removed from the metal surface by each adsorption surfactant molecule.

However, absorbtion of solvent molecules to the metal surface, preventing corrosion. Because the energy of reaction for amphiphilic molecules and intermetallics is greater than the energy of water molecules and intermetallics (Xiang *et al*. 2011), Tian *et al.*

2017). The effectiveness of corrosion inhibitor materials is greatly influenced by the concentration of critical micelles. The prevention of corrosion is the most critical process, especially in industrial applications. It is possible to find successful ways to prevent decay depending on the type of metal that cause corrosion. Generaly, many techniques have been used for protectioning of metal from corrosion are elaborated below (Heselmans *et al.* 2013, Shreir 2013).

A. Protection of metal by designing or shaping the surface

Corrosion can be controlled by the appropriate choice of material in the working environment and also a selection of good engineering designs. Therefore, the use of different metals should be avoided for friction purposes with the electrolyte. It can be caused by galvanic corrosion (Córdoba *et al.* 2016).

B. Chemical coating

There are multiple types, including metallic coating, which was based on the mixing of the resistant material in coating between the metal and the corrosion medium, such as iron coating with zinc. Moreovere, the organic coating works to insolates the metal from the corrosive environment by making an insulating layer. It contains corrosion inhibitors such as resin, plastics and rubber, as well as an inorganic coating like ceramic coatings or silica. However, chemical conversion coating is an improvement in the metal surface to develop resistance against corrosion with chemical treatments of a protective layer composed of a mixture of chromium oxides, metal oxides and phosphate in the steel coating (Heselmans *et al.* 2013, Shreir 2013).

C. Anodic and cathodic protection

Anodic protection involves making the metal surface the anode in an electrochemical cell. This is done by applying a current to the metal surface that is positive with respect to the surrounding environment. As a result, the metal becomes the anode and corrodes

preferentially, protecting other metals surf ace in the same environment. Anodic protection can be used to protect metals such as copper, nickel, and aluminum in acidic environments. İn addition, the prevention of cathode is the most important way. It depends on making the material be protected as a cathode in an electric cell by applying a direct electrical current or galvanic cell, connecting an active electrode material, which work as an anode (Urquidi-Macdonald and Macdonald 2014).On the other side, Cathodic protection involves making the metal surface the cathode in an electrochemical cell. This is done by applying a current to the metal surface that is negative with respect to the surrounding environment. As a result, the metal becomes the cathode and is protected from corrosion by the more reactive anodic metal in the same environment. Cathodic protection can be used to protect metals such as steel and iron in environments such as seawater. Anticorrosive materials are often used in both anodic and cathodic protection methods. These materials can be applied to the metal surface as coatings or paints, or they can be used as sacrificial anodes. Sacrificial anodes are made of more reactive metals, such as zinc or magnesium, and are connected to the metal to be protected. The sacrificial anode corrodes preferentially, protecting the metal from corrosion. While anodic protection relies on exposing the metal to a current that results in a configured protective layer on the metal surface, reducing the rate of corrosion. However, it is not a widely used method, because of risk of corrosion. As the the protective layer collapsing under the influence of chloride ions. (Hussain and Ishida 2011).

2.11 Inhibitors

Inhibitors are organic or inorganic additives that are added to a reaction in minimal amounts, up to part per million (ppm), to reduce the rate of corrosion and metal loss (Tian *et al.* 2017, Urquidi-Macdonald and Macdonald 2014). In addition, inhibitors are categorized into four different groups; such as organic inhibitors, inorganic inhibitors, surfactant inhibitors, and Inhibitors of mixed materials. Moreover, surfactants used in mixture solution are the most effective, low in cost, having low toxicity level, very simple to manufacture and have high rate of coorosion preventions (Shreir 2013).

3 . MATERIALS AND METHODS

3.1 Chemicals and Reagents

All the chemical materials used in this study were of analytical grade and of high purity. The list of chemicals is given below in Table 3.1.

Table 3.1 List of chemicals and reagents with theiry purity and manufacturers

Chemical Materials	**Purity**	**The Manufacturer**
Thionyl chloride	99.5%	CDH
Ethylene di amine tetra acetic acid(EDTA)	99%	BDH
Naphthalic Anhydride	99%	Sigma
N,N-Methylenebis(Acryl amide)	99%	Sigma
Acrylamide (2-Popenamide, Acrylic Acid Amide	99%	Sigma
Di Methyl For Amine (DMF)	99%	CDH
Acetone	99%	GCC
Toluene	99.9%	GCC
Ethanol	99.9%	GCC
HCL	99.9	TB
$AgNO_3$		CDH
Xylene	99.9	GCC

3.2 The Instruments and Equipments

Tabe 3.2 List of Instruments and equipments used in this study.

No.	Instruments	Company
1	Sensitive balance	EXACTA
2	Thermo-Circulator	LAB TECH COMPANY
3	Hotplate stirrer	LAB TECH COMPANY
4	Conductivity meter	HANNA
5	Water bath	GEMMYCO
6	Digital viscometer	BROOKFIELD LVDVE
7	Fourier transform infrared spectroscopy (FT-IR)	THERMO
8	Density device	RULOPH
9	Salt device	SETA
10	Water device	AQUA
11	Sulfur device	HORIBA
12	Asphalten device	COSMO
13	Viscosity device	OMNITEK
14	Reid vapour pressure device	ERALYTICS
15	H2s dissolved in crude oil device	EHC
16	Refractive index device	RUDOLPH
17	Water and sediment device	SIGMA
18	HNMR Proton nuclear magnetic resonance	BRUKER

3.3 Preparation of Stock Solution of EDTA

0.5g (0.0017 moles) of EDTA was dissolved in 20 ml of dimethyl formamide (DMF) and reflux the solution at 60 °C for 1 hour. This stock solution was further used in the synthesis process of surfactents.

3.4 Preparation of Ethylenediaminetetraacyle Chloride

For the synthesis of ethylene diamintetra acetic acid (0.0068 moles, 0.49 mL) of thionyl chloride was taken in one nick round size (100 ml) botel. From already prepared stock solution (0.24ml) of EDTA (0.017 mole 0.5g) was added into the thionyl chloride solution. after mixing both solution at magnetic stirrer, reflex them at 80 °C for 4 hours. After completion of reflux process, thin layer chromatography (TLC) technique was applied. The precipitate of final product was filtered and washed with ethanol several time to remove unreacted content. the obtained precipitates were recrystallized with ethanol. A white powder was obtained, and achieve the yield of reaction upto 97.2 %, as shown in the Figure 3.1.

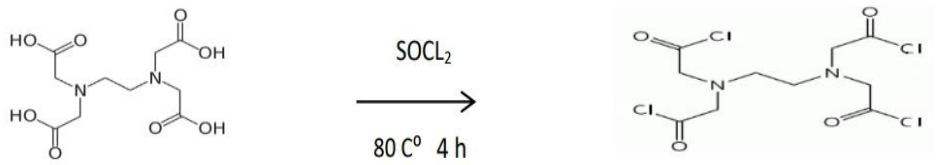

Figure 3.1 Preparation of ethylene diaminetetra acyle chloride.

3.5 Synthesis of Polymeric Surfactant (F1)

For the synthesis of surfactant (F1), already prepared 15 ml of ethylene diaminetetra acyle chloride 0.5g (0.0013 moles) was taken in round bottom flask. 0.85g (0.0055 moles) from N, N-methyl bis acrylamide was heated in 10 ml of DMF and mixed in EDTA solution. mixture of both solutions was refluxed at of 60°C for 3 hours. The reaction was monitored using TLC, and obtained precipitates were filtered and washed

with ethanol. The precipitate of final product recrystallized with ethanol. A white powder obtained weighed as 1.75g. schematic representation of synthesis shoen in Figure 3.2.

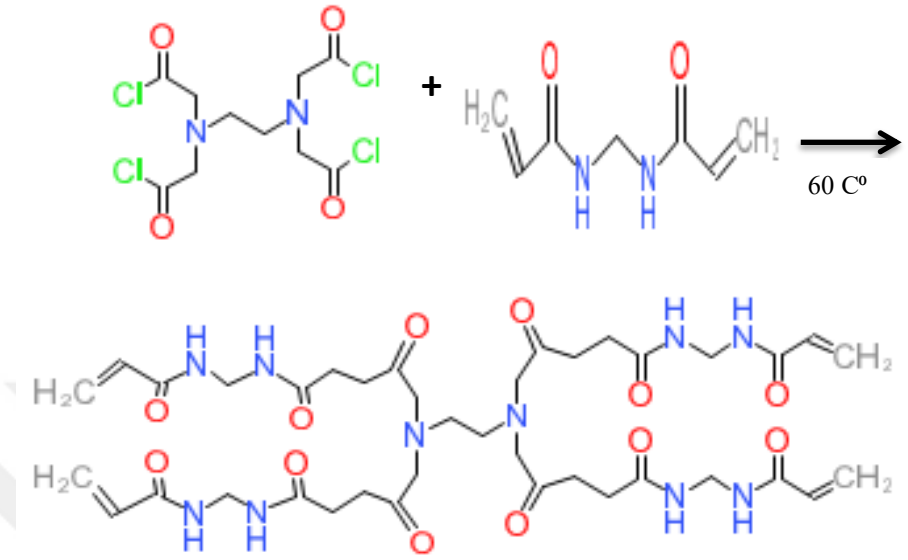

Figure 3.2 Synthesis of polymeric surfactant (F1)

3.6 Synthesis of Polymeric Surfactant (F2)

For the synthesis of surfactant (F2), 15 ml of ethylene diaminetetra acyle chloride was taken in round bottom flask 0.5g (0.0013 moles) into round bottom 100 ml of flask. 0.39g (0.0055 mole) from 2-propenamide (acrylamide) was melted in 10 ml of DMF and added into ethylene diaminetetra acyle chloride solution. both solutions were mixed well and refluxed them at 60C° for 3 hours. The reaction was monitored using TLC, and the precipitates were filtered and washed with ethanol. The obtained precipitates were recrystallized with ethanol. A white powder obtained around 1.75g, schematic representation of reaction shwn in Figure 3.3.

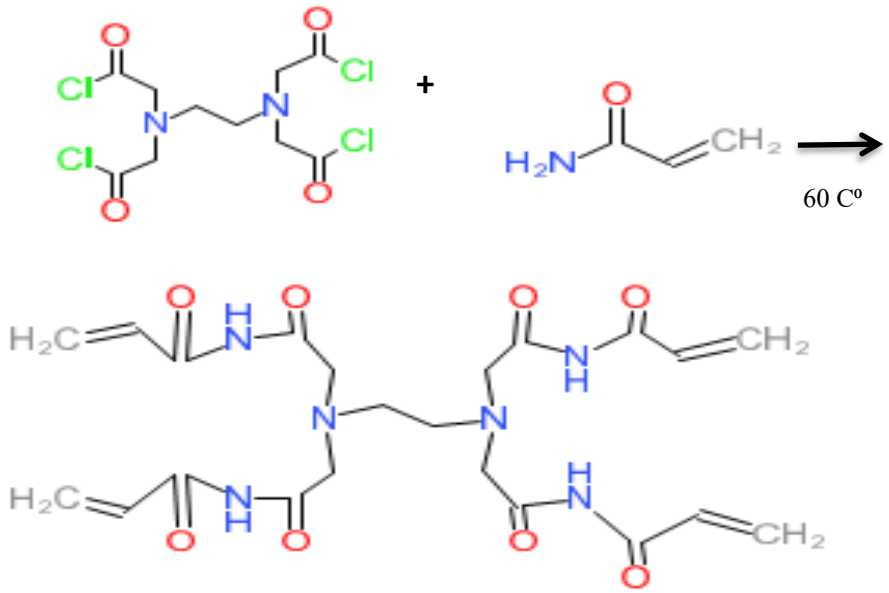

Figure 3.3 Synthesis of of polymeric surfactant (F2)

3.7 Synthesis of Polymeric Surfactant (F3)

For the synthesis of surfactant (F3) 0.5g of (0.0029 moles) from naphthalic acid was added in 100 ml of round bottom flask. Then 15 ml of solvents (DMF) was added to the naphtalic acid containing flask. Added 0.44g of (0.0029 moles) N, N-methyl bis acrylamide after melted it in 10 ml of DMF. Then mixture was reflexed at a temperature of 60°C for 3 hours. The reaction was monitored using TLC, and the precipitate was filtered and washed with ethanol. Then obtained precipitates were recrystallized with ethanol. A 1.75g of white powder product obtained, schematic representation of synthesis of F3 surfactant shown in Figure 3.4.

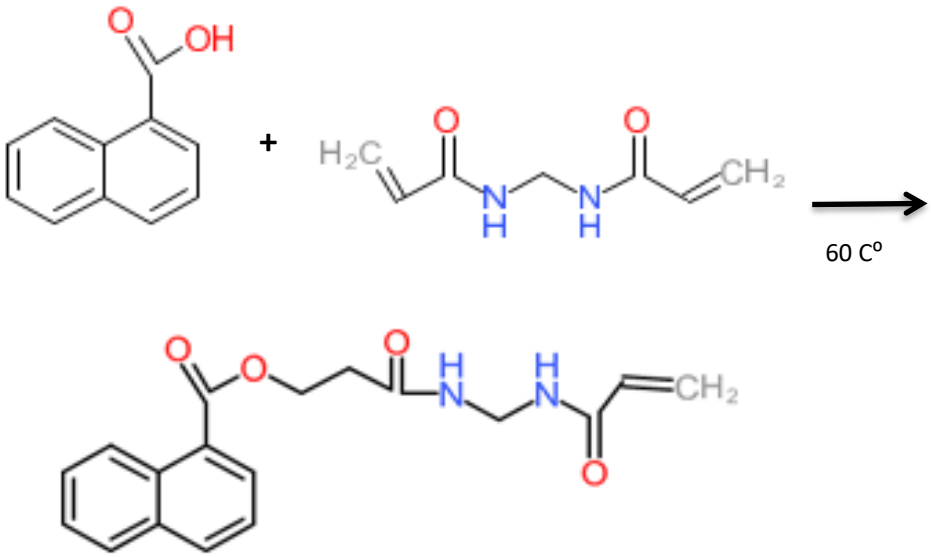

Figure 3.4 Synthesis of polymeric surfactant (F3)

3.8 Synthesis of Polymeric Surfactant (F4)

For the synthesis of polimeric surfactant (F4), 0.5g of (0.0029 moles) Naphthalic acid was added into 100 ml of round bottom flask and dissolved into 15 ml of DMF. 0.2g (0.0029 mole) from 2-propenamide (acrylamide) was added in molten form in 10ml of DMF. The reactants were reflexed at 60 ^{o}C for 3 hours. The reaction was monitored using TLC, and the precipitate was washed after filteration in small batches with ethanol. They were recrystallized with ethanol. A white powdered product obtained was weighed as 1.75g. the process of synthesis of F4 polimeric surfactant is shown in Figure 3.5.

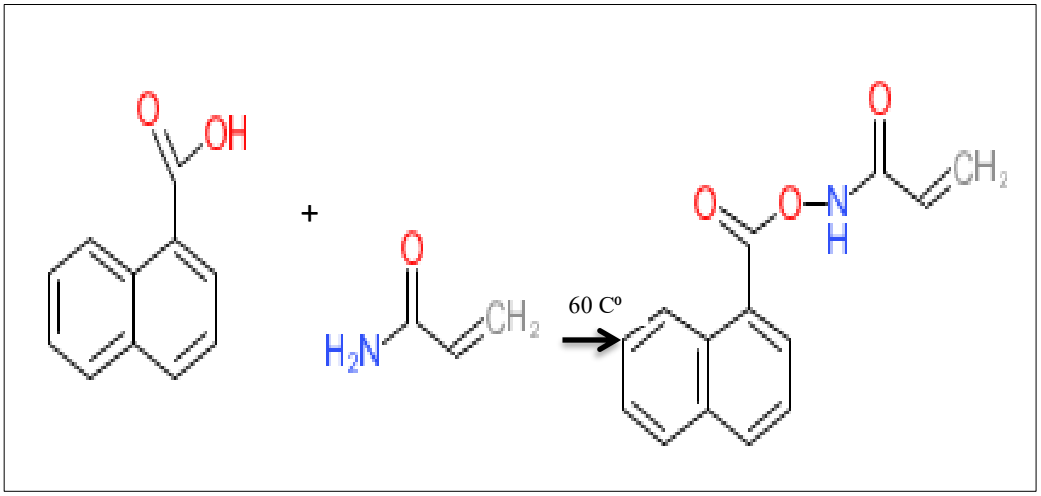

Figure 3.5 Synthesis of polymeric surfactant (F4)

3.9 Demulsification Process

A. Preparation of wet crude oil in a laboratory

For the preparation of wet crude oil, 15% of distilled water and 85% of dry oil was combined at laboratory scale in a mechanical stirrer for two hours (Feng *et al.* 2012).

B. Preparation of demulsifiers

For the preparation of demulsifier, four different F1, F2, F3, and F4 polimeric surfactant were used. A mixture of polymeric surfactant and crude oil (20g: 80mL) ratio was mixed, and then divided into smaller proportions containing (0.2g: 0.8mL) of both. The components were melted in (0.8mL) toluene solvent with 0.1g of surfactant, 0.05g of Castrol oil, and 0.05g of silver nitrate. The mixture was then used to make a good laboratory demulsifier (Abdulbari *et al*. 2011, Kailey and Feng 2013).

C. Measurment of separation efficiency of prepared demulsifiers

For the measurement of separation of demulsifier, Karl Fisher's method of separation was applied. A 0.02 ml of already prepared demulsifire was poured into 10 ml of test

tube. Following that, the samples are submerged in a 45°C of water bath. After (20, 40, 60, 80, 100 and 120 minutes), the volume of separated water was measured (Fang *et al.* 2017).

D. Preparation of Anti-Corrosion Solution

The (0.5M) hydrochloric acid solution was used to make solutions of (10, 20, 30, 40) ppm for each of the surfactants (F1, F2, and F4) respectively.

E. Study of the corrosion speed (The weight loss method)

To investigate weight loss due to corrosion a (carbon steel) alloy models were cut into square shapes with dimensions of (3, 3, 0.2) cm by length, width, and thickness, respectively. The models were washed several times with distilled water and ethanol, and dried to remove moisture (Migahed *et al.* 2018, Nuer *et al.* 2020). The models were then refined using calcium carbide papers with cold water to avoid high temperatures to remov the layer of oxides from the metal surface, as per standard specifications.

A four decimal mattresses (Malik *et al.* 2011, Nuer *et al.* 2020), models were weighted using a sensitive balance. For an hour, the models were immersed in a corrosion medium (0.5M hydrochloric acid solution). After removing the corrosive part on the surface with a piece of cotton, the models were cleaned. After cleaning and drying, the models were weighed and calculated the corrosion rate by following the given equation below, shown in Equation (3.1) (Nuer *et al.* 2020).

$$R\text{corr}=\frac{\Delta W}{S.corr\times t} \qquad (3.1)$$

Where: R_{corr} = corrosion speed ($gm.cm^{-2}.hr^{-1}$) = weight loss (g), S_{corr} = surface space for the model (cm^2), t = time (hr), g- Restart steps (a-f) after adding anti-corrosion (surfactant) concentration of (10, 20, 30, 40) ppm and h- Restart steps (a-g) at 2 hours and 3 hours.

3.10 Using Electric Conductivity, Calculation of CMC of Prepared Polymeric Surfactants

At a temperature of (25C°), the conductivity cell was calibrated with (0.1M) potassium chloride (KCl). The surfactants were then concentrated in series using distilled water. These solutions electric conductivity was measured in the lab at room temperature, and the CMC values are listed in the Table 4.8 (Fuguet *et al.* 2005, Scholz, Behnke, and Resch-Genger 2018).

3.11 The Molecular Weight of Prepared Polymeric Surfactants was Determined Using Their Viscosity

A viscometer was first used to measure viscosity and the time it took for the water to descend as a solvent. The descent time for each polymer was then determined using a series solution. The viscosity was calculated and compared to digital viscometer measurements. In chapter three, the (Mark-Houwink) equation was then applied to the resulting values, as shown in Tables 4.7 (KamelAl-Khalidi and Al-Lami 2017, *Gupta et al.* 2005).

4 . RESULTS AND DISCUSSION

4.1 FT-IR Spectra of Prepared Materials

Fourier Transform Infrared Spectroscopy (FT-IR) tests were measured in the range of 4000 to 400 cm^{-1} to find out the available functional groups to determine the compositions of prepared EDTA-DA and polymeric surfactants (F1, F2, F3, and F4) (Pavia *et al.* 2014) as shown in Table 4.1.

4.1.1 Fourier transform infrared spectroscopy (FT-IR) of prepared (EDTA-TETRA CL)

FT-IR of the polymer (EDTA-TETRA CL) shown amine bands (NH-C=O) at (3400 cm^{-1}) and gives a (CH-Ar) peak at 3014 cm^{-1}. A (CH- alpha) peakes are shown at 2794 and 2851 cm^{-1}, two stretching bands for (C=O) at 1694 cm^{-1} and (C=C) at 1410 cm^{-1} as shown in Figure 4.1.

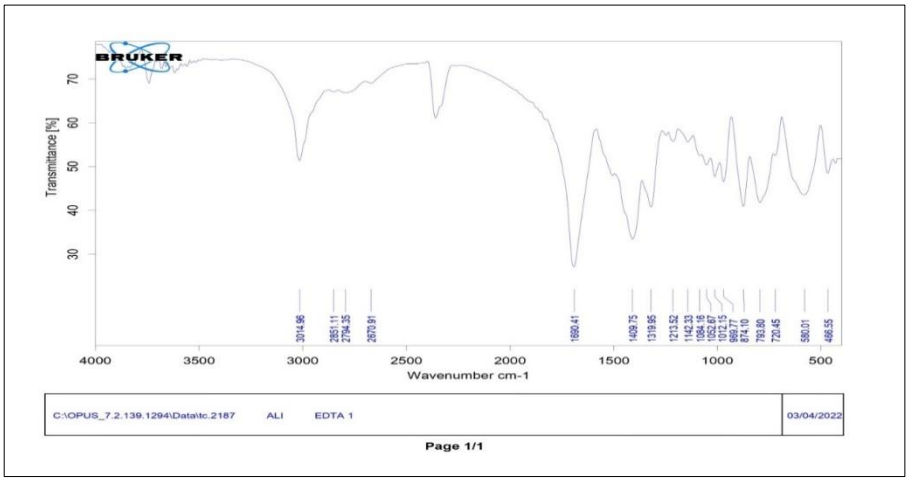

Figure 4.1 FT-IR spectrum of EDTA-TETRA CL

4.1.2 Fourier transform infrared spectroscopy (FT-IR) of polymeric surfactant (F1)

FT-IR of the polymer (F1) shown in Figure 4.2 have amine bands (NH-C=O) at 3400 cm^{-1} and give a CH-Ar peak at 3016 cm^{-1}. Moreover, CH- alpha peaks are observed at 2795 and 2669 cm^{-1}, two stretching bands for (C=O) at 1694 cm^{-1}, and (C=C) at 1410 cm^{-1}.

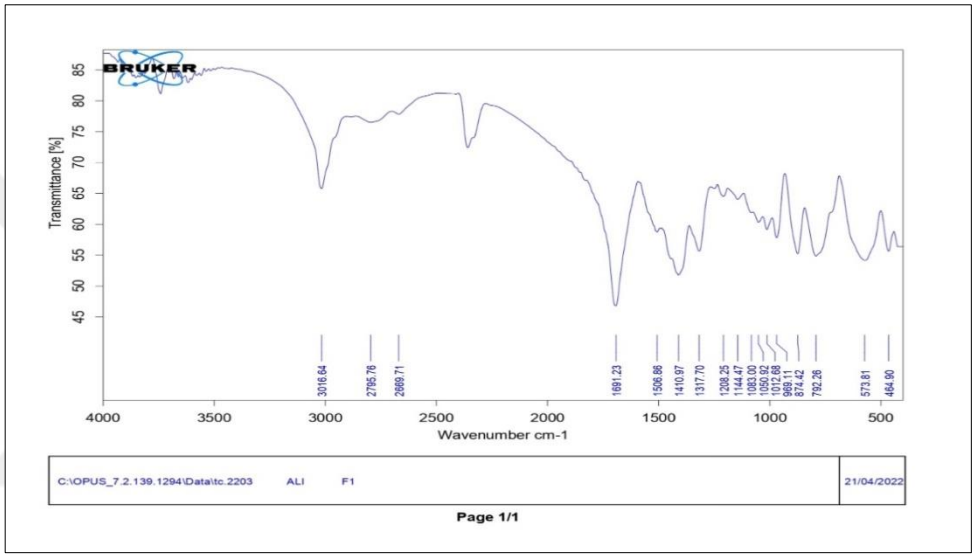

Figure 4.2 FT-IR spectrum of polymer (F1)

4.1.3 Fourier fransform infrared spectroscopy (FT-IR) of polymeric surfactant (F2)

FT-IR spectra of polymer (F2) as shown in Figure 4.3, the peak of amine bands (NH-C=O) was observed at 3400 cm^{-1}, while peak belong to CH-Ar-HC=CH was observed at 3018 cm^{-1}. The CH- alpha peak was observed at 2893 and 2800 cm^{-1}, two stretching bands for (C=O) were observed at 1694 cm^{-1} and (C=C) at 1447 cm^{-1}.

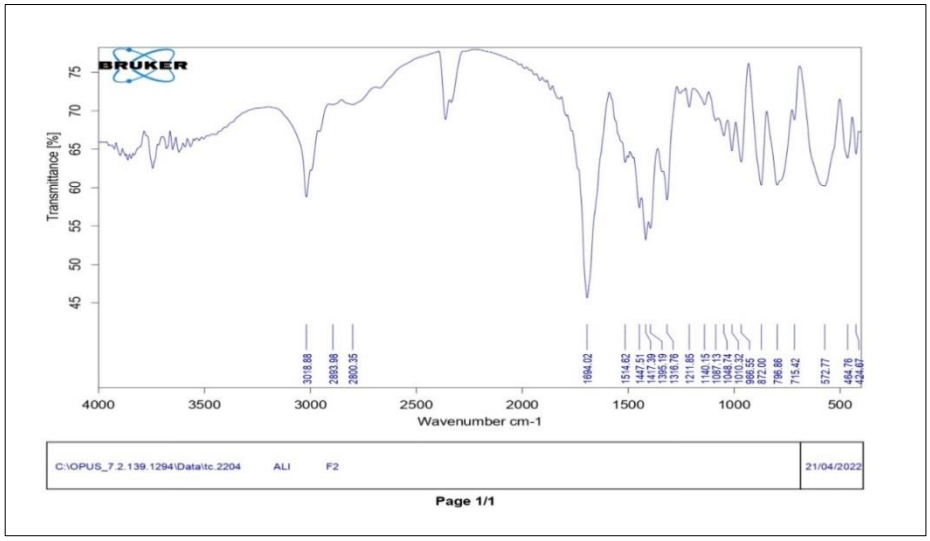

Figure 4.3 FT-IR spectrum of polymer (F2)

4.1.4 Fourier transform infrared spectroscopy (FT-IR) of polymeric surfactant (F3)

FT-IR of the polymer (F3) shown in Figure 4.4 showd band belongs to amine group (NH-C=O) at 3700 cm^{-1} and (CH-Ar) at 3065 cm^{-1}. Moreover, two stretching bands belongs to (C=O) at 1769 and 1738 cm^{-1} and (C=C) at 1575 cm^{-1} and asymmetric vibration of (C-O-C) at 1338 cm^{-1}. In addition, a symmetric vibration of (C-O-C) was observed at 1070 cm^{-1} and for phanel at 841 cm^{-1}.

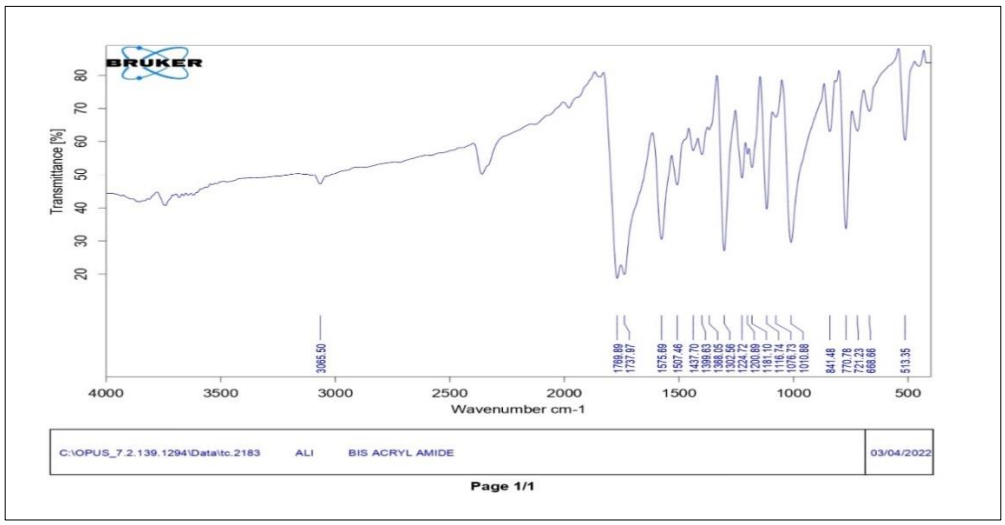

Figure 4.4 FT-IR spectrum of polymer (F3)

4.1.5 Fourier transform infrared spectroscopy (FT-IR) of polymeric surfactant (F4)

FT-IR spectra of the polymer (F4) shown in Figure 4.5 give spectral amine bands (NH-C=O) at 3700 cm^{-1} and aromatic C-H stretching (CH-Ar) at 3065 cm^{-1}. Moreover, two stretching bands for (C=O) was observed at 1769 and 1738cm^{-1} and (C=C) at 1574 and 1506 cm^{-1}. A asymmetric streatching of (C-O-N) and symmetric streaching of same group was observed at 1301 cm^{-1} and 1115 cm^{-1}respectively, and a peak for phanel was observed at 839 cm^{-1}.

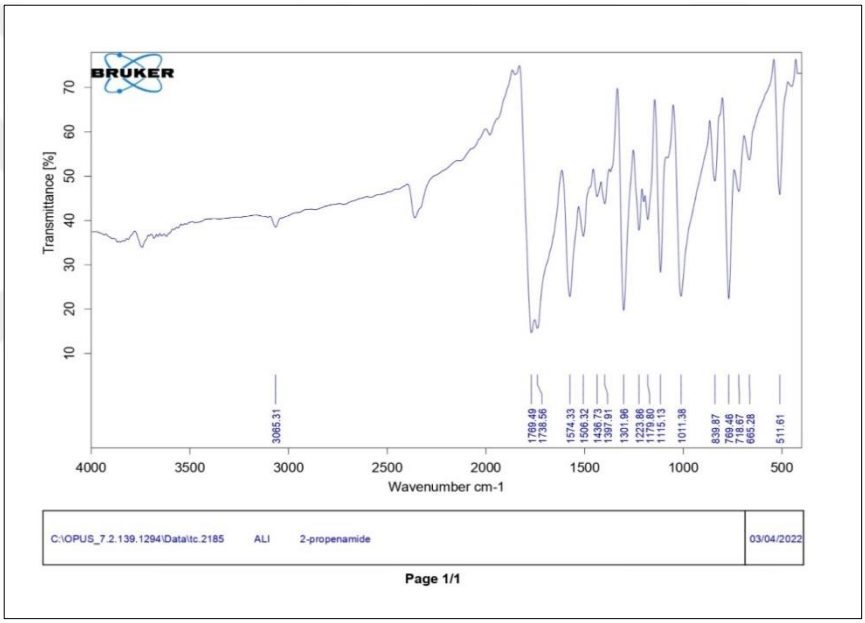

Figure 4.5 FT-IR spectrum of polymer (F4)

Furthermore, FTIR spectral data related to functional groups for all the polymeric surfactant ar given in Table 4.1 below.

Table 4.1 FT-IR spectra of table prepared polymer

Comp.	υNH amide cm^{-1}	υCH Ar. cm^{-1}	υCH Cyc. cm^{-1}	υCH Aliph. cm^{-1}	υC=O amide cm^{-1}	υC=O ester cm^{-1}	υC=O anhyd. cm^{-1}	υC=C Ar. cm^{-1}	υC-N cm^{-1}	υC-O cm^{-1}
EDTA	3600	3014	----	2851	-----	----	1690	1690	---	----
F_1	3703	3020	----	2795	-----	1694	------	1417	----	----
F_2	3700	3018	----	2893	-----	1694	------	1447	----	----
F_3	3698	3065	----	2890	-----	1769	------	1575	----	1368
F_4	3705	3035	----	----	-----	1769	------	1574	----	1301

4.2 ^{1}H-NMR Spectra of Gemini Surfactants

^{1}H-NMR of F1, F2,F3, F4 are given in Figure between 4.6-4.9 were analysed to find out the position of hydrogen in different gemini surfectent. All the samples were analysed in chloroform -d. the peak at 1.6ppm belongs to solvent in all spectras.

4.2.1 ^{1}H-NMR spectra of gemini surfactants (F1)

The ^{1}H-NMR spectra of the compound F1 was examined as shown in Figure 4.6, and the results shown in Figure 4.6. The spectrum at (0.1 ppm) belongs to (O=C-CH_2-CH-C=O), while peaks observed at (1.25 ppm) belongs to CH_2-N. Moreover, peakes belong to CH_2 was observed at 1.6 ppm and for NH at 7.3 ppm (Macomber 1998, Aiad *et al*. 2012, Luo, Liu, and Chen 2013)

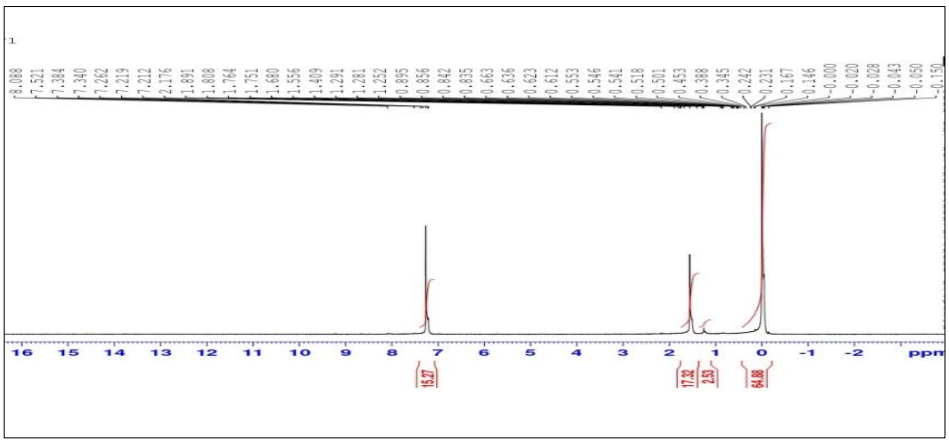

Figure 4.6 ^{1}H-NMR spectra of (F1) polymer

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4.2.2 ^{1}H-NMR spectra of gemini surfactants (F2)

The ^{1}H-NMR spectra of compound F2 was examined as shown in Figure 4.7. The spectrum reveals four signals, the first one belongs to (CH_2-CH_2) observe at 0.1 ppm, the second one belongs to CH=CH was observed at 1.3 ppm. Moreover, the signals appeared at 1.6 ppm belongs to chloroform-d and 7.3 ppm were belonge to CH=CH and NH respectively. (Macomber 1998, Aiad *et al*. 2012, Luo, Liu, and Chen 2013).

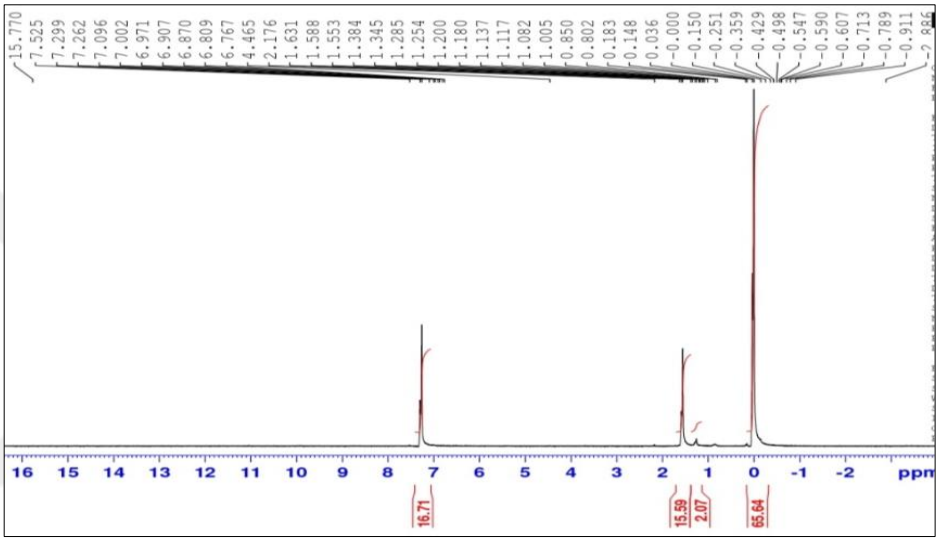

Figure 4.7 ^{1}H-NMR spectra of (F2) polymer

4.2.3 ^{1}H-NMR spectra of gemini surfactants (F3)

The ^{1}H-NMR spectra of compo pfund F3 was examined as shown in Figure 4.8. There were five signals observed belong to hydrogen atom in five different environments. The peak observed at 0.1 ppm was belong to (-CH_2-CH_2-) and 1.6 ppm belongs to chloroform-d. Moreover, peak observed between 2.2-2.9 ppm belongs to CH=CH and at 7.3 ppm represent presence of NH group, while naphthalic peaks were observed at the range of 7.3 - 8.7ppm (Macomber 1998, Aiad *et al*. 2012, Luo, Liu, and Chen 2013).

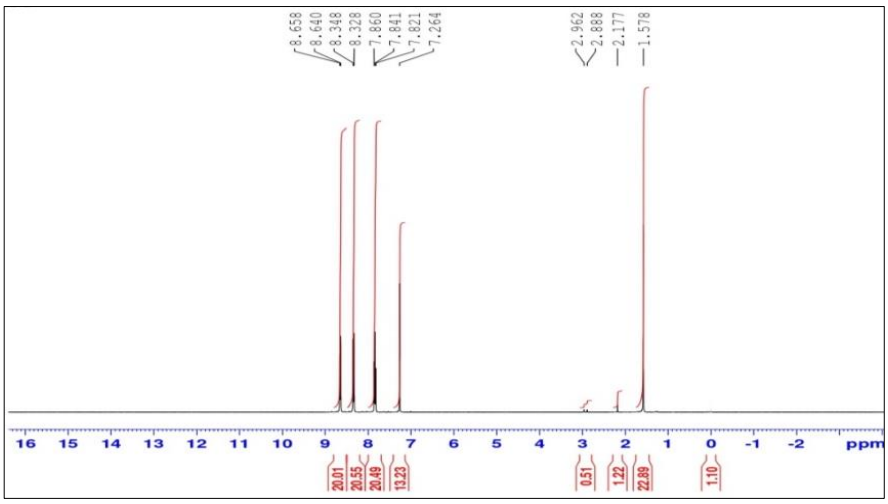

Figure 4.8 ^{1}H-NMR spectra of (F3) polymer

4.2.4 ^{1}H-NMR spectra of gemini surfactants (F4)

The ^{1}H-NMR of compound F4 was examined, and the results shown in Figure 4.9. The spectrum reveals the fifth signal, the peaks observed at 2.9 ppm belongs to CH_2=CH and 7.25 were belongs to NH group. The signals observed at in between (7.85 - 8.7 ppm) were belongs naphthalic groups (Macomber 1998, Aiad *et al.* 2012, Luo, Liu, and Chen 2013).

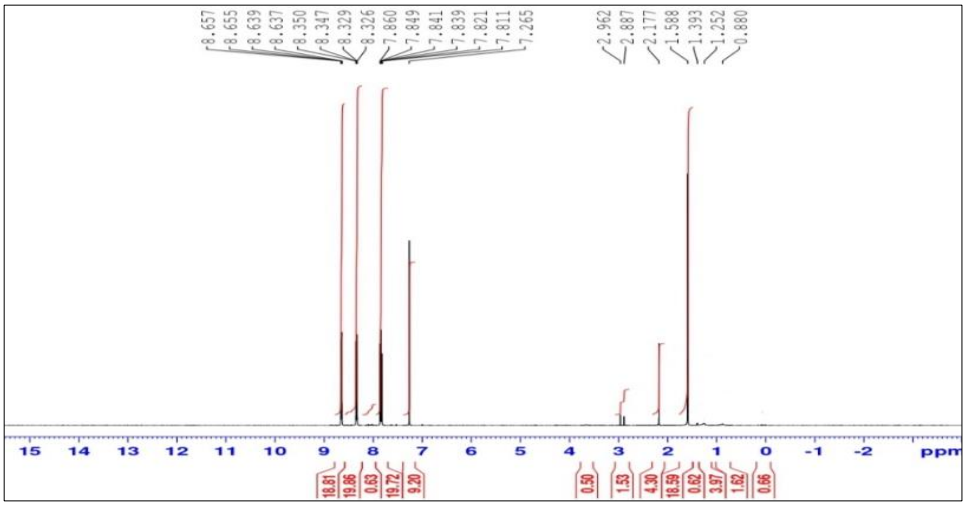

Figure 4.9 ^{1}H-NMR spectra of (F4) polymer

4.3 Determine of Average Molecular Weight by Viscosity (Mv) Measurment

The molecular weight of a polymer can be determined in several ways, including viscosity. Viscosity is an internal property of a fluid that resists the flow or flow of a fluid due to the internal friction of the molecules. The viscosity depends mainly on the nature and temperature of the liquid. To measure the viscosity of a fluid, a viscometer can be used, and a device used to determine the viscosity, i.e., the resistance of a liquid to the movement of something in it. Many methods are available to measure the viscosity of a polymer solution, and this experiment depends on the capillary tube viscometer method. There are two well-known viscometers: the Ubbelohde Viscometer and the Ostwald Viscometer. The Ostwald method is one of the simplest methods. In this method, the time of flow of the same volume for different concentrations of a solution has measured for polymer between the two lines (A and B) shown on the tube of the viscometer called the Ostwald Viscometer so that it will be the one used in this experiment.

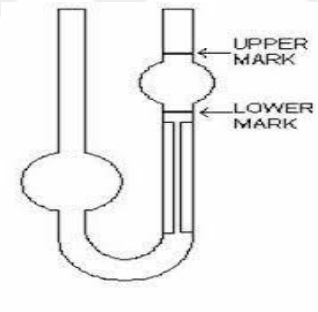

Figure 4.10 Viscometer

Water was used as a solvent to create a series of solutions for each polymer, and the descent time was recorded by applying Equation(4.1) to calculate the relative viscosity (Ovejero *et al*. 2007, Kwaambwa *et al*. 2007).

$$ŋr=t/t_{\circ} \qquad (4.1)$$

Where: ŋr=relative viscosity, t=time flow of solution, $t_{\circ}$= time flow of solvent.

In addition, the specific viscosity was also calculated by applying the following Equation (4.2).

$$ŋsp=t/t_{\circ}-1=ŋr-1 \qquad (4.2)$$

Where: ŋsp = specific viscosity

Equation (4.3) was used to calculate the reduced viscosity (sp /C), and a graph between (sp /C) and the intrinsic viscosity value was used to determine the intrinsic viscosity value (C).

$$ŋ=\lim\left(\frac{ŋsp}{C}\right) \qquad (4.3)$$

Where: ŋ= intrinsic viscosity, C= concentration (g/100mL).

Then a Mark and Houwink Equation (4.4) was applied (KamelAl-Khalidi and Al-Lami 2017, Gupta *et al.* 2005), $[ŋ]= K.Mv^{\alpha}$. Moreover, Mv= viscosity average molecular weight. K, α = The constants change with temperature, solvent type, and polymer type.

4.3.1 Viscosity measurement for F1, F2, F3 and F4 polymer

All the synthesized polymer (F1, F2, F3 and F4) were taken in different amount of (0.5, 0.55, 0.6, 0.65, and 0.7 g) into 10ml of xylene and measured its viscosity (specific viscocity and relative viscosity) at different flow rate of solution, results are shown into Table 4.2.

Table 4.2 Measurement of viscosity of F1 surfactant

Contact	t	t_o	ŋr	ŋsp	ŋ
0.5	32	28	1.14285	0.14285	0.28
0.55	34	28	1.21428	0.21428	0.38
0.6	36	28	1.28571	0.28571	0.47
0.65	37	28	1.32142	0.32142	0.49
0.7	39	28	1.39285	0.39285	0.56

From Table 4.2 it was found that at the concentration of 0.7 g of surfactant F1, the measured maximum relative viscosity was about 1.39285 and specific viscosity of 0.39285 as the overall viscosity for highest amount of F1 polymer is 0.56. which increase from (0.5, 0.55, 0.6, 0.65, and 0.7 g) concentration of polimer F1 to (0.28, 0.38, 0.47, 0.49 and 0.56) respectively.

Table 4.3 Measurement of viscosity of F2 surfactant

Contact	t	t_o	ŋr	ŋsp	ŋ
0.5	31	28	1.10714	0.10714	0.21
0.55	33	28	1.17857	0.17857	0.32
0.6	34	28	1.21428	0.21428	0.35
0.65	36	28	1.28571	0.28571	0.43
0.7	38	28	1.35714	0.35714	0.51

From Table 4.3 it was found that at the maximum concentration of 0.7 g for surfactant F2 have maximum viscosity of 0.51. in addition, the measured maximum relative viscosity found to be 1.3514 and specific viscosity of 0.35714. it was observed that with the increase in concentration of polymer from (0.5, 0.55, 0.6, 0.65, and 0.7 g) viscosity of polymer F2 also increase (0.28, 0.38, 0.47, 0.49 and 0.56) respectively.

Table 4.4 Measurement of viscosity of F3 surfactant

Contact	t	t_o	ŋr	ŋsp	ŋ
0.5	30	28	1.07142	0.07142	0.14
0.55	32	28	1.14285	0.14285	0.25
0.6	33	28	1.17857	0.17857	0.29
0.65	35	28	1.25	0.25	0.38
0.7	37	28	1.32142	0.32142	0.45

The data about measured viscosity especially the relative and specific viscosity are given into Table 4.4 fo F3 polymer. It was found that, at the concentration of 0.7 g of surfactant F7 have maximum viscosity of 0.45. in addition the relative visocity was about 1.32142 and specific viscosity of 0.32142. it was found that the increase from (0.5, 0.55, 0.6, 0.65, and 0.7 g) concentration of polimer F1 there was increase into the viscosity from (0.28, 0.38, 0.47, 0.49 and 0.56) respectively.

Table 4.5 Measurement of viscosity of F4 surfactant

Contact	**t**	**t_o**	**ŋr**	**ŋsp**	**ŋ**
0.5	33	28	1.17857	0.17857	0.35
0.55	34	28	1.21428	0.21428	0.38
0.6	35	28	1.25	0.25	0.41
0.65	36	28	1.28571	0.28571	0.43
0.7	38	28	1.35714	0.35714	0.51

Similarly, the data about measured viscosity of F4 was shown in table 4.5 also showed the same results for F4 polymer. However, at the same concentration, the measured relative viscosity and specific viscosity was 1.35714 and 0.35714 respectively. From the above results of Tabel (4.2 to 4.5) the molecular weight of the surfactents were measure as given in Tabel 4.6.

Table 4.6 Average molecular weight (Mv) of surfactant measured by viscosity

Polymers (surfactants)	**Intrinsic viscosity (ŋ) Where k=0.0002 and α=0.76**	**Average Molecular Weight (Mv)**
F1	0.47	27265
F2	0.35	18499
F3	0.29	14444
F4	0.41	22780

For all polymer of F1, F2, F3 and F4 the measured intrinsic viscosity (ŋ) was found to be 0.47, 0.35, 0.29 and 0.41 with average molecular weight of 27265, 18499, 14444, 22780 respectively (Mv).

4.4 Determination of Critical Micelles Concentration (CMC) by Electrical Conductivity Method

Different dilutions of surfactant were prepared at the concentrations of 20-200 ppm for determination of CMC of solutions by electrical conductivity (G) values at (25C°). Specific conductivity (L) was determined by appling the values in Equatin (4.5) given below.

$$L = A\ G \qquad (4\text{-}5)$$

Where: L= specific conductivity. A=cell constant and G= electrical conductivity.

We noticed that as the concentration increased, the conductivity changed linearly because there was direct relation of release in amphiphilic molecules compared to concentration. Hence, it effects the concentration of micelles formation to reach a critical level (CMC). This significant increase in free ions released in the solution, as shown in Table 4.7, and Figure 4.11 to 4.14, caused the enormous increase in conductivity.

Table 4.7 Values of specific conductivity of surfactants

Conc. (ppm)	**Specific Conductivity of Surfactants (L*10^{-3}) Ohm.cm^{-1}**			
	F1	**F2**	**F3**	**F4**
20	1.1	1.01	1.12	0.9
40	1.15	1.1	1.18	1.14
60	1.2	1.17	1.24	1.19
80	1.23	1.22	1.29	1.26
100	1.28	1.28	1.35	1.32
120	1.32	1.31	1.38	1.39
140	1.37	1.38	1.41	1.45
160	1.41	1.44	1.46	1.49
180	1.46	1.49	1.51	1.53
200	1.51	1.53	1.57	1.58

From Tabel 4.7. it was found that, the specific conductivity of surfactants F1 to F4 at concentration of 200 ppm give maximum conductivity value of 1.51, 1.53, 1.57 and 1.58 Ohm.cm^{-1} respectively (Perinelli *et al.* 2020, D. Liu *et al.* 2018).

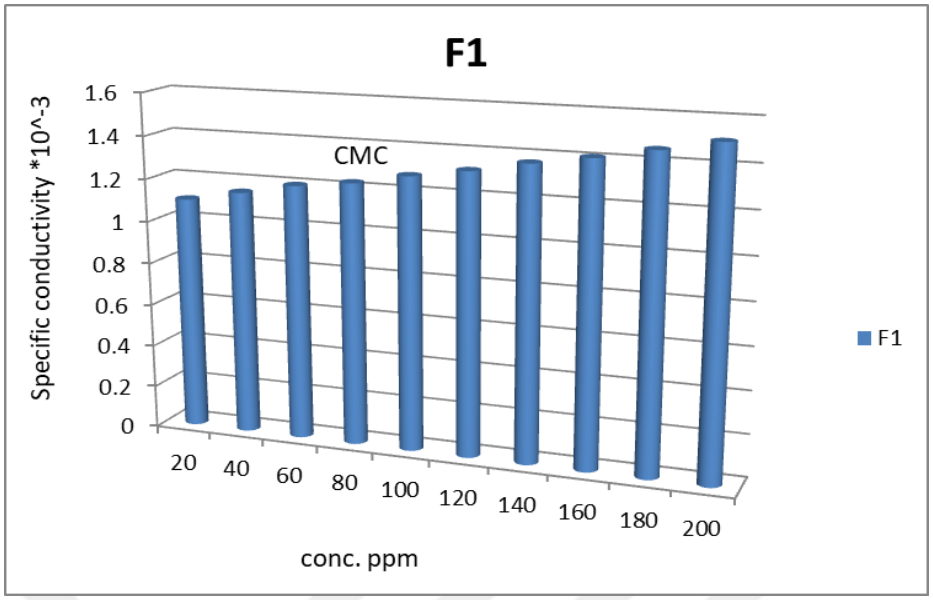

Figure 4.11 Critical micelles concentration of surfactant (F1)

According to Table 4.8, the plot was drawn for F1 polymer as shown in Figure 4.11, which showed that, with the increase in concentration from 20 to 200 ppm of F1 polymer, the specific conductivity increases is directly affecting the CMC value.

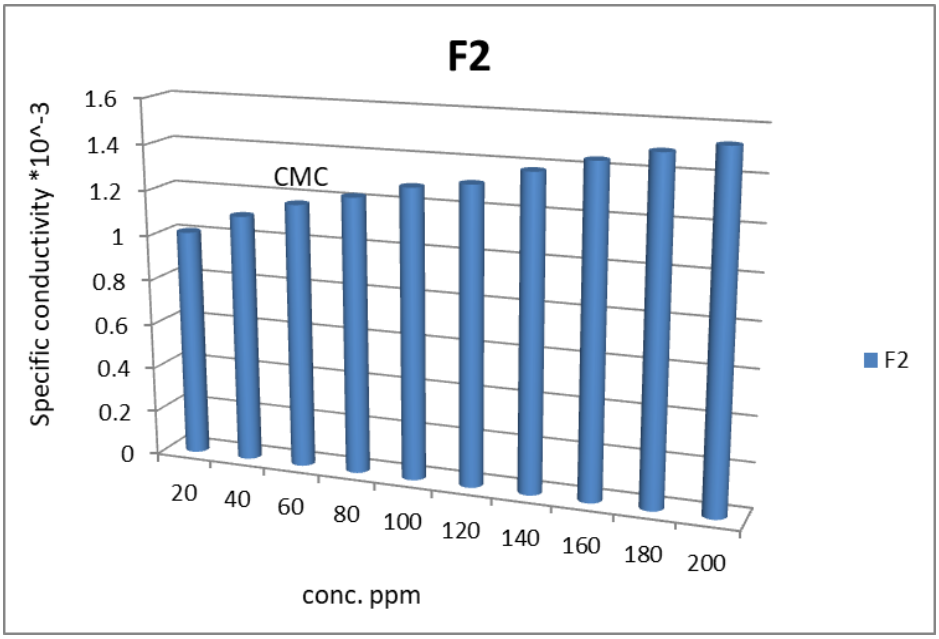

Figure 4.12 Critical micelles concentration of surfactant (F2)

Similar to F1 polymer's specific conductivity, the observed data for the formation of CMC was determined by increasing the concentration of F2 polymer from 20 to 200 ppm and measuring the specific conductivity of the poymer. It was found that with the increase the concentration, the value of specific conductivity increases, which have direct effect on CMC value.

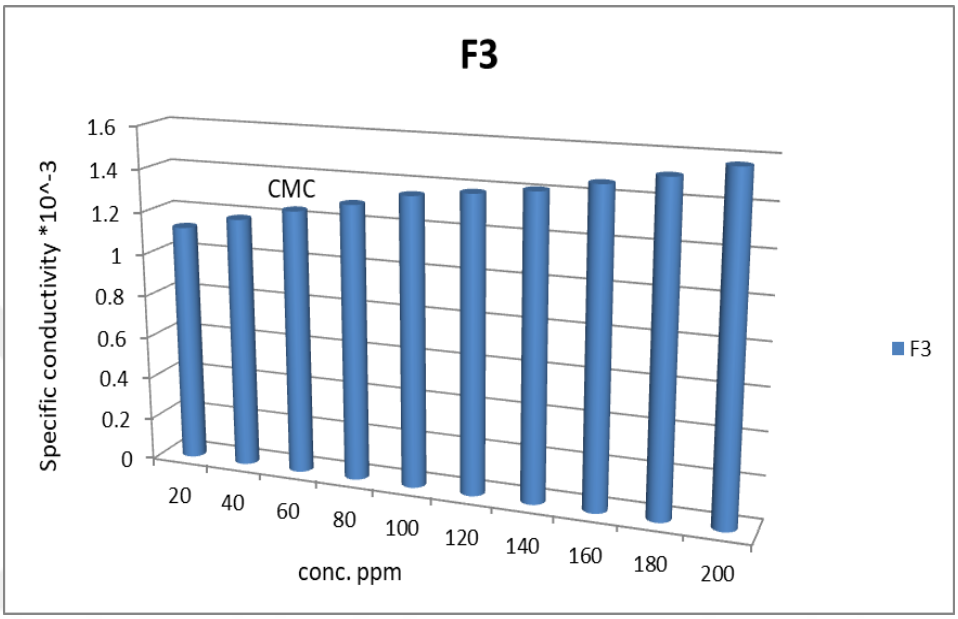

Figure 4.13 Critical micelles concentration of surfactant (F3)

The specific conductivity of F3 polymer was measured against variable concentration as shown in Figure 4.13. it was found that, with the increae in polymer concentration for 20-200 ppm the specific conductivity of F3 polymer also affect. This increase in conductivity has direct effect on CMC formation.

Almost similar results were observed for F4 polymer, when specific conductivity was measured against polymer concentration from 20 to 200 ppm. The results are ploted are shown in Figure 4.14 shoed the increase in concentration of polymer directly effect the value of specific conductivity as well as CM formation value.

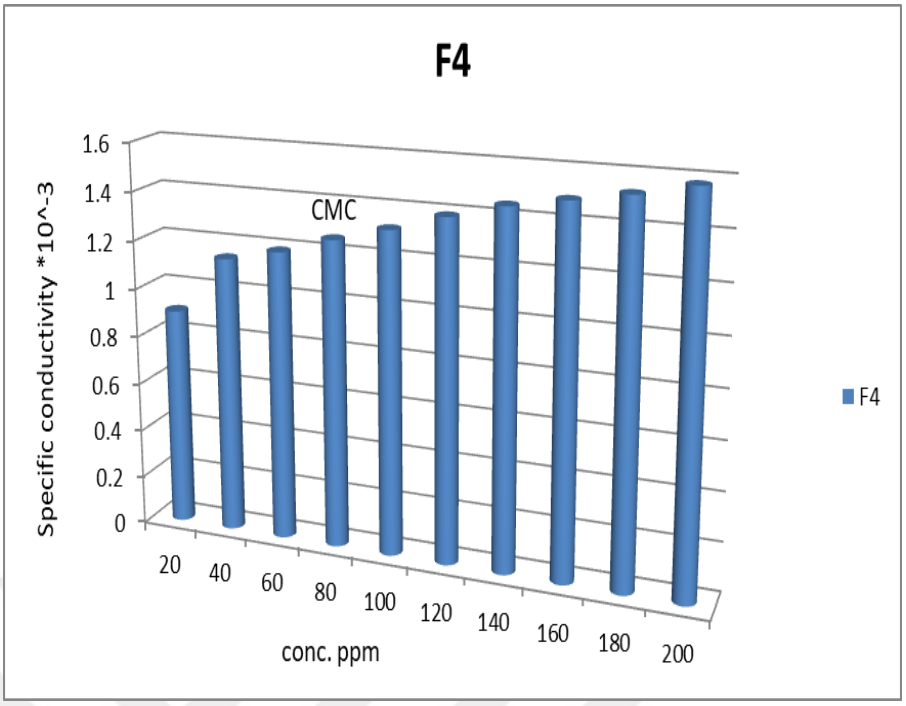

Figure 4.14 Critical micelles concentration of surfactant (F4)

Table 4.8 Critical micelles concentration (CMC) of prepared surfactants

Surfactant	CMC (ppm)
F1	80
F2	60
F3	60
F4	80

Hence, from the obtained specific conductivity of polemers F1-F4 against their variable concetration, it was found that the criticle micelles concentration was 80, 60, 60 and 80 ppm respectively.

4.5 Determination of Hydrophilic-Lipophilic Ratio (HLB)

The HLB value of any polimer tell the solubility of the polymer into solvent either water or organic one. By comparing the HLB value of the polymer with HLB-vlaue table we determined the amount of solvent required for solubility. Therefore, HLB values of prepared surfactants F1-F4 were calculated using the acidity function by following the Equation (2.4) given before. It was found that, the HLB values found by

applied method was significant for prepared surfactant. For that purpose that was driven from Table 2.2 and the calculatedHLB value is given in Table 4.9 (Severino *et al.* 2011, McCrary *et al.* 2013).

Table 4.9 Values of (HLB) of synthesized surfactants

Surfactant	Values of HLB	Use
F1	11.80	(O/W) emulsion
F2	12.38	(O/W) emulsion
F3	7.99	(W/O) emulsion
F4	7.21	(W/O) emulsion

From the above thble it was found that the maximum HLB value was for F2 surfactent of 12.38 which showed its solubility in oil as compare to F4 with HLB value of 7.21 close to water in oil solubility value.

4.6 Demulsifiers Preparation

By combining already preprepared surfactants (F1, F2, F3, and F4) with demulsifiers, (DF1, DF2, DF3, and DF4) were developed. They were formed by combining the 0.2g of surfactant, 0.05 g of heavy natural oil and 0.05 g of silver nitrate ($AgNO_3$) into (0.8 ml) of toluene as a solvent. Afterthat, the separation efficiency of prepared demulsifer was investigated on wet crude oil. The chemical structure of surfactant and additions of demulsifier influence separation efficiency. Therefor, there is aneed to examine the ideal circumstances for removing water from an emulsion (Nikkhah *et al.* 2015).

4.6.1 Effective quantity of prepared demulsifiers and factors influencing separation efficiency

This study followed standard protocols such as time and quantity of water separated at desire concentration of 50, 100, 150, 200, and 250 ppm used to calculate the separation efficiency. At constant temperature, the percentage of separation (E percent) was calculated using Equation (4.6) (Yu *et al*. 2020, Li *et al.* 2018).

$$E\% = V1(mL)/V2(mL)*100 \quad (4.6)$$

Where, E%= percent of the separation, V1= size of separated water prepared demulsifier, V2= size of commercial demulsifier in separated water (theoretical).

A variety of factors influence the efficiency of water split from crude oil. It was analyesed and supported by already published experimental data, such as: demulsifier quantity, time and additives, (Taleghani *et al*. 2019, Feitosa *et al.* 2019).

4.6.2 Additives

To improve the separation process, several additives were combined with demulsifiers, including heavy oils and silver nitrate ($AgNO_3$). Such as, castor oil is a thick, very dense, natural standard oil (0.05g). It has many advantages, including the ability to prevent ignition and ensure safe storage and transportation. Demulsifiers are substances that increase viscosity and flow. It also increases the effectiveness of separation. It works to strengthen its connection to crude oil. In addition, use of large amount 0.05 g of silver nitrate ($AgNO_3$) It helps to strengthen the bond with the water phase as a result water phase became more polarized.

4.6.3 Effect of quantity of demulsifier and time

At concentrations of 50, 100, 150, 200, and 250 ppm of the surfactants, the chemical compounds were studied and compared to the theoretical results of a commercial demulsifier (Rp\6000). An increase in quantity leads to an increase in separation efficiency under the same conditions. In addition, the effect of time on emulsion water separation efficiency was also investigated. According to the findings, it was found that time had a significant impact on separation effecieny. However, the value of time is immeasurable but, according to commercialy available demulsifier separation process, the established standard time was of 120 minuts (Rp\6000). In our study we studied (20,40,60,80,100and 120 minutes) as a time, against separation effecieny of water from emulsion as shown in Table 4.10- 4.19 and Fig 4.15-4.24, that the water separation

efficiency of all prepared demulsifiers increases with time. The efficiency calculation was with a commercial emulsifier product.

Table 4.10 Separation of water by applying 50 ppm of synthesized demulsifiers

Time (minutes)	F1 (H_2O) mL	F2 (H_2O) mL	F3 (H_2O) mL	F4 (H_2O) mL
20	0.9	1.1	0.40	0.30
40	1	1.4	0.50	0.40
60	1	1.7	0.60	0.60
80	1	1.7	0.93	0.96
100	1	1.7	0.93	0.97
120	1	1.7	0.93	0.97

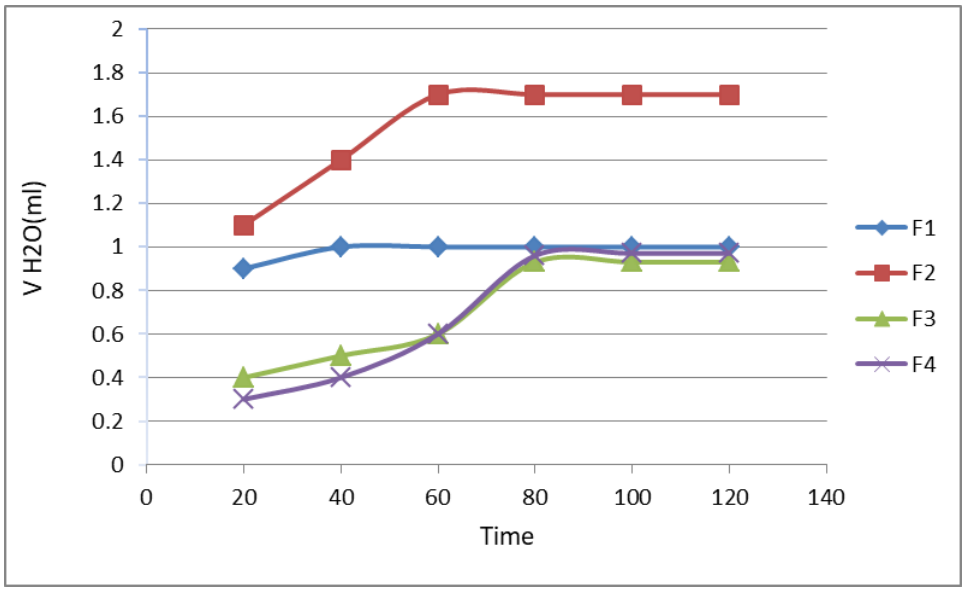

Figure 4.15 Graphical representation of water separation from emulsion at 50 ppm of concentration of demulsifier against time

The volume of water removed with surfactant at the concentration of 50 ppm against time of 20-120 minutes was shown in Tabel 4.5 and ploted as shown in Figure 4.16. it was found that among F1, F2, F3 and F4, maximum removel of water was done almost 1.7 ml in 40 minutes compared to others. The efficiency calculation was with a commercial emulsifier product.

Table 4.11 Separation efficiency of surfectents at 50 ppm concentration of demulsifiers

Time (minutes)	**%E F1**	**%E F2**	**%E F3**	**% E F4**
20	91.83	112.24	40.81	30.61
40	102.04	142.85	51.02	40.81
60	102.04	173.46	61.22	61.22
80	102.04	173.46	94.89	97.95
100	102.04	173.46	94.89	98.97
120	102.04	173.46	94.89	98.97

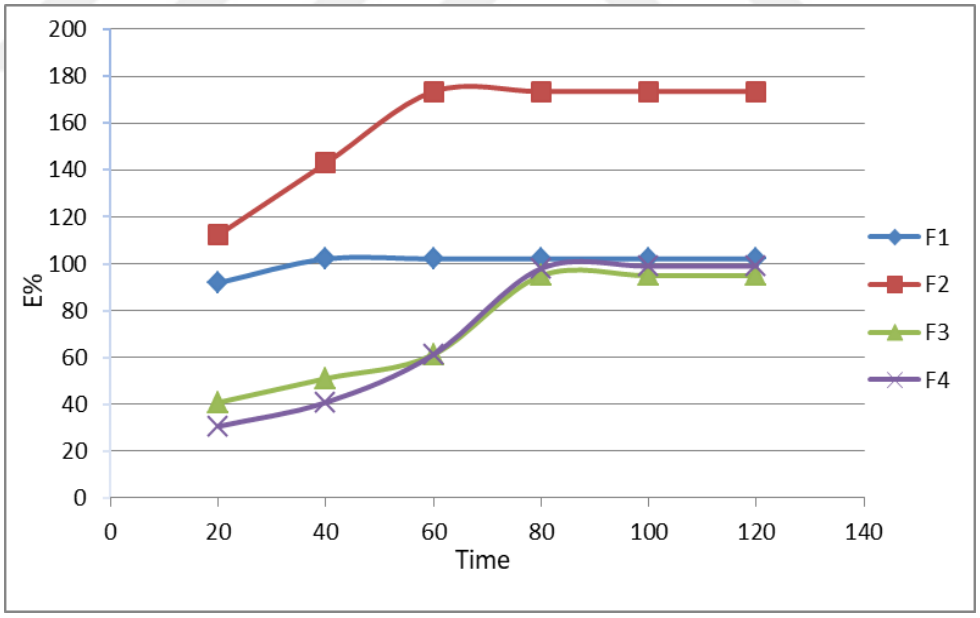

Figure 4.16 Graphical representation of water separation effeciency of surfactant from emulsion at 50 ppm of concentration of demulsifier against time

The efficiency of removel of water at 50 ppm of surfactant concentration was given in Table 4.11 and a plot was drawn from that data given in Figure 4.17. it was found that

F2 polymer have 173.46 % of efficiency of separation of water at 50 ppm concentration of surfactant.

Table 4.12 Separation of water by applying 100 ppm of synthesized demulsifiers

Time (minutes)	**F1 (H_2O) mL**	**F2 (H_2O) mL**	**F3 (H_2O) mL**	**F4 (H_2O) mL**
20	1	1.5	0.30	0.41
40	1.2	1.9	0.60	0.47
60	1.5	2	0.65	0.71
80	1.5	2.1	0.89	0.94
100	1.5	2.1	0.92	0.97
120	1.5	2.1	0.93	0.97

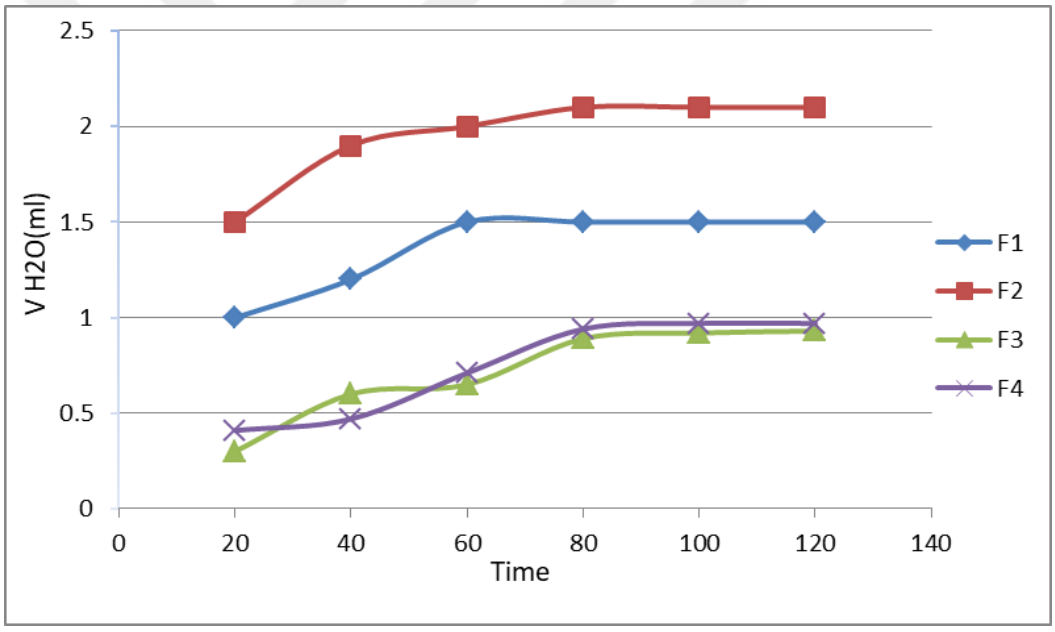

Figure 4.17 Graphical representation of water separation from emulsion at 100 ppm of concentration of demulsifier against time

From the data given in Table 4.12, it was found that, F2 demulsifier at the concentration of 100 ppm effectively removed 2.1 ml of water compare to other surfactants. The effectiveness of separation of water from emulsion was took place with in 80 minutes.

Table 4.13 Separation efficiency of surfectents at 100 ppm concentration of demulsifiers

Time (min)	% E F1	%E F2	%E F3	%E F4
20	102.04	153.06	30.61	41.83
40	122.44	193.87	61.22	47.95
60	153.06	208.08	66.32	72.44
80	153.06	214.28	90.81	93.87
100	153.06	214.28	94.89	98.97
120	153.06	214.28	94.89	98.97

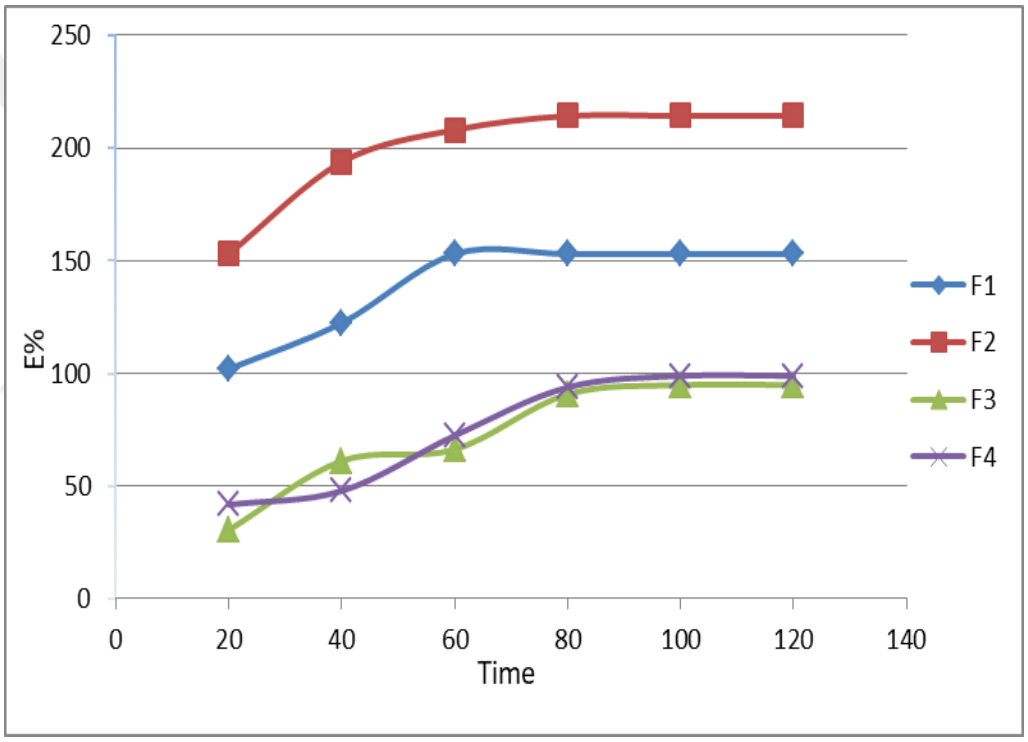

Figure 4.18 Graphical representation of water separation effeciency of surfactant from emulsion at 100 ppm of concentration of demulsifier against time

The efficiency of F2 polymer in separation of water at 100 ppm was found mmximum as compared to other synthesized polymer as shown in Table 4.13 and drawn in Figure 4.18. it was found that, 214.28 % of F2 polymer was effective with in 80 minutes compared to affectivness of other polymers.

Table 4.14 Separation of water by applying 150 ppm of synthesized demulsifiers

Time (min)	F1 (H_2O) mL	F2 (H_2O) mL	F3 (H_2O) mL	F4 (H_2O) mL
20	1.2	1.5	0.53	0.47
40	1.4	1.9	0.62	0.66
60	1.7	2.1	0.84	0.81
80	1.7	2.3	0.98	0.99
100	1.7	2.3	0.98	0.99
120	1.7	2.3	0.98	0.99

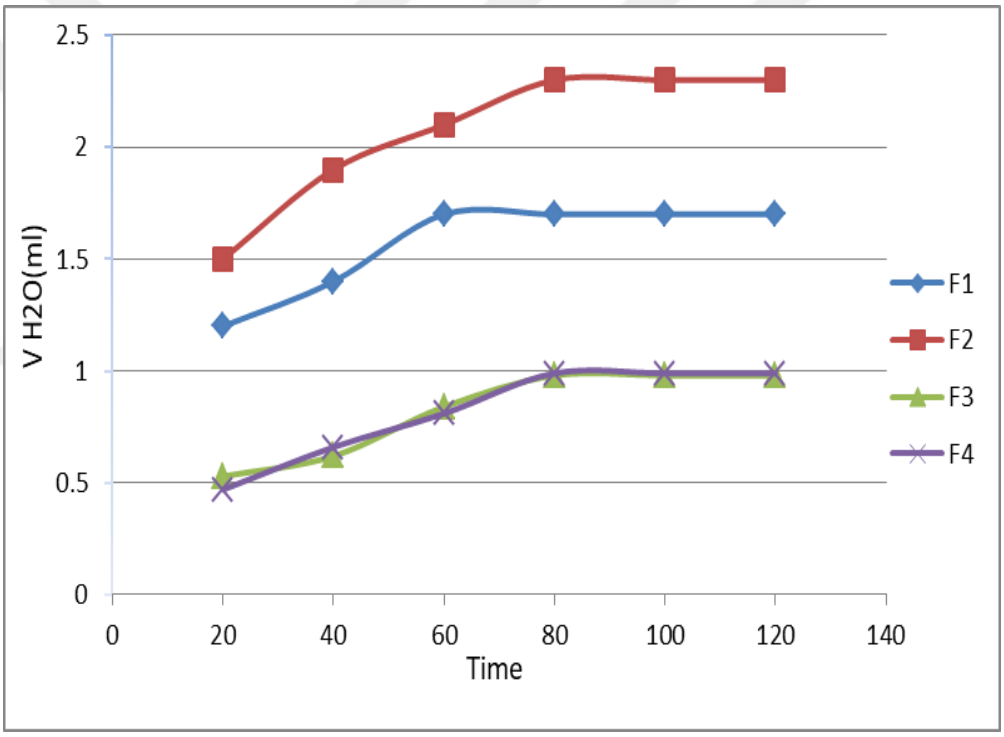

Figure 4.19 Graphical representation of water separation from emulsion at 150 ppm of concentration of demulsifier against time

Similar results were found at 150 ppm concentration of demulsifier as given in Table 4.14 and drawn as shown in Figure 4.19. It was found that, 2.3 ml of water was removed in 80 minutes by F2 polymer compare to others.

Table 4.15 Separation efficiency of surfectents at 150 ppm concentration of demulsifiers

Time (min)	%E F1	%E F2	%E F3	% E F4
20	122.44	153.06	50.08	47.95
40	142.85	193.87	63.26	66.34
60	173.46	214.28	85.71	82.65
80	173.46	234.69	100	101.02
100	173.46	234.69	100	101.02
120	173.46	234.69	100	101.02

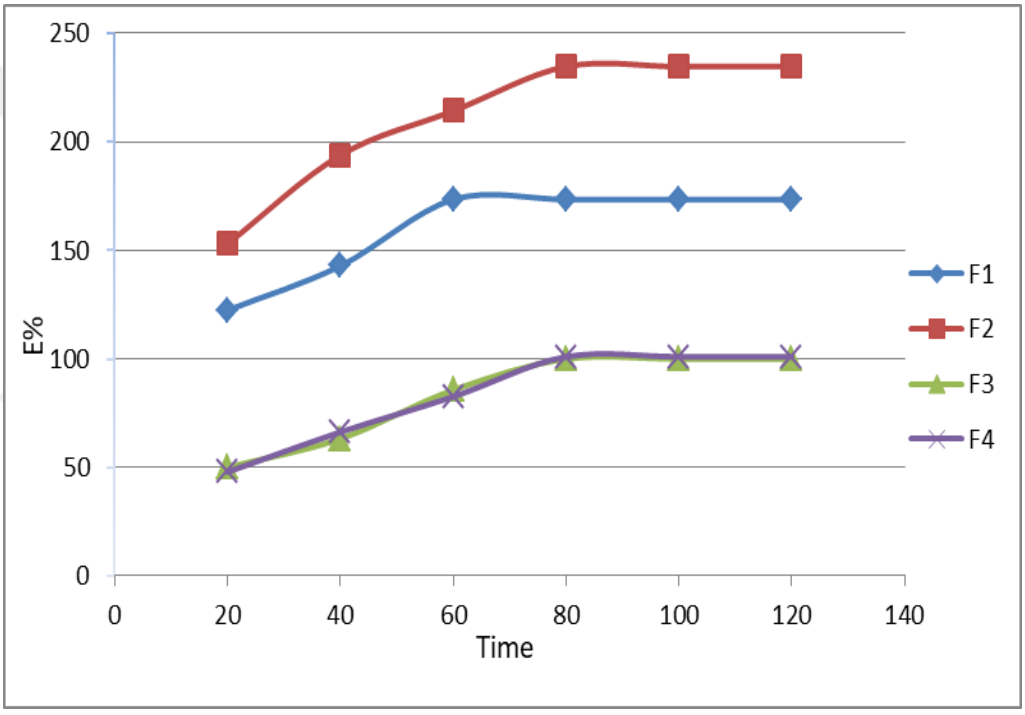

Figure 4.20 Graphical representation of water separation effeciency of surfactant from emulsion at 150 ppm of concentration of demulsifier against time

The effectiveness of surfactant at 150 ppm of concentration are given in Table 4.15 and graphically represented in Figure 4.20, from the obtained data, it was found that, the efficiency of F2 polymer was maximum of 234.28 % in 80 minutes, compare to other demulsifieres.

Table 4.16 Separation of water by applying 200 ppm of synthesized demulsifiers

Time (min)	F1 (H_2O) mL	F2 (H_2O) mL	F3 (H_2O) mL	F4 (H_2O) mL
20	1.5	1.7	0.55	0.95
40	1.9	2	0.62	0.95
60	1.9	2.4	0.79	1.00
80	1.9	2.4	0.90	1.00
100	1.9	2.4	1.00	1.00
120	1.9	2.4	1.00	1.10

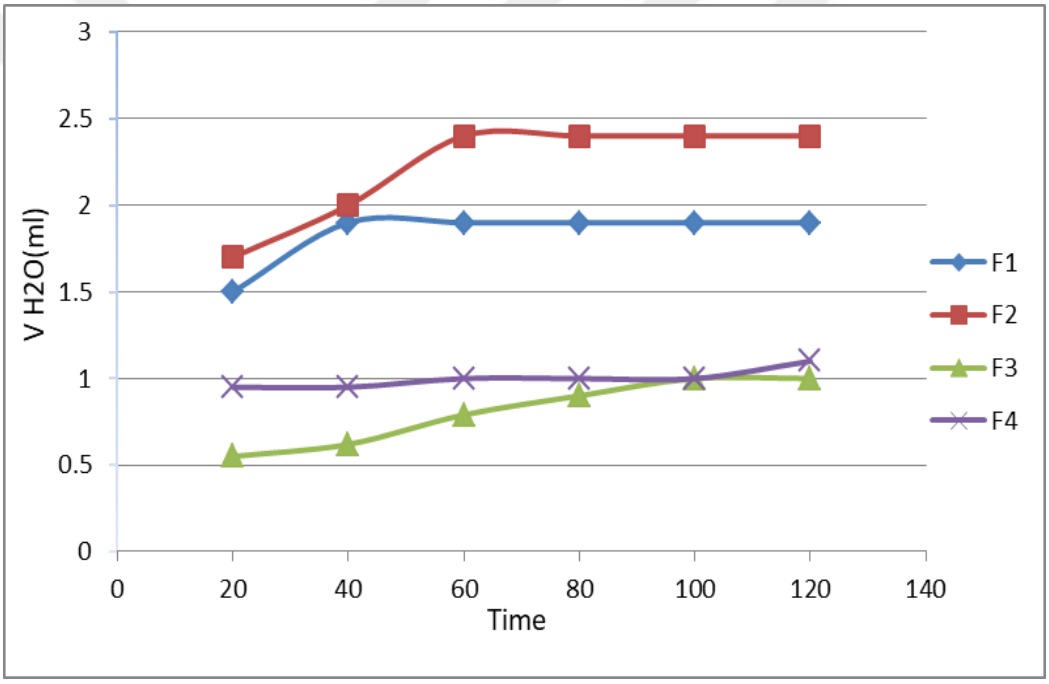

Figure 4.21 Graphical representation of water separation from emulsion at 200 ppm of concentration of demulsifier against time

The separation of water by applying 200 ppm of surfactant in concentration, data shown in Table 4.16 and graphically represented in Figure 4.21, showed that, 2.4 ml of water was removed with the increased amount of 200 ppm of surfactant F2 was maximum compared to other demulsifier in 60 minutes.

Table 4.17 Separation of water by applying 200 ppm of synthesized demulsifiers

Time (min)	%E F1	%E F2	%E F3	%E F4
20	153.06	173.46	56.12	96.93
40	193.87	204.08	63.26	96.93
60	193.87	244.89	80.61	102.04
80	193.87	244.89	91.83	102.04
100	193.87	244.89	102.04	102.04
120	193.87	244.89	102.04	112.24

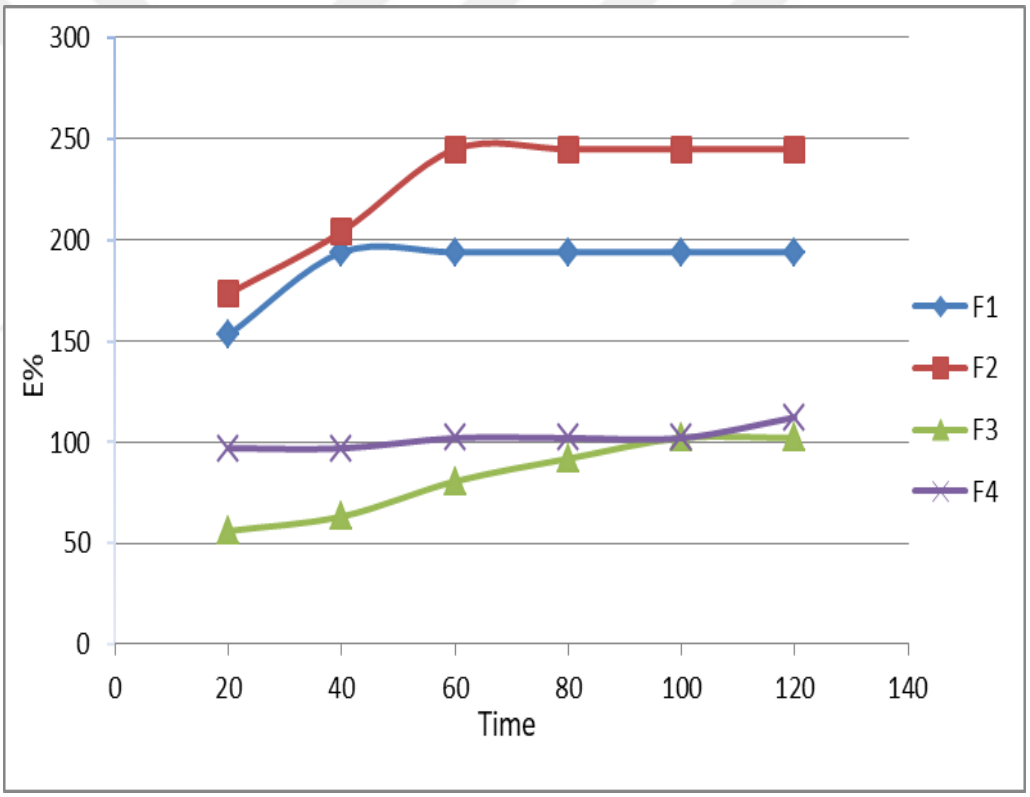

Figure 4.22 Graphical representation of water separation effeciency of surfactant from emulsion at 200 ppm of concentration of demulsifier against time

The efficiency of separation of water by using 200ppm of emulsifier shown in Table 4.17 and plot was drawn in Figure 4.22. maximum efficiency was shon by F2 surfactent compare to other with separation efficiency of 244.89 % at 40 minutes.

Table 4.18 Separation of water by applying 250 ppm of synthesized demulsifiers

Time (min)	F1 (H_2O) mL	F2 (H_2O) mL	F3 (H_2O) mL	F4 (H_2O) mL
20	1.7	1.8	0.89	0.88
40	2	2.1	0.96	0.99
60	2	2.6	1.10	1.10
80	2	2.7	1.10	1.15
100	2	2.7	1.10	1.15
120	2	2.7	1.10	1.15

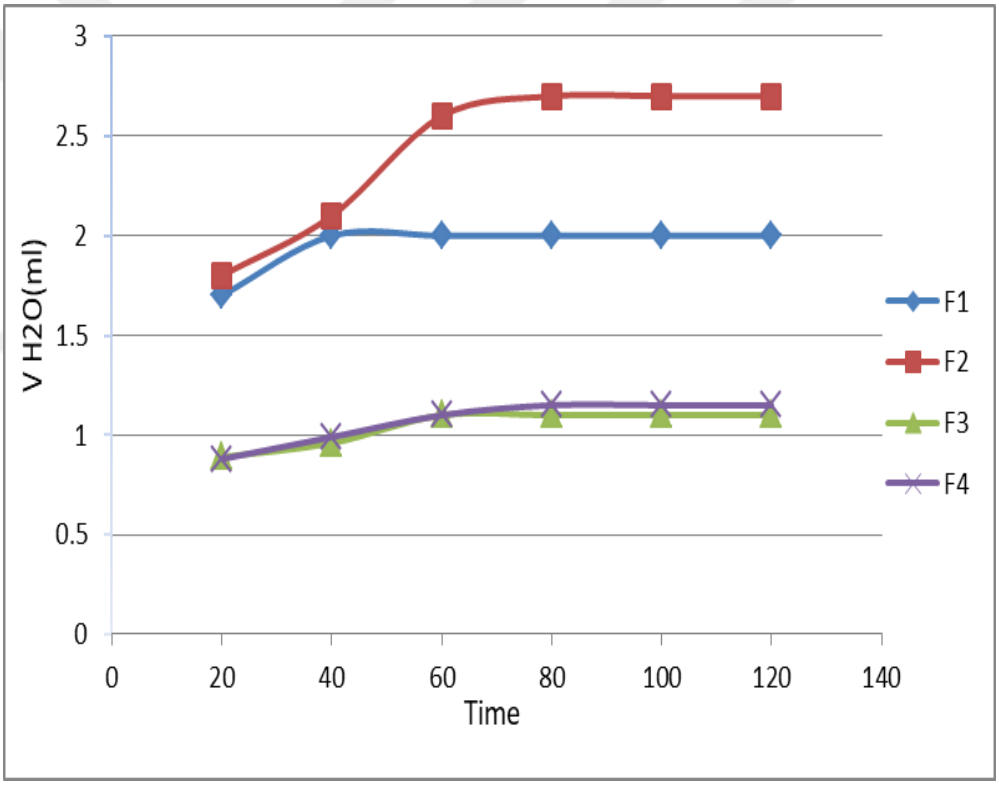

Figure 4.23 Graphical representation of water separation from emulsion at 250 ppm of concentration of demulsifier against time.

The data obtained for concentration of 250 ppm of surfactents, applied for the separation of water from emulsion are shown in Table 4.18 and drawn a plot given in Figure 4.23. it was found that volume of separation of water was 2.7 ml after 80 minutes, compared to other emulsifiers.

Table 4.19 Separation of water by applying 250 ppm of synthesized demulsifiers

Time (min)	E% AF1	E% AF2	E% AF3	E% AF4
20	173.46	183.67	90.81	89.79
40	204.08	214.28	97.95	101.02
60	204.08	265.30	112.24	112.24
80	204.08	275.51	112.24	117.34
100	204.08	275.51	112.24	117.34
120	204.08	275.51	112.24	117.34

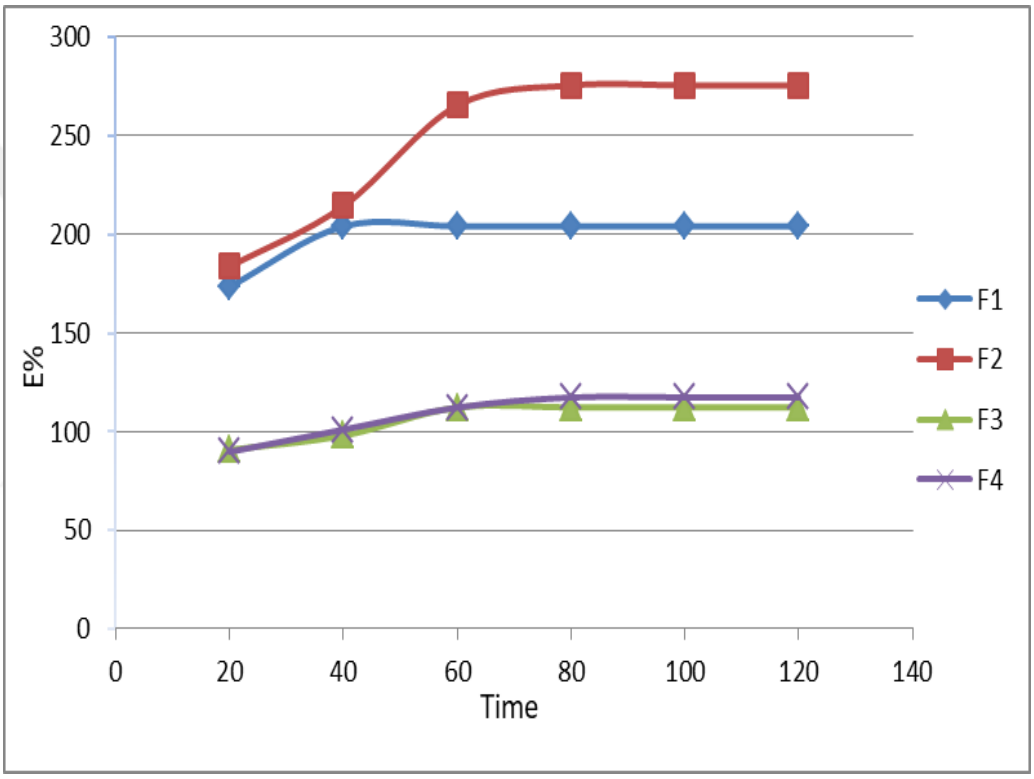

Figure 4.24 Graphical representation of water separation effeciency of surfactant from emulsion at 250 ppm of concentration of demulsifier against time

Similar results was found for 250 ppm concentration of demulsifers. The efficiency of surfactant F2 was found maximum as given in Table 4.19 and plot was drawn shown in Figure 4.24. Overall results showed that, by Increasing the quantity of surfavtent, lowers the surface tension for water drops in the emulsion and make it easier for penetration into drops. Because prepared demulsifiers melt faster in water, they are becoming increasingly important (Hajivand and Vaziri 2015, Feitosa, Alves, and de Sant'Ana 2019). From the obtained data from our study we can easily say that, the separation

efficiency of prepared demulsifier enhance according to DF2>DF1>DF3>DF4 respectively for all prepared surfactants.

4.7 Application of Demulsifiers on Crude Oil Emulsions

Compared to commercial demulsifiers (Rp6000), the demulsifiers have a water separation efficiency of up to 100 percent in crude oil emulsions. Specification of crude oil is given in Table 4.20. The prepared demulsifiers can be distinguished by their ease of utilization, low of cost, as well as their constant separation ability. It was detrmined by one weak of separation test of water, which remains constant. Several factors contribute to its effectiveness (Adem and Kreamid 2010) such as chemical structure, HLB value and addition of right amount of additives.

Table 4.20 Wet Crude Oil Specifications

IT	Characteristics	Standard Methods	Result
1	Density	ASTM D-5002	0.9044
2	Specific gravity @ 60 F°	=	0.9053
3	API Gravity	=	24.80
4	Water Content, vol. %	ASTM D-4928	2.8
5	Water & Sediment, VOL.%	ASTM D-4007	9.578
6	Salt Content, 1b/1000/br1	ASTM D-3230	358.5 PTB–1343 PPM
7	Sulfur Content, wt%	ASTM D-4294	0.8689
8	H_2S Dıssolved in crude oil PPM	ASTM D-7621	0.04
9	Flash Point, C°	ASTM D-7094	-1
10	Asphalten Content, wt%	IP 143	1.843
11	Pour Point, C°	ASTM D-97	-45
12	Reid Vapour Pressure@100 F°-Psi	ASTM D-6377	4.88
13	C° Ref Refractive Index	ASTM D-1218	1.487
14	Kinematic Viscosity, cSt 1-@ 70 F° (21.1 C°) 2-@100 F° (37.8 C°) 3-@ 120 F° (48.9 C°) 4-@140 F° (60.0 C°)	ASTMD-445	 52.54 33.87 25.76 16.87

4.8 A Study of Gemini Surfactants as a Demulsifier

Several factors with a direct impact on water separation fraction were investigated for the determination of separation efficiency of Gemini surfactant-based demulsifiers compared to commercial demulsifier (RP6000). There are following variables considered:

A. Demulsifier concentration: Demulsifier (50, 100, 150, 200, and 250 ppm) was used in various concentrations to achieve an effective dosage for breaking of emulsification.

B. Effect of temperature on separation efficiency were studied at 25°C in the winter and 50°C in the summer.

C. The volume of water separated was measured to investigate the effect of time in (20, 40, 60, 80, 100, and 120 minutes).

The demulsifier is one of the most affecting analyte used to remove water from oil and affect on the stability of the emulsifier (Adem and Kreamid 2010, Mosayebi and Abedini 2013) because it breaks the film that surrounds the continuous phase and allows the mixture to be separated from one another. The emulsion separation efficiency also influenced by the structure of the Gemini surfactant and with the addition of additives used in the demulsifier composition. The separation performance of water from crude oil as a function of concentration, time, and temperature was calculated using Equation (4.7) (Azim *et al.* 2010, Ramalho *et al.* 2010):

$$\%E\ Separation=(VS/VT)x100 \qquad (4.7)$$

However, the percentage stability of emulsion was calculated by using Equation (4.8):

$$\%Emulsion\ stability=[1-(VS/VT)]x100 \qquad (4.8)$$

Where: % E Separation: The percentage of separation efficiency of water from crude oil. V S: Volume of water separated (mL). V T: Total volume of brine (15 mL) in crude oil.

4.8.1 Separation efficiency of (F1) as demulsifier

The water separation efficiency of the (F1) demulsifier with crude oil for treating W/O emulsions is shown in Table 4.21. If the demulsifier concentration is 250 ppm and the volume of separated water is 0.59 ml, the proportion of best separation efficiency (% E) at a temperature of 25 °C is (60.20 percent). At 50°C, the (percent E Separation) was (204.08 percent) in 250 ppm concentrations, with th e volume of water separated being (2 mL).

Table 4.21 Water and crude oil separation using demulsifier (F1)

Temp. (°C)	Con. ppm	Water Separated (mL)						%Sep, Effeciency
		20 minutes	40 minutes	60 minutes	80 minutes	100 minutes	120 minutes	
25 °C	50	0.1	0.15	0.21	0.25	0.3	0.35	35.71
	100	0.17	0.22	0.28	0.31	0.37	0.4	40.81
	150	0.19	0.25	0.32	0.38	0.41	0.43	43.87
	200	0.2	0.28	0.34	0.39	0.47	0.52	53.06
	250	0.27	0.33	0.41	0.48	0.52	0.59	60.20
50 °C	50	0.9	1	1	1	1	1	102.04
	100	1	1.2	1.5	1.5	1.5	1.5	153.06
	150	1.2	1.4	1.7	1.7	1.7	1.7	173.46
	200	1.5	1.9	1.9	1.9	1.9	1.9	193.87
	250	1.7	2	2	2	2	2	204.08

It was found that the best separation condition for the demulsifier F1 is a concentration of 250 ppm, at ideal temperature of 50°C with efficiency ratio of (204.08%) and suitable time of 40 minutes. It has different effects on breaking of crude oil interfacial film, with the increase in concentration of surfactant, but at a certain point, the film thinning rate and stability does not reduce (Feng *et al.* 2012). As a result, when determining the nature of the crude oil film, it is difficult to tell the amount of demulsifier (Atta *et al.* 2012).

4.8.2 Separation efficiency of (F2) as a demulsifier

The water separation efficiency of the (F2) demulsifier with crude oil for treating W/O emulsions was shown in Table 4.22. The percentage of best separation efficiency (% E Separation) at a temperature of 25 °C was (53.06 %). The concentration of demulsifier 250 ppm and the volume of separated water 0.52 ml was calculated. The percentage of separation at 50°C of water was 275.51% at 250 ppm of concentrations and separated volume of 2.7 ml was determined.

Table 4.22 Water and crude oil separated using a demulsifier (F2)

Temp. (°C)	**Con. ppm**	**Water Separated (mL)**						**% Sep. Effeciency**
		20 minutes	**40 minutes**	**60 minutes**	**80 minutes**	**100 minutes**	**120 minutes**	
25°C	50	0.13	0.19	0.25	0.3	0.37	0.41	41.83
	100	0.15	0.21	0.29	0.33	0.39	0.44	44.89
	150	0.16	0.21	0.27	0.34	0.42	0.45	45.91
	200	0.19	0.25	0.29	0.36	0.42	0.49	50
	250	0.21	0.27	0.33	0.40	0.47	0.52	53.06
50°C	50	1.1	1.4	1.7	1.7	1.7	1.7	173.46
	100	1.5	1.9	2	2.1	2.1	2.1	214.28
	150	1.5	1.9	2.1	2.3	2.3	2.3	234.69
	200	1.7	2	2.4	2.4	2.4	2.4	244.89
	250	1.8	2.1	2.6	2.7	2.7	2.7	275.51

It was found that the best separation process for the demulsifier F2 is a concentration of 250 ppm, the ideal temperature of 50°C and the separation efficiency ratio of 275.51% in 80 minutes. It was found that, F2 surfactant has different effects on breaking of interfacial film of crude oil with the increase in concentration of surfactant. However, it was observed that, at a certain point, the film thinning rate and stability did not be reduced (Feng *et al.* 2012).

4.8.3 Separation efficiency of (F3) as demulsifier

The water separation effeciency of Polymer F3 as demulsifier was shown in Table 4.23. on crude oil to treat W/O emulsions. The percentage of maximum separation efficiency

(% E) at 25 °C temperature, the concentration of demulsifier of 250 ppm and the volume of separated water was about 0.63 ml calculated. However, 64.28 percent efficiency of water separation was observed for 250 ppm of concentrations at 50 °C with 112.24 % and 1.10 ml of water was separated.

Table 4.23 Water and Crude Oil Separated Using a Demulsifier (F3)

Temp. (°C)	**Con. ppm**	**Water Separated (mL)**						**%Sep. Effeciency**
		20 minutes	**40 minutes**	**60 minutes**	**80 minutes**	**100 minutes**	**120 minutes**	
25 °C	50	0.05	0.09	0.1	0.19	0.24	0.31	31.63
	100	0.06	0.1	0.17	0.21	0.28	0.34	34.69
	150	0.1	0.17	0.23	0.29	0.34	0.39	39.79
	200	0.18	0.24	0.30	0.37	0.44	0.5	51.02
	250	0.3	0.39	0.46	0.51	0.58	0.63	64.28
50 °C	50	0.40	0.50	0.60	0.93	0.93	0.93	94.89
	100	0.30	0.60	0.65	0.89	0.92	0.93	94.89
	150	0.53	0.62	0.84	0.98	0.98	0.98	100
	200	0.55	0.62	0.79	0.90	1	1	102.04
	250	0.89	0.96	1.10	1.10	1.10	1.10	112.24

It was found that the best separation ability for F3 demulsifier with 250 ppm of concentration, at 50 °C of ideal temperature with in 60 minutes have maximum separation efficiency ratio of 112.24% was calculated. Moreover, different effects on breaking of interfacial film in crude oil was measured with increase in concentration of surfectant, however, it was found that at a certain point, the thinning rate of film and its stability did not reduce (Feng *et al.* 2012).

4.8.4 Studying the efficiency separation of (F4) as demulsifier

The separation efficiency of F4 polymer as demulsifier was measured and optimized data was obtained as given in Table 4.24 for the separation of water and effectiveness of the demulsifier was calculate. The W/O emulsions formed by crude oil was tested. However, it was found that the maximum separated volume of 0.5 ml water was measured at the concentration of 250 ppm of demulsifier at 25 °C of significant separation efficiency of 51.02%. Moreover, at 50°C, the percentage separation

efficiency at 250 ppm of concentrations was measured as 117.34% with removing volume of 1.15 ml of water.

Table 4.24 Water and crude oil separated using a demulsifier (F4)

Temperature	Con. ppm	Water Separated (mL)						%E Separation
		20 minute	**40 minute**	**60 minute**	**80 minute**	**100 minute**	**120 minute**	
25 °C	50	0.07	0.08	0.1	0.18	0.26	0.34	34.69
	100	0.08	0.1	0.15	0.2	0.26	0.37	37.75
	150	0.1	0.14	0.2	0.26	0.33	0.4	40.81
	200	0.14	0.21	0.29	0.35	0.4	0.47	47.95
	250	0.2	0.27	0.33	0.39	0.45	0.5	51.02
50 °C	50	0.30	0.40	0.60	0.96	0.97	0.97	98.97
	100	0.41	0.47	0.71	0.94	0.97	0.97	98.97
	150	0.47	0.66	0.81	0.99	0.99	0.99	101.02
	200	0.95	0.95	1	1	1	1.10	112.24
	250	0.88	0.99	1.10	1.15	1.15	1.15	117.34

It was found that the best concentration of demulsifier for separation of water was 250 ppm for F4 demulsifier, at ideal temperature of 50°C, and separation efficiency of 117.34 % with in 60 minutes. Moreover, different effects on destroying the interfacial film of crude oil were observed with the increase in concentration of demulsifier, however, after certain point, the thining rate of film and its stability stay unaffected (Feng *et al.* 2012).

4.9 Anti-Corrosion Activity of Polymeric Surfactants

4.9.1 Development of corrosive enviorment for carbon steel without inhibitor

The rate of corrosion of carbon steel was studied by applying weight loose method. According to the method, a gauge was used for determination of coorosion of carbon steel in developed environment. The main component of corrosive enviorment are listed below in Table 4.25. For studing the corrosion, first of all 0.5 M of hydrochloric acid solution was added over carbon steel material at 45°C, and data was analysed from (1-3 hours) by time. The results are given in Tables 4.26- 4.29 are summarized and data

showed that the intensity of corrosion of metal part accelerates with the passage of time (Ekott and Etukudo 2014).

Table 4.25 Chemical composition of (Carbon steel)

Composition in percentage (%)	**Calibration**	**Result**
C	0.240	0.253
Si	0.68	0.237
Mn	1.72	0.47
P	0.0272	0.0193
S	0.089	0.0136
Cr	16.77	0.0164
Mo	0.297	(C<)0.0070
Ni	9.65	0.0185
Al	0.0148	0.0074
Co	0.136	(C<)0.0110
Cu	0.41	0.0132
Nb	(C<)0.0110	(C<)0.0110
Ti	0.233	(C<)0.0020
V	0.073	(C<)0.0040
W	(C<)0.040	(C<)0.040
Pb	(C<)0.0110	(C<)0.0110
Sn	0.0131	0.0033
Mg	0.0206	0.0050
As	0.0238	0.0090
Zr	(C>)0.0070	(C>)0.0070
B	0.0023	(C>)0.0005
Fe	69.5	98.8

4.9.2 Development of corrosive enviorment for carbon steel in the presence of corrosive inhibitor

The corrosion rate of Carbon steel was placed in 0.5 M of HCl solution was investigated at 45 °C between 1-3 hours of time scale. In addition, (F1, F2, F3, and F4) as a corrosive inhibitor, synthesized polymeric surfactant was added over it. The rate of coorosion was measured against time. The experimental results were presented in Table 4.26-4.29, and Figure 4.25- 4.27 represent the inhibition percentage efficiency (IE%) of polymeric surfactant., and data was analysed by following the Equation (4.9) (Ali *et al.* 2005).

$$\%IE = [1-\frac{Wadd}{W\,free}]x100 \qquad (4.9)$$

Where, W_{add}= weight loss with inhibitors.and W_{free}= weight loss without inhibitors. However, for measurement of surface degradation after covering the surface layer was measured by Equation (4.10) given below:

$$\theta=[1-\frac{Wadd}{Wfree}] \qquad (4.10)$$

it was found that each inhibitor surfactant have unique effect on the material surface, with high level of effeciency. Moreover, it was found that, the inhibitors' potency of polymer increased over time.

4.9.3 Effect of concentration of corrosive inhibitor and their efficiency

Each inhibitor was studied at four different concentrations of (10, 20, 30, and 40) ppm. According to the findings, inhibition efficiency increases with increase in concentration up to a critical micelle concentration point (CMC). In addition, it was found that, efficiency of surfactant at the CMC point was more efficient, with slight or continuous change, the data about study is given in Table 4.26- 4.29 and Figure 4.25- 4.27.

Table 4.26 Inhibitor (surfactant – F1) corrosion test at 45 °C

t./hr	1 hr				2 hr				3 hr			
Conc. ppm	Wt. loss (g)	R_{corr} $g.cm^{-2}.hr^{-1}$	%IE	Ɵ	Wt. loss (g)	R_{corr} $g.cm^{2}.hr^{-1}$	%IE	Ɵ	Wt .loss(g)	R_{corr} $g.cm^{2}.hr^{-1}$	%IE	Ɵ
Blank	0.1214	$1.34*10^{-2}$	----	-----	0.082	$4.60*10^{-3}$	----	----	0.1037	$3.84*10^{-3}$	----	---
10	0.0904	$1.005*10^{-2}$	25.5	0.255	0.050	$2.80*10^{-3}$	38.9	0.389	0.0492	$1.82*10^{-3}$	52.5	0.525
20	0.0902	$1.003*10^{-2}$	25.6	0.256	0.050	$2.79*10^{-3}$	39.3	0.393	0.0490	$1.81*10^{-3}$	52.7	0.527
30	0.0901	$1.002*10^{-2}$	25.7	0.257	0.050	$2.78*10^{-3}$	39.5	0.395	0.0488	$1.80*10^{-3}$	52.9	0.529
40	0.0900	$1.003*10^{-2}$	25.8	0.258	0.049	$2.77*10^{-3}$	39.7	0.397	0.0486	$1.80*10^{-3}$	53.1	0.531

The data presented in Table 4.26, showed that, addition of surfactant F1 as corrosion inhibitor a promising result were obtained. The addition of 40 ppm of F1 anti crossion agent reduced the rate of corrosion with maximum efficiency of 53.1 after 2 hours, compared to blank where metel completely depleted. More over total weight loss observed after 2 hours was 0.0486 compared to blank with 0.1037 of weight loss. In addition, the rate of corrosion reduced to $1.80*10^{-3}$ after 2 hours compared to $3.84*10^{-3}$ of blank.

Table 4.27 Inhibitor (surfactant – F2) corrosion test at 45 °C

t./hr	1 hr				2 hr				3 hr			
Conc. ppm	Wt loss (g)	R_{corr} ($g.cm^{-2}.hr^{-1}$)	%IE	Ɵ	Wt .loss (g)	R corr ($g.cm^{-2}.hr^{-1}$	%IE	Ɵ	Wt. loss (g)	R corr ($g.cm^{-2}.hr^{-1}$)	%IE	Ɵ
Blank	0.1214	$1.34*10^{-2}$	----	----	0.0828	$4.60*10^{-3}$	----	-----	0.1037	$3.84*10^{-3}$	----	----
10	0.0730	$8.11*10^{-3}$	39.8	0.398	0.0428	$2.38*10^{-3}$	48.2	0.482	0.0496	$1.83*10^{-3}$	52.1	0.521
20	0.0729	$8.1*10^{-3}$	39.9	0.399	0.0426	$2.36*10^{-3}$	48.5	0.485	0.0493	$1.83*10^{-3}$	52.3	0.523
30	0.0727	$8.08*10^{-3}$	40.0	0.400	0.0423	$2.35*10^{-3}$	48.8	0.488	0.0491	$1.82*10^{-3}$	52.6	0.526
40	0.0725	$8.05*10^{-3}$	40.2	0.402	0.0421	$2.34*10^{-3}$	49.1	0.491	0.0489	$1.81*10^{-3}$	52.8	0.528

It was found from Table 4.27 that, F2 surfactant as anti-corrosive agent at 40 ppm of concentration gave promising results. The data collected after two hours showed that, the rate of corrosion $1.81*10^{-3}$ as compared to $3.84*10^{-3}$ control group. The loss of

weight for carbon steal was observed as 0.04891 after 2 hours compared to complete depletion of control group.

Table 4.28 Inhibitor (surfactant – F3) corrosion test results at 45 °C

t./hr	1 hr				2 hr				3 hr			
Conc. ppm	Wt. loss (g)	R corr g.cm^{-2}.hr^{-1})	%IE	Ɵ	Wt. loss (g)	R $_{corr}$ g.cm^{-2}.hr^{-1}	%IE	Ɵ	Wt. loss (g)	R $_{corr}$ g.cm^{-2}.hr^{-1}	%IE	Ɵ
Blank	0.1214	1.34*10^{-2}	---	---	0.0828	4.60*10^{-3}	----	-----	0.1037	3.84*10^{-3}	----	----
10	0.0910	1.011*10^{-2}	25.06	0.250	0.0512	2.84*10^{-3}	38.10	0.3810	0.0502	1.86*10^{-3}	51.5	0.515
20	0.0908	1.009*10^{-2}	25.18	0.251	0.0509	2.83*10^{-3}	38.50	0.3850	0.0499	1.85*10^{-3}	51.8	0.518
30	0.0906	1.006*10^{-2}	25.39	0.253	0.0506	2.81*10^{-3}	38.88	0.3888	0.0495	1.83*10^{-3}	52.2	0.522
40	0.0902	1.003*10^{-2}	25.67	0.256	0.0501	2.78*10^{-3}	39.42	0.3942	0.0492	1.82*10^{-3}	52.5	0.525

Similar results were observed after inhibition of corrosion with F3 surfactent polymer at 40 ppm of concentration as shown in Table 4.28. the data showed that, rate of corrosion was reduced to 1.82*10^{-3} with F3 inhibition at 40 ppm till 2 hours comare to 3.84*10^{-3} rate of control group. Moreover, the weight loss observed for carbon steel at 40 ppm was 0.04921 with maximum inhibition efficiency of 52.56 compare to control group, which completely depleted with no efficiency of inhibition from corrosion.

Table 4.29 Inhibitor (surfactant – F4) corrosion test results at 45 °C

t./hr	1 hr				2 hr				3 hr			
Conc ppm	Wt. loss (g)	R corr g.cm^{-2}.hr^{-1}	%IE	Ɵ	Wt. loss(g)	R $_{corr}$ g.cm^{-2}.hr^{-1}	%IE	Ɵ	Wt. loss(g)	R $_{corr}$ g.cm^{-2}.hr^{-1}	%IE	Ɵ
Blank	0.1214	1.34*10^{-2}	-----	-----	0.0828	4.60*10^{-3}	-----	-----	0.1037	3.84*10^{-3}	-----	----
10	0.0749	8.32*10^{-3}	38.3	0.383	0.0508	2.82*10^{-3}	38.66	0.3866	0.0422	1.56*10^{-3}	59.30	0.593
20	0.0745	8.28*10^{-3}	38.59	0.385	0.0504	2.80*10^{-3}	39.05	0.3905	0.0418	1.55*10^{-3}	59.64	0.596
30	0.0740	8.23*10^{-3}	39.0	0.390	0.0501	2.78*10^{-3}	39.44	0.3944	0.0414	1.53*10^{-3}	60.04	0.6004
40	0.0738	8.21*10^{-3}	39.1	0.391	0.0499	2.77*10^{-3}	39.75	0.3975	0.0410	1.52*10^{-3}	60.40	0.604

Similar results were observed after inhibition of corrosion with F4 surfactent polymer at 40 ppm of concentration as shown in Table 4.29. the data showed that, rate of corrosion was reduced to 1.52*10^{-3} with F4 inhibition at 40 ppm till 3 hours comare to 3.84*10^{-3} rate of control group. Moreover, the weight loss observed for carbon steel at 40 ppm

was 0.04108 with maximum inhibition efficiency of 60.40 compare to control group, which completely depleted with no efficiency of inhibition from corrosion.

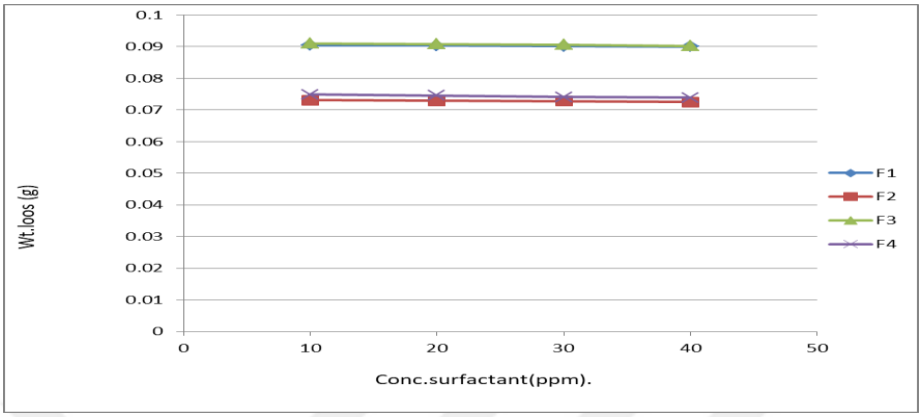

Figure 4.25 Graphical representation of weight loss (g) against Conc. Of surfactant in the range of 10-40 ppm at 45 °C for an hour

The weight loss against inhibition concentration of surfactants were ploted in Figure 4.25. Data presented in plot was collected after 1 hour of reaction at 45 °C at 10, 20, 30 and 40 ppm concentration of F1, F2, F3, and F4 surfactant. It was observed that, F1, F2 and F4 were almost protective t same rate of inhibition and observable weight loss was less then F3 corrosion inhibitor.

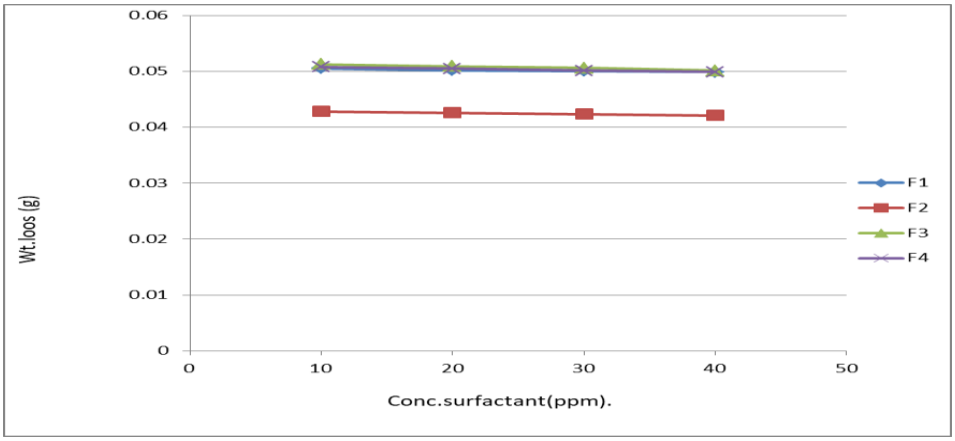

Figure 4.26 Graphical representation of weight loss (g) against Conc. Of surfactant in the range of 10-40 ppm at 45 °C for 2 hours

However, there was a variation observed after 2 hour at same temperature against same amount of corrosion inhibition agent. It was found the weight loss of metal was reduced with F2 compare to other surfactant polymer as shown in Figure 4.26.

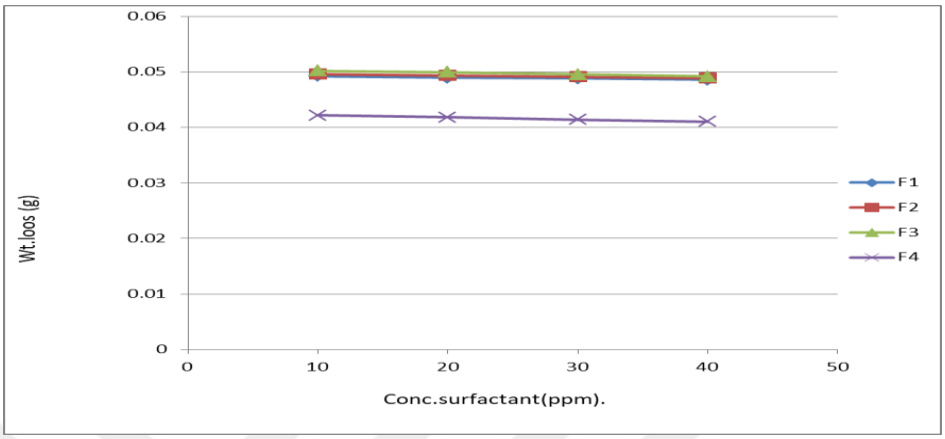

Figure 4.27 Graphical representation of weight loss (g) against Conc. Of surfactant in the range of 10-40 ppm at 45 °C for 3 hours

However, a change was observed again after passage of time as shown in Figure 4.27. it was seen that, F4 was more affective compare to other corrosion inhibitor. F4 was mor affective to inhibit the processs of corrosion and therefor there was reduction in weight loss.

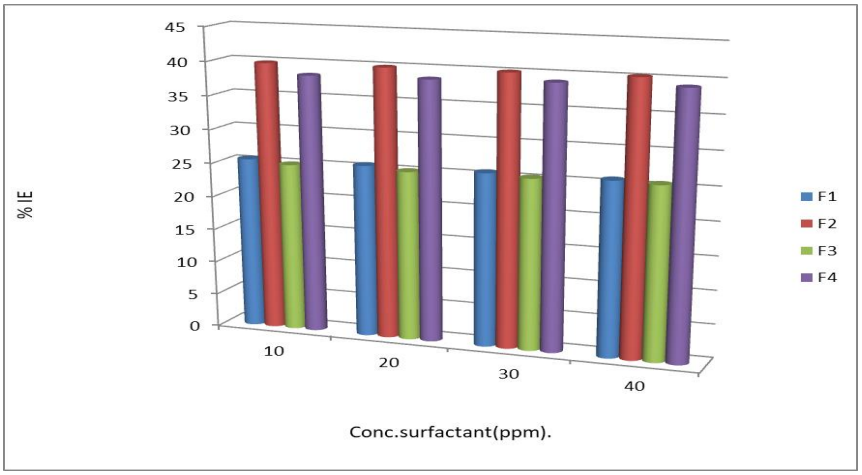

Figure 4.28 Inhibition efficiency of surfactant against different Conc. Of surfactant between 10-40 ppm at 45 °C for an 1 hour.

A graph belong to inhibition efficiency of all surfactant was ploted (Figure 4.28) against concentration values of corrosion inhibitor. At different concentration of 10, 20, 30 and 40 ppm, it was found that, F2 and F4 showed maximum inhibition efficiency compare to F1 and F2 at 45 °C and 1 hour time period.

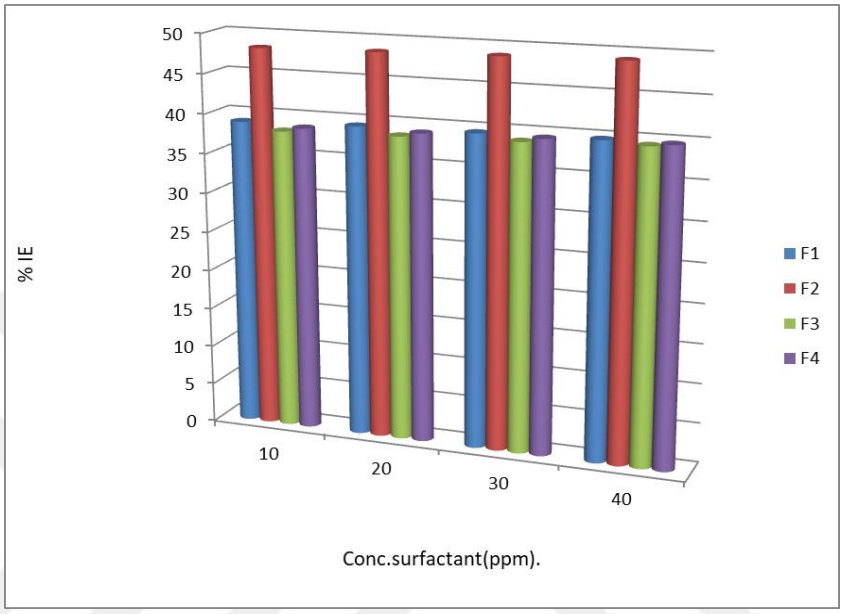

Figure 4.29 Inhibition efficiency of surfactant against different Conc. Of surfactant between 10-40 ppm at 45 °C for 2 hours

The graphical representation of results obtained after 2 hours of experiment at 45 °C, the efficiency of inhibitor F2 was found maximum compare to all other F1, F3 and F4 experiment against corrosion inhibition as shown in Figure 4.29.

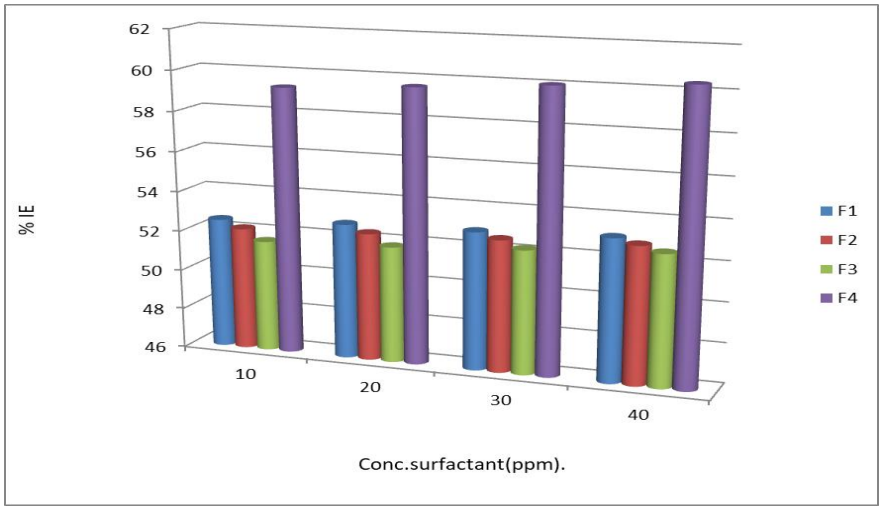

Figure 4.30 Inhibition efficiency of surfactant against different Conc. Of surfactant between 10-40 ppm at 45 °C for 3 hours.

A surprising result were obtained after 3 hours of reaction completion at 45°C for all surfactant. It was found that F1 have maximum efficiency of inhibitor compare to all other surfactants. However, this strange event takes a long time of 3 hours duration. Therefore, overall data analysis showed that, F2 surfactant as corrosion inhibitor was more efficient even in short time scale, and low dose.

5 . CONCLUSIONS AND RECOMMENDATION

5.1 Conclusion

In conclusion, it was found that, polymeric surfactants have potential as demulsifiers and anti-corrosive agent. Moreover, our synthesized demulsifiers have a lower impact on crude oil properties compared to commercially available demulsifiers. In addition, it was found that, our synthesized demulsifier have high efficiency, while dealing with crude oil emulsions. According to the finding of our study, the synthesized polymeric surfactants can be used as effective corrosion inhibitors. Moreover, very economic, easy to synthesized and have high efficiency compared to commercially available anticorrosive materials. Hence, our designed polymeric surfactant improved the crude oil specification.

5.2 Recommendations

1. There are possibilities of expanding the study in terms of treating industrial wastewater through prepared demulsifiers such as oil stains demulsification in water contributes to reducing water pollution.
2. It is possible to get benefits from this study by applying them on bacteria which adversely affect crude oil properties.
3. It can provide support for removel or reduction of excessive elements from raw materials such as reduction in content of sulfur in crude oil.
4. It gives new dimension of synthetic chemistry for the engineering of synthetic material and its application in various sectors of industrial fields.

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