University of Stavanger

Faculty of Science and Technology

MASTER'S THESIS

Study program/ Specialization: Environmental Technology, Offshore Environmental Engineering	Spring semester, 2014 Open/Restricted access
Writer: Corinne Fredvang Smith	………………………………………… (Writer's signature)
Faculty supervisor: Torleiv Bilstad External supervisor(s): Stig O. Keller	
Thesis title: Destabilization and Separation of Drilling Mud by Utilizing Chemicals and Mechanical Equipment	
Credits (ECTS): 30	
Key words: - Drilling fluids and drilling fluid functions - Drilling fluid waste treatment - Chemical destabilization - Centrifugal separation - Retort analysis - Separation Efficiency, Oil-in-Water	Pages: 89 + enclosure: 9 Stavanger, 25.06/2014

Destabilization and Separation of Drilling Mud by Utilizing Chemicals and Mechanical Equipment

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25.06.2014

Environmental Technology, Offshore Environmental Engineering

University of Stavanger and Norwegian-Group AS

Acknowledgements

Abstract

The research of this thesis was aimed to test new chemical mixtures for drilling fluid waste treatment and optimize them for different type of oil-based drilling muds. The research included two steps: pre-screening and main experiment. Pre-screening included identification and selection of the most efficient chemical mixtures on various industrial oil-base mud samples, as well as definition of optimum centrifugation conditions. Main experiment included determination of the most efficient conditions such as temperature and dosage of the selected chemical mixtures in order to destabilize chosen drilling muds down to clear 3-phase condition. During main experiment, oil-based muds were made into lab slop-muds to be more representative as mud waste. First destabilization of drilling slop-mud was done by adding small dosage of the chemical mixture to the drilling mud; then mechanical separation was done by a lab centrifuge; expecting to get clear 3-phase separation (oil, water and solid phases).

Decrease of viscosity of the mud samples was done by increasing operating temperature in some of the samples. Lower viscosity had significant improvement on the separation process as it provided better conditions during centrifugation and higher water recovery. Centrifugation parameters such as g-force and retention time were also evaluated. The results indicated that increase on g-force had dramatic effect on higher water recovery percentage, while retention time did not proof to be a considerable parameter in this work. Dosage of the chemical mixtures was another main factor, providing satisfying results during the separation process. By increasing the concentration of chemical mixtures phase separation was enhanced after centrifugation.

The main experiment resulted in successful destabilization and clear 3-phase separation on different oil-base slop-muds by MudSplit001, MudSplit003 and Mudsplit005. Mudsplit001 and Mudsplit005 concluded to be most effective chemicals with respect to dosing. Representative samples, giving satisfying result at lowest possible dosing, were analyzed for separation efficiency. Oil- and solids phases were analyzed by retort analysis, giving fractions of contaminants in the phases. The retort analysis method provided proper understanding on the content of the different phases. GC- Fid analysis by Intertek Westlab on decanted water phases showed low content of oil in the samples.

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Abbreviations

CMC - Carboxymethyl Cellulose

HTHP - High Temperature High Pressure

NAF - Non-Aqueous drilling Fluids

NCS - Norwegian Continental Shelf

OBM - Oil-Based Mud

OWR - Oil-Water-Ratio

RCF - Relative Centrifugal Force

ROP - Rate of Penetration

RPM - Revolutions per Minute

SBM - Synthetic-Based Mud

WBM - Water-Based Mud

1   Introduction

Waste management and sustainable technologies have become one of the biggest milestones for companies involved in the oil and gas industries (Kirkness and Garrick, 2008). One of their challenges is discarding/treating large quantities of waste drilling fluids having high toxicity and complex composition. The main goal of these companies – "zero harmful discharge" can be reached by using environmental friendly chemicals and/or reusing and recycling the produced waste as much as possible in the system efficiently.

1.1   Drilling Wastes

Water based drilling fluid and drill cuttings from drilling with water-based mud can be discharged to sea, while oil-based muds (OBMs) need proper treatment and disposal of the hydrocarbon contaminated wastes generated (Paulsen et al., 2003). OBM waste is categorized as hazardous waste and cannot be discharged. Drill cuttings and other wastes from offshore drilling operations have been treated onshore for many years, in accordance with regulatory restrictions governing offshore disposal. Transporting the wastes from offshore locations to onshore treatment facilities is commonly referred to as "skip and ship". However, "skip and ship" of large tonnages of hydrocarbon contaminated cuttings and drilling waste give extensive environmental and safety implications for the drilling industry (Kirkness and Garrick, 2008). "Skip and ship" is also considered costly and not practical regarding logistics, storage etc. It often includes mud slops being stored at the rigs/platforms in tanks before pumped to the supply vessel tanks and shipped to shore as hazardous waste. Service companies normally provide temporary storage on the supply base before waste is handed over to hazardous waste collectors. The hazardous waste collectors normally mix and blend different waste streams with the target to separate it into 3 phases: oil, water and solids. Thereafter, some of the oil is sold to other locations, providing proper treatment; some of the contaminated slop water is shipped to Denmark or Germany for further treatment. Tanker bottom solids are sent to a cuttings treatment plant, which there are several of in Norway, operated by companies such as SAR-Group, Halliburton, Franzefos and TWMA, among others (Keller, 2014).

1.2 Cost Savings and Advantages (Issues)

Finding environmental friendly, economical and sustainable methods for treating hazardous waste from the oil industry is one of the top priorities in the last decade. Processing of the contaminated wastes offshore would limit many of the problems and challenges associated with "skip and ship", additionally improving safety, reduce environmental impact, costs and logistics (Kirkness and Garrick, 2008). Among possible ways of cost saving the following options are given:

- Save cost by freeing up rig space and reduce weight due to no need to storage of slop and cuttings. Save cost due to significant reduction in supply ship use. Serve more rigs per supply vessel.
- Save cost due to less downtime in drilling, related to bad weather and transshipment of cuttings skips.
- Save costs by reuse of the reclaimed oil.
- Reduce the amount of hazardous waste sent to shore.
- HSE cost savings related to:

  - Loading and unloading by crane to tankers. No long-term storage, avoiding H_2S production. By avoiding transshipment of slop to shore, people entering tanks for washing are avoided.

  - Less logistics, consequently less CO_2 emissions. The Operators have control on the waste being processes (Keller, 2014).

1.3 Norwegian-Group AS

Norwegian-Group AS vision is to provide waste management solutions to the oil and gas drilling industry, onshore and offshore treatment. The main long-term target for Norwegian-Group AS is *"to develop an all integrated compact and sustainable treatment solution, that can separate oil contaminated water (slop water) for discharge to sea (less than 1 ppm oil in water), reduce oil on cuttings (less than 1 %) so that cuttings can be discharged to seabed, and to destabilize contaminated oil-based mud (OBM) into 3 phases: oil, water and bottom sludge"* (Keller, 2014). Figure 1.1 gives an overview of suggested waste management solution.
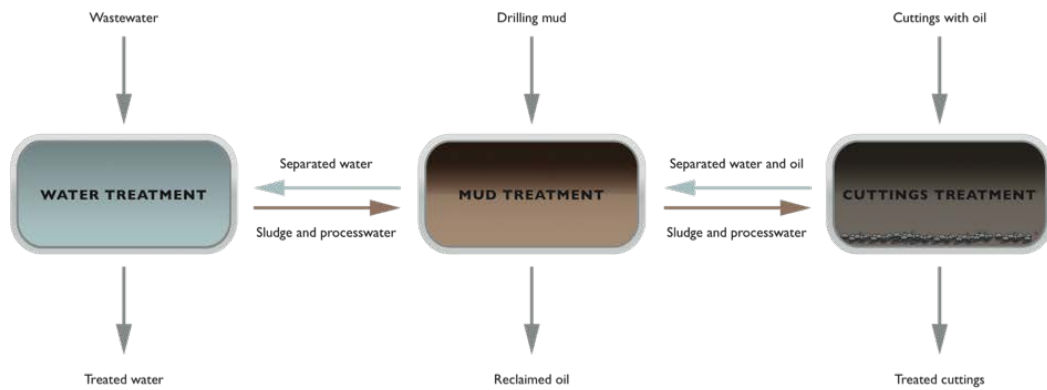
Figure 1.1 *Waste management solution, a combination of water-, mud- and cuttings treatment (Keller, 2014).*

A combination of water, drilling mud and cuttings treatment allows for water and cuttings to be discharged to sea and only oil to be sent to shore.

1.4 Target of the Project

The main target of this project is to test the process of destabilization and 3-phase separation (oil, water and solids) of different drilling muds with help of new chemical mixtures, as well as identify the best possible conditions for clear 3-phase separation. The study was performed to develop and optimize a method that allows for onsite separation of contaminated OBMs into three phases with less than 1.5 % oil-in-water. The experiment started with literature study, covering the drilling operation, separation equipment involved in treatment of drilling fluid, drilling fluids and rheology, clay and polymer chemistry and different chemicals for destabilization of OBM. The lab experiment was build up to test chemical mixtures, identified in the literature study, on actual OBM from different service companies, on waste mud taken from tanks at a hazardous waste collector, as well as on lab slop-mud[1].

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[1] Lab slop-mud is a mixture of synthetic seawater and OBM in a ratio of 1:1, being more representative as mud waste.

2 Drilling Fluids

Drilling fluid, often referred to as drilling mud, is a key requirement in oil and gas drilling operations. Drilling fluid is defined by The American Petroleum Institute (API), as "a circulating fluid used in rotary drilling to perform any or all of the various functions required in a drilling operation" (Irfan, 2014). Drilling fluids help providing safe and productive oil and gas wells and serve many important functions during drilling operations. The most important functions that drilling fluids serve are controlling formation pressures, removal of drill cuttings from the borehole, maintaining wellbore stability, cooling and lubrication of the drill bit, keep drill cuttings in suspension when drilling is ceased and controlling fluid loss to the formation. Drilling fluids also transmit hydraulic energy downhole to drill bit, tools, and help facilitate data logging and give information about the formation being drilled (Williamson, 2013).
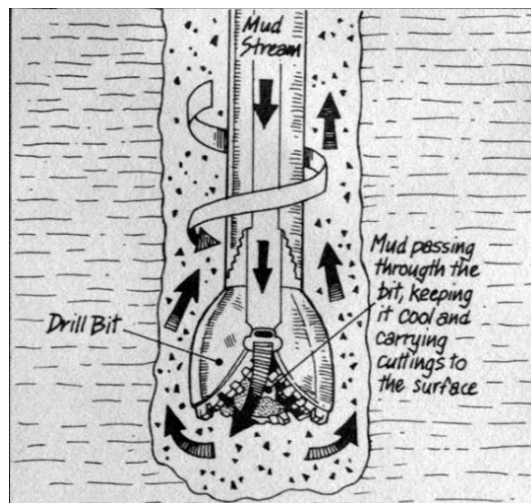
Figure 2.1 *Downhole view of drilling fluid circulation (Seadrill, 2013).*

2.1 Drilling Fluid Circulation System

Drilling fluid is pumped from mud surface tanks (mud pit) and downhole by use of powerful pumps. The mud flows inside the drill string on its way downhole to the drill bit where it exits from high speed jet nozzles to remove drilled solids, referred to as drill cuttings, away from the bit. Figure 2.1 illustrates downhole view of drilling fluid circulation. The mud is circulated back to the surface through annulus, with drill cuttings entrained in the fluid. An overview of the circulation system for drilling mud is shown in Figure 2.2.

The mud reaches the surface degraded by downhole conditions, dehydrated and loaded with formation solids and other unwanted components from the formation. Treatment and processing are necessary for the mud to maintain its properties. Solids are removed at the surface solids control system and new additives are blend in to ensure the mud to meet specifications (Williamson, 2013). After solids control system, the mud flows back into the mud pits where it can continue its circulation process.
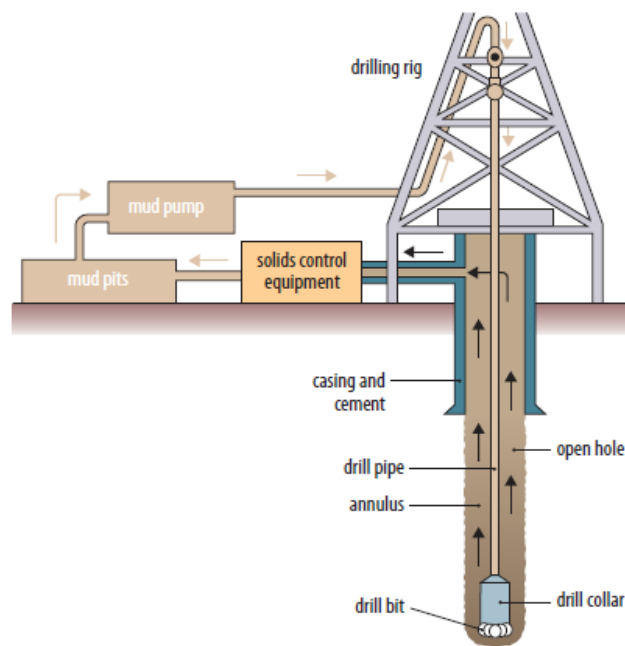
Figure 2.2 *Simplified overview of drilling fluid circulating system of a rig and well (IPIECA/OGP, 2009).*

2.2 Drilling Fluid Surface Treatment

Drilling fluid that returns to the rig surface is processed and treated by use of different mechanical equipment to allow efficient and practical reuse of drilling fluids. The mechanical separation equipment varies from rig to rig, and technologies such as shale shakers, settling pits, degasser, cyclones, centrifuges, cuttings dryers, etc. are normally included. Brief descriptions of some of the most common technologies are given in the subsequent chapters.

2.2.1 Shale Shaker

Shale shaker is a general term for a vibrating device used to screen solids from a circulating drilling fluid. Shale shakers are the most important solids-removal equipment. Drilling fluid flows over a screen, particles larger than the openings in the screen are removed while the liquid mud and fine solids pass through (ASME, 2005) (see Figure 2.3).
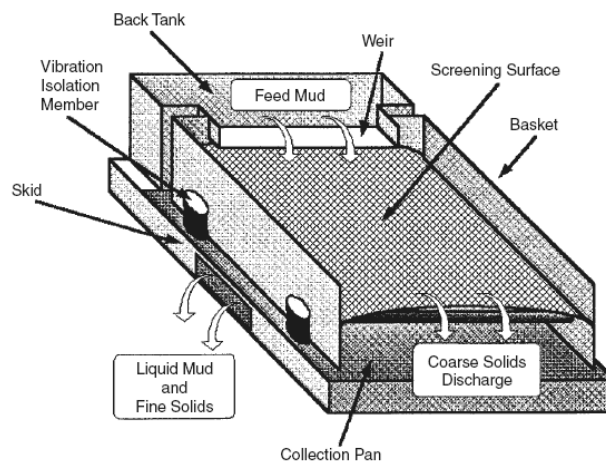
Figure 2.3 *Shale shaker (GN Solids Control, 2010a).*

After shale shaker, the underflow (liquid mud and fine solids) go through additional stages using desanders, desilters, centrifuges, among others, to remove finer solids down to 1 um range (Fink, 2003).

2.2.2 Degasser

Degasser removes entrained gas from the drilling fluid preventing problems related to gas in the drilling mud. The degasser is a tank in which a vacuum and/or spray removes entrained gas from the mud system (ASME, 2005).

2.2.3 Hydrocyclones

Hydrocyclones use centrifugal force to separate suspended solids, 15- to 80-micron range, from drilling fluid. The action inside the hydroyclone can multiply gravitational force by as much as 200 times. Fluid enters tangentially and spins inside the cone. Heavier solids settle to the walls of the cone and move downward to the discharge point. The spinning fluid travels partway down the cone and back up to exit at the top of the cone. Generally, a desander and a desilter are available as part of a rig's hydrocyclone equipment (ASME, 2005). Figure 2.4 shows the principle of operation for hydrocyclones.
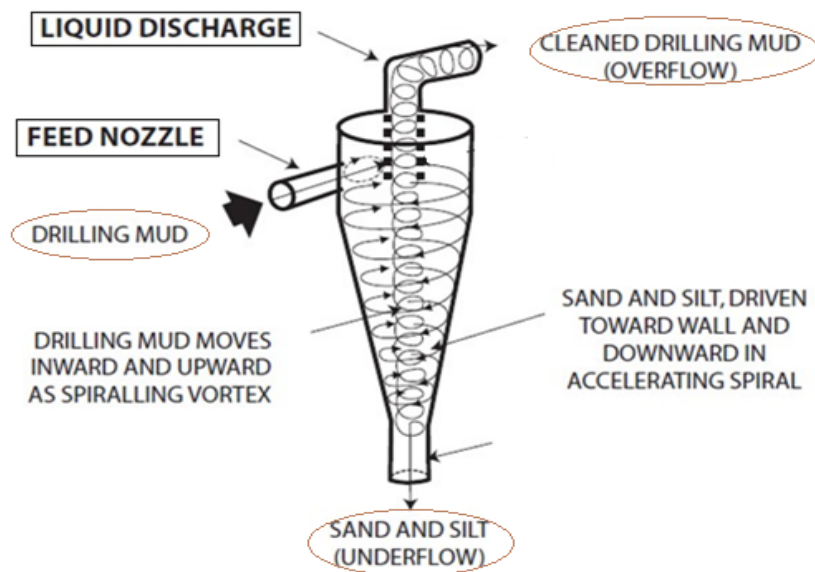
Figure 2.4 *Hydrocyclone schematic, principle of operation (GN Solids Control, 2010b).*

2.2.4 Centrifuge

A centrifugal separator uses high-speed mechanical rotation for separation of materials of different masses. Centrifuges can provide an artificial centrifugal force of 2-5000 g (ASME, 2005). Conventional centrifuges are either decanter centrifuges or disc-stack centrifuges. Disc-stack centrifuge are mainly for separation of oil and water, while decanter centrifuges are primarily for separation of solids from liquid slurries (Statoil, 2013).

In a decanter centrifuge, feed enters the bowl through a concentric tube. The liquid phase migrates to the larger radius end of the bowl where it is discharged continuously, while the solids are continuously transported to the other end of the bowl by a helical screw conveyor (see Figure 2.5) (Alfa Laval, 2013).
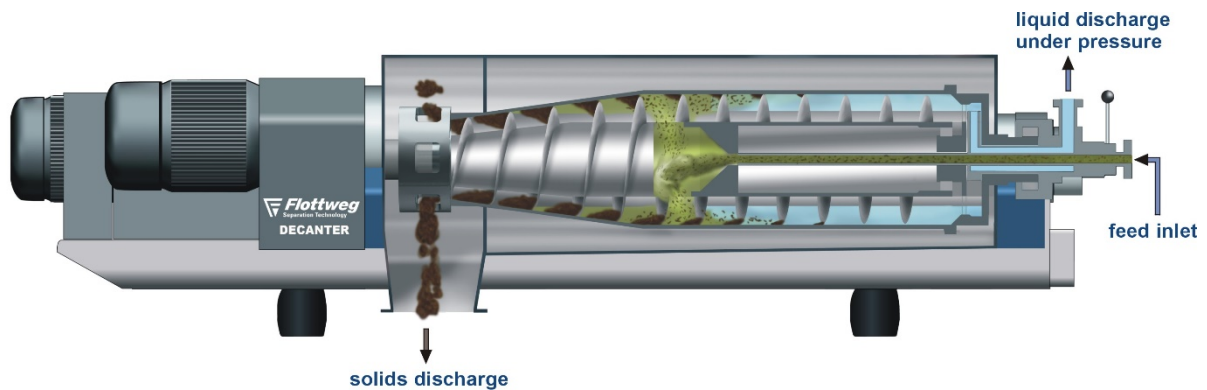
Figure 2.5 *Decanter centrifuge, principle of operation (Flottweg, 2014).*

2.2.4.1 Stoke's Law

Stoke's law defines the relationship between parameters that control the settling velocity of particles in viscous liquids, such as drilling mud. This is relevant in equipment as hydrocyclones and centrifuges. Hydrocyclones and centrifuges increase settling rates by application of increased centrifugal force, equivalent to higher gravity force (ASME, 2005). Stoke's law:

$$V_s = \frac{d^2 \, g \, (p_S - p_L)}{18\mu}$$

V_s	Settling velocity, m/s
d	Particle diameter
g	Acceleration (gravity 9.81 m/s^2 for settling, or apparatus angular velocity)
$\boldsymbol{p_S}$	Density of solids (barite etc.) kg/m^3
$\boldsymbol{p_L}$	Density liquid phase, kg/m^3
$\boldsymbol{\mu}$	Viscosity of the mud, Ns/m^2 = kg/ms

For a particle which is forced to spin in a circular path, it can be shown that the gravity acting on that particle is proportional to the square of the angular velocity:

$$g = (\omega^2 r)$$

Where r is the radius of the circular path and ω is the angular velocity. For centrifugal separation Stoke's law becomes:

$$V_s = \frac{d^2 \, \omega^2 r \, (p_S - p_L)}{18\mu}$$

This gives a tremendous potential for increasing the terminal settling velocity, and is utilized in all types of centrifugal separation (Statoil, 2013).

Separation can be increased by understanding the factors influencing the process. The parameters in Stoke's law can be altered for optimized separation. Reducing the viscosity of the drilling mud, increasing gravitational forces, increasing the particle size, or increasing the effective particle size with flocculation or coagulation, increased separation can be achieved. High density difference is beneficial for separation, and heavier particles settle faster.

2.2.5 MudCube

MudCube is a new solids control equipment that can replaces traditional shale shakers with a single enclosed system (see Figure 2.6). *MudCube* operates by the use of vacuum and conveyer belt. The system can also eliminate degasser, settling pits and cuttings dryers among others (Cubility, 2011). To date, the MudCube has been introduced to drilling sites in the North Sea and in the U.S. Marcellus shale play (Cubility, 2014).

Figure 2.6 *The MudCube (Cubility, 2011).*

2.3 Drilling Fluid Classification

Drilling fluids are commonly classified by their continuous phase, or base fluid. Main categories are aqueous drilling fluid and non-aqueous drilling fluid. Water is the continuous phase of aqueous drilling fluid, generally referred to as water-based mud (WBM). Non-aqueous drilling fluid includes synthetic-based mud (SBM) and oil-based mud (OBM): containing mineral oil or diesel as base fluid (Williamson, 2013). Figure 2.7 gives an overview of drilling fluid classification.
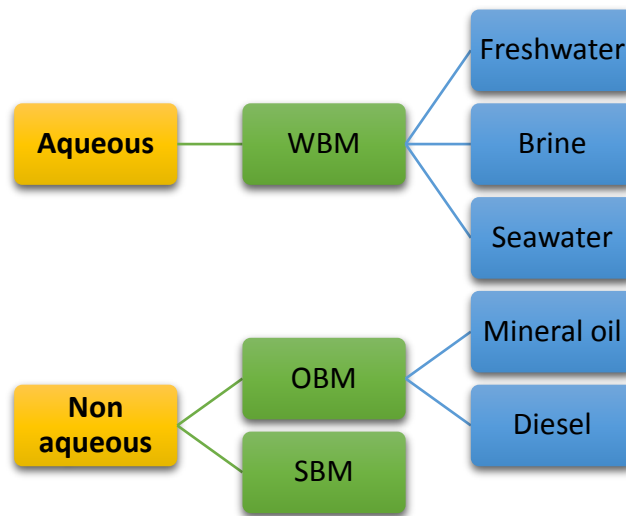
Figure 2.7 *Drilling fluid classification (Williamson, 2013).*

Drilling fluid, or drilling mud, also contains significant amounts of suspended solids in the base-fluids. In addition to base fluids and suspended solids, emulsified water or oil, and chemical additives are present to modify properties of the drilling fluids (Caenn et al., 2011).

2.3.1 Aqueous Drilling Fluid

Aqueous drilling fluid, or water-based mud, has a continuous phase composed of freshwater, seawater or brine. Oil may also be emulsified in the water base. Various solids and chemicals are added to the fluid, necessary to achieve the desired properties of the drilling mud (IPIECA/OGP, 2009). General composition of WBM is shown in Figure 2.8.
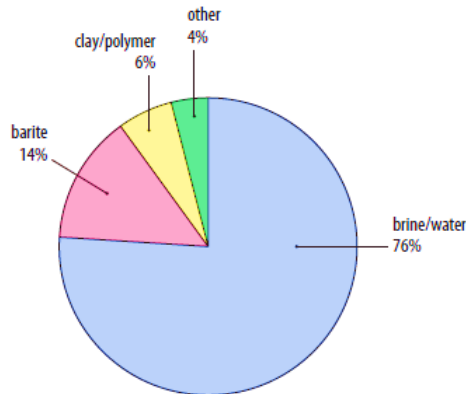
Figure 2.8 *Composition of water based mud – chemical components %, by weight (IPIECA/OGP, 2009).*

2.3.2 Non-Aqueous Drilling Fluid

Non-aqueous drilling fluids are mostly emulsions. The continuous or external phase is the non-aqueous base fluid and the internal phase is water. Solids and other chemical additives are present, necessary to achieve the desired properties of the drilling fluid (JWSL, 2009). General composition of non-aqueous drilling fluids is shown in Figure2.9.
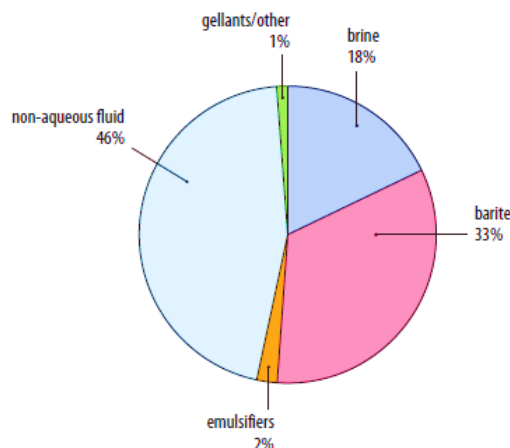
Figure 2.9 *Non-aqueous drilling fluids – chemical components %, by weight (IPIECA/OGP, 2009).*

2.3.2.1 Oil-Based Mud

Oil-based mud has a continuous phase being diesel oil or mineral oil (Garrett, 2014g). Water or brine is dispersed throughout the oil and an emulsifier is added for stabilizing the emulsion (Bloys et al., 1994). Oil-based drilling fluids normally have an oil/water ratio (OWR[2]) in the range of 60/40 to 90/10 (McCosh et al., 2009). Oil-based mud also contains solid particles that are suspended throughout the base fluid. The solids and other chemical additives are used to achieve the desired properties of drilling fluids and for stabilizing the emulsion system. However, there are also oil-based mud systems being 95-99 % oil (Garrett, 2014f).

2.3.2.2 Synthetic-Base Drilling Fluid

Synthetic-base drilling fluids or synthetic-base muds (SBM) have base fluids which are synthetic hydrocarbons. These fluids are reaction products of specific, purified chemical feedstock materials (e.g. ethylene) instead of being distilled or refined from petroleum (OGP, 2003). They are generally organic liquids, and the most common are ethers, esters, polymerised olefins and synthetic paraffins (ASME, 2005). SBM are analogous to OBM and therefore they are often called "pseudo[3] oil-base mud" (Garrett, 2014f).

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[2] OWR = Ratio of the volume percent oil to the volume percent water in an oil mud, where each is a percent of the total liquid in the mud (Garrett, 2014).

[3] *From Greek, "lying, false".*

2.3.3 Emulsions

An emulsion normally consists of two immiscible liquids, with one of the liquids dispersed in the other (dispersed- and continuous phase, respectively) (Schramm, 1992). There can also be more than two phases, where other phases of liquid, liquid crystalline or solid state are present. An emulsifier is needed to achieve stability between the immiscible liquids; it works by reducing surface tension (interfacial tension) between the liquids. The emulsifier is important for the emulsion, not only for the formation but also for the stability over time. Emulsions can be classified based on the structure of the system (Schramm, 1992):

- Nature of the disperse and continuous phase
    - oil-in-water (O/W)
    - water-in-oil (W/O)
    - oil-in-oil (O/O)
- Microemulsions
- Macroemulsions
- Double and multiple emulsions
- Mixed emulsions
- Bilayer droplets

Emulsions of O/W and W/O are illustrated in Figure 2.10.
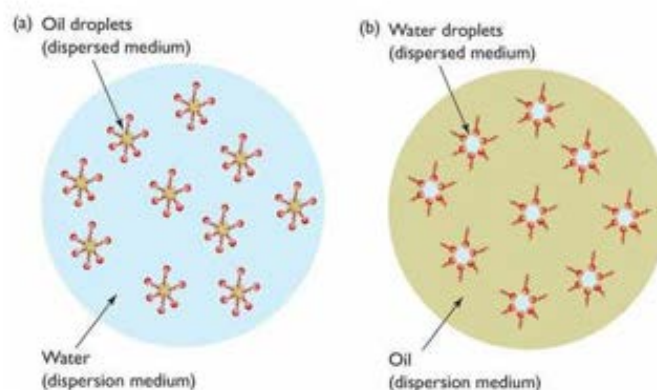
Figure 2.10 *Oil-in-water emulsion (left) and water-in-oil emulsion (right) (Pack, 2011).*

Water-based drilling mud (WBM) is an oil-in-water emulsion, where oil is dispersed as fine droplets throughout the water base fluid, while oil-based mud (OBM) is a water-in-oil emulsion, also called invert emulsion (Garrett, 2014d). In addition, solids are present in the drilling fluids.

Oil, water and solids forms a special type of emulsions, called Pickering emulsions. These emulsions are stabilized by fine particles. The particles form a close-packed structure at the oil-water interface that provides a barrier to coalescence (see Figure 2.11) (Schramm, 1992). Clay minerals are known to produce quite stable Pickering emulsions (Mikkelsen, 2014). Starch and Carboxymethyl Cellulose (CMC), common additives to drilling mud, also aid emulsion mud stability.
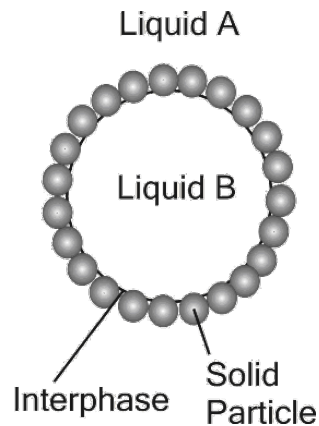
Figure 2.11 *Pickering emulsions, e.g. by colloidal solids (clay) in drilling mud (Vleugels, 2010).*

2.3.4 Development and Usage

In the late 1980s synthetic-based mud (SBM) was introduced to replace OBM (PetroWiki, 2013). SBMs offered strong drilling performance like OBMs, withstanding high temperatures and more suited for deep water drilling environments, but were closer to WBMs in environmental impact by having lower toxicity, faster biodegradability and lower bioaccumulation potential (Harto, 2001). Synthetic fluids have been used as an alternative to oil-base fluids, however, they are not much in use at the Norwegian Continental Shelf (NCS) today. Water-based drilling fluid is the most commonly used drilling fluid at the NCS (NEA, 2013). Oil-based drilling fluid is used only where properties of WBF are insufficient, such as in deeper parts of a well. Non-aqueous drilling fluids have many advantages compared to water-based drilling fluid, they provide excellent borehole control, thermal stability, lubricity and penetration rates. In addition, they are more used for HTHP (high temperature high pressure) wells (Williamson, 2013).

2.4 Basic Properties & Drilling Fluid Additives

Drilling fluids have certain basic properties, physical and chemical, to perform all the important functions during drilling. These are properties such as density, rheology (viscosity and gel strength), fluid-loss control, solids content, pH and shale inhibition (Bloys et al., 1994). Different chemical additives are used to achieve the desired properties (see Table 2.1). Many different additives are available, depending on the type of drilling fluid.

Table 2.1 *Drilling fluid properties and associated common additives (IPIECA/OGP, 2009).*

Property	Additive(s)	NAF	WBM
Density	Weighting material	*Barite, $CaCO_3$, $FeCO_3$, Hematite, Ilmenite*	*Barite, $CaCO_3$, $FeCO_3$, Hematite, Ilmenite*
Viscosity and gel strength	Viscosifiers and gelling material: Colloids	*Organophilic[4] clay* *Polymers*	*Bentonite clay, or other clays* *Polymers*
	- thinner/deflocculant	*Organic sulfonates*	*Lignosulfonates, lignites, anionic polymers, tannins, etc.*
Fluid-loss	Fluid-loss materials (also called filtrate-reducing materials)	*Polymers, clay*	*Polymers, clay*
Shale inhibition	Salts, shale inhibition chemicals	*$CaCl_2$, formate.*	*Salts, glycols, etc.*
pH	Acid/base	-	*NaOH, KOH, $Ca(OH)_2$, Citric acid, $NaHCO_3$*
Other	Surfactants	*Emulsifiers Wetting agents*	*Emulsifier Wetting agents Lubricants*
	Scavengers of O_2, CO_2, H_2S		
	Corrosion inhibitors	-	
	Biocides		

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[4] Organophilic coating, for clay to become dispersible in oil-base muds (Garrett, 2014 h).

2.4.1 Density

Drilling fluid density, or mud weight, is one of the most important drilling fluid properties. It controls formation pressure and is important in providing well control (Bloys et al., 1994). The mud column in the well exerts a hydrostatic pressure (P_s) that balances pore pressure (the pressure exerted by the fluids in the pores of the formation), is illustrated in Figure 2.12 Hydrostatic pressure (P_s) is given by $\rho * g * h$, where ρ represents density, g is gravitational acceleration and h is the height of mud column. Thus, drilling fluid controls formation pressure by its density.
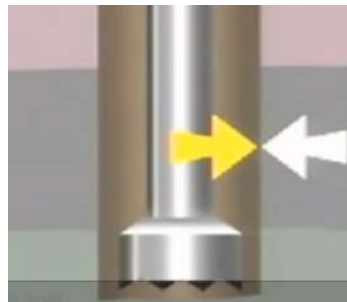
Figure 2.12 *Hydrostatic pressure exerted by the mud column (yellow) controls the pore pressure exerted by the formation fluids (white) (William, 2012).*

Density is defined as weight per unit volume, or compared to the weight of an equal volume of water, as specific gravity (SG). Mud density varies according to the formation pressure in the wellbore. A mud density of 1.08 kg/dm^3 may be required at normal pressure conditions, and an extremely high pore pressure may need a density of 2.16 kg/dm^3 or higher (Garrett, 2014e).

2.4.1.1 Weighting Material

Weighting material is added to drilling mud to provide sufficient hydrostatic pressure. Barite is the most used weighting material (Bloys et al., 1994), used in both water-based mud and non-aqueous mud. Other common weighting materials are hematite and ilmenite, calcium carbonate and iron carbonate (see Table 2.2) (IPIECA/OGP, 2009). Due to environmental reasons barite is often replaced by hematite or ilmenite (Skaugen, 1997).

Table 2.2 *Specific gravity of common weighting materials used in drilling mud.*

Weighting material	Specific gravity
Calcium carbonate ($CaCO_3$)	2.70
Iron carbonate ($FeCO_3$)	3.90
Barite ($BaSO_4$)	4.20 – 4.40
Ilmenite ($FeTiO_3$)	4.50
Hematite (Fe_2O_3)	5.26

2.4.2 Solids Content

Desired solids are added to drilling fluids to achieve properties such as density, rheology, and fluid-loss control (Bloys et al., 1994). Included are high gravity solids such as weighting materials, and colloidal solids such as mineral clays and polymers. Drilled solids, rock fragments from the formation, are also added continuously to the drilling fluid during drilling.

2.4.2.1 Silt

A large proportion of solids in drilling muds fall in the silt size range of 4 -74 μm in diameter (ASME, 2005). These particles are either natural silts from the formation, or larger particles comminuted by the bit or from barite added as weighting material. Particles in this size fraction are commonly called the inert solids. However, high enough concentrations makes the inert solids influence the viscous properties of the mud (Caenn et al., 2011).

2.4.2.2 Colloids

The colloidal size range is in general defined as less than two microns (ASME, 2005). The ability of drilling fluids to perform important functions and exhibit desirable properties is dependent on the colloidal nature of the materials used in the mud formulation (Lummus and Azar, 1986). Colloids affect mud flow properties such as viscosity, gel strength and fluid-loss control. The colloids in drilling mud usually constitute a small proportion of the total solids, but they have a relatively high influence on mud properties because of their high degree of activity. Colloidal activity depends on surface phenomena: specific surface and the surface potential (Caenn et al., 2011). With the extremely small particle size of colloids a large amount of surface area is exposed for a small amount of mass (specific surface). The surface potential varies with atomic structure. Molecules in the surface layer are not in electrostatic balance, so the surface carries an electrostatic charge. Colloids include a wide variety of materials that can remain suspended in another medium due to the extremely small particle sizes and unusual surface forces (Lummus and Azar, 1986).

Examples of colloids in drilling mud are *clay minerals* (such as Bentonite clay) and *organic polymers* (such as starch, Carboxy Celluloses, natural or modified gums, and polyacrylamide derivatives). These polymers are macromolecules or long-chain molecules, whose size give them colloidal properties (Caenn et al., 2011).

2.4.2.2.1 Clay

Clays fall within in the colloidal size range with maximum particle size of two micrometers (Caenn et al., 2011). Clay provides the colloidal base of nearly all aqueous muds, and is also used in oil-based muds (organophilic). Bentonite clay (principally the clay mineral montmorillonite) is the colloid of major interest in mud chemistry (Lummus and Azar, 1986). In addition to bentonite, formation clays will be added naturally to drilling fluids when drilling in shale rich formations, increasing viscosity of the drilling fluid.

Clay Structure

Clay minerals are hydrous aluminum silicates of a layer-type lattice structure (see Figure 2.13), with magnesium, iron, and potassium located either between the layers or substituted within the lattice.
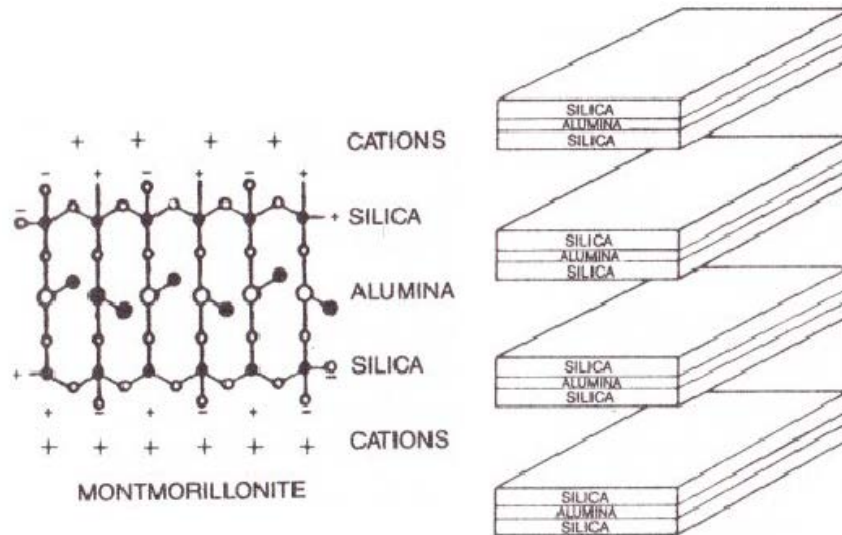
Figure 2.13 *Structure of Montmorillonite. Layers of silica and alumina forms a plate (Lummus and Azar, 1986).*

Bentonite clay is composed of flat, plate-like particles with two "different surfaces"; planar- and edge- surface. The face or planar surface is negatively charged, and the edge surface is positively charged (surface potential). Bentonitic clays also have cations surrounding the clay molecule. The combination of these positively charged cations and negative and positive charges associated with the clay structure make up the electrical field around the clay particles, determining how they interact. Bentonite has more planar surface exposed, with more negative charges, and consequently the electrical field surrounding bentonite is predominately negative. This negatively charged field causes a net particle-repelling effect (Lummus and Azar, 1986).

Cation Exchange Capacity

To neutralize the negative charges in the basal plane cations are adsorbed to the clay surface. In presence of water the adsorbed cations can exchange with cations of another species in the water, they are therefore known as *exchangeable cations*. The total amount of cations adsorbed, expressed in milliequivalents per hundred grams of dry clay, is called the cation exchange capacity (CEC). The CEC of clay and the species of cations in the exchange positions are a good indication of the colloidal activity of the clay. A clay such as montmorillonite, that has a high cation exchange capacity, swells greatly and forms viscous suspensions at low concentrations of clay (Caenn et al., 2011).

Clay Particle Association in Drilling Mud

Clay interaction and association in solution is important to drilling mud technology as it affects important properties such as viscosity, yield and fluid-loss of the mud. The mechanisms are dispersion, flocculation, deflocculation and aggregation (see Figure 2.14) (Strand, 2001). Whether or not drilling mud is in one of these conditions depends on mechanical forces, but mostly on the electrical interaction between clay crystals in the mud. The electrostatic interactions between the clay crystals are in turn dependent on the pH and electrolyte concentration of the solution (Caenn et al., 2011).
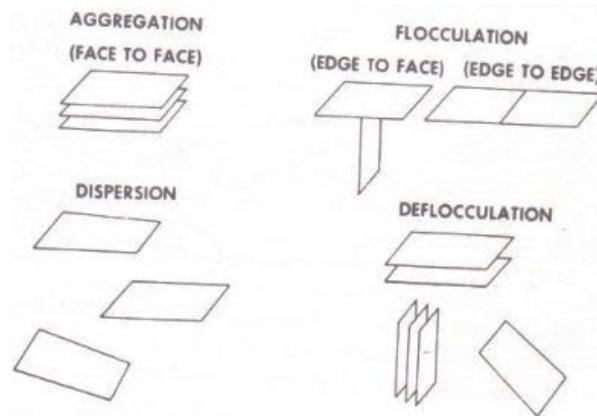
Figure 2.14 *Clay associations; Aggregation, dispersion, flocculation and deflocculation (Lummus and Azar, 1986). Each plate resemble three layers (silica, alumina).*

Dispersed State

Dispersed state occurs when the clay platelets separate into individual platelets. The clay platelets break apart and disperse into the water due to loss of attractive forces as water forces the platelets farther apart (Garrett, 2014b).

Aggregated System

Aggregated system occurs when clay platelets are stacked parallel to each other. In aggregated state the platelets are bind together forming aggregates - groups or clusters of particles in a fluid. As an consequence, viscosity and gel strength of the fluid decrease (ASME, 2005). Aggregation of dispersed clay is a result of ionic or thermal conditions. By altering the hydrational layer around clay platelets it is possible to remove the deflocculant from positive edge charges and allows platelets to assume a face-to-face structure. Alum,

$Al_2(SO_4)_3$ – a common inorganic coagulant, and also polymers, can cause colloidal particles to aggregate, allowing easier separation (Garrett, 2014b).

Unhydrated clay exists in aggregated state. When the clay contacts water, the nature of the cations holding the clay platelets together may or may not allow dispersion to take place. Aggregated or dispersed clays can undergo *flocculation* or *deflocculation*.

Flocculation

Flocculation occurs when there are net attractive forces between the clay platelets. The particles can associate to form loose structures. Individual clay particles, or aggregates, bind together edge against edge, or surface to edge (Strand, 2001). When flocculated, particles are often large enough to separate and settle out of solution. Several mechanisms exist by which flocculation can occur. It is generally caused by changes in electrolyte concentration, temperature and solids crowding (Baker Hughes, 2006). High pH and addition of a flocculant can also cause clay flocculation. Flocculation of suspended clay particles in drilling mud also begins when mechanical shearing stops, and platelets previously dispersed come together due to the attractive force of surface charges on the platelets (Garrett, 2014b).

Deflocculation

Deflocculation is the reverse of the flocculation process. Deflocculation occurs when there are netto repulsive forces between the individual clay particles. This way the clay particles remain independent and unassociated with adjacent particles (Garrett, 2014b). This is normally achieved by creating conditions in which the particles carry the same charges, which is usually negative. Deflocculation can also be achieved by adding a deflocculant, a temperature stabilizer or a dilution fluid (Baker Hughes, 2006). Chemical deflocculants are thought to adsorb on edges of clay, and thereby neutralizing positive edge charges and allowing the clay platelets to separate (Caenn et al., 2011).

Well-functioning bentonite mud is dispersed and deflocculated. Alkaline pH in drilling mud ensure net negative charge of clay particles (also the edge charges) in order to keep the attractive electrostatic forces at a minimum (Strand, 2001). Table 2.3 gives an overview of effect of clay on mud properties.

Table 2.3 *Effect of clays in drilling fluid systems (Strand, 2001).*

	Viscosity	**Yield**	**Fluid-loss**
Dispersed	Normal*	Normal	Normal
Flocculated	High	High	High
Deflocculated	Normal	Low	Low
Aggregated	Low	Low	Low

*Viscosity of a dispersed system increases as more and more clay platelets are being dispersed.

Knowledge on clay chemistry is important in maintaining colloidal stability providing a well-functioning mud, and is also useful for destabilizing and separating the colloids in mud.

2.4.2.2.2 Polymers

Polymers are long chains, macromolecules, made up of repeating units, monomers. Polymers added to drilling mud exercise a relatively high influence on mud properties even though they are present in small proportions. The polymers discussed here are organic colloids (Caenn et al., 2011).

Polymers serve many functions as drilling fluid additives. Molecular size (weight) influences how a specific polymer performs in a given type of mud. A small polymer may be a deflocculant, whereas a large polymer of the same type may be a flocculant; some are viscosifiers and others are fluid-loss control additives while others are multifunctional (Garrett, 2014i). Polymers can be positive, negative or neutrally charged, providing flexibility in performance and properties. In solution, polymer chains can create networks, giving complex viscosity behavior. The polymers form links between dispersed clay platelets, increasing viscosity (Caenn et al., 2011).

Most polymers added to muds are synthetic, made by modifying natural polymers (e.g. xanthan gum, guar gum and starch). Examples of synthetic or modified natural polymers are Carboxymethyl Cellulose (CMC), hydropropyl starch, lignosulfonate, polyacrylates, polyacrylamides and polyalphaolefins (Garrett, 2014i).

2.4.3 Rheology, Viscosity and Gel Strength

Rheology is the study of flow and deformation of fluids. There are several rheology values, but gel strength and viscosity are in the focus of this research. These values are important for drilling fluid's flow properties and the success of a drilling operation (Caenn et al., 2011).

2.4.3.1 Viscosity

Viscosity is defined as a property of fluids that indicates their resistance to flow (Garrett, 2014j). Viscosity is an important property for drilling fluids, for the hole cleaning capability. As the drill bit rotates and cut into the surface during drilling, drill cuttings are produced. If not removed, the cuttings pile up around the bit and can cause stop in drilling. Drilling fluids are designed highly viscous to provide sufficient carrying capacity of drill cuttings. The drill solids together with suspended weighting materials are entrained in the highly viscous drilling fluid and further transported to the surface for solids removal (Bloys et al., 1994). However, too high viscosity of the drilling fluid will cause problems with friction. Viscosity is commonly measured in Poise (P), corresponding to Pa*s (equivalent to Ns/m^2) in SI-units (Skaugen, 1997).

2.4.3.2 Gel Strength

Another important function of drilling fluids is to suspend drill solids and weighting material. This is important in case of stop in circulation, preventing drilled solids and weighting material falling downhole and piling up around the drill bit. The ability of the drilling fluid to suspend drilled solids and weighting materials depends on the gel strength (Skaugen, 1997).

The gel structure of some muds, fresh-water clay muds, increases with time after agitation has ceased. By resuming agitation viscosity decreases and gel structure is broken up until an equilibrium viscosity is achieved (Caenn et al., 2011).

2.4.3.2.1 Viscosity Regulators and Gelling Materials

To ensure sufficient carrying capacity of drill cuttings and sufficient gel strength, viscosity regulators and gelling material are added to the drilling mud (Garrett, 2014j).

Viscosity is mostly dependent on the type of mud and its solids content (Bloys et al., 1994). Any increase in solid content in drilling mud as barite, drill solid, clay, polymers, etc will result in higher plastic viscosity. Normally, higher mud weight gives higher viscosity. However, increasing viscosity without mud weight change means an increase in ultra-fine drill solid content in the mud system. However, main viscosity regulators are colloids, such as clay and polymers. Commercially mined clay minerals are commonly added to both aqueous- and non-aqueous drilling fluids for viscosity control. Bentonite clays are typically used viscosifiers for water-based drilling fluids. Other clays can also be used but they are not as effective as Bentonite (Skaugen, 1997). Alternative additives for viscosity control in aqueous drilling fluids include organic colloids such as polymers, biopolymers, cellulose polymers, xanthan gum (polysaccharide) and starch (Bloys et al., 1994). Alternative viscosity regulators for non-aqueous drilling fluids are fatty acids and synthetic polymers. However, in NAF viscosity can also be controlled by altering the oil water ratio (OWR). Emulsified water in oil base drilling fluid will act like a solid and increase the viscosity dramatically (Caenn et al., 2011).

Gel strength depends on the presence of colloidal clays. In water-based clay muds, clay platelets align themselves by bringing their positively charged edges towards their negatively charged basal surfaces (see Figure 2.15) and create a gel structure.
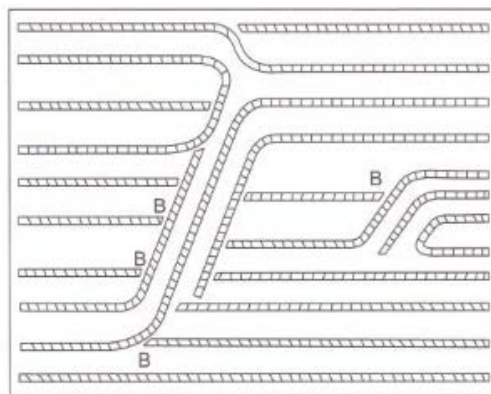
Figure 2.15 *Schematic representation of a clay gel structure. B: edge-to-surface bonds (Caenn et al., 2011).*

2.4.3.2.2 Deflocculants

Deflocculants are thinning agents used for reducing viscosity or preventing flocculation of water-based drilling fluids, clay muds (Garrett, 2014c). Deflocculants are mostly low-molecular weight anionic polymers that interact with the charges on clay particles (neutralize positive charges on clay edges). Thinners are added to the mud to reduce its resistance to flow and to stifle gel development. They are typically acrylates, polyphosphates, lignitic materials, lignosulfnates or synthetic polymers (Garrett, 2014c). There are seldom thinners in NAF.

2.4.4 pH

Mud properties depend on pH, normally pH more than 9 is required (Baker Hughes, 2006). High pH helps reduce corrosion, bacterial growth, as well as neutralizes possible H_2S gas from the formation. Organic dispersants and filtration control agents generally achieve maximum effectiveness in an alkaline environment. Thermal stability of lignosulfonate systems may be improved at a pH of 10.0 or above (Baker Hughes, 2006). Basic additives to achieve alkaline environment in drilling mud are NaOH, KOH or $Ca(OH)_2$. Too high pH is regulated by adding citric acid or $NaHCO_3$ (IPIECA/OGP, 2009).

2.4.5 Shale Inhibition

Clays, as claystones, shales and intermixed with sands and sandstones make up the largest percentage of minerals drilled while exploring for oil and gas (Garrett, 2014a). Borehole instability can easily occur during drilling in shale rich formations with the use of water-based mud. Shales are easily hydrated and can cause the borehole to collapse (see Figure 2.16).
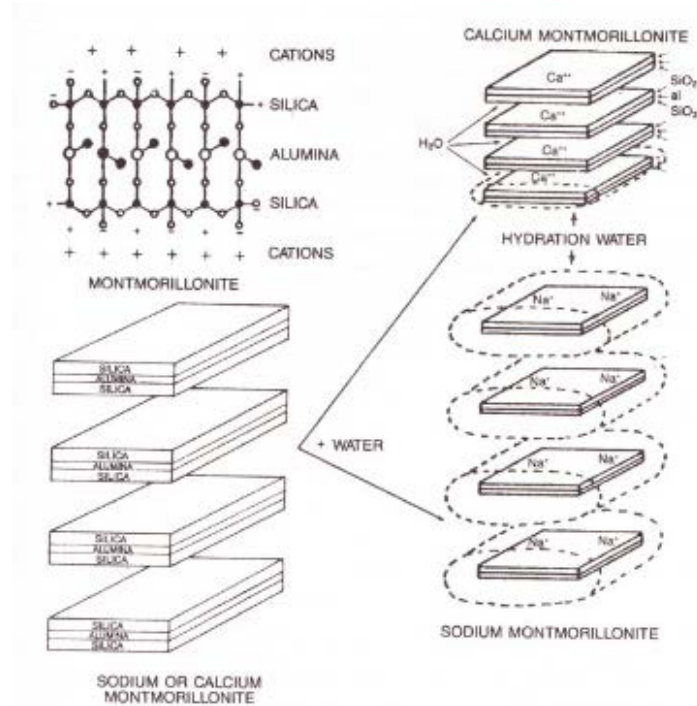
Figure 2.16 *Clay-water interaction: clay packets in shales absorb water and swell (Lummus and Azar, 1986).*

2.4.5.1 Shale Inhibition Additives

Salts are added to water-based mud to prevent interaction with shale and problems with swelling. Salts dissolve into cations and anions that prevent clay to dissolve in water (Skaugen, 1997). Shale inhibitors may also be used. Shale inhibitors prevent water uptake by clays, so swelling can be reduced despite usage of WBM (M-I Swaco, 2014b).

Problems with swelling are avoided by using non-aqueous drilling fluid due to neutral oil molecules that do not interact with clays in shale. However, due to the internal water phase in OBM emulsions $CaCl_2$ or formate ($HCOO^-$) are added for shale inhibition (Skaugen, 1997).

2.4.6 Fluid-Loss Control

Fluid-loss can be a problem when drilling in porous formations. As the hydrostatic pressure of the mud column exceeds the formation pressure, fluid is lost to the formation. To prevent fluid-loss the drilling fluid is designed to form a form a low-permeability filter cake, also called mud cake. The mud cake seals between the formation and the wellbore thereby preventing fluid-loss (see Figure 2.17), provides wellbore stability and prevents the well from caving in (Skaugen, 1997).
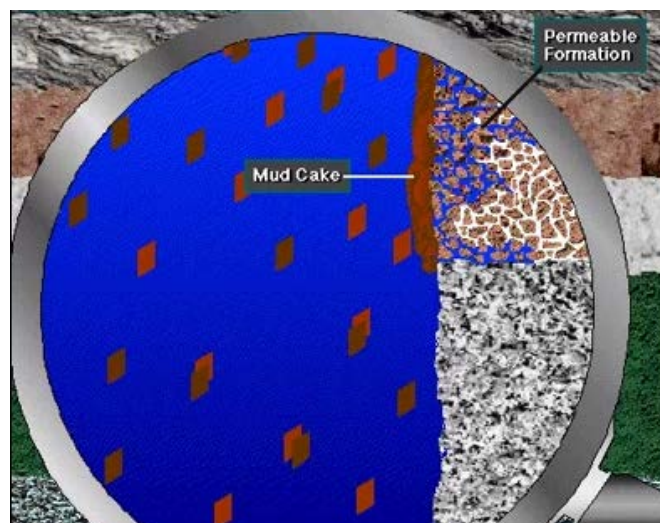
Figure 2.17 *Illustration of mud cake build-up on the formation wall (Rachain and Coleman, 2014).*

2.4.6.1 Fluid-Loss Material

Common fluid-loss materials are polymers, added to NAFs and WBMs. Examples are modified lignites (IPIECA/OGP, 2009). Bacteria cultures may also be added, they can form natural polymers and prevent fluid-loss (Fink, 2003). For WBM, polymers such as celluloses (e.g. CMC), and starch (OGP, 2003), or synthetic polymers can be used. Addition of clays in drilling fluids also provides fluid loss control.

2.4.7 Specialized Chemical Additives

Other specialized chemical additives such as surfactants in various forms, corrosion inhibitors, O_2/CO_2/H_2S scavengers and biocides are commonly added to drilling fluids to provide a safe and productive oil well (IPIECA/OGP, 2009). Surfactants serve many purposes in drilling fluids and are explained more in the text below.

2.4.7.1 Surfactants

Surface active materials tend to concentrate at an interface of an emulsion or a solid/liquid interface. Surfactants are added to drilling fluids in forms of emulsifiers, foamers and defoamers, wetting agents, lubricants and corrosion inhibitors, among others (Bloys et al., 1994).

2.4.7.1.1 Emulsifiers

Emulsifiers are added to drilling fluids to stabilize the emulsions and mix immiscible liquids. In OBM oil-mud emulsifiers are added, forming water-in-oil emulsions. Water droplets in the oil phase are stabilized by the use of surfactants, and prevent the droplets to coagulate and separate out. The internal water phase present in the oil-based mud are loaded with excess emulsifying agents so that formation water can be dispersed into the OBM during drilling (Skaugen, 1997). Oil-based emulsifiers can be calcium fatty-acid soaps or derivatives (Baker Hughes, 2006). These emulsifiers surround water droplets, with the fatty acid component extending into the oil phase. Figure 2.18 shows the surfactant structure of soaps.
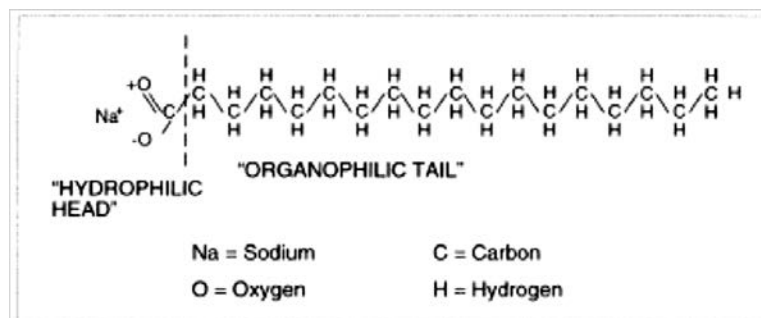
Figure 2.18 *Surfactant structure, soaps (Baker Hughes, 2006).*

For water-based muds numerous types of emulsifiers will disperse oil into water muds, including: sulfonated hydrocarbons, ethyoxylated nonylphenols, alkali-metal fatty-acid soaps, lignosulfonate, among others (Baker Hughes, 2006).

2.4.7.1.2   Wetting Agents

Wetting agents are commonly added to OBMs. Mineral solids and clays are normally ionic and polar, naturally preferring the water phase. As these solids water-wet, they stick together forming agglomerates, which settle out. By the use of wetting agents that adsorb on to the solids, they alter the ionic surface and promote oil wetting instead. This way solids can be dispersed in the nonpolar oil phase (Baker Hughes, 2006).

2.4.7.1.3   Lubricants

Lubricants are chemicals formulated to reduce the coefficient of friction of water-based drilling fluids, which minimizes torque and drag. Lubricants are employed to reduce stuck pipe tendencies of water-based drilling fluids and increase rate of penetration (ROP) during drilling (M-I Swaco, 2014a). Normally the base fluid provides sufficient lubricity to Non-aqueous drilling fluid, eliminating the need for lubricating agents (OGP, 2003).

2.5   Drilling Fluid Waste

There has been an increased focus and emphasis on environmental issues in the oil and gas exploring industry the recent years. Attention has been on the wastes generated during drilling, and with focus on discharges as the oil and gas industry is moving towards zero harmful discharge (Ivan and Dixit, 2006).

Non-aqueous drilling fluids are utilized to drill multiple holes during their lifetime. In doing so, colloidal particle concentration increases over time resulting in dramatic changes in fluid properties, eventually giving unusable drilling fluid. In most cases the drilling fluid is diluted and reformulated to minimize the concentration of the colloidal solids and restore the properties of the fluid. Eventually, when the mud can no longer be diluted and reformulated, it ends up as waste (Ezell and Harvey, 2008).

2.5.1   Slop-Mud

An average of 16 000 - 80 000 liters (≈100-500 bbl) of slop is produced on a daily basis during normal drilling activities (Ivan and Dixit, 2006). Slop-mud is waste streams generated when non-aqueous drilling fluids are contaminated with large quantities of water (McCosh et al., 2009). The large amounts of water can be emulsified into the mud due to excess of emulsifying agents already present in the invert-emulsion mud. The contamination of water makes the slop mud contain 50 to 90 vol % water, and 10 to 50 vol % of the original drilling fluid, decreasing the OWR of the OBM (Ivan and Dixit, 2006). The drilling fluid is no longer usable when it is slop-mud. Slop muds require treatment as they contain harmful constituents and cannot be discharged to sea.

2.5.2   Separating Slop Muds

Field muds are often over-treated and contain widely varying concentrations of emulsifiers, formation solids and property additives. Excess emulsifiers in the mud systems make it more challenging to destabilize and separate the mud waste. Often heat and demulsifiers are needed prior to settling or mechanical separation (ASME, 2005). However, the widely varying composition of the mud waste makes each mud require different treatment (Ezell and Harvey, 2008).

2.5.2.1 Demulsifiers

Demulsifiers (or emulsion breakers) are surfactants. An emulsifier is widely defined as a chemical, any agent added to an emulsion that causes or enhances the rate of breaking of the emulsion. Demulsifiers may act on a number of different mechanisms. Conventional demulsifiers are commonly formulated from the following chemistries: polyglycols and polyglycol esters, ethoxylated alcohols and amines, ethoxylated resins, ethoxylated phenol formaldehyde resins, ethoxylated nonylphenols, polyhydric alcohols, and sulfonic acid salts. Commercial emulsion breakers may contain one type of active ingredient or intermediate or a variety (Schramm, 1992).

Critical for breaking an emulsion, the demulsifier must reach the interface on an emulsified droplet and the surrounding liquid. In addition, a critical dose of demulsifier is needed to compete the emulsifiers already present. At that point, the emulsion breaker disrupts the interfacial tensions between the present phases. For slop mud wastes, demulsifying components destabilize the slop mud and aid in water-wetting the originally oil-wet solids present in the slop-mud (Ezell and Harvey, 2008).

Promising demulsifiers were tested during this project (see Chapter 3).

2.5.2.2 Flocculating Polymers

Flocculation is a method of removing colloidal-size drilled solids (Lummus and Azar, 1986). Flocculating polymers are used for agglomeration of colloidal solids for removal by screening or settling. The polymers are long chain molecules, which wrap themselves around the colloidal particles, so the particles form flocks and can be separated (see Figure 2.19)
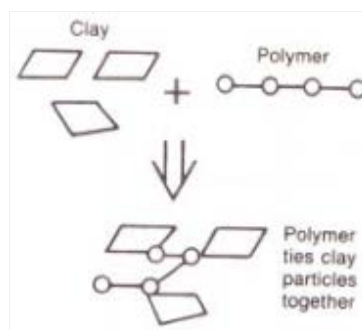
Figure 2.19 *Mechanism of flocculation; Flocculating polymer and colloidal clay (Lummus and Azar, 1986).*

2.5.3 Key Chemicals of the Research

Almost all the chemicals used in the project are confidential, and no detailed information was given on composition. Key chemical groups tested are MudSplit Chemicals, SAS Chemicals and GSA MudFloc Polymers.

SAS chemicals are *MudClean* splitting products that break and separate mud waste. The chemistries of SAS products utilize microemulsion technology, which is extremely effective at separating oil, water and solids. The SAS chemistry reduces the viscosity of the wastes, water-wet drill solids and gives distinct layers of oil, water and solids (Surface Active Solutions, 2013).

Information of the other chemicals was not available from the supplier.

3   Materials and Methods

Laboratory work was performed aiming to destabilize and separate mud by utilizing chemicals and mechanical equipment (lab centrifuge). The intention was to identify chemicals that together with increased centrifugal force gave successful 3-phase separation of mud waste with respect to chemical dosage (lowest possible), applicable g-force and retention time. A screening of different destabilization chemicals, tested on various mud-characteristics, was done. Laboratory testing was done in two steps; pre-screening and main experiment.

Various muds, provided from different suppliers, were tested (Table 3.1) Retort analyses of the various muds were performed to determine the volume fractions of oil, water, and solids in the mud. Retort analysis is described in Chapter 3.3.

Table 3.1 *Overview of muds used during the experiment.*

Mud character	Status of mud at arrival to UiS lab	Supplier	Mud name	Pre-screening		Main experiment	
				Base mud	Lab slop	Base mud	Lab slop
OBM	Fresh	Baker Hughes	*Carbosea*	✓			✓
		"	*Carbosea LC-LUBE Fine Premix*[5]				✓
		M-I SWACO	*Versatec*	✓			✓
	Degraded mud waste	SAR Treatment AS	*SAR-mud*	✓			

During the main experiment the base-muds were made into lab-slop muds to be more representative as mud waste. Lab slop mud was made by mixing synthetic seawater and OBM in a ratio of 1:1 (McCosh et al., 2009). Recipe of the synthetic seawater is given in Appendix D. By shaking the mud and synthetic seawater vigorously for a few minutes, the mixture became highly viscous. Emulsified water in oil base drilling fluid acts like a solid and increase the viscosity dramatically (Caenn et al., 2011).

Two centrifuges were used for mechanical separation. Relevant data for the centrifuges are given in Table 3.2, including details on equipment and operational parameters.

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[5] Carbosea LC-LUBE Fine Premix arrived late in the project, therefore no testing during pre-screening.

Table 3.2 *Data for centrifugal separation.*

		Pre-screening	Main experiment
Centrifuge details	Model	*Sorvall RC-5B Superspeed Centrifuge*	*Hettich Rotofix 46 Benchtop Centrifuge*
	Rotor	SS 34	5694
	Radius, rotor (mm)	107	173
	Volume of centrifuge bottles (mL)	50	290
Operational parameters			
Relative Centrifugal Force (RCF)*		2000	
		3000	3000
		7700	
Retention time (minutes)		1	
		2	2
		3	
		6	

*Conversion formula and conversions of RCF to RPM (revolutions per minute) are given in Appendix A.

3.1 Pre-Screening

Pre-screening was performed in order to specify and find optimal conditions for further testing. Eight chemical mixtures were tested on three different types of oil-based muds (Carbosea, Versatec and SAR-mud) to find an acceptable range of dosage. Variations in g-force and retention time were evaluated to optimize operational parameters for centrifugal separation. Pre-screening also included jar-testing with variation in temperature.

3.1.1 Centrifugal Separation

Variation in centrifugal force and retention time was studied during pre-screening. A *Sorvall RC-5B* lab centrifuge (Figure 3.1) at the University of Stavanger was used. Details regarding the centrifuge are given in Table 3.2 in addition to an overview of the different parameters adjusted during pre-screening.
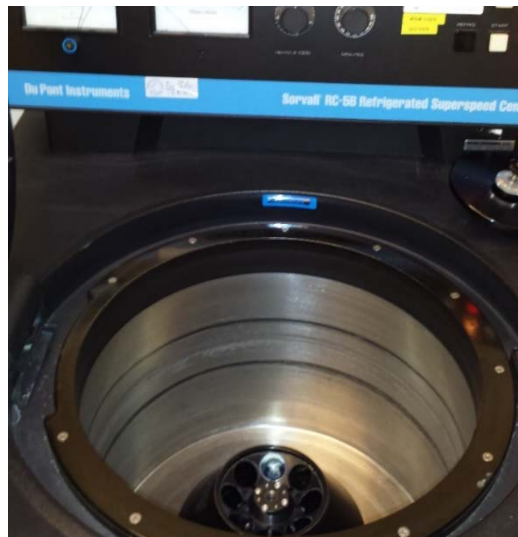
Figure 3.1 *Sorvall RC-5B Superspeed Centrifuge, with the SS34 rotor.*

3.1.3 Destabilization and Separation

The destabilization and separation were done in order to find out the most efficient chemical mixtures for main experiment.

3.1.3.1 Lab-Procedure

Lab-procedure included several steps:

1) Mud samples of 30 mL were put into centrifuge bottles by pouring or by a spatula.
2) Chemical mixtures were pipetted into the centrifuge bottles at different concentrations.
   a. The chemical mixtures and muds were mixed by shaking the centrifuge bottles for one minute by hand.
   b. Observation of possible changes in viscosity.
3) Mechanical separation by lab centrifuge. Centrifuge bottles containing mud and chemicals were balanced on a balance-weight before put into the centrifuge.
4) Results were reported in terms of successful 3-phase separation.
5) For most tests the liquid phase was poured into a graduated glass cylinder. Volumes of total liquid, oil and water were noted.

3.1.3.2 Destabilizing Chemical Mixtures

An overview of the chemical mixtures tested on the different muds during pre-screening is given in Figure 3.2.
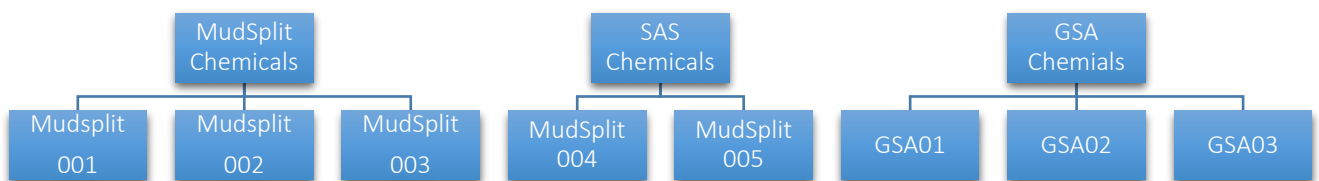
Figure 3.2 *Chemicals tested during pre-screening.*

3.1.4 Effect of Temperature

Pre-screening included jar-testing with variation in temperature. Drilling mud (*SAR-mud*) was poured into a glass beaker and heated to 80 °C. A magnetic stirrer with hotplate was used for heating; a lab thermometer was used for measuring the temperature. Mud was poured into two centrifuge bottles after reaching 80 °C. Temperature, measured in the centrifuge bottles prior to centrifuging, was approximately 50 °C.

Viscosity is dependent on temperature, decreasing with increasing temperature. By decreasing viscosity prior to centrifuge, separation will increase according to Stoke's law.

Instead of addition of chemicals (Step 2, Chapter 3.1.2.1), the samples were heated. The rest of the lab-procedure was performed as described in Chapter 3.1.2.1.

3.2 Main Experiment

The tests of main experiment part were performed and optimized, based on the results from pre-screening. Muds, used for testing, were made into lab slop-muds to be more representative as mud waste. Additionally, jar-testing with variation in temperature was performed. Analytical work was done on representative samples after successful 3-phase separation, with the intention to define separation efficiency.

3.2.1 Centrifugal Separation

Mechanical separation was performed by the *Hettich Rotofix 46 Centrifuge* (Figure 3.3) at the UiS lab. The centrifuge was run at fixed retention time and g-force (2 minutes and 3000 g, respectively). Details regarding the centrifuge and operational parameters are given in Table 3.2.

Figure 3.3 *Hettich Rotofix 46 Centrifuge.*

3.2.2 Destabilization and Separation

The destabilization and separation were done in order to find out the most efficient dosage of chemical mixtures for sufficient 3-phase separation.

3.2.2.1 Lab-Procedure

Lab-procedure included the following steps:

1) Mud samples of 200 mL were put into centrifuge bottles.
2) Chemical mixtures were pipetted into the centrifuge bottles at different concentrations.
   a. The chemical mixtures and mud were mixed by shaking the centrifuge bottles by hand for one minute.
   b. Observation of possible changes in viscosity.
3) Mechanical separation by lab centrifuge. Centrifuge bottles with mud and chemical mixtures were balanced on a weight before put into the centrifuge.
4) Results were registered in terms of successful 3-phase separation.
5) Analytical work on representative samples for reporting separation efficiencies (see Chapter 3.2.4).

3.2.2.2 Destabilizing Chemical Mixtures

Chemical mixtures were tested at specified concentration range during main experiment. An overview of the chemical mixtures tested on the different slop-muds is given in Figure 3.4.
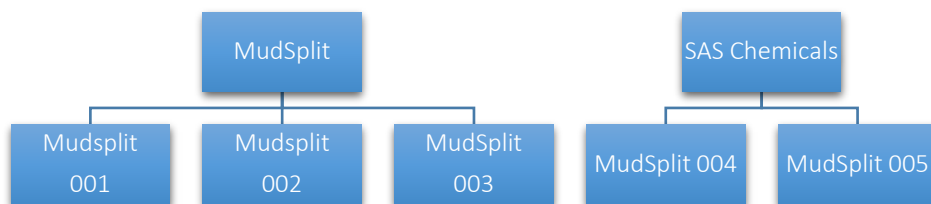
Figure 3.4 *Chemical mixtures studied during main-experiment.*

3.2.3 Effect of Temperature

Jar-testing with variation in temperature was performed in order to study the effect of temperature on separation process. Drilling mud (*Versatec OBM*) was poured into three different glass beakers and heated to 40, 60 and 80 °C respectively. Magnetic stirrers with hotplates were used for heating; a lab thermometer was used for measuring the temperature in each beaker. When the desired temperature was reached, additional heating was done prior to pouring the mud into centrifuge bottles. The additional temperature rise was in order to account for the temperature drop during transferring the mud and centrifuging. Heating was done instead of addition of chemicals (Step 2, Chapter 3.2.2.1). The rest of the lab-procedure was performed as described in Chapter 3.2.2.1

3.2.4 Analytical Work on Separation Efficiency

Analytical work was performed on a few representative samples after successful 3-phase separation. The intention was to determine separation efficiencies. Based on visual observations, samples with good results at lowest possible dosing were chosen (Table 3.3).

Table 3.3 *Samples for reporting separation efficiency.*

Sample	Mud	Dosing, MudSpli001
1	Carbosea (50/50)	0.5 % vol/vol
2	Carbosea Lube Fine Premix (50/50)	0.5 % vol/vol
3	Versatec (50/50)	0.5 % vol/vol

Each of the three samples was prepared in a two-fold, according to the lab-procedure described in Chapter 3.2.2.1. After centrifuging the liquid phases (from the two centrifuge bottles of same composition) were decanted into a glass bottle for overnight settling to obtain clear, distinct oil- and water phases. Figure 3.5 gives an overview of the procedure, repeated for all the three samples.
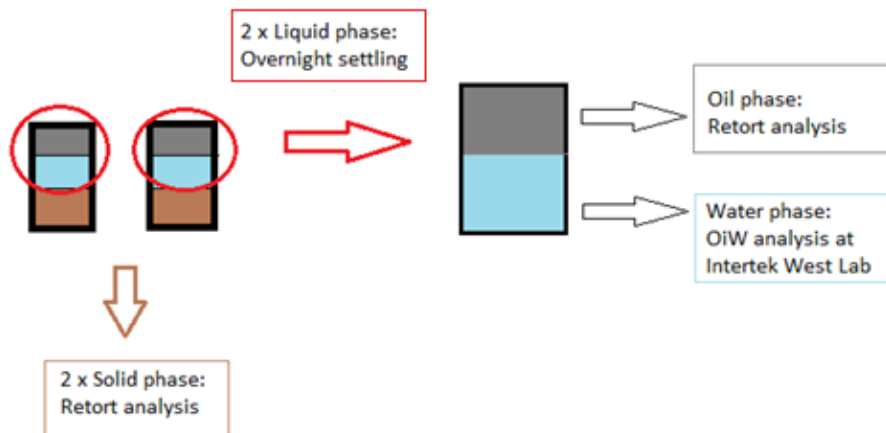
Figure 3.5 *Procedure for reporting on separation efficiency.*

After overnight settling the oil-phase was pipetted into the retort cup and analyzed. The water phase was sent to *Intertek West Lab* for oil in water analysis. The solid phases were mixed to one sample, thereafter transferred to the retort cup by a spatula and analyzed by the retort.

3.3 Retort Analysis

Volume fractions of oil, water, and solids in mud are determined by a retort analysis. A known volume of a mud sample is heated in the retort; the liquid components are vaporized, condensed and collected in a graduated cylinder. The volumes of water and oil are read in the cylinder. The solids remain, and the volume of solids is obtained by subtracting the liquid volume from the total sample volume (Baker Hughes, 2006).

3.3.1 Equipment and Procedure

The retort apparatus used during the project is shown in Figure 3.6.

Figure 3.6 *Retort apparatus.*

Retort analysis was performed by the following steps:

1. The retort cup was filled with 50 mL mud sample (fixed volume).
2. The retort cup assembly was put together with the condenser mounted.
3. The retort cup was put inside the heating chamber with the condenser on the outside.
4. A receiving graduated cylinder was put under the condenser.
5. Temperature set at 480 °C before the analysis can be started.
6. The total volume liquid, oil and water (mL) are recorded from the graduated cylinder after analysis.

Calculations are necessary to obtain the volume fractions and oil-water ratio, see Appendix B.

4   Results

This part of thesis contains tables, photos and graphically presentations of the results. Discussion of results follows in Chapter 5.

4.1 Pre-Screening

4.1.1 Centrifugal Separation

Results from centrifugal separation, with variation retention time and centrifugal force are presented graphically in Figure 4.1 – 4.5. After centrifuging the liquid was poured into graduated cylinders and volumes were read (raw data and conversions of RCF and RPM are given in Appendix A). Each value in the curves represents average numbers from two bottles of blank SAR-mud put in the centrifuge.

4.1.1.1 Fixed Centrifugal Force, Variations in Retention Time

From Figure 4.1 and 4.2 it is clear that at 2000 g retention time does affect much on separation efficiency, while at 7000 g a more dynamic picture is observed. Phase separation increases significantly.
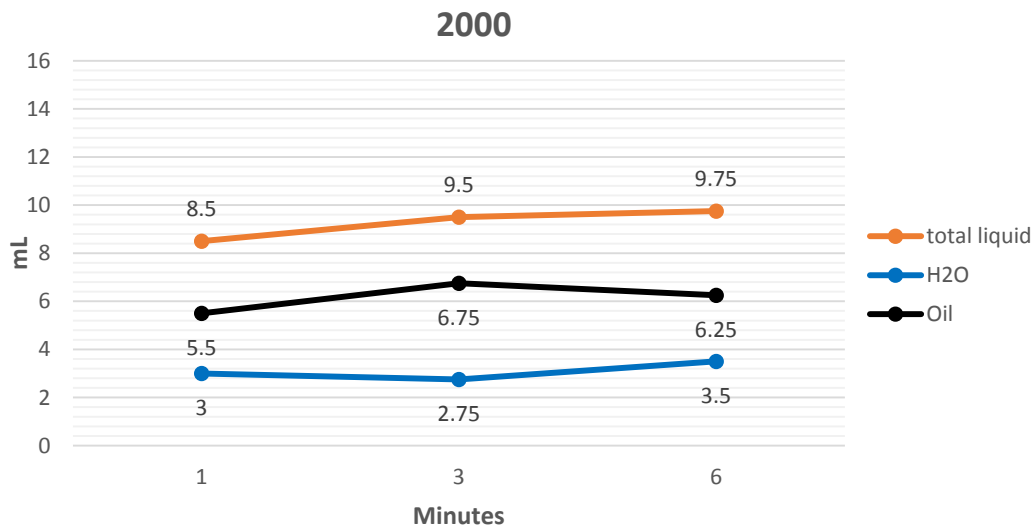
Figure 4.1 *Fixed centrifugal force, 2000 g. 1, 3 and 6 min. retention time.*
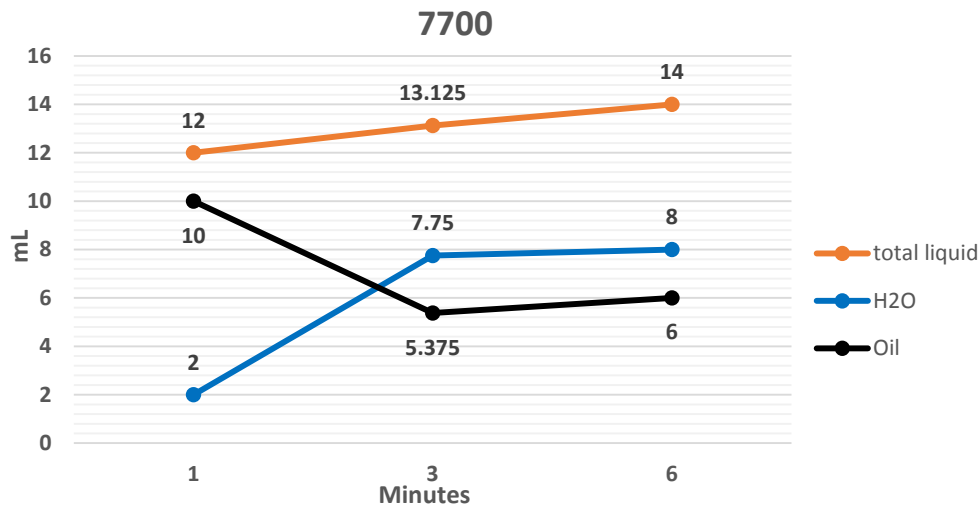
Figure 4.2 *Fixed centrifugal force, 7700 g. 1, 3 and 6 min. retention time.*

4.1.1.2 Fixed Retention Time, Variations in Centrifugal Force

From Figures 4.3 – 4.5 it can be seen that centrifugal force is the most important factor for obtaining satisfying phase-separation. Increasing centrifugal force from 2000 to 7700 g, gives significant increase in total liquid recovery, as well as improved phase-separation.
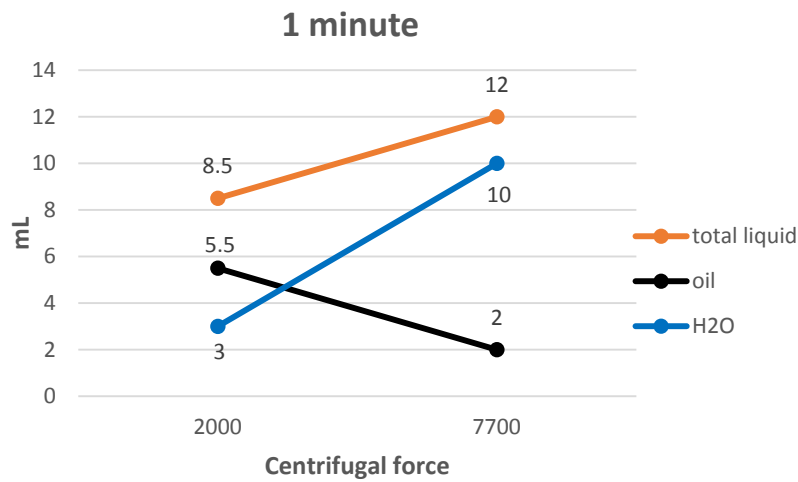
Figure 4.3 *One minute retention time, variation in RCF.*
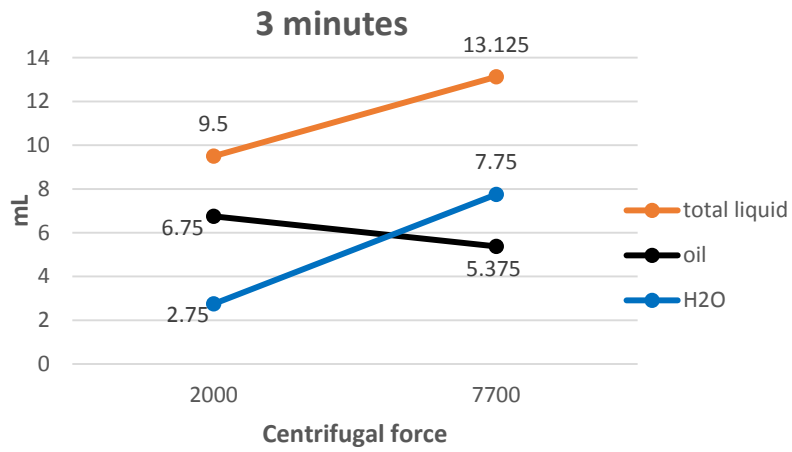
Figure 4.4 *Three minutes retention time, variation in RCF.*
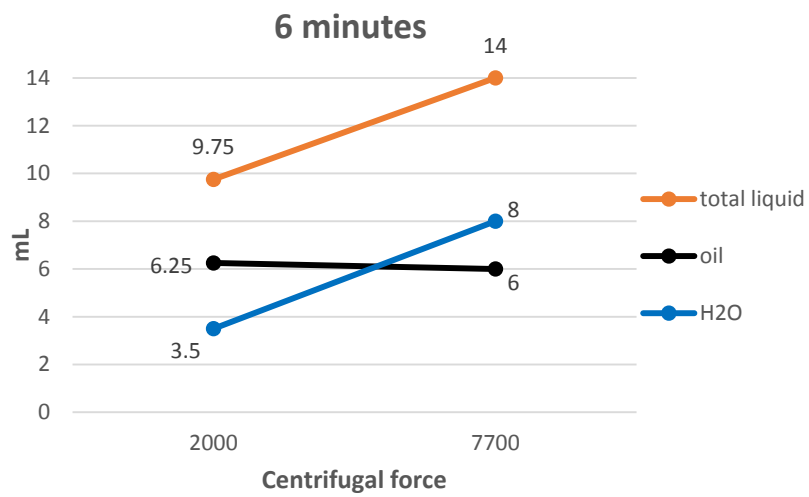
Figure 4.5 *Six minutes retention time, variation in RCF.*

4.1.2 Destabilization and Separation

Results in Table 4.2 – 4.4 report on the effect of the destabilizing chemical mixtures listed in Chapter 3.1.2.2. Symbols for explanation of the results are given in Table 4.1.

Table 4.1 *Symbols for reporting results.*

-	Test not performed
N	Negative, only 2-phase separation
P	Positive, 3-phase separation
P+	Enhanced 3-phase separation
μ↓	Decrease in viscosity after mixing chemicals and mud.

Table 4.2 *Effect of MudSplit chemicals for destabilization.*

Chemical	Concentration range (vol/vol %)	Carbosea OBM	Versatec[6] OBM	SAR-mud
MudSplit001	*Blank*	N	N	P
	0.10	N	-	P
	0.30	N	-	**P+ (μ↓)**
	0.60	**P** (μ↓)	-	**-**
	1.00	**P+ (μ↓)**	**-**	**P+ (μ↓)**
	1.50	**P+ (μ↓)**	**-**	**-**
	2.00	**-**	**-**	**P+ (μ↓)**
MudSplit002	0.50	N	-	**-**
	0.75	N	-	**-**
	1.00	N	-	**P+ (μ↓)**
	1.50	N	-	**P+ (μ↓)**
	2.00	N	-	**-**
	3.00	N	-	**P+ (μ↓)**
	5.00	N	-	**-**
MudSplit003	0.50	N	-	**-**
	1.00	N	-	**P+ (μ↓)**
	1.50	N	-	**-**
	2.00	N	-	**P+ (μ↓)**
	3.00	-	-	**P+ (μ↓)**

From Table 4.2 it can be read that the combination of MudSplit001 and Carbosea, as well as SAR-mud gives satisfying 3-phase separation. A combination of MudSplit002 and 003 with Carbosea did not provide desirable phase-separation.

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[6] Versatec OBM arrived at the end of pre-screening, thus only a few tests performed.

From Table 4.2 it is clear that blank samples of SAR-mud gave 3-phase separation. However, some of the chemical mixtures gave enhanced separation. Figure 4.6 gives a good depiction of the difference of positive 3-phase separation (**P**) and enhanced 3-phase separation (**P+**) of SAR-mud.
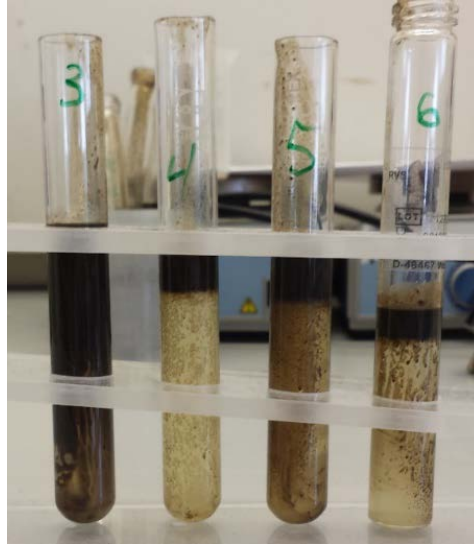
Figure 4.6 *SAR-mud and MudSplit001 at concentrations of (from left to right): 0.10 (# 3), 0.30 (# 4), 1.00 (# 5) and 2.00 vol/vol % (# 6).*

Tube # 3 shows 3-phase separation, similar result as blank samples gave (noted as **P** in Table 4.2). Water is apparent at the bottom of the glass tube, but distinctive phases are not observed. However, distinctive phases are observed in tube # 4 – 6, the 3-phase separation is significantly improved (noted as **P+** in Table 4.2).

Results from testing different concentrations of SAS Chemicals on chosen muds are given in Table 4.3.

Table 4.3 *Effect of SAS chemicals used for destabilization.*

Chemical	Concentration range (vol/vol %)	Carbosea OBM	Versatec OBM	SAR-mud
	Blank	*N*	*N*	*P*
MudSplit004	0.25	N	N	*P*
	0.50	N	N (μ↓)	*P*
	1.00	N (μ↓)	N (μ↓)	**P** (μ↓)
	2.00	N (μ↓)	N (μ↓)	**P** (μ↓)
MudSplit005	0.25	N	N	**P+**
	0.50	N	N (μ↓)	**P+**
	1.00	N (μ↓)	N (μ↓)	**P+** (μ↓)
	2.00	**P** (μ↓)	**P** (μ↓)	-

The effect of MudSplit005 at 2.00 vol/vol % on Carbosea OBM, compared to a sample of blank Carbosea OBM, is shown in Figure 4.7.

Figure 4.7 *Centrifuge bottles directly after centrifuging. Blank sample of Carbosea OBM (# B) and with 2.00 % MudSplit005 (# 9).*

Two-phase separation is observed for # B, while 3-phase separation with a clear water phase is apparent in # 9.

Results from testing different concentrations of Global Solution chemicals (GSA) on chosen muds are given in Table 4.4.

Table 4.4 *Effect of Global Solution chemicals used for destabilization.*

Chemical	Concentration range (vol/vol %)	Carbosea OBM	Versatec OBM	SAR-mud
	Blank	N	N	*P*
GSA01	0.05	N	-	-
	0.10	N	-	-
	0.15	N	-	-
	0.20	N	-	-
	0.30	N	-	-
	0.60	N	-	-
GSA02	0.05	N	-	-
	0.10	N	-	-
	0.15	N	-	-
	0.20	N	-	-
	0.30	N	-	-
	0.60	N	-	-
GSA03	0.05	N	-	-
	0.10	N	-	-
	0.15	N	-	-
	0.20	N	-	-
	0.30	N	-	-
	0.60	N	-	-

No effect was observed by testing Global Solution chemicals on Carbosea OBM. It can be concluded that GSA chemicals, at given concentrations, are not able to destabilize Carbosea OBM.

4.1.3 Effect of Temperature

Results after heating SAR mud (performed by the procedure described in Chapter 3.1.3) are given in Figure 4.8. A blank sample, representing 20 °C, was compared with Sample # 1 and Sample 2 that were heated to 80 °C and put in centrifuge at 50 °C (labeled as "80 °C → 50 °C"). Viscosity drop of SAR-mud was evident reaching 70 °C.
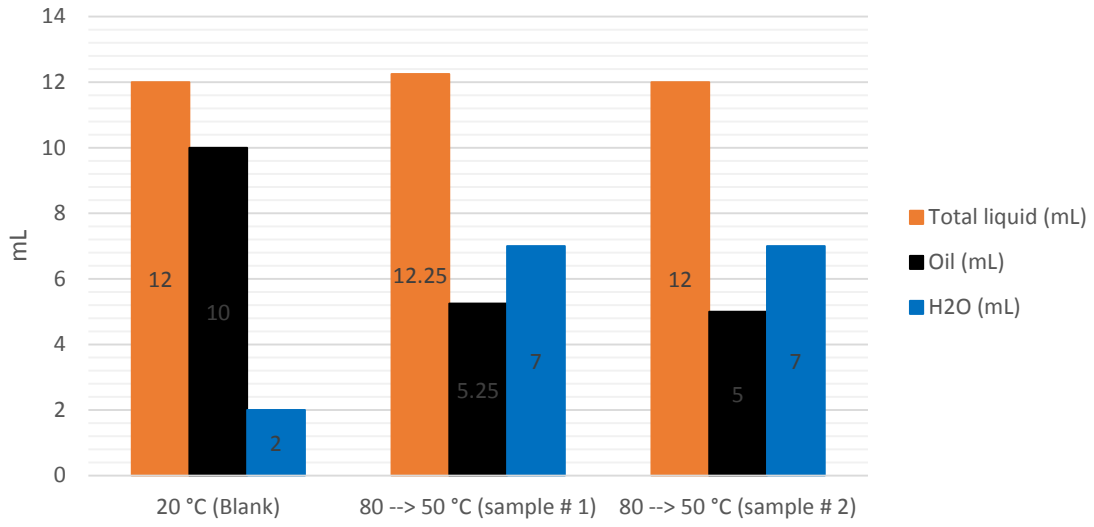
Figure 4.8 *Effect of temperature on separation of SAR-mud.*

4.2   Main Experiment

Results from the main experiment are given in subsequent chapters. Muds used for testing were lab slop-muds, labelled "(50/50)".

4.2.1   Centrifugal Separation

Centrifugal separation was performed by a centrifugal force of 3000 g and 2 minutes retention time.

4.2.2   Destabilization and Separation

Results are presented in Figure 4.10 – 4.20 with associated descriptions in Table 4.5 – 4.15. Photos are taken after overnight settling. Results report on testing of the destabilizing chemical mixtures listed in Chapter 3.2.2.2 on three different muds: Carbosea OBM (50/50), Versatec OBM (50/50) and Carbosea LC-LUBE Fine Premix OBM (50/50). For all tests, possible changes in viscosity observed prior to centrifuging are described. Figure 4.9 shows some of the destabilizing chemicals used for testing.
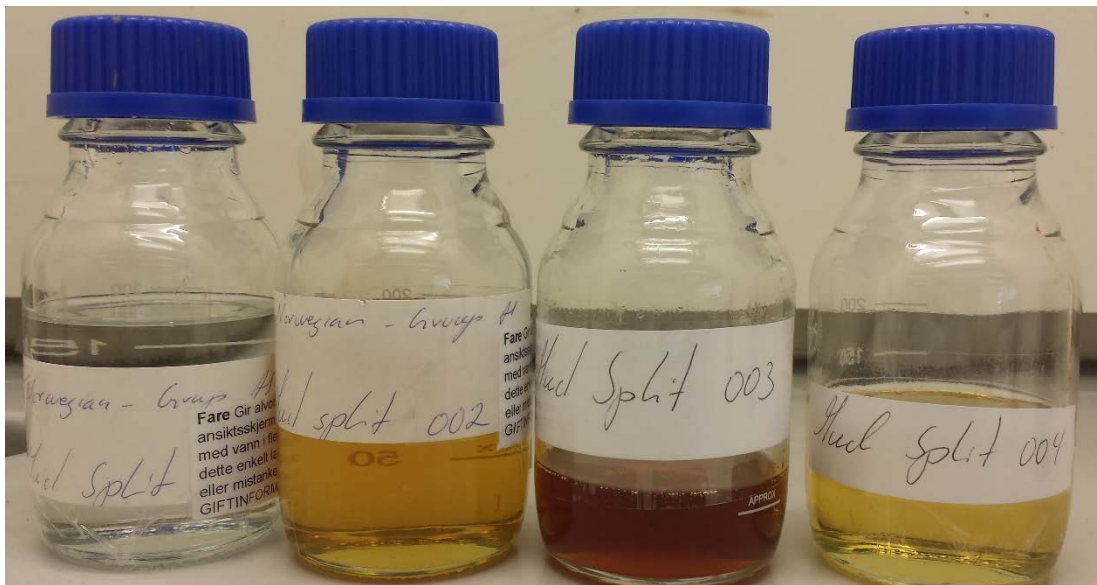
Figure 4.9 *MudSplit chemicals.*

4.2.2.1 Carbosea OBM (50/50)

This chapter presents results from testing MudSplit001-005 on Carbosea OBM (50/50).

4.2.2.1.1 MudSplit001

Figure 4.10 shows the effect of MudSplit001 at different concentrations on Carbosea OBM (50/50). Table 4.5 explains effect of different concentrations of MudSplit001 added to Carbosea OBM (50/50).

Figure 4.10 *Carbosea OBM (50/50) + MudSplit001 added at following concentrations (left to right) Blank (# 1), 0.50 (# 2), 2.00 (# 3) and 5.00 vol/vol % (# 4).*

Table 4.5 *Carbosea OBM (50/50) + MudSplit001.*

Sample #	Concentration (vol/vol %)	Effect
1	Blank	2-phase separation. Dense solid phase at bottom.
2	0.50	3-phase separation. Oil phase on top; water and solids emulsified into the oil-phase. Water phase with flocks. Dense solid phase at bottom.
3	2.00	3-phase separation. High water recovery, turbid water phase (suspended solids and oil emulsified in water phase). Dense solid phase at the bottom.
4	5.00	4-phase separation. i) Oil phase on top. ii) Turbid, dark, water phase with emulsified oil and solids. iii) An "extra" water phase is apparent being white and turbid, with suspended solids. iv) Dense solid phase at bottom.

It was observed that viscosity decreased with increasing dosage of MudSplit001. At 2.00 % and 5.00 % the viscosity drop was evident.

4.2.2.1.2 MudSplit002

No effect was observed by testing different concentrations of MudSplit002 on Carbosea OBM (50/50). From Figure 4.11 and Table 4.6 it can be seen that there is no 3-phase separation.

Figure 4.11 *Carbosea OBM (50/50) + MudSplit002 added at following concentrations (left to right) Blank, 0.50 (# 5), 2.00 (# 6), 3.00 (# 7) and 5.00 vol/vol % (# 8).*

Table 4.6 shows effect of different concentrations of MudSplit001 added to Carbosea OBM (50/50).

Table 4.6 *Carbosea OBM (50/50) + MudSplit002.*

Sample #	Concentration (vol/vol%)	Effect
5	0.50	2-phase separation, similar to blank.
6	2.00	"
7	3.00	"
8	5.00	"

It was observed that viscosity decreased slightly at highest dosing of MudSplit002.

4.2.2.1.3 MudSplit003

Figure 4.12 shows successful 3-phase separation at 2.00 and 5.00 % vol/vol concentration of MudSplit003 added to Carbosea OBM (50/50).

Figure 4.12 *Carbosea OBM (50/50) + MudSplit003 added at following concentrations (left to right) Blank, 0.25 (# 1), 0.50 (# 2), 2.00 (# 3) and 5.00 vol/vol % (# 4).*

Table 4.7 explains the effect of different concentrations of MudSplit003 added to Carbosea OBM (50/50).

Table 4.7 *Carbosea OBM (50/50) + MudSplit003.*

Sample #	Concentration (vol/vol %)	Effect
1	0.25	2-phase separation, similar to blank. Dense solid phase at bottom.
2	0.50	"
3	2.00	3-phase separation. Oil phase with emulsified water and solids. Clear water phase*. Dense solid phase at the bottom.
4	5.00	4-phase separation. i) Oil phase with emulsified water and solids. ii) Clear water phase*. iii) Water with solids. iv) Dense solid phase *at the bottom*.

*Water phase is clear, however some oil stuck on centrifuge bottle makes it appear more turbid.

It was observed that viscosity decreased with increasing dosage of MudSplit003. At 2.00 % and 5.00 % dosing viscosity drop was evident.

4.2.2.1.4 MudSplit004

Mudsplit004 was not tested on Carbosea lab slop mud (50/50). Decision was made based on negative results during pre-screening and negative results on Versatec lab slop mud (50/50) during main experiment (described in Chapter 4.2.2.2.4).

4.2.2.1.5 MudSplit005

Figure 4.13 shows successful 3-phase separation at 1.00 and 2.00 % vol/vol MudSplit005 on Carbosea OBM (50/50). However, at 1.00 % dosing 3-phase separation is not optimized.

Figure 4.13 *Carbosea OBM (50/50) + MudSplit005 added at following concentrations (left to right): Blank, 0.25 (# 1), 0.50 (# 2), 1.00 (# 3) and 2.00 vol/vol % (# 4).*

Table 4.8 *Carbosea OBM (50/50) + MudSplit005.*

Sample #	Concentration (vol/vol %)	Effect
1	0.25	2-phase separation, similar to blank. Dense solid phase at bottom.
2	0.50	"
3	1.00	3-phase separation, but not distinctive phases. Oil on top. Water phase with an evident oil/solids emulsified internal phase. Dense solid phase at bottom.
4	2.00	Perfect 3-phase separation. Oil on top. Clear water phase. Dense solid phase at bottom.

*Water phase is clear, however some oil stuck on centrifuge bottle makes it appear more turbid.

It was observed that viscosity decreased with increasing dosage of MudSplit005. At 1.00 % and 2.00 % viscosity drop was evident.

4.2.2.2 Versatec OBM (50/50)

This chapter presents results from testing MudSplit001-005 on Versatec OBM (50/50).

4.2.2.2.1 MudSplit001

Figure 4.14 shows successful 3-phase separation on 0.50, 2.00 and 5.00 % vol/vol MudSplit001 on Versatec OBM (50/50).

Figure 4.14 *Versatec OBM (50/50) + MudSplit001 added at following concentrations (left to right) Blank, 0.25 (# 1ˣ), 0.50 (# 2ˣ), 2.00 (# 3ˣ) and 5.00 vol/vol % (# 4ˣ).*

Table 4.9 describes the effect of MudSplit001 on Versatec OBM (50/50).

Table 4.9 *Versatec OBM (50/50) + MudSplit001.*

Sample #	Concentration (vol/vol %)	Effect
No label	Blank	2-phase separation, dense solid phase at the bottom.
1ˣ	0.25	2-phase separation, similar to blank.
2ˣ	0.50	Perfect 3-phase separation. Oil phase is black, contains some emulsified water and solids. Clear water phase*. Dense solid phase.
3ˣ	2.00	Perfect 3-phase separation. Black oil phase. Perfectly clear water phase. Dense solid phase at the bottom.
4ˣ	5.00	Perfect 3-phase separation. Oil phase contains more emulsified water and solids than lower dosing. Flocks floating at the oil/water interface. Perfectly clear water phase. Dense solid phase at the bottom.

*Water phase is clear, however some oil stuck on centrifuge bottle.

Viscosity decreased with increasing concentration of MudSplit001. Already at 0.50 % dosing, viscosity drop was evident.

4.2.2.2.2   MudSplit002

Figure 4.15 and Table 4.10 show no effect of MudSplit002 on Versatec OBM (50/50) at all tested concentrations.

Figure 4.15 *Versatec OBM (50/50) + MudSplit002 added at following concentrations (left to right) Blank, 0.50 (# 5ˣ), 2.00 (# 6ˣ), 3.00 (# 7ˣ) and 5.00 vol/vol % (# 8ˣ).*

Table 4.10 *Versatec OBM (50/50) + MudSplit002.*

Sample #	Concentration (vol/vol %)	Effect
5ˣ	0.50	2-phase separation, similar to blank.
6ˣ	2.00	"
7ˣ	3.00	"
8ˣ	5.00	"

Only at highest concentrations (3.00 and 5.00 %) of MudSplit002, minor changes in viscosity were observed by mixing with Versatec lab slop mud.

4.2.2.2.3 MudSplit003

Figure 4.16 shows successful 3-phase separation at 2.00 and 5.00 % dosage of MudSplit003 added to Versatec OBM (50/50).

Figure 4.16 *Versatec OBM (50/50) + MudSplit003 added at following concentrations (left to right) Blank, 0.25 (# 5), 0.50 (# 6), 2.00 (# 7) and 5.00 vol/vol % (# 8).*

Table 4.11 describes the effect of MudSplit003 on Versatec OBM (50/50) at different concentrations.

Table 4.11 *Versatec OBM (50/50) + MudSplit003.*

Sample #	Concentration (vol/vol %)	Effect
5	0.25	2-phase separation, same as blank.
6	0.50	"
7	2.00	3-phase separation. Large oil phase, with emulsified water and solids. Turbid water phase*, low water recovery. Dense solid phase at the bottom.
8	5.00	" Similar to 2.00 % (bottle # 7) - see explanation.

*Seems more turbid than it is due to oil stuck on centrifuge bottle.

It was observed that viscosity of the mud decreased at 2.00 % and 5.00 % vol/vol of MudSplit003.

4.2.2.2.4 MudSplit004

No effect was observed during tests with different concentrations of MudSplit004 on Versatec OBM (50/50), as shown in Figure 4.17 and Table 4.12.

Figure 4.17 *Versatec OBM (50/50) + MudSplit004 added at following concentrations (left to right): Blank, 0.25 (# A1), 0.50 (# A2), 1.00 (# A3) and 2.00 vol/vol % (# A4).*

Table 4.12 *Versatec OBM (50/50) + MudSplit004.*

Sample #	Concentration (vol/vol %)	Effect
A1	0.25	2-phase separation, similar to blank.
A2	0.50	"
A3	1.00	"
A4	2.00	2-phase separation. Almost same as blank, some changes in sediments compared to lower dosing - Observed by difference in colors, looks like oil stuck on centrifuge bottle. The mud is not destabilized.

It was observed that viscosity of the mud decreased slightly at 2.00 % dosing of MudSplit004.

4.2.2.2.5 MudSplit005

Figure 4.18 shows successful 3-phase separation of Versatec OBM (50/50) at 1.00 and 2.00 % concentrations of MudSplit005.

Figure 4.18 *Versatec OBM (50/50) + MudSplit005 added at following concentrations (left to right): Blank (# A), 0.50 (# B), 1.00 (# C) and 2.00 vol/vol % (# D).*

Effect of MudSplit005 on Versatec OBM (50/50) is described in Table 4.13.

Table 4.13 *Versatec OBM (50/50) + MudSplit005.*

Sample #	Concentration (vol/vol %)	Effect
A	Blank	2-phase separation. Dense solid phase at the bottom.
B	0.50	2-phase separation, similar to blank.
C	1.00	3-phase separation. Oil phase with emulsified water and solids. Clear water-phase*. Flocks on top of dense solid phase.
D	2.00	Perfect 3-phase separation. Clear, black oil phase. High water recovery*. Dense solid phase at the bottom.

*Water phase is clear, however some oil stuck on centrifuge bottle.

Viscosity decreased with increasing dosage of MudSplit005. Evident drop in viscosity was observed at 1.00 % and 2.00 % vol/vol.

4.2.2.3 Carbosea LC-LUBE Fine Premix OBM (50/50)

Only Mudsplit001 and Mudsplit005 were tested on Carbosea LC-LUBE Fine Premix OBM due to lack of mud. Minor volume Carbosea LC-LUBE Fine Premix OBM was delivered for testing.

4.2.2.3.1 Mudsplit001

Figure 4.19 shows successful 3-phase separation on Carbosea LC-LUBE Fine Premix OBM (50/50) at 0.5 and 0.75 % concentrations of MudSplit001.

Figure 4.19 *Carbosea LC-LUBE Fine Premix OBM (50/50) + Mudsplit001 added at following concentrations (left to right) 0.25 (# 1), 0.50 (# 2), 0.75 vol/vol % (# no label).*

Table 4.14 gives descriptions of the effect by testing MudSplit001 on Carbosea LC-LUBE Fine Premix OBM (50/50).

Table 4.14 *Carbosea LC-LUBE Fine Premix OBM (50/50) + Mudsplit001.*

Sample #	Concentration (vol/vol %)	Effect
1	0.25	2-phase separation, somewhat different from blank (Blank is shown in Figure 4.20). Dense solid phase at the bottom. The mud is not destabilized.
2	0.50	3-phase separation. Oil phase with emulsified solids and water**. Water phase is turbid and white. Oil emulsified in the water. Dense solid phase.
No label (0.75 %)	0.75	3-phase separation. Oil phase with emulsified solids and water**. High water recovery, with white and turbid water. Dense solid phase.

**White/grey powder on oil surface.

It was observed that the viscosity of the mud decreased with increasing dosage of Mudsplit001. Viscosity drop was observed even at lowest concentration.

4.2.2.3.2 Mudsplit005

Figure 4.20 shows successful 3-phase separation of Carbosea LC-LUBE Fine Premix OBM (50/50) at 1.00 % and 2.00 % concentration of MudSplit005.

Figure 4.20 *Carbosea LC-LUBE Fine Premix OBM (50/50) + MudSplit005 added at following concentrations (left to right) Blank, 1.00 % and 2.00 vol/vol %.*

Table 4.15 describes the effect of MudSplit005 on Carbosea LC-LUBE Fine Premix OBM (50/50).

Table 4.15 *Carbosea LC-LUBE Fine Premix OBM (50/50) + MudSplit005.*

Sample #	Concentration (vol/vol %)	Effect
No label	Blank	2-phase separation. Dense solid phase at the bottom.
"	1.00	Perfect 3-phase separation. Oil phase appears clear**. High water recovery, clear water phase*. Dense solid phase at the bottom.
"	2.00	Perfect 3-phase separation. Oil phase appears clear**. High water recovery, clear water phase. Dense solid phase at the bottom.

*Water phase is clear, however some oil stuck on centrifuge bottle. ** White/grey powder on oil surface.

It was observed evident viscosity drop of the Carbosea LC-LUBE Fine Premix OBM (50/50) by mixing with MudSplit005 at 1.00 % and 2.00 % vol/vol.

4.2.3 Effect of Temperature

Results after heating of Versatec lab-slop mud (procedure described in Chapter 3.2.3) are presented in Figure 4.21.

It was observed that the viscosity of Versatec OBM (50/50) decreased when temperature increased.

The results report on better separation by increased temperature, where successful 3-phase separation is observed at 60 °C and 80 °C. Minor separation can be observed already at 40 °C.

Figure 4.21 *Bottles with following temperature adjustments (left to right) 20°C (# A), 40°C, 60 °C and 80 °C.*

The water phases are clear, but oil stuck on centrifuge bottles gives a misleading picture on turbidity.

4.2.4 Separation Efficiency

Separation efficiency reports on concentrations of oil in water; fractions of water and solids in the oil phase; as well as fractions of oil and water in the solid phase.

4.2.4.1 Solid-Phase

The solid phases from two centrifuge bottles were mixed to one sample and analyzed by the retort. Results are given in Table 4.16 – 4.18. The retort analysis of the solid phases reports on high volumes of liquid. It is clear that more oil is stuck on solids, giving high oil-water-ratio (OWR).

Table 4.16 *Retort analysis of solid phase Carbosea OBM (50/50) + 0.5 % MudSplit001.*

	Carbosea OBM (50/50)	
	Vol (mL)	Vol %
Total liquid	23.5	47
Oil	19.5	39
H_2O	4	8
Solids	-	53
OWR	$\dfrac{83}{17}$	

Table 4.17 *Retort analysis of solid phase Carbosea LC-LUBE Fine Premix (50/50) + 0.5 % MudSplit001.*

	Carbosea LC-LUBE Fine premix (50/50)	
	Vol (mL)	Vol %
Total liquid	27	54
Oil	18.5	37
H_2O	8.5	17
Solids	-	46
OWR	$\dfrac{69}{31}$	

Table 4.18 *Retort analysis of solid phase Versatec OBM (50/50) + 0.5 % MudSplit001.*

	Versatec OBM (50/50)	
	Vol (mL)	Vol %
Total liquid	30	60
Oil	18.5	37
H_2O	11.5	23
Solids	-	40
OWR	$\dfrac{62}{38}$	

4.2.4.2 Oil-Phase

The oil phases from two centrifuge bottles were combined to give one sample and analyzed by the retort. Results are given in Table 4.19 – 4.21. The retort analysis of the oil phases reports on high volumes of water for Carbosea OBM (50/50) and Versatec OBM (50/50), at 32 and 64 % respectively. Carbosea LC-LUBE Fine Premix OBM contains only 14 % water. Generally, minor volumes of solids are found in all the samples at 3, 6 and 10 % for Carbosea OBM, Versatec OBM and Carbosea LC-LUBE Fine Premix OBM.

Table 4.19 *Retort analysis of oil phase Carbosea OBM (50/50) + 0.5 % MudSplit001.*

	Carbosea OBM (50/50)	
	Vol (mL)	Vol %
Total liquid	48.5	97
Oil	32.5	65
H$_2$O	16.0	32
Solids	-	3
OWR	$\dfrac{67}{33}$	

Table 4.20 *Retort analysis of oil phase Carbosea LC-LUBE Fine Premix (50/50) + 0.5 % MudSplit001.*

	Carbosea LC-LUBE Fine Premix (50/50)	
	Vol (mL)	Vol %
Total liquid	45.0	90
Oil	38.0	76
H$_2$O	7.0	14
Solids	-	10
OWR	$\dfrac{84}{16}$	

Table 4.21 *Retort analysis of oil phase Versatec OBM (50/50) + 0.5 % MudSplit001.*

	Versatec OBM (50/50)	
	Vol (mL)	Vol %
Total liquid	47.0	94
Oil	15.0	30
H$_2$O	32.0	64
Solids	-	6
OWR	$\dfrac{32}{68}$	

4.2.4.3 Water-Phase

Concentrations of oil-in-water, for sample 1-3, were estimated by mass-balance. Results from retort analysis (oil- and solids phases) were used to calculate remaining fractions of oil in the water phases. However, uncertain data from retort analyses of oil- and solids phases, gave imprecise oil-in-water concentrations estimated by mass-balance (Chapter 5.2.4). Theoretical oil-in-water concentrations are given in Appendix C.

Results from oil in water analysis, performed by Intertek West Lab, are given in Table 4.22, with detailed laboratory rapport in Appendix E. The water samples analyzed are shown in Figure 4.22

Table 4.22 *Oil-in-water concentrations found by Intertek West Lab.*

Sample	Mud	Dosing, MudSpli001 (% vol/vol)	Oil-in-Water (mg/L)
1	Carbosea (50/50)	0.50	120
2	Carbosea Lube Fine Premix (50/50)	0.50	58
3	Versatec (50/50)	0.50	16

Figure 4.22 *Water samples sent to Intertek West Lab for OiW analysis.*

Figure 4.22 shows more yellow water phases in bottles with higher concentration of OiW, as expected.

4.3 Retort Analysis

Results from retort analysis, reporting oil, water and solid fractions of the various base muds used for testing, are given in Table 4.23.

Table 4.23 *Retort analysis data, original base mud.*

		Carbosea OBM	Carbosea LC-LUBE Fine Premix OBM	Versatec OBM	SAR-mud
Vol (mL)	Total liquid	38.0	44.0	42.0	39.5
	Oil	30.5	15.5	32.0	14.5
	H_2O	75.0	28.5	10.0	25.0
Vol%	Oil	61.0	31.0	62.0	29.0
	H_2O	15.0	57.0	20.0	50.0
	Solids	24.0	12.0	16.0	21.0
OWR		$\dfrac{80}{20}$	$\dfrac{35}{65}$	$\dfrac{76}{24}$	$\dfrac{37}{63}$

Figure 4.23 shows emulsions at the oil-water-interfaces from retort analysis.
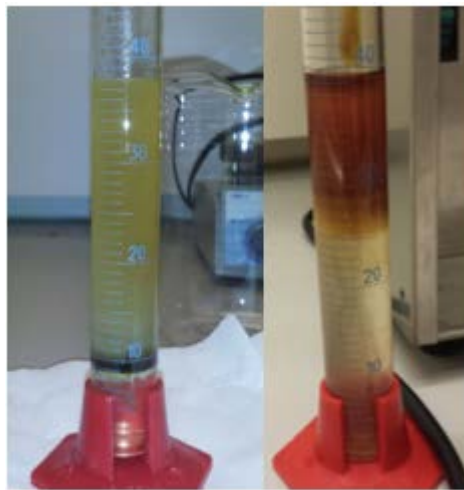
Figure 4.23 *Retort analysis of Carbosea OBM (left) and SAR-mud (right). Emulsions are evident at the oil-water-interfaces.*

5 Discussion

This chapter provides analysis and discussion of the results presented in Chapter 4.

5.1 Pre-Screening

5.1.1 Centrifugal Separation

From Figure 4.1 and 4.2 it can be seen that increased retention time gives minor increase in total liquid recovery. 15 % increase in total liquid are observed from one to six minutes at 2000 g. 17 % increase in total liquid are observed from on to six minutes at 7700 g.

High water content of the mud should give increased water recovery and less oil at increased retention time. Due to average numbers, there is no clear trend in oil/water recovery in Figure 4.1. Bottle # 2 (see raw data, Appendix A) shows increased water recovery at increased retention time, while Bottle # 1 gives misleading values. Additional testing should have been performed to obtain more results. Deviation calculations are given in Chapter 5.4.2.

Phase separation is significantly improved from one to three minutes retention time, seen clearly in Figure 4.2. After one-minute retention time the OWR is close to 80/20. However, after three minutes the OWR is close to 40/60. The same result is apparent at six minutes, an OWR close to 40/60. The individual results (raw data, Appendix A), Bottle # 1 and # 2, give good matching, thus average numbers presented in Figure 4.2 are concluded realistic.

Figure 4.3 – 4.5 shows that total liquid volume increases significantly at increased centrifugal force. Also, phase-separation is significantly improved by increased centrifugal force (see Table 5.1).

Table 5.1 *Effect of centrifugal force on phase-separation, OWR.*

	1 min		3 min		6 min	
Centrifugal force	2000	7700	2000	7700	2000	7700
OWR	65/35	17/83	71/29	41/59	64/36	43/57

Table 5.1 shows how phase-separation can be improved by increasing centrifugal force and retention time. At six minutes retention time and centrifugal force of 7700 g the OWR is close to the original OWR of the mud. Original OWR of SAR-mud found by retort analysis is 37/63. At 2000 g high OWR is found, representing high oil content, while at 7700 g the OWR is lowered, giving higher water recovery.

In general, it is concluded not much gain by increasing retention time from one to six minutes. Centrifugal force is most important factor increasing separation, giving significantly better phase-separation and higher total liquid recovery when increased.

Results were taken into consideration for the main experiment. Alfa Laval's recommendations for swinger centrifuges, 3000 g's at 3 minutes, were also considered. Alfa Laval's values are industrial values and should provide good separation (Keller and Alfa Laval, 2014). A g-force of 3000 and 2 minutes retention time were concluded for further testing. Two minutes is considered a reasonable value, and higher retention time will demand higher energy- and time consumption for operation.

5.1.2 Destabilization and Separation

5.1.2.1 Screening of MudSplit Chemicals

- MudSplit001

Viscosity drop of both OBMs (Carbosea and SAR-mud) was observed by adding MudSplit001. Even at low concentrations of MudSplit001 the viscosity drop was clear, and it increased with increasing chemical concentration. Testing with MudSplit001 concluded to destabilize and give 3-phase separation of Carbosea OBM and SAR-mud at 0.60 % and 0.30 % vol/vol, respectively. SAR-mud was already degraded, hence lower dosing needed. Higher dosing of MudSplit001 gave better separation, and clearer phases compared to blank samples. SAR-mud samples were turbid and cloudy after centrifuge. By overnight settling clear 3-phase separation was achieved.

The effect seen by adding MudSplit001 was that solids "flipped" and became water-wet. At this point viscosity decreased, and breaking of the fluid was obvious. Observations for MudSplit001 may indicate presence of demulsifier. Demulsifiers can be added to destabilize and aid in water-wetting originally oil-wet solids of slop-mud (Ezell and Harvey, 2008).

- MudSplit002

Testing with MudSplit002 concluded not to be able to destabilize Carbosea OBM. MudSplit002, tested on SAR-mud, decreased viscosity of the mud; viscosity drop increased by introducing higher dosing of MudSplit002. SAR-mud was destabilized by MudSplit002 and 3-phase separation was enhanced compared to blank.

- MudSplit003

Testing with MudSplit003 concluded not to be able to destabilize Carbosea OBM. MudSplit003 tested on SAR-mud gave viscosity drop at increasing dosing. SAR-mud was destabilized by MudSplit003 with enhanced 3-phase separation compared to blank.

In general, best effect on Carbosea and SAR-mud obtained by MudSplit001 at 0.60 % and 0.30 % dosing, respectively.

The chemical composition of MudSplit chemical mixtures is unknown, what can be a reason for the poor results for MudSplit002 and 003.

5.1.2.2 Screening on SAS Chemicals

Dosing range for SAS chemicals was based on recommendations from Surface Active Solutions (Harrison, 2014).

- MudSplit004

Testing with MudSplit004 concluded not to be able to destabilize the OBMs. Viscosity decreased slightly at highest dosing prior to centrifuge, but only 2-phase separation observed after centrifuge for Versatec and Carbosea. For SAR-mud, 3-phase separation was similar to blank.

- MudSplit005

Viscosity decreases of the different muds at increased dosing of MudSplit005 prior to centrifuge. Testing on MudSplit005 concluded to be able to destabilize Carbosea and Versatec, however, successful 3-phase separation is achieved only at highest dosing, 2 vol/vol %. For Versatec 3-phase separation was not observed directly after centrifuge, but after overnight settling. For SAR-mud it could be seen that blank was more turbid than the samples with MudSplit005.

In general, for SAS chemicals best effect was observed by MudSplit005. Acceptable 3-phase separation was achieved for Versatec OBM, Carbosea OBM and SAR-mud at 2 vol/vol %. Viscosity decreases with increasing dosing of SAS chemicals. Results confirmed SAS chemistry. By adding SAS "*MudClean*" chemicals the viscosity decreases. The solids originally oil-wet turned water-wet and the fluid falls apart. The mud is destabilized and further allows the centrifuge to pull out as much of the solids as possible (Surface Active Solutions, 2013).

5.1.2.3 Screening on GSA Chemicals

Testing with chemicals from Global Solution concluded not to be able to destabilize the oil-based mud. The chemicals, being high molecular weight flocculating polymers, were not able to form aggregates of the colloidal particles and destabilize the mud. According to Stoke's law increasing the particles, or increasing the effective particle size with flocculation or coagulation can increase the separation (ASME, 2005). From (Lummus and Azar, 1986): "*Flocculants are polymers used for agglomeration of solids for removal by screening or settling. Flocculation is a method of removing colloidal-size drilled solids*". However, no effect was observed by testing Global Solution MudFloc chemicals on Carbosea OBM. The polymers from Global Solution were tested in the start-up phase of the project. A different strategy for testing was used during start-up, resulting in testing at a lower concentration range (max. 0.60 vol/vol %) than the other chemicals. The concentration range should, however, be sufficient according to industrial standards for the polymers (Keller, 2014). Overdosing of polymers can give unwanted viscosity increase which is undesirable for separation. However, only one mud was used for testing of Global Solution chemicals, and testing was not performed as extensively as for the other chemicals.

5.1.3 Effect of Temperature

The viscosity of SAR-mud decreased by heating, as expected. According to Stoke's law, decrease in viscosity (dependent on temperature) increases separation. After adjusting temperature and put into centrifuge, the same amount of total liquid as blank samples (also giving 3-phase separation) was recovered. However, phase separation was significantly improved, more water was recovered from the mud being heated. The phases were also less turbid after adjusting temperature. The results were as expected, increased separation by increasing temperature and consequently decreasing viscosity. High water content of the mud used for testing (SAR-mud) was more obvious in samples exposed to heating.

5.2 Main Experiment

5.2.1 Centrifugal Separation

Parameters set for centrifugal separation during main experiment were concluded sufficient to obtain acceptable results during testing of chemical mixtures on muds. By implementing the higher capacity *Hettich* centrifuge, test conditions were improved and more efficiently testing was performed during the main-experiment. Results were easier to observe due to larger volume bottles.

5.2.2 Destabilization and Separation

Effect of chemical mixtures on three different lab slop-muds (50/50) is discussed in following chapters.

5.2.2.1 Carbosea OBM (50/50)

5.2.2.1.1 MudSplit001

After mixing MudSplit001 and Carbosea OBM (50/50), solids «flipped» and became water-wet. Viscosity decreased dramatically and breaking of the fluid was obvious. Viscosity drop increased by introducing higher dosing, as expected. 3-phase separation of Carbosea OBM (50/50) was achieved at 0.5 vol/vol % MudSplit001. However, 0.5 % dosing (sample # 2) gave low water recovery compared to bottles with higher dosing. Water and solids remained emulsified in the oil-phase. This could simply be the result of scarce dosing, or incomplete reaction of chemicals and mud. Samples with higher dosing gave less oil, thus more water recovered and colloids separated from the oil phase. Results indicate that the oil-wet solids have been washed, turned water-wet and separated from the oil phase. Four phases were observed in sample # 4 (5.0 % dosing). There can be an optimal range for dosing of MudSplit001, and by exceeding this range, undesirable reactions/complexes can be formed. Mores testing should have been performed to confirm or disprove the result. Bottle # 3 and 4 gave turbid water phases, but after longer periods of settling the turbidity decreased as solids settled. According to industrial standards and from an economical point of view, lowest possible dosing is preferred. Based on results, optimal dosing of Mudsplit001 on Carbosea (50/50) is concluded close to 0.5 vol/vol %.

5.2.2.1.2 MudSplit002

By mixing MudSplit002 and Carbosea OBM (50/50), viscosity decreased only at highest dosing. No evident viscosity drop was observed and the chemical was not able to destabilize the mud. After centrifuging no effect was observed different from blank. MudSplit002 concluded not to be able to destabilize and give 3-phase separation of Carbosea OBM.

5.2.2.1.3 MudSplit003

After mixing MudSplit003 and Carbosea OBM (50/50) viscosity decreased at increasing dosing of Mudsplit003. Viscosity drop was clear for 2 % and 5 % vol/vol, resulting in breaking the stability of the mud. After centrifuging 3-phase separation was achieved for 2 % and 5 % vol/vol. Water recovery was higher for 2 % than 5 % dosing (Bottle # 3 and 4, respectively). However, the oil-phase at 2 % (Bottle # 3) contained emulsified water and oil-wet solids, not being washed. The oil phase is smaller with 5 % dosing, indicating that the solids have been washed and turned water-wet. The water-wet solids now form an additional phase on top of the dry, dense solid phase (Figure 4.12).

Results concluded that MudSplit003 destabilized and separated Carbosea OBM with optimal dosing in the range of 0.5 – 2.0 % vol/vol. Results show that dosing under 0.5 % vol/vol MudSplit003 (Bottle # 2) is insufficient to destabilize the mud. Further testing should be performed with intervals that are more frequent.

5.2.2.1.4 MudSplit004

Based on negative results from pre-screening and testing on Versatec OBM (50/50), MudSplit004 was not tested on Carbosea lab slop-mud (50/50) during main experiment.

5.2.2.1.5 MudSplit005

After mixing MudSplit005 with Carbosea (50/50), viscosity decreased with increasing dosing of MudSplit005. Viscosity drop was clear at 1 % and 2 % dosing, solids became water-wet and the fluid fell apart. After centrifuging, 3-phase separation was achieved at 1 % and 2 % dosing (Bottle # 3 and 4, respectively). Bottle # 3 shows destabilized mud with 3-phase separation, but the phases were not fully distinctive. Dosing might be scarce, or it might be a result of improperly mixing of chemicals and mud. Perfect 3-phase separation is found at 2 % vol/vol MudSplit005, clear distinct phases are observed with high water recovery. It is concluded successful destabilization and separation of Carbosea (50/50) by MudSplit005, with optimal dosing in the range of 1-2 vol/vol %.

Summary Carbosea OBM (50/50)

MudSplit001, MudSplit003 and MudSplit005 destabilized and gave 3-phase separation of Carbosea OBM (50/50). MudSplit001 gave 3-phase separation at lowest dosing (0.5 vol/vol %), compared to the other chemicals. MudSplit002 was concluded not to be able to destabilize the mud. MudSplit004 was not tested.

The chemical composition of MudSplit chemical mixtures is unknown, what can be a reason for the poor results for MudSplit002. Observations from Mudsplit001, 003 and 005 showed that solids "flipped" and became water-wet. Viscosity decreased, and breaking of the fluid was obvious. Observations for MudSplit001, 003 and 005 may indicate that these chemical mixtures can be (or can contain) demulsifiers.

5.2.2.2 Versatec OBM (50/50)

5.2.2.2.1 MudSplit001

After mixing MudSplit001 with Versatec OBM (50/50), solids "flipped" and became water-wet. Viscosity decreased with more evident viscosity drop for samples with highest dosing. The mud was destabilized and the fluid fell apart. Perfect 3-phase separation was observed for Versatec at 0.5 % vol/vol and higher dosing of MudSplit001. By increasing dosing from 0.5 % to 2.0 %, clearer water phase and enhanced phase-separation were obtained. Even clearer water phase was observed at 5.0 % dosing, however, flocks floating at the oil/water interface indicates exceeding optimal dosing. Results report on successful destabilization and 3-phase separation of Versatec OBM (50/50) by MudSplit001 at optimal dosing (lowest possible) in the range of 0.25 - 0.5 vol/vol %.

5.2.2.2.2 MudSplit002

By mixing MudSplit002 with Versatec OBM (50/50) viscosity decreased slightly at highest dosing (5.0 %) of chemical mixture, but the chemical was not able to destabilize the mud. After centrifuging, no effect was observed different from blank. MudSplit002 was concluded not to be able to destabilize and give 3-phase separation of Versatec OBM (50/50).

5.2.2.2.3 MudSplit003

After mixing MudSplit003 with Versatec OBM (50/50) viscosity drop was observed for 2.0 % and 5.0 % vol/vol, and destabilization of the mud was achieved. After centrifuging, 3-phase

separation was obtained for 2.0 % and 5.0 % vol/vol. The two samples (Bottle # 7 and 8, respectively) gave equal results, with low water recovery and large amounts of water and solids emulsified into the oil-phase. The water phase appears turbid, what can be explained by oil stuck on centrifuge bottle. Solids remain in the oil-phase because of insufficient washing, thus not turned water-wet. The relatively poor separation with contaminated oil phase can be a result of scarce dosing and incomplete reaction of chemicals and mud, or insufficient mixing of chemicals and mud. Shear force during mixing was found influential for results. It was concluded that MudSplit003 destabilized and separated Versatec OBM (50/50) at dosing higher than 2.0 % vol/vol. Further testing should be performed with intervals being more frequent.

5.2.2.2.4 MudSplit004

By mixing MudSplit004 with Versatec OBM (50/50), viscosity decreased only at 2.0 % vol/vol dosing. No evident viscosity drop was observed and the chemical was not able to destabilize the mud. After centrifuging no effect was observed different from blank, only minor change in color of sediments was observed at 2.0 % dosing of MudSplit004 (Bottle # A4). This gives the impression that oil, washed off the originally oil-wet solids, is now stuck on the centrifuge bottle. MudSplit004 was concluded not to be able to destabilize and give 3-phase separation of Versatec OBM (50/50).

5.2.2.2.5 MudSplit005

After mixing MudSplit005 with Versatec OBM (50/50), viscosity decreased with increasing dosing of MudSplit005. Viscosity drop was obvious at 1.0 % and 2.0 % dosing, solids became water-wet and the fluid was destabilized. After centrifuging, 3-phase separation was achieved at 1.0 % and 2.0 % dosing (Bottle # C and D, respectively). Bottle # D gave enhanced 3-phase separation compared to # C, related to higher dosing of MudSplit005. Bottle # C (1.0 % dosing) shows destabilized mud with 3-phase separation, but the oil phase contains emulsified water and solids. The water phase is clear, but appears more turbid than actual due to oil stuck on centrifuge bottle. At 2.0 % dosing (Bottle # D), solids are washed, become water-wet and separate from the oil phase. Also, water phase is clear in Bottle # D with sufficient dosing of chemicals to wash oil from centrifuge bottle. Perfect 3-phase separation is found at 2.0 % vol/vol MudSplit005 where clear distinctive phases and high water recovery are observed. It was concluded to be successful destabilization and separation of Versatec (50/50) by

MudSplit005. Optimal dosing is in the range of 0.5 - 1.0 % vol/vol, being lowest possible. Further testing should be done with more intense intervals.

Summary Versatec OBM (50/50)

MudSplit001, MudSplit003 and MudSplit005 are concluded to destabilize and give 3-phase separation of Versatec OBM (50/50). Best effect was observed by MudSplit001 and 005, while MudSplit003 required higher dosing to destabilize the mud. MudSplit002 and MudSplit004 were concluded not to be able to destabilize the mud. Pre-screening included testing of only MudSplit004 and MudSplit005 on Versatec OBM (base mud), whereby just MudSplit005 destabilized and separated the mud. So, MudSplit005 proved overall successful.

5.2.2.3 Carbosea LC-LUBE Fine Premix OBM (50/50)

5.2.2.3.1 MudSplit001

After mixing MudSplit001 with Carbosea LC-LUBE Fine Premix OBM (50/50) viscosity decreased already at lowest dosing (0.25 % vol/vol). The viscosity drop increased by introducing higher dosing of chemical mixture. Solids became water-wet and the mud was destabilized. After centrifuging Bottle # 1, containing 0.25 % dosing, appeared to differ from blank. Minor changes in sediments and colors of the mud were observed, but the mud was not destabilized. Perfect 3-phase separation was achieved for 0.5 % and 0.75 % vol/vol MudSplit001. Different from the other OBMs, the recovered water phases from Carbosea LC-LUBE Fine Premix appeared white. This can be explained by presence of macro-emulsion, as most macro-emulsions scatter light strongly and appear white and opaque (G. Barnes, 2011). Results report on successful destabilization and separation of Carbosea LC-LUBE OBM (50/50) by MudSplit001 at optimal dosing in the range of 0.25 - 0.5 % vol/vol.

5.2.2.3.2 MudSplit005

By mixing of MudSplit005 and Carbosea LC-LUBE (50/50) viscosity drop was intense at 1 % and 2 % dosing. Solids were washed, became water-wet and the fluid was destabilized. After centrifuge the bottles containing 1.0 % and 2.0 % vol/vol dosing showed perfect 3-phase separation. High water recovery was observed for both samples. Equal results were obtained, except for some oil stuck on centrifuge bottle at 1.0 % dosing, while 2.0 % shows enough dosing to wash oil from centrifuge bottle. Results report on successful destabilization and

separation of Carbosea LC-LUBE Fine Premix OBM (50/50) by MudSplit005 at 1.0 % vol/vol.

Summary Carbosea LC-LUBE Fine Premix OBM (50/50)

Both chemicals report on successful 3-phase separation. MudSplit005 gave clearer water phases than MudSplit001, but required higher dosing. For all samples white/grey powder was observed on top of the oil phase[7]. Wider dosing range, especially lower concentrations of MudSplit005, needs to be tested. However, low volumes of mud put limitations on testing of Carbosea LC-LUBE. No pre-screening was done due to late arrival of Carbosea LC-LUBE Fine Premix OBM, thus there are no results for comparison.

5.2.3 Effect of Temperature

According to Stoke's law, lowering the viscosity of the mud will improve separation. The results confirmed that increase in temperature (viscosity drop) gives increased separation. The viscosity of Versatec OBM decreased by heating, as expected. After centrifuging 3-phase separation was achieved at 40, 60 and 80 °C. For 20 °C only 2-phase separation was achieved, while 40 °C showed 3-phase separation with not distinctive phases. At 60 and 80 °C 3-phase separation with distinctive phases was obtained, however, there is no trivial difference in separation between these two temperatures. Slightly turbid water phases were observed for samples of 60 and 80 °C due to oil stuck on centrifuge bottles.

Acceptable 3-phase separation was achieved by increasing temperature only, without addition of chemicals. It can be concluded that for successful 3-phase separation the temperature must exceed 40°C.

Results from adjusting temperature are not considered fully precise due to methods of testing. Temperature dropped rapidly after transferring the mud into centrifuge bottles and during centrifuging.

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[7] LC-LUBE™ Fine is the material name of synthetic graphite, an additive applicable in WBM, SBM, or OBM. It appears as grey colored, solid powder (Baker Hughes, 2006 b).

5.2.4 Separation Efficiency

The liquid phases from the three samples settled for some days prior to analysis. However, after this period of time the samples still contained impurities in the oil- and water phases, indicating that stable emulsions were present. Analytical work on separation efficiencies was based on a small number of samples.

5.2.4.1 Oil-Phase

The oil phases proved to be stable emulsions with high water content and some solids emulsified in the oil phase after settling over time. Some water is emulsified into the oil phase due to emulsifiers and solids helping to stabilize emulsions. However, the water content for Versatec OBM (50/50) oil phase was very high and concluded to be imprecise. This can be explained by accidentally including water during pipetting of oil into the retort-cup.

5.2.4.2 Solid-Phase

Results from solid phases showed evident oil- and water content. Prior to mixing, the solid phases were dense and dry at the bottom. However, oil and water were present at the solid surfaces and became included into the retort analysis. Thus, results can give the impression of being misleading regarding solid dryness, due to presence of liquid.

5.2.4.3 Water-Phase

Prior to analysis water phases appeared relatively transparent, though a little yellow. Figure 4.22 shows more yellow water phases in bottles with higher concentration of OiW, as expected. Consequently, it can be assumed that the water phases were stable emulsions, still containing emulsified oil after over time settling.

Content of solid phases and oil-phases (due to uncertain data) led to high concentrations of OiW, estimated theoretically from mass-balance, thus concentrations of OiW were concluded imprecise.

Additionally, OiW concentrations were validated by an external accredited laboratory, Intertek West Lab, which results were concluded accurate and realistic. OiW concentrations found by Intertek confirmed theoretical concentrations unrealistic. Deviations of Intertek analysis are given in the laboratory report (see Appendix E).

5.3 Retort Analysis

- For *Carbosea OBM* an OWR of 80/20 was found, what seems realistic value related to information on Carbosea being a low toxicity mineral oil-based mud, emulsion drilling fluid (Baker Hughes, 2014).

- For *Carbosea LC-LUBE Fine Premix OBM* the oil-water ratio was very low compared to OWR range[8] for oil-base muds. Arild Fjogstad, Baker Hughes, confirmed the mud being oil-based, but no information was given on OWR (Fjogstad, 2014). The result can be explained by accidentally performing the retort analysis on lab slop-mud (50/50) of Carbosea LC-LUBE, instead of on base mud.

- For *Versatec OBM* an OWR of 76/24 was found, proving information on Versatec OBM, being a low toxicity mineral oil with emulsified brine as internal phase, and average OWR of 74/26 (Rapp, 2009).

- OWR for *SAR-mud* was found to be lower than normal OWR range for OBMs. The result is concluded realistic, since SAR-mud is a mixture of different OBM wastes. These wastes are put on tank and stored for longer periods onshore, thus partly biodegraded. Also, the oil-based muds are contaminated with water, crude, hydraulic oil, detergents, rust, grease etc., confirmed by high water content and darker oil phase observed in the graduated cylinder (Figure 4.23).

Emulsions at the oil-water interface were observed in the graduated cylinder after retort analysis of all the muds, confirming oil-based emulsion muds. Retort analysis was concluded an effective and successful method determining oil, water and solid content of each mud.

Retort analysis for Carbosea LC-LUBE Fine Premix OBM was not repeated due to low volume delivery of mud.

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[8] OWR of oil-based mud is normally in the range of 60/40 to 90/10.

5.4 Pre-Screening and Main-Experiment Comparison

Results found during pre-screening were reproducible during main-experiment. Chemicals, proved to be effective on destabilizing and separating the mud during pre-screening, also proved to be effective during main-experiment. However, dosing required for 3-phase separation was higher during pre-screening. Pre-screening included testing on base-muds whereas the main-experiment included lab slop-muds for testing. Lab slop mud has lowered OWR compared to uncontaminated OBM, thus the stability of the emulsion slop-mud decreases, making the mud easier to separate (McCosh et al., 2009).

5.5 Standard Deviations

During pre-screening the *Sorvall RC-5B* centrifuge caused standard deviations due to being old. Settings were adjusted manually. Indicators showing RPM and time were not always steady during operation of the centrifuge, probably impacted by vibrations.

Results from centrifugal separation in pre-screening, presented graphically in Chapter 4.1.1, are given by average numbers. Deviations are given in Table 5.2 and 5.3.

Table 5.2 *Deviation for centrifugal separation at 2000 g.*

2000 G		Sample a)	Sample b)	Average	Deviation
1 min	oil	5.50	5.50	5.50	*0.00*
	H_2O	3.00	3.00	3.00	*0.00*
3 min	oil	7.50	6.00	6.75	*1.06*
	H_2O	2.00	3.50	2.75	*1.06*
6 min	oil	6.75	5.75	6.25	*0.71*
	H_2O	3.00	4.00	3.50	*0.71*

Table 5.3 *Deviation for centrifugal separation at 7700 g.*

7700 G		Sample a)	Sample b)	Average	Deviation
1 min	oil	10.25	9.75	10.00	*0.35*
	H_2O	2.00	2.00	2.00	*0.00*
3 min	oil	5.00	5.75	5.38	*0.53*
	H_2O	8.00	7.50	7.75	*0.35*
6 min	oil	6.00	6.00	6.00	*0.00*
	H_2O	8.00	8.00	8.00	*0.00*

5.6 Obstacles of Laboratory Testing

The *Hettich Rotofix 46* centrifuge, originally intended for the project, was delayed by post-delivery for several weeks. Thus, the lower capacity *Sorvall RC-5B* centrifuge was used during pre-screening, and the main experiment did not start until the *Hettich Centrifuge* arrived.

6 Conclusions

The main objective of this study was to destabilize and separate different contaminated oil-based muds into its original phases, by utilizing different destabilizing chemicals and a lab centrifuge. The study performed indicated satisfying results to convert viscous oil-based mud waste into oil, water and solids.

- Pre-screening concluded on five destabilizing chemicals for further testing. MudSplit001 reported on best effect during pre-screening with respect to dosing.

- Evaluations of centrifugal force and retention time for centrifugal separation reported on minor improvements by increasing retention time from one to six minutes. Increasing centrifugal force from 2000 to 7700 g gave significantly improved separation efficiencies. Operational parameters for centrifugal separation concluded on two minutes retention time and centrifugal force of 3000 g for further testing.

- Results from the main experiment reported on destabilization and successful three-phase separation of the different oil-based lab slop-muds by MudSplit001, 003 and 005. MudSplit001 and MudSplit005 proved most effective with respect to dosing. Overall, MudSplit001 concluded most effective on destabilizing and separating the mud even at 0.5 % dosing.

- Factors, as mud composition, chemical dosing, shear force when mixing mud and chemicals, temperature adjustments, centrifugal force and retention time, were critical to the process of destabilizing and separating the mud waste.

  Higher dosing of chemicals resulted in enhanced phase-separation with clearer phases. However, for some samples it could be seen that exceeding optimal dosing range gave undesirable products and poorer separation.

  Heating of mud gave increased phase-separation. Higher temperature exposure of the mud gave better separation, observed both in pre-screening (SAR-mud) and during main-experiment (Versatec OBM). Temperatures above 40 °C provided satisfying 3-phase separation.

  A clear correlation was found between viscosity drop and phase separation of mud (both during pre-screening and main-experiment), obeying Stoke's law.

Shear force during mixing chemicals and mud proved important for results. Chemicals can be sensitive to over-mixing, applying too much shear caused separated water to re-emulsify back into the slop-mud.

Scarce dosing of chemicals and overdosing of chemicals, as well as too high shear force for mixing resulted in poor phase separation.

- Representative samples with good result of 3-phase separation at lowest possible dosing of chemicals were analyzed for separation efficiency. Each of the samples contained different oil-based muds with same dosing of chemical mixture, 0.5 % vol/vol MudSplit001. Separation efficiency was reported through concentrations of oil in water, found to be 120, 58 and 16 mg/L for Carbosea (50/50), Carbosea LC-LUBE Fine Premix (50/50) and Versatec (50/50) respectively.

- According to the purpose, the study was concluded successful, and laboratory testing was concluded effective and satisfactory for obtaining observable and good results. Further studies on optimizing are recommended.

7   Further Recommendations

Based on recommendations from the supplier the experiment for this research was performed quite broadly with respect to chemical concentrations. The following recommendations are suggested for further studies:

- From an academically point of view, dosing should be performed more consistently overall.
- For more representative data extended analytical work on separation efficiency is recommended.
- Temperature testing can be performed more comprehensively. An option is water baths to maintain a steady temperature of samples prior to centrifuging, in addition to adjusting temperature digitally in the centrifuge to maintain a steady temperature during centrifugal separation.
- Retort analysis should be repeated on Carbosea LC-LUBE Fine Premix OBM to confirm or disprove the OWR.
- A combination of coagulants with the flocculating polymers from Global Solutions can be tested.
- Testing of lab slop mud prepared by fresh water and mud in a ratio of 1:1 for destabilization can be done.
- Destabilization and separation of WBM and SBM should be tested.
- Detailed pH testing with respect to base and acid should be performed.

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APPENDIX

Appendix A: Centrifugal Separation

RPM/RCF conversion

$$RCF = \left(\frac{RPM}{1,000}\right)^2 \times r \times 1.118 \quad \Leftrightarrow \quad RPM = \sqrt{\frac{RCF}{r \times 1.118}} \times 1,000$$

RCF = relative centrifugal force

RPM = rotational speed (revolutions per minute)

r   = centrifugal radius in mm = distance from the center of the turning axis to the bottom of the centrifuge.

Figure 0.1 *Conversion formula, RPM vs. RFC (Hettich, 2014).*

Table 0.2 *RPM (Revolutions per Minute) to RCF (Relative Centrifugal Force), parameters relevant for testing.*

Pre-screening		Main experiment	
RCF	RPM	RCF	RPM
2000	4089		
3000	5008	3000	3938
7700	8023		

Raw Data from Pre-Screening: Variations in Retention Time and G-Force

Two bottles of 30 mL mud samples (SAR-mud) put in the centrifuge.

Table 0.3 *Raw data from centrifugal separation, run at 2000 RCF.*

		1 min	3 min	6 min
Sample 1	**total liquid**	8.50	9.50	9.75
	H2O	3.00 *	2.00	3.00
	Oil	5.50	7.50	6.75
Sample 2	**total liquid**	8.50	9.50	9.75
	H2O	3.00	3.50	4.00
	Oil	5.50	6.00	5.75
Average	**total liquid**	**8.50**	**9.50**	**9.75**
	H2O	**3.00**	**2.75**	**3.50**
	Oil	**5.50**	**6.75**	**6.25**

*oil on cylinderwall. only 1 mL clear water phase

Table 0.4 *Raw data from centrifugal separation, run at 7700 RCF.*

		1 min	3 min	6 min
Sample 1	**total liquid**	12.25	13.00	14.00
	H2O*	2.00	8.00*	8.00
	Oil	10.25	5.00	6.00
Sample 2	**total liquid**	11.75	13.25	14.00
	H2O	2.00	7.50	8.00
	Oil	9.75	5.75	6.00
average	**total liquid**	**12.00**	**13.13**	**14.00**
	H2O	**2.00**	**7.75**	**8.00**
	Oil	**10.00**	**5.38**	**6.00**

* 5 mL clear water phase

Appendix B: Retort Analysis - Vol % Calculation Formulas

$$\% \text{ oil} : \frac{vol \% oil}{vol \% oil + vol \% water} = x$$

$$\% \text{ water} : \frac{vol \% water}{vol \% oil + vol \% water} = y$$

$$\text{OWR} = \frac{x}{y}$$

OWR = Ratio of the volume percent oil to the volume percent water in an oil mud. OWR is calculated directly from the retort analysis of an oil mud (Garrett, 2014h).

Appendix C: Oil-in-Water, Mass-Balance

Figure 0.5 – 0.7 gives OiW for Carbosea, Carbosea LC-LUBE and Versatec. Explanations of numbers (in red color) are given in Figure 0.8.

CARBOSEA	1 Basemud	2 50/50 lab slop mud	4 Oil phase	4 Solids phase		Water phase
%oil	61	30,5	65	39		10,4 %
%water	15	57,5	32	8		
%solids	24	12	3	53		
tot=	100	100				7
		3	5	5		
oil (mL)		61,3	39,8	9,4	6	12,0
water (mL)		115,6				
solids (mL)		24,1				

Figure 0.5 *Mass-balance giving percentage oil-in-water for Carbosea (50/50) with 0.5% dosing of MudSplit001.*

Carbosea LC-LUBE	1 Basemud	2 50/50 lab slop mud	4 Oil phase	4 Solids phase		Water phase
%oil	31	15,5	76	37		1,9 %
%water	57	78,5	14	17		
%solids	12	6	10	46		
tot=	100	100				7
		3	5	5		
oil (mL)		31,2	23,7	4,5	6	3,0
water (mL)		157,8				
solids (mL)		12,1				

Figure 0.6 *Mass-balance giving percentage oil-in-water for Carbosea LC-LUBE (50/50) with 0.5% dosing of MudSplit001.*

Versatec	1	2		4	4			
	Basemud	50/50 lab slop mud		Oil phase	Solids phase		Water phase	
%oil	60	30		30	37		28,9 %	
%water	21	60,5		64	23			
%solids	19	9,5		6	40			
tot=	100	100						
							7	
		3		5	5			
oil (mL)		60,3		18,1	7,1	6	35,1	
water (mL)		121,6						
solids (mL)		19,1						

Figure 0.7 *Mass-balance giving percentage oil-in-water for Versatec (50/50) with 0.5% dosing of MudSplit001.*

1. Base mud analyzed by retort					
2. Mass-balance for 50/50 slop mud					
3. Sample of 200 mL slop mud (50/50) + 0,5% mudsplit001,				201	mL
Should give following volumes (mL)					
4. Oil and solids phases are analyzed by retort					
5. Oil (mL)of each phase is found					
6. Remaining oil (mL) in water phase is found					
7. Percentage oil-in-water					

Figure 0.8 *Explanations for Figure 0.5 – 0.7.*

Appendix D: Synthetic Seawater

Recipe for the synthetic seawater used for mixing with OBM in making lab slop-mud is given in Table 0.9.

Table 0.9 *Synthetic seawater, by Brujewicz, Subow, 1931, (UCP, 1982-2004).*

Salt	g/kg
$NaCl$	26.518
$MgCl_2*6H_2O$	2.447
$MgSO_4$ anhydrous	3.305
$CaCl_2$	1.141
KCl	0.725
$NaHCO_3$	0.202
$NaBr$	0.083

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cc:

Vår ref:	2014-04306
Utgave	1
Dato:	05.jun.2014
Side:	1 av 3
Deres ref:	olje i vann

Laboratorierapport

Hensikt:	Analyse av olje i vann.
Prøvested:	n/a
Prøve tatt av:	Norwegian-Group AS
Mottatt dato:	03.jun.2014
Analysert:	05.jun.2014

Ved spørsmål angående denne rapporten, ta kontakt med undertegnede.

Med hilsen
Intertek West Lab AS

Teknisk ansvarlig

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P.O. Boks 139, N-4098 TANANGER, Besøksadresse: Oljevn. 2, N-4056 TANANGER
Testresultatene relateres bare til prøvene som er testet. - Rapporten kan ikke reproduseres, utenom i sin helhet, uten skriftlig godkjennelse fra laboratoriet.

Intertek

Laboratorierapport

Prøveinformasjon

Prøve nr	Prøvepunkt	Prøve tatt dato
2014-04306	n/a	
-001	Prøve 1 Carbosea + 0.5 % Mudsplitt 001	03.jun.2014
2014-04306	n/a	
-002	Prøve 2 Carbosea Lube + 0.5 % Mudsplitt 001	28.mai.2014
2014-04306	n/a	
-003	Prøve 3 Vergatec + 0.5 % Mudsplitt 001	03.jun.2014

Resultater

Komponent	Enhet	001	002	003
Olje i vann (C7-C40)	mg/l	120	58	16

Tegnforklaring: " = ikke akkreditert analyse
(n) = Antall replikater rapportert hvor n er replikat nummer.

Kommentarer

Prøve nr.
   Prøve kommentar

VIII

Intertek

Laboratorierapport

Metode referanse

Parameter	Enhet	PKG Nedre	PKG Øvre	Metode	Standard	Usikkerhet
Olje i vann (C7-C-40), GC/FID						
Olje i vann (C7-C40)	mg/l	0,4		M-039	Mod. NS-EN ISO 9377-2 / OSPAR 2005-15	±15% / ±0,2

Tegnforklaring: * = Ikke akkrediterte analyser, PKG = Praktisk kvantifiseringsgrense. # = Analysen er utført av underleverandør.

Usikkerheten er angitt med 95% konfidensintervall. Der det er oppgitt både relativ og absolutt usikkerhet gjelder det argumentet som til enhver tid representerer størst usikkerhet.

Vår ref.: 2014-04306
Utgave: 1
Rapportert dato: 05.jun.2014
Rapportformat: RL-06
Kontrollert av: Anny Yrke
Side: 3 av 3

IX