International Journal of Engineering & Technology
CENTRE OF PROFESSIONAL RESEARCH PUBLICATIONS
ISSN 2049-3444
International Journal of Engineering and Technology Volume 5 No. 9, September, 2015

# Experimental Study of the Effect of Temperature on Demulsification

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## ABSTRACT

The major problem, when dealing with the crude oil is the formation of persistent water in the crude oil emulsion thus performed the stable emulsion. Emulsions occurred when there is water as immiscible liquids present in the crude oil. Water is normally present in crude oil reservoirs or is injected as steam to stimulate oil production. Water and oil can mix while rising through the well and when passing through valves and pumps to form in most cases relatively stable dispersions of water droplets in crude oil.This thesis investigates the stability of emulsion using crude oil sample from the Niger Delta and simulation of Stoke's Law of settling was investigated. In this study, two crude oil samples were obtained and emulsion was prepared on a laboratory scale. Thermal method (microwave heating) was used in the demulsification of emulsion while simple spreadsheet model using Oracle Crystal Ball was used in the simulation study. The stability studies were carried out by analysing operating conditions such as stirring intensity, types of surfactant used, effect of viscosity on shear rate using varied ratio of water-in-oil emulsion. The performance of microwave heating demulsification method was compared with conventional demulsification methods such as chemical demulsifiers and gravity separation which shows that chemical demulsifier offers appreciable separation but less than effect of microwave heating while gravity settling offered no appreciable separation. For simulation study, a simple model analysis was carried out on Stoke's Law and from the results obtained using plots of Sensitivity chart, Spider and Tornado charts, it shows that the difference in density of water and oil is the most sensitive parameter which impacts the forecast (settling velocity) more than viscosity of oil and the mean diameter of droplets in agreement with Stoke's Law.

**Key words***: Water In Oil Emulsion, Emulsion Stability Using Stoke's Law of Settling Velocity, Demulsification Methods, Microwave Heating, Spider and Tornado, And Sensitivity Chart.*

## 1. INTRODUCTION

It is well recognized that the energy consumption per capital and crude oil play an important role in providing energy supply of the world because crude oil is the most readily exploitable source of energy available to humankind, and also source of raw materials for feed stocks in many of the chemical industries (Holmberg; 2001). However, there are about 80% of exploited crude oils exist in an emulsion state all over the world (Xia et al., 2004) and to avoid issues relating to quality of crude oil during the refining process, which would lead to enormous financial losses and economic penalties, the crude oil have to be treated.

In the oil field, the two basic types of emulsions are water-in-oil (w/o) and oil-in-water (o/w). Ali and Alqam, 2000 stated that more than 95 % of the crude oil emulsion formed in the oil field are of the w/o type. The concept of microwave heating of emulsions was first suggested by Klaila, 1983 and Wolf, 1986. In the past 20 years, Microwave (MW) energy has been widely applied in food and chemical processing for heating, thawing (melting), sintering of ceramics and many others (Ayappa et al., 1992 and Kim, et al., 1996)

An efficient method of resolving crude oil emulsions, the chemical method, isbased on the addition of reagents (demulsifiers) which destroy the protective action of hydrophobic emulsifying agents and allow the water droplets to coalesce. There are anionic, cationic and nonionic surfactants that have been used as demulsifiers (Selvarajan et al., 2001). These demulsifiers permit agglomeration, coalescence and gravity settling of the water droplets (Staiss et al., 1991).

## 2. CHALLENGES OF WATER IN OUL EMULSION

The major problem, when dealing with the crude oil is the formation of persistent water in the crude oil emulsion thus performed the stable emulsion. Emulsions occurred when there is water as immiscible liquids present in the crude oil. Water is normally present in crude oil reservoirs or is injected as steam to stimulate oil production. Water and oil can mix while rising through the well and when passing through valves and pumps to form in most cases relatively stable dispersions of water droplets in crude oil. Emulsions of crude oil and water can be encountered at many stages during drilling, producing, transporting and processing of crude oils and in many locations such as in hydrocarbon reservoirs, well bores, surface facilities, transportation system and refineries. Microwave offer a fast heating treatment with uniform temperature distribution to the subjected sample. This study is aimed to investigate the effect of microwave radiation in the breaking of the emulsion and separation of water from crude oil and how the increase in temperature will affect coalescence.

## 3. PREVIOUS WORK

Recent work indicates that the viscosity of an emulsion is related to its stability (NRT Science & Technology Committee, 1997). Stability is widely used to refer to the persistence of an emulsion in the environment, and has been identified as an important characteristic of water-in-oil emulsions.

Stability is a consequence of the small droplet size and the presence of an interfacial film on the droplets in emulsions, which make stable dispersions. That is the suspended droplets do not settle out or float rapidly, and the droplets do not coalesce quickly. According to Schramm, 2007 the definition of emulsion stability, it is considered against three different processes; creaming (sedimentation), aggregation and coalescence" as shown in Figure (2.3)

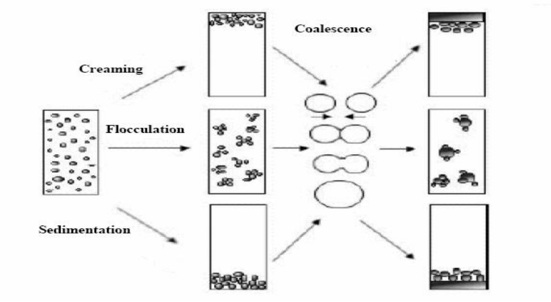


**Figure1: Processes Taking Place in an Emulsion Leading to Emulsion Breakdown and Separation [Auflem, 2002].**

According to NRT Science & Technology Committee, 1997, emulsion can be categorized into stable, unstable and meso-stable emulsions according to stability:

Stable emulsions will persist for days, weeks and longer. In addition, stable emulsion will increase with viscosity over time. Increasing alignment of asphaltenes at the oil-water interface may cause increasing of viscosity.

Unstable emulsions usually persist for only a few hours after mixing stops. These emulsions are ready to separate into oil and water due to insufficient water particle interactions. However, the oil may retain small amounts of water, especially if the oil is viscous.

Meso-stable emulsions are probably the most common emulsion that was formed in the fields. These emulsions can be red or black in appearance. This emulsion has the properties between stable and unstable emulsions. It is suspected that these emulsions contain either insufficient asphaltenes to render them completely stable or contain too many destabilizing materials such as smaller aromatics. The viscosity of the oil may be high enough to stabilize some water droplets for a period of time. Particles, which are water-wet, tend to stabilize (O/W) emulsions while those oil-wet tend to stabilize (W/O) emulsions (Auflem, 2002).

There are two factors that affect the emulsion stability stated by Kim, 1995:

Viscosity: The application of heat and the addition of demulsifiers can reduce the viscosity. As the results, the rate of water droplets settlement and the mobility of water are increased and lead to collisions, coalescence, and further increase in the rate of separation.

Density difference: Heat application to the emulsion will decrease the density of the oil at a greater rate than that of water and thus allows more rapid settling of the water. This is because the difference in densities of the two liquid phases may be increased. Dehydration of heavier oil is typically more difficult compared with light oil, as its density is closer to that of water. The stability of emulsion is determined by the interaction between the particles during the collisions. As studied from previous researchers (Sullivan and Kilpatrick, 2002; Ariany, 2004; Abdurahman et al., 2007) there are at least four mechanisms by which emulsions are stabilized; electrostatic repulsion, steric repulsion, the Marangoni- Gibbs effect, which retards film drainage and thin film stabilization.

## 4. METHODS OF EMULSION BREAKING

The breaking of emulsion is necessary in emulsion treatment. Methods currently available for demulsification can be classified as chemical, electrical and mechanical (Abdurahman et al., 2007). In generally, methods to induce phase separation for water-in-oil emulsions can be applied using chemical or heating the emulsion. Chemical demulsifiation is common method used and since the 1930s, demulsifier has been used in treating emulsion (Wu et al., 2003). Conventional thermal heating involved the breaking of emulsion due to reduction of viscosity of continuous phase. However, currently microwave has been studied an alternative method to treat the
emulsion. The concept of microwave demulsification is first introduced by Klaika (1978) and Wolf (1986).

## 5. MICROWAVE MECHANISMS AND THEORY

It has been known for long time that microwaves can be use for heat materials. In fact, the development of microwave oven for the heating food has been more than a 50-year history (Wu, 2003). Recently, microwave is investigated as an alternative method to break the emulsion. Microwave irradiation is being studied as tool for demulsification. In the electromagnetic spectrum, the microwave radiation is located between infrared radiation and radio waves. Microwaves have wavelengths of 1 mm -1m, corresponding to the frequencies between 0.3 and 300 GHz (Lidstrő́m et al., 2001). From Lidstrő́m et al. (2001), in general in order to avoid interference, the frequency at which industrial and domestic corresponding to 2.45 GHz, but other frequency allocations do exists. The heating of liquids using microwaves can be explained by the interaction of matter with the electric field of the incident radiation, causing the movement of ions as well as that of induced or permanent molecule dipoles. The movement of such species can cause heat generation.
Microwave is very short waves of electromagnetic by penetrate the electromagnetic energy through materials. Since microwave energy transfer involve electric and magnetic field, the interaction materials is selected based on electric and magnetic component.

### 4.1. Method of Study

To accomplish the objectives of scopes of this research, the study was carried out in three stages namely, emulsion stabilization, microwave batch process and simulation.

 

The first and second stages are experimental while third stage is simulation. Experimental methods involve the procedure for emulsion sample preparations, experimental set-up for microwave equipment, sample characterization using microwave processes (sample analysis, measurement of emulsion density, droplet size and viscosity. Details of the experimental are discussed below.

**4.2. Work Flow of Study**

The study has been conducted by using two types of crude oil from lighter to heavier crude oil obtained from a field in the Niger Delta region. Basically this study is summarized by three parts. Firstly each crude oil was characterized in term of physical properties. The emulsion would be prepared depend on the requirement of volume fraction and each prepared emulsion was done by using dilution and conventional drop tests. Only water in oil emulsion has been used in the emulsion stability part. The most stable emulsion was selected in this screening part before adding chemical demulsifiers and finally, heat was applied by using conventional microwave heating.

**5.1 Material Selection**

Two types of crude samples, denoted as crude oil A (lighter oil) and crude oil B (heavier oil), obtained from the Niger Delta region were used. Two chemical are selected to characterize and investigate the emulsion stability. In this study, sodium dedocyl sulfate (SDS) 10% in water and Propylene oxide were used to stabilize the emulsion while 99% hexylamine 98% pentylamine act as demulsifier.

**5.2 Sample Preparation**

Basically emulsion was prepared by adding water to crude oil in prescribed ratio. About 50 ml of emulsion sample was prepared in this study. Firstly, about 0.1% emulsifier of crude oil added to crude oil (mixing solution) and was stirred vigorously for 1 minute. Water is added gradually to the mixing solution and was agitated vigorously at a temperature of 28o C for 5 minutes. Then, the type of emulsion (W/O or O/W) was inferred by a conventional drop test (Evdokimov et al., 2008). The concentrations of water (internal phase) in the samples were varied by volume. Emulsions were observed over a period of time to provide a qualitative measure of the stability.

**8. DEMULSIFICATION PROCEDURES**

The stable emulsion would be treated in demulsification part. The demulsification experiments were performed batchwise using conventional heating (microvawe. The demulsification efficiency was evaluated by measuring the percentage of water separated from the emulsion as a function of time. The amount of separated water is the most appropriate measure of the emulsion stability of water-in crude oil (W/O) emulsions (Jiang et al., 2007).

Emulsion was prepared by comparing two ratios; 30-70 % W/O and 50-50 % W/O emulsion. The chemical demulsifier was added into the emulsion at 1500 rpm in 30 seconds to perform homogenous solution. The effectiveness of demulsifier was evaluated by observing the percentage of water separation from gravity settling. In conventional heating method, the prepared emulsion was heated using microwave heating method. Temperatures at surface and bottom beaker were measured using thermometer. While for gravity settling, emulsion was prepared and separated by sedimentation.

## 6. DATA ANALYSIS

The experiment was analyzed by observation free water forms at the bottom of measuring cylinder and calculated as percentage water separation as shown in Equation 3.6. This is the best indicator in measuring stability of emulsion and used from previous researcher (Jiang et al., 2007). The amount of water separated or response of yield (%) was calculated as equation below:

Water separated (%) =

$$\frac{vol.\ of\ \ water(V),ml}{Original\ Vol\ \ of\ \ water(V_o),ml} \times 100 \quad \ldots\ldots\ldots$$

(eqn.1)

Each data obtained would be plotted using Microsoft excel tool.

## 7. SENSITIVITY ANALYSIS

The results from the demulsification of microwave heating were used as the variable in simulating the formulation in order to investigate the most sensitive parameter on settling velocity (Equation 3.7) in breaking the prepared emulsion by using an experimental design. An experimental design determined the effect of the independent variables on the dependent variable (settling velocity) by simulating the independent variables and observing the impact of each of these variables on the response (settling velocity). In this section, the software Oracle Crystal Ball version 11 was employed for simulation and data analysis.

$$v_m = \frac{gD^2(\rho_w - \rho_o)}{18\mu_o}$$

…….….…………………….…. (eqn.2)

Where:

v is the settling velocity of the water droplets

D is the mean diameter of the droplets

g is the acceleration caused by of gravity

r is the radius of the droplets

(ρw – ρo) is the density difference between the water and oil μ is the oil viscosity

### 8.1 Simulation Design

Crystal Ball is a software tool that measures the input of each model variable one at a time, independently, on a target forecast or for assessing the impact or risk associated with a particular situation or managing risk in a dynamic business environment. Assessing the impacts of inputs on a target forecast using crystal ball relies on developing a mathematical model in Excel that represents a situation of interest. The crystal ball uses the point estimates with probability distribution assumptions and forecast distribution of the output using Monte-Carlo Simulation. Using Crystal Ball/Monte-Carlo simulation, we need to determine which element has the most influence on the parameter of interest and which element is more important. To use Crystal Ball for this purpose, the following steps are required:
Build a mathematical flow model of the intended target/project in a spreadsheet.

• Identify the independent variables.
• Define a realistic model
• Generate the distribution or the sensitivity of the parameter criteria.

## 9. RESULTS AND DISCUSSION

Each crude oil has its own complexity nature in characteristics and offers individual problems. The API gravity, the viscosities and water content of each crude samples were determined by using a laboratory glass viscometer (150-601B) and the viscometer constant which is temperature dependent was obtained from glass viscometer tables. In this study the value used is 0.03641492 at ambient temperature of 29oC.

**Table 1: Physical properties of crude samples**

| | Crude oil A | Crude Oil B |
|---|---|---|
| Density (g/cm$^3$) | 0.835 | 0.924 |
| $^o$API density | 37.96 | 21.64 |
| Viscosity (cp) | 26.2 | 62.5 |
| Water content (%) | 8 | 3.5 |

Results had shown that crude oil B was heavier and the lighter was crude oil A. This trend has good confirmation with Ariany (2004), which viscosity changes in small magnitude usually proportional to density change, whether caused by temperature or pressure. The different physical properties of crude oil are obviously showed from API density which strongly agreed that crude oil B is the heavier crude oil.

### 9.1 Effect of surfactant types on stability of emulsion

Emulsion was prepared at different surfactants to discover the most stable emulsion as problems encountered in industry. The most important task in preparation of emulsions is the selection of suitable surfactant which will satisfactorily emulsify the chosen ingredients at a given temperature. It has also been recognized that that there is a relation between the contributions of the polar hydrophilic head and the nonpolar lipophilic tail which related to the polarity of surfactant (Chen and Tao, 2005).

As observed from the figure 4.1, Sodium dodecyl sulfate (SDS) is anionic group and water-soluble obtained the highest percentage of water separation (55% after 60mins) while Propylene oxide (PO) with appreciable solubility in water obtained a lesser percentage of water separated compared to SDS.

### 9.2 Stirring intensity (rpm) effect on emulsion stability.

Emulsion stability is also affected by the intensity at which it's being stirred. During emulsification process, the interfacial area between phase's increases hence energy in the form of mechanical energy is required to reduce the interfacial area and proceed with emulsification. The more the energy required to produce small droplets size using very intense agitation thus makes barriers of droplets to coalesce. According to Hannisdal, 2005, the break-up of droplets strongly depends on the type and intensity of the flow which is determined by the rotational speed and geometry of the agitator.

The percentage of water separated as observed in figure 4.2 shows effect of varied stirring intensity on emulsification. In this study, three different intensity rates were considered which are 500rpm, 1000rpm and 1500rpm respectively.

At lower intensity rate of 500rpm, after 15 minutes, the amount of water separated was 26% when compared to 35% and 41.6% for 1000rpm and 1500rpm respectively. It shows that at lower intensity rate, the surfactant does not mix well with the crude oil which can be classified as being heterogeneous. However, at higher stirring rate of intensity, the effectiveness of surfactant to adsorb onto the droplets was increased as observed in the 1500rpm used in this study.

### 9.3 Effect of stirring intensity on droplet size.

The effect of stirring rate also influences the diameter of droplets and this equally provides vital information on the efficiency of emulsification process. Figure 4.3 demonstrates the mean of droplets diameter, d at varied phase ratio for the crude sample where the droplets size (mean) is inversely proportional with stirring intensity. Both phases ratio showed discrepancy for droplets mean diameter where 50-50% w/o emulsion has higher diameter compared to 30-70% w/o emulsion. The intensity used ranges from 500rpm to 2000rpm.

### 9.4 Rheological property of w/o emulsion (effect of shear rate)

Plots of apparent viscosity versus shear rate as shown in Figure 4.4. It was used to test the effect of phase ratio in examining emulsion behaviour. Two different ratios of 55-45% w/o and 60-40% of emulsion of sample B were prepared and tested. Emulsion in both ratios exhibited non-Newtonian behaviour due to trend of graph obtained. The gradual break-up of interparticles structure may be primarily responsible for this behaviour.

### 9.5 Microwave demulsification study on water-in-oil (w/o) emulsion

Both water and crude oils were used in microwave heating. The crude oil samples and water were place in microwave for heating to test the effect of temperature increase on the samples. From figure 4.5 obtained, it was observed that the temperature of water rapidly increase compare to that of the crude samples. This may be due to the dipole nature of water. However at temperature of about 102oC and at 82 sec, temperature for water was observed to be constant because rise in water is more uniform more than oil due to defined convective mechanisms of heat transfers than oil. The continuous increase in temperature as observed in the crude oil sample may be due to the presence of wax and asphaltenes as detected by Gunal and Islam, 2000. In this study, the chemical compositions of the crude samples were not studied.

### 9.6 Effect of temperature distribution on varied water-oil ratios

Only the heavier crude sample was test for effect of temperature distribution on emulsion formulation ratios. Three phase ratios of 50-50%, 30-70% and 20-80% were tested respectively. Figure 4.6 show that the 20-80% emulsion had the lowest temperature increase at 100 seconds with maximum temperature of 52oC as compared with 72oC for 50-50% ratio within the same time intervals. It can be said that as the phase ratio increases higher temperature is attained due to more readily microwave irradiation absorbed through heating.

### 9.7 Effect of microwave power on w/o in emulsion

The effectiveness of microwave power on water separation was carried out. Two microwaves with power ratings of 360 and 450 watts were used in this study. Figure 4.7 shows the efficiency of water-oil separation using varying microwave power ratings. It is observed that increase in power rating causes substantial efficiency in phase separation.

### 9.8 Comparison of microwave heating with conventional methods

In this study, the efficiency of water separated from both microwave heating, gravity settling and chemical demulsification were considered. As shown in figure 4.8a and 4.8b, the microwave demulsification offered rapid separation compared to chemical demulsification while gravity settling offered no appreciable separation. This was because of microwave quickly heat emulsion, huge temperature rises and reducing the viscosity of continuous phase, thus favouring the contact among water droplets and coalesce the droplets. Besides, microwave is non-destructive method on emulsion, do not required addition of chemical agents, which do not implies for further waste water treatment (Rajakovic and Skala; 2006).

### 9.9 Simulation Study

Crystal Ball was used to run a simulation study. A simple model using Equation 3.7 was built into a spreadsheet. The density of water used in the study is 1.193g/cm3 while that of oil is 0.835g/cm3, (density of crude sample A). Assumptions were defined on the inputs and the impacts on settling velocity were investigated using the results of the Tornado and Spider charts obtained from the simulation study. The simple spreadsheet model for simulation is as shown in Table 4.2.

Oil/water separation is usually based on a gravitational separation from theory. Because water has a higher density than oil, water droplets have a tendency to settle down. Stokes' Law approximates the settling rate of water droplets as stated in equation 3.7.

The law suggests that the settling velocity is increased by:

- Increasing the density difference between water and oil
- Reducing the viscosity
- Increasing the droplet size

In this study however, the simulation results obtained as shown in Figures 4.9, 4.10 and 4.11, shows that the difference in density of water and oil is the most sensitive parameter which impacts the forecast (settling velocity) more than viscosity of oil and the mean diameter of droplets in agreement with Stoke's Law. Hence, it means that if larger difference in density of oil phase and water phase can be achieved this will improve efficiency of both microwave heating and other conventional methods considered in this study.

 

## 10. CONCLUSION

The behaviour of water-in-oil emulsion was investigated by studying the stabilisation and demulsification of emulsions by various methods (microwave heating and chemical demulsifications).

The following conclusions could be obtained:

• The stability of emulsion was influenced by surfactant type. This is due to the contributions of the polar hydrophilic head and the nonpolar lipophilic tail in relation to the polarity of the surfactants. In this study surfactant SDS gave higher separation efficiency than PO.

• The rate of stirring of emulsion (stirring intensity) also affects emulsion stability. The more the energy, the more the barriers of droplets breaks causing more droplets to coalesce. The 1,500rpm provides better separation efficiency than 500rpm and 1,000rpm as observed in this study.

• The behaviour of emulsion observed through plotting graph of apparent viscosity versus shear rate indicated emulsion behave as non-Newtonian as seen in figure 4.4.

• Analysis of demulsification was conducted by comparing microwave heating with conventional methods such as chemical demulsification, and settling gravity. Chemical demulsifiers were added to both crude samples before enhancing separation using microwave heating. It was observed that increase in from 360W to 450W power rating causes substantial efficiency in phase separation.
Also, when microwave heating was compared with chemical demulsification and gravity settling, it was observed that chemical demulsifier offers appreciable separation but less than effect of microwave heating while gravity settling offered no appreciable separation.

• In the simulation study, a simple model analysis was carried out on Stoke's Law and from the analysis of results obtained using plots of Sensitivity chart, Spider charts, it shows that the difference in density of water and oil is the most sensitive parameter which impacts the forecast (settling velocity) more than viscosity of oil and the mean diameter of droplets in agreement with Stoke's Law.

**Recommendations**

Based on this research work, there is need to study the effect of mixing on emulsion stability as well as the effect of demulsifier overdosing on emulsion stability. Further study on the effect of dielectric properties such as wavelength and depth penetration of microwave heating on emulsion stability is also recommended.

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# APPENDIX

**Table 1: Simple spreadsheet model for Stoke's Law simulation study**

| Input | Value | unit | Symbol | Assumptions | | |
|---|---|---|---|---|---|---|
| viscosity of oil | 26.2 | cp | $\mu_o$ | Uniform | 20 | 30 |
| Mean diameter of droplet | 60 | $\mu m$ | D | Normal | 60 | 2 |
| Density difference between oil and water | 0.358 | $g/cm^3$ | $(\rho_w - \rho_o)$ | Uniform | 0.1 | 0.4 |
| Acceleration caused by gravity | 9.8 | $m/s^2$ | g | Constant | | |

| Output | | |
|---|---|---|
| Settling Velocity ($v$) | 26.7817 | $m/s^2$ |

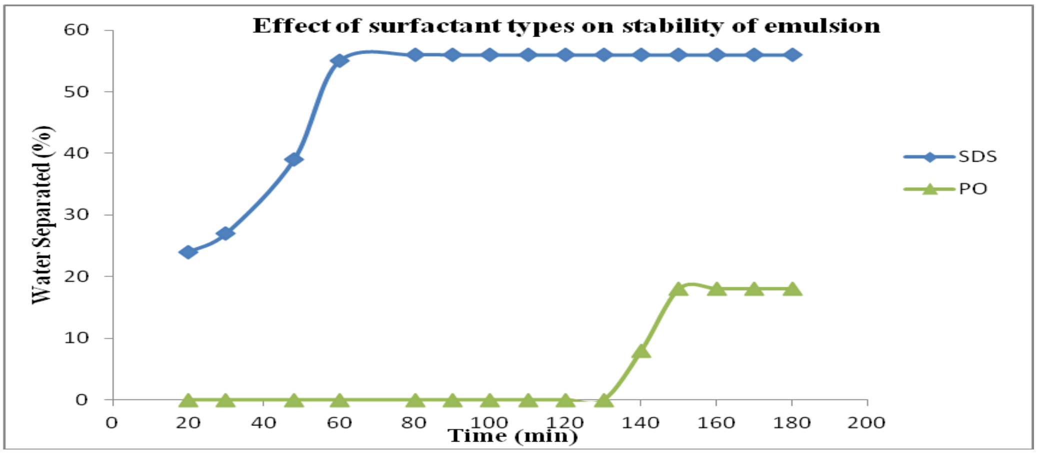


**Figure.1: Effect of surfactant types on stability of emulsion**

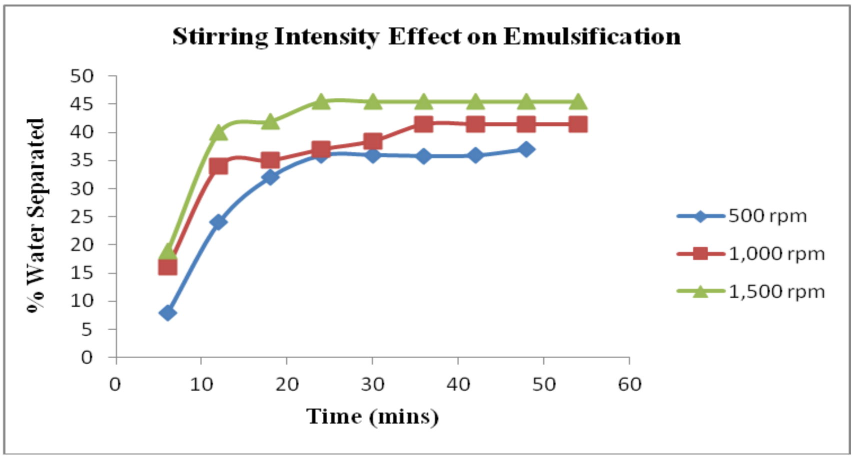


**Figure.2: Stirring Intensity Effect on Emulsification**

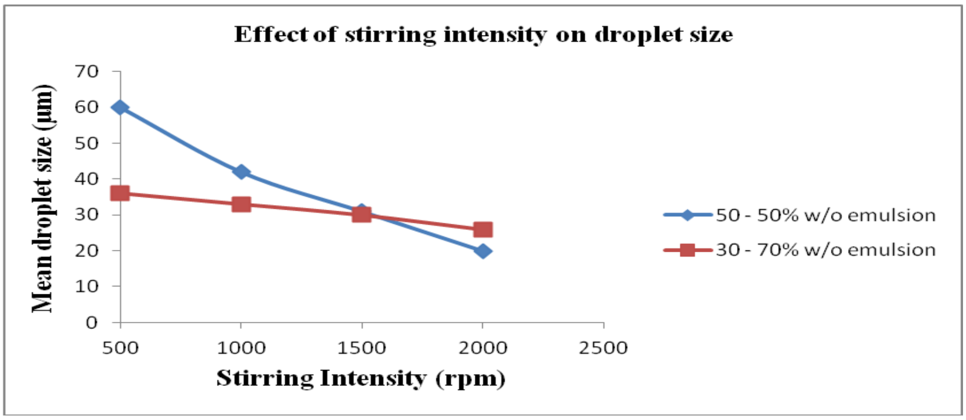


Figure 3: Effect of stirring intensity on droplet size

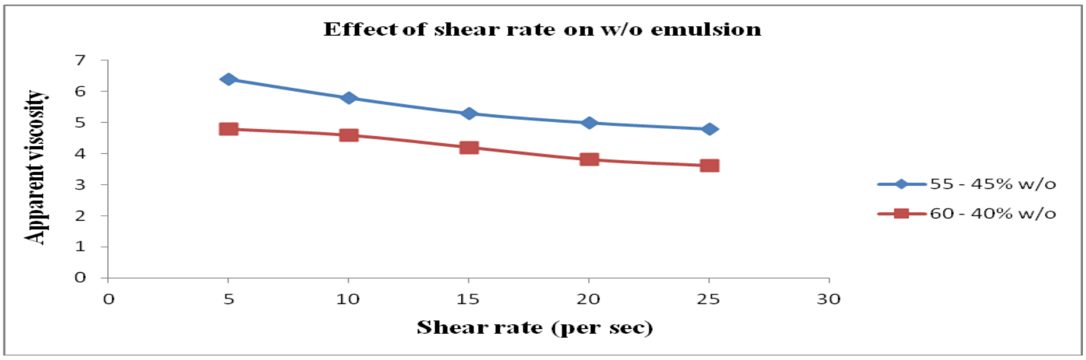


Figure 4: Effect of shear rate on w/o emulsion

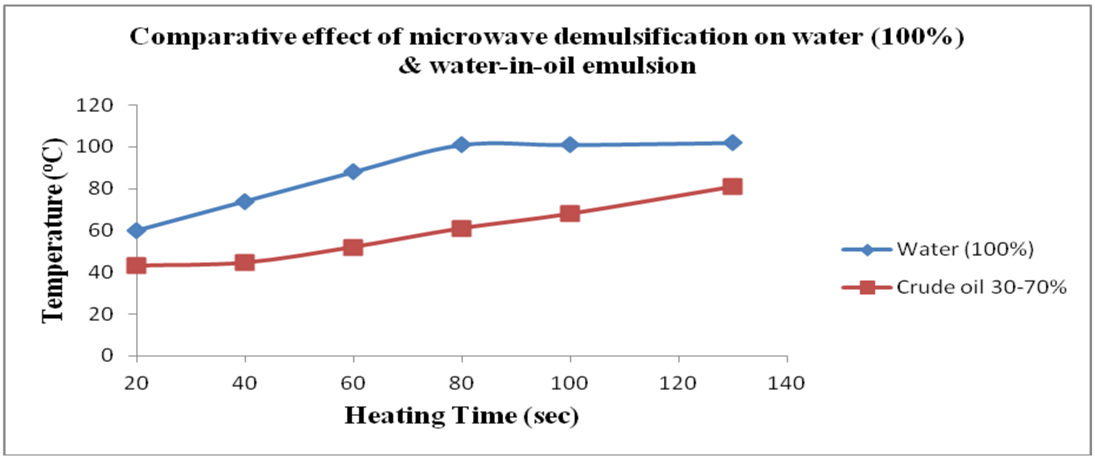


Figure.5: Comparative effect of microwave demulsification on water (100%) & water-in-oil emulsion

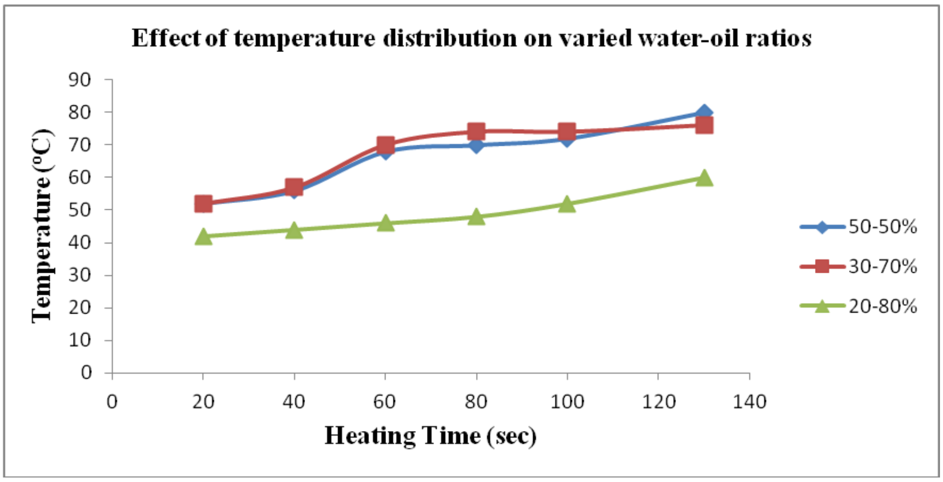


Figure.6: Effect of temperature distribution on varied water-oil ratios

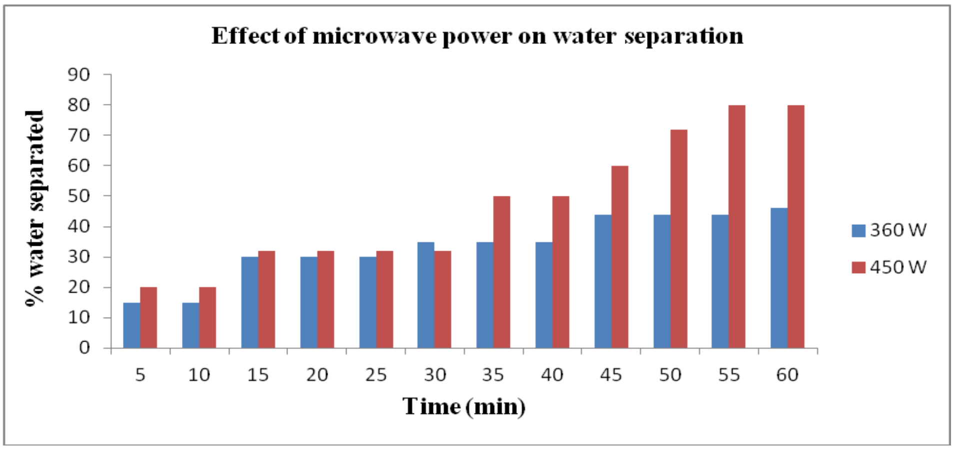


Figure 7: Effect of microwave power on w/o in emulsion

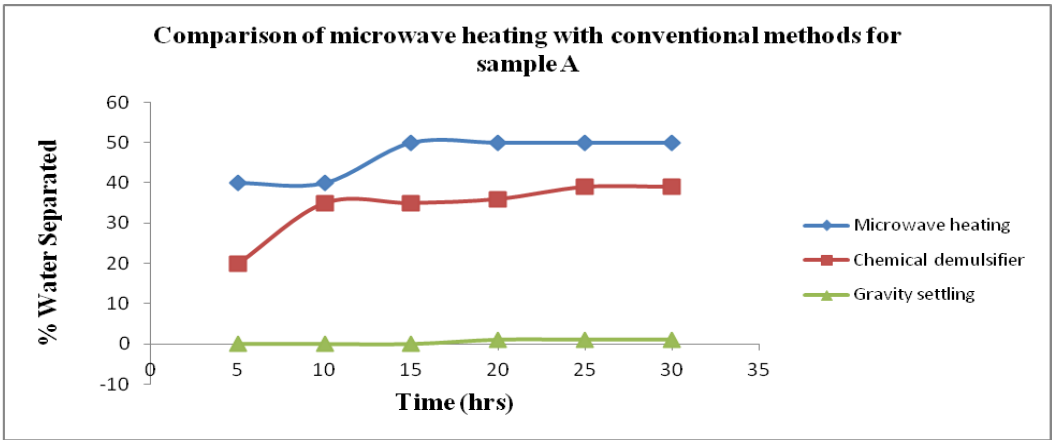


Figure 8a: Comparison of microwave heating with conventional methods.

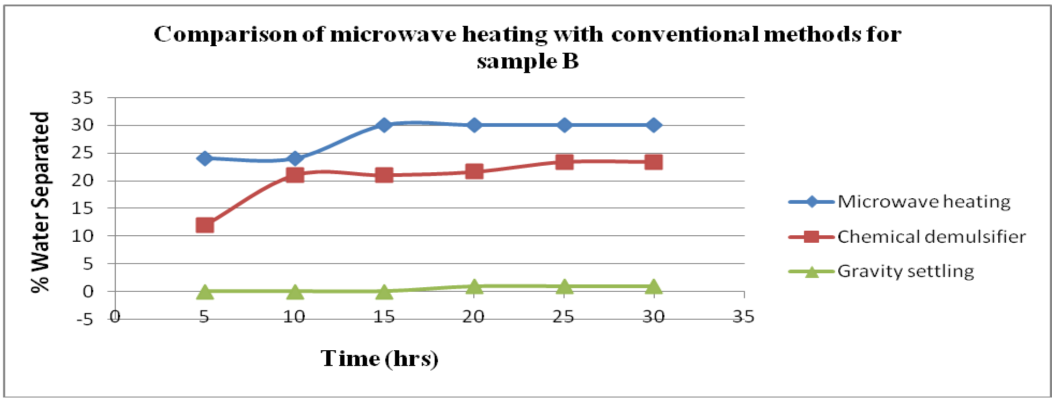


Figure 8b: Comparison of microwave heating with conventional methods.

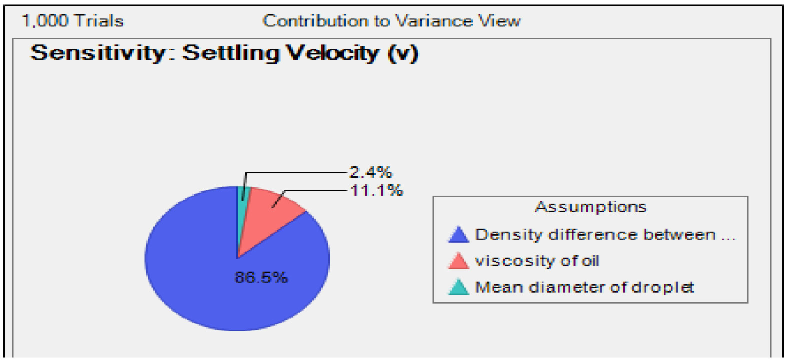


Figure 4.9: Sensitivity chart showing contribution to variance

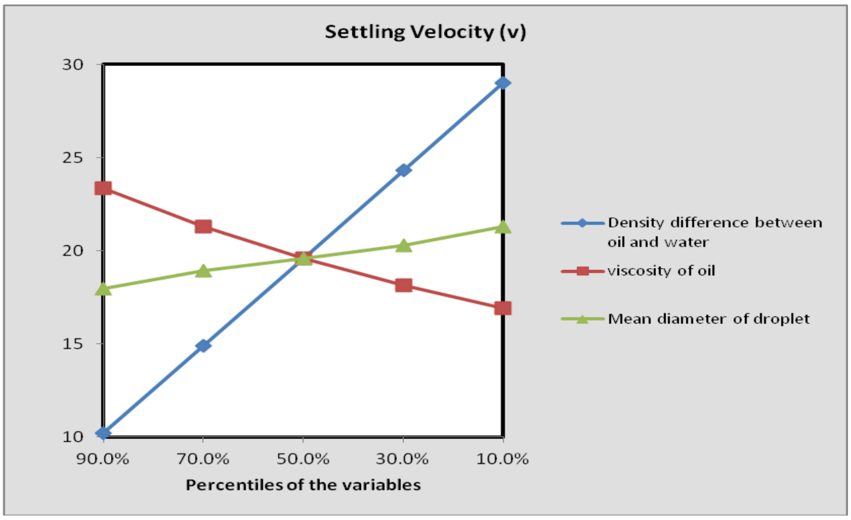


Figure10: Spider chart showing degree of variance of parameters on settling velocity