### **Treatment of Crude Oil Emulsion Using Locally Made Demulsifiers**

### Oloro John

Delta State University, Abraka, Delta State, Nigeria.

#### Abstract

Life of most oil wells, produces more water with the crude oil than acceptable standard to the producer or customer. This may be due to the perforation depth at that point in time which allows water to enter the well. In some cases, it is impossible to prevent water contamination regardless of its source. Some of the water produced combines with the oil to form an emulsion, hence requiring special methods to separate them.

In this study ,treatment of crude oil emulsion using locally made emulsifiers was carried out by formulation of an emulsion breaking chemical or demulsifier in the laboratory from materials obtained locally and the experimental investigation of its effectiveness in breaking crude oil emulsion was also . Materials used include Liquid soap, starch, camphor, alum, and castor oil. Two different demulsifier was formulated and tested on the crude oil emulsion sample from umusadege field and was treated at a temperature of  $50^{\circ C}$ .

Foreign demulsifiers, Separol and servo of the same quantity and under the same experimental condition, served as a basis for comparison. The result of the treatment was a successful separation of oil and water. The separated water volume of umusadege well 1 after treating for 120minutes by LD1 was 46ml and when the foreign demulsifier (separol) was used the separated volume of water was 0ml. Also umusadege well 2, after treating for 120minutes by LD1 the volume of water separated was 136ml, while when separol was used the volume of water separated was 49ml and servo was105ml.

Keywords: Demulsifier, formulation, Treatment, emulsion List of Abbreviations

API	-	American Petroleum Institute
BS & V	V -	Basic Sediment and Water
LD1	-	Local Demulsifier Formulation 1
LD2	-	Local Demulsifier Formulation 2
O.T.R	-	Obtained T-ratio
T.T.R	-	Tabled T-ratio

#### **1.0** Introduction

Crude oil is a complex fluid containing asphaltenes, resins and napthenic acid. Asphaltenes is the heaviest and most polar fraction in the crude oil and responsible in rising up the variety of nuisances and stabilized the water in oil emulsion that occurred during crude oil production. It is widely known that deposition and flocculation of asphaltenes may be occurred when the thermodynamic equilibrium is disturbed.

The water and oil phases are co-produced during oil production and transportation. The dispersion of water droplets in oil or oil droplets in water will be formed by sufficient mixing energy from the refinery. The interfacial active agents in the crude oil such as asphaltenes, resins and naphtenic acid may accumulate at the water-oil interface and hinder the droplets to separate. Among these components, asphaltenes are believed to be the major causes in stabilizing the emulsion. This is

Corresponding author: Oloro John, E-mail: joloroeng@yahoo.com , Tel.: +2348052756328

because 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. Consequently, the formation of particularly stable water-in-crude oil emulsion is facilitated.

Emulsion problems in crude oil productions resulted in a demand for expensive emulsion separation equipment such as water treaters, separators and coalescers. Hence, chemical demulsification is the suitable method from both operational and economic point of view to break the crude oil emulsion. A chemical agent typically acts on the interfacial film by either reacting chemically with the polar crude oil components or by modifying the environment of the demulsification.

Among chemical agents, interfacial-active demulsifiers, which weaken the stabilizing films to enhance droplets coalescence, are preferred due to lower additions rates needed.

Emulsions of oil and water are one of many problems directly associated with the petroleum industry, in both oil-field production and refinery environments.

Whether these emulsions are created inadvertently or are unavoidable, as in the oil field production area, or are deliberately induced, as in refinery desalting operations, the economic necessity to eliminate emulsions or maximize oil-water separation is present[1].

The aim of this project is to treat crude oil emulsion sample gotten from umusadege field using locally made demulsifiers.

The major objectives are: 1.To determine the API Gravity of the sample before and after treatment.2.To determine the Basic Sediment and Water (BS&W) of the sample before and after treatment.3.To determine the amount of extracted water, free water, and oil volume.

The use of local materials for the production of demulsifiers in the breaking of crude oil emulsion is relevant in oil industry because it helps to reduce the exclusive dependence on the high profile foreign demulsifiers, which are expensive and imported.

## 2.0 Theory and Method

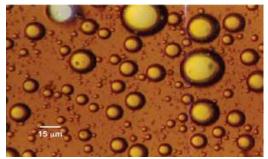
Crude oil emulsion is defined as the mixture of two immiscible liquids (oil and water) one which is dispersed in the form of droplets in the other. The liquid in an oil emulsion that is broken into droplets is known as the internal or dispersed phase while the continuous or external phase is the phase in which the droplet is suspended.

### 2.1 Types of Emulsion

Depending on which phase is dispersed and which phase is continuous. Emulsions are classified into three groups. They are;

- 1. Water-in-oil emulsion (W/O) or Regular emulsion
- 2. Oil-in-water emulsion(O/W) or Reverse emulsion
- 3. Multiphase or Mixed emulsion
- Water in Oil Emulsion (Regular Emulsion)

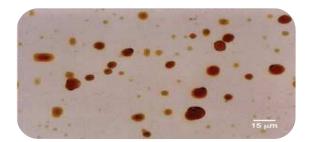
In this type of emulsion, water is the dispersed or internal phase and oil is the continuous or external phase, water is dispersed in the oil as shown in Fig1. When considering crude oil emulsion, water-in-oil emulsions are treated with great concern because it is the most common type of crude oil emulsion encountered in the oilfield



**Fig. 1:** Photomicrograph of a Water-In-Oil Emulsion.

### 2.2 Oil in Water Emulsion (Reverse Emulsion)

This is an emulsion type in which oil is dispersed in water. The continuous or external phase is water while oil is the dispersed or internal phase. This emulsion type is often called reverse emulsion. It is encountered in some heavy oil production/ formation zones, such as that found in areas of Canada, Venezuela, California, and other areas.



### Fig. 2: Photomicrograph of an Oil-In-Water Emulsion.

### 2.3 Multiphase or Mixed Emulsion

Multiple emulsions are more complex and consist of tiny droplets suspended in bigger droplets that are suspended in a continuous phase. For example, a water-in-oil-in-water emulsion consists of water droplets suspended in larger oil droplets that, in turn, are suspended in a continuous water phase. **Fig.3.0** shows an example of a multiple emulsion.

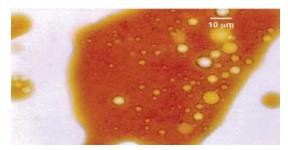


Fig. 3: Photomicrograph of a Water-In-Oil-In-Water Emulsion.

For crude oil emulsion to be formed, there are three basic conditions. These are;

1. The two liquids forming the emulsion must be immiscible (oil and water).2. An emulsifying agent must be present

3. There must be sufficient agitation to disperse one liquid as droplet in the other [2].

### 2.4 Emulsion Stability

A stable emulsion will not break or separate without some form of treatment. Several factors can contribute to the stability of an emulsion. This factors include;

- 1. emulsifying agent,
- 2. Specific/API gravity,
- 3. Total dissolved solid,
- 4. Viscosity,
- 5. water percentage,
- 6. agitation
- 7. Age of emulsion.

### 2.5 Emulsifying Agents

Produced oil field water-in-oil emulsions contain oil, water, and an emulsifying agent. Emulsifiers stabilize emulsions and include surface-active agents and finely divided solids.

Surface-active agents

Surface-active agents (surfactants) are compounds that are partly soluble in both water and oil. They have a hydrophobic part that has an affinity for oil and a hydrophilic part that has an affinity for water. Because of this molecular structure, surfactants tend to concentrate at the oil/water interface, where they form interfacial films. This generally leads to a lowering of the interfacial tension (IFT) and promotes dispersion and emulsification of the droplets. Naturally occurring emulsifiers in the crude oil include higher boiling fractions, such as:

- 1. Asphaltenes and resins
- 2. Organic acids
- 3. Bases

These compounds have been shown to be the main constituents of interfacial films that form around water droplets in many oilfield emulsions. Other surfactants that may be present are from the chemicals injected into the formation or wellbores, such as:

1. Drilling fluids

J of NAMP

- 2. Stimulation chemicals
- 3. Corrosion inhibitors
- 4. Scale inhibitors
- 5. Wax

6. Asphaltene control agents

### 2.6 Finely divided solids

Fine solids can act as mechanical stabilizers. These particles, which must be much smaller than emulsion droplets (usually submicron), collect at the oil/water interface and are wetted by both oil and water. The effectiveness of these solids in stabilizing emulsions depends on factors such as;

i Particle size

- ii Interparticle interactions
- iii Wettability of the particles

## 2.7 Finely Divided Solids Found in Oil Production include:

i Clay particles ii Sand iii Silt iv Asphaltenes and waxes v Corrosion products vi Shale particles vii Mineral scales that collect at the oil/water interface viii Drilling muds.

# 2.8 Specific Gravity and API Gravity

The specific gravity of a substance is the weight of a given amount of that substance (liquid) at a given temperature compared to the weight of an equal volume at the same temperature. The difference in specific gravity between the oil and water has a bearing on the stability of an emulsion. The greater the differences, the faster the water can settle out, and stabilize. Crude oil with low API gravity (high density) form the more stable and higher percentage volume of emulsion, while crude oil with high API gravity (low density) will form less stable and lower percentage of emulsion. Oil with a low API, tends to keep water droplets in suspension creating a more stable emulsion. Asphaltic based oils have tendency to emulsifying more rapidly than paraffin-base oil.

$$API = \frac{141.5}{32} - 131.5$$

Where  $\mathbf{S.G}^{SG}$  is the specific gravity of the oil.

(1)

Total Dissolved Solid

Total dissolved solid or salinity of the water also influences settling rates. The higher the amount of solid in an emulsion, the faster the water is separated from the emulsion. But the lower the amount of solid in an emulsion the lower the water is separated from the emulsion the heavier the water, the faster the stabilization or settling time of the emulsion.

## 2.9 Viscosity

Viscosity is defined as fluids resistance to flow. High viscosity crude oil usually forms a more stable emulsion than low viscosity oil. Emulsion of high viscosity crude is very stable and difficult to treat. Because the viscosity of the oil hinders or prevents movement of the dispersed water droplets and thus reduces or retards their coalescence. High viscosity oil contained more emulsifiers than lighter oils. An oil of high viscosity required more time for the water droplet to coalescence and settle out, at the bottom of the container because of the high resistance to flow.

## 2.10 Water Percentage

Smaller percentage of water in an emulsion means greater stability. High percentage of water in an emulsion tends to make the water to coalescence quickly and then settle out.

## 2.11 Agitation

Emulsion stability will be reduced by reducing or eliminating the agitation of oil-and-water mixture. The amount of mixing depends on several factors and is difficult to avoid. In general, the greater the mixing, the smaller the droplets of water dispersed in the oil and the tighter the emulsion. The effectiveness of any demulsifier added to treatment system, is directly dependent upon its making optimum contact with the emulsion.

Therefore, the emulsion must be sufficiently agitated after the chemical demulsifierhas been added. Increase of the mild agitation, is beneficial in promoting coalescence. Re-emulsification may occur if an emulsion is agitated severely once it has broken into oil and water.

# 2.12 Age of the Emulsion

The longer an emulsion remains untreated, the harder it becomes to break .The emulsifying agent that surrounds the surface of the droplets with a film that becomes thicker with time, hence making it difficult to break. An emulsion generally tends to increase in stability with age. It is therefore advised to treat emulsion that is fresh from the flow lines or well head rather than to allow it spend more time in the storage tank before treating [3].

## 2.13 Characteristics and Physical Properties

Oilfield emulsions are characterized by several properties including:

i Appearance and color

```
Journal of the Nigerian Association of Mathematical Physics Volume 32, (November, 2015), 373 – 396
```

### ii BS&W

iii Droplet size

### iv Bulk and interfacial viscosities

#### 2.14 Appearance and Color

Color and appearance is an easy way to characterize an emulsion. The characterization becomes somewhat easy if the emulsion is transferred into a conical glass centrifuge tube. The color of the emulsion can vary widely depending on:

- i Oil/water content
- ii Characteristics of the oil and water

The common colors of emulsions are dark reddish brown, gray, or blackish brown; however, any color can occur depending on the type of oil and water at a particular facility. Emulsion brightness is sometimes used to characterize an emulsion. An emulsion generally looks murky and opaque because of light scattering at the oil/water interface. When an emulsion has small diameter droplets (large surface area), it has a light color. When an emulsion has large diameter droplets (low total interfacial surface area), it generally looks dark and less bright. Understanding the characteristics of an emulsion by visual observation is an art that improves with experience.

### 2.15 Basic Sediment and Water

Basic sediment and water (BS&W) is the solids and aqueous portion of an emulsion. It is also referred to as BS&W, bottom settlings and water, or bottom solids and water. Several methods are available to determine the amount of water and solids in emulsions. Standard methods have been proposed by several organizations including the;

- 1. Institute of Petroleum
- 2. American Petroleum Institute
- 3. American Society for Testing Materials

The most common technique for the determination of oil, water, and solids consists of:

- 1. Adding a slight overdose of a demulsifier to an emulsion
  - 2. Centrifuging it
  - 3. Allowing it to stand
- The amount of solids and water separated is measured directly from specially designed centrifuge tubes.

#### 2.16 Droplet Size and Droplet-Size Distribution

Produced oilfield emulsions generally have droplet diameters that exceed 0.1  $\mu$ m and may be larger than 100  $\mu$ m. Emulsions with smaller drops are more stable than those in larger drops. Because smaller drops will not coalesce easily and settle out as water and oil.

### 2.17 Interfacial Viscosity

The previous discussion on viscosity was limited to bulk emulsion viscosity. A closely related and very important property, especially for demulsification, is the interfacial viscosity, or the viscosity of the fluid at the oil/water interface. As mentioned previously, water-in-oil emulsions form rigid interfacial films encapsulating the water droplets. These interfacial films stabilize an emulsion by lowering IFT and increasing interfacial viscosity. These films retard the rate of oil-film drainage during the coalescence of water droplets, thereby greatly reducing the rate of emulsion breakdown. The oil-drainage rate depends on the interfacial shear viscosity. High interfacial viscosities significantly slow the liquid drainage rate and thus have a stabilizing effect on the emulsion.

## 2.18 Demulsification and Principles of Demulsifiction

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:

- 1. Rate or the speed at which this separation takes place.
- 2. Amount of water left in the crude oil after separation.
- 3. 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 obviously 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 company and pipeline specifications. The salt is insoluble in oil and associated with residual water in the treated crude. Low BS&W and salt content is 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 in refinery equipment. The salts are removed by washing or desalting the crude oil with relatively fresh water.

### 2.19 **Principles of Demulsification**

Three basic steps or Demulsification principles are required to separate a water-in-oil emulsion into a bulk phase of oil and water. These are;

Destabilizing emulsions

Oilfield emulsions possess some kinetic stability. This stability arises from the formation of interfacial films that encapsulate the water droplets. To separate this emulsion into oil and water, the interfacial film must be destroyed and the droplets made to coalesce. Therefore, destabilizing or breaking emulsions is linked directly to the removal of this interfacial film. Temperature

Application of heat promotes oil/water separation and accelerates the treating process. An increase in temperature has the following effects.

- 1. Reduces the viscosity of the oil.
- 2. Increases the mobility of the water droplets.
- 3. Increases the settling rate of water droplets.
- 4. Increases droplet collisions and favors coalescence.
- 5. Weakens or ruptures the film on water droplets because of water expansion and enhances film drainage and coalescence.
- 6. Increases the difference in densities of the fluids that further enhances water-settling time and separation.

All of these factors favor emulsion destabilization and oil/water separation; however, heat by itself is not a cure-all and even has some disadvantages (e.g., loss of light ends from the crude oil). An economic analysis should be performed that takes into consideration factors such as:

- 1. Heating costs
- 2. Reduced treating time
- 3. Residual water in the crude

An increase in temperature also can be achieved by burying crude-oil pipelines or by insulating them. These factors should be evaluated carefully during development, especially at facilities where emulsion problems are anticipated [4].

### 2.20 Coalescence

Coalescence is defined as the combination of two or several droplets to form a large drop. It takes place when the thin film of continuous phase between two drops breaks and they fuse rapidly to form a single droplet. The stability of an emulsion system, therefore, depends on the rate of coalescence and it is obvious that the properties of this film will determine the stability of the emulsion.

### 2.21 Gravity Separation

A period of setting must be provided to allow the coalesced drop to settle out at the oil because of the difference in density between the water and oil. This is accomplished by providing a sufficient resident time and favorable flow patterns in tank vessels that will allow the coalesced drop out water to separate from the oil.

## 2.22 Methods of Treating Crude Oil Emulsion

In the oil industry, crude oil emulsion must be separated completely before the oil can be transported and processed further. All the various treating method listed before are directed at increasing the size of the water droplet, increasing the gravity difference between the water and oil. Therefore oil produce from reservoir must be treated or demulsified, before they are pumped to the refineries or off-loaded at the terminals. 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:

- 1. Adding chemical demulsifiers (chemical method).
- 2. Increasing the temperature of the emulsion (thermal method).
- 3. Applying electrostatic fields that promote coalescence (electrical method).
- 4. Reducing the flow velocity that allows gravitational separation of oil, water, and gas. This is generally accomplished in large-volume separators and desalters (mechanical method).

## 2.23 Chemical Methods

Dehydration chemicals, or demulsifiers, are chemical compounds that are widely used to destabilize, and assist in the coalescence of crude-oil emulsions. This treatment method is popular because the chemicals are easily applied, usually are reasonable in cost, and usually minimize the amount of heat and settling time required.

The chemical counteracts the emulsifying agent, allowing the dispersed droplets of the emulsion to coalesce into larger drops and settle out of the matrix. To work, demulsifiers

- 1. Must be injected into the emulsion
- 2. Must mix intimately with the emulsion and migrate to all the protective films surrounding all the dispersed droplets.
- 3. Must displace or nullify the effect of the emulsifying agent at the interface.

For the oil and water to separate, there must also be a period of continual, moderate agitation of the treated emulsion to produce contact between coalescence of the dispersed droplets as well as a quiet settling period.

### Journal of the Nigerian Association of Mathematical Physics Volume 32, (November, 2015), 373 – 396 Treatment of Crude Oil... Oloro J of NAMP

Actions of a chemical demulsifier

The mechanisms to consider for a chemical demulsifier are

- 1. Strong attraction to the oil/ water interface. The demulsifier must be able to migrate rapidly through the oil phase to reach the droplet interface where it must counteract the emulsifying agent.
- 2. Flocculation. The demulsifier must have an attraction for water droplets with a similar charge and bring them together. In this way, large clusters of water droplets gather, which under a microscope look like bunches of fish eggs.
- 3. Coalescence. After flocculation, the emulsifier film remains continuous. If the emulsifier force is weak, the flocculation force might be enough to cause coalescence; however, this is usually not true, and the demulsifier must enable coalescence by neutralizing the emulsifier and promoting rupture of the droplet interface film. In the flocculated emulsion, the rupture causes increasing water- drop size.
- 4. Solid wetting. Iron sulfides, clay and drilling muds can be made water-wet, which causes them to leave the interface and be diffused into the water droplets. Paraffin and asphaltenes can be dissolved or altered by the demulsifier to make their film less viscous, or they can be made oil-wet so that they will be dispersed in the oil [5].

## 2.24 Selection of Demulsifier

Selection of the right demulsifier is crucial in the emulsion breaking process, and still viewed as an art rather than a science. Demulsifiers are chemicals that contain solvents (e.g. benzene, toluene, xylene, short-chain alcohols, and heavy aromatic naphtha), surfactants, flocculants, and wetting agents. The demulsifiers act by total or partial displacement of the indigenous stabilizing components (polar materials) of the interfacial film surrounding the emulsion droplets. This displacement also brings about a change in properties such as interfacial viscosity or electricity of the protecting film, thus enhancing destabilization. In some cases, the chemicals act as a wetting agent and alter the wettability of the stabilizing particles, which lead to a breakup of the emulsion film. Testing procedures are available to select appropriate demulsifiers. These tests include bottle tests, dynamic simulators, and actual plant tests. These tests also determine the amount of chemical to be added. Too little dosage of demulsifier will leave the emulsion unresolved. On the other hand, a high dosage of demulsifier (an over treating condition) may be detrimental to the treatment process. Because demulsifiers are surface-active agents too like emulsifiers, an excess quantity of demulsifier may also produce very stable emulsions. In this case, the demulsifier simply replaces the natural emulsifiers at the interphase[6].

### 2.25 Chemistry of Demulsifier

Crude oil emulsion treatment dates back to the time when the first oil well was drilled. Chemical demulsifier's history reveals that prior to the knowledge of polyether condensate synthesis, chemicals such as sulphuric acid, sulphated castor oil, and polyhydric alcohols were used directly as demulsifiers. Demulsifiers maybe specific for a given emulsion and completely ineffective for another emulsion. The Table 1. below shows the development and evaluation of demulsifiers.

Year	Demulsifiers
1920-1930	Soap, naphtenic acid salts and sulphated castor oil.
1930-1940	Petroleum sulphonates, derivatives of sulpho-acid oxidized castor
	Oil and sulphosucinic acid ester.
1940-1950	Fatty acids, fatty alcohols, alkylphenols.
1950-1960	Ethylene oxide/propylene oxide copolymer, Alkoxylated cyclic palkylphenol
	Formaldehyde resins.
1960-1970	Amine alkoxylate.
1970-1980	Alkoxylated cyclic p-alkylphenol formaldehyde resins
1980-1990	Polyesteramine and blends.
1990-2013	Blends of any combination of polyglycol esters, low molecular weight resin derivatives,
	sulfonates, polymerized oils and esters, high molecular weight resin derivatives,
	alkanolamine condensates, oxyalkylated phenols, polyamine derivatives and various blends
	of surfactants.

 Table 1: Historical Development of Chemical Demulsifier

 Vacan
 Demulsificant

## 2.26 Physical Methods

Physical treatment techniques include heating, electrical, or a mechanical method [7].

### 2.27 Thermal Methods

Heating alone does not cause an emulsion to breakdown, except in rare instances.

Usually the application of heat is an auxiliary process to enhance the breaking or separation of emulsion. It reduces the viscosity of the oil and increases the water settling rate according to Stokes law. Increase in temperature also results in the destabilization of rigid films caused by reduced interfacial viscosity. Furthermore, the coalescence frequency between water droplets is increased because of the higher thermal energy of the droplets. Increasing the temperature has some negative effects. Firstly, it costs money to heat the emulsion stream.

Second, it can result in the loss of light ends from the crude oil, reducing its American petroleum institute (API) gravity and the treated oil volume. Finally, increasing temperature leads to an increased tendency toward scale deposition and an increased potential for corrosion in treating vessels.

Application of heat for emulsion breaking should be based on an overall economic analysis of the treatment facility. The cost effectiveness of adding heat should be balanced against longer treatment time (larger separator), loss of light ends and a resultant reduced oil price, chemical costs, and the costs of installation of electrostatic grids, or retrofitting. Some common types of heaters in the oil industry include Tubular heaters, fluid-jacket heaters, internal firebox heaters, volume, or jug-type heaters.

### 2.28 Electrical Methods

High voltage electricity (electric Grids) is often an effective means of breaking emulsion. It is generally theorized that water droplets have an associated charge and, when an electric field is applied, the droplets move rapidly, collide with each other, and coalesce. The electric field also disturbs the rigid interfacial film by rearranging the polar molecules thereby weakening the tight film and enhancing coalescence. The electrical system consists of a transformer and electrodes that provide high voltage alternating current alternating current.

The electrodes are placed in such a way as to provide an electric field that is perpendicular to the direction of flow. The distance between the electrodes – in some designs – adjustable so that the voltage can be varied to meet the requirement of the emulsion being treated.

Electrostatic dehydration is rarely used as a method of breaking emulsions. It is generally used in conjunction with chemical and heat requirements. Invariably, the use of electrostatic dehydration will result in a reduction of heat addition. Lower temperatures result in fuel economy, reduced problems with scale and corrosion formation, and reduced light ends loss. Electrostatic grids also can lead to a reduction in the use of emulsion-breaking-chemicals [8].

### 2.29 Mechanical Method

Mechanical methods have to do with either separating or slotting from water or vice versa. These are different mechanical equipment used for water settling. They are:

- 1. Settling tank.
- 2. Free Water Knock Out drums.
- 3. Skim pits.
- 4. Guns Barrel or Wash Tank.
- 5. Production Traps or three phase separator
- 6. Desalters.

Settling Tank

The settling tank was the basic vessel used in emulsion in the early days. This involves allowing the emulsion to remain in a container for a long time enough for the water droplet to settle to the bottom of the container. In the emulsion, the water has a heavier density than oil, therefore the oil floats on top of the water. This method often provides more adequate when economy is a prime factor.

Free Water Knock Out Drum

Free-water knockout drums separate the free water from the crude oil/water mixture. Some of the associated gases may be separated in these drums. Free-water knockout drums are supplementary equipment that aid in the treatment of produced crude oil emulsions.

Skim pit

A skim pit is an earthen pit lined concrete into which large volumes of well fluid are produced. Due to high density of water, oil floats on top of the water and the water settles at the bottom of the pit. The oil is skimmed off by series of baffles as water flows across the pit.

Gun Barrel or Wash Tank

This is another type of settling tank that is fatted with an internal or external boot or flume. The gun barrel is composed of different parts. They are;

The inlet pipe: This pipe conducts the emulsion (water and oil) from the oil and gas separator to the gun barrel.

*The conductor pipe*: This is also known as the boot or flume . This is a large pipe through which the emulsion passes before getting to the bottom of the pit.

*Body or Tank:* This part holds the water wash or water layer emulsion and clean oil layers and allow time for the oil and water to separate.

*Water Outlet:* It provides outlet for the separated water and it is used to regulate the amount of water held in the gun barrel. Production Trap or Three Phase separator

Three phase separator are used to separate the production fluid into oil, water, and gas. The separator could either be horizontal or vertical in configuration. Separator efficiency can be improved upon by installing coalesce pack/pack as they increase the travelling of the fluid through the separator and spreader/spreader which increase the collision frequency between droplet. The main part of the separator include: heater section, wash tank, electrostatic grids and stabilizing section.

Desalters

The oil from the separator is generally "off-spec" (i.e., it still contains unacceptably high levels of water and solids). It must be further treated to meet crude specifications. For the refinery, the salt level must be further reduced. Refinery crude should contain no more than a specified amount of inorganic solids (salts). This is generally expressed in pounds per thousand barrels. The industry standard is 1 pound per thousand barrels. The removal of the salts, along with the remaining water, is the process of desalting.

## 3.0 Methodology

#### Experimental Procedures and Analysis

The crude oil emulsion sample used in this research, was obtained from an oil field located at kwale in Ndokwa West local Government Area of Delta state, Nigeria. The following experiments were conducted to determine the BS&W, API & SG of the sample before and after treatment and also to analyze the effectiveness of the treatment. The Bottle test method is used to determine the chemical/ formulation most effective in breaking the emulsion.

Determination of Free Water in Emulsion by Gravity Separation

### **3.1** Apparatus/Materials

- 1. Retort stand
- 2. Crude oil emulsion sample
- 3. Separating funnel
- Procedure/Method
- 1. The separating funnel was fixed properly to the retort stand.
- 2. Samples (2.25 liters) from umusadege well 1 and (3.5 liters) from umusadege well 2, were poured into the separating funnels and allowed for some time to separate free water by gravity.

#### Determination of Percentage BS&W of Untreated Emulsion Sample

Basic Sediments and Water is the percentage of impurities/foreign matter within the crude oil sample. This is done to ascertain the sediment and water quantity of the sample to be treated.

- Apparatus/Materials
- 1. Centrifuge Machine
- 2. Centrifuge Tube
- 3. Crude Oil Emulsion (Sample)
- 4. Stop Watch

Procedure/Method

- 1. 10ml of Samples were poured into the calibrated centrifuge tubes.
- 2. Samples were centrifuged thoroughly at 5000 rev/min for 5 minutes in the centrifuge machine and readings were recorded.

Determination of specific gravity and API gravity of untreated crude oil emulsion sample (using hydrometer method)

AIM: To determine the specific/ API gravity of crude of emulsionsample using Hydrometer method.

SCOPE: This experiment covers the determination of API/Specific gravity using hydrometer method.

Significance: Accurate determination of density, specific gravity and API of the emulsion sample is necessary for the conversion of measured volume 60f/60f

Apparatus

- 1. Measuring cylinder
- 2. Hydrometer
- 3. Thermometer
- 4. Crude sample

Procedure

- 1. Crude oil sample (well 1 and 2) was transferred into a clean hydrometer cylinder
- 2. The cylinder containing the sample was positioned vertically in a free location to avoid air contact and inclination of any disturbance.
- 3. The hydrometer was carefully lowered into the sample. As soon as a stabilized reading was reached, the point at which the sample cut the graduated line was recorded as the specific gravity and the temperature reading of the sample was taken and recorded.

Formulation of Local Demulsifier (LD)

The materials used in formulating LD1 and LD2 are shown in Table 2.

## Treatment of Crude Oil...

Oloro J of NAMP

Table 2:	Showing	LD1 and	l LD2 Material	S
----------	---------	---------	----------------	---

Demulsifier	Content	Weight/Volume	Function
	Alum	5g	To facilitate settling of sediments
	Castor Oil	50ml	It acts as a solvent for the camphor and improves on the
LD1			lipophilic properties
	Industrial	5g	Coalescing of the tiny water droplets
	Starch		
	Liquid Soap	50ml	Destabilization of emulsion film
	Camphor	5g	This forms the lipophilic end of the demulsifier, and
			improves on the demulsifier performance
	Alum	5g	To facilitate settling of sediments
	Castor Oil	50ml	It acts as a solvent for the camphor and improves on the
LD2			lipophilic properties
	Camphor	5g	Coalescing of the tiny water droplets
	Liquid	50ml	Destabilization of emulsion film
	Soap		
	Comart	5g	This forms the lipophilic end of the demulsifier, and
	renew cold		improves on the demulsifier performance
	water Starch		

### 3.2 Apparatus

(i)Digital Weigh balance (ii)Measuring Cylinder(50ml graduated)(iii)Beaker(iv)Magnetic Heat-Stirring machine (v)Filter paper(vi)Prescription bottles (200ml graduated)(vii)Thermostatic Water Bath(viii)Stop watch(ix)Syringe(x)Micropipette

### 3.3 LD1 formulation Procedure

- 1. A solution of 5g of camphor dissolved in 50ml of castor oil was prepared in a beaker.
- 2. The camphor and castor oil solution was stirred and heated (220°F) in a Magnetic heating and stirring machine until homogeneity of solution is obtained. See figure 4.



Fig. 4: Magnetic Heat & Stirrer Machine

- i 5g of industrial starch, 5g of alum, and 50ml of detergent was added to the camphor-castor oil solution.
- ii The new mixture above was further stirred and heated for 15 minutes to achieve homogeneity, after which all precipitates or sediments were filtered off.

### 3.4 LD2 formulation Procedure

i A solution of 5g of camphor dissolved in 50ml of castor oil was prepared in a beaker. ii The camphor and castor oil solution was stirred and heated (130°F) in a Magnetic heating and stirring machine until homogeneity of solution is obtained. iii 5g of comart renew cold water starch, 5g of alum, and 50ml of detergent was added to the camphor-castor oil solution. iv The new mixture above was further stirred and heated for 15 minutes to achieve homogeneity, after which all precipitates or sediments were filtered off.

Determination of the Effectiveness of Separol and Local Demulsifiers Using the Bottle Test Method

Apparatus/Materials

i Forty- two prescription bottles

ii Crude oil emulsion samples

iii Thermostatic Water Bath

iv Demulsifiers

v Syringe vi Stop watch Procedure

- 1. A 100ml of emulsion was poured into each of the 42 prescription bottles.
- 2. All prescription bottles were labeled according to the concentration of demulsifier to be used for the analysis. For well 1; eighteen prescription bottles was used, six bottles for each demulsifier (LD1, LD2 and separol). For well 2; twenty- four prescription bottles were used, six bottles for each demulsifier (LD1, LD2, servo and separol) were used with concentrations of 0.2ml, 0.4ml, 0.6ml, 0.8ml, 1.0ml, and 1.2ml of demulsifiers respectively.
- 3. All bottles containing samples with varying dose of demulsifier were agitated to achieve proper mixture.
- 4. The bottles were placed in the water bath, with water level at the 100ml mark and heated to 50°C. See figure 2.



Fig. 5: Samples in the Thermostatic Water Bath.

5. Observations of the samples were made every 30 minutes for two hours to identify any separation while being heated and was recorded.

Determination of Percentage BS&W of Treated Emulsion Sample

- Apparatus/Materials
- 1. Centrifuge Machine
- 2. Centrifuge Tube
- 3. Crude Oil (Treated Emulsion Samples)
- 4. Stop Watch

#### Procedure/Method

- 1. From each bottle, 10ml oil of treated Samples were extracted and poured into the calibrated centrifuge tubes.
- 2. Samples were centrifuged thoroughly at 5000 rev/min for 15 minutes in the centrifuge machine, and readings werd recorded.

Determination of API Gravity and Specific Gravity of Emulsion Sample after Treatment (Hydrometer Method) Apparatus

(i)Measuring cylinder (ii) Hydrometer(iii)Thermometer(iv)Crude sample (treated)

- Procedure
- 1. 400ml of oil from each treated sample (well 1 and 2) was transferred into a clean hydrometer cylinder.
- 2. The cylinder containing the sample was positioned vertically in a free location to avoid air contact and inclination of any disturbance.
- 3. The hydrometer was carefully lowered into the sample. As soon as a stabilized reading was reached, the point at which the sample cut the graduated line was recorded as the specific gravity and the temperature reading of the sample was taken and recorded.

## 4.0 **Results and Discussions**

Table 3: Result for volume of free water by Gravity separation before treatment

	Sample	Volume (liters)
Umusadege well 1	Emulsion	2.25
	Free water	0
	Emulsion	3.25
Umusadege well 2	Free water	0.25

Table3.shows the results of the volume of free water from emulsion by gravity separation before treatment.

0.25 liters of free water was separated from umusadege well 2 emulsion sample while umusadege well 1 sample shows 0 water separation . This indicates that well 2 contains more water than well 1.

### Treatment of Crude Oil... Oloro

#### Oloro J of NAMP

	Sample	Component (ml)	Component (%)
Umusadege well 1	Oil	10	100
	Sludge	0	0
	Water	0	0
Limusodogo well 2	Oil	9.8	98
Umusadege well 2	Sludge	0.2	2
	Water	0	0

**Table 4:** Showing the Results for BS&W Before Treatment

Table4. shows the results of the BS&W of samples before treatment. A manual centrifuge was used to determine the BS&W before treatment. This is to ascertain if the samples to be considered is an emulsion. From Table 4. above, it indicates that well 2 sample contains more emulsion than well 1 sample.

#### Table 5: BS&W of treated oil with LD1 (Umusadege well 1)

Volume of injected Separol	Volume of Water	Volume of Water	Volume of Oil	Volume of Oil
(ml)	( <b>ml</b> )	(%)	( <b>ml</b> )	(%)
0.2	0.05	0.5	9.95	99.5
0.4	0.0	0	10	100
0.6	0.0	0	10	100
0.8	0.0	0	10	100
1.0	0.0	0	10	100
1.2	0.0	0	10	100

Table5. shows the results of the BS&W of treated oil with LDI (umusadege well 1 samples). A manual centrifuge was used to determine the BS&W after treatment. This is to ascertain if the top oil still contains water. From Table 5. above, only 0.2ml of LD1 indicates 0.5% of water in top oil.

#### Table 6: BS&W of Oil treated with LD1 (umusadege well 2)

Tuble of Dow () of on fictured () in DDT (unitsbudege () on 2)						
Volume of injected	Volume of Water	Volume of Water	Volume of Oil	Volume of Oil		
LD1	(ml)	(%)	(ml)	(%)		
(ml)						
0.2	0.1	1.0	9.9	99.0		
0.4	0.05	0.5	9.95	99.5		
0.6	0.05	0.5	9.95	99.5		
0.8	0.0	0.0	10.0	100.0		
1.0	0.0	0.0	10.0	100.0		
1.2	0.05	0.5	9.95	99.5		

Table6. shows the results of the BS&W of treated oil with LDI (umusadege well 2 samples). A manual centrifuge was used to determine the BS&W after treatment. This is to ascertain if the top oil still contains water. From Table 6.0 above, 0.8ml and 1.0ml indicates 0% of water in top oil.

#### Table7: BS&W of Oil treated with LD2 (umusadege well 1)

Volume of injected LD1 (ml)	Volume of Water (ml)	Volume of Water (%)	Volume of Oil (ml)	Volume of Oil (%)
0.2	0.05	0.5	9.95	99.5
0.4	0.05	0.5	9.95	99.5
0.6	0.0	0.0	10.0	100.0
0.8	0.0	0.0	10.0	100.0
1.0	0.0	0.0	10.0	100.0
1.2	0.05	0.5	9.95	99.5

Table7. shows the results of the BS&W of Oil treated with LD2 (umusadege well 1). A manual centrifuge was used to determine the BS&W after treatment. This is to ascertain if the top oil still contains water. From Table 7.0, 0.6ml, 0.8ml & 1.0ml indicates 0% water in top oil.

Table 6 DSee W of On treated with LD2 (unusadege with 2)						
Volume of injected	Volume of Water	Volume of Water	Volume of Oil	Volume of Oil		
LD1	(ml)	(%)	(ml)	(%)		
(ml)						
0.2	0.1	1.0	9.9	99.0		
0.4	0.05	0.5	9.95	99.5		
0.6	0.0	0.0	10.0	100.0		
0.8	0.0	0.0	10.0	100.0		
1.0	0.0	0.0	10.0	100.0		
1.2	0.05	0.5	9.95	99.5		

#### Table 8.: BS&W of Oil treated with LD2 (umusadege well 2)

Table8. shows the results of the BS&W on oil treated with LD2 (umusadege well 2 sample). A manual centrifuge was used to determine the BS&W after treatment. This is to ascertain if the top oil still contains water .From Table 8.0, 0.6ml, 0.8ml & 1.0ml indicates 0% water in top oil.

#### Table 9.: BS&W of Oil treated with separol (umusadege well 1)

Volume of injected	Volume of Water	Volume of Water	Volume of Oil	Volume of Oil
LD1	(ml)	(%)	(ml)	(%)
(ml)				
0.2	0.05	0.5	9.95	99.5
0.4	0.1	1.0	9.9	99.0
0.6	0.1	1.0	9.9	99.0
0.8	0.1	1.0	9.9	99.0
1.0	0.05	0.5	9.95	99.5
1.2	0.05	0.5	9.95	99.5

Table9. shows the results of the BS&W of Oil treated with separol (umusadege well 1) .A manual centrifuge was used to determine the BS&W after treatment. This is to ascertain if the top oil still contains water. Samples still showing trace of water in top oil.

#### Table 10.: BS&W of Oil treated with separol (umusadege well 2)

Tuble 100 DbW () of on the deduce ((indubudege () of 2)						
Volume of injected	Volume of Water	Volume of Water	Volume of Oil	Volume of Oil		
LD1	(ml)	(%)	(ml)	(%)		
(ml)						
0.2	0.1	1.0	9.9	99.0		
0.4	0.1	1.0	9.9	99.0		
0.6	0.1	1.0	9.9	99.0		
0.8	0.1	1.0	9.9	99.0		
1.0	0.1	1.0	9.9	99.0		
1.2	0.1	1.0	9.9	99.0		

Table10. shows the results of the BS&W of Oil treated with separol (umusadege well 2). A manual centrifuge was used to determine the BS&W after treatment. This is to ascertain if the top oil still contains water. Sample still showing trace of water in top oil.

Table 11.: BS&W of Oil treated with servo (umusadege well 2)

Volume of injected	Volume of Water	Volume of Water	Volume of Oil	Volume of Oil
LD1	(ml)	(%)	(ml)	(%)
(ml)				
0.2	0.1	1.0	9.9	99.0
0.4	0.05	0.5	9.95	99.5
0.6	0.05	0.5	9.95	99.5
0.8	0.05	0.5	9.95	99.5
1.0	0.05	0.5	9.95	99.5
1.2	0.05	0.5	9.95	99.5

Table11. shows the results of the BS&W of Oil treated with servo (umusadege well 2). A manual centrifuge was used to determine the BS&W after treatment. This is to ascertain if the top oil still contains water. Samples still showing trace of water in top oil.

#### Results of specific/ API gravity before and after treatment with various demulsifiers

Tuble 12. Speenle Gruvhymi i Result Obtained Defore Treatment								
Samples	T(°F)	⊿T (° <b>F</b> )	SG <sub>TT</sub>	SG <sub>STD</sub>	API			
Well 1	84	24	0.811	0.8206	40.9348			
Well 2	84	24	0.807	0.8166	41.7795			

#### Table 12: Specific Gravity/API Result Obtained Before Treatment

Table12. shows the results of the Specific Gravity/API Result Obtained Before Treatment. Before the treatment of each samples using various demulsifiers, the API gravity of umusadege well 1, reads 40.9348 and the API gravity of umusadege well 2, reads 41.7795.

#### Table 13.: Specific Gravity/API Result Obtained after Treatment with LD1

Samples	T(°F)	ΔT (° <b>F</b> )	SG <sub>TT</sub>	SG <sub>STD</sub>	API
Well 1	86	26	0.802	0.812	42.675
Well 2	86	26	0.791	0.801	45.660

Table13. shows the results of the Specific Gravity/API Result Obtained after Treatment with LD1. The API gravity in well 1 and well 2 samples increased after treatment with LD1, due to the separation and extraction of water from samples.

#### Table 14.: Specific Gravity/API Result Obtained after Treatment with LD2

Samples	T(°F)	⊿T ( <b>°F</b> )	SG <sub>TT</sub>	SG <sub>STD</sub>	API
Well 1	84	24	0.806	0.816	41.992
Well 2	85	25	0.792	0.802	44.934

Table14. shows the results of the Specific Gravity/API Result Obtained after Treatment with LD2. The API gravity in well 1 and well 2 samples increased after treatment with LD2, due to the separation and extraction of water from samples.

#### Table 15.: Specific Gravity/API Result Obtained after Treatment with separol and servo

Samples	T(°F)	⊿T ( <b>°F</b> )	SG <sub>TT</sub>	SG <sub>STD</sub>	API
Well1(separol)	84	24	0.811	0.8206	40.9348
Well2(separol)	86	26	0.802	0.812	42.675
Well 2(servo)	84	24	0.801	0.812	43.062

Table15. shows the results of the Specific Gravity/API Result Obtained after Treatment with separol and servo. The API gravity in well 1 and well 2 samples increased after treatment with LD2, due to the separation and extraction of water from samples.

### Treatment of Crude Oil...

Oloro J of NAMP

Volume of Demulsifier (ml)	Sample Components	0 minutes	30 minutes	60 minutes	90 minutes	120 minutes	Temp.°C
0.2	Oil	95.0	93.0	92.0	91.0	91.0	50
	Sludge	4.0	6.0	7.0	7.0	7.0	
	Water	1.0	1.0	1.0	2.0	2.0	
0.4	Oil	92.0	91.0	92.0	92.0	92.0	50
	Sludge	1.0	1.0	2.0	2.0	2.0	
	Water	7.0	8.0	6.0	6.0	6.0	
0.6	Oil	86.0	86.0	86.0	86.0	86.0	50
	Sludge	4.0	4.0	4.0	4.0	4.0	
	Water	10.0	10.0	10.0	10.0	10.0	
0.8	Oil	90.0	89.0	88.0	88.0	88.0	50
	Sludge	2.0	2.0	0.0	0.0	0.0	
	Water	8.0	9.0	12.0	12.0	12.0	
1.0	Oil	91.0	90.0	90.0	90.0	90.0	50
	Sludge	0.0	1.0	0.0	0.0	0.0	
	Water	9.0	9.0	10.0	10.0	10.0	
1.2	Oil	92	90.0	90.0	90.0	90.0	50
	Sludge	4.0	4.0	4.0	4.0	4.0	
	Water	4.0	6.0	6.0	6.0	6.0	

**Results of Heat and Chemical Treatment using Various Demulsifiers Table 16:** Result of Heat and Chemical Treatment using LD1 (*Umusadege well 1*)

Table16. shows the results of Heat and Chemical Treatment using LD1 (*Umusadege well 1*). After the injection of LD1 demulsifying chemical on the given crude oil emulsion sample, which was heated for 120minutes at field temperature of 50°c, readings were taken at 30minutes interval.

Effect on well 1; the separated water in the bottles containing the emulsion with concentration of 0.2ml, 0.4ml, 0.6ml, 0.8ml, 1.0ml & 1.2ml were 1.0ml, 8.0ml, 0.0ml, 11.0ml, 4.0ml, 8.0ml respectively.

Volume of Demulsifier (ml)	Sample Components	0 minutes	30 minutes	60 minutes	90 minutes	120 minutes	Temp.□
0.2	Oil	68.0	65.0	65.0	65.0	65.0	50
	Sludge	22.0	25.0	25.0	25.0	25.0	
	Water	10.0	10.0	10.0	10.0	10.0	
0.4	Oil	72.0	71.0	71.0	71.0	71.0	50
	Sludge	6.0	7.0	6.0	6.0	6.0	
	Water	22.0	22.0	23.0	23.0	23.0	
0.6	Oil	75.0	73.0	72.0	72.0	72.0	50
	Sludge	5.0	6.0	5.0	5.0	5.0	
	Water	20.0	21.0	23.0	23.0	23.0	
0.8	Oil	72.0	71.0	71.0	71.0	71.0	50
	Sludge	3.0	2.0	1.0	1.0	1.0	
	Water	22.0	27.0	28.0	28.0	28.0	
1.0	Oil	75.0	74.0	73.0	73.0	73.0	50
	Sludge	3.0	2.0	0.0	0.0	0.0	
	Water	22.0	24.0	27.0	27.0	27.0	
1.2	Oil	76.0	75.0	75.0	75.0	75.0	50
	Sludge	0.0	1.0	0.0	0.0	0.0	
	Water	24.0	24.0	25.0	25.0	25.0	

Table 17: Result of Heat and Chemical Treatmen	nt using LD1 (Umusadege well 2)
Tuble 177 Result of ficut and Chemical fication	t using EDT (Chubuucge wen 2)

Table17. shows the results of Heat and Chemical Treatment using LD1 (Umusadege well 2). After the injection of LD1 demulsifying chemical on the given crude oil emulsion sample, which was heated for 120minutes at field temperature of 50°c, readings were taken at 30minutes interval.

Effect on well2; the separated water in the bottles containing the emulsion with concentration of 0.2ml, 0.4ml, 0.6ml, 0.8ml, 1.0ml & 1.2ml were 10.0ml, 19.0ml, 26.0ml, 25.0ml, 27.0ml, & 25.0ml.

Volume of Demulsifier (ml)	Sample Components	0 minutes	30 minutes	60 minutes	90 minutes	120 minutes	Temp.□
0.2	Oil	97.0	96.0	96.0	96.0	96.0	50
	Sludge	2.0	0.0	0.0	0.0	0.0	
	Water	1.0	4.0	4.0	4.0	4.0	
0.4	Oil	89.0	89.0	89.0	89.0	89.0	50
	Sludge	5.0	5.0	5.0	5.0	5.0	
	Water	6.0	6.0	6.0	6.0	6.0	
0.6	Oil	93.0	92.0	92.0	92.0	92.0	50
	Sludge	1.0	1.0	0.0	0.0	0.0	
	Water	6.0	7.0	8.0	8.0	8.0	
0.8	Oil	95.0	94.0	92.0	92.0	92.0	50
	Sludge	1.0	1.0	0.0	0.0	0.0	
	Water	4.0	5.0	8.0	8.0	8.0	
1.0	Oil	91.0	90.0	90.0	90.0	90.0	50
	Sludge	0.0	1.0	0.0	0.0	0.0	
	Water	9.0	9.0	10.0	10.0	10.0	
1.2	Oil	95.0	93.0	93.0	93.0	93.0	50
	Sludge	1.0	2.0	2.0	2.0	2.0	
	Water	4.0	4.0	4.0	4.0	4.0	

 Table 18: Result of Heat and Chemical Treatment using LD2 (well 1)

## 4.1 Effect of locally formulated demulsifier (LD2) on the crude oil emulsion sample

After the injection of LD2 demulsifying chemical on the given crude oil emulsion sample, which was heated for 120minutes at field temperature of 50°c, readings were taken at 30minutes interval.

Effect on well 1; the separated water in the bottles containing the emulsion with concentration of 0.2ml, 0.4ml, 0.6ml, 0.8ml, 1.0ml & 1.2ml were 4.0ml, 0.0ml, 8.0ml, 7.0ml, 10.0ml, & 4.0ml respectively.

Volume of Demulsifier (ml)	Sample Components	0 minutes	30 minutes	60 minutes	90 minutes	120 minutes	Temp. 🗆
0.2	Oil	70.0	68.0	68.0	68.0	68.0	50
	Sludge	20.0	22.0	22.0	22.0	22.0	
	Water	10.0	10.0	10.0	10.0	10.0	
0.4	Oil	76.0	75.0	75.0	75.0	75.0	50
	Sludge	4.0	5.0	4.0	6.0	6.0	
	Water	20.0	20.0	21.0	19.0	19.0	
0.6	Oil	78.0	75.0	75.0	74.0	74.0	50
	Sludge	2.0	2.0	1.0	0.0	0.0	
	Water	20.0	23.0	24.0	26.0	26.0	
0.8	Oil	79.0	78.0	77.0	74.0	74.0	50
	Sludge	1.0	1.0	0.0	0.0	0.0	
	Water	20.0	21.0	23.0	26.0	26.0	
1.0	Oil	72.0	72.0	72.0	72.0	72.0	50
	Sludge	2.0	2.0	1.0	1.0	1.0	
	Water	26.0	26.0	27.0	27.0	27.0	
1.2	Oil	79.0	77.0	75.0	75.0	75.0	50
	Sludge	1.0	0.0	0.0	0.0	0.0	
	Water	20.0	23.0	25.0	25.0	25.0	

 Table 19: Result of Heat and Chemical Treatment using LD2 (well 2)

## 4.2 Effect of locally formulated demulsifier (LD2) on the crude oil emulsion sample

After the injection of LD2 demulsifying chemical on the given crude oil emulsion sample, which was heated for 120minutes at field temperature of 50°c, readings were taken at 30minutes interval.

Effect on well 2; the separated water in the bottles containing the emulsion with concentration of 0.2ml, 0.4ml, 0.6ml, 0.8ml, 1.0ml & 1.2ml were 10.0ml, 19.0ml, 26ml, 25ml, 27ml, & 25.0ml respectively.

Volume of Demulsifier (ml)	Sample Components	0 minutes	30 minutes	60 minutes	90 minutes	120 minutes	Temp. 🗆
0.2	Oil	99.0	97.0	97.0	97.0	97.0	50
	Sludge	1.0	3.0	3.0	3.0	3.0	
	Water	0.0	0.0	0.0	0.0	0.0	
0.4	Oil	98.0	96.0	96.0	96.0	96.0	50
	Sludge	2.0	4.0	4.0	4.0	4.0	
	Water	0.0	0.0	0.0	0.0	0.0	
0.6	Oil	96.0	95.0	95.0	95.0	95.0	50
	Sludge	4.0	5.0	5.0	5.0	5.0	
	Water	0.0	0.0	0.0	0.0	0.0	
0.8	Oil	95.0	94.0	94.0	94.0	94.0	50
	Sludge	5.0	6.0	6.0	6.0	6.0	
	Water	0.0	0.0	0.0	0.0	0.0	
1.0	Oil	98.0	98.0	98.0	98.0	98.0	50
	Sludge	2.0	2.0	2.0	2.0	2.0	
	Water	0.0	0.0	0.0	0.0	0.0	
1.2	Oil	98.0	98.0	98.0	98.0	98.0	50
	Sludge	2.0	2.0	2.0	2.0	2.0	]
	Water	0.0	0.0	0.0	0.0	0.0	]

 Table 20: Result of Heat and Chemical Treatment using separol (Umusadege well 1)

## 4.3 Effect of foreign demulsifier (separol) on the crude oil emulsion sample

Effect on well 1; after treating for 120minutes, it was observed that separol was not really effective as a treatment chemical for umusadege well 1. This may be due to the inability to remove water from emulsion.

Volume of Demulsifier (ml)	Sample Components	0 minutes	30 minutes	60 minutes	90 minutes	120 minutes	Temp.□
0.2	Oil	90.0	94.0	95.0	95.0	95.0	50
	Sludge	9.0	3.0	1.0	1.0	1.0	
	Water	1.0	3.0	4.0	4.0	4.0	
0.4	Oil	97.0	95.0	92.0	93.0	93.0	50
	Sludge	3.0	4.0	0.0	0.0	0.0	
	Water	0.0	1.0	5.0	7.0	7.0	
0.6	Oil	96.0	96.0	94.0	94.0	94.0	50
	Sludge	4.0	4.0	0.0	0.0	0.0	
	Water	0.0	0.0	6.0	6.0	6.0	
0.8	Oil	92.0	92.0	92.0	92.0	92.0	50
	Sludge	8.0	7.0	1.0	1.0	1.0	
	Water	0.0	1.0	7.0	7.0	7.0	
1.0	Oil	92.0	91.0	88.0	88.0	88.0	50
	Sludge	7.0	7.0	2.0	2.0	2.0	
	Water	1.0	2.0	10.0	10.0	10.0	
1.2	Oil	80.0	80.0	80.0	80.0	80.0	50
	Sludge	18.0	12.0	10.0	10.0	10.0	
	Water	2.0	8.0	10.0	10.0	10.0	

 Table 21: Result of Heat and Chemical Treatment using separol (Umusadege well 2)

## 4.4 Effect of foreign demulsifier (separol) on the crude oil emulsion sample

Effect on well 2; the separated water in the bottles containing the emulsion with concentration of 0.2ml, 0.4ml, 0.6ml, 0.8ml, 1.0ml & 1.2ml were 17.0ml 8.0ml, 4.0ml, 7.0ml, 8.0ml, & 10.0ml.

Volume of Demulsifier (ml)	Sample Components	0 minutes	30 minutes	60 minutes	90 minutes	120 minutes	Temp.
0.2	Oil	95.0	94.0	92.0	92.0	92.0	50
	Sludge	2.0	1.0	1.0	0.0	0.0	
	Water	3.0	5.0	7.0	8.0	8.0	
0.4	Oil	94.0	84.0	84.0	84.0	84.0	50
	Sludge	0.0	0.0	0.0	0.0	0.0	
	Water	6.0	16.0	16.0	16.0	16.0	
0.6	Oil	81.0	80.0	80.0	80.0	80.0	50
	Sludge	0.0	0.0	0.0	0.0	0.0	
	Water	9.0	20.0	20.0	20.0	20.0	
0.8	Oil	85.0	79.0	79.0	79.0	79.0	50
	Sludge	1.0	0.0	0.0	0.0	0.0	
	Water	14.0	21.0	21.0	21.0	21.0	
1.0	Oil	80.0	80.0	80.0	80.0	80.0	50
	Sludge	1.0	0.0	0.0	0.0	0.0	
	Water	19.0	20.0	20.0	20.0	20.0	
1.2	Oil	80.0	80.0	80.0	80.0	80.0	50
	Sludge	1.0	0.0	0.0	0.0	0.0	
	Water	19.0	20.0	20.0	20.0	20.0	

 Table 22: Result of Heat and Chemical Treatment using servo (umusadege well 2)

### 4.5 Effect of foreign demulsifier (servo)on the crude oil emulsion sample

After the injection of servo demulsifying chemical on well 2 sample at various concentration of 0.2ml, 0.4ml, 0.6ml, 0.8ml, 1.0ml & 1.2ml, showed separated water after 120minutes as 18.0ml, 8.0ml, 20.0ml, 16.0ml 20.0ml, & 20.0ml

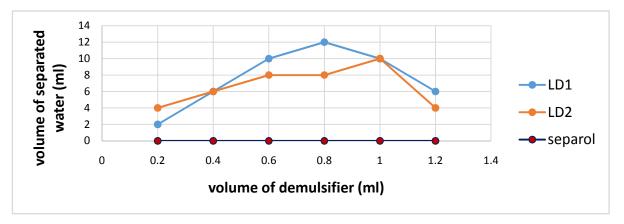


Fig.6: A plot of volume of separated water against volume of demulsifier (well 1)

Fig 6 above shows the volume of separated water from umusadege well 1 emulsion sample, after treament with LD1, LD2 and Separol. Though a little deviation from normal was observed in some of the samples, as shown in Fig 6. As volume of demulsifier increased, ideally the volume of separated water ought to have increased or be maintained, not decreased. This may be due to sludge/ sediment content in the emulsion sample in that particular bottle.

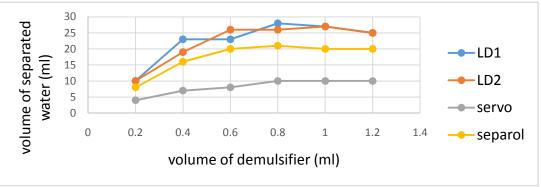


Fig.7: A plot of volume of separated water against volume of demulsifier (well 2)

Fig 7 above shows the volume of separated water from umusadege well 2 emulsion sample, after treament with LD1, LD2, Separol and servo. Though a little deviation from normal was observed in some of the samples, as shown in Fig 7. As volume of demulsifier increased, ideally the volume of separated water ought to have increased or be maintained, not decreased. This may be due to sludge/ sediment content in the emulsion sample in that particular bottle.

## 4.6 The T-tests.

The T-test or t-distribution was developed by William Gosset in 1908 who wrote under the pen name "Student". The t-test is used for testing hypothesis using statistical analysis. Hence the t-test will be used in this work to compare the effectiveness of the various demulsifiers on the given emulsion samples. Local formulated demulsifier (LD1) will be compared with Foreign (separol) on well 1 and LD1 will be compared with foreign (servo) on well 2.

Table 23: showing the amount of water removed l	by LDI and Separol on well 1
---	------------------------------

LD1	2	6	10	12	10	6
Separol	0	0	0	0	0	0

Tble 23 above shows the amount of water separated from well 1 sample, after treatment with 0.2ml,0.4ml...1.2ml of LD1 and separol respectively. This result will be used in table 4.5.2 below, to analyze both demulsifiers using the T-test method.

α-values	Obtained	Table t-ratio	Status	Conclusion
	t-ratio			
0.10	5.148	1.812	O.T.R > T.T.R	Not pass
0.05	5.148	2.228	O.T.R > T.T.R	Not pass
0.01	5.148	3.169	O.T.R > T.T.R	Not Pass

#### Table 24: result from t-test

O.T.R= obtained t-ratio

T.T.R= table t-ratio

NB: Calculations on how these values and results were obtained are contained in the Appendix B section.

## 4.7 Discussion on the effectiveness of LD1 and Separol using the T-test

From the table above, O.T.R was greater than T.T.R in all three  $\alpha$ -values indicating that there is a significant difference between LD1 and separol in comparison for effectiveness in the given crude oil emulsion treatment. Thus cannot be used as a substitute in treating umusadege well 1.

#### Table 25 : showing the amount of water removed by LDI and Servo on well 2

LD1	10	23	23	28	27	25
Servo	8	16	20	21	20	20

Table 25 above shows the amount of water separated from well 2, after treatment with 0.2ml,0.4ml...1.2ml of LD1 and servo respectively. This result will be used in table 26 below, to analyze both demulsifiers using the T-test method.

α-values	Obtained t-ratio	Table t-ratio	Status	Conclusion			
0.10	1.553	1.812	0.T.R >T.T.R	Pass			
0.05	1.553	2.228	O.T.R > T.T.R	Pass			
0.01	1.553	3.169	0.T.R >T.T.R	Pass			

Table. 26: result from t-test analysis.

From the table above, O.T.R was less than T.T.R in all three  $\alpha$ -values indicating that there is no significant difference between LD1 and servo in comparison for effectiveness in the given crude oil emulsion treatment. Thus can be used as a substitute in treating umusadege well 2.

### 5.0 Conclusion

Considering the volume of water removed from crude oil emulsion by different demulsifiers, it was concluded that:-From the plots and data's obtained, it is obvious that the local demulsifier (LD1), was best in treating well 1 and well 2. From the T-test carried out on the samples, it was concluded that LD1 when compared with separol on well 1, does not measure up to the standard set by the T-test analysis. Thus LD1 is not a good match to separol.

For well 2, LD1 when compared with servo, proved to be a good match to servo for the given crude oil emulsion treatment. The cost of any treatment option may determine the economic viability of such reservoir. Cheap local chemicals with demulsification properties can be used to successfully break emulsions. Since the local formulation, LD1 was able to break the crude oil emulsion from umusadege well 2, considering the properties as the time the crude was collected, it can be recommended. Furthermore, students should be encouraged to carry out more research on local products that can be used as substitute for imported ones. I also recommend that the institutions' Petroleum and Gas Laboratory should be well equipped with new, standard and modern equipment / facilities in order to carry out various analysis and practical.

## 6.0 References

- [1] Abdurrahman, et al, (2007) "Stability and demulsification of the water- in- crude oil emulsions via chemical method", university of Malaysia Pahang. Page 7-8.
- [2]. Society of Petroleum Engineers (SPE), (accessed 1<sup>st</sup> July, 2015): "crude oil emulsion". Available from petrowiki.org/oil\_emulsion.
- [3] Tharwat, F.T. (2013): 'Emulsion Formation and Stability', Wiley-VCH Verlag GmbH & Co. KGaA, London, First Edition, pp.3.
- [4] Chevron Nigeria Limited, (2005): "Manual on Emulsion Treatment"; chevron training program. Page. 2-10.
- [5] HTS Consultants, (2004): "Demulsification" in Oilfield Production Chemicals and Microbiology page 1 20.
- [6] Kokal. S.L. (2005): "crude oil emulsion". Petroleum Engineering Handbook, SPE. Richardson, Texas.
- [7] Petroleum Extension service (PES), (1990). "Treating Oil Field Emulsion" 4th edition, Austin Texas. page 1-10.

[8] Gary, W.S and Moshen. Z., (1999). "The practiced art of emulsion Resolution in electrostatic processes". Presented at spring meeting, Houston