DISSERTATION

NANO-EMULSION FOR PRODUCTION ENHANCEMENT PURPOSE

by

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ABSTRACT

Efficient wellbore cleaning to remove OBM (oil-based mud) residue is important in order to prevent contamination of clean completion brines. Contaminated completion brine is detrimental to the reservoir productivity. If left in place oil reacts with brine to form excessive skin or even form emulsion blockages that can damage the formation eventually leads to a reduction in oil and gas production rate. The best solution to overcome this problem is to remove all OBM residues and replacing the casing to water wet. As a result, the brine can be maintained clean thus reducing the potential of skin damage and emulsion blockage. Conventional clean-up system requires large volume of surfactant and solvent, turbulent flow and incomplete wetting phase change. Recent development in nano-technology has resulted in creation of nanoemulsion fluid for removing OBM residue. Nanoemulsion is ideal for remediating formation damage by removing emulsion blockage and re-water wetting the reservoir. This project studies the potential of nanoemulsion technology in removing OBM residue during wellbore cleaning process. Two sets of experiment will be conducted to compare the cleaning efficiency between nanoemulsion and conventional cleaner fluid : 1)Test at ambient condition using standard API-35 Viscometer and 2)Test at High Pressure High Temperature condition using HPHT Filter Press. The data obtain from these experiments will be studied and analyzed to compare the cleaning efficiency between nanoemulsions and conventional cleaner fluid. Then, the feasibility of using nanoemulsion can be determined in term of cleaning efficiency at different well conditions.

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Nanotechnology (also known as "Nanotech") is the art of manipulating matter on atomic and molecular scale. Commonly, nanotechnology is used to develop materials, devices, or other structures with at least one dimension sized ranging from 1-100 nanometers. The application of nanotechnology is very diverse, ranging from modification of conventional device to completely new approaches based upon molecular self-assembly, from developing new nanoscale material to direct control of matter on atomic scale. The technology is being applied on various fields including the electronics, biomedical, pharmaceutical, materials and manufacturing, aerospace, photography, etc. Most applications are limited to the use of "first generation" passive nanomaterials which allows tennis ball to last longer, golf balls to fly straighter, bowling balls to become more durable and have a harder surface, bandages to heal faster, computer to become cheaper, faster and larger memory size. Thanks to nanotechnology for the advancement in daily life. In like manner, nanotechnology is also being implemented in oil and gas industry.

In oil and gas industry, nanotechnology has great potential to introduce revolutionary change in the area of exploration, drilling, production, enhanced oil recovery, refining and distribution. For instance, nanosensors can provide more detail and accurate reservoir data, nanoparticles can be used as scale inhibitor, nanomaterial could allow the development of better equipment which is lighter, reliable and durable, and nanomembrane could enhance the gas separation and help removing impurities from oil and gas streams [1]. In addition, another development in petroleum industry is the creation of new types of "smart fluid" for enhanced oil recovery (EOR) and drilling purposes.

Nanotechnology has opened the door to the development of new type of drilling fluid known as "smart fluid" for drilling, production and stimulation-related applications. From drilling perspective, such smart fluids will help to enhance drilling process by forming thinner and impermeable mud cake which in turns will reduce the formation damage while drilling [2]. Due to smart fluids high surface to volume ratio, the layer of mud cake can be easily removed by conventional cleaning system during well completion stage [2]. As for these reasons, the formation damage is virtually eliminated thus increasing hydrocarbon production.

Besides of drilling fluid, nano-technology is also being implemented in completion fluid. Completion fluid is a solids-free liquid used to complete an oil or gas well [3]. Nano-emulsion is introduced in completion fluid to provide better well cleaning efficiency through its ability to significantly reduce oil-water interfacial tension (IFT) thus allowing easy removal of oil residue on the wellbore surface. As a result of efficient cleaning process, the formation damage such as mudcakes can be reduced therefore improving the well productivity. Brines (chlorides, bromides and formats) are the typical completion fluid. Drilling fluid is not suitable to be used as completion fluid due to its solid content, pH and ionic composition. Drill-in-fluids might be suitable for both purposes in some cases [3].

1.2 Problem Statement

It is important to have efficient OBM residue removal, filter cake removal and formation clean up prior for well cementing, production and injection wells for primary, secondary and tertiary oil recovery. OBM residue must be removed completely in order to prevent brine contamination that can cause emulsion blockage, thus reducing well productivity. The problem with conventional clean up system is it requires large volume of surfactant and solvent, turbulent flow to solubilize residue efficiently, cause incomplete wetting phase of casing surface and last but not least, the escalating cost of surfactant. In order to overcome this problem, a new type of emulsion known as 'Nano-emulsion' is introduced in the industry. This product of nano-technology is believed to have higher cleaning efficiency in term of removing OBM residue, hence improving the well productivity.

1.3 Objective and Scope of Study

The objectives of this study are:

- To study the effect of nanoemulsion on completion fluid behaviour and properties
- To compare the mud removal efficiency between nanoemulsion and common cleaner fluid
- To suggest a design improvement to further increase mud removal efficiency

The scope of study includes:

- Application of nanoemulsion fluid for cased hole completion during completion stage

CHAPTER 2

LITERATURE REVIEW

2 Literature Review

The study is focusing on how nano-emulsion help increasing hydrocarbon recovery by efficient removal of oil based mud (OBM) residue. Basically, this literature review will cover the fundamental theory and concept related to OBM residue, surfactant and nanoemulsion.

2.1 OBM Residue

Oil Based Mud (OBM) residue is the leftover mud on the casing or formation surface after well bore cleanup operation. In open hole completion (without casing) the OBM residue is formed as filter cake deposited on the formation whereas for cased hole completion (with casing) a thin oily film is formed on casing surface.

In order to clean the casing from mud, a proper cleaning fluid and transition spacers must be injected in correct order [8, 14]. Cleaning is first initiated by base fluid, normally water and some brine to push the mud out of the hole through the annular space. Then, the wash fluid consisting of brines and surfactant is injected to clean the remaining residue from the pipe. The wash fluid and base fluid must be separated by viscous pill to prevent them mixing together. After finished cleaning the well, a completion fluid is used to fill up the well completely. Viscous pill is used once again here as a separator to prevent mixing of completion fluid with wash fluid. Regardless of the techniques, it is very difficult to achieve 100% cleaning efficiency since the residue has tendency to stick on solid surface due to wettability effect.

Commonly, the casing is oil wet. The wetting phase is determined by the contact angle. Contact angle ,θ , is a quantitative measure of the wetting of a solid by a liquid. It is defined geometrically as the angle formed by a liquid at the three phase boundary where a liquid, gas and solid intersect as shown below:

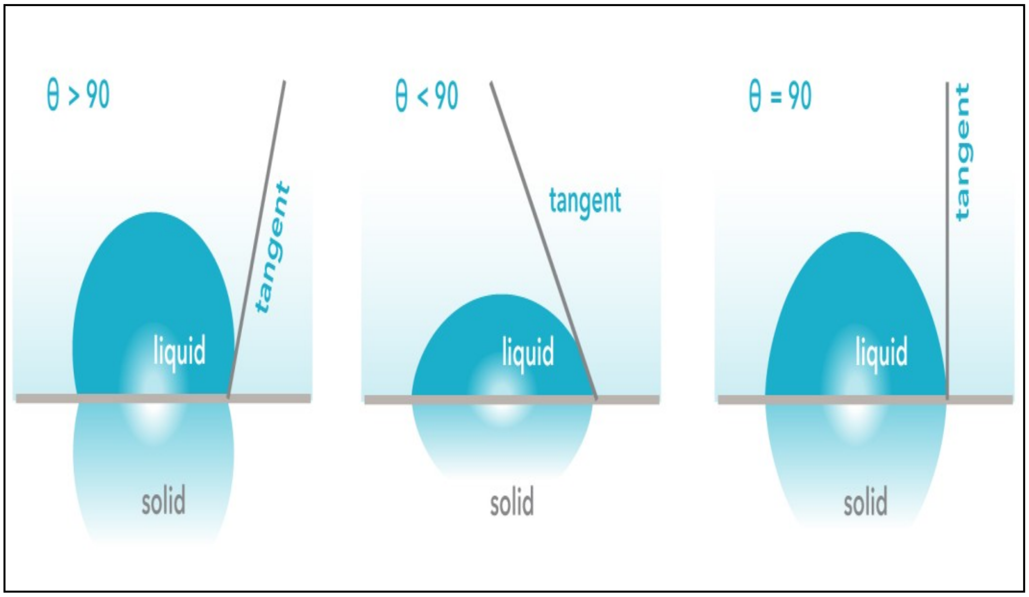

Figure 1. Relation between contact angle and wettability

It can be seen from this figure that a low values of contact angle (θ) indicates that the liquid spreads, or wets well, while a high contact angle indicates poor wetting. If the angle θ is less than 90 degrees the liquid is said to wet the solid. If it is greater than 90 degrees it is said to be non-wetting. A zero contact angle represents complete wetting. Wettability can be influenced by temperature and pressure. However, it is still in debate that temperature and pressure effects balance each other in term of comparing degree of wettability in laboratory and reservoir condition [9].

Failure to remove OBM residue is detrimental to the well. If the issue is unresolved the oil may react with brine water which is used as completion fluid. The oil contaminates the brine by forming undesirable emulsion. Formation of emulsion in the wellbore will be easiest at high shear rates and high emulsifiers in the mud residue [10]. This emulsion may block the reservoir or formation after the well is perforated. If this happen, it will lead to decrease in well productivity.

2.2 Surfactant

Surfactant is an organic substance that is capable of reducing interfacial tension (IFT) between two medium for example gas/liquid, liquid/liquid or solid/liquid. Emulsifiers are considered class of surfactants. Surfactant lowers the surface tension of bulk liquid thus allowing it to be more compatible with the hydrophobic material or oil. An emulsifier interacts with hydrophobic part, giving it some type of coating which 'hides' the oil from the water, thus making the surface of oil resemble the one of water[11].

Surfactant consists of two main parts, one is a water-soluble (hydrophilic part), and the other is oil-soluble (hydrophobic part) (figure 1) [4, 15]. When surfactant is mixed into completion fluid, for example brine, the hydrophilic head and hydrophobic tail groups will be adsorbed at the interfacial surface thus reducing the IFT(figure 2)[4]. However, in order to reduce Gibss free energy, surfactant molecule tends to self-arrange themselves, forming a 3-D molecular cluster defined as 'micelle' (figure 3)[4].

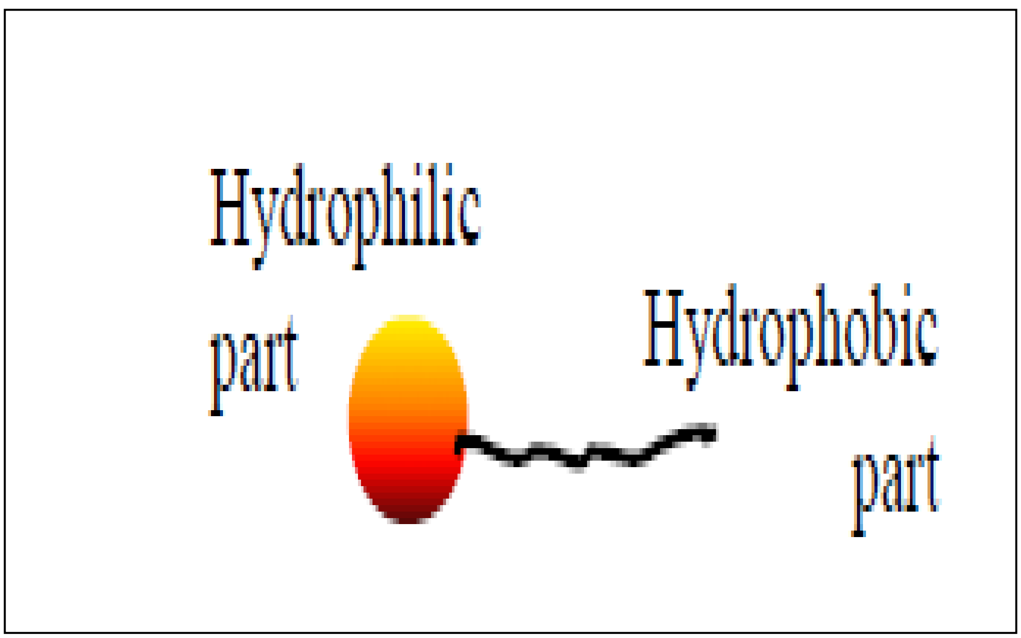

Figure 2. Hydrophilic head and hydrophobic head (Courtesy of Chun et al., 2011)

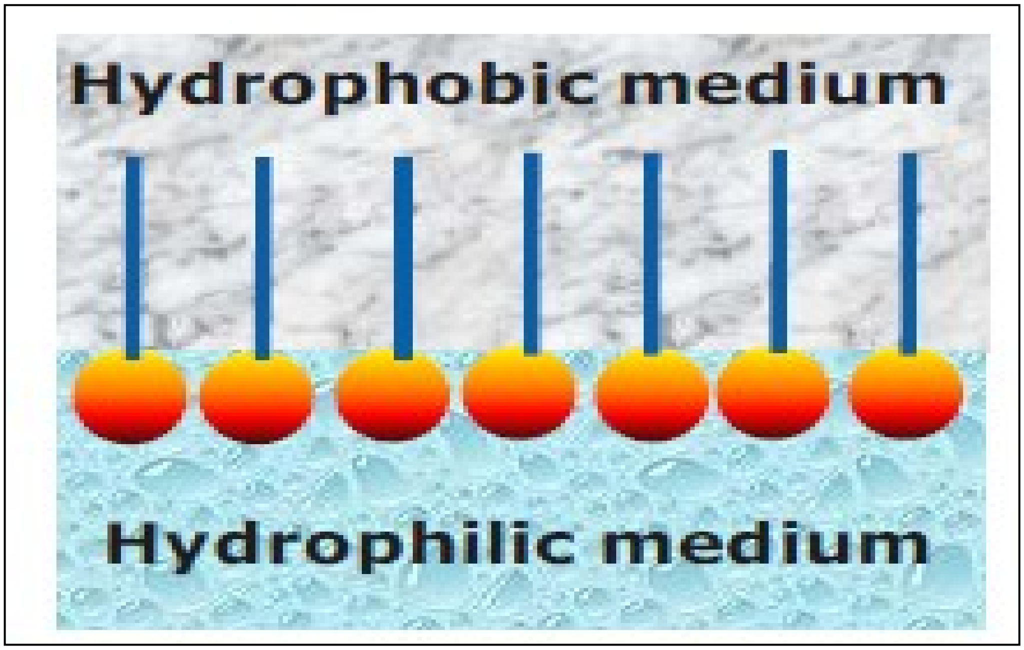

Figure 3. Adsorption of surfactant molecule at the interfacial surface (Courtesy of Chun et al., 2011)

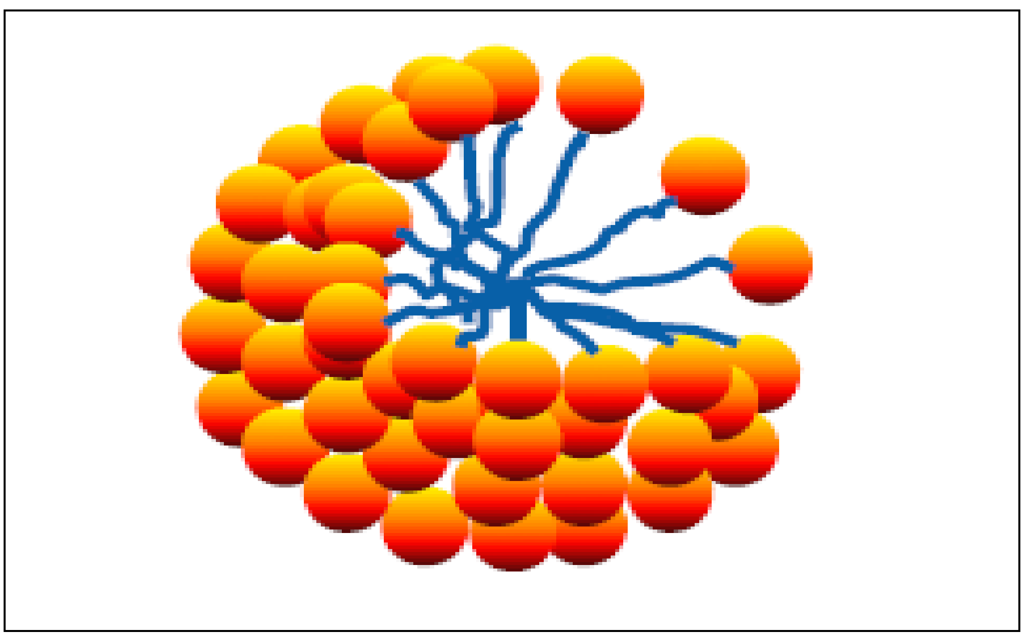

Figure 4. Strucuture of micelle in 3-D (Courtesy of Chun et al., 2011)

The concentration of surfactant is inversely proportional to the IFT (figure 4) [4]. Increase in surfactant concentration means decrease in IFT. In spite of that, the IFT can only be reduced up to minimum interfacial tension. The concentration at this point is defined as critical micelle concentration (CMC) of the surfactant. In colloidal and surface chemistry, the critical micelle concentration is described as the concentration of surfactans above which micelles form and almost all additional surfactants added to the system go to micelles [12]. Above this concentration, the surface tension could not be reduced further. Further addition of surfactant will only result in formation of new micelles since the interfacial surface is already being saturated with surfactant molecules.

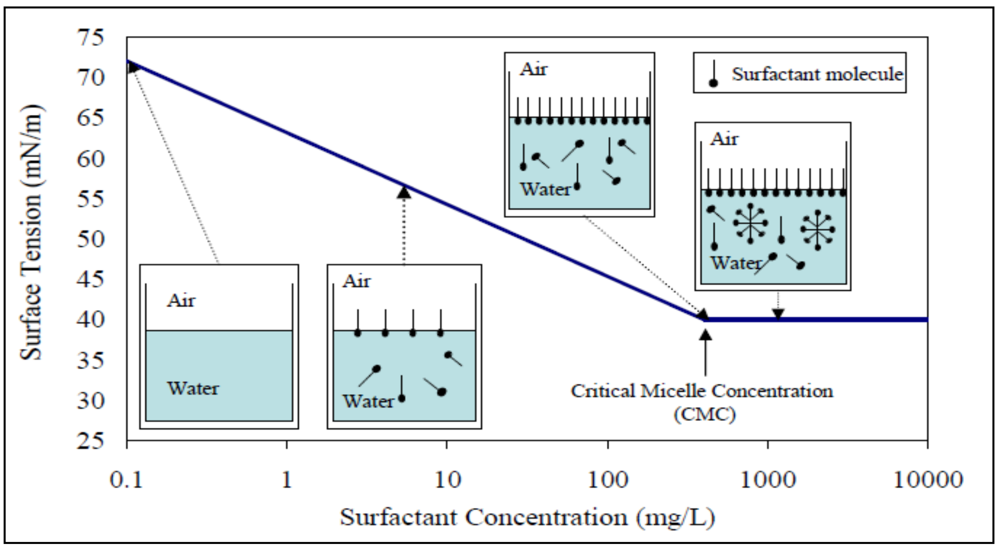

Figure 5. Graph of surface tension versus surfactant concentration (Courtesy of Chun et al., 2011)

2.3 Nano-emulsion

Emulsion is formed when two immiscible fluid (eg.water and oil) are mixed together. Nano-emulsion is considered as special type of emulsion with extremely small particle size range of 1-10 nm which is smaller than the wavelength of light (400nm $\leq \lambda_{light} \leq$ 700nm). For this reason, light is able to pass through the emulsion, creating weak scatters of light. This explains their transparent properties.

In contrast to microemulsion phases, relatively little is known about creating and controlling nanoemulsions. This is due extreme shear, well beyond the reach of conventional mixing devices, must me be apllied to overcome the effects of surface tension to rupture the droplets into the nanoscale regime [13]. In industry, nanoemulsions play an increasingly important role since they can typically be formulated using significantly less surfactant than is required using microemulsion.

Nano-surfactant reduces the interfacial tension between aqueous and hydrocarbon phase in nano-emulsion down to 0.0001 mN/m, compared with ordinary or micro emulsion (20-50 mN/m.) (Chun et al., 2011)[4]. Nano-emulsion can be classified into four type according to Winsor's nomenclature (Zanten et al., 2010) [5].

1. Windsor I – Two phase system, O/W micellar dispersion, excess oil phase, water dispersed in oil (figure 5a)[5]
2. Windsor II – Two phase system, W/O micellar dispersion, excess water phase, oil dispersed in water (figure 5b) [5]
3. Windsor III- Three phase system, bicontinuos dispersion, excess water and oil phase (figure 5c) [5]
4. Windsor VI- Single phase system, swollen micelles dispersed in aqueous phase

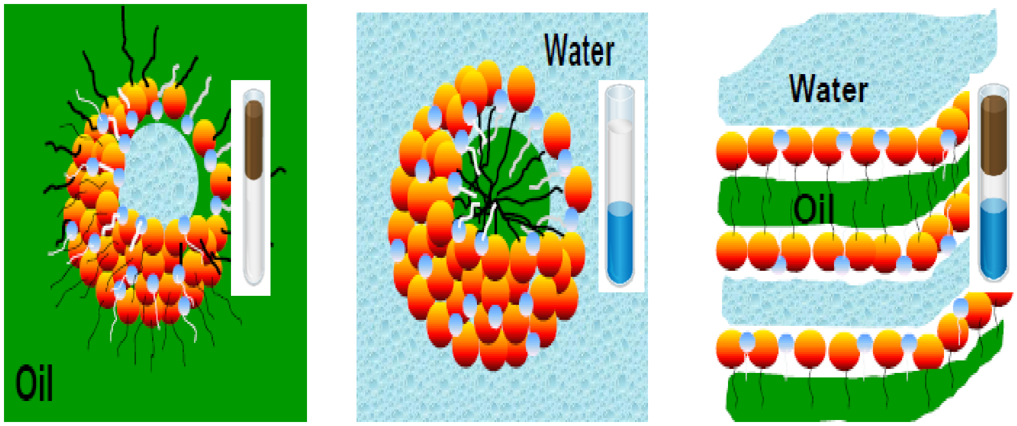

Figure 6. From the left, figure 5(a), 5(b) and 5(c) showing the molecule structure of Windsor type I, II and III respectively (Courtesy of Chun et al., 2011)

The surface tension reduction varies for different types of nanoemulsion (figure 6). In term of surface tension, the lowest value is achieved by middle phase nanomaterial. Ultralow interfacial tension is highly desirable to promote displacement of oil from surfaces and porous media.

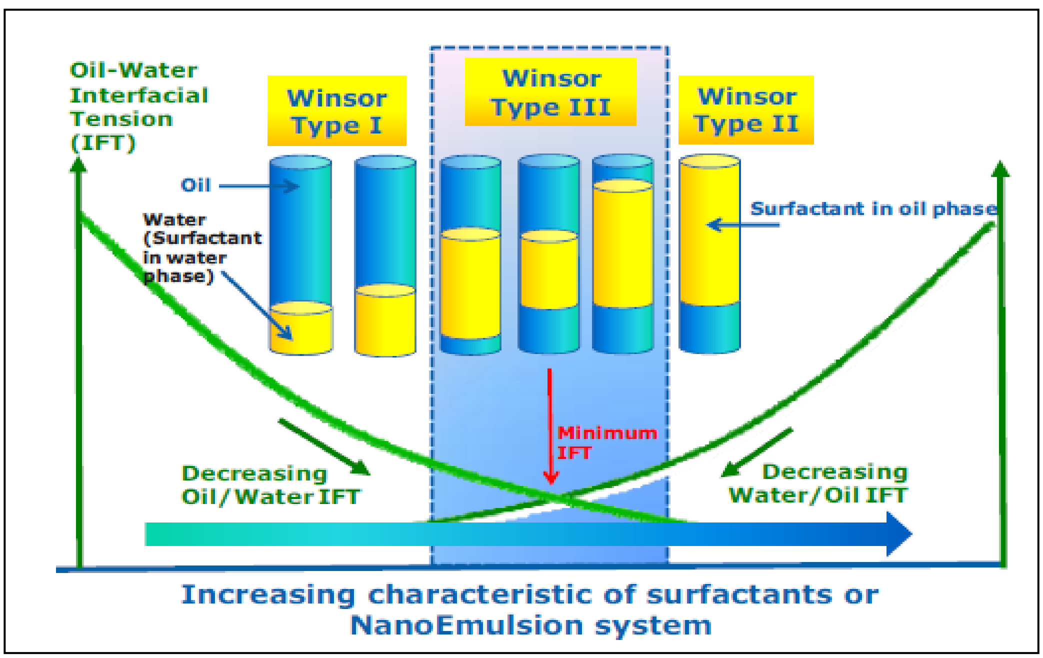

Figure 7. Phase transition of nano-emulsion effect on interfacial tension (IFT)

CHAPTER 3

METHODOLOGY

3 Project Methodology

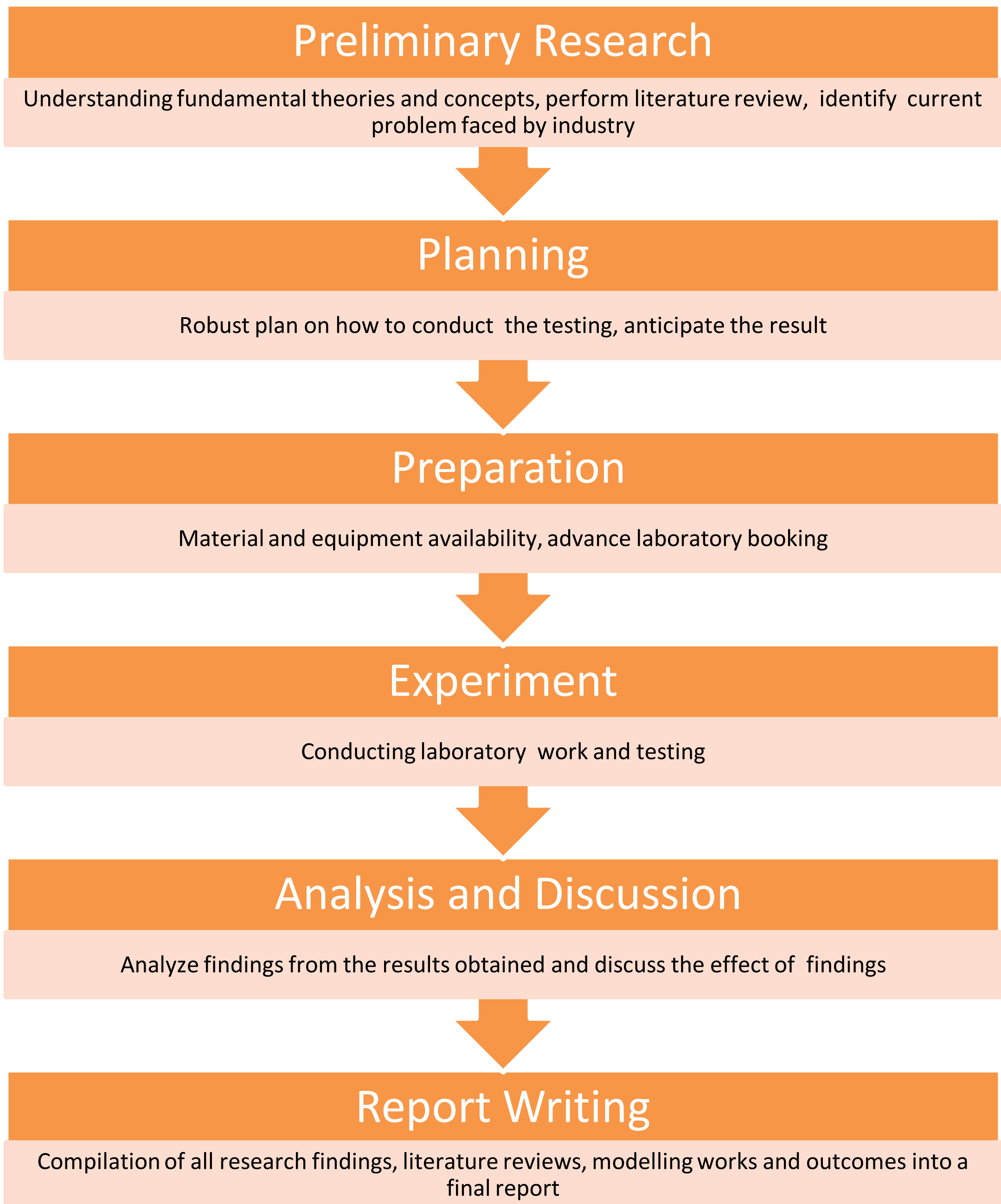

Figure 8 Process flow of work

3.1 Key Milestone

Table 1. Key Milestone for Project

Week	Objectives
FYP I	
5	Completion of preliminary research work
6	Submission of extended proposal
9	Completion of proposal defence
12	Confirmation on lab material and equipment for conducting experiment
13	Submission of Interim draft report
14	Submission of Interim report
FYP II	
5	Finalized the experiment procedure
6	Conducting experiment
7	Result analysis and discussion
8	Submission of progress report
9	Preparation for Pre-SEDEX
11	Pre-SEDEX
12	Submission of draft report
13	Submission of technical paper and dissertation
14	Oral presentation
15	Submission of project dissertation

3.2 Gantt Chart

Table 2. Proposed Gantt chart for the project implementation for both FYP I and FYP II.

TOPIC	PROJECT ACTIVITIES	WEEKS																											
		Final Year Project 1														Final Year Project 2													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	Project Scope Validation																												
	Project Introduction																												
	Submission of Extended Proposal																												
	Identify material and equipment																												
	Training on how to conduct experiment																												
	Proposal Defence																												
	Detail Study																												
	Submission of Interim Draft Report																												
	Finalized Procedure																												
	Conducting Experiment																												
	Result analysis and discussion																												
	Submission of progress report																												
	Preparation for Pre-SEDEX																												
	Pre-SEDEX																												
	Submission of draft report																												
	Submission of technical paper and dissertation																												
	Oral presentation																												
	Submission of project dissertation																												

Proposed Gant chart for the project implementation for both FYP I and FYP II. Based on the Gant Chart, the project is feasible to be completed within the given amount of time.

3.3 Material and Testing Procedure

❖ Material

1. Surfactant
2. Nanoemulsion fluid
3. Non-nanoemulsion fluid (detergent based common cleaner fluid)
4. Distilled water
5. Salt: Industrial grade $CaCl_2$
6. Non-aqueous mud (oil : water = 70 :30)

❖ Testing

1) Mud Removal Efficiency Test

-To measure the nanoemulsion efficiency in removing OBM residue

-Tested by standard API Fan35Rheometer Sleeve Testing

Figure 9. Standard API-35 Viscometer

Test Condition

Table 3. Test condition for Mud Removal Efficiency Test

Parameter	Condition
OBM	(Oil : Water = 70 :30)
Temperature	60 fahrenheit
Pressure	14.7 psia
Rotation speed	600 rpm
Contact time	10 minutes

Test Procedure

1. Weight a clean beaker. Pour mud into beaker, and weight the beaker again. Calculate initial mud weight, ***W_i***
2. Pour nanoemulsion water base cleaner into beaker. Mix the fluid inside the beaker using Fan35 Rheometer @ 300 rpm
3. Test is conducted at ambient room temperature and pressure
4. Let the fluid to mix for 10 minutes
5. Pour all the fluid out
6. Measured the weight of retained mud inside the beaker, ***W_f***
7. Repeat the same for common detergent base cleaner

Calculation

-Mud removal efficiency can be calculated using formula below:

%MRE = {{W_i-W_f))/W_i*100 Eq. (1)

Where MRE is the efficiency, W_f is the final mud weight loss and W_i is the initial mud weight.

2. ***HPHT Filtration Test***

-To measure the efficiency of nanoemulsion in removing mudcake for HPHT condition

-Tested by HPHT Filter Press

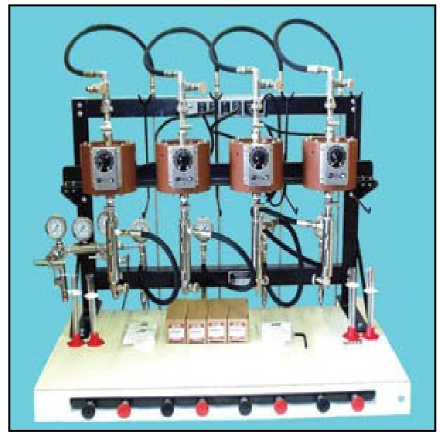

Figure 10. HPHT Filter Press

Test Condition

Parameter	Condition
OBM	(Oil : Water = 70 :30)
Filter cake	200 degree fahrenheit, 500 psi
Pressure	500 psi
Contact time	4 hours

Test Procedure

1. Measure the weight of empty HPHT cell. Pour OBM into the cell and reweight the cell. Calculate the initial mud weight, ***W_i***
2. Conduct HPHT test allowing formation of filter cake at API standard 500 psi and 250 degree fahrenheit for 30 minutes
3. Pour out the mud from HPHT cell and replaced with 5 v/v% nanoemulsion. Observe the mud cake appearance on filter paper. Retain the filter cake
4. Allow soaking for 3 hours. API standard 500 psi and 250 degree fahrenheit

5. After soaking, open the bottom valve of HPHT chamber to start filtration. Collect the filtration liquid
6. Pour out nanoemulsion from HPHT cell and reweight the cell. Calculate the final mud weight, ***W_f***.
7. Remove the filter paper and observe the mud cake appearance again after soaking

Calculation

-Mud removal efficiency can be calculated using formula below:

%MRE = {{W_i-W_f))/W_i*100 Eq. (1)

Where MRE is the efficiency, W_f is final mudcake in HPHT cell and W_i is the initial mud weight in the cell

CHAPTER 4

RESULT AND DISCUSSION

4 RESULT AND DISCUSSION

4.1 Mud Removal Efficiency Test

Test was conducted using standard API Fann35 Rheometer at ambient temperature and pressure. Two separate tests were performed to test the cleaning efficiency of nano-emulsion and non-nanoemulsion fluid respectively. Both fluids were tested on 11.5 ppg mud after hot-rolling for 16 hours at 250°F. A thin layer of mud coating was purposely deposited on the inner surface of 250 ml beaker to resemble the skin formed on casing surface at completion stage. The first cleaning was initiated by brine solution prepared at 10 weight-percent Calcium Chloride, $CaCl^2$. Cleaning was then continued using nano and non-nano cleaning fluid at 5 volume-% concentration. The beaker weight and surface appearance was observed and recorded for both experiment.

Non-nanoemulsion

Table 4. Result for cleaning efficiency test of non-nanoemulsion

Run	Weight	Mud Retain	Mud Removed	Efficiency
Empty beaker	108.4	0	-	-
Initial (mud+beaker)	231.5	123.1	-	0.00
Before cleaning with brine	118.5	10.1	-	91.80
After cleaning with brine	117.9	9.5	0.6	92.28
After cleaning with nano	116.8	8.4	1.1	93.18

Nanoemulsion

Table 5. Result for cleaning efficiency test of nanoemulsion

Run	Weight	Mud Retain	Mud Removed	Efficiency
Empty beaker	108.4	0	-	-
Initial (mud+beaker)	232.1	123.7	-	0.00
Before cleaning with brine	118.4	10	-	91.92
After cleaning with brine	117.8	9.4	0.6	92.40
After cleaning with non-nano	112.4	4	5.4	96.77

Non-nanoemulsion

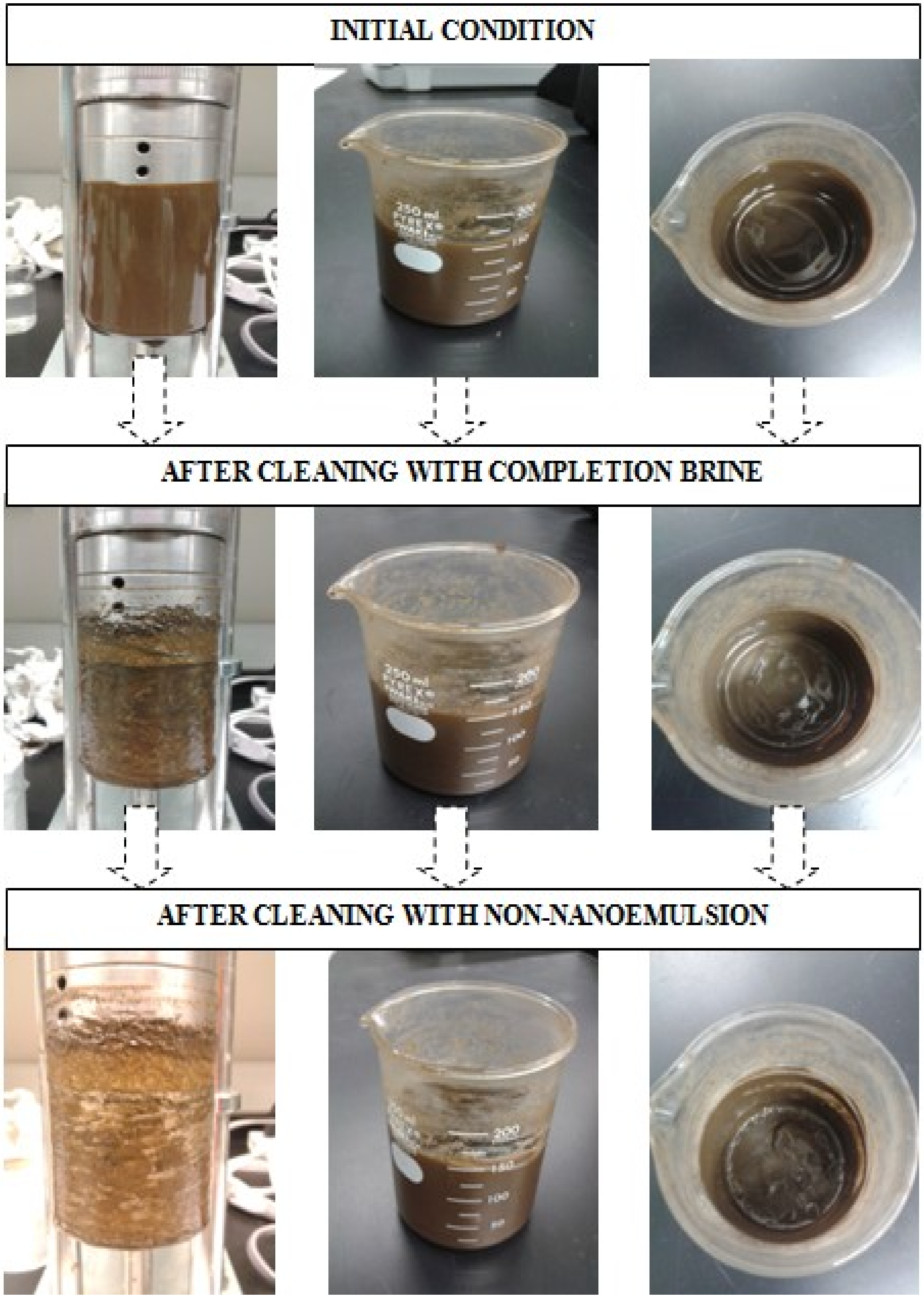

Figure 11. Retained mud observed on the rheometer sleeve, outer and inner side of 250 ml beaker for nano testing

Nanoemulsion

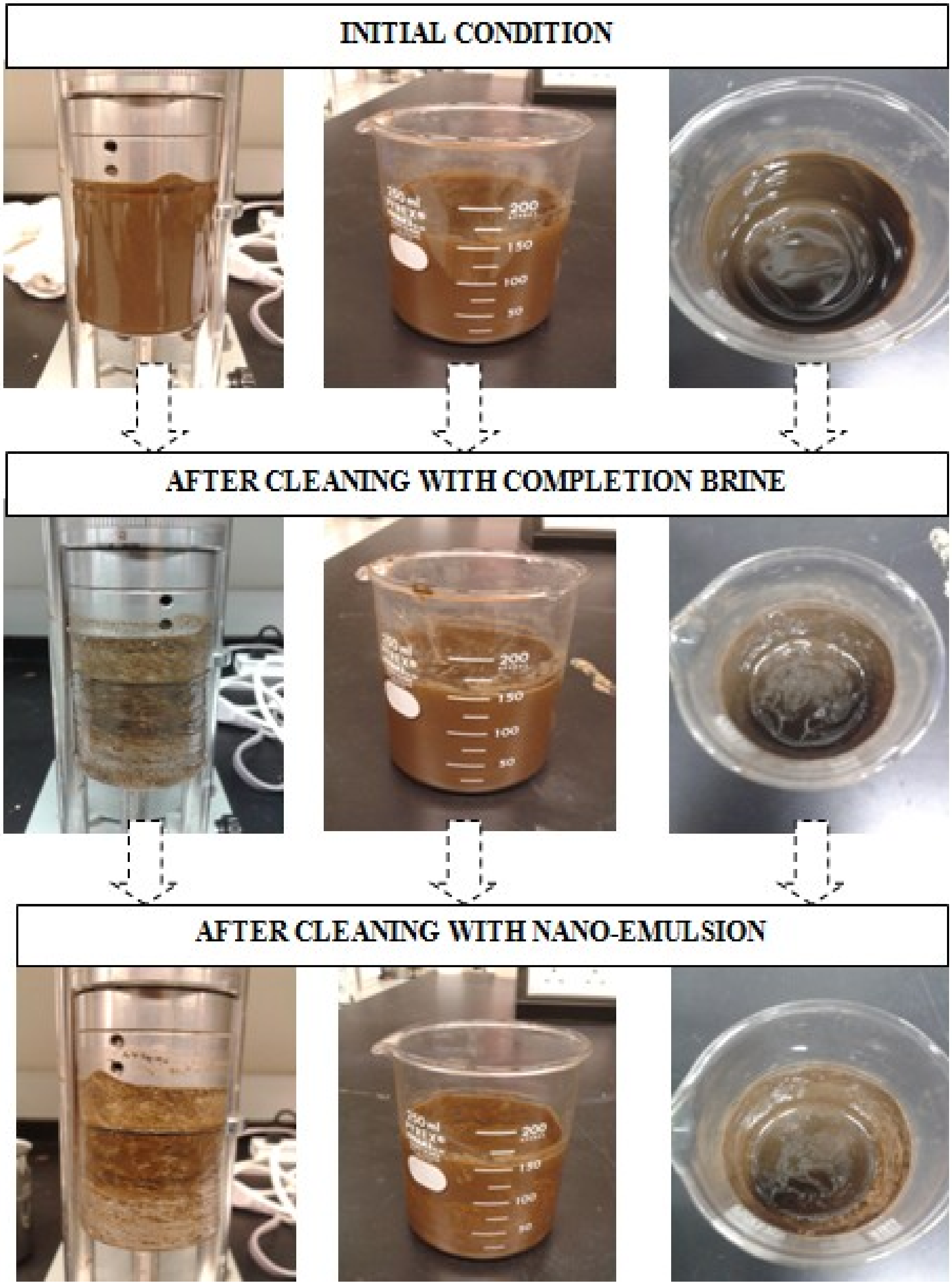

Figure 12. Retained mud observed on the rheometer sleeve, outer and inner side of 250 ml beaker for non-nano testing

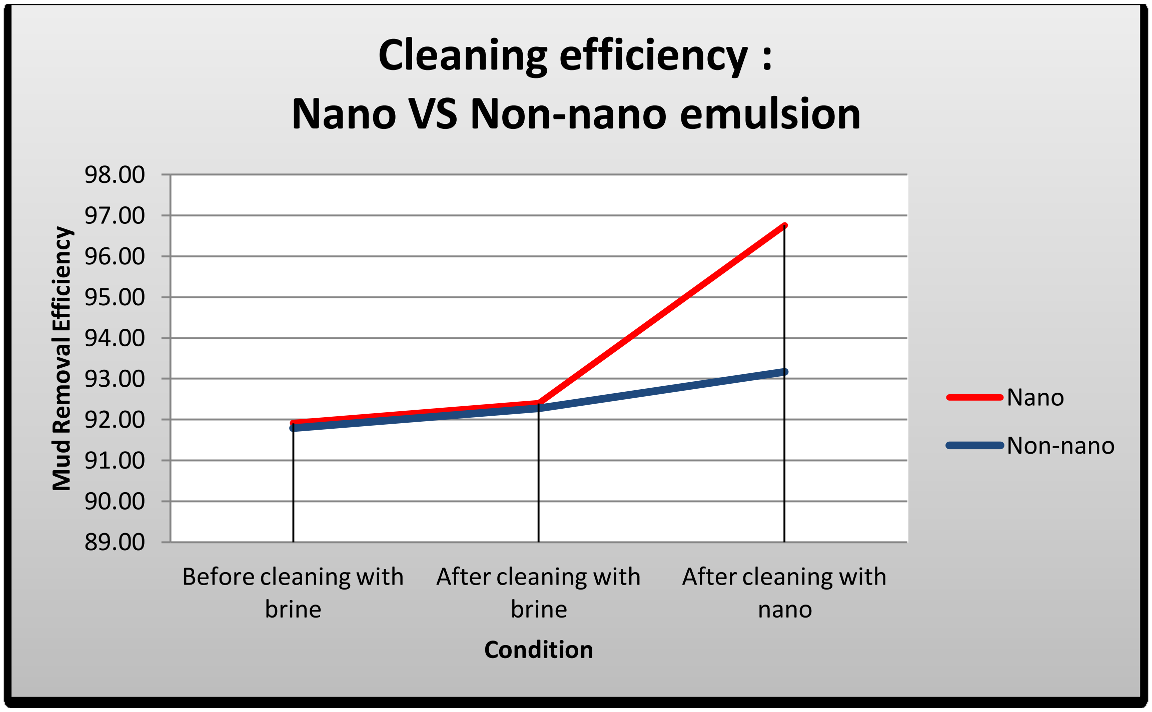

Figure 13. Graph showing mud removal efficiency of nanoemulsion VS non-nanoemulsion

Based on graph above, we could conclude that nanoemulsion has better cleaning effect compared to non-nanoemulsion. At 5% surfactant concentration, nanoemulsion and non-nanoemulsion are capable of removing the mud up to 97% and 93% respectively. The final amount of mud retain after cleaning with surfactant were 4 gram and 8.4 gram respectively. Lower amount of mud retain after cleaning with surfactant indicates better cleaning efficiency. In this case, nano-emulsion had successfully removed 5.4 gram of mud whereas non-nanoemulsion only managed to remove 1.1 gram of mud. The amount of mud removed at this final cleaning stage for nanoemulsion is almost 5 times more than the one removed by non-nanoemulsion. This experiment concludes that at 5% surfactant concentration, nanoemulsion is 5 times better than non-nanoemulsion at removing mud residue.

4.2 HPHT Filter Press Test

The effectiveness of nanoemulsion and non-nanoemulsion for cleaning mud residue at high temperature and pressure was tested using HPHT Filter Press Test. Soaking test was conducted for 3 hours at pressure 250 psia and temperature 250°F. The condition of HPHT cell after the soaking test was observed for cleaning with nanoemulsion and non-nanoemulsion respectively. In this case, the surfactant concentration was increased to 10 volume-% of surfactant for clearer observation inside HPHT holding cell.

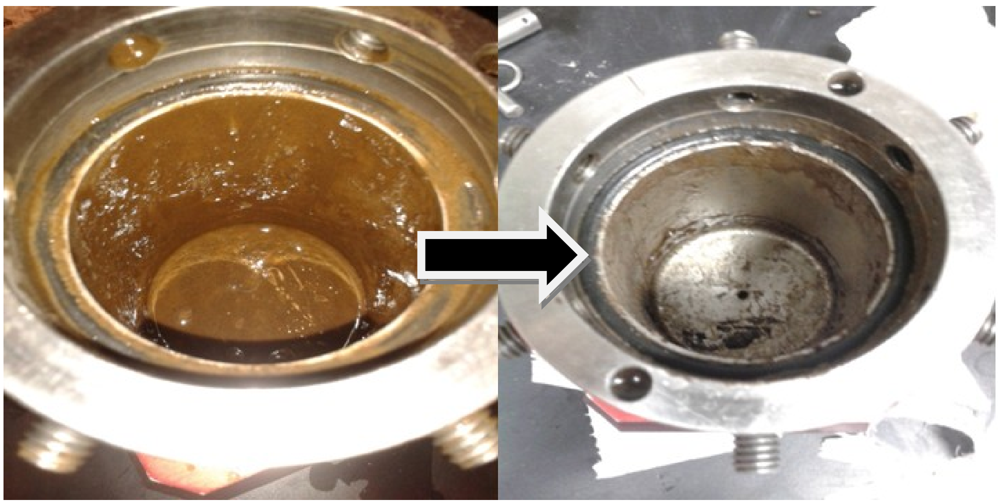

Figure 14. Inside view of HPHT cell before and after cleaning with nanoemulsion

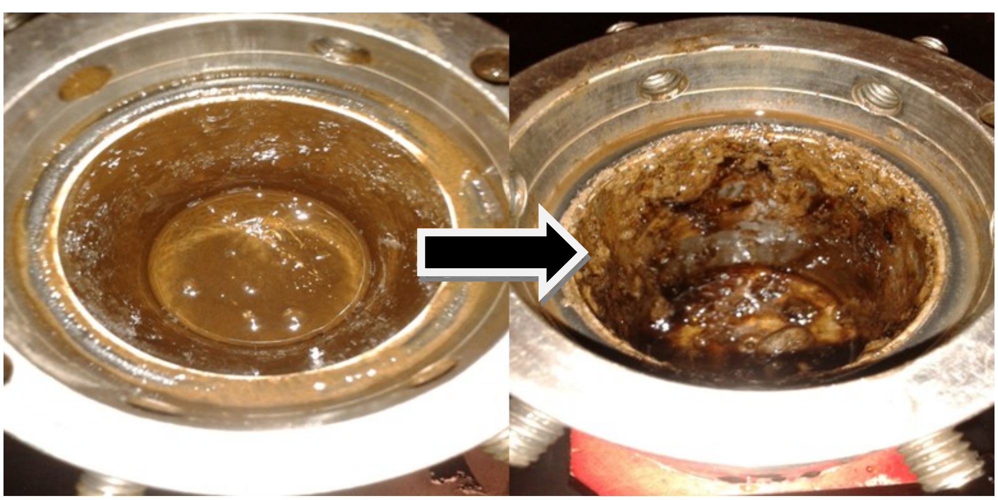

Figure 15. Inside view of HPHT cell before and after cleaning with non-nanoemulsion

The test result revealed that temperature does not impact the cleaning efficiency of nanoemulsion. Unlike non-nanoemulsion, the cleaning efficiency of nanoemulsion was still high even at high temperature (see figure 14). The inner side of HPHT cell soaked with nanoemulsion was clearer and cleaner compared to the one soaked with non-nanoemulsion. The soaking period allows enough time for the nanoemulsion to interact with mud particles, thus dissolving the remaining mud residue into the nanoemulsion. The mixture of mud and nanoemulsion was then collected as a filtrate during the filtration test (see figure 16). The brownish dirty colour of filtrate collected from nanoemulsion test indicates higher amount of mud dissolved in the solution. This explains the cleaner surface of HPHT cell. In contrast, the filtrate solution collected from non-nanoemulsion test is lighter in colour and cleaner, which indicates less dissolved mud residue. This in other hand explains the poor performance of non-nanoemulsion for cleaning the HPHT cell at high temperature and pressure(see figure17).

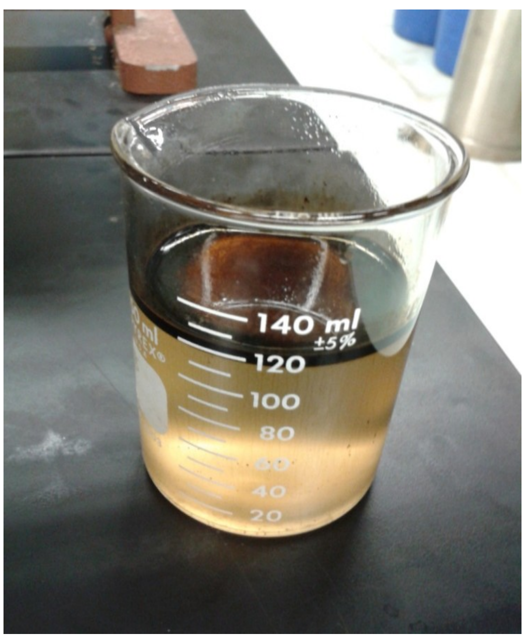

Figure 17. Filtrate collected for Non-nano test

Figure 16. Filtrate collected for Nano test

After soaking process for 3 hours, the experiment was continued with filtration test at pressure differential of 250 psia. For nanoemulsion test it took about 40 minutes for the fluid to be filtered out completely. However, the return fluid amount was less than the initial amount due to some fluid loss through evaporation. Conversely, it took more than 40 minutes for non-nanoemulsion fluid to be filtered out completely under the same pressure and temperature. Due to very slow filtration process, the pressure differential was increased to 500 psi after 40 minutes filtration time in order to speed up the process.

Table 6. Filtration test result for nanoemulsion

Time (sec)	Volume (ml)	Time (sec)	Volume (ml)
0.016667	10	15	40
0.166667	10	20	50
0.5	10	25	70
1	10	30	88
2	15	35	110
5	20	40	138
10	30	45	40

Table 7. Filtration test result for non-nanoemulsion

Time (sec)	Volume (ml)	Time (sec)	Volume (ml)
0.016667	8	15	10
0.166667	8	20	10
0.5	8	25	10
1	8	30	10
2	8	35	10
5	8	40	10
10	9	45	10

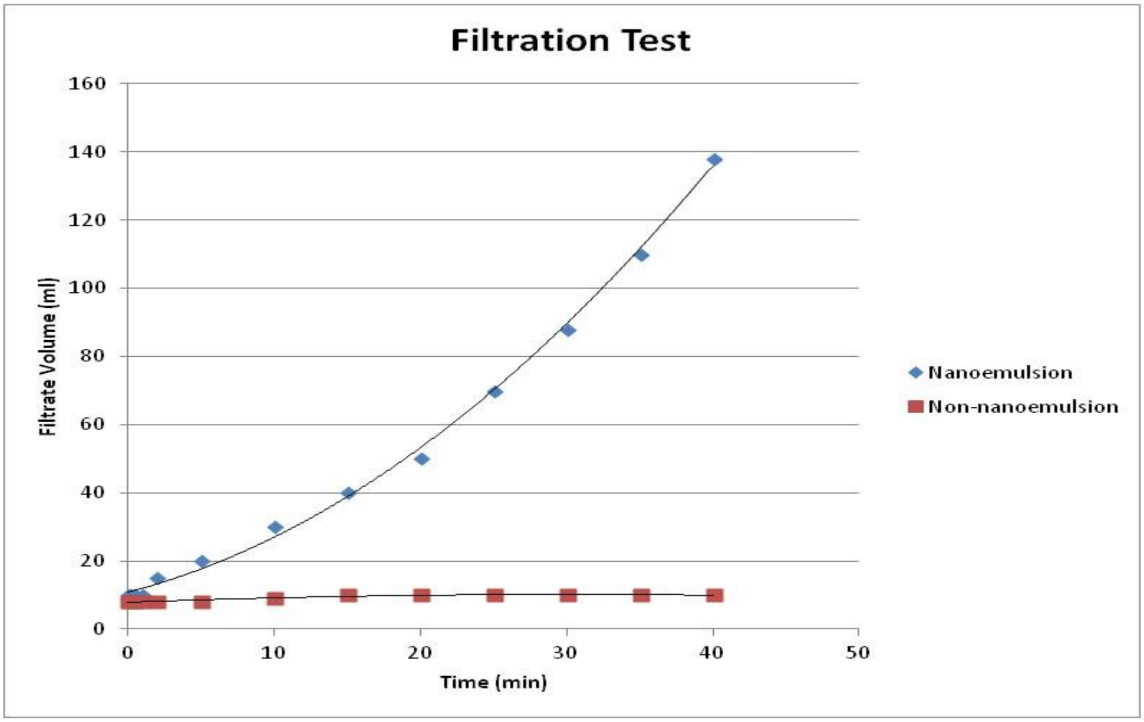

Figure 18. Filtration test result for nanoemulsion and non-nanoemulsion

Based on graph in figure 18, it was observed that the the filtration process for nanoemulsion was significantly faster than non-nanoemulsion. This is due to better cleaning performance of nanoemulsion which dissolved the mud residue more efficiently than the non-nanoemulsion. As a result, the filtration process was smoother and faster for nanoemulsion test. In the other hand, the same process was very slow for non-nano emulsion due to its poor cleaning performance which leads to formation of small mud lumps which then was deposited on the filter paper. Consequently a thicker mud cake was formed (figure 20), causing slower filtation process.

Figure 19. Mud cake deposited by nanoemulsion cleaning fluid

Figure 20. Mudcake deposited by non-nanoemulsion cleaning fluid

CONCLUSION

In conclusion, nanoemulsion offers promising technology for oil-based mud removal during cleaning stage. Nanoemulsion is more stable and it works efficiently at different operation condition; from ambient to HPHT condition. At same surfactant concentration, cleaning process using nanoemulsion results in better performance than cleaning with non-nanoemulsion. As a result, the amount of surfactant usage can be reduced through cleaning with nanoemulsion, therefore increasing its potential to be used in industry as replacement for non-nanoemulsion.

RECOMMENDATION AND SUGGESTION

Due to insufficient amount of nanoemulsion and non-nanoemulsion, this experiment can be further improved in the future by conducting cleaning efficiency test at different surfactant concentration; from 5 to 15 volume percent. From this experiment, further analysis can be done to compare the efficiency between nano and non-nanoemulsion at various concentrations. In addition, the temperature can also be manipulated using heating jacket, to see the effect on cleaning efficiency at various temperatures; from 60-250 degree Fahrenheit.

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