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Effect of Temperature and Concentration of Demulsifier on Crude Oil of Emulsion Break

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Abstract
Produced crude is associated with aqueous salt solutions, particularly sodium chloride in the form of emulsions. It is also a fact that the presence of chloride and water in emulsions (in crude) presents several problems during recovery as well as during crude processing. Hence, this study aimed at investigating and evaluating the concentration of the demulsifierconcerningtemperature that will be effective to break the emulsion. A mixture of 50 mls to 50mls of crude and freshwater was used under different temperatures range of 80°F (29°C), 120°F (49°C), and 150°F (66°C) respectively, and concentrations of 0, 0.5, and 1.0ml of demulsifier were added at intervals of 5 and 10 minutes continuously until total separation was observed. Analysis of results obtained shows that sludge was associated with water and oil at a temperature of 80°F and 120°F at different time intervals whereas, at a temperature of 150°F, there was an absence of sludge at different time intervals. Furthermore, analyses of results also showed that an increase in temperature led to a gradual destabilization of the crude oil/water interfacial films. Therefore, the higher the temperature the better the separation of the water component from the emulsion,and the higher the concentration of surfactant in the emulsion, the better the water is separated from the emulsion.

Keywords: *Demulsifier, temperature, surfactant, destabilization, sodium chloride*

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I. Introduction

Petroleum produced from the depths of the earth is associated with water or aqueous solutions of salts, particularly sodium chloride, in the form of emulsions. Emulsions are also produced during the processing of crude and also during spills, especially over waters. The presence of water in emulsions (in crude) presents several problems during processing as well as during recovery-such as the increased viscosity and hence increased pumping costs, pipeline corrosions, hampering of skimmer operations, increased handling of oily waste disposal and storage problems, etc. An emulsion is an intimate mixture of two liquid phases, such as oil and water,in which the liquids are mutually insoluble and where either phase may be dispersedin the other. Also, Becher, P. (1965), defined ‘‘an emulsion as a heterogeneous system, consisting of at least one immiscible liquid intimately dispersed in another in the form of droplets, whose diameters, in general, exceed 0.1mm. Such systems possess minimal stability, which may be accentuated by such additives as surface-active agents (surfactants), finely-divided solids, etc.’’ In this respect, surfactants may be present as monolayer or liquid crystals; polymers are another class of emulsifiers. There are two categories of simple emulsions namely: oil-in-water (O / W) and water-in-oil (W/ O). There are also two kinds of multiple (double) emulsions: W/O/W and O/W/O. The so-called aqueous phase may contain inorganic or organic solutes and the oily phase, often a mixture of species can be of mineral, vegetable, or animal origin. In water chemistry, two types of emulsions are commonlyfound, oily wastewater (oil emulsified in water or O/W emulsions) and waste oil emulsions (water emulsified in oil or W/O emulsions).Oily waste and waste oil emulsions can usually be differentiated visually.O/W emulsion appears to be just oily, dirty water; a drop of the emulsion addedto water disperses as shown in Figure 1(a). A W/O emulsion is usually thick and viscous; a drop of this emulsion added to water does not disperse as shown in Figure 1(b). It also happens that emulsions, desirable or not, are formed temporarily during industrial processes (e.g., polymerization, oil extraction).

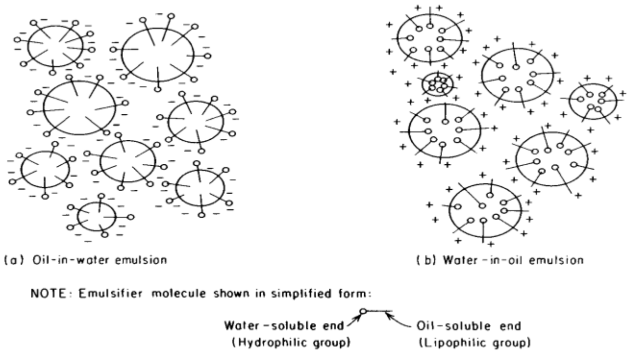

Figure 1: Chemical stabilization by surfactants having hydrophilic and lipophilic groups(.Becher, P. (1965).

From a thermodynamic point of view, an emulsion is an unstable system because there is a natural tendency for a liquid/liquid system to separate and reduce its interfacial area and, hence, its interfacial energy. However, most emulsions demonstrate kinetic stability (i.e., they are stable over some time). Produced oilfield emulsions are classified based on their degree of kinetic stability which includes:Loose emulsions separate in a few minutes, and the separated water is free; medium emulsions separate in tens of minutes; and tight emulsions separate (sometimes only partially) in hours or even days. These emulsions may contain as much as 80% water and are frequently extremely stable due to the presence in the emulsion of a variety of emulsifiers such as asphaltenes.

According to Auflem (2002), asphaltenes are the heaviest and most polar fraction in crude oil and it plays a leading role in causing a variety of nuisances such as the stabilization of the water-in–oil-emulsion that occurs during crude oil production. They tend to adsorb at water-in-crude oil interfaces to form a rigid film surrounding the water droplets and protect the interfacial film from rupturing during droplet-droplet collisions, giving rise to the formation of particularly stable water-in-crude-oil emulsion (Sjoblom, *et al*, 1990). Hence it is necessary to remove the water before processing and handling.

Selvarajan, *et al*, (2001) simply defined de-emulsification as the breaking or destabilization of crude oil emulsions to separate them into two clear immiscible phases i.e. oil and water phases. Of the four principal methods- mechanical, chemical, electrical and thermal, chemical de-emulsification is generally the most suitable method to break crude oil emulsions from both the operational and economic points of view. This is done by treating the emulsions with de-emulsifiers under different conditions of temperature and concentrations. The choice of de-emulsifiers to be used for a particular oil is usually from a selection of what has been used for other crudes. De-emulsifiers proposed for use may include organic substances such as sulphonates, polyglycol ethers, and oxylated phenols, e.g. alkanolamine and nonylphenol ethoxylate derivatives (Easton, *et al* 1989) relatively recent additions include the organosiloxanes which are regarded as the most efficient for crude oil de-emulsification.

De-emulsifiers are interfacial-active agents which weaken the stabilizing films to enhance droplet coalescence. They are preferred to other chemical agents due to the lower addition rates needed. The separation rate of W/O emulsion is usually a function of the de-emulsifier used, the emulsion stability, the temperature, the concentration, the process residence time, and the mixing energy (Sunil, 2006). Because crudes vary in composition according to their location, the best de-emulsifier for one crude sample may not be the same for another. Another factor affecting the performance of the de-emulsifier is the water content of the emulsion. Therefore, it is imperative to have an understanding of the behavior of emulsion breakers under different temperatures and concentrations to time duration because of the prevalent challenges associated with production operations. It is obvious that crude produced from the depths of the earth is associated with aqueous fresh or salt solutions, particularly sodium chloride in the form of emulsions; the presence of chloride and water in emulsions (in crude) presents several problems during processing as well as during recovery. It is therefore important that the concentration of the de-emulsifier concerning a temperature that will be cost-effective to break the emulsion be evaluated.

Oil-in-Water Emulsions

An oily waste emulsion is a type of emulsion where oil is dispersed in the water phase, it may contain any of various types of oil in a wide range of concentrations. These oils are defined as substances that can be extracted from water by hexane, carbon tetrachloride, chloroform, or fluorocarbons. In addition to oils, typical contaminants of these emulsions may be solids, silt, metal particles, emulsifiers, cleaners, soaps, solvents, and other residues. The types of oils found in these emulsions will depend on the industry. They may be fats, lubricants, cutting fluids, heavy hydrocarbons such as tars, grease, crude oils, and diesel oils, and also light hydrocarbons including gasoline, kerosene, and jet fuel. Their concentration in the wastewater may vary from only a few parts per million to as much as 5 to 10% by volume.

A stable O/W emulsion is a colloidal system of electrically charged oil droplets surrounded by an ionic environment. Violent mixing and shearing of oily wastewater in transfer pumps disperses these minute oil droplets throughout the water.

Emulsion stability is maintained by a combination of physical and chemical mechanisms. These emulsions are similar in behavior to the colloidal systems encountered in swamps (color) and rivers (silt). One such stabilizing mechanism, ionization, is brought about by the addition of surface-active agents, such as organic materials or cleaners, which aid in maintaining a stable colloidal system. These molecules usually carry an electric charge and seek out the oil/water interface of the emulsified droplet. Here, the accumulated charges cause the emulsion to be stabilized through the repulsion of the commonly charged droplets. Neutral (nonionic) surfactants can also stabilize an emulsion since these molecules are bifunctional: one end is soluble in water, and the other end is in hydrocarbon, so the molecule bridges the interface and stabilizes it.

Emulsions can also be stabilized by friction between the oil and water phases created by vigorous mechanical or physical agitation. Static electric charges developed by this action tend to collect at the oil/water interface. An emulsifier is usually a complex molecule, often having a hydrophilic (water-loving) group at one end and a lyophilic (oil-loving) group at the other as shown in Figure 2. Emulsifiers disperse oil droplets in the water phase because they have an affinity for both water and oil that enables them to overcome the natural forces of coalescence. Most emulsifiers are surfactants having either anionic or nonionic polar groups. Petroleum sulfonates and sulfonated fatty acids are common anionic emulsifiers, and ethoxylated alkyl phenols are common nonionic emulsifiers. Examples of naturally occurring surfactants are organic sulfur compounds, various simple esters, and metal complexes. Alkaline cleaners containing surfactants

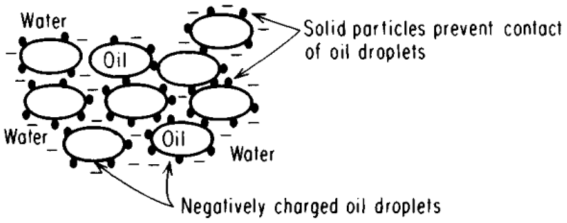

Figure 2: Physical emulsion stabilization by finely divided solids. Illustrated with O/W emulsion (Becher, P. (1965)

Mechanisms involved in Demulsification

Demulsification is the breaking of a crude oil emulsion into oil and water phases. From a process point of view, the oil producer is interested in three aspects of demulsification which include the rate or speed at which this separation takes place**, the a**mount of water left in the crude oil after separation,and the quality of separated water for disposal.

A fast rate of separation, a low value of residual water in the crude oil, and a low value of oil in the disposal water are desirable. Produced oil generally has to meet company and pipeline specifications. For example, the oil shipped from wet-crude handling facilities must not contain more than 0.2% basic sediment and water (BS&W) and 10 pounds of salt per thousand barrels of crude oil. This standard depends on the company and pipeline specifications. The salt is insoluble in oil and is associated with the residual water in the treated crude. Low BS&W and salt content are required to reduce corrosion and deposition of salts. The primary concern in refineries is to remove inorganic salts from the crude oil before they cause corrosion or other detrimental effects on refinery equipment. The salts are removed by washing or desalting the crude oil with relatively fresh water.

Methods of emulsion breaking or demulsification

Crude oil emulsions must be separated almost completely before the oil can be transported and processed further. Emulsion separation into oil and water requires the destabilization of emulsifying films around water droplets. This process is accomplished by any, or a combination, of the following methods: adding chemical demulsifiers, increasing the temperature of the emulsion, applying electrostatic fields that promote coalescence, and reducing the flow velocity that allows gravitational separation of oil, water, and gas. This is generally accomplished in large-volume separators and desalters.

Chemical methods

The chemical method is the introduction of demulsifiers for emulsion treatment and it is the most common method widely used. These chemicals are designed to neutralize the stabilizing effect of emulsifying agents. Demulsifiers are surface-active compounds that, when added to the emulsion, migrate to the oil/water interface, rupture or weaken the rigid film, and enhance water droplet coalescence. Optimum emulsion breaking with a demulsifier requires a properly selected chemical for the given emulsion; an adequate quantity of this chemical; adequate mixing of the chemical in the emulsion; and sufficient retention time in separators to settle water droplets. It may also require the addition of heat, electric grids, and coalescers to facilitate or completely resolve the emulsion.

Chemical selection

Some researchers have stated that the selection of the right demulsifier is crucial to emulsion breaking (Bobra, M. 1990; Kokal, S. et al 1999; Svetgoff, J.A. 1989; Eley, D.D., et al 1988 and Kokal, S.L et al 1995). The selection process for chemicals is still viewed as an art rather than a science. However, with the increasing understanding of emulsion mechanisms, the availability of new and improved chemicals, and new technology, research, and development efforts, the selection of the right chemical is becoming more scientific. Many of the failures of the past have been eliminated. Demulsifier chemicals contain the following components such as solvents, surface-active ingredients, and flocculants.

- Solvents, such as benzene, toluene, xylene, short-chain alcohols, and heavy aromatic naphtha, are generally carriers for the active ingredients of the demulsifier. Some solvents change the solubility conditions of the natural emulsifiers (e.g., asphaltenes) that are accumulated at the oil/brine interface. These solvents dissolve the indigenous surface-active agents back into the bulk phase, affecting the properties of the interfacial film that can facilitate coalescence and water separation.
- Surface-active ingredients are chemicals that have surface-active properties characterized by hydrophilic-lipophilic balance (HLB) values. The HLB scale varies from 0 to 20. A low HLB value refers to a hydrophilic or water-soluble surfactant. In general, natural emulsifiers that stabilize a water-in-oil emulsion exhibit an HLB value in the range of 3 to 8 Kimbler, O.K., et al (1966). Thus, demulsifiers with a high HLB value will destabilize these emulsions. The demulsifiers act by total or partial displacement of the indigenous stabilizing interfacial film components (polar materials) around the water droplets. This displacement also brings about a change in properties such as interfacial viscosity or elasticity of the protecting film, thus enhancing destabilization. In some cases, demulsifiers act as wetting agents and change the wettability of the stabilizing particles, leading to a breakup of the emulsion film.
- Flocculants are chemicals that flocculate the water droplets and facilitate coalescence. A detailed process for selecting the appropriate demulsifier chemicals includes the following steps:
- Characterization of the crude oil and contaminants includes the API gravity of the crude oil, type, and composition of oil and brine, inorganic solids, amount and type of salts, and contaminant type and amounts.
- Evaluation of operational data includes production rates, treating-vessel capabilities (residence time, electrostatic grids, temperature limitations, etc.), operating pressures and temperatures, chemical dosage equipment and injection points, sampling locations, maintenance frequency, and wash-water rates.
- Evaluation of emulsion-breaking performance: experience and operating data including oil, water, and solids content during different tests; composition and quality of interface fluids; operating costs; and amounts of water generated and its disposal. According to Bobra, M. (1990), testing procedures are available to select appropriate chemicals and these tests include Bottle tests; Dynamic simulators, and Actual plant tests.

Mixing/agitation

For the demulsifier to work effectively, it must make intimate contact with the emulsion and reach the oil/water interface. Adequate mixing or agitation must be provided to thoroughly mix the chemical into the emulsion. This agitation promotes droplet coalescence; therefore, the point at which the demulsifier is added is critical. Once the emulsion has broken, agitation should be kept to a minimum to prevent re-emulsification. There should be sufficient agitation in the flow stream to allow the chemical to mix thoroughly, followed by a period of gentle flow inside the separator to promote gravity separation.

Dosage

The demulsifier's effectiveness also depends on its dosage. An increase in demulsification rate is generally observed with increasing demulsifier concentration up to a critical concentration (the critical aggregation concentration). This is attributed to the monolayer adsorption of the demulsifier at the interface (simultaneously displacing the indigenous crude oil surfactant film). Eley, D.D. et al (1988) and Davies, G.A. et al (1996) noted that higher concentrations beyond this critical concentration (overdosing) result in different types of behavior.

The solubility of the demulsifier in oil and water, or its partitioning, is also very crucial in determining the effectiveness of the demulsifier. The partitioning of the surfactant is measured either by the partition coefficient or by its HLB value. Several studies (Svetgoff, J.A. 1989; Tambe, D.E. et al 1993; Menon, V.B. et al 1988; Coppel, C.P. 1975 and Kokal, S. et al 2000) have tried to link the demulsifier effectiveness to its partition coefficient. For the demulsifier to be fairly active, it must be an amphiphile with a partition coefficient of unity Menon, V.B. et al 1988; Coppel, C.P. 1975 (i.e., the demulsifier should partition equally between the oil and water phases). The surface adsorption rate is faster when the demulsifier has a partition coefficient of close to one. Because of this criterion and the fact that demulsifiers are added to the continuous oil phase, demulsifiers that are soluble in water only (low partition coefficient or low HLB) are not very effective in breaking water-in-oil emulsions.

However, the amount of chemicals added is also important. Too little demulsifier will leave the emulsion unresolved. Conversely, a large dose of demulsifier (an overtreat condition) may be detrimental. Because demulsifiers are surface-active agents like emulsifiers, excess demulsifiers may produce very stable emulsions. The demulsifier simply replaces the natural emulsifiers at the interface. Furthermore, some of the chemicals come in different concentrations (the active ingredient in a carrier solvent). The amount or dosage of demulsifier required is very site-specific and depends on several factors, some of which are discussed in this chapter. Based on an evaluation of the literature, the demulsifier rates quoted vary from less than 10 to more than 100 ppm (based on total production rates). These numbers are provided for primary or secondary oil-recovery emulsions. During tertiary oil recovery (especially during surfactant or micellar flooding), demulsifier rates typically can be in the hundreds of ppm and even higher in extreme cases.

Materials and Procedure

This study was aimed at evaluating the effects of temperatures and concentrations on the emulsionbreak of crude oil. A crude oil emulsion sample for this work was obtained from an oil company situated in Rivers State, Nigeria. A commercial de-emulsifier as referred to as D^Z, (used as the standard for comparison), was also obtained from the same company.

The apparatus used includeda 100ml measuring cylinder, 50ml beaker, surfactant (De-emulsifier), treated water, regulated heater, water bath,and stopwatch.

Procedure

The procedure used in achieving the aim of this study includes: 50 ml of crude oil was mixed with 50 ml of freshwater and allowed to stand. Thereafter, a 0.5ml increment of demulsifier was added at intervals of 5 minutes and 10 minutes, concerning different temperatures continuously until total separation was observed.The interfaces between oil and water; water and sludge were recorded in ml. The processes were repeated continually at varied concentrations of demulsifier and varied temperature increases until the total separation of crude oil and water at equal volume occurred clearly without any sludge

II. Results Presentation and Analysis

To evaluate the effect of temperatures and concentrations on emulsionbreak; water and crude oil at a ratio of 50ml to 50ml were vigorously mixed to obtain a homogenous mixture. 0.5ml increment of demulsifier was also added at stipulated intervals.

Figure 3 to **Figure 6** shows the effect of temperatures on emulsion treatment for surfactantsat different concentrations. It can be observed from (**Figure 3** to **Figure 6**) that the percentage separation of the oil component from the emulsion decreases with an increase in temperature for a 0.5ml concentration of surfactant at a retention time of 5 minutes (**Figure 3**), while the percentage separation of the water component from the emulsion decreases as the temperature increases. The sludge component of the emulsion decreases with an increase in temperature for the same amount of surfactant and retention time.

A similar observation was noted in (**Figure 4)** between the separation percentages of the emulsion and the temperature when the 0.5ml concentration of surfactant was used but the retention time increased to 10 minutes. When the concentration of the surfactant was increased to 1ml(**Figure 5**), the trend observed with 0.5ml concentration was still noted for 5 minute retention time. For 10 minute retention time (**Figure 6**), there was no sludge observed, as the emulsion was made up of water and oil only, but a similar trend was observed for the percentage separation and the temperature.

Conversely, the results have shown that the temperature of the emulsion affects the percentage separation of the emulsion such that the higher the temperature of the emulsion the better the separation of the water component from the emulsion.

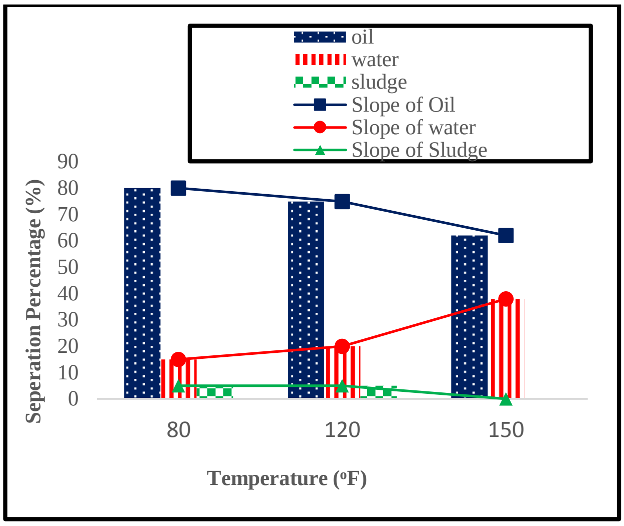

Figure 3 Effect of temperature on emulsion treatment for 0.5ml surfactant for a 5-minute duration

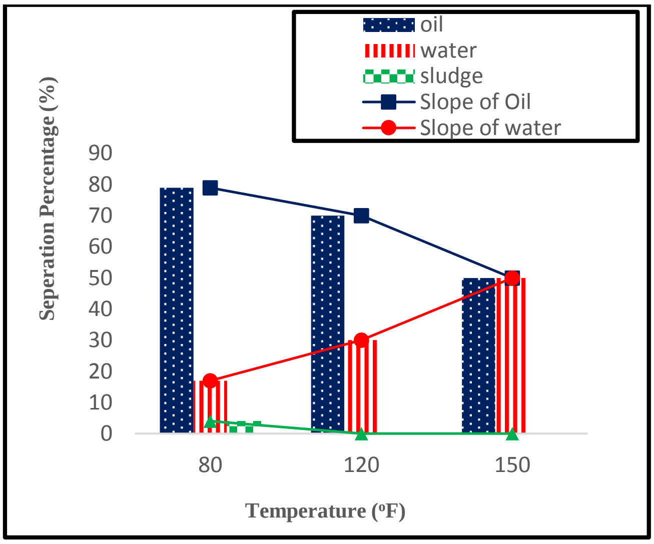

Figure 4 Effect of temperature on emulsion treatment for 0.5ml surfactant for a 10-minute duration

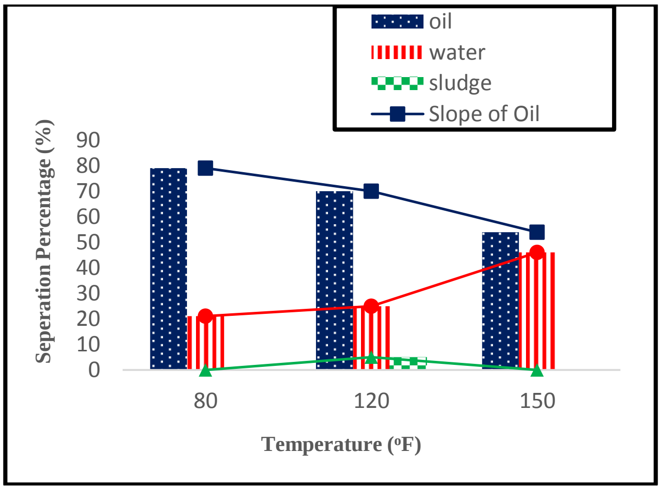

Figure 5 Effect of temperature on emulsion treatment for 1ml surfactant for a 5-minute duration

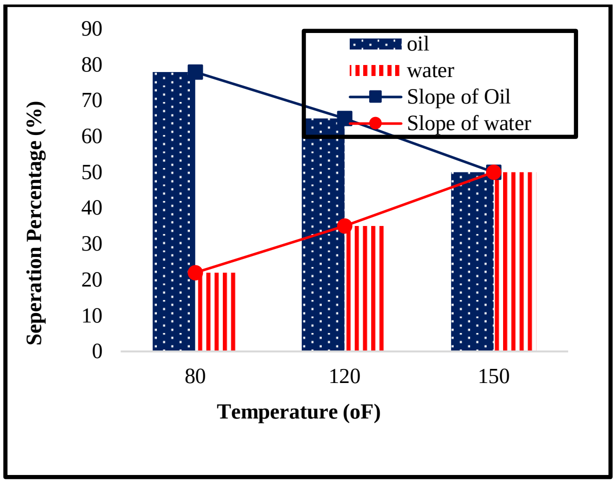

Figure 6 Effect of temperature on emulsion treatment 1ml surfactant for the 10-minute duration

Similarly, **Figure 7** to **Figure 12** showed the effect the level of concentration has on the percentage of separation. It was observed that the higher the concentration of surfactant in the emulsion, the better the water is separated from the emulsion. This could be observed for all cases apart from **Figure 11**, where the amount of concentration did not affect the percentage separation.

It was also observed that at 0.5ml of surfactant, the separation percentage of water was 50% and at 1ml surfactant, the level of water separation from the emulsion was still 50%. This observation was only noticed at the emulsion temperature of 150°F and the retention time of 10 minutes.

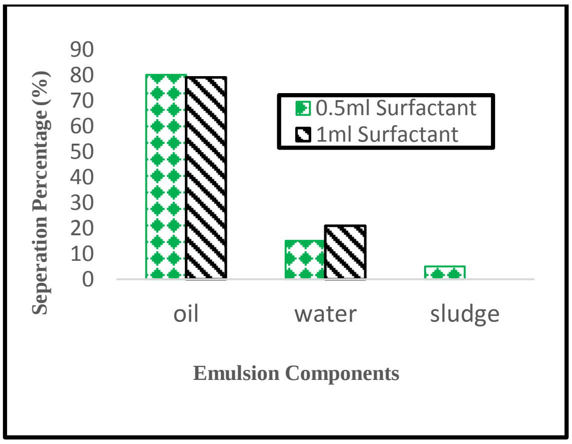

Figure 7 Effect of concentration of Surfactant on emulsion treatment for a temperature of 80°F for a 5-minute duration

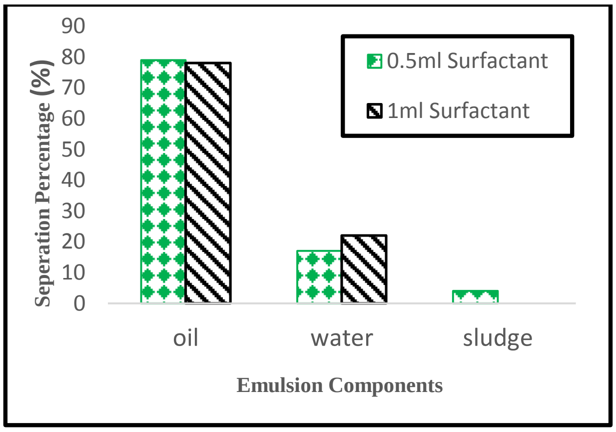

Figure 8 Effect of Concentration of Surfactant on emulsion treatment for a temperature of 80°F for a 10-minute duration

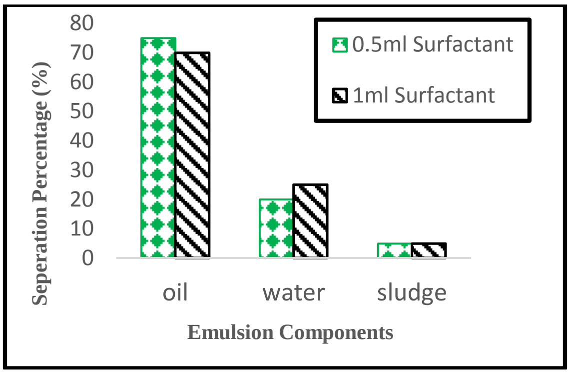

Figure 9 Effect of Concentration of Surfactant on emulsion treatment for a temperature of 120°F for a 5-minute duration

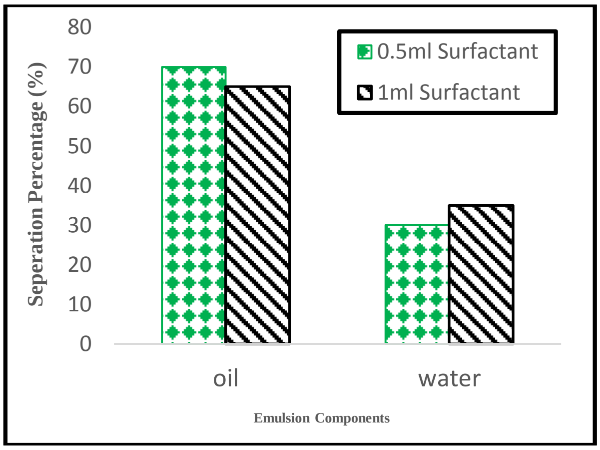

Figure 10: Effect of Concentration of Surfactant on emulsion treatment for a temperature of 120°F for a 10-minute duration

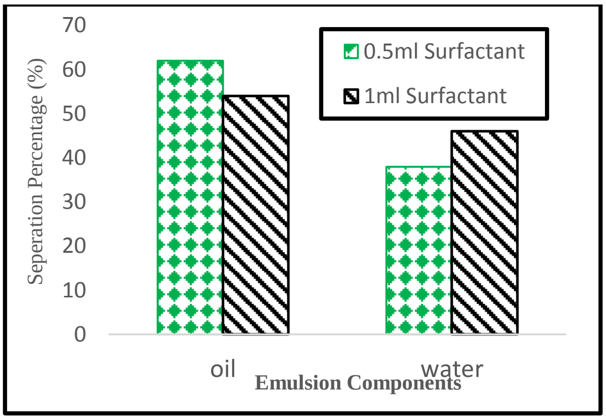

Figure 11 Effect of Concentration of Surfactant on emulsion treatment for a temperature of 150°F for a 5-minute duration

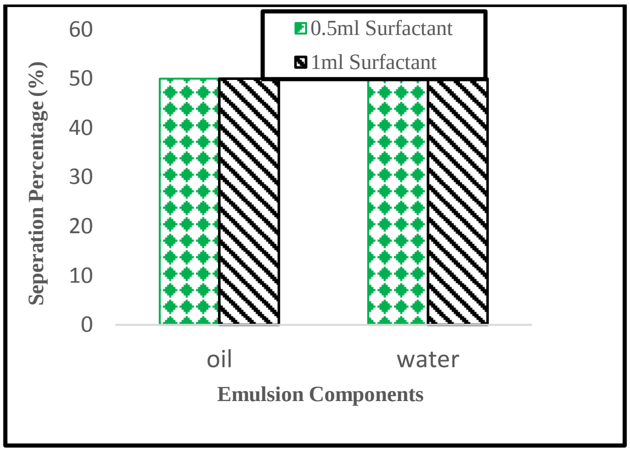

Figure12 Effect of Concentration of Surfactant on emulsion treatment for a temperature of 150°F for a 10-minute duration

III. Conclusion

An experiment aimed at evaluating the effect of temperature and demulsifier on crude oil of emulsion break has been carried out in the laboratory. However, based on the results obtained,the following conclusion is drawn as follows:

- Analyses of results have shown that an increase in temperature led to a gradual destabilization of the crude oil/water interfacial films meaning temperature aids in emulsion break.

- There was a clear separation emulsion with an increase in the concentration of the demulsifierover time.
- It is essential to have an understanding of the efficacy of demulsifiers on emulsion break to mitigate challenges associated with production operations.
- However, a lot of production problems can be avoided if the optimum values of concentrations are known at a given temperature.

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Table 1 Effect of de-emulsifier concentration and separation time on the performance

S/N	**Surfactant (ml)**	**Temperature(°F)**	**Time (mins)**	**Observation**	**Break**	**Interface**	**Sediment**
1.	**0.5**	**80**	**0**	Separation begins slowly	Not clear	Not clear	None
			5	Oil =80mls Water =15mls Sludge =5ml	Not clear	Not clear	None
			10	Oil =79mls Water =17mls Sludge = 4ml	Not clear	Clear	None
	1.0		**0**	Separation starts	Not clear	Not clear	None
			5	Oil =79mls Water =21mls Sludge = 0	Not clear	Not clear	None
			10	Oil =78mls Water =22mls	Not clear	Clear	None
S/n	**Surfactant (ml)**	**Temperature(°F)**	**Time (mins)**	**Observation**	**Break**	**Interface**	**Sediment**
2.	**0.5**	**120**	**0**	Separation begins	Clean	Clean	None
			5	Oil =75mls Water =20mls Sludge = 5	Clean	Clear	None
			10	Oil =70mls Water =30mls	Clean	Clear	None
	1.0		**0**	Separation starts	Clean	Clean	None
			5	Oil =70mls Water =25mls Sludge =5mls	Very clean	Very clear	None
			10	Oil =65mls Water =35mls	Very clean	Very clear	None
3.	**0.5**	**150**	**0**	Separation starts	Clean	Clean	None
			5	Oil =62mls Water =38mls Sludge = 0	Very clean	Very clear	None
			10	Oil =50mls Water =50mls	Very clean	Very clear	None
	1.0		**0**	Separation starts	Very clean	Very clear	None
			5	Oil =54mls Water =46mls	Very clean	Very clear	None
			10	Oil =50mls Water =50mls	Very clean	Very clear	None