Optimization of Heat and Chemical Applications toward Effective Emulsion Resolution

by

Umoh Etimbuk Bassey

15649

Dissertation submitted

In partial fulfilment of the requirement for the

Bachelor of Engineering (Hons)

(Petroleum)

SEPTEMBER 2014

Univerisit Teknologi PETRONAS

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CERTIFICATION OF APPROVAL

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Approved by,

Dr. Sulaimon Aliyu Adebayor

CERTIFICATE OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is mine except where specified in references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

UMOH ETIMBUK BASSEY

ACKNOWLEDGEMENT

Foremost, I would like to thank the Almighty God for guiding and guarding me hitherto. The strength and will to carry on this far comes only from God.

My appreciation goes to my wonderful supervisor, Dr Sulaimon Aliyu Adebayor, whose thorough guidance made this work what it is. Furthermore, I would also like to thank Mr. Bamikole Joshua for his unending will to help at all times.

I am indebted to my lovely family for their unending support in my career path. They have been supporting me financially, spiritually and otherwise. They have been a great source of inspiration to me.

Not forgetting my university, Universiti Teknologi Petronas (UTP) for having created a citadel of learning to churn out well rounded students. I do appreciate the provided platform, facilities and funds to make this project a success.

ABSTRACT

Water-in-oil emulsions are unstable colloidal dispersions with water as the dispersed phase and oil as the continuous phase. Water-in-oil emulsions are the most common emulsions found in the oil field. It occurs in almost every phase of the oilfield from the sand face to the surface. Emulsion constitutes numerous problems in the oil and gas industry ranging from flow problems due to high viscosity of emulsions to increase in cost in the use of demulsifiers. In resolving emulsions, heat, chemical or a combination of both methods can be used. Demulsifiers destabilizes the emulsifier film in the emulsion and thus rupture the film to resolve the emulsion. While heat decreases the viscosity of the emulsion by increasing the entropy of the system. The heat excites the enclosed water molecules and thus rupture the film.

Research has shown that the use of chemical and heat to resolve emulsion is effective. In this study, synthetic emulsions in water-oil ratios of 20/80 and 50/50 were formed in the laboratory. Emulsions were separated into 10 ml measuring cylinders, then 0.5 ml of demulsifier was added before exposing it to heat from a domestic microwave oven. Three cases were experimented; the use of microwave heat only, demulsifier only and the use of both demulsifier and microwave heat.

The results obtained showed the effectiveness in the combination of heat and chemical in emulsion resolution. 70% water was recovered for the case of 20/80 water-oil ratio at a microwave exposure of medium low microwave power (336 Watt) for 60 seconds. In the 50/50 water-oil ratio, 100% water was recovered with just demulsifier use.

In resolving tight water-in-oil emulsions, the synergy effect of heat and chemical is required for effectively resolving the emulsions. The findings showed an optimal exposure time to which the emulsion should be heated, after which it is a waste of energy to continue heating. It is recommended that this procedure be repeated for varying amount of demulsifier. Furthermore, various water-oil ratios such as 10/90, 30/70, 40/60 should be experimented using this method to understand and analyse the effect on water-oil ratio in resolving emulsions using a combination of microwave and demulsifier.

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CHAPTER 1

INTRODUCTION

1.1. Background

Emulsions are thermodynamically unstable colloidal dispersions containing at least one immiscible fluid. In simplified terms, emulsion is the result of mixing two or more liquids with at least one of it being immiscible. In the process of this mixing or agitation, small droplets form due to the immiscibility of the liquids. Emulsions usually have two phases, the dispersed or internal phase and the continuous or external phase. The continuous phase suspends or carries the dispersed phase. The dispersed phase is contained in small droplets formed and this is carried or suspended by the continuous phase. Emulsions are found in various industries such as oil and gas, food and chemical industries. In the food industry for instance, emulsions are induced to form gelatinous foods. In the oil and gas industry which is the focus of this research, emulsions are formed naturally and not induced.

Two common types of emulsions are found in the oil and gas industry, water-in-oil emulsions and oil-in-water emulsions. The former has oil as the continuous phase and water as the dispersed phase while the later has oil as the dispersed phase and water as the continuous phase. For the purpose of this research, water-in-oil emulsion is the focus because it is the most abundant type of emulsion found in the oil and gas industry.

Emulsions can be resolved by various means which includes but not limited to the use of heat, chemical and electricity. In the course of this research, the effective means of combining heat and chemical to resolve emulsions is being looked into.

1.2. Problem Statement:

Emulsions constitute a major problem in the oil and gas industry. Emulsion is almost inevitable in the industry. About two thirds of produce from new fields have water-in-oil emulsion problems. Emulsions have higher viscosity than oil and water, this causes flow problems by causing high pressure drop in flow lines, as increase in viscosity means a higher resistance to flow. Also, emulsions causes increase in use of demulsifier, this in turns contributes to cost of production. Other than that, emulsion reduces the quality of oil produced and sometimes makes it unsalable, thus the emulsion has to be resolved so as to enable the produced oil meet the international standard of contained basic sediment and water (Al-Ghandi et al., 2007, 2009; Nguyen & Sadeghi, 2011; Dosunmu et al., 2012).

1.3. Project Objectives:

At the end of this research the following objectives are expected to be met;

1. Investigate and discuss the kinetics behind the reaction involved in using chemical method and the mechanism of heat transfer involved in using heat method.
2. Develop an approach to give maximum yield of emulsion resolution using a combination of heat and chemical methods.

1.4. Scope of study:

The scope of this study covers problem identification, intensive literature review of past works done that is related to the project, planning a comprehensive experimental design that will aid in solving the problem at hand and also meeting the research objectives. Experimenting and reporting the combined effect of heat and chemical on water-in-crude oil emulsion is included in the scope of study. That is, using a demulsifier together with heat from a microwave oven at varying amount, then observing and reporting the corresponding effect of varying these parameters. The water-oil ratios that were investigated are 20/80 and 50/50. Where 20/80 was for tight emulsion and 50/50 for weak emulsions.

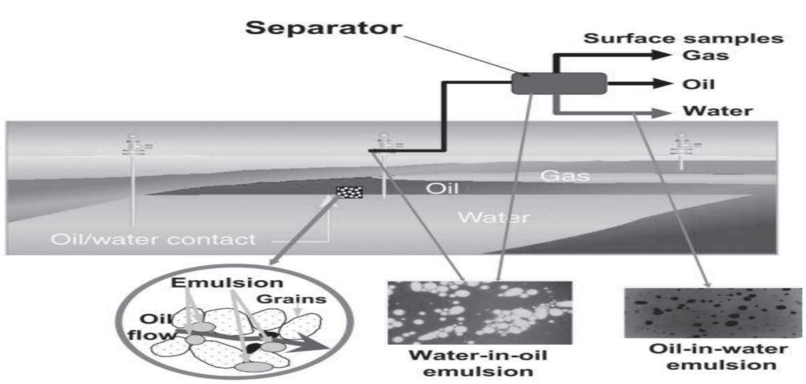

Figure 1.1: Phases of emulsion formation (Kokal & Al-Dokhi, 2008)

CHAPTER 2

LITERATURE REVIEW

2.1. Defining Emulsions

Emulsions are described as a type of colloidal dispersion where at least one immiscible fluid is present and dispersed in the other in the form of small or tiny droplets. These droplets are measured in micro meters (µm) and the size of the droplets range from 0.1 µm for tight emulsions to 100 µm for loose emulsions. Emulsions are thermodynamically unstable and also exhibit kinetic stability as it becomes stable overtime. Emulsions have two phases, the dispersed or internal phase and the continuous or external phase. The dispersed phase is the phase in droplets while the continuous phase is the phase that suspends the droplets. Emulsions are majorly of two types which includes water-in-oil and oil-in-water emulsions.

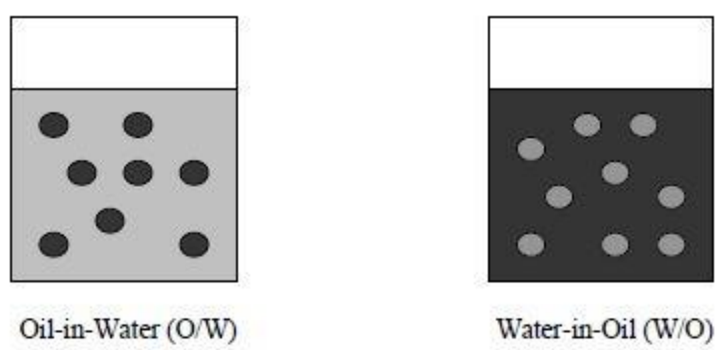

Figure 2.1: Schematics showing two types of emulsion (Hanapi et.al, 2006)

Describing these types in terms of phases, water-in-oil emulsions have water as its dispersed phase and oil as the continuous phase. On the other hand, oil-in-water emulsions have oil as its dispersed phase and water as the continuous phase (Emuchay et al., 2013; Fingas & Fieldhouse, 2008; Hanapi et al., 2006).

2.2. Forming Emulsions

Emulsions form in the presence of oil, water, emulsifiers and agitation or mix. Thus, in the presence of the above mentioned, there is bound to be emulsion formation. The emulsifiers create a film or layer which encapsulates the dispersed phase. This film inhibits the coalescence of the dispersed phase thus keeps the emulsion stable. This film usually has viscoelastic properties which influences its stability. For instance, the more elastic the film, the more stable the emulsion as the elasticity is able to shield the dispersed phase from external interferences.

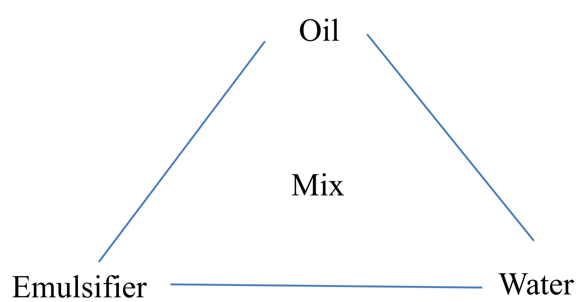

Figure 2.2: Emulsion Triangle

In the oil and gas industry, these conditions exist in almost every phase, from the reservoir to surface facilities. Oil is produced alongside with water as they both coexist in the reservoir, also in the course of production, there is a mixture of these fluids and oil and water being immiscible, with natural emulsifiers such as asphaltenes, waxes and resins present in oil, the formation of emulsion is almost inevitable. Asphaltene as the primary emulsifier, with the help of resins which keep the asphaltene in solution where it functions best, form rigid and elastic films which encloses the dispersed phase (Dosunmu et al., 2012; Gallup et al., 2010; Fingas & Fieldhouse, 2008, 2011; Kokal & Al-Dokhi, 2008).

2.3. Resolving Emulsions

In resolving of emulsions, physical, chemical or a combination of both methods are used. Physical method includes the use of agitation (use of centrifugal or enhanced gravity), settling time, electricity and heat. On the other hand, chemical method involves the use of demulsifiers to break emulsions.

2.3.1. Chemical Resolution of Emulsions

Chemical method involves the use of chemicals called demulsifiers. These are surfactants that attacks the film/layer that encloses the dispersed phase, thereby breaking the film and allowing for coalescence. Essentially, demulsifiers counterbalances the effect of emulsifiers and breaks the emulsion. Demulsifiers are generally classified into water soluble or oil soluble. As the name implies, water soluble demulsifiers dissolve in water while oil soluble dissolves in oil. The use of oil soluble demulsifier is common in resolving water-in-oil emulsions because oil is the continuous phase, thus the demulsifier is able to dissolve in the oil thereby attacking the emulsion globules. Alternatively, water soluble demulsifiers are sometimes preferred because the demulsifier dissolves in the water and is excreted alongside with it, thus avoiding unnecessary contamination of the oil (Jones et. al, 1978; Hanapi et al., 2006; Gallup et al., 2010).

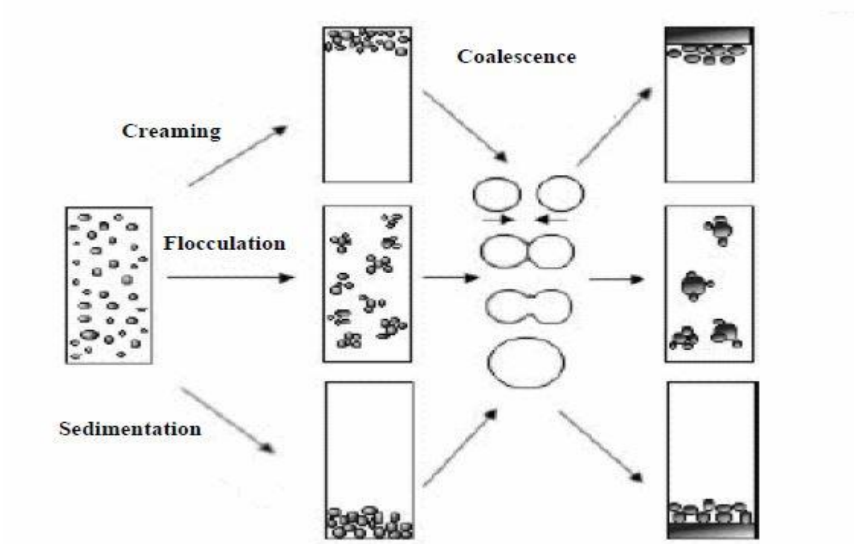

Figure 2.3: Process of emulsion breakdown (Auflem, 2002)

The figure 2.3 shown above, describes the process of emulsion breakdown. Sedimentation is when the heavier liquid settles down due to the density differences. Creaming occurs as a direct opposite of sedimentation, forming a concentration gradient of droplets. Coalescence is when the droplets join together to from a bigger droplet or unit, thus reducing the total surface area.

Demulsifiers usually have the hydrophilic and hydrophobic parts. The hydrophilic part attaches itself to the water while the hydrophilic part joins with the oil. Consequently, it activates the breaking of the film that encapsulates the dispersed phase by reducing the interfacial tension gradient and also interfacial shear viscosity thus, enhancing the breaking of the film.

Demulsifier resolves emulsions by flocculation and coalescence. Flocculation is the process by which the globules of the dispersed phase joins together. If viewed in a high resolution microscope, it resembles a bunch of fish eggs. After Flocculation, coalescence follows. In this stage, the actual breaking of the emulsifier film occurs, the dispersed phase unites and settles out. This process is shown in figure 2.4 below (Soffian & Niven, 1993, Hanapi et al., 2006).

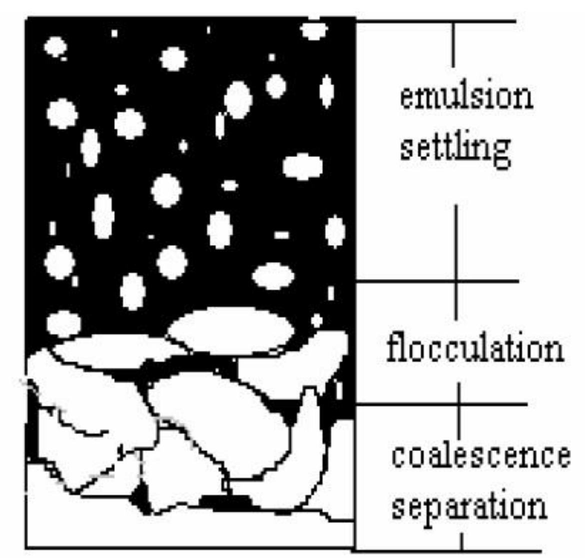

Figure 2.4: Flocculation and coalescence process (Hanapi et al., 2006)

2.3.2. Heat Resolution of Emulsions

Heat destabilizes emulsion by increasing the entropy of the system. Due to the increase in heat, the system becomes more disordered and this affects the interfacial film that was formed by the emulsifier, this effect causes the emulsion to be resolved. Although heat helps break emulsions significant increase in heat leads to loss of light components in the crude oil. Also, some emulsions get more stable as temperature significantly increases (Bansbach, 1965; Martínez-Palou et al., 2013).

Heat from microwave can also be applied in resolving emulsions. Microwave heating uses microwave radiation to perform bulk heating of emulsions. Microwave heating resolves oil-in-water emulsions by acting in two ways. Firstly, it increases temperature which in turn decreases viscosity of emulsion. By so doing, it causes the film encapsulating the dispersed phase to be disturbed and eventually ruptures. After the rupturing, coalescence begins. From Stoke's law, for water-in-oil emulsions where oil is the continuous phase, the settling velocity of water droplets through oil is given by the equation below:

$$V_w = \frac{(\rho_w - \rho_o) * g * D^2}{18 * \mu_o} \quad \text{.................................} \quad (2.1)$$

where:

V_w = Settling velocity of water

ρ_w = Density of emulsified water

ρ_o = Density of oil

g = Gravitational constant

D = Droplet diameter

μ_o = Viscosity of continuous phase (oil)

Equation 2.1, shows that the settling velocity is directly proportional to the density difference, gravity and square of droplet diameter, and inversely proportional to the viscosity of the continuous phase, in this case being oil. By heating, viscosity decreases rapidly in comparison to the density difference, this leads to the separation of the dispersed phase (Abdurahaman & Rosli, 2006).

Secondly, it causes the coagulation of the dispersed droplets. Due to increase in temperature and decrease in viscosity, this enhances coagulation of the water (dispersed) phase. This coagulation results in larger diameter particles, this increase in diameter particles leads to rapid separation. This is proven by Stokes law (equation 1) where the settling velocity is directly proportional to the square of the droplet diameter (Abdurahaman & Rosli, 2006; Anisa et al., 2011).

Microwave energy causes the bulk and direct heating of the dispersed (water) phase through interaction with the molecules caused by the electromagnetic field the microwave creates. Also, microwave rearranges the electrical charge distribution of water molecules as it rotates, therefore moving the ions around the droplet. Water being dipolar, allows selective heating by these microwaves. Due to this, the problem of lighter components in oil evaporating because of excessive heating is reduced significantly as the heat affects only the dipolar water molecules (Vega et al., 2002; Binner et al., 2013; Martínez-Palou et al., 2013)

CHAPTER 3

METHODOLOGY

3.1. Introduction

Persistent with the highlighted objectives of this study, this research adopts an experimental research design. To meet these objectives, a number of materials were used. Also, experimental and analytic procedures were adopted from the reviewed literature. These materials and procedures are discussed in details in the subsequent sections of this chapter.

3.2. Materials and Equipment

All the materials (except for the demulsifier which was obtained from Akzo nobel) used in the course of this research were obtained from the core analysis laboratory of the Petroleum Engineering department of Universiti Teknologi PETRONAS. The list of materials and equipment used is given below.

Equipment:

- 700 watt Elba domestic microwave oven
- Constant speed mixer
- Digital scale
- Stop watch
- Magnetic stirrer
- 10 ml graduated measuring cylinder
- 100 ml graduated measuring cylinder
- 900 ml graduated beaker

Materials:

- TBCO crude oil sample
- Distilled water
- Sodium Chloride (NaCl)
- Demulsifier

3.3. Experimental Procedure

This section discusses the procedures adopted to carry out the experiment. The method of preparing emulsion as well as the demulsification process is discussed.

3.3.1 Emulsion Preparation

In preparing the synthetic emulsion, synthetic brine was used together with the TBCO crude oil sample. The synthetic brine was made by dissolving sodium chloride (NaCl) in distilled water. The salinity equation, equation 3.1 is shown below (Hanapi et al., 2006).

$$Y = 8.3566X - 0.3582 \qquad (3.1)$$

where Y = Salinity (%w/w); % per thousand

X = NaCl concentration (g/100ml)

Adopting salinity of 9.4% as used by, the equation becomes;

$$9.4 = 8.3566X - 0.3582, \text{ this yields } X = 1.1672 \text{ g.}$$

Therefore, 1.1672g of Sodium chloride was dissolved in 100 ml of distilled water to form a synthetic brine of 9.4% salinity. This percentage salinity was adopted from the Tabu oilfield in Malaysia.

After the synthetic brine was made, the synthetic emulsion was made by mixing the brine with the TBCO crude oil in its appropriate proportions using a constant speed mixer at 1800 rpm for 5 minutes.

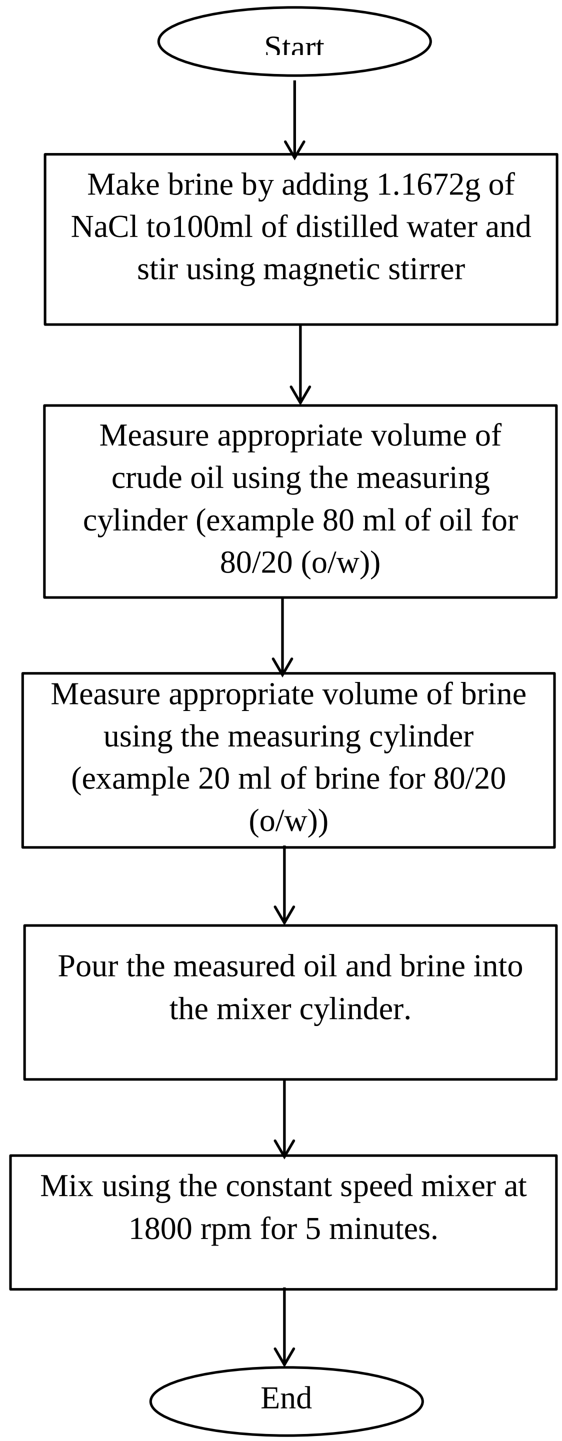

Figure 3.1: Process flowchart for preparing emulsion

3.3.2 Selection of water-oil ratio

To select the appropriate proportion of water and oil to be used, a stability test was done using three different water-oil-ratios. Water-oil-ratios of 80/20 (oil/water), 60/40 (oil/water) and 50/50 (oil/water) were used. Emulsions corresponding to these ratios were prepared and left at room temperature for a week, after which no water was separated from the 80/20 (oil/water) and 60/40 (oil/water) emulsions. For the 50/50 (oil/water) emulsion, 40% water was separated. Thus, two water –oil-ratios were selected to conduct further experiments in this research. The 80/20 (oil/water) was selected to serve as a strong or tight emulsion while the 50/50 (oil/water) was chose to act as a weak or loose emulsion.

3.3.3 Demulsification procedure

In carrying out the actual demulsification process, three conditions were experimented. The first condition involved the use of microwave heating only, the second condition involved a combination of both microwave heating and the use of demulsifier, and the third condition was the use of demulsifier only. In each of the cases, after the corresponding treatment, the sample was observed over separation time of 60 minutes. The volume of water separated was recorded at five (5) minutes interval.

For the first condition, microwave heating only was used. The emulsion sample was put in 10 ml graduated cylinder, covered the top of the cylinder with aluminum foil, the power level of the microwave oven was set, then the emulsion sample was put in the microwave oven for an exposure time of 30, 60, 90 and 120 seconds. For this, four (4) levels of microwave power were used. Low power level corresponds to 17% of the total 700 Watt which gives 119 Watt. Medium low power level corresponds to 48% of the total 700 Watt which gives 336 Watt. Medium power level corresponds to 66% of the total 700 Watt which gives 462 Watt. High power level corresponds to 100% of the total 700 Watt which gives 700 Watt. For each power level, four different samples were used for the four different exposure time of 30, 60, 90 and 120 seconds. After which, it was observed over separation time of 60 minutes where readings of volume of water separated were recorded at five (5) minutes interval.

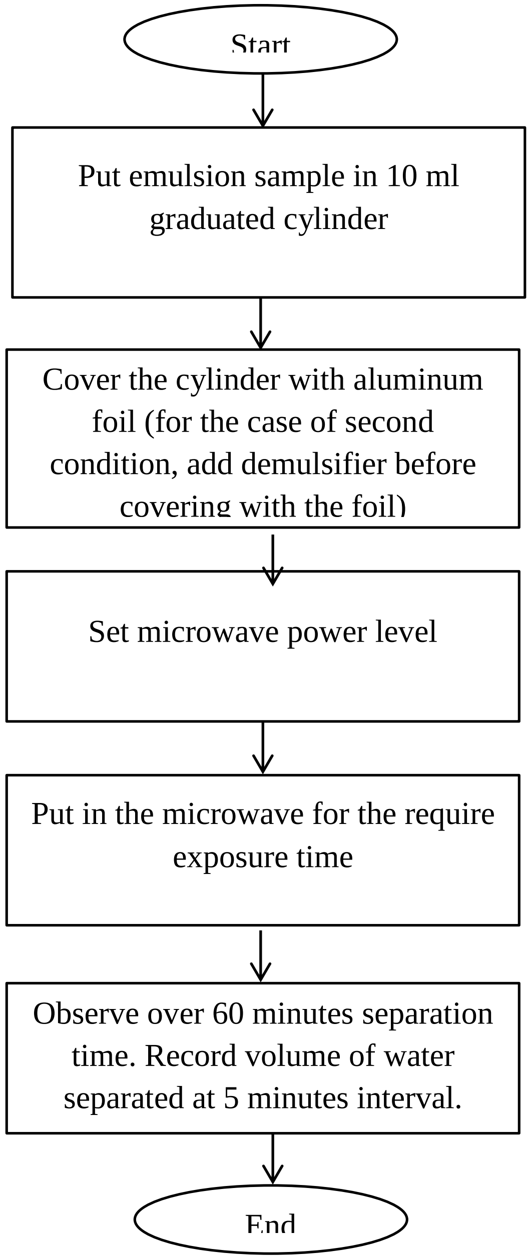

Figure 3.2: Process flowchart for demulsification procedure

For the second condition, the combined effect of microwave heating and use of demulsifier was experimented. In this case, after the emulsion sample was poured into the 10 ml graduated cylinder, 0.5 ml of non-ionic resin oxyalkylate demulsifier was added then aluminum foil was used to cover it before placing it in the microwave oven for different power levels and different exposure times as described for the first condition above.

In the case of the third condition, the effect of demulsifier use only was tested. Here, the prepared emulsion sample was placed in a 10 ml graduated measuring cylinder, 0.5 ml of non-ionic resin oxyalkylate demulsifier was added. After which it was placed at a constant temperature in the oven at 40°C and it was observed over separation time of 60 minutes where readings of volume of water separated were recorded at five minutes interval.

Figure 3.3: Figure showing experimental setup

3.4. Project activities

The major project activities and the flow is as shown below:

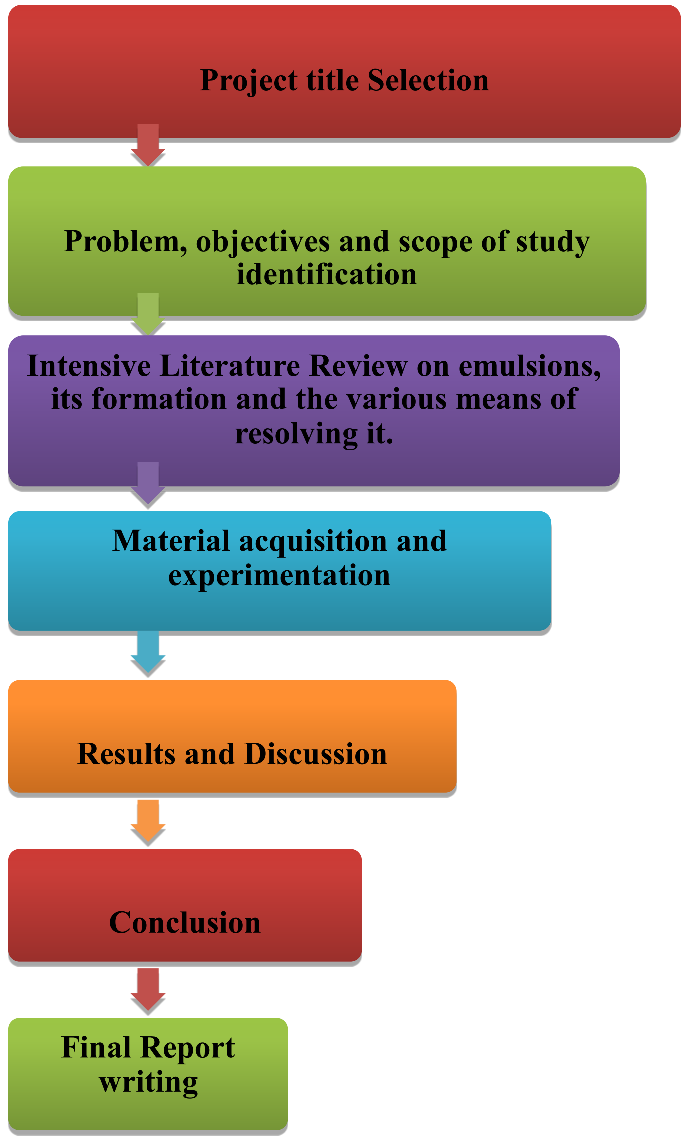

Figure 3.4: Project Activities involved

3.5. Gant Chart

Weeks / Activities	2	4	6	8	10	12	14	16	18	20	22	24	26	28	30	32
Problem Identification	■	■														
Intensive Literature review			■	■	■											
Developing method to solve the problem					■	■	■									
Material acquisition and Experimentation								■	■	■	■					
Results, findings and discussion											■	■	■			
Conclusions														■	■	
Report writing															■	■

CHAPTER 4

RESULT AND DISCUSSION

4.1. Introduction

This chapter reports the result obtained from the experiments. The results are presented in two parts. The first part is the results obtained for the tight emulsion, that is, the 80/20 (oil/water) emulsion. The second part is the results obtained for the weak emulsion, that is, the 50/50 (oil/water) emulsion.

4.2. Results for Tight Emulsion

This section is divided into three subsections. Each subsection discusses a demulsification condition as described in the methodology. The conditions are; microwave heating only, microwave heating with demulsifier and application of demulsifier only. The volume of separated water is calculated in percentage using the equation (4.1) (Abdurahman et al., 2006).

$$\text{Separated water (\%)} = \frac{recovered\ water\ volume\ (ml)}{Original\ water\ volume\ (ml)} * 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots (4.1)$$

4.2.1. Microwave heating only

In this subsection, the effect of using microwave heat only in resolving tight emulsions is observed. Table 4.1 shows that for low microwave power (119 watt), no water was recovered for the different exposure times of 30, 60, 90 and 120 seconds. This indicates that the heat provided by the microwave at this level is not sufficient to break the emulsion.

Table 4.1: Separated water (%) in separation time (mins) for low microwave power

	Separated water (%) in Separation time (mins)												
Te \ Tsep	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
60 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
90 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
120 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

Table 4.2 indicates that for medium low microwave power (336 watt), its result follows the same pattern for the low microwave power level. No water was recovered for the different exposure times of 30, 60, 90 and 120 seconds. From this, it can be said that the energy provided by the microwave is not enough to break the emulsion.

Table 4.2: Separated water (%) and separation time (mins) at a power of 336W

	Separated water (%) in Separation time (mins)												
Te \ Tsep	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
60 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
90 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
120 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

Figure 4.1 shows that for medium microwave power (462 watt), no water was recovered except for the exposure time of 120 seconds. At this microwave power level, given that watt is joules/second, this indicates that the 55.4 kJ was required to break the emulsion.

At this point only 20% of the water was separated. This percentage recovery is very low as higher percentage recovery is required in the industry.

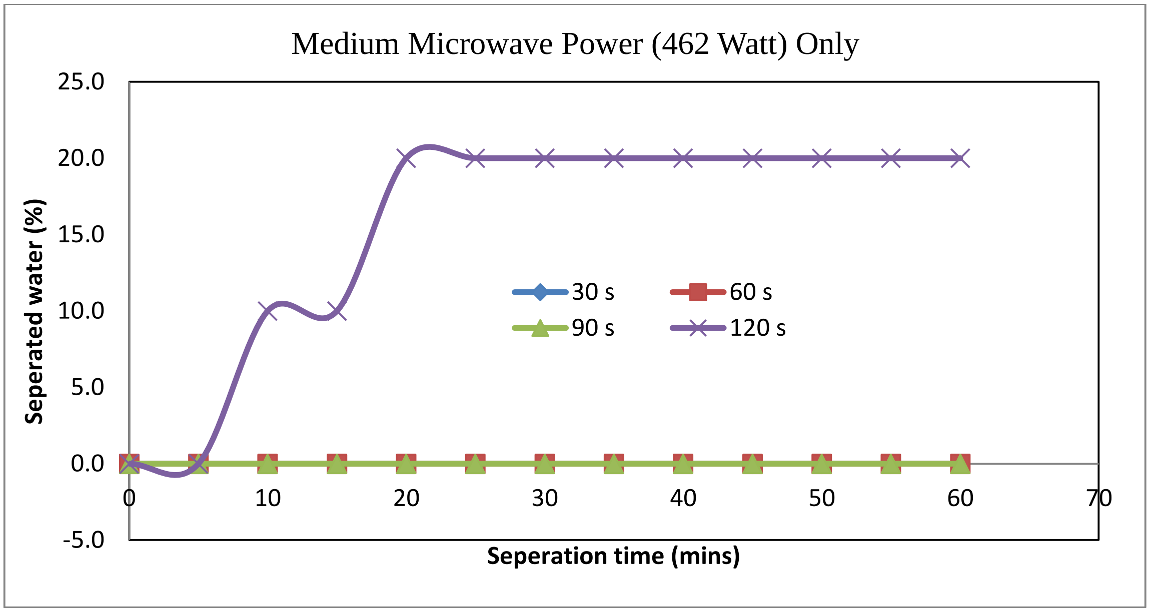

Figure 4.1: Separated water (%) vs separation time (mins) at a power of 462W

Table 4.3 elaborates that for high microwave power (700 watt), no water was recovered for the different exposure times. This result seems to be abnormal as water is expected to have separated because the medium power level of 462 watt had 20% separated at 120 seconds. This abnormality could be due to the extremely high energy that the microwave heating provided to the emulsion sample. Excessive heat can sometimes stabilize the emulsion instead of breaking it.

Table 4.3: Separated water (%) in separation time (mins) for high microwave power

	Separated water (%) in Separation time (mins)												
Te \ Tsep	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
60 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
90 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
120 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

4.2.2 Microwave heating with demulsifier

In this subsection, the effect of using both microwave heat and demulsifier is seen.

In the Figure 4.2 below, it is observed that a combination of low microwave power (119 watt) and 0.5 ml of demulsifier yields some amount of water. The highest being for the 90 seconds exposure time which yields 50% separated water at separation time of 5 minutes. This indicates that 10.7 kJ of heat was used together with the demulsifier effect to yield this amount of separated water. The 30 seconds exposure yields no separated water at all. This could be because of insufficient heat to break the emulsion. The 120 seconds exposure time yields only 5% of separated water as compared to 90 seconds exposure time which yielded 50%. The heat provided at 120 seconds could have been too much for the emulsion sample, thus giving a possibility of re-stabilizing the emulsion. For this scenario, it is seen that 90 seconds is optimal as it yields a considerable 50% water recovery.

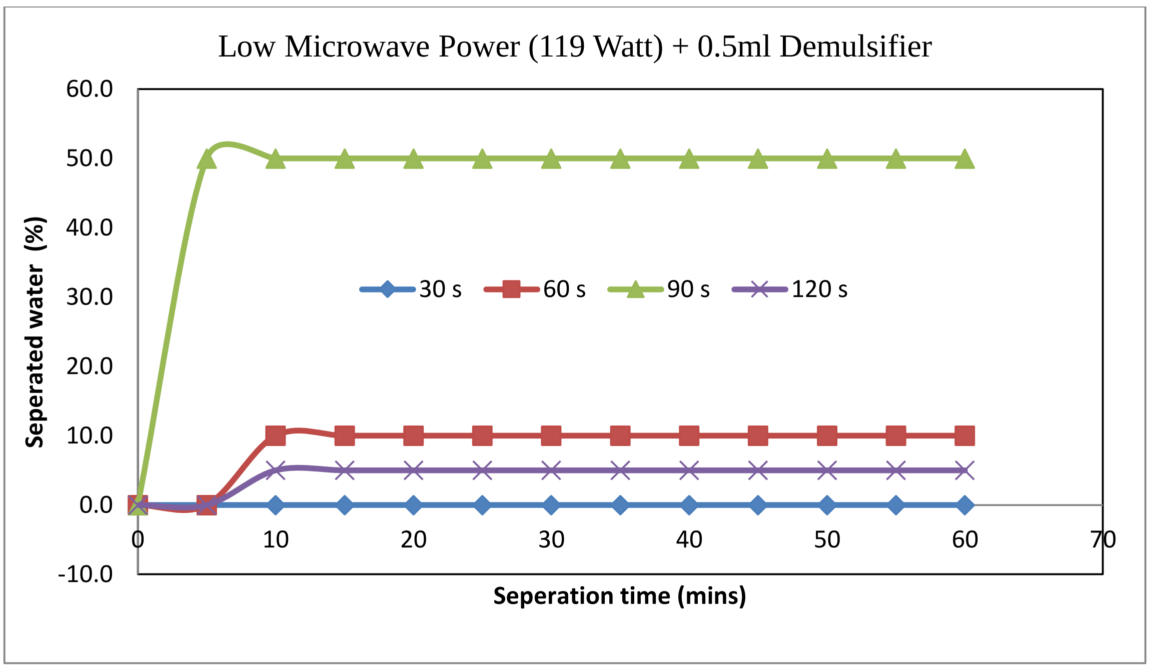

Figure 4.2: Graph of separated water (%) vs separation time (mins) for low microwave power (119 watt) + 0.5 ml demulsifier

Figure 4.3 shows a combination of medium low microwave power (336 watt) and 0.5 ml of demulsifier separates water. The highest separated water for this case goes to the 60 and 90 seconds exposure time which yields 70% separated water at separation time of 25 and 35 minutes respectively. Here, 20. 2 kJ and 30.2 kJ of heat were used to achieve this feat. It can also be observed that in relation to low microwave power heating, a jump from 50% to 70% of separated water is seen.

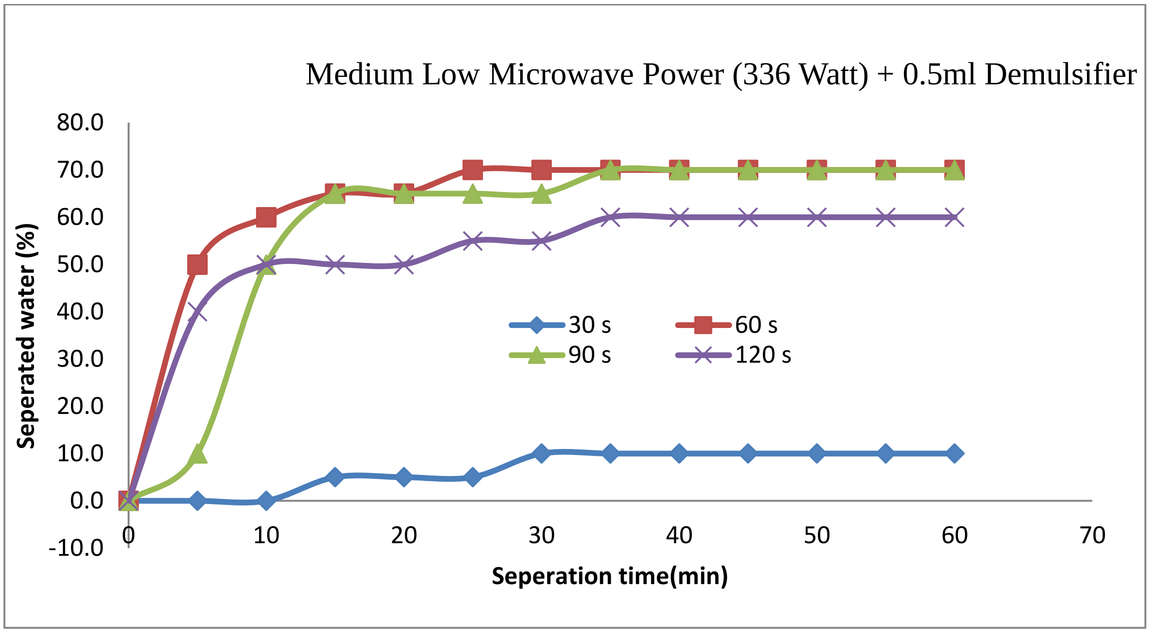

Figure 4.3: Graph of separated water (%) vs separation time (mins) for medium low microwave power (336 watt) + 0.5 ml demulsifier

In the Figure 4.4 below, it is seen that a combination of medium microwave power (462 watt) and 0.5 ml of demulsifier separates water. The highest separated water is 70% observed at separation time of 35 minutes for the sample exposed for 120 seconds. The exposure time of 90 seconds follows closely with 60% separated water at 30minutes separation time. The 30 and 60 seconds exposure time have a tie of separated water of 50% at separation time of 35minutes. Relating this to the previous results, it can be said that the separated water has climaxed at 70% as there is no difference in the highest amount of water separated between medium microwave power and medium low microwave power.

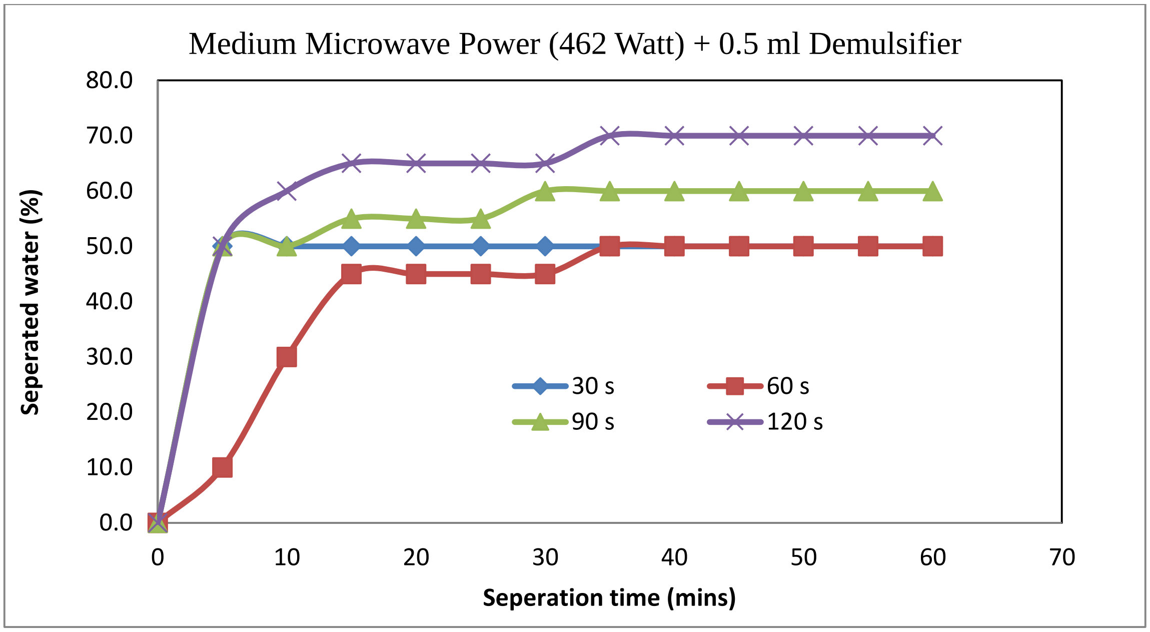

Figure 4.4: Graph of separated water (%) vs separation time (mins) for medium microwave power (462 watt) + 0.5 ml demulsifier

Figure 4.5 points out a combination of high microwave power (700 watt) and 0.5 ml of demulsifier separates water. The highest separated water seen here is 50% observed at separation time of 15 minutes for the sample exposed for 60 and 120 seconds. The 90 seconds exposed sample reaches 50% separated water at 30 minutes. In general, it is observed that the highest amount of separated of water has declined in the case of the high microwave power. The peak of 70% separated water is achieved with medium low and medium microwave power. This further buttresses the point that, the separated water has climaxed at 70% for the scenario where medium low and medium microwave power is used. For this section, a combination of medium low microwave power (336 watt) and 0.5 ml of demulsifier is recommended.

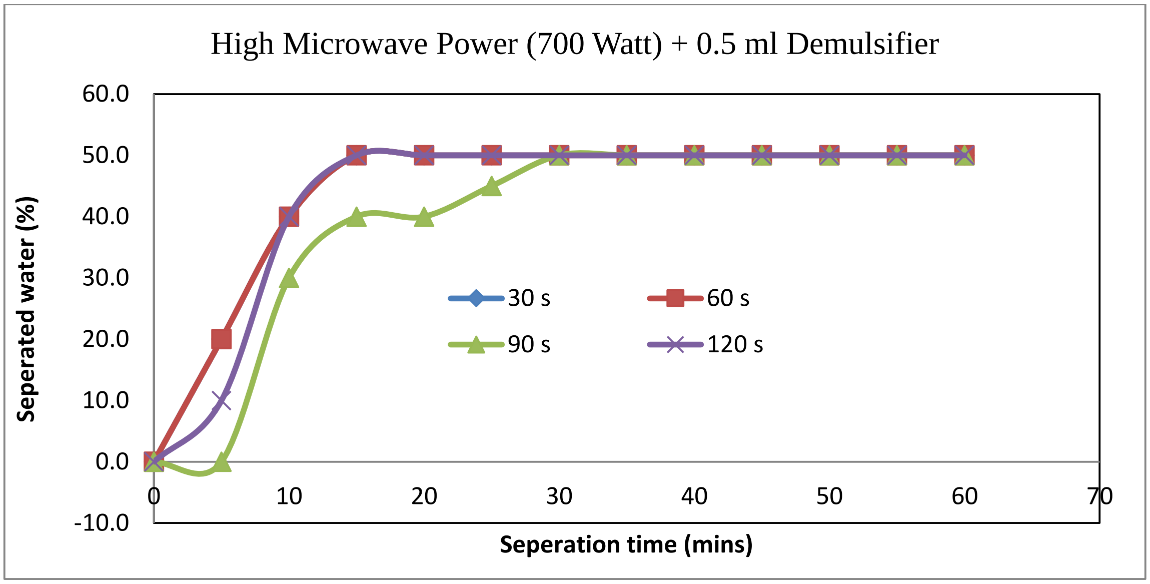

Figure 4.5: Graph of separated water (%) vs separation time (mins) for high microwave power (700 watt) + 0.5 ml demulsifier

4.2.2.1 Comparison Based on Exposure Time

In this subsection, the effect of exposure time is observed in resolving tight emulsions. The results obtained from the different power levels are grouped into the four (4) different exposure time of 30, 60, 90 and 120 seconds. The results are discussed subsequently.

From the figure 4.6 below, it is noticed that for 30 seconds exposure time, the highest water separated is 50% which is observed at the combination of medium microwave power and 0.5 ml demulsifier. This 50% yield is also seen at the combination of high microwave power and 0.5 ml demulsifier. 50% water separation is not desirable as opting for the highest possible water separated is always the case. Therefore, thirty seconds exposure is not sufficient to break the emulsion.

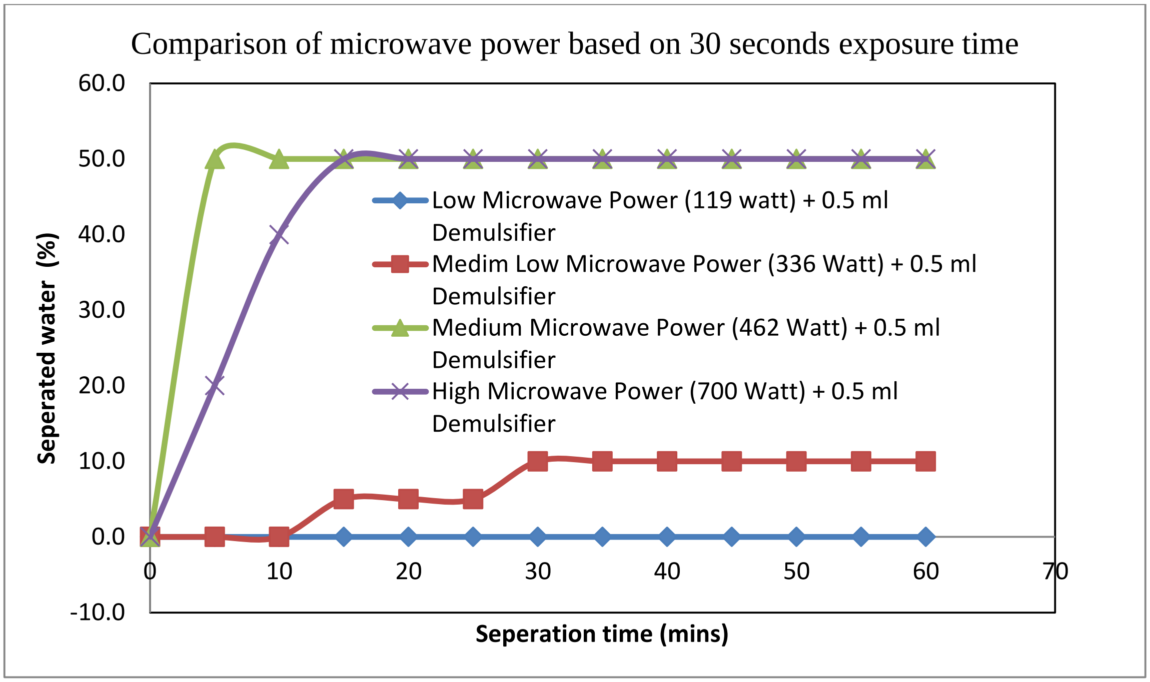

Figure 4.6: Graph of separated water (%) vs separation time (mins) for 30 seconds exposure time at different microwave power levels

Figure 4.7 below, it is noticed that for 60 seconds exposure time, the highest water separated is 70% which is noted at the combination of medium low microwave power and 0.5 ml demulsifier at separation time of 25 minutes. High microwave power and medium microwave power combined with 0.5 ml demulsifier follow with 50% separated water. At the highest yield of 70%, 20.16 kJ of heat is used to achieve this. This result appears optimal as not much energy is used and the amount of water separated is good.

In figure 4.8, for 90 seconds exposure time, the highest water separated is 70% which is noted at the combination of medium low microwave power and 0.5 ml demulsifier at separation time of 35 minutes. Medium microwave power combined with 0.5 ml demulsifier follows closely with 60% separated water. Low and high microwave power combined with 0.5 ml demulsifier converge at separation time of 30 minutes where it yields 50% separated water. In comparison with the 90 seconds exposure plot, medium low microwave still maintains the highest yield of 70% separated water. But the medium low power performs better at 60 seconds as it reaches peak separated water at 25 minutes separation time as compared to 35 minutes separation time of 90 seconds exposure time.

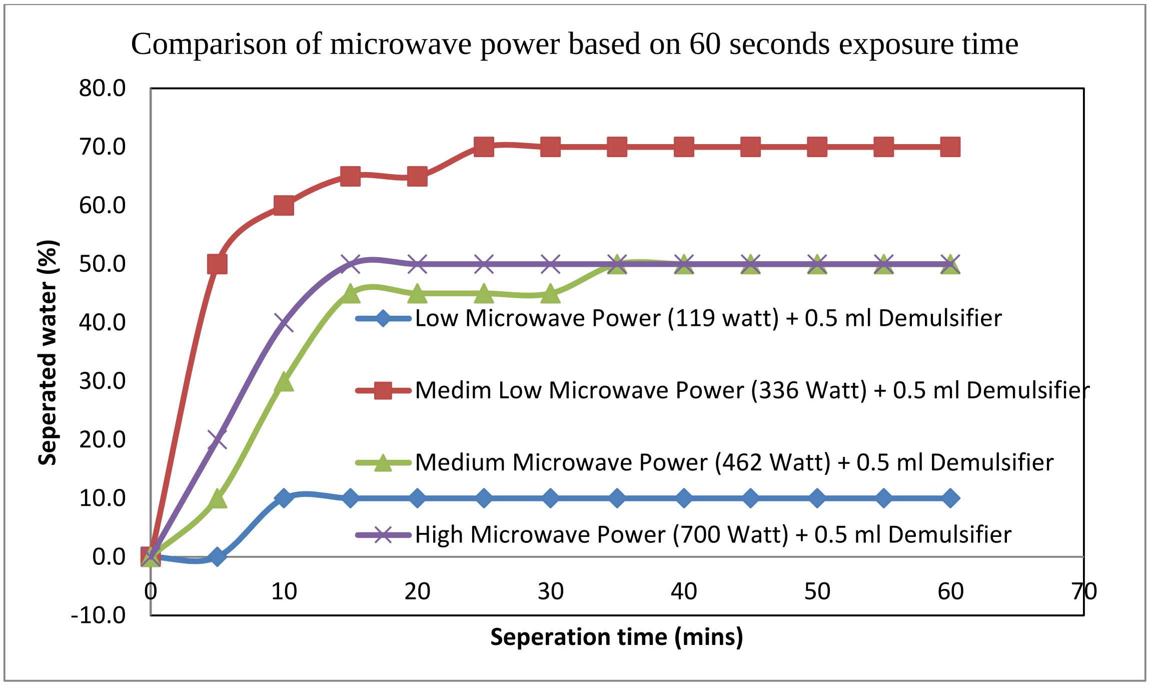

Figure 4.7: Graph of separated water (%) vs separation time (mins) for 60 seconds exposure time at different microwave power levels

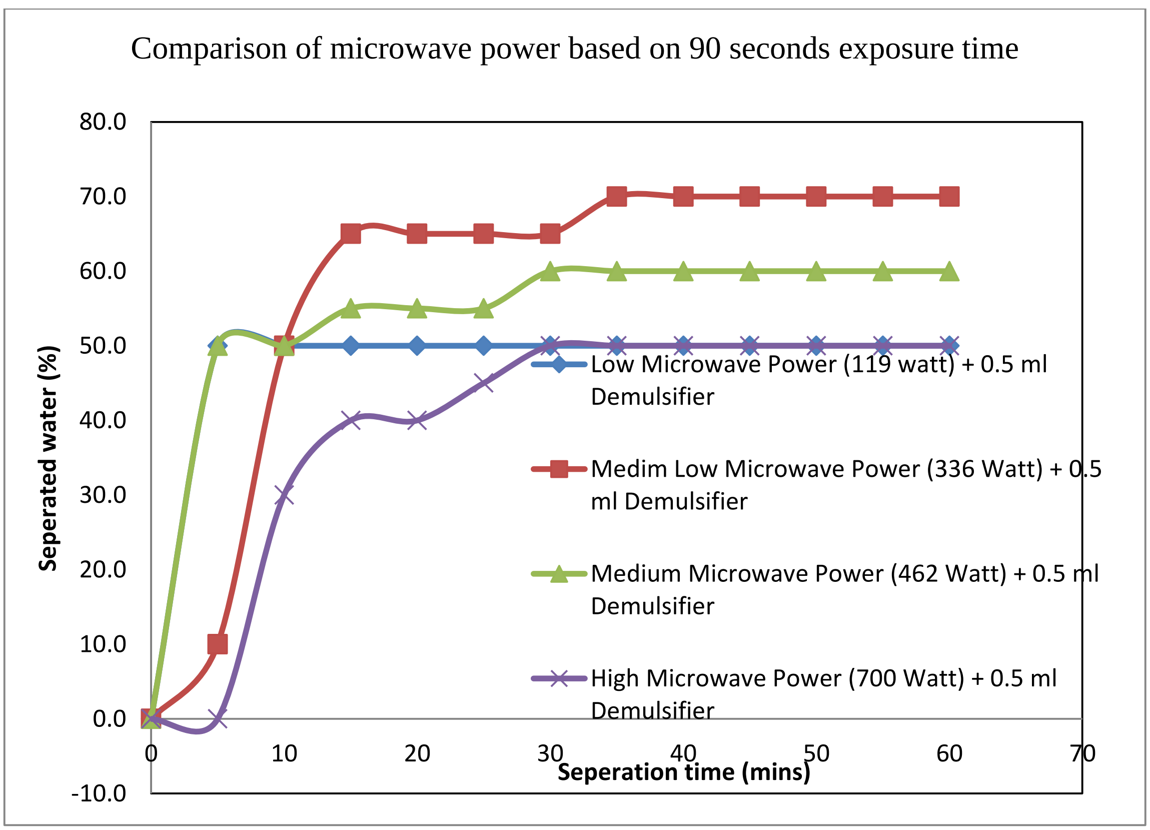

Figure 4.8: Graph of separated water (%) vs separation time (mins) for 90 seconds exposure time at different microwave power levels

Figure 4.9 shows that for 120 seconds exposure time, the highest water separated is 70%, observed at the combination of medium microwave power and 0.5 ml demulsifier. The lowest yield is 5% separate water, noticed at the combination of low microwave power and 0.5 ml demulsifier. In comparison with previous exposure time plots, this result is the least favorable as a high amount of heat (55.4 kJ) is required to give the highest yield of 70% separated water.

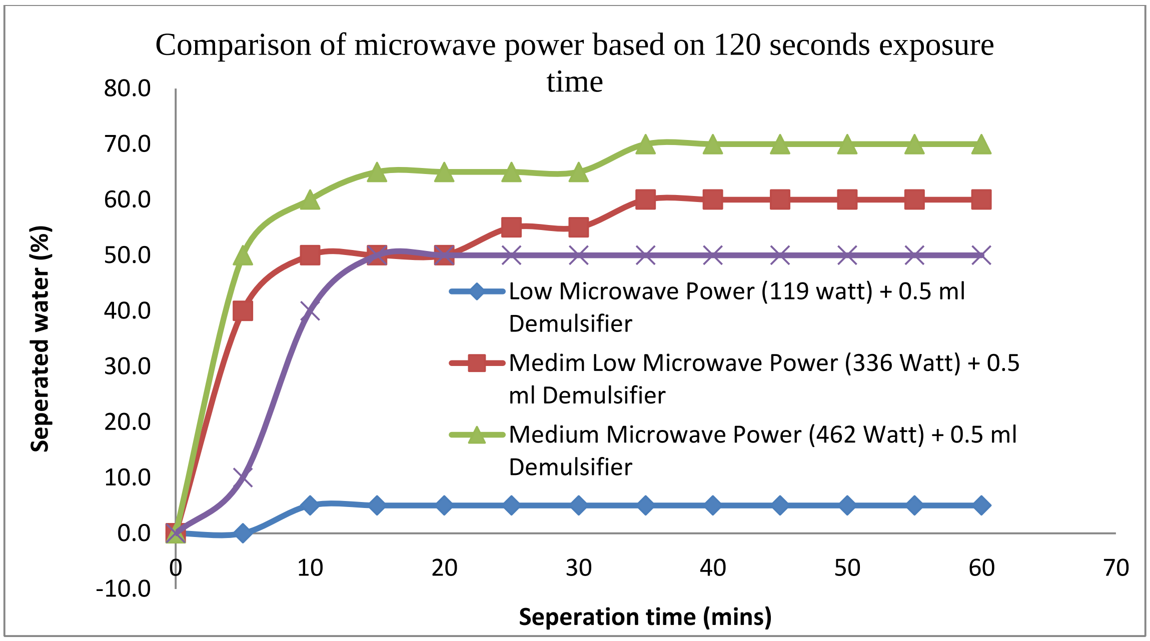

Figure 4.9: Graph of separated water (%) vs separation time (mins) for 120 seconds exposure time at different microwave power levels

4.2.3 Demulsifier only

In this subsection, the effect of using 0.5ml of neat demulsifier only is observed.

From the table 4.4 below, it is seen that for 0.5 ml demulsifier only, there is no water separated. This suggests that the use of demulsifier alone for this type of emulsion is not sufficient.

Table 4.4: Separated water (%) in separation time (mins) for 0.5 ml demulsifier only

T Sep (mins)	0	5	10	15	20	25	30	35	40	45	50	55	60
Separated water (%)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

4.3. Results for weak emulsion

Similar to section 4.2, this section is divided into three subsections. Each subsection discusses a demulsification condition as described in the methodology. The conditions are; microwave heating only, microwave heating with demulsifier and application of demulsifier only. The volume of separated water is calculated in percentage using the equation (3) (Abdurahman et al., 2006).

$$\text{Separated water (\%)} = \frac{recovered\ water\ volume\ (ml)}{Original\ water\ volume\ (ml)} * 100 \ \ldots\ldots\ldots\ldots\ (4.1)$$

4.3.1. Microwave heating only

In this subsection, the effect of using microwave heat only in resolving weak emulsion is observed. In figures 4.10, 4.11, 4.12 and 4.13 below, it is observed that for the four (4) different microwave power levels (low, medium low, medium and high), 100% water recovery is observed for the different exposure times. It is worthy to further note that this separation occurs rapidly, almost as soon as the emulsion is taken out of the microwave. It is observed that the average separation time is 8 minutes. That is, on average, by eight minutes separation time, 100% water recovery is observed. This is an indication that indeed 50:50 water-oil-ratio results in a weak or unstable emulsion thus the less energy required to break it.

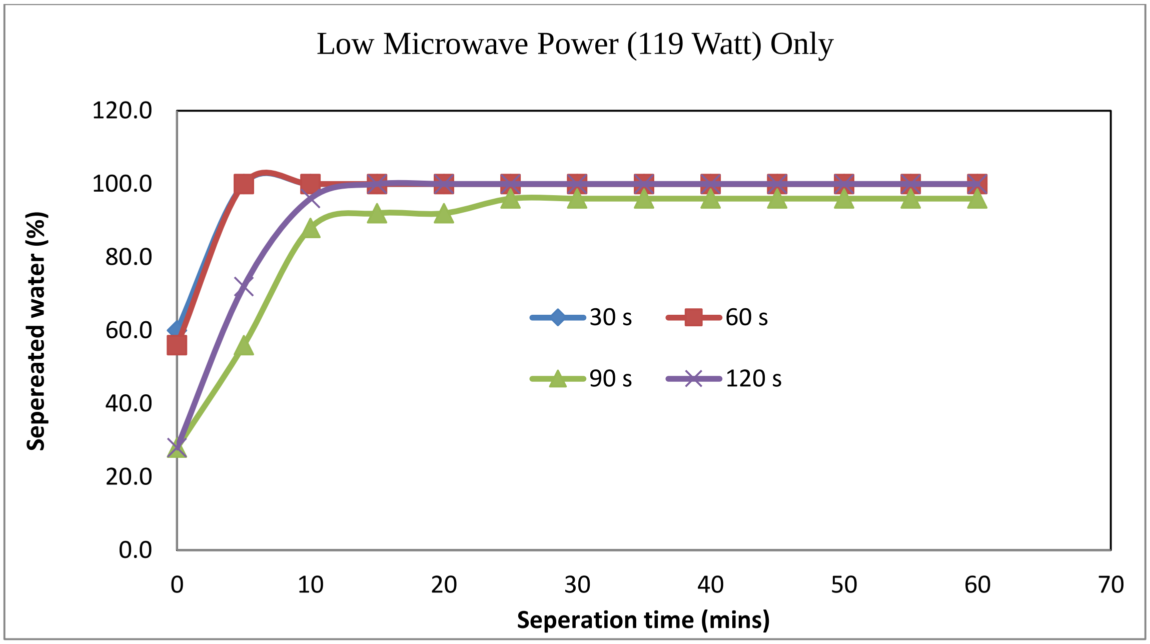

Figure 4.10: Graph of separated water (%) vs separation time (mins) for low microwave power (119 watt) only

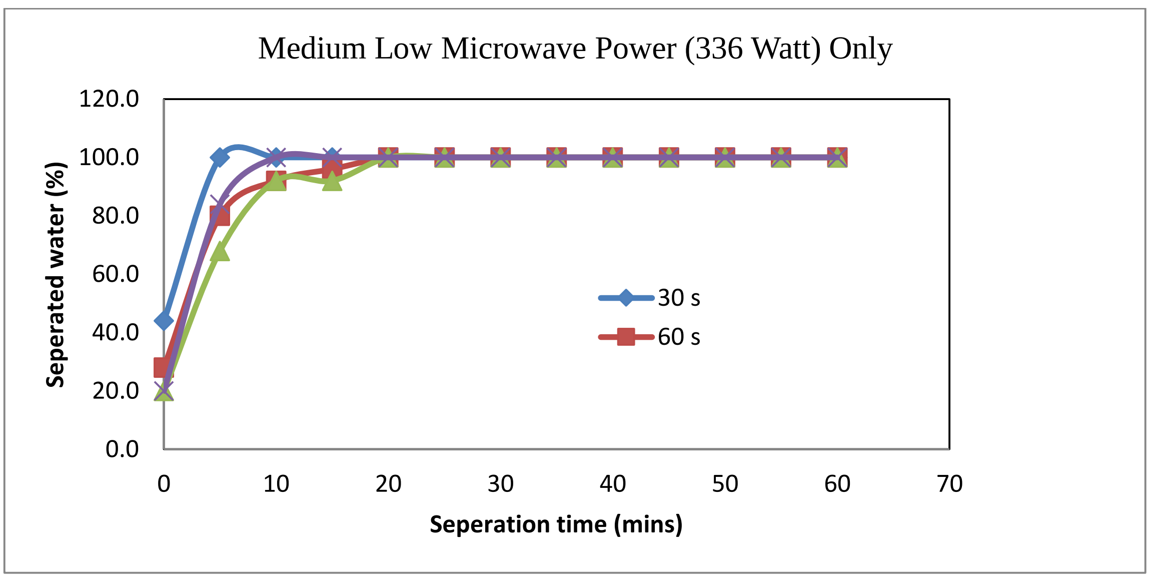

Figure 4.11: Graph of separated water (%) vs separation time (mins) for medium low microwave power (336 watt) only

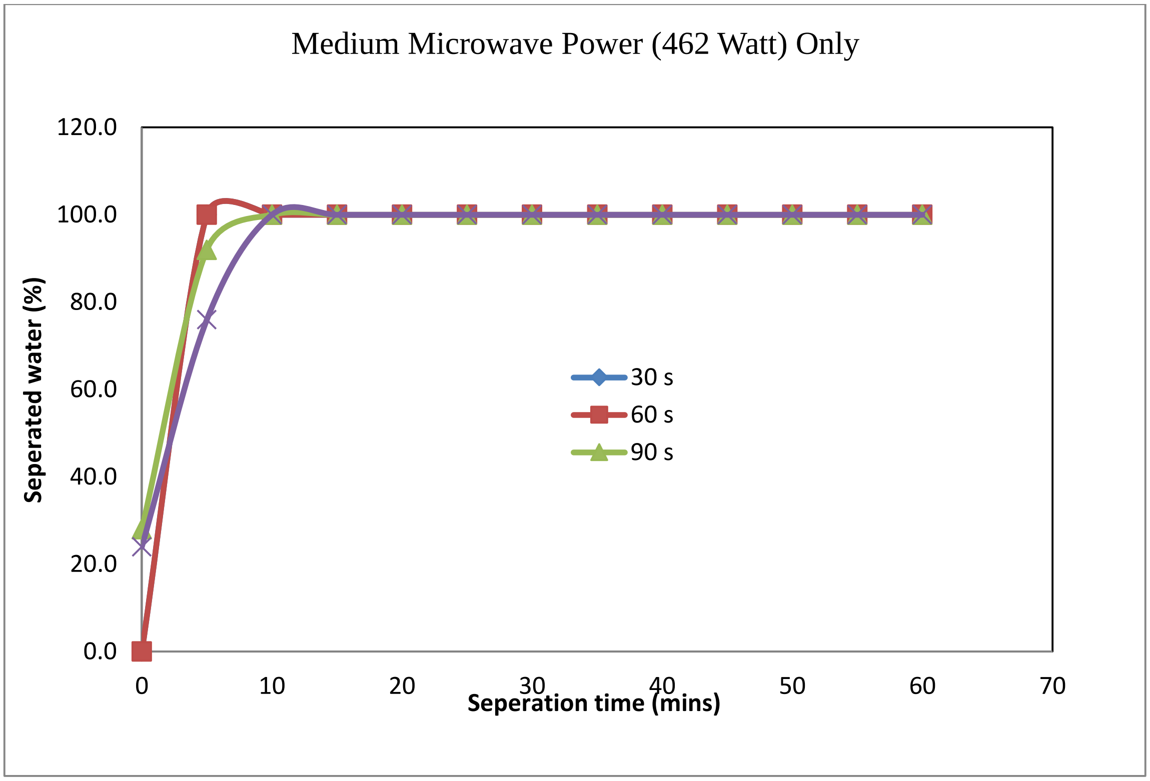

Figure 4.12: Graph of separated water (%) vs separation time (mins) for medium microwave power (462 watt) only

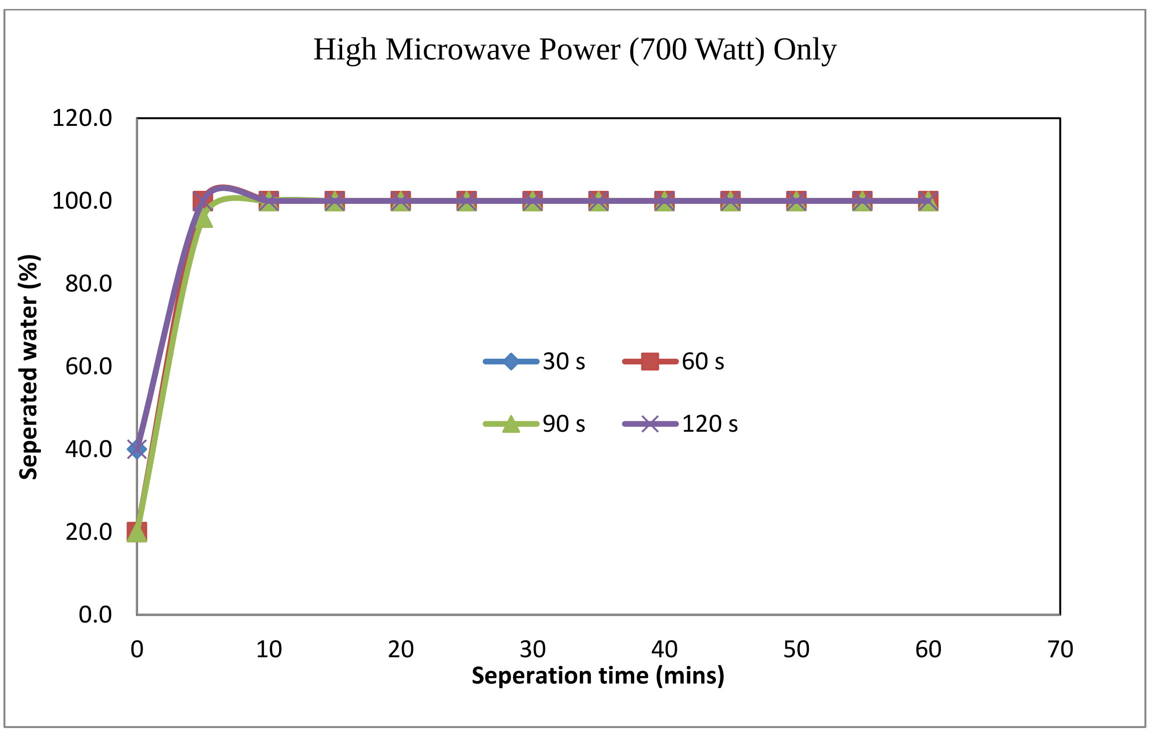

Figure 4.13: Graph of separated water (%) vs separation time (mins) for high microwave power (700 watt) only

4.3.2 Microwave heating with demulsifier

In this subsection, the combined effect of microwave heat and demulsifier on weak emulsion is investigated.

In the figures below; 4.14, 4.15, 4.16 and 4.17 corresponding to the four different microwave power levels of low, medium low, medium and high respectively, it is seen that 100 % water recovery is obtained in all cases. Although some cases are slightly more efficient such as under the case of low and high microwave power where 60 seconds exposure time yields 60% and 90% of water at zero separation time. That is, this water recovery is observed as soon as the emulsion is taken out of the microwave. These cases are the more efficient cases, other than that, for other power levels, a 100% water recovery is observed over the course of the separation time. This further buttresses the point that 50:50 water-oil ratio results in a loose or unstable emulsion. As already observed from section 4.3.1, there is no need to add demulsifier as the use of microwave only suffice to break the weak emulsion.

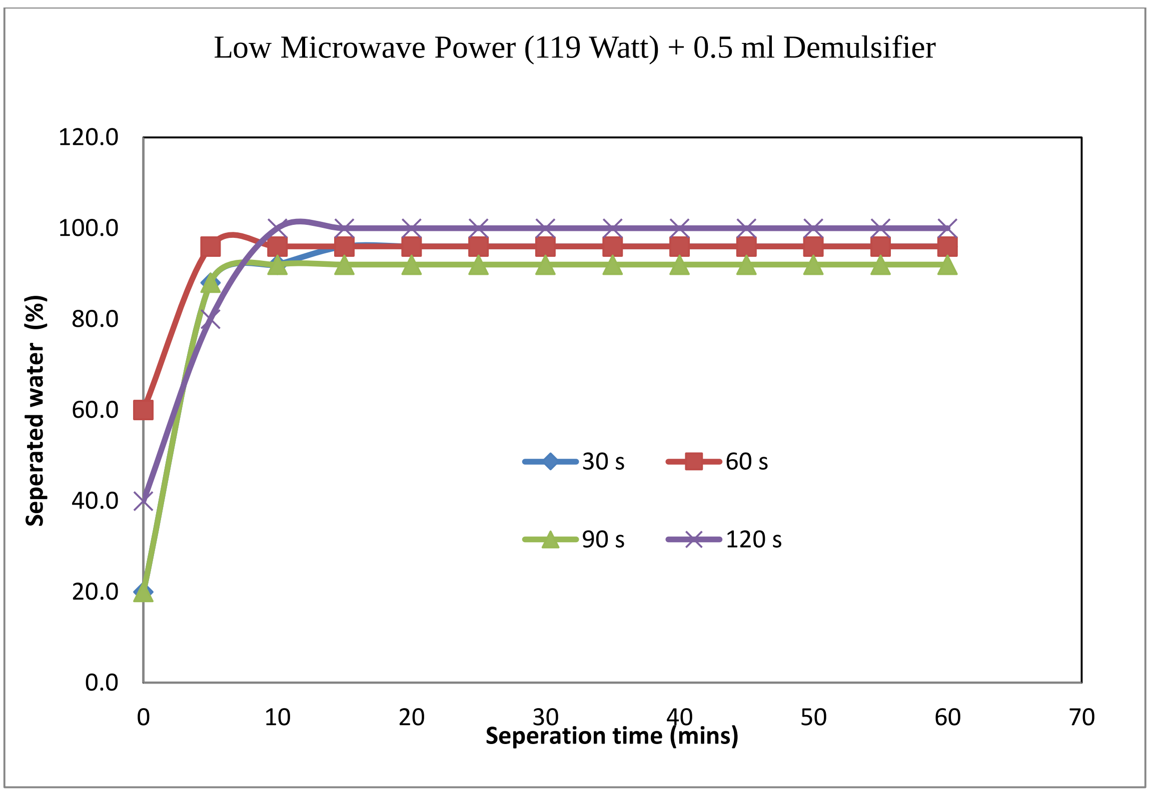

Figure 4.14: Graph of separated water (%) vs separation time (mins) for low microwave power (119 watt) + 0.5 ml demulsifier

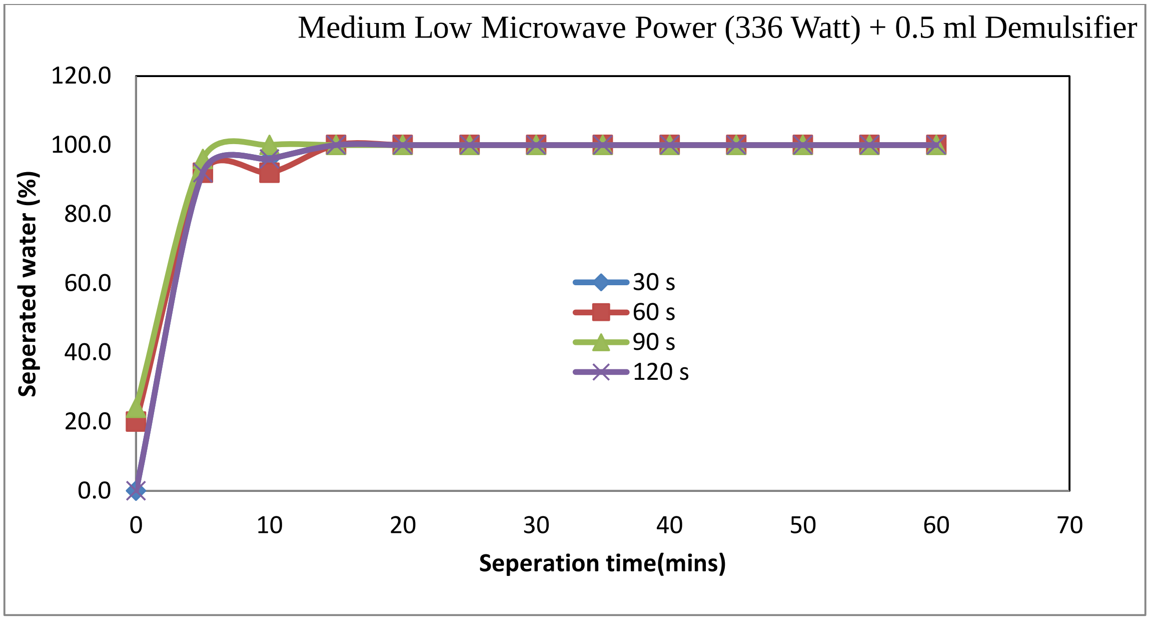

Figure 4.15: Graph of separated water (%) vs separation time (mins) for medium low microwave power (336 watt) + 0.5 ml demulsifier

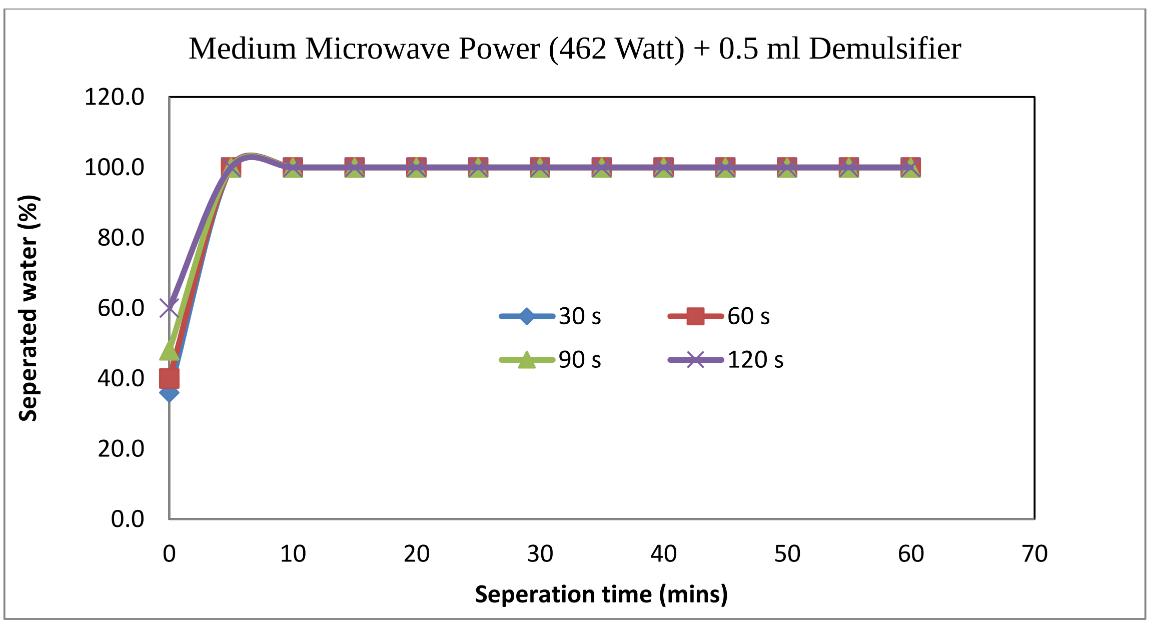

Figure 4.16: Graph of separated water (%) vs separation time (mins) for medium microwave power (462 watt) + 0.5 ml demulsifier

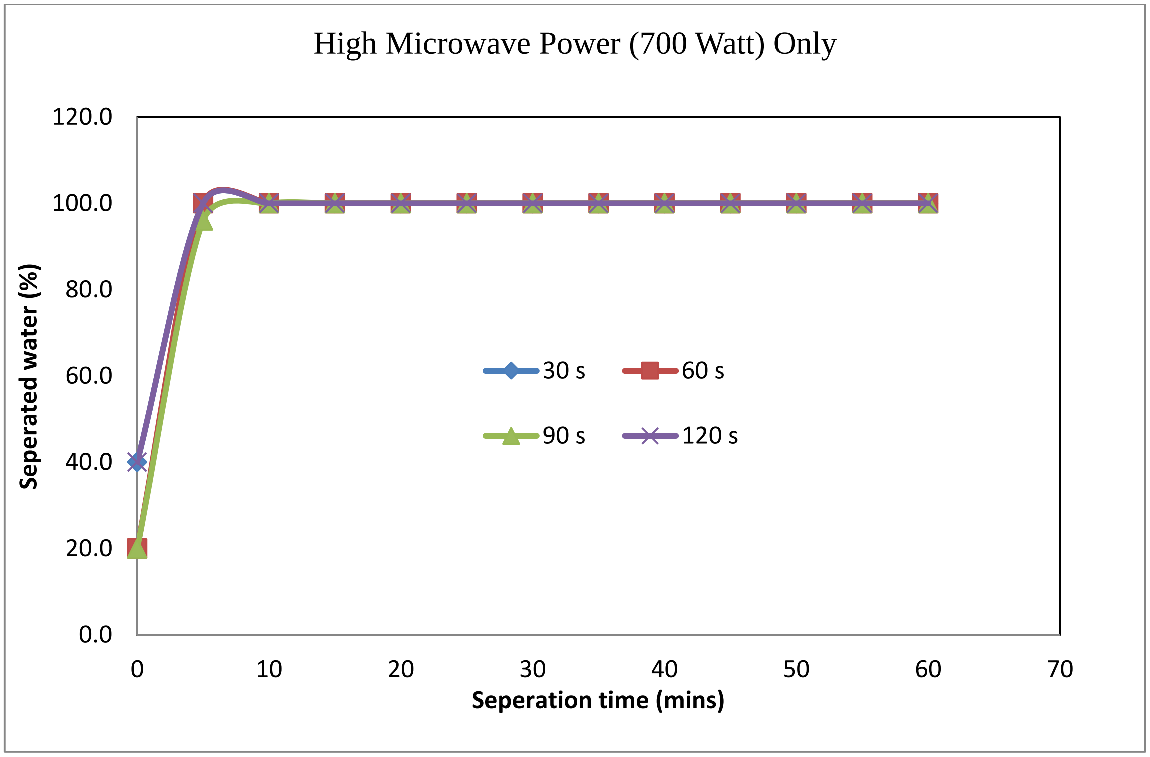

Figure 4.17: Graph of separated water (%) vs separation time (mins) for high microwave power (700 watt) + 0.5 ml demulsifier

4.3.2.1 Comparison Based on Exposure Time

In this subsection, the different results obtained from the various microwave power levels are grouped under exposure time basis and the results are shown in the subsequent graphs shown below.

In the following figures 4.18, 4.19, 4.20, 4.2.1, a comparison is done based on exposure time. It is seen that a steep slope is observed in all the four cases of 30, 60, 90 and 120 seconds exposure time. After the steep slope, a plateau is observed. This shows that a maximal water recovery of 100% has been reached. It is also observed that for loose emulsions, there is no need to expose it in the microwave for more than 30 seconds as the results obtained for the 30 seconds exposure time is good enough so as to discontinue from further heating.

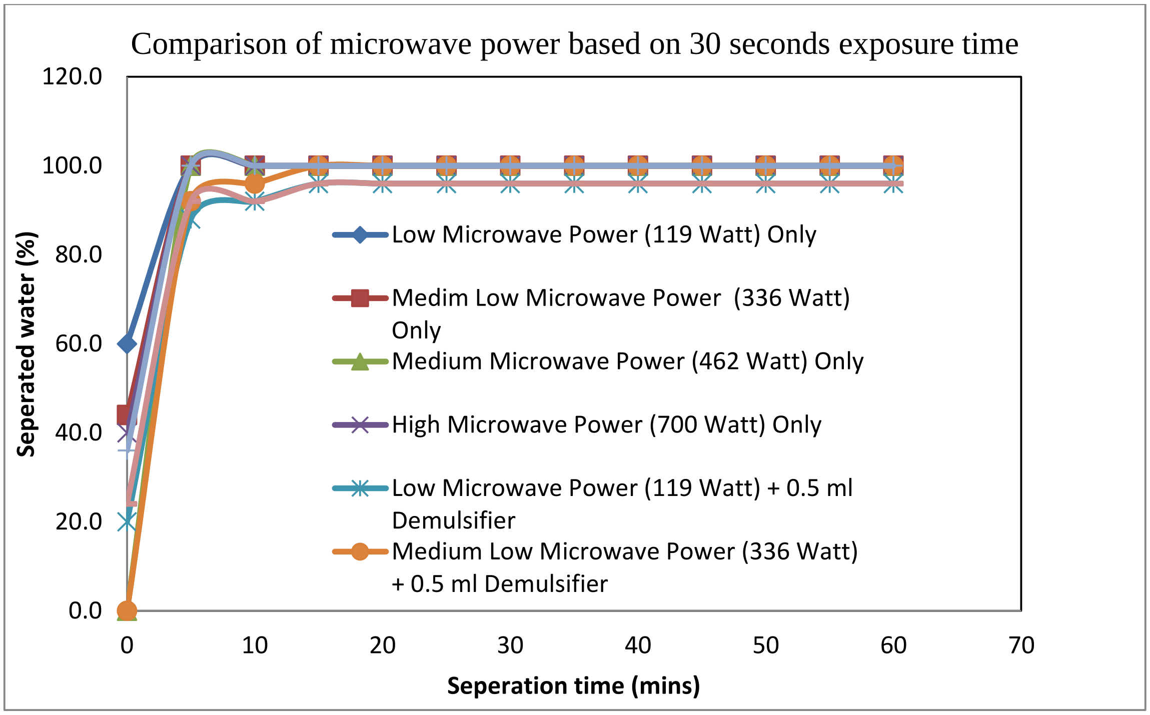

Figure 4.18: Graph of separated water (%) vs separation time (mins) for 30 seconds exposure time at different microwave power levels

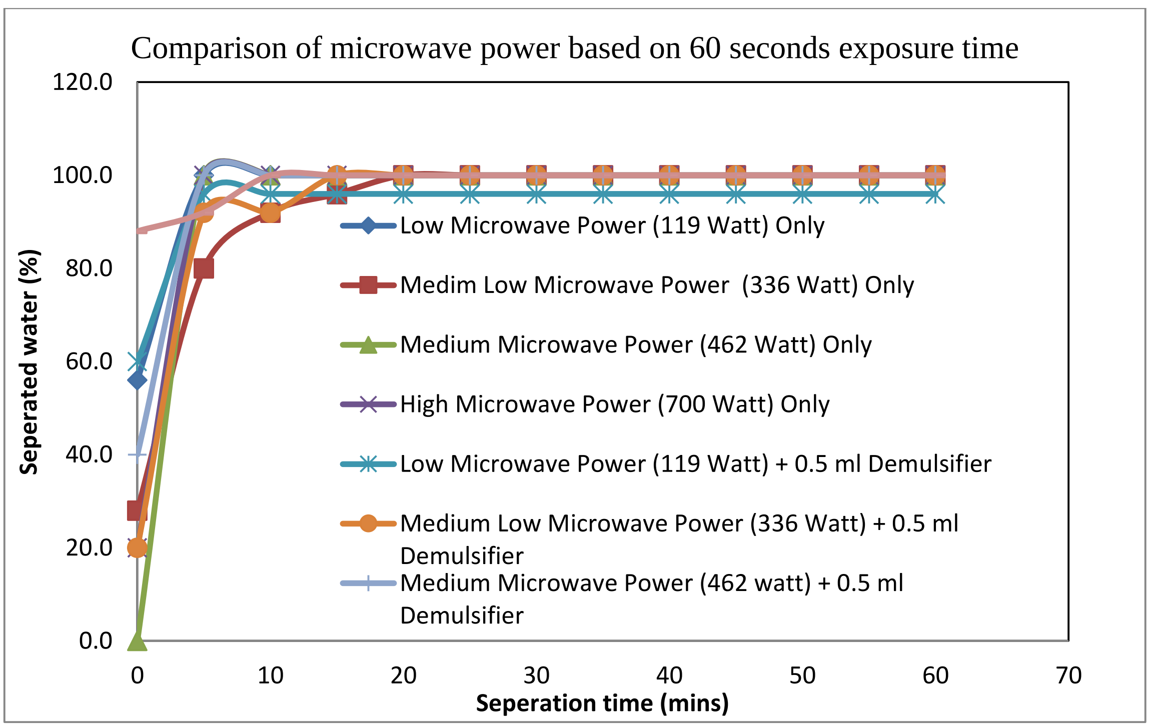

Figure 4.19: Graph of separated water (%) vs separation time (mins) for 60 seconds exposure time at different microwave power levels

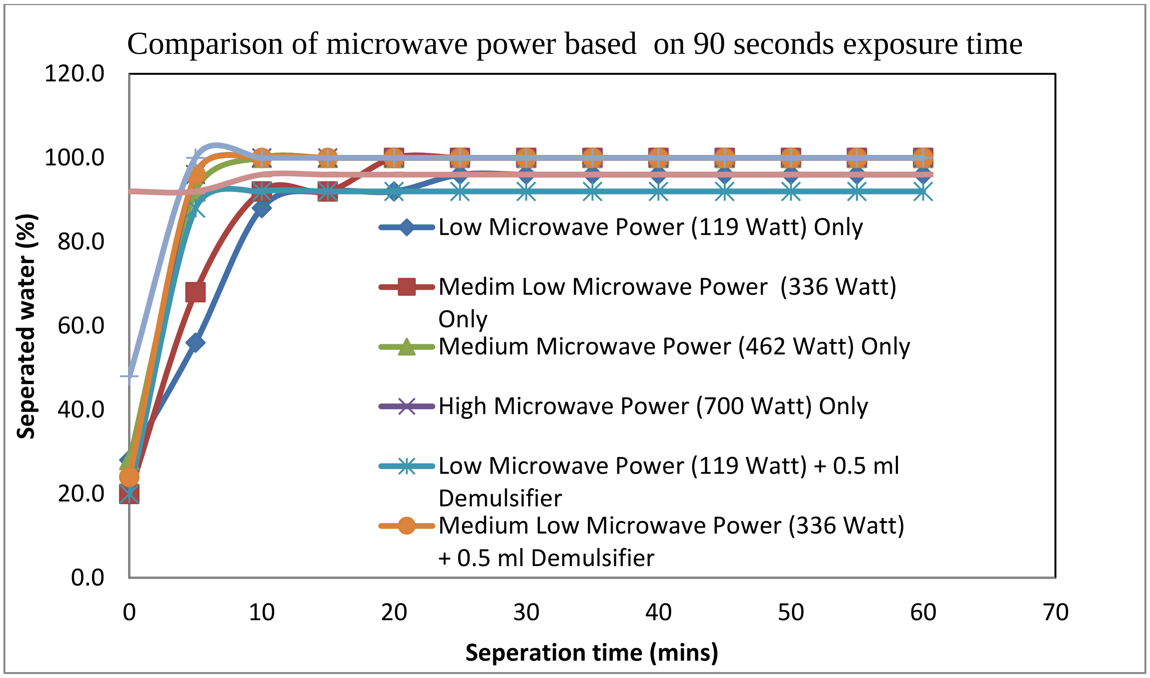

Figure 4.20: Graph of separated water (%) vs separation time (mins) for 90 seconds exposure time at different microwave power levels

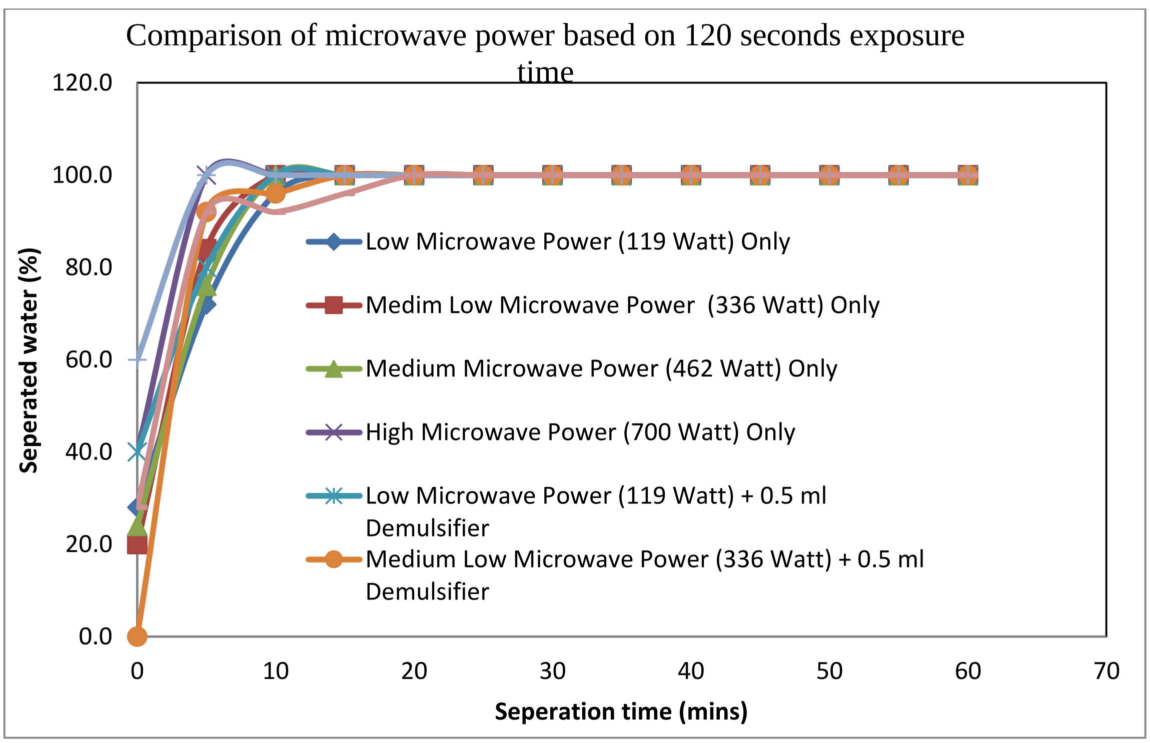

Figure 4.21: Graph of separated water (%) vs separation time (mins) for 120 seconds exposure time at different microwave power levels

4.3.3 Demulsifier only

In this subsection, the effect of using demulsifier only to resolve weak emulsion is observed.

Figure 4.22 indicates that between zero (0) and ten (10) minutes separation time, a steep slope is observed. At ten minutes separation time, a climax of 100% water recovery is achieved. This further ascertains that there a 50-50 water-oil ratio emulsion is a loose emulsion as demulsifier use only is enough to resolve it.

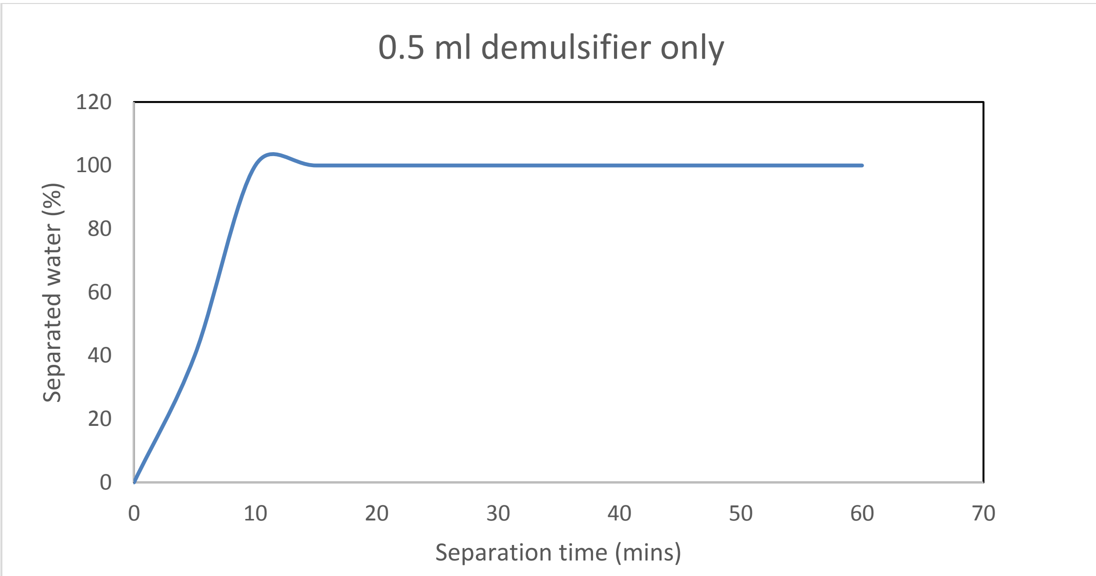

Figure 4.22: Graph of separated water (%) vs separation time (mins) for 0.5 ml demulsifier only

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1. Conclusion

Water-in-oil emulsions constitute a major problem in the oil and gas industry. These emulsions are resolved to avoid flow problems as well as to meet international market standards. To resolve emulsions, heat, chemical or a mix of both can be used. This project investigated and observed the combination of heat and chemical in effectively resolving emulsions.

- Results show that microwave heating alone or the use of demulsifier alone is not sufficient in resolving tight water-in-oil emulsions. The synergy effect in the combination of heat (in this case from microwave radiation) and use of chemical was observed. The use of microwave only or demulsifier only is sufficient to break weak or unstable emulsions in little separation time. Thus, the application of both heat and chemical is not necessary and will amount to a waste of energy or resources if both are used for unstable emulsions.

- The kinetics of the mechanism was observed to be that, as the demulsifier attacks the emulsifier film, and tries to rupture it, the bulk heating provided by the microwave increases the entropy of the encapsulated water molecules. The combination of these effects, leads to the rupturing of this emulsified film which leads to coalescence of the water.

- It is worthy to conclude that the synergy effect of heat from microwave and chemical increases the effectiveness of the emulsion resolution. In doing this, cost is saved as little amount of demulsifier is used and 20.16 kJ (60 seconds exposure time at medium low microwave power level of 336 watt) of heat is required to achieve this feat.

5.2. Recommendations

To test and further justify these results, the following points are recommended:

- This same procedure should be carried out where the amount of demulsifier used is varied as well as the microwave power levels. This is to obtain a more accurate optimal point for amount of demulsifier use.
- Repeating the same procedure for oil-in-water emulsion. This is to observe the combined effect of heat and chemical on oil-in-water emulsion.
- This same procedure should be repeated for various water-oil ratios. Such as 10/90, 30/70, 40/60. This is to further understand the effect on water ratio in resolving emulsion using a combination of microwave and demulsifier.

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APENDICES

TIGHT EMULSIONS

Microwave heating only:

Separated water (%) in separation time (mins) for medium microwave power

	Separated water (%) in Separation time (mins)												
Tsep / Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
60 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
90 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
120 Seconds	0.0	0.0	0.0	10.0	10.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0

Microwave heating with demulsifier:

Separated water (%) in separation time (mins) for low microwave power

	Separated water (%) in Separation time (mins)												
Tsep Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
60 Seconds	0.0	0.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
90 Seconds	0.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
120 Seconds	0.0	0.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0

Separated water (%) in separation time (mins) for medium low microwave power

	Separated water (%) in Separation time (mins)												
Tsep Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	0.0	0.0	5.0	5.0	5.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0

60 Seconds	0.0	50.0	60.0	65.0	65.0	70.0	70.0	70.0	70.0	70.0	70.0	70.0	70.0
90 Seconds	0.0	10.0	50.0	65.0	65.0	65.0	65.0	70.0	70.0	70.0	70.0	70.0	70.0
120 Seconds	0.0	40.0	50.0	50.0	50.0	55.0	55.0	60.0	60.0	60.0	60.0	60.0	60.0

Separated water (%) in separation time (mins) for medium microwave power

	Separated water (%) in Separation time (mins)												
Tsep / Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
60 Seconds	0.0	10.0	30.0	45.0	45.0	45.0	45.0	50.0	50.0	50.0	50.0	50.0	50.0
90 Seconds	0.0	50.0	50.0	55.0	55.0	55.0	60.0	60.0	60.0	60.0	60.0	60.0	60.0
120 Seconds	0.0	50.0	60.0	65.0	65.0	65.0	65.0	70.0	70.0	70.0	70.0	70.0	70.0

Separated water (%) in separation time (mins) for high microwave power

	Separated water (%) in Separation time (mins)												
Tsep \ Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	20.0	40.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
60 Seconds	0.0	20.0	40.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
90 Seconds	0.0	0.0	30.0	40.0	40.0	45.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
120 Seconds	0.0	10.0	40.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0

LOOSE EMULSIONS

Microwave heating only:

Separated water (%) in separation time (mins) for low microwave power

	Separated water (%) in Separation time (mins)												
Tsep \ Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	60.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

60 Seconds	56.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
90 Seconds	28.0	56.0	88.0	92.0	92.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0
120 Seconds	28.0	72.0	96.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Separated water (%) in separation time (mins) for medium low microwave power

	Separated water (%) in Separation time (mins)												
Tsep / Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	44.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
60 Seconds	28.0	80.0	92.0	96.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
90 Seconds	20.0	68.0	92.0	92.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
120 Seconds	20.0	84.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Separated water (%) in separation time (mins) for medium microwave power

	Separated water (%) in Separation time (mins)												
Tsep Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
60 Seconds	0.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
90 Seconds	28.0	92.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
120 Seconds	24.0	76.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Separated water (%) in separation time (mins) for high microwave power

	Separated water (%) in Separation time (mins)												
Tsep Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	40.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

60 Seconds	20.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
90 Seconds	20.0	96.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
120 Seconds	40.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Microwave heating with demulsifier:

Separated water (%) in separation time (mins) for low microwave power

	Separated water (%) in Separation time (mins)												
Tsep / Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	20.0	88.0	92.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0
60 Seconds	60.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0
90 Seconds	20.0	88.0	92.0	92.0	92.0	92.0	92.0	92.0	92.0	92.0	92.0	92.0	92.0

120 Seconds	40.0	80.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Separated water (%) in separation time (mins) for medium low microwave power

	Separated water (%) in Separation time (mins)												
Tsep / Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	0.0	92.0	96.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
60 Seconds	20.0	92.0	92.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
90 Seconds	24.0	96.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
120 Seconds	0.0	92.0	96.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Separated water (%) in separation time (mins) for medium microwave power

	Separated water (%) in Separation time (mins)												
Tsep Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	36.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
60 Seconds	40.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
90 Seconds	48.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
120 Seconds	60.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Separated water (%) in separation time (mins) for high microwave power

	Separated water (%) in Separation time (mins)												
Tsep Te	0	5	10	15	20	25	30	35	40	45	50	55	60
30 Seconds	24.0	92.0	92.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0

60 Seconds	88.0	92.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
90 Seconds	92.0	92.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0
120 Seconds	28.0	92.0	92.0	96.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Demulsifier Only:

Separated water (%) in separation time (mins) for 0.5 ml demulsifier only

T Sep (mins)	0	5	10	15	20	25	30	35	40	45	50	55	60
Separated water (%)	0.0	40.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0