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Evaluation of Using Coagulant for Treatment of Highly Viscous Slop Water Containing Xanthan Gum

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Abstract

Exploration and development drilling activities in offshore Oil and Gas industry generates waste streams, which slop water are among the significant wastes. Slop water is formed when wastewater originating from multiple sources becomes contaminated with oil/synthetic-based drilling fluid components and have to be treated or disposed prior to discharge due to the strict regulations in Norwegian Continental Shelf, NSC. The slop water is collected in tanks where it is contaminated with different components which cause varying composition. The different contaminants are mainly in the from Oil-in-Water (O/W) emulsions, which may cause problem in to stay in compliance with discharge limits, 30 mg oil per liter of water. In some instance the varying slop can be more complex to treat, one such instance is high viscous slop water. The cause of high viscous slop has been identified as slop containing Xanthan Gum (XG). The main focus of this study is on evaluating treatment methods for complex slop water containing XG. The understanding of polymers applied offshore for different purpose has been important for the performance of this study. Furthermore, analysis of possible slop water treatment technologies and combination of them used offshore and onshore is carried out in this study.

A major research based on literature study is presented in this thesis to support the experimental study. In the experimental part chemical treatment combined with centrifugation is evaluated as a representative for high viscous slop water. The results of the experimental part showed that Nature NIC and Nature NSC coagulants combined with centrifugation were an alternative for treatment of high viscous slop and increased the quantity of water recovered. Dilution of $XG_{(aq)}$ with slop or seawater had a significant effect on the viscosity of $XG_{(aq)}$, in addition to increased separation of water from $XG_{(aq)}$ which was obtained. Increased addition of NaOH increased the separation of water from $XG_{(aq)}$.

Dilution of $XG_{(aq)}$ with 75 % seawater combined with 2.0 ml/L of NaOH and 4.0 ml/ L coagulants and centrifuged were chosen as potential solution for the high viscosity of slop water, 116.40 g of water was separated from $XG_{(aq)}$ and sample dried in oven showed 96.5 % water hold by XG floc.

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Stavanger, June 2018

Hirra Ejaz

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Abbreviations

BAT – Best Available Technology

CMC – Sodium Carboxyl Methyl Cellulose

COD – Chemical Oxygen Demand

CTU – Compact Treatment Unit

DAF – Dissolved Air Flotation

EC – Electrocoagulation

EOR – Enhanced Oil Recovery

GG – Guar Gum

HEC – Hydroxyl Ethyl Cellulose

HOCNF – Harmonized Offshore Chemical Offshore

LCM – Lost Circulation Material

MF – Microfiltration

NCS – Norwegian Continental Shelf

NF – Nanofiltration

NPD – Norwegian Parliament Directorate

OBM – Oil Based Mud

O.E – Oil equivalent

O/W – Oil-in-Water

PAC – Polyaluminium Chloride

PEC – Predicted Environmental Concentration

PLONOR – Pose Little or No Risk to the Environment

PNEC – Predicted No Effect Concentration

PSA – Petroleum Safety Authorities

RO – Reverse Osmosis

SBM – Synthetic Based Mud

STU – Sludge Treatment Unit

TOC – Total Organic Content

XG – Xanthan Gum

1. Introduction

In oil and gas exploration industry the focus of attention is the drilling wastes due to the rigorous environmental rules and regulations moving towards zero discharge. The drilling activities offshore generates significant volume of waste, which includes, produced water, drilling fluids and cuttings, slop water and solid wastes (Ivan & Dixit, 2006). The focus in this study is on slop water and problem associated with it. Slop water is known as wastewater contaminated with invert-emulsion drilling fluid. Slop water generated varies due to varying drilling conditions from one drilling operations to another. Cleaning of rig, boat, deck floor, storage tanks, and shale shakers can also generate slop water, and a mix of this form a slop emulsion. Varying mix of slop water typically contain weighting agents, fluid-loss additives, viscosity regulators, and other chemicals.

Different problems are encountered due to complex mix of slop water. One such problem met is high viscous slop. A study by McCosh et al. (2009) reported that high viscosity cause the particles and other hazardous components to stay in suspension, which cause difficulties in treatment of slop and to meet the strict regulations (McCosh, Kapila, Dixit, Way, & Phipps, 2009). The aim of this thesis is to find a fast and effective process of decreasing the high viscosity of slop water and separate excess water. To achieve this, an increased understanding of the process was required and laboratory research was carried out. The treatment of high viscous slop water may be done using traditional water treatment technologies, such as addition of coagulants, flocculants and other technologies before discharged to sea or sewer.

1.1 Statement of the problem

Drilling activity generates multiple waste streams, one of the major liquid fraction is often referred to as “slop water”. These waste streams are by products of cleaning deck floor, shaker room, pump room, pit cleaning and other areas where spillage and interfaces during displacement occur. It can either be treated, discharged or disposed depending on the composition of the slop water, usually it is treated before it is discharged or disposed. Different rig configuration and operation practice influence greatly the composition of the produced slop water and in some instances the slop can be more complex to treat, one such instance is high viscosity slop. This thesis has been a part of Nature Oil & Gas AS project, where the focus has been to treat Xanthan Gum solution which cause high viscous slop. Different chemicals has been tested to see which of them is more efficient, and how efficient they decreases the viscosity of slop water solution. The following questions were answered under this project:

- ✓ Which chemicals have an effect on Xanthan Gum solution?
- ✓ Does a combination of coagulants have an effect on Xanthan Gum solution?
- ✓ How does water separate from Xanthan Gum solution?
- ✓ Does increased pH have any effect on the separation of water from Xanthan Gum solution?
- ✓ Which effect does dilution of Xanthan Gum solution have on the separation of water?
- ✓ Which parameter are optimal for decrease of viscosity of Xanthan Gum and separation of water?

1.2 Objectives

The aim of this thesis has been to find a potential treatment method for decreasing the viscosity of slop water. The objective of the thesis is to study the problem of high viscous slop generated in the offshore Oil and Gas industry, and find optimal solution for its treatment. The industrial partner for this thesis, Nature Oil and Gas AS, is a slop treatment company located at Stavanger, Norway, which has encountered issues with treatment of high viscous slop water. The cause has been identified as slop containing Xanthan Gum. The purpose of the master thesis, in this regard is to find a solution for the existing problem of high viscosity slop water using Nature Oil and Gas laboratory. The objective of finding a solution, was achieved by identifying and fulfilling the following steps:

- To understand and describe possible slop water treatment technologies used offshore and onshore, which also include a number technologies utilized by Nature Oil & Gas AS
- Research the buildup of polymers, specifically the composition containing Xanthan Gum
- To determine potential solutions to the problem of high viscosity of slop water
- To determine and evaluate the effect of different concentrations of coagulants and pH applied on high viscous slop water
- To choose the optimal dosage of chosen coagulants and pH

It was also found necessary to examine waste produced from the Oil & Gas industry and get an overview of the related laws and regulations. In addition, a review of the use and buildup of polymers in the oil & gas industry, specifically the use of Xanthan Gum was examined.

1.3 Nature Oil & Gas

This study was performed in cooperation with Nature Oil & Gas AS. The company was founded in 1999 and their headquarters are in Stavanger. Nature Oil & Gas is an innovative group with specialization in the treatment of offshore liquid waste and utilizes proven and reliable technologies. They provide advice to companies with solution to the problem of treating and removing oil-containing slop water at source. The company has utilized a technology that allows the treatment of slop water in the site of operation, this includes offshore installation, a drilling or production ship or even an industrial facility onshore. Technologies utilized by Nature Oil & Gas AS have small footprints and all are manufactured in accordance with NORSOK Z-015 standard and approved for ATEX II/3G.

In this study, treatment of high viscosity of slop water was conducted, which has been one of the challenges Nature Oil & Gas have met.

1.4 Thesis Structure

This master thesis is divided into eight chapters, where chapter 1 gives an introduction regarding this study of high viscous slop. Chapter 2 provides background information, an overview of oil and gas industry in Norwegian Continental Shelf and waste associated with this industry. The source of the waste from offshore industry and Norwegian rules and regulations of handling this waste is also discussed in this Chapter. A description of slop water and the treatment technologies available offshore and onshore are presented in Chapter 3, with focus on chemical treatment. Chapter 4 presents a description of different polymers, with focus on Xanthan Gum and the use of them in the offshore industry. Approaches, materials and methods are described in Chapter 5. Chapter 6 presents the results and discussions from the tests conducted in Chapter 5. The conclusion and recommendations made for the study are given in Chapter 7 and 8.

2. Theoretical Background

This chapter present an overview of the Oil and Gas industry in Norwegian Continental Shelf (NSC) and waste associated with this industry. Further, Norwegian rules and regulations for discharges of oily wastewater from offshore activity are discussed and the Environmental Impact Factor, EIF. In the end, Oil-in-Water (O/W) emulsion is presented and the waste generated from exploration and production drilling.

The basic understanding of this chapter is important for the experimental part of the thesis. Exploration and production drilling in Oil and Gas industry in NSC generates a lot of waste, such as produced water and slop water. One of the challenges associated with slop water during drilling is O/W emulsion. It is therefore important to understand the O/W emulsion and how to break this emulsion to separate the oil. The wastewater containing oil, hazardous chemicals and other pollutants are not allowed to discharge due to strict rules and regulations. These rules and regulation includes a reduction on the environmental impact from produce water release to “zero effect release”. To achieve this, EIF has been developed.

2.1 Oil and Gas Industry – Norwegian Continental Shelf

The NCS is a relatively young Oil and Gas province compared to the UK, US and other areas, such as the Middle East region. The first discovery was made in 1969 of the coast of Norway, which later became the Ekofisk field. In the subsequent years a number of discoveries were made and a new and important industry was founded in Norway. In the development of Norwegian oil and offshore industry, Norwegian government has played an active and important role. They have characterized strategies for resource management and industrial development by gradualism (Mohn, 2008).

As seen from *Figure 1* reservoir usually contain of three layers in varying amount. Produced water increase as the production time increase. Oil content decreases with time and to maintain the reservoir pressure produced water, sea water or other types of water are injected.

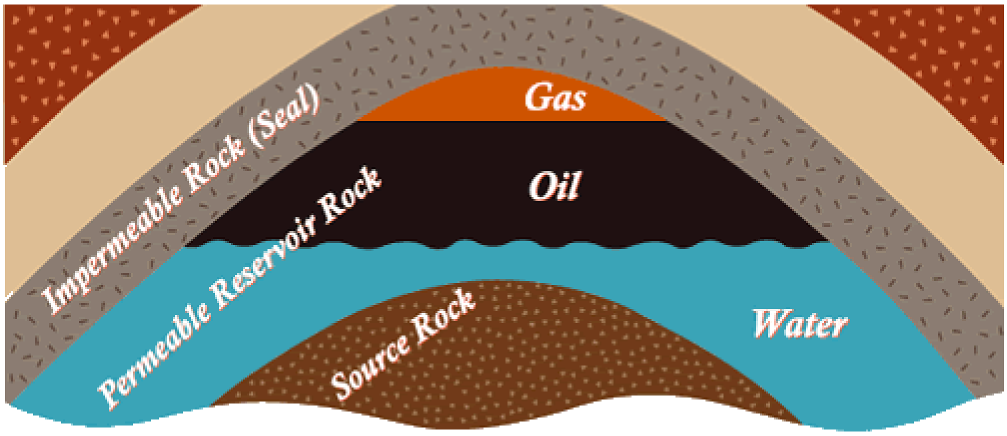

Figure 1: Typical Gas, Oil and Water reservoir (Source Rock as a Reservoir, a personal odyssey, 2018

From the Oil and Gas activity on the NCS there are produced; gas, natural gas liquids, condensate and oil. In 2017, the total production increased for the fourth straight year, leading to a sale of 236.4 million Sm^3 O.E. (Norwegian Petroleum Directorate, 2018). This is 2.7 % more than in 2016, as seen in *Figure 2*. The production from different reservoirs also contains oil, gas and water in various combinations. The production stream from the reservoirs must be separated and treated to get marketable products. The hydrocarbon composition, and volume of produced water varies from reservoir to reservoir and over time (Norsk Petroleum, 2018). The sale of petroleum was record-breaking in 2004, where 264.1 million Sm^3 was sold as seen in *Figure 2*. The production is expected to increase the next years.

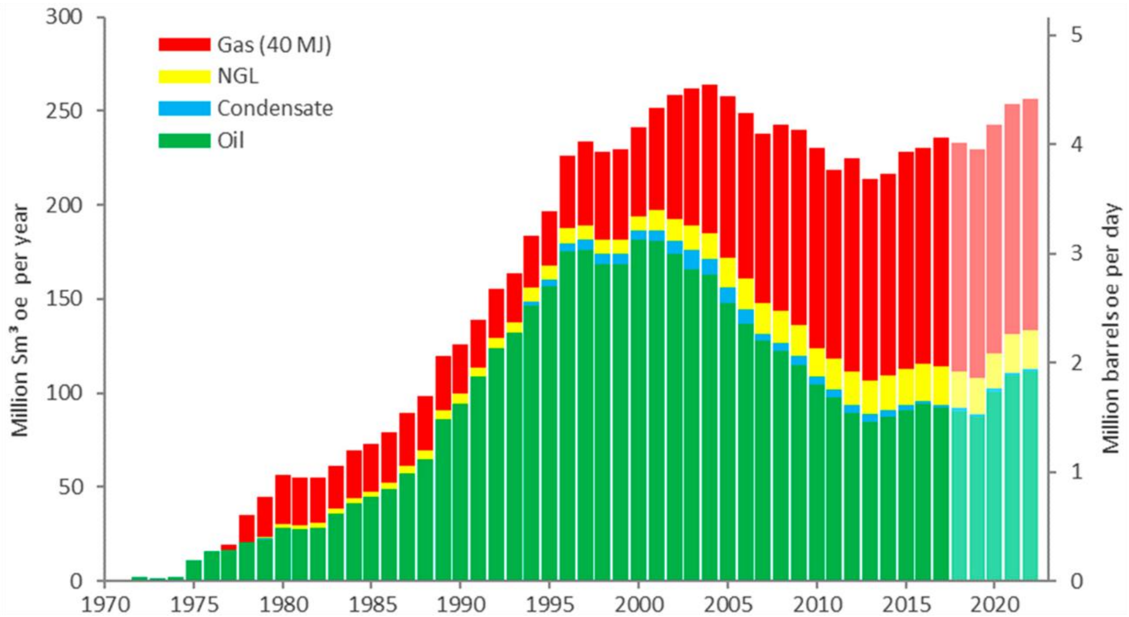

Figure 2: Actual and projected sale of petroleum 1971-2022 (Norwegian Petroleum Directorate, 2018)

Crude oil is composed of different types of hydrocarbons, where the specific composition varies from field to field. The composition of crude oil also determines how light or heavy (viscous) the oil is (Norsk Petroleum, 2018). The quality of the oil depends on what and how much it contains of other substances than hydrocarbons, such as wax, asphaltenes and Sulphur. These components can potentially also be present as a contaminants in produced water.

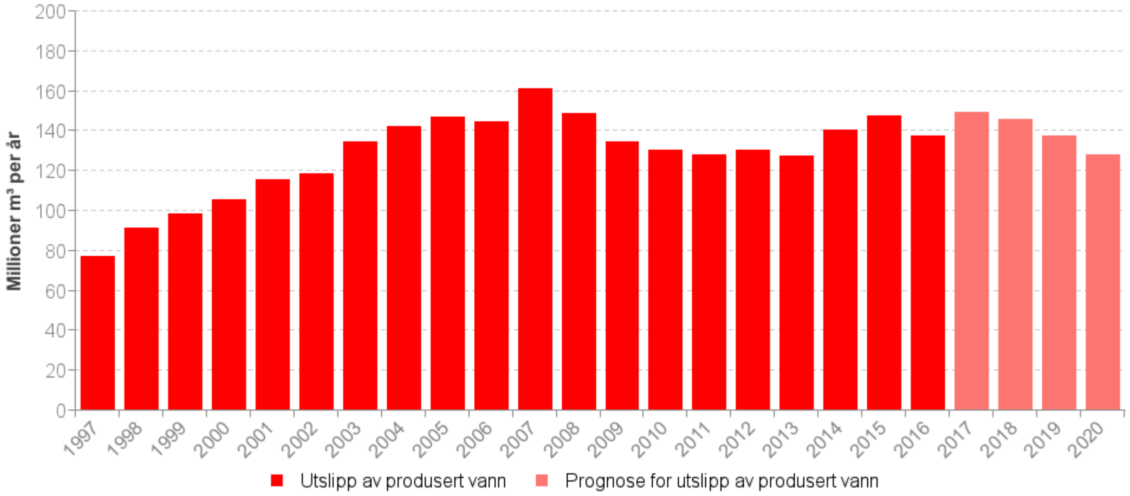

Figure 3: Historical data and forecast for discharge of produced water 1971-2022 (Norwegian Petroleum Directorate, 2018)

2.2 Waste from Oil & Gas Industry – Norwegian Continental Shelf

During the drilling and production of Oil and Gas, water from underground formations is brought to the surface which is defined as produced water. Produced water is also referred to as formation, brine or saltwater. Formation water is the water that occurs naturally within the pores of rocks. 134.2 million Sm^3 of produced water was discharged in 2017 (Norwegian Petroleum Directorate, 2018). Produced water is by far the largest volume byproduct or waste stream associated with Oil and Gas production (Abdol Hamid, Kassim, El Hishir, & El-Jawashi, 2008).

The NSC discharged an overall 134.2 million Sm^3 of produced water in 2017 and under 41.0 million Sm^3 of this volume was injected, which has a declining trend since 2016 (Dragsund, 2018). In 2016, the overall amount of produced water discharged was 138 million Sm^3 and 43 million Sm^3 was injected. Sources other than produced water, such as drainage water discharged in 2017 was 0.98 million Sm^3, and 0.14 million Sm^3 was injected. This volume is less than 1% of the volume of produced water (Miljødirektoratet, 2017).

As seen in Error! Reference source not found. The amount of produced water increased from 1997 and stabilized in between 120-150 million m^3 discharged per year since 2002. The amount of produced water discharges is expected to decrease over the next years – probably because a number of new fields are coming in production (Norsk Olje & Gass, 2017).

Produced water composition and physical properties varies significantly depending on the geographic location of the field, the reservoir formation with which the produced water have been in contact with for several of years and the type of hydrocarbon product being produced (Abdol Hamid et al., 2008; Alkhudhiri, Darwish, & Hilal, 2013). This would also be the case for slop water. In general, produced water is mainly water, contaminates usually composed of dispersed oil, dissolved organic compound, production chemicals, heavy metals and natural radioactive metals. Some of these components appear naturally in the produced water, while others are added for well-control purposes. Properties of produced water can be varying throughout the lifetime of the reservoir. The produced water volume from the Oil and Gas industry does not remain constant over time; it increases over the life of the oil or gas well. When the well is new, small amount of produced water is expected. To maintain the sufficient pressure in a reservoir for oil production, produced water or other waters may be injected to the reservoir (Alkhudhiri et al., 2013).

The regulatory agencies have discovered the negative potential impact produced water discharges can have (depending on the composition) on the environment and they have restricted the discharges in most onshore or near-shore locations (Abdol Hamid et al., 2008). The rules and regulations, which have been in development the recent years have set an increasing rigorous requirements on the discharge of produced, drain and slop water containing oil and environmental hazardous chemicals.

2.3 Norwegian Rules and Regulations for Discharge of Oily Wastewater from Offshore Activity

In the initial years of offshore oil and gas development, the oceans were perceived and treated as a limitless dumping grounds (Ball, Stewart, & Schliephake, 2012). More or less all liquid wastes were discharged from the platforms directly into the ocean. Undesirable effects on local ecology was observed during the 1970s and 1980s and as the evidence mounted, there was an indication that the oil-based drilling fluids and cuttings were the reason for this effect. In the benthos adjacent the oil and gas platforms in the North Sea, ecotoxicological changes were observed (Ball et al., 2012). Today the discharge of oily water to the sea is strictly monitored and regulated nationally through the Pollution Control Act and HSE regulation by the Petroleum Safety Authority in Norway, and similarly globally. This environmental focus was initiated by the Oslo-Paris (OSPAR) convention in 1972, and has since then had a gradually higher focus.

In 1972, the Norwegian Parliament took the decision to create the Norwegian Parliament Directorate (NPD). The NPD was later split into two independent agencies, creating Petroleum Safety Authority (PSA) with responsibility for safety supervision, and the NPD continued primarily with a resource management role. The new agency PSA reports to the Ministry of Petroleum and Energy. Today PSA cooperates with a number of other government agencies. PSA are responsible for coordinating the work of other agencies that have an independent regulatory responsibility for health, safety and the environment. The PSA regulatory are important in order to avoid offshore Petroleum industry to meet conflicting requirements from different authorities (Petroleum Safety Authority Norway, 2018).

To summarize, there are a number of different regulations governing pollution, production and waste management related to offshore activities.

2.3.1 Regulations on Pollution

The process of exploration drilling generates a large volume of waste, and among these are drainage water and slop water. There are regulations in place to ensure that handling of hazardous wastes shall be conducted in such a way that there is a non-negative effect on the external environment. The Activities regulation stipulates that (Petroleum Safety Authority Norway, 2017), before discharging the oily water to sea it has to be treated and the oil content in the water should be as low as possible. Some relevant sections of the regulations are:

§ 60 The oil content in produced water discharged to sea in any event, shall not exceed 30 mg oil per liter of water as a weighted average for one calendar month.

§ 60a Oily drainage water and other oily water may be discharged to sea subsequent to treatment. The oil content in water discharged to sea, shall be as low as possible. As mentioned in the first subsection the oil content shall not exceed 30 mg oil per liter of water as a weighted average for one calendar month.

Additional requirements can be set by the Norwegian Environment Agency regarding discharges of drainage water and other oily waters, on an individual basis (Petroleum Safety Authority Norway, 2017).

The cooperation of 15 Governments and the EU, OSPAR Commission is the mechanism to protect the marine environment of the North-East Atlantic. OSPAR Convention is the base for national laws governing discharged into the North Sea, including offshore drilling wastes.

OSPAR is aimed at protecting and conserving the North-East Atlantic and its Marine resources and environment, which Norway is a contracting part of (OSPAR Commission, 2018). The OSPAR commission has put in place a number of measures to reduce discharge from the oil and gas industry (OSPAR Commission, 2013b). For example, although the amount of produced water and displacement water discharged continues to increase, the total quantities of dispersed oil discharges to the sea is decreasing (OSPAR Commission, 2013b).

The rules and regulations regarding Oil & Gas offshore industry in Norway are based upon the agreed terms of OSPAR. Slop, drain and produced water have to be treated with the Best Available Technology (BAT) before discharged to the sea. The regulatory statement for the oil concentration in slope water/drainage water discharged is set to be as low as possible. However, the threshold for discharge to the sea is 30 mg/ L weighted over a month. To meet the strict regulations the oil concentration in wastewater is decreased to lower than 30 mg/L before discharged to the sea, which is also the case for produced water.

2.3.2 Product Regulations

Product regulations relates to restrictions on the manufacture, import, export, sale and use of chemicals and other hazards to health and the environment. The main purpose of the regulations are to prevent certain hazardous substances or preparations from causing injury to health or environmental damage, in additional all chemicals used in EU are subject to REACH. OSPAR 2017 adopted (OSPAR Commission, 2017b) a harmonized mandatory control system for use and reduction of offshore chemicals promoting the shift toward the use of less hazardous or preferably non – hazardous substances as the previous years (Petroleum Safety Authority Norway, 2017).

Harmonized Offshore Chemical Notification Format (HOCNF) has developed some rules and regulations on the use of chemicals offshore. The chemical suppliers must according to the HOCNF provide data and information about chemicals to be used and discharged offshore to the national authorities (OSPAR Commission, 2017b).

Based on the information sent by the chemical supplier to the national authorities who carries out the pre-screening, OSPAR has developed guidelines on how to complete the format. The

National authorities carries out the pre-screening and take the appropriate regulatory action, such as issuing discharge permits.

OSPAR guidelines for toxicity testing of substances, and protocol on methods for the testing of chemicals used in the offshore oil industry have to be followed by chemical suppliers. The OSPAR provides a list of substances or preparations used and discharged offshore which are considered to Pose Little or No Risk to the Environment (PLONOR) (OSPAR Commission, 2013a). The PLONOR list contains substances, which chemicals use and discharge offshore are subject to expert judgment by the competent national authorities. These chemicals are categorized as green chemicals.

The HOCNF scheme is an initiative of the OSPAR commissions, and concern offshore oil and gas operations within the North Sea and the northeast Atlantic, where they standardize the requirements for the testing and reporting of all chemicals. In Norway, the operator shall ensure that chemicals that are used or discharged offshore have been tested regarding to their ecotoxicological properties. The ecotoxicological testing of chemicals includes testing of biodegradability, bioaccumulation potential and acute toxicity. In Norwegian Activities Regulations (Section 62), the following HOCNF data set on ecotoxicology testing has to be followed by chemicals suppliers and operators using chemicals (OSPAR Commission, 2017a):

- **Bioconcentration potential**: N-octanol water partitioning data (log P_{OW}) must be provided for all organic substances.
- **Biodegradation potential:** Aerobic degradation test data must be provided for all organic substances. Biodegradation is the most important mechanism for the total removal of chemicals from the environment.
- **Toxicity potential:** Toxicity test data must be provided for all substances. The following toxicity tests are required:
    1) *Skeletonema costatum test*
    2) *Acartia Tonsa test*
    3) *Fish Scophthalmus maximus test*
    4) *Corophium volutator test.*

At Norwegian Oil and Gas fields operators have to categorize all chemicals that are used during offshore operations, for toxicity level. OSPAR (PLONOR) has categorized these offshore chemicals using colour code (OSPAR Commission, 2013a). The following colours are used for the categorization of offshore chemicals:

- **Green:** Chemicals allowed to discharge without permission
- **Yellow:** Chemicals need discharge permission. If there are acceptable environmental properties, then application will usually go through.
- **Red:** Chemicals need discharge permission. These chemicals are phasing out. Norwegian authorities banned to discharge these chemicals from 2005.
- **Black:** No discharge of chemicals are permitted.

In Norway, the authorities do not authorize individual offshore chemicals, and are not responsible for registration. However, the operator shall obtain a permit from the Norwegian Environment Agency covering use and discharge of chemicals, and report the actual consumption accordingly. The Environment Agency regulates the use and discharge of chemicals through this permitting, and follow up through compliance monitoring and spot checks in the NEMS database (OSPAR Commission, 2017a).

The chemicals used in this study are categorized as green chemicals, such as Xanthan Gum, Nature NSC and Nature NIC. These chemicals are allowed to be discharged without permission, because they are in the OSPAR list of Substance Used and Discharged Offshore (PLONOR). Other chemicals used in this study are categorized as yellow chemicals, which are allowed to be used due to their acceptable environmental properties, given a reasonable explanation for its use.

2.4 Environmental Impact Factor, EIF

The amount of produced water discharged to the sea on the NCS has exceeded 100 million tons per year and have therefore become a concern for the operators such as oil companies and for the Norwegian Governmental Bodies (RYE, REED, DITLEVSEN, BERNTSEN, & GARPESTAD, 2004). Norwegian Authorities have therefore established strict regulations towards the discharge of produced water to the environment. The operators on the NCS have agreed to follow the regulations and work together with the Norwegian Authorities towards a reduction on the environmental impact from produced water releases to “Zero effect release”. Environmental Impact Factor (EIF) has been developed to achieve this “Zero effect release”.

Produced water discharge may have different potential impacts on the environment depending on where it is discharged, and its composition. If the produced water is discharged into small streams it may have larger environmental impact than when discharged into the open ocean. In open ocean the produced water is diluted which may have less potential environmental impact.

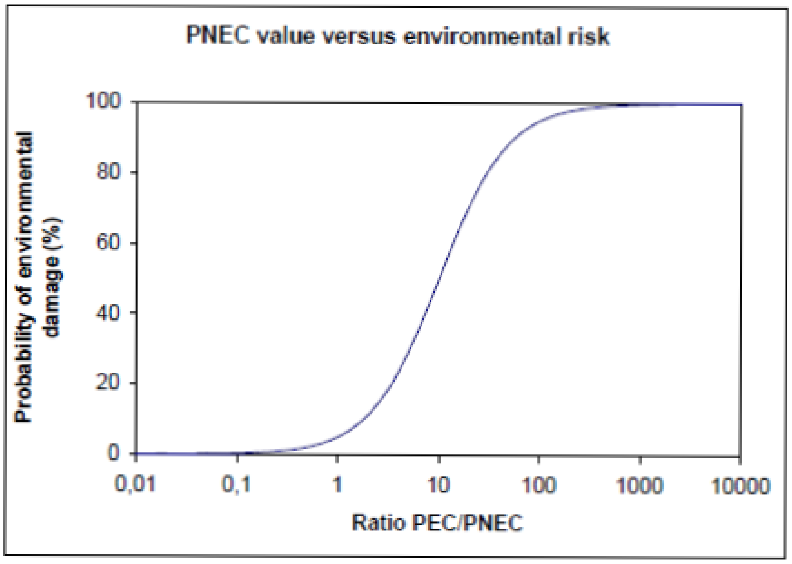

Figure 4: The relation between the PEC/PNEC level and the risk level probability for damage on biota (RYE, REED, DITLEVSEN, BERNTSEN, & GARPESTAD, 2004)

EIF is an indicator for the potential impact on environment from produced water discharges and is developed by the operators on the NCS (RYE et al., 2004). This method is based on the calculation on the EIF according to the DREAM model and the EIF method is based on a PEC/PNEC approach (RYE et al., 2004). The Predicted Environmental Concentration (PEC) is in the recipient the three-dimensional and time variable concentration that is caused by the discharge of the produced water. The lower limit where effects in the marine biota in the recipient may be experienced is the Predicted No Effect Concentration (PNEC). For each component in the produced water the PNEC level is given. EIF is characterized as the water volume where the PEC/PNEC >1, divided by a unit volume equal to 10^5 m^3 (100 x 100 x 10) of recipient water (D Smit, Frost, & Johnsen, 2011).

There may be a risk for damage if the PEC is larger than the threshold PNEC, and the risk for a damage is considered to be acceptable when the PEC is lower than the PNEC threshold. When PEC/PNEC = 1, this corresponds to a level of probability of pollution equal to 5%. The relation between the PEC/PNEC-ratio and the probability of pollution is shown in *Figure 4.*

Untreated produced water and slop water discharged offshore cause PEC/PNEC larger than 1. PEC/PNEC larger than 1 means larger impact on water volume offshore. Discharge of produced and slop water have increased and the possible adverse effects of its constituents on the marine environment. Treatment of produced and slop water is essential to have minimal EIF, environmental damage and PEC/PNEC <1.

2.5 Oil-in-Water Emulsion

A homogeneous mixture of two (or more) immiscible liquids is known as an emulsion. It consists of at least one immiscible liquid, the internal phase, dispersed as droplets in another continuous phase, and is more a heterogeneous system. In an O/W emulsion the oil droplets are dispersed as droplets in a continuous water phase. Formation of emulsion is an action of the viscosity, dielectric constants, relative concentration, and specific gravity of both phases, emulsifier (surfactant). It is also influenced by the pH of the aqueous phase (Coca-Prados & Gutiérrez-Cervelló, 2011).

These O/W emulsions are typically observed in slop water generated during drilling. It is composed of drilling or displacement fluids, wash water from cleaning operations, rain water runoff contaminated with drilling fluid components, which are captured and stored in tanks. The composition of slop water can vary widely, from lightly oil-contaminated water to complex water-in-oil or oil-in-water emulsions. This complex emulsion mixture can be treated by chemically and physically treatment methods (Mueller, Andrade, & Massam, 2013).

The homogeneous mixture of water and oil separates normally, into two layers by gravity. However, a fraction of the oil will in some cases remain as tiny droplets suspended in water, and not form a distinct layer, due to stokes law. Surfactant added to an O/W mixture can form a stable O/W emulsion. This is due to the surface tension of the liquid, which is in small amount, lowered by the long organic molecules of surfactants. Separation of oily wastewater can be extremely challenging due to the presence of a surfactants, which cause the enhanced solubility of the emulsified oil. Interfacial tension reduces significantly with presence of a surfactant, and a stable emulsion will be formed, seen in *Figure 5*.

Interfacial tension of a liquid is often determined as the work required to increase the area of a surface isothermally and reversibly by a unit amount (Coca-Prados & Gutiérrez-Cervelló, 2011).

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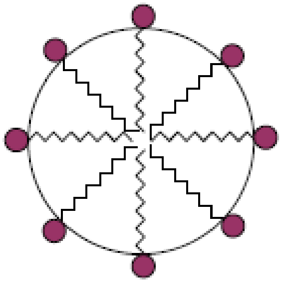

Figure 5: Surfactant stabilized micelles, Oil-in-water (O/W)

Several properties playing a key role in the stability of an O/W emulsion have to be measured to analyze separation processes. Zeta potential (ζ) play an important role in the stability of O/W emulsions, and is measured by the velocity of charged droplets or colloids in an electrical potential field of known strength. The emulsion stability increases with large values of ζ (positive or negative). This cause difficulty for coalescence of droplets and particles (Hunter, 2013, pp. 220-254). Zeta potential is a key factor in understanding the performance of physical processes such as flocculation and sedimentation. It affects the size and density of flocs formed in wastewater. The ζ of particles varies depending on what wastewater contains.

The ζ of a particle in solution is characterized as the value of the electrostatic potential at the shear plane, see *Figure 6*, i.e. plane which ions are no longer dragged along with a moving droplet or particle. High ζ value, increased interfacial shear viscosity, and relatively decreased interfacial tension are largely caused by surfactants. Coalescence can occur if ζ tends to zero, to reach this polyvalent ions are added with opposite charge than on the emulsion droplet or particles, the electrically repulsive force will be neutralized (Coca-Prados & Gutiérrez-Cervelló, 2011).

The ζ is affected by different factors, i.e. pH and thickness of double layer. The pH of the sample is one of the most important factors that affect its ζ. The ζ will be positive with decreased pH and negative at increased pH. The concentration of ions chose the thickness of the double layer.

The double layer becomes more compressed with the higher ionic strength, and is also influenced by the valence of the ions.

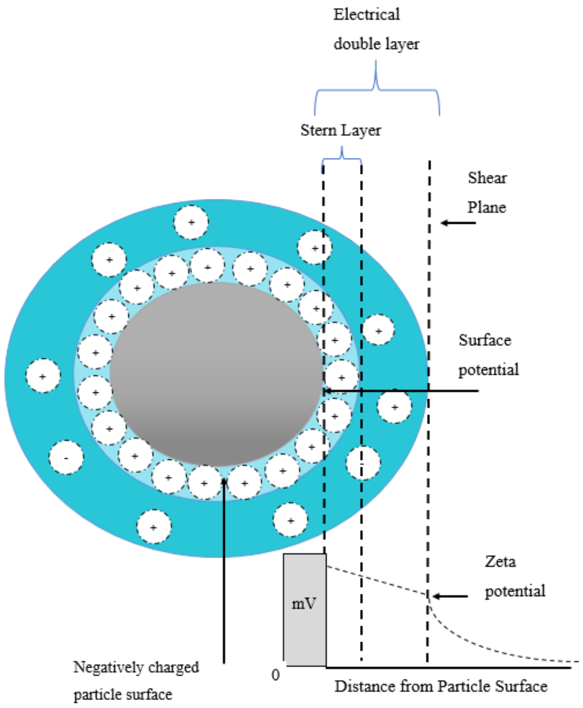

Figure 6: Electrical double layer around a negatively charged colloid and the distribution of electrical potential.

Main physical treatment processes included in wastewater and slop water treatment are sedimentation, flotation and filtration. All these processes are depended on the principles relating the size, density and the charge of the particles to be removed. More rapid flocculation of particles in wastewater is obtained, as the density of flocs increases. The electrostatic interactions between particles reduce with decreasing zeta potential, which cause production of more compact flocs by colliding particles. A study done by Morfesis et al. 2009, shows that there is a clear relationship between lower zeta potential that results in higher removal efficiencies (Morfesis et al., 2009).

2.6 Exploration Drilling

Oil fields are typically examined by seismic exploration, which is based around the geological knowledge of the ground formation. An exploratory well is drilled to confirm the seismic and geological expectation on a new oil or gas reservoir (Leffler, Pattarozzi, & Sterling, 2011, pp. 240-250).

The exploration well is drilled with a conductor pipe that is driven into the ground with a pile driver. The inside of the conductor will be full of rubble as the bottom of the conductor is open while hammering it into the ground. A drill bit is slightly smaller than the conductor and is run on drill collars down to the top of the rubble. The bit is rotated and lowered when mud is pumped down the drill string. The mud returns and flows upwards in the annulus lifting the bit up to the surface and breaks the rubble up. The well is drilled until it reaches the depth to run the surface casing, but the mud is still circulated around until all of the drill cuttings are cleaned out of the well (Devereux, 2012).

After several years of high exploration activity, Norwegian Petroleum Directorate reported 20 fewer exploration wells in 2016 than the preceding years, in total 36 exploration wells were drilled (Norwegian Petroleum Directorate, 2017). The reason for this is a lower oil price and cost cuts. In these situations oil companies priorities exploration drillings in familiar areas where discoveries are more likely, but often small.

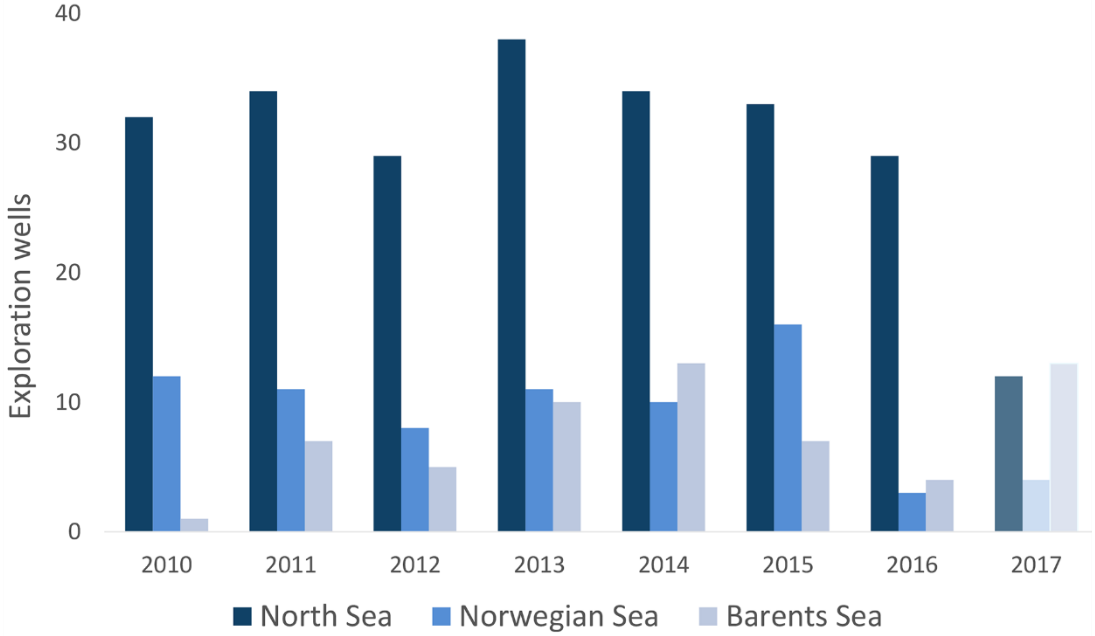

Figure 7: Spudded exploration wells (Norwegian Petroleum Directorate, 2017)

In 2017, a decline in the number of exploration wells were expected as seen in Error! Reference source not found.. Discoveries made in 2016 were 18 compared to 11 in 2017. In the last few years a number of exploration wells have decreased this appears clearly in Error! Reference source not found. (Norwegian Petroleum Directorate, 2018)**.** In 2017, 34 wells were completed, 23 were wildcat wells and eleven were appraisal wells compared with 2016 where 28 were wildcat wells and eight were appraisal wells (Norwegian Petroleum Directorate, 2017 2018)

An offshore drilling service Diamond Offshore claims, drilling an exploratory (wildcat) well offshore may take as 15 days or as long as 12 months, of round the clock, seven day per week operation to drill a single well depending on the complexity of the project (Diamond Offshore, 2017). A rig generates a lot more slop during active drilling. Due to slop containing waste from the drill floor and potentially excess drilling fluids.

2.6.1 Lost Circulation

When drilling a surface hole, the most common problem experienced is the drilling mud disappears downhole into the rock either into fractures or into very permeable rock, this phenomena is known as lost circulation. Drilling through very permeable formations cause this problem, because the mud cannot form an effective filter cake (Messenger, 1981). The losses occur while drilling the surface hole, and have two common causes:

- When the whole mud seep through the pore spaces since the formation is very permeable.
- Mud that leave the wellbore due to fractures. Severe or total losses are likely with fractures.

According to Devereux one way to treat the losses of mud into a permeable formation is to mix lost circulation material (LCM) with mud before pumping it into the well. LCM may create a plaster of material at the exposed face of the formation. There are several techniques available to achieve and use LCM, such as, reducing the density of the drilling fluid itself or reducing the density of the drilling fluid contaminated with cuttings (Devereux, 2012, pp. 303-308; Hyne, 2012, p. 285). Some other possible ways to increase the density is to increase the viscosity of the mud at the shear rates present in the annulus so cuttings are more efficiently brought to surface by the mud. The viscosity is normally increased with polymers, one such polymer used is Xanthan Gum.

While drilling, the drilling mud is pushed upwards annulus between drill collar and borehole and transports with all loose drill cuttings and dust from the bottom. The drill cuttings will sink in the drilling mud when the production is stopped. Particles sink faster in thin liquid (low viscosity) compared to thick liquid (high viscosity) (Skjeggestad, 1989).

Drill cutting have to be separated from the drilling mud when it returns to the surface of the rig, especially when the drilling mud contains a lot of drill cuttings and other contaminants.

Different drilling muds are used at different times during drilling. Changing from one mud to another will require extensive cleaning of the pits which the muds are kept in. This flushing and cleaning leads to slop waste and in some cases complex slop water.

2.7 Production Drilling

When exploration drilling is completed and an offshore field has officially been discovered, there is a varying period of time before the field is actually developed. An example of this is Martin Linge field which was discovered in 1978, but the plan for development and operation were approved in 2012, the platform is under construction and the estimated production start is 2019 (Oljedirektoratet, 2018).

A well is brought into production after the well has been drilled to the depth where oil or gas is expected to be found. A number of operations are involved in production drilling, such as penetration of the casing and cleaning out water and sediment from the pipeline so that flow is unimpeded. By electrical impulses charges are discharged through casing into reservoir to create opening for the oil and gas to flow into the well and to the surface. The oil and gas produced are separated, and water and sediment are removed from the crude oil (Devereux, 2012, pp. 52-60).

Almost all oil crude oil and gas are under natural pressure, which decreases as Oil and Gas is removed from the reservoir. The reservoir life cycle goes through three phases. The first phase is the natural pressure from the oil, gas and water in the reservoir, where the productivity of the production decreases with decreased pressure. Second phase is artificial lift, which involves pumping of pressurized gas into the reservoir when the natural pressure is expended. The third phase is stripper or marginal production (Hyne, 2012, pp. 391-406).

Sheng, 2010, discovered that productivity of oil and gas reservoir may be improved by a variety of recovery methods, both physical and chemical methods may be used. Injection of water and gas into the reservoir is one of the recovery methods, this to maintain working pressure by natural displacement. A secondary recovery method includes displacement by pressure, artificial lift and flooding (Sheng, 2010, pp. 1-4). Using various secondary methods in multiple and different combinations is called Enhanced Oil Recovery, EOR (Fink, 2011, p. 459). Some of the methods are explained briefly below:

Acidizing

The output of a well is increased by pumping acid directly into a producing reservoir. This may open flow channels through the reaction of chemicals and minerals, this method is known as acidizing. Depending on the type of rock or minerals in the reservoir hydrochloric acid is used together with hydrofluoric acid, formic acid and acetic acid. These combinations of acids are currently used to clean perforations which have been plugged with drilling mud and to restore damaged permeability near the well hole (Fink, 2011, pp. 471-473).

Fracturing

Method used to increase the flow of oil or gas through a reservoir into wells by force or pressure is described by fracturing. Underground channels opens by fracturing forces. Fluid treated with special propping agents is pumped into the reservoir under high pressure to open fissures. Normally, massive fracturing is used to open gas wells where the reservoir formations are so dense that gas cannot pass through them (Fink, 2011, pp. 519-524).

Polymer flooding

Polymer flooding is an enhanced recovery method used to reduce the surface tension of crude oil which will improve water injection. A miscible fluid is injected into a reservoir. In polymer flooding a detergent is used to wash the crude oil. To move the crude oil towards the producing well a high viscosity fluid is injected behind the detergent (Sheng, 2010, pp. 101-233). Typically polymer used in polymer flooding is a biologically produced polymer known as Xanthan Gum (Chang, 1978).

3. Slop Water Management

The process of drilling oil and gas wells offshore generates significant volumes of waste, which includes fluids and cuttings, slop water and solid wastes. Drilling waste with physical properties similar to water that is contaminated with oil/hydrocarbon from drilling is colloquially called “slop water”, and when having a sludge like character it is defined as “slop mud”. “Slop mud” is typically generated from OBM/SBM saturated with water from the drilling process (Ivan & Dixit, 2006). The volume and type of slop generated varies from one drilling operation to another and also during the drilling campaign due to varying drilling activities, fluids and formation conditions. The slop can either be treated offshore or sent to shore for treatment or disposal (SorbWater Technology, 2014).

Collected “slop water” also has contributions from water that is collected from the various drains onboard a rig, such as rain water, water containing oil or water-based drilling mud from the drains at the drill floor, soap from cleaning of pipe threads at deck, mud and chemical residuals from the mud pump room and mud mix room. This mixture of slop water is treated offshore with the use of different treatment technologies, i.e. DAF, membrane, centrifuge, hydrocyclone and et cetera (Nature Oil & Gas AS, 2013 -a). The treatment technologies developed for offshore, treats slop water according to the rule and regulations at the respective geographical location.

A number of options are available for handling slop water. Slop water may be treated and discharged offshore, transported to shore for treatment and disposal or offshore re-injection. Only the treated water meeting discharge regulations may be discharged (Okiemute & Brattebø, 2015). *Figure 8* shows methods to treat slop water and all the methods are described in the sections below.

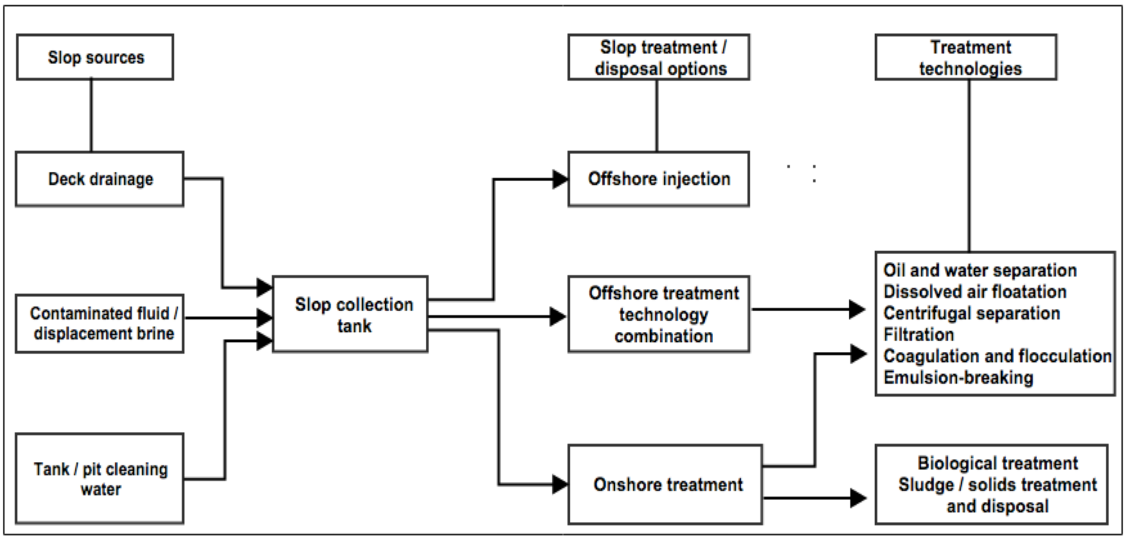

Figure 8: Flow sheet of slop water sources and management approaches (Okiemute & Brattebø, 2015)

Figure 8 shows typical slop sources, slop treatment/disposal options and technologies to treat the slop. All water from different sources is collected in a slop tank. Treatment technologies and where to treat the slop are selected depending on the composition of slop water. Mostly slop water is treated offshore with different combinations of technologies. Also, offshore injection and onshore treatment are used.

3.1 Onshore Slop Water Treatment

The main parameter for water discharged offshore is its hydrocarbon content. Meanwhile for water discharged onshore many more parameters have to be taken into consideration. All types of water which is not discharged, such as produced water, slop water and drilling fluid, collected from the offshore are mixed together and sent onshore for treatment. Discharge water parameters taken into consideration before discharged onshore cover total organic content (TOC), pH, chemical oxygen demand (COD), heavy metals, PAH and PCBs, PFAs to meet the discharge criteria. Onshore facilities are similar to those used offshore except that the treatment unit onshore handle and treat significantly higher volumes and organics. The liquid onshore is typically pretreated to remove suspended solids and free oil. This is usually achieved by sedimentation, and separation by centrifuge, DAG etc. An added process used for onshore treatment is biological treatment that is capable of removing COD, TOC and oil content from wastewater (Ivan & Dixit, 2006).

Biological process is based on the effective contact between microorganism and the organic matter contained in the mixed water, in such a way that it can be used as food for the microorganisms. The organic matter is converted into carbon dioxide, CO_2, water and cellular material by the microorganisms (Sperling, 2007, pp. 180-183).

The presence of oxygen is a fundamental component of the aerobic process and is required for the biological decomposition of the organic matter. High efficiency of biological treatment can in addition be achieved by temperature, pH, and contact time which are important parameters in this process (Wiesmann, Choi, & Dombrowski, 2007, pp. 119-121).

For the treatment of oily wastewater from offshore, biological treatment has successfully been used both in a form of aerobic and anaerobic, especially for the removal of aromatic compounds. Anaerobic biodegradability is a complex process compared to an aerobic treatment. Completely anaerobic decomposition of organic matter is achieved by passing different steps in series, where product from one process is used as substrate in the next process. This requires that the processes are in balance with each other, because polluted and accumulation may destroy the processes. Due to occurrence of these problems anaerobic treatment process is not widely used compared to aerobic (Tchobanoglous, Metcalf, Eddy, & Aecom, 2014, pp. 655-660. 1070-1075). For these treatments different reactors have been used, where the most common ones are fluidized bed reactor, moving bed biological reactors, submerged fixed film reactors, and fixed activated sludge. The main disadvantage of this method is the amount of sludge produced: approximately 1600-3200 Kg DS/ day per 8000 m^3/day of produced water (Freese, Trollip, & Nozaic, 2003; Sperling, 2007, pp. 183-193)

Biological treatment onshore also requires the addition of macro nutrients (Nitrogen and Phosphorus) as these are not commonly present in this waste.

3.2 Offshore Slop Water Treatment

The focus on slop water treatment with suitable technologies has increased due to stricter environmental regulations and challenges with slop injection, in addition so that there is a significant cost to onshore treatment. Treating slop waste offshore reduces the volume of waste that needs to be shipped onshore for treatment. The nature of slop water varies significantly and can be very complex. The choice of optimal treatment methods depends on slop water properties. There are two main categories for treatment of slop water, which either is a physical or chemical process, or a combination of both. Depending on the contamination level of the slop water being treated, the treatment processes can include one or two main stages (Ivan & Dixit, 2006; Okiemute & Brattebø, 2015). These two stages are described below (Ivan & Dixit, 2006):

- **Pre-treatment:** In this stage processes that separate mud and water are used. The separated mud and water are transferred to separate holding or treatment vessels for further processing. The separation process may be chemical, physical or combination of both.
- **Water treatment stage:** If discharge limits of separated slop water collected directly during drilling do not meet the discharge regulation, it has to be treated further until it meets the regulations. Common treatment methods used are physical or chemical and are described below.

3.2.1 Physical Treatment Processes

Physical treatment cannot be applied as the only treatment on all types of slop wastewater, due to complex emulsion. It often has to be combined with other methods, such as pretreated by chemical or biological treatment (only used onshore), which are discussed below. Typical physical processes for slop water treatment are gravity separation, centrifugal separation, filtration and membrane separation. In addition these are sometimes aided by temperature (heating). The particle`s size and the viscosity of the fluid are some of the factors that affect the separation and sedimentation of particles in the slop water.

Sedimentation is defined by George Stokes in Stoke`s law as following (Bouse, 2005):

$$v = \frac{kgD^2(d_s - d_f)}{\mu} \tag{3.1}$$

Where;

v = terminal velocity

k = a constant that is dependent on the units in use

g = the gravitational constant

D = the diameter of the solid particle

d_s = the density of the particles

d_f = the density of the fluid

μ = the viscosity of the fluid

From Stoke`s law equation 3.1 it is clear that particles settle more rapidly in less viscous fluids, and that heavy particles settle more rapidly than light particles. Stoke`s law is also applied to oil droplets. The oil droplets rises to the surface of a tank and are skimmed off. The rises of particles are based on a density difference.

3.2.1.1 Gravity Separation

Gravity separation is the most common process used in water and wastewater treatment, which historically has meant sedimentation, then achieved by simply storing the fluid in a tank/vessel. Sedimentation follows coagulation and flocculation, which will settle out over time (Crittenden, Trussell, Hand, Howe, & Tchobanoglous, 2012a; Vigneswaran & Ben-Aim, 1989).

One of the first devices used to control particulate emissions was the gravity settler. It is primarily vertical static tanks allowing the particles to settle out under the action of gravity. Settling is mostly used for separation of mud, sludge and water (and free oil if this is present in larger quantities). Gravity settling can be aided by pretreatment such as chemical, and has a low treatment capacity and high footprint which make it non-efficient as a primary type of slop water treatment and offshore treatment unit, *Figure 9* show a typical primary settling tank used for treatment of wastewater (Martínez-González et al., 2009; Theodore, 2008, pp. 317-320).

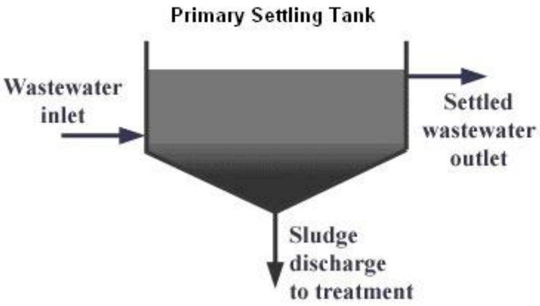

Figure 9: Primary settling tank

Settling is indirectly used as a pre-treatment step, as the slop will separate when stored on tanks prior to treatment. Oil, or heavy oil contaminated water will float to the surface, and heavier fractions of the slop (i.e. slop sludge) will settle to the bottom of the tank.

3.2.1.2 Dissolved Air Flotation

Another process for removing particles in suspension from liquid is called Dissolved Air Flotation (DAF) and is done by bringing the particles to the surface of the liquid. In a saturator air is dissolved at high pressure and when water is released in the flotation cell at atmospheric pressure, formation of microbubbles appears. The microbubbles attach onto the surface of the suspended particles and increase their buoyancy allowing them to rise to the surface and the accumulated flocs on the water surface layer are skimmed or scraped off (Al-Shamrani, James, & Xiao, 2002; Suzuki & Maruyama, 2005). DAF has been shown to be an effective treatment process for water containing low-density particles and for low turbidity water. DAF with flocculation has proven efficient removal of emulsified oil from oily wastewater (Zouboulis & Avranas, 2000). Due to oil being directly brought to the surface by the microbubbles or indirectly by attachment to particles.

3.2.2 Centrifugal Forces

Fluid is sent tangent inside a round vessel in a centrifuge where the liquid is rotated in a rotating cylinder. The separation of gas, oil, water and solids result from the application of artificial gravity caused by the centrifugal force in the spinning liquid. Different treatment units are developed where this centrifugal force is used, such as decanter centrifuge, disc stack centrifuge and hydrocyclone.

3.2.2.1 Decanter Centrifuge

Decanter centrifuges are a mechanical process used for separation of solids from liquids, by mean of sedimentation accelerated by the centrifugal force. It can be used at high solids concentrations (sludge) (Kinnarinen & Häkkinen, 2013). A decanter centrifuge is built up of two rotating elements, one is a solid bowl that act as the sedimentation medium and the second is a helical screw that is used to remove sediment solids from the conical end of the bowl. A schematic of a decanter centrifuge is presented in *Figure 10*. The separation is achieved by accelerated sedimentation. The fluid passes through a rapidly rotating bowl and the centrifugal force pushes the heavier particles to the bowl wall, where they are scraped towards the discharge ports by a conveyor, which rotates at a slightly slower rate than the bowl (Bouse, 2005).

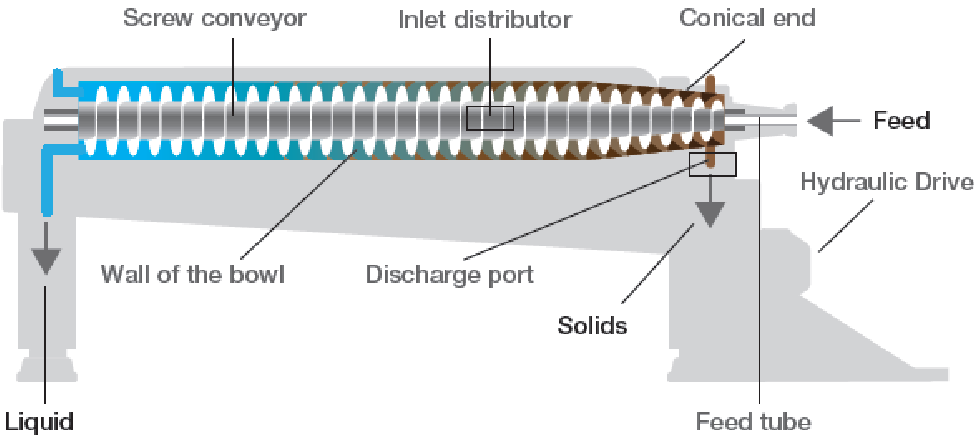

Figure 10: Horizontal centrifuge with a screw conveyor in center for rejection of solids

Decanter centrifuges greatest advantage is that they are suitable for dewatering hard-to-filter slurries containing fine particles (<1 µm) (Kinnarinen & Häkkinen, 2013). Different characteristics of wastewater such as solids concentration, type of solid, liquid loading, type of liquid, pH, viscosity and temperature affect the efficiency of dewatering. The Decanter Centrifuge is one of the most commonly selected types of process equipment used to separate a liquid from a solid, because of their capacity, process containment capability and reliability.

The sedimentation process is accelerated by the centrifugal force applied by the rotation of the bowl. Under normal gravitation force of 1 G in an undisturbed container separation might take hours or days, but may be achieved in seconds at 400-3000 G generated by the centrifuge force, by spinning the container at 3-4000 RPM. For the proper use of centrifuge an understanding of the factors influencing the process is required in view of the fact that sedimentation is used to achieve the separation.

Decanter centrifuge is widely used in the offshore industry for dewatering of sludge, or removing solids from muds. An advantage with this process is the unit's capacity to handle high viscosity and high solids fluid. (Bouse, 2005; Wakeman. J, 2007). However, it has limitation if the density and boundary layer of solid, water and oil in the slop is close together.

3.2.2.2 Disc Stack Centrifuge

Self-cleaning separators with disc stack and automatic solids ejection are used when the percentage of solids in the oil is too high for manual cleaning. These machines are mainly used for the clarification and purification of fuel oils such as gas oil, diesel oil, and heavy fuel oil and lube oil (GEA Mechanical Equipment, 2013). They can be applied to slop water, if the slop has low solid content.

Water containing oil droplets enters the bowl in the center through a still-standing pipe, flows down to the bottom of the bowl where the liquid is fed to the disc stack through feed holes in the discs, *Figure 11*. The heavier water phase and particles are forced towards the underside of the next disc and move towards the outer wall of the bowl. The lighter oil phase forms a layer on top of the discs and move towards the center of the bowl. The liquid phases are discharged continuously through pump impellers (Maybury, Mannweiler, Titchener-Hooker, Hoare, & Dunnill, 1998; Meixner et al., 2015).

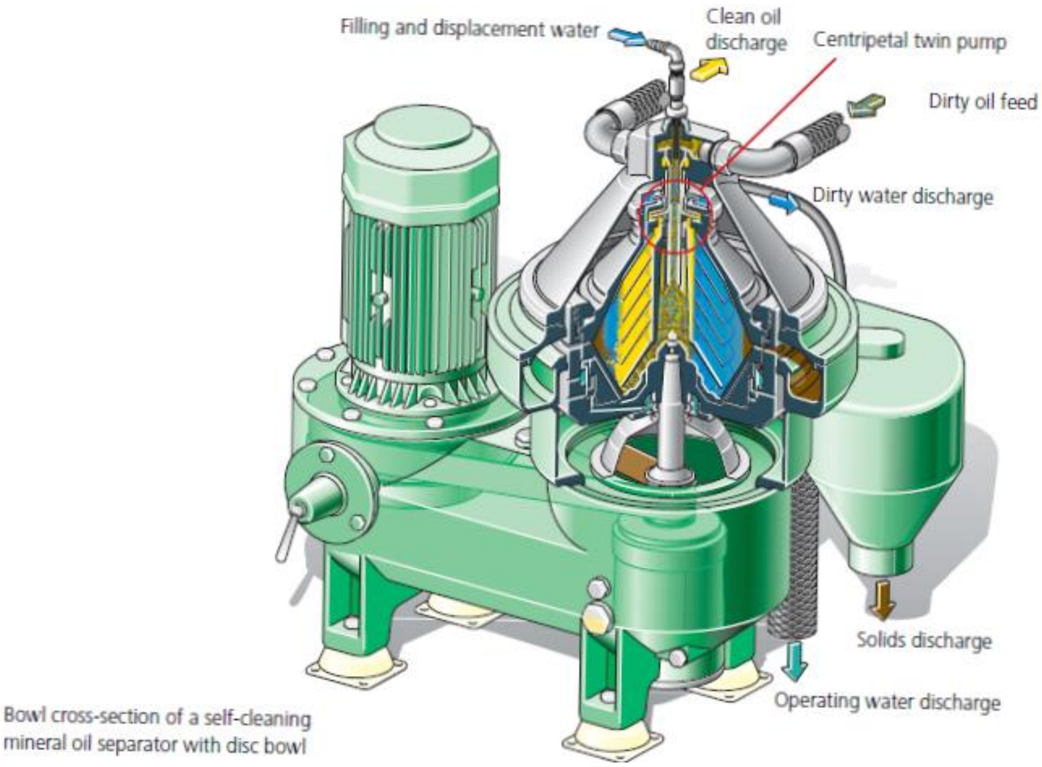

Figure 11: Typical disc stack centrifuge (Equipment, 2013)

Fluid the containing particles, discharge particles at intervals through the outer edge of the bowl. Particles concentration (> 100 mg/l) may be solved by use of nozzle centrifuge discharging particles continuously through nozzle in the bowl edge.

For the optimum separation the discs in the disc stack have spacers with correct distance, typically between 0.4 and 3 mm. To achieve optimum interface and flow pattern location of feed holes and design of spacers are important, if not it will have issues with solid loads and complex oil emulsions that are not influenced by gravity/G-force.

The Disc Stack centrifuge efficiently removes small droplets due to high gravity force, up to 9000 G. The limitation with use of this treatment unit is the limited capacity per unit: 40 – 100 m^3/h (Maybury et al., 1998).

3.2.3 Filtration – Membrane Separation

Filtration is the method used to separate particulate of solute components in a fluid suspension or solution depending on size by flowing under a pressure differential through a porous medium. There are two categories of filtration, conventional (also called dead-end filtration) and crossflow filtration. In relation to the filter medium they differ according to the direction of the fluid feed. The fluids flow perpendicular to the medium in conventional filtration, which results in a cake of solids depositing on the filter medium. However, in crossflow filtration the fluid flows parallel to the medium to minimize the cake of solids on the medium (Harrison Roger, Todd Paul, Rudge Scott, & Petrides Demetri, 2015; Vigneswaran & Ben-Aim, 1989). Following *Figure 12* shows illustration that compares conventional filtration and crossflow filtration.

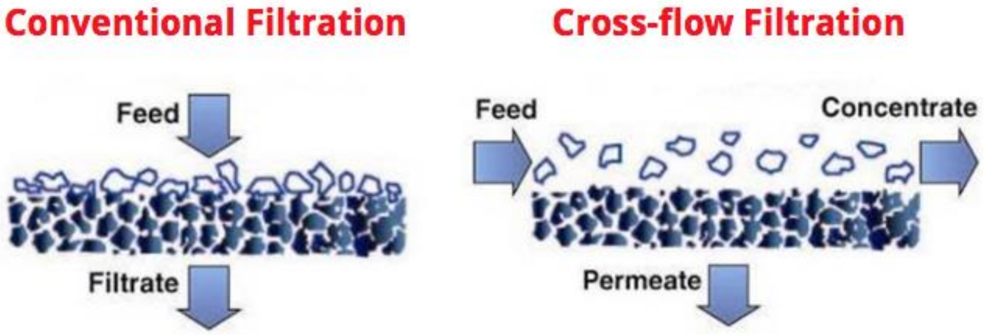

Figure 12: Typical disc stack centrifuge (Equipment, 2013)

Membrane separation technology separate a fluid from its contaminated components. Thin film made of artificial organic or inorganic porous material is used. The differential pressure through the membrane is the driving force of separation, and determines the degree of separation. The different types of membrane that is used in the industry are categorized as, ultrafiltration (UF), micro-filtration (MF), nano-filtration (NF) and reverse osmosis (RO). These different types of membranes remove different size of particles, such as MF membranes are used to remove suspended particles, UF membranes are used to remove macromolecules and dissolved components can be removed by RO membranes (Crittenden, Trussell, Hand, Howe, & Tchobanoglous, 2012b; Yu, Han, & He, 2013).

UF membranes technique is highly efficient at removing oil from produced water. Any chemical additive in UF treatment is not needed (Benjamin & Lawler, 2013, pp. 731-735). However, it has limitations with regards to solid content and sludge.

The strict regulations to remove dissolved organic components from oil wastewater in onshore treatment, cannot be achieved by MF and UF process, other methods need to be used is RO membrane. Dissolved organic compound and heavy metals are removed from produced water by using RO membrane. The disadvantages with using RO membranes for their operation is the energy consumption which is needed to produce the high pressure (Benjamin & Lawler, 2013, pp. 731-740; Crittenden et al., 2012b). In addition there are two main technical drawbacks of membranes, fouling and the short lifetime of the membrane material, which cause low treatment capacity.

Like all other technologies membranes separation also has advantages and disadvantages. Membranes are selected so that the species of interest will not pass through these membranes. If the concentration of particles is big in the bulk phase it will build up next to the membrane surface. High concentration of species may precipitate on the membrane surface and impeding the flow of fluid through the membrane. The solubility of solute can be exceeded with high solute concentration, resulting in the precipitation of the solute and the formation of a solids or gel layer on the membrane surface. Some advantages with the use of different membranes are the removal of macromolecules and dissolved components.

3.2.4 Chemical Treatment

Chemical treatment is an efficient treatment method for wastewater. The physical, chemical and biological nature of wastewater is changed by chemical reactions. There are different chemical unit processes to treat wastewater. These includes chemical coagulation, chemical precipitation, chemical oxidation, ion exchange, and chemical neutralization and stabilization (Bahadori, 2013; Holloway, 1991).

3.2.4.1 Chemical Precipitation

The leading method used for removing dissolved metals from wastewater solutions either from municipal or industrial metals contaminated liquid waste is chemical precipitation. The dissolved metals are converted into solid particle form by adding precipitation reagent to the mixture. The occurrence of chemical reaction is triggered by the reagent forms the solid particles from dissolved solids. The particles can then be removed from the mixture by using different treatment methods, e.g. filtration, centrifuge etc. How well chemical precipitation works depends on metal present, the concentration of the metal, type of reagent used (Bahadori, 2013; Holloway, 1991). However, this is effectively utilized for treatment of most slops.

3.2.4.2 Chemical Coagulation

Chemical coagulation is the process of destabilizing mutual repulsion of particles and cause particles to bind together to bigger flocs. The electric surface charge of the fine solid particles dispersed in wastewater is negative, which prevents the particles to bind together to bigger flocs. These negative charged particles are destabilized by chemical coagulation. Positively charged coagulants reduces negative charge of particles resulting in charge neutralization, this phenomena are mentioned in section 2.5 in zeta potential paragraph. The particles bind to big flocs once the charge is reduced, and is bound to even bigger flocs by adding flocculant to the mixture. These bigger flocs are created when the flocculant reacts against the positively charged solution, (*Figure 13*), where it either creates bridges between the particles or neutralizes the particle groups (Bahadori, 2013; Holloway, 1991; Hubbe, Sundberg, Mocchiutti, Ni, & Pelton, 2012). The purpose of coagulation is to convert small particles that agglomerates into larger particles, and overcome the electrostatic repulsive surface forces.

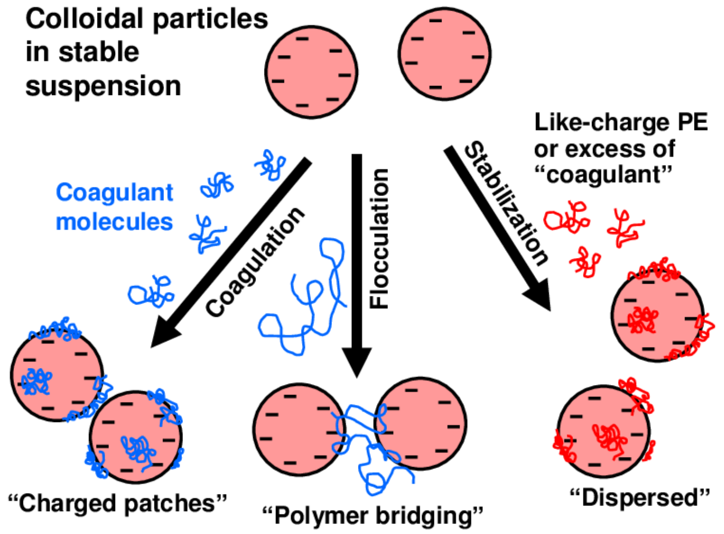

Figure 13: Three ways in which the addition of a polyelectrolytes affect the colloidal stability or negatively charged particles in an aqueous suspension (Hubbe et al., 2012). Red indicates negatively charge; blue indicate positive charge.

Chemicals used to produce agglomerations of colloidal particles are known as coagulants. These chemicals are used in effluent water treatment processes for solids removal, water clarification, lime softening, sludge thickening, and solids dewatering. There are different groups of coagulants, these includes aluminium salts, iron salts, and polyelectrolytes. A widely used aluminium salt is polyaluminium chloride (PAC) and aluminium sulphate, known as

Alum. The dominating coagulating agents used are, inorganic salts of iron (Fe^{3+}) and aluminium (Al^{3+}) (Kvinnesland, 2002). In this study inorganic salts of Aluminium (Al^{3+}) and Iron (Fe^{3+}) have been used during lab testing.

Coagulants depress the effective width of the double layer around the particles and reduce the surface charge on the particle by cation addition**,** see *Figure 13***.** This leads to reduction of the zeta potential, and particle flocs start to form. The coagulation of particles is increased by reducing the surface charge of the particles. The surface charge is reduced by adding inorganic species, which increases the conductivity of the continuous phase.

The purpose of the coagulation process and selection of coagulants depends on the further use of water, whether it will be dumped or reused. Another factor it depends on is the source of the water and the nature of the suspended, colloidal, and dissolved organic constituents (Bahadori, 2013; Crittenden et al., 2012a). It is very important to select proper and the most effective chemical since coagulation is an important part of conventional water treatment. The strict rules and regulations on the discharge of chemicals are deciding the choice of coagulants (Crittenden et al., 2012a).

3.2.4.3 Chemical Flocculation

Flocculation is the process of forming large agglomerates from small particle agglomerates that are already formed as a result of coagulation. Flocculation takes place through high molecular weight polymeric materials, and no substantial change of surface charge is accomplished. In the case of flocculation, the flocs are of larger size, strongly bound, and porous, whereas the agglomerates formed by coagulation are compact and loosely bound, see Figure 14 14. Flocs separation can be done by gravity sedimentation and/or filtration (Crittenden et al., 2012a; Zouboulis & Avranas, 2000).

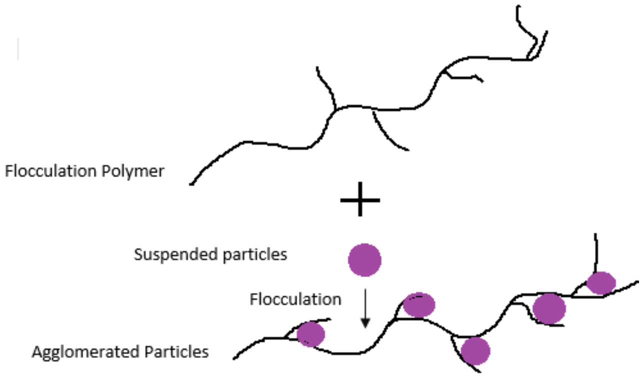

Figure 14: The Process of Flocculation

Flocculation is normally executed chemically, by adding flocculants, which are anionic or cationic synthetic polymers, or a combination of both charges. Flocculation is also executed through electrocoagulation. The polymers are commonly synthetically produced, but can also be natural organic biopolymers such as alginate and Xanthan gum. The basic flocculation mechanism is the same as the other flocculation methods, and also needs a certain stirring intensity and a certain residence time (Ødegaard, Norheim, & Norsk Vann, 2014).

Flocculation is commonly grouped into two types, microflocculation and macroflocculation. These may be accomplished without adding any chemical to the wastewater. The process of microflocculation is when particle aggregation is brought about by the random thermal motion of fluid molecules. Macroflocculation is the process when particle aggregation is brought about by inducing velocity gradients and mixing in the fluid containing the particles to be flocculated. Macroflocculation can also occur with differential settling in which larger particles overtake small particles to form larger particles (Bahadori, 2013; Crittenden et al., 2012a).

Synthetic polymer flooding is being increasingly used worldwide as a viable option for increasing recovery rates. Synthetic polymers are also used for chemical treatment of waste water or in drilling fluid increasing the viscosity. Back produced polymers from polymer flooding are ideally disposed of by reinjection or injection to a deposit well (Opsahl & Kommedal, 2017). The impact and fate of the polymers released to the sea are largely unknown beyond standardized acute toxicity and biodegradability data (Opsahl & Kommedal, 2017). Within the Norwegian Offshore Environmental regulations biodegradability is conclusive for chemical classification. Biopolymers satisfy the strict Norwegian rules and regulations criteria, while the non-biodegradability synthetic polymers do not and are prohibited from use (Opsahl & Kommedal, 2017). Modern polymer flooding uses synthetic polymers due to different factors, including the use of biocide.

3.2.4.4 Electrocoagulation

The process of electrocoagulation (EC) is electrochemical production of metal ions (such as Al and Fe) which is effective for the destabilization of dispersed fine particles and removal of dissolved matters, such as heavy metals and organics. Coagulants are formed by the electrolytic oxidation of the sacrificial electrodes, such as aluminum and iron. The EC process leads to neutralization of electric charge for removing pollutants, oppositely charged particles bond together like small magnets to form a mass. EC has been productively applied for the treatment of wastewater containing foodstuff wastes, oil wastes, dyes, chemical and mechanical polishing waste, suspended particles and heavy metals, to name some of them (Kathiravan & Muthukumar, 2011). High particulate removal efficiency, a compact treatment facility and relatively low cost are some of the advantages of EC. In addition no supplementary addition of chemical is required and this reduces sludge production (Kathiravan & Muthukumar, 2011; Sahu, Mazumdar, & Chaudhari, 2014; Yavuz & Ögütveren, 2018). However it will require the wastewater to be conductive.

The three following stages summarized below are involved in the EC process (Yavuz & Ögütveren, 2018):

1) Electrolytic oxidation of the electrode to form coagulants.
2) Destabilization and neutralization of the electrical charged contaminants and particulate suspension, and breaking of emulsions.
3) Formation of flocs by aggregation of the destabilized particles.

An EC reactor in the simplest form is made up of an electrolytic cell with one anode and one cathode. The anode material is electrochemically corroded due to oxidation when the cell is connected to an external power source (Kathiravan & Muthukumar, 2011; Sahu et al., 2014). The water and wastewater is continuously mixed with a stirrer to keep it uniform in the reactor. Cathodic reactions are encountered in the negative side, while the positive side undergoes anodic reactions during electrolysis. The charge of the particles are neutralized by the released ions, thereby coagulating. The undesirable contaminants are removed by the released ions either by chemical reaction and precipitation or by causing the colloidal materials to coalesce, which can be removed by floatation. The mechanism of EC is described in *Figure 15*, EC process involves the formation of hydroxyl ions and hydrogen gas at the cathode. The flocculated particles are with the help of generated gas raised to the water surface (Moreno-Casillas et al., 2007).

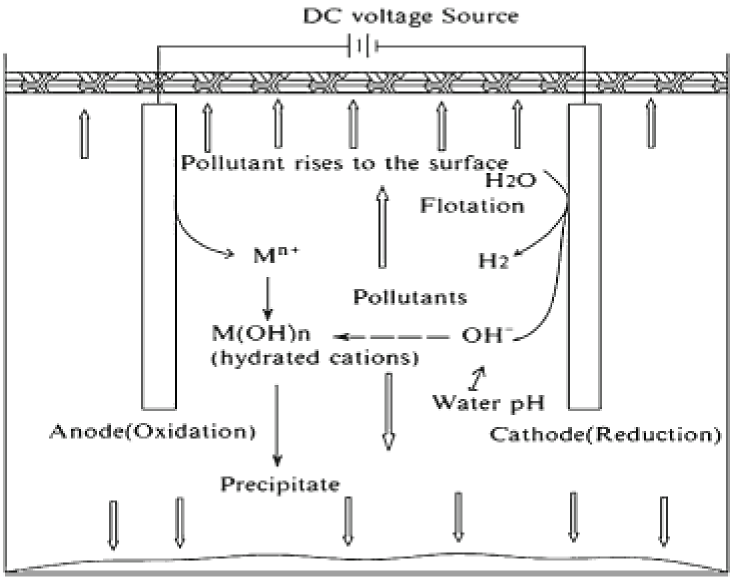

Figure 15: The mechanism of electrocoagulation (Sahu, Mazumdar, & Chaudhari, 2014)

3.2.4.5 Emulsion-breaking

Emulsion-breaking is the process of demulsifying oil/water mixture by using emulsion breaking chemicals (e.g. surfactants), in a liquid to form stable layer between the phases. Emulsions can consist of oil, water and solids combined in oily water emulsions. The surfactant is mixed in the wastewater tank to break the emulsion. The emulsion breaking surfactant will rise to the top of the tank and skimmed off, the sludge settles to the bottom of the tank. Water-soluble surfactants are preferred if the system designed to reuse separated fluids. On the other hand, oil soluble surfactants may affect the properties of drilling fluid by retaining in the recovered fluid (Ivan & Dixit, 2006; Suzuki & Maruyama, 2005; Zouboulis & Avranas, 2000).

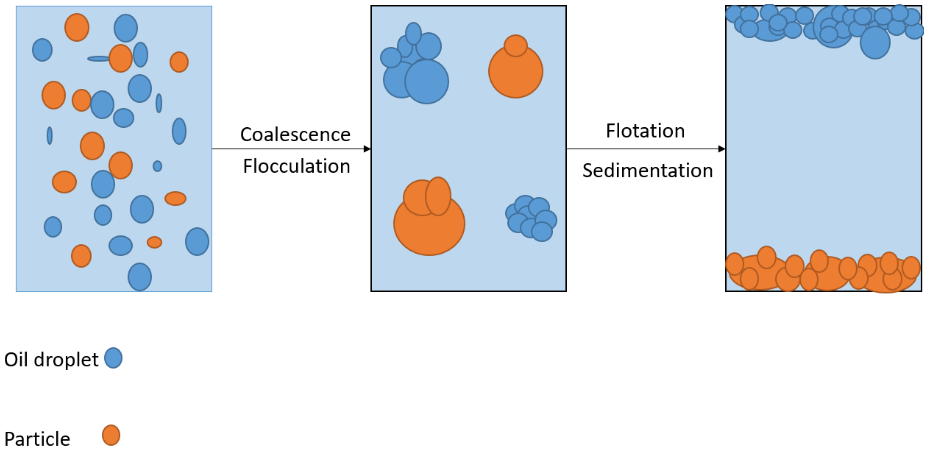

Figure 16: Emulsion breaking

Coalescence shown in the *Figure 16* involves the merging of oil droplets to form a single larger droplet, which may float and lead to phase separation, and breaking of emulsion. Particles flocculate together to bigger flocs and cause sedimentation. Distinct layer of oil droplets on top layer of an O/W emulsion, this due to the lower density of oil droplets than the surroundings, and sedimentation of particles occurs as it is illustrated in the *Figure 16* above.

3.3 Compact Offshore Treatment Units from Nature Oil & Gas AS

This section provides overviews of the compact units for offshore slop water treatment supplied to the market by Nature Oil & Gas AS, whose main interest lays in the slop water treatment.

3.3.1 Nature Long Beach Decanter Centrifuge

Long Beach Decanter Centrifuge also known as Sludge Treatment Unit (STU) is designed for treating heavier slop waste fractions such as sludge, water saturated mud etc. In combination with a decanter centrifuge this separation technology includes an emulsion breaker injection system. The decanter is designed with a high slenderness ratio, which is beneficial for slop mud. The unit is equipped to separate the solids from the water, and has been developed with the goal of producing treatable water effluent. The effluent water is further treated with the CTU. STU is best utilized in situations where there is not enough tank capacity or time on the rig to support natural gravity settlement of the heavy slops in water and solids, or the waste to shore has to be absolute minimized (remote locations). The solid fraction will be separated from the slop water and the lighter fraction will transfer into the solid control process and the water part will go to the CTU for further treatment (Nature Oil & Gas AS, 2013 -b).

STU has a small footprint, 14 ft. and is containerized as shown in *Figure 17*. The benefits with this treatment unit offshore are claimed to be:

- Cost reduction on transport of sludge/mud to shore and hazardous waste handling
- More efficient when dewater the CTU sludge
- The deck space can be saved by placing the STU on top of the CTU, due to the extra ISO fitting on STU.

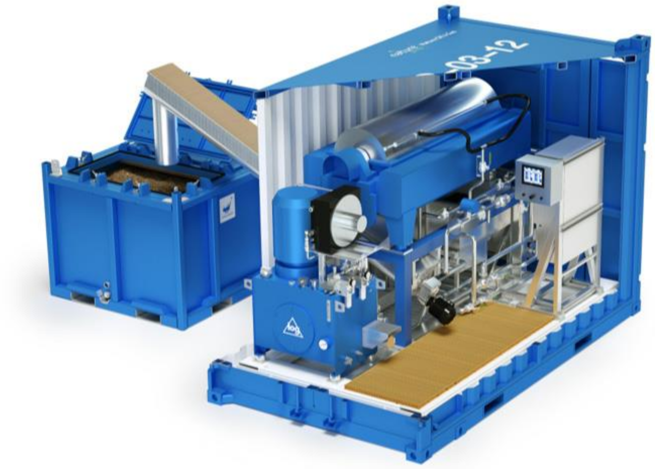

Figure 17: Nature`s 14 ft. Long Beach Decanter Centrifuge in a container

3.3.2 Nature Compact Treatment Units (CTU)

The Compact Treatment Unit (CTU) developed by Nature Oil & Gas AS is a containerized slop water processing unit. This unit enables an operator to process, and discharge, the slop water generated on a rig offshore, rather than transporting it to a treatment facility onshore (*Figure 18*). The advantages are reduced cost and risk to the environment. The treatment system is built into a 12 ft. container in compliance with the NORSOK Z- 015 standard and approval for ATEX 11/3G (Nature Oil & Gas AS, 2013 -a).

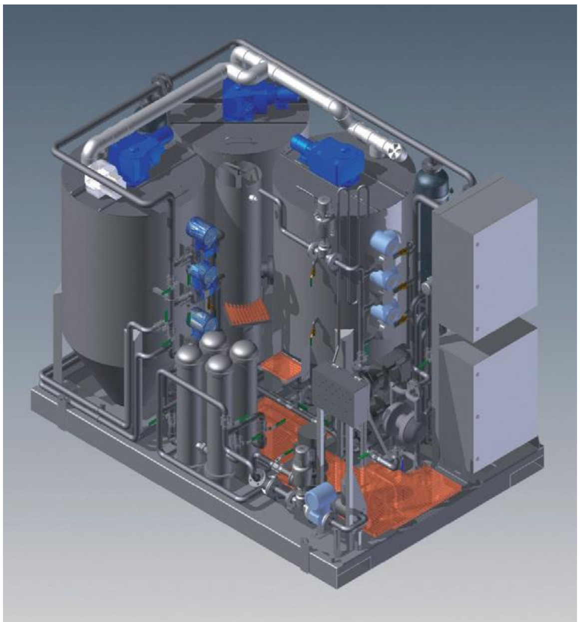

Figure 18: Nature CTU in a container with small footprint

Slop collected in a tank is connected with CTU for treatment. The CTU treats the slops from the slop tank and processes it into a waste stream with low oil content and a small sediment fraction. The process is based on a chemical and physical coagulation and flocculation process performed in a 1-3 stage DAF system (Nature Oil & Gas AS, 2013 -a).

The CTU separation technology includes coagulation, flocculation, dissolved air flotation with dosing pumps and control system. Dissolved air in water is brought into the chambers for flotation of the flocculated particles. The produced sludge on the top of the water is skimmed off and discharged to sludge skip. Depending on the water characteristics, CTU offshore reduces onshore delivery of hazardous waste from the rig up to 95%. In addition, the production of H_2S during transportation and storage is reduced. The water treated by CTU is released offshore. The *Figure 19* shows the different treatments in the CTU.

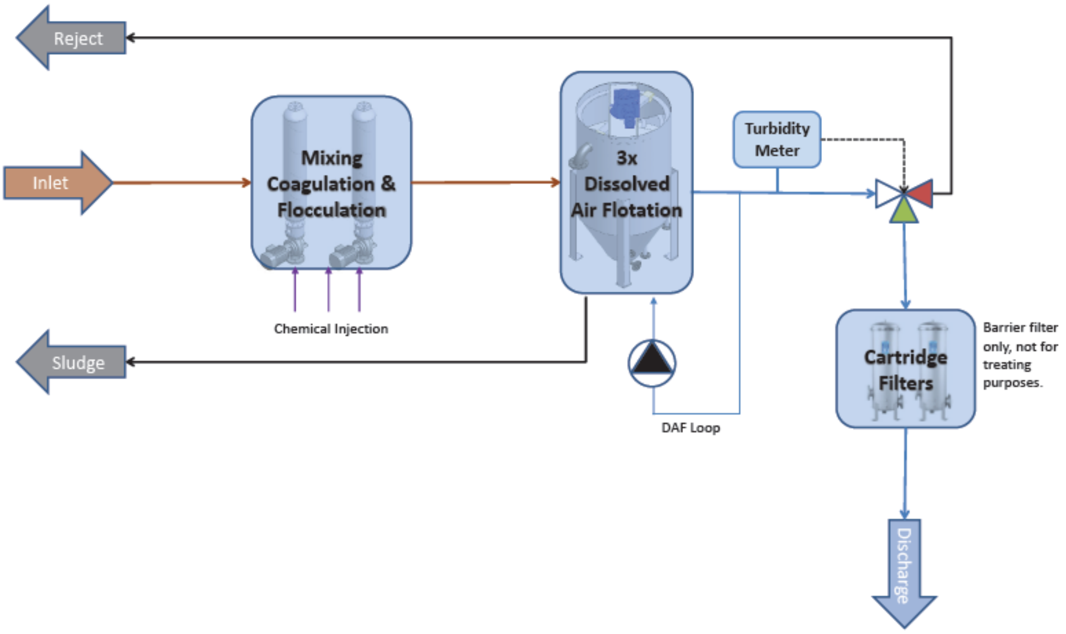

Figure 19: Operation system of a CTU

3.4 Comparison of Treatment Technologies Used Offshore

In this section different combination treatment technologies used offshore are compared in *Table 1*. In addition, companies providing the technologies, application area and advantages and disadvantages are summarized in *Table 1*.

Table 1: Comparison of treatment technologies for offshore wastewater treatment provided by different companies

Technology	Company	Treated	Treatment Method	Application	Advantages	Disadvantages
Compact Treatment Unit, CTU	Nature Oil & Gas AS	Offshore	Coagulation/Flocculation + Dissolved Air Flotation	Slop water and produced water treatment	- High treatment capacity - Low power consumption - Good effluent quality	- Requires chemicals - Complex operation
Sludge Treatment Unit, STU	Nature Oil & Gas AS	Offshore	Coagulation/Flocculation + Chemical Emulsion Breaker + Decanter Centrifuge	Sludge and mud treatment	- High treatment capacity	- Requires chemicals - Cannot separate low density permeate
Water Treatment Unit, WTU	Halliburton	Offshore	Coagulation/Flocculation + Dissolved Air Flotation	Slop and produced water treatment	- Easy to enter the marked	- Varying results - High footprint - Low treatment capacity - Equipment built for general use, not specific for slop treatment
Slop Treatment Unit	OTS AS (Rental company, main client Soiltech)	Offshore	Decanter Centrifuge + Centrifuge Separator + Particle/Oil Adsorbing Filter	Slop treatment	- Easy to operate - No chemicals - All in one unit	- High footprint - Low treatment capacity - Still under development

Decanter Unit	OTS AS (Rental company, main client Soiltech)	Offshore	Decanter Centrifuge	Slop treatment	- Easy to operate - No chemicals	- High footprint - No internal process
Renapure PW and Renapute SW	Rena Technology, part of IKM	Offshore	Membrane Filtration	Produced water treatment	- Easy to operate - No chemicals	- Costly replacement filters - Sensitive for Sludge/mud - High footprint - Not proven technology
Renapure SW with decanter	Rena Technology, part of IKM	Offshore	Membrane Filtration + Decanter Centrifuge	Slop treatment	- Easy to operate - No chemicals - All in one unit	- Costly replacement filters - Very low capacity for sludge treatment - High footprint - Not proven technology
Slop Treatment Unit	Soiltech AS	Offshore	Decanter Centrifuge + Centrifuge Separator + Particle/Oil Adsorbing filter	Slop treatment	- Easy to operate - No chemicals All in one unit	- High footprint - Still under development - Low treatment capacity
Envirounit	MI Swaco	Offshore	Settling	Slop treatment	- Robust - For sludge and water	- Produce a lot of sludge - Very high footprint - Very low treatment capacity

4. Polymers

A polymer is a large molecule that is buildup of one or more repeat units of small molecules known as monomers. Polymers can be anionic, cationic or non-ionic dependent on which reacting group are bounded to the polymer. The polymerchain can be changed by chemical treatment or cross-linking, which changes the physical, chemical and mechanical properties of the polymer. A polymer is water soluble only if it has the ionic property. The charges make polymers more dependent on the pH (optimum at pH = 8.5-9.0) and the concentration of salt to work satisfactory. In Oil and Gas industry non-ionic polymers are mostly utilized (Hodne & Saasen, 2015 ; Rellegadla, Prajapat, & Agrawal, 2017).

Polymers have a different function in the drilling fluid, i.e. bring particles in suspension, increase the viscosity and control lost circulation. Biopolymers added in drilling fluids may be exposed to biodegradation, therefore biocides are used.

Polymers may be dissolved in seawater, but the properties of synthetic polymers decreases with increasing salt concentration and causes larger quantities of polymers used. Some requires removal of divalent ions, such as Ca^{2+} and Mg^{2+} before use. High pH (over 11) may destroy the polymer and pH less than 5, the polymers may not act optimal.

Different types of polymers are used in the Oil and Gas industry depending on the purpose. The polymers varies widely, such as Starch, Cellulose and Cellulose derivatives, Xanthan Gum, Carboxyl Methyl Cellulose, and Poly non-ionic Cellulose (PAC), and Hydroxyl Ethyl Cellulose.

4.1 Xanthan Gum

The important research publications on the production of XG appeared in 1961. It was discovered by the research laboratories of the US Department of Agriculture, that the pathogen bacterium Xanthomonas campestris found on cabbage plants produces an extracellular heteropolysaccharide (EPS= Xanthan Gum) under aerobic conditions with exceptional rheological properties (Bergmann, Furth, & Mayer, 2008; Katzbauer, 1998; Tako, Teruya, Tamaki, & Ohkawa, 2010). A number of improvements and research in polysaccharide manufacture has been done. Commercially XG is the most important microbial polysaccharide.

XG has been used in the wide range of contexts. Xanthan solutions show thickening properties, with a pseudoplastic behaviour, which are used in food, cosmetic, pharmaceutical, paper, textile, mining and oil drilling industries (Diana, Carolina, Isabel, Margarida, & Fernão, 2016; Tako et al., 2010). Similarly to cellulose xanthan is stable over a wide range of pH and changes in temperature. By combining XG with other polysaccharide or proteins it is possible to change the mechanical and rheological properties of XG (Diana et al., 2016).

In some cases it is important to break the viscosity of an aqueous fluid. In U.S. Pat.NO 5,054,552 researched on breaker system for aqueous fluids containing XG, which is particularly useful in a fractured formation. They invented a breaker system for aqueous fluid containing xanthan gum thickeners wherein a combination of two oxidizers, an organic acid, and two catalysts were used as a breaker. This invention does not only reduce the viscosity but provides a more complete break of the gel (Hall, Szemenyei, & Gupta, 1991).

4.1.1 Xanthan Gum Structure

Xanthan Gum represents an anionic, branched hetero- polysaccharide. The primary structure of XG is as in cellulose a (1→4)-linked β –D-glucan backbone, and every second glucose unit carries a trisaccharide side chain composed of β-D-Mannose-(1→4)- β-D-Glucuronic Acid-(1→2)-α-D-Mannose-(1→is substituted at O-3 of alternate D-glucosyl residues. The internal D-mannose of side chain is substituted at C-6 with an acetyl group (Bergmann et al., 2008; Diana et al., 2016; Katzbauer, 1998; Tako et al., 2010; Yajima, Imai, Poplawsky, Nukada, & Yabuta, 2010). On approximately one half of the terminal D-mannose residues a pyruvic acid moiety is joined by a ketal linkage. The schematic representation of a representative section of Xanthan molecule is presented in *Figure 20*.

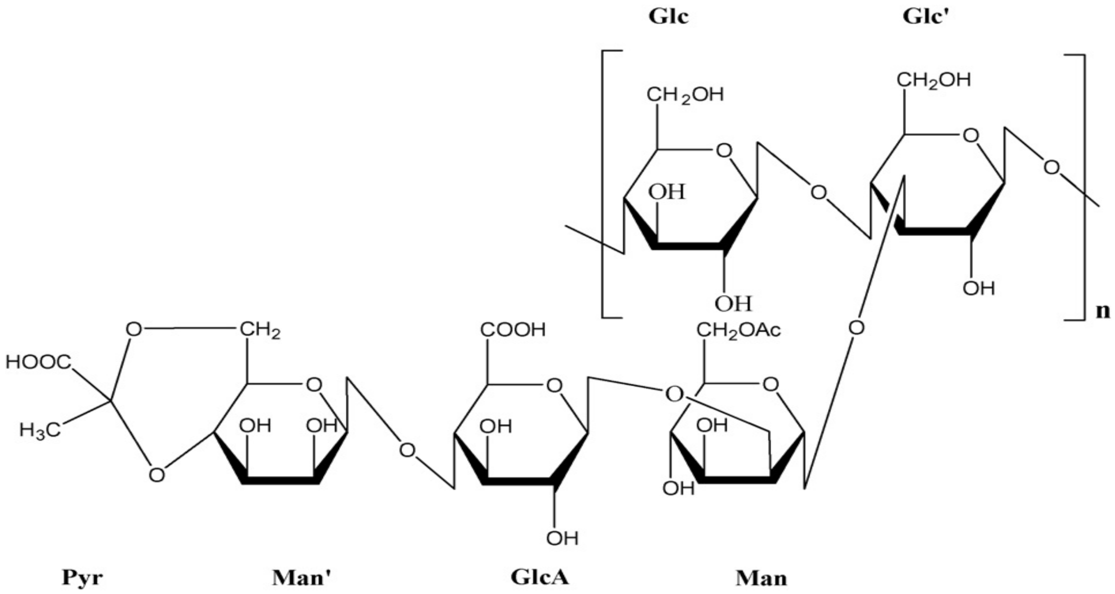

Figure 20: Representative section of Xanthan gum structure (Bergmann, Furth, & Mayer, 2008; Rellegadla, Prajapat, & Agrawal, 2017)

4.1.2 Production of Xanthan Gum

In the production of XG, Xanthomonas Campestris is used. Xanthomonas Campestris is cultured in a well- aerated and well-agitated fermenter. The media contains carbohydrate source, such as glucose, a nitrogen source that suits and a nutrient salts. Both compounds are heated when the fermentation is completed to kill the bacteria and the XG is recovered by precipitation with isopropyl alcohol (Katzbauer, 1998; Tao et al., 2012).

4.1.3 Xanthan Gums Properties

In many industries XG is employed as a thickener. In the paper mill and textile industries xanthan is stabilizing the suspensions and the emulsions. Xanthan`s ability to solve in hot or cold water, high viscosity at low concentration, and stability in acid system is the cause of employing it in the food industry. It also has excellent suspending properties owing to a high yield value and also an ability to provide good freeze-thaw stability (Casas, Santos, & García-Ochoa, 2000; Diana et al., 2016).

Bacterial growth conditions, operational conditions and the Xanthomonas Cp. used may affect the levels of pyruvic acid and acetal substitutions in the molecule. The change in polymer nature affect the xanthan solution rheological properties, they depend on average molecular weight and acetate and pyruvate contents (Casas et al., 2000). In aqueous solution, solvation of xanthan leads to a partial expansion of the molecular structure connected to a corresponding change in solution viscosity (Bergmann et al., 2008).

4.1.4 Xanthan Gums Use in Oil and Gas Production

XG is used for different purpose in the oil and gas industry, such as in drilling fluid or under drilling operations. XG in drilling fluid is used to have the right viscosity of the fluid to bring the drill cutting to the platform. Another purpose the XG in the Oil and Gas industry is for polymer flooding for Enhanced Oil Recovery (EOR), and for slurryfication (injection).

4.1.4.1 Xanthan Gum Used in Drilling Fluid

Drilling fluid with XG is an important component circulating in the well bore to facilitate in the drilling process for different purpose. The main and most important functions of drilling fluids are to transport the drill cuttings, lubricate the drill bit, avoid and prevent the influx of lost circulation and maintain the hydrostatic pressure (Jha, Mahto, & Saxena, 2016; Sheng, 2010, p. 207). Different types of drilling fluids are used during the drilling operation for oil and gas, and type of drilling fluid chosen depends mainly on the formation being drilled, depth, pressure of the well bore and the mechanical resistance (Jha et al., 2016; Masi, Molaschi, Zausa, & Michelez, 2011).

One type of drilling fluid is emulsion drilling fluids which consist of a water-insoluble liquid such as oil, a colloidal material, viscous material and water together with a suitable emulsifier which can stabilize the emulsion systems (Jha et al., 2016). The main purpose of the emulsifiers is to support the emulsion system and give a colloidal stability, and an emulsifier may be molecular surfactants, such as polymer, proteins or fatty acids or alcohols (Jha et al., 2016).

Key parameters which influence important aspects of drilling operations are rheological and filtration properties of drilling fluids. The drilling fluids cycle of flow in the well experience wide range of shear rate, therefore recommended that fluids have strong shear thinning behavior. XG works as a viscosity modifier and emulsifier in O/W emulsions drilling fluids, the rheological properties of these solutions are constant over a wide range of salt concentrations. Other properties that make XG an effective additive for drilling fluids are its potential for cross linking and shear thinning features, and ability to stabilize emulsions and reduce dispersion and temperature stability (Jha et al., 2016; Masi et al., 2011).

4.1.4.2 Xanthan Gum Used for Enhanced Oil Recovery

First stage of oil production is the differential pressure between the reservoirs and wellbore that are responsible for driving oil out of the production well. This is referred as primary production since this process only recovers about 10% of the original oil in place (OOIP). As time pass the pressure in the reservoir decreases, also the oil recovery decreases which leads to implementation of secondary recovery. The injection of an external fluid, such as water and gas, is the part of secondary recovery, which is done to maintain reservoir pressure and displacing oil towards the wellbore. To increase oil production the oil industries adopted enhanced oil recovery (EOR) process (Rellegadla et al., 2017; Sheng, 2010). This is done by improving oil flow and sweep efficiency in the reservoir. The sweep efficiency is improved by polymer-based EOR and the water to oil mobility ratio is lowered. Different types of polymers and polymer-related profile control systems are used in EOR. Mostly two different types of polymers are currently used, a synthetic polymer such as polyacrylamide and a biologically produced polymer such as XG and cellulose. Biopolymers lowers the water to oil mobility ratio, it also selectively plugs the high-permeability hidden zones and leads to a redirection of the water-flood to unreachable oil zones (Rellegadla et al., 2017).

Some important properties with the use of XG in EOR are the less sensitivity to relative high concentrations of salt and its tolerance to high mechanical shearing, which may not affect the viscosity of XG solution. High mechanical shearing tolerance makes XG easier to handle in field injection pumps, transportation and related oilfield equipment. Some disadvantages associated with the use of XG are the formation plugging characteristics and the highly sensitive to susceptibility to bacterial attack. Remediating by filtration or additional processing can be applied to prevent plugging and addition of biocides bacterial degradation can be prevented (Chang, 1978; Rellegadla et al., 2017).

However, polyacrylamide is less susceptible to bacterial attack than XG, because XG chains are broken down from polysaccharides to monosaccharides by bacteria, which then can be used in metabolism. On the other hand, polyacrylamides cannot be broken down extracellularly and used in the metabolism (Chang, 1978). This is one of the reasons why XG is on the PLONOR list and not polyacrylamide (OSPAR Commission, 2013a).

XG solution appears to have better injectivity rather than powder forms. Increased shipping costs and extra storage facilities are associated disadvantages. XG is more severe than polyacrylamide since XG contains more than 96 % water (Chang, 1978). Polyacrylamide is highly sensitive to mechanical degradation, which presents practical problems in field operations. Mechanical shear applied to polyacrylamide solution is minimized by use of special techniques during mixing and delivering.

4.2 Hydrolyzed polyacrylamide

Hydrolyzed polyacrylamide (HPAM) is one of the polymer that is widely used in EOR applications. This polymer solutions have provided significantly greater oil recovery (Sheng, 2010). HPAM solutions exhibit significantly greater viscosity than xanthan solutions and are therefore widely used for EOR.

HPAM is a copolymer of acrylamide and acrylic acid. Due to charge repulsions of the carboxylic group in fresh water the viscosity is high. This because the HPAM flexible chains structure is stretched. HPAM flexible chains are compressed in saline water, since the charge is neutralized or shielded, which results in low viscosity. For large-scale production HPAM has been used in many fields because less cost. Other polyacrylamide-derived polymers used in EOR includes salinity tolerant polyacrylamide and hydrophobically associating polymers. Due to its low biodegradability, HPAM containing chemicals are categorized as a red chemical.

4.3 Less Commonly Used Polymers

Different polymers are used for various purposes in the Oil and Gas industry. Some polymers are used for EOR, while others are used to prevent lost circulation and to bring drill cutting to the top of the platforms. Some polymers are less used in the Oil and Gas industry and are roughly described in the sections below (Hodne & Saasen, 2015).

4.3.1 Guar Gum

Guar Gum (GG) is the powdered endosperm of the seeds of the Cyamopsis tetragonolobus that contain galactomannan, which is a complex polysaccharide, organically grown. This polymer is hydroxyl group rich with high molecular weight, which consists of d-mannose backbone, on which d-galactose units are attached as side-chains. Hydrogen-bonds are formed between these functional groups and water to impart viscosity and thickening to the solution. GG specialty is its ability to hydrate quickly to produce viscous solution even in cold water (Thombare, Jha, Mishra, & Siddiqui, 2017).

The Cross-linking of GG and its derivatives have revealed tremendous potential to be exploited as flocculating agents (Rellegadla et al., 2017; Thombare et al., 2017). By cross-linking GG with borax hydrogel may be produced. This cross-linking can be synthesized at room temperature in single step. The mechanism of synthesis of borax cross-linked guar gum hydrogels is described in *Figure 21*.

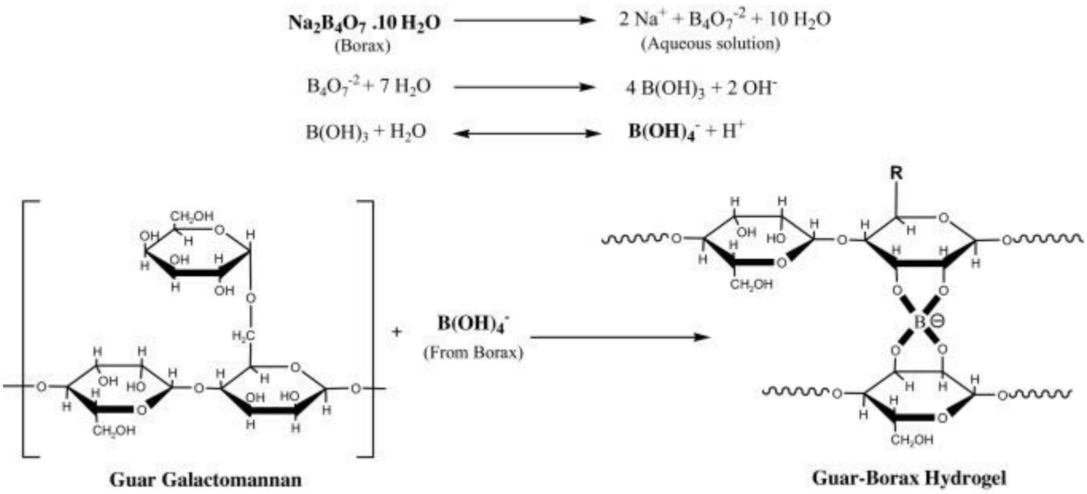

Figure 21: Mechanism of synthesis of borax cross-linked guar gum hydrogels (Thombare, Jha, Mishra, & Siddiqui, 2017)

4.3.2 Sodium Carboxyl Methyl Cellulose

Carboxyl Methyl Cellulose (CMC) is produced from wood and cotton in a chemical reactor in an organic solvent and addition of sufficient amounts of NaOH and Monochloroacetic acid to provide the desired properties. By adding different chemicals to CMC various properties may be obtained, and high viscosity is build-up by long chains. CMC is stable to 150°C. The effectivity of CMC may decrease with increasing salt concentrations. Its resistance to Ca^{2+} is low (Hodne & Saasen, 2015).

4.3.3 Hydroxyl Ethyl Cellulose

Hydroxyl Ethyl Cellulose (HEC) is produced by soaking cellulose in NaOH and then react with Ethylene Oxide. HEC is a non-ionic but is water soluble due to the hydroxyl ethylene groups, which increases the possibility of hydrogen binding in the molecules.

Compared to CMC the viscosity of HEC is lower. HEC also give lower gel strength and therefore have poor ability to hold the particles in suspension. Drilling fluids containing HEC may make foam and antifoam have to be used.

4.4 Comparison of Polymers

In this chapter polymers characteristics and the purpose of using them in petroleum industry are compared and presented in *Table 2*.

Table 2: Comparison of polymers

Name	**Type of Polymers**	**Characteristics**	**Purpose in Petroleum Industry**
Xanthan Gum	Biopolymer	- Thickening properties - Dissolves in cold and hot water - Stable over a wide range of pH and temperature - Biodegradable	- EOR - Drilling fluid for particles in suspension - Reduce lost circulation
Hydrolyzed polyacrylamide	Homopolymer (synthetically based)	- Exhibit significant greater viscosity than XG - Flexible chain structure compressed in saline water - Less costly	- EOR
Guar Gum	Biopolymer	- Organically grown - Hydrate quickly to produce viscous solutions even in cold water - Cross-linking of GG exploited as flocculating agents	- Drilling fluid for suspension of particles
Sodium Carboxyl methyl cellulose (CMC)	Homopolymer (Plant origin)	- Produced from wood and cotton - Property easily changed by adding different chemicals	- EOR - Injection in wells

		- Effectivity is salt sensitive	
Hydroxyl Ethyl cellulose	Homopolymer (Non-ionic)	- Water soluble because of hydroxyl ethylene - Low viscosity - Lower gel strength	- Widely used in EOR - Less used in fluid

4.5 Polymers in Slop Water

Polymers have several different functions in Oil and Gas industry, these functions includes (Devereux, 2012) (Lucas, Mansur, Spinelli, & Queirós, 2009);

- Increase in the viscosity of the fluid. It has to be taken in to account that a lot of pressure is needed to pump a highly viscous fluid through a pipe than a fluid with lower viscosity.
- Increase gelation properties. A gel is formed when the fluid becomes stationary. Suspended solids and drill cuttings in the mud are allowed to be suspended within the gel so that they do not sediment through the mud.
- Decrease fluid loss into the formation.
- Act as a surfactant. Oil and water are mixed together to an emulsion.
- Highly shear – thinning. Xanthan Gum is for example used to provide high viscosity at very low shear rates.

When drilling the first section hole during the exploration drilling large amount of rock cuttings with mud will be produced and returns to the platforms. Mud returns to the platform may contain drill cuttings and different chemicals, such polymers.

Polymers used for different proposes in drilling operations returns polluted to the platform with changed properties, and therefore are routed as wastes in slop containers. Properties of polymers will change due to high pressure, high temperature, reaction with other chemicals and other factors.

5. Materials & Methods

In this chapter experimental commitment was formed to examine the potential for improving the treatment of high viscous slop water. It was found out that high viscous slop water contained XG. The testing was therefore executed on XG. The aim of the experiment is to find an optimal treatment method of the viscosity of XG. Due to the rheological properties of XG, the testing of XG has to be based on empirical laboratory tests with chemical treatment and centrifuge. This chapter describes materials and procedure of the experiments. Various parameters are considered for the study of XG properties, and the effect of XG mixed with slop.

5.1 Materials

Jar testers, Stuart SW-6 with two and six paddles were used for mixing in this study. For pH measurement, a pH-meter Hanna Instruments HI98127 was used. A 1-5 ml pipette (Thermo Scientific) was used for chemical addition. Same analytical balance was used for all experiment done in the lab, and was mostly used for weighing plastic bottles before and after centrifuge, solid Xanthan Gum and chemicals. *Table 3* summarizes all chemicals used and the purpose.

Table 3: Chemicals used in the experiment

Chemical	**Formula**	**Purpose**	**Colour categorized**
Ammonium Peroxodisulfate	$(NH_4)_2S_2O_8$	Oxidizer, breakdown XG structure	
Citric Acid Monohydrate	$C_6H_8O_7$	Organic Acid, breakdown XG structure	
Ethylene diamine Tetracetic Acid, EDTA	$C_{10}H_{16}N_2O_8$	Catalyst, breakdown XG structure	
Ferrous Sulfate	$FeSO_4 \times 7H_2O$	Catalyst, breakdown XG structure	
Floc 3 (Flotreat DR-11506)		Flocculation	Red
Hydrogen chloride, 5M	HCl	Decrease pH	Green

Masava Tank Cleaner		Protein dissolving property	Yellow
Monoethyleneglycol	$C_2H_6O_2$	Decrease XG viscosity??	Green
Nature NIC		Coagulation	Green
Nature NSC		Coagulation	Green
Nature NSP		Flocculation	Green
Sodium hydroxide, (Nature pH^+)	25 % v/w NaOH	Increase pH	Yellow
Sodium Percarbonate	Na_2CO_3	Oxidizer, breakdown XG structure	Red
Vinegar Essence 33%	CH_3COOH	Decrease XG viscosity	Green
Zirconium Oxychloride Solution (Zirconyl chloride dissolved in water)	$ZrOCl_2*8H_2O$	Decrease XG viscosity	Yellow

Coagulants focused in this study were Nature NIC and Nature NSC.

Nature NIC consist of:

Di Iron (III) Sulphate: 40 – 50 %

Iron (II) Sulphate: 0.1 – 1.5 %

Sulfuric Acid: 0 – 1 %

Nature NSC main components was:

PAC – Poly Aluminium Chloride

Reaction described in equation 5.1 occurs when NaOH is added to Nature NIC. Ferrous Sulphate and Sodium Sulphate are soluble in water, this reaction cause a solid Ferrous Hydroxide to be precipitated. Ferrous (III) hydroxide is brown, and a brown/red precipitate is formed.

$$FeSO_{4\,(aq)} + 2\,NaOH_{(aq)} \rightarrow Fe(OH)_{2(s)} + Na_2SO_{4}{}_{(aq)} \qquad 5.1$$

5.2 Methods

In this section the processes executed in the lab is described. A compound used to produce “viscosity pills”, Xanthan Gum (Duo –TEC$^+$ NS) dissolved in freshwater was tested in the lab. This was done to simulate the fluid utilized in offshore drilling activities. Before collecting a sample the XG $_{(aq)}$ was stirred thoroughly in the original container. The pH of non-treated XG was measured to be at 4.65 and for other samples pH was increased with a NaOH 25 % w/v. It was observed air bubbles in samples collected from the container in the start compared to those collected later.

5.2.1 Processes

Process executed in the lab is described in this section. *Figure 22* shows an overview of what was done in lab, and the processes are explained in details below.

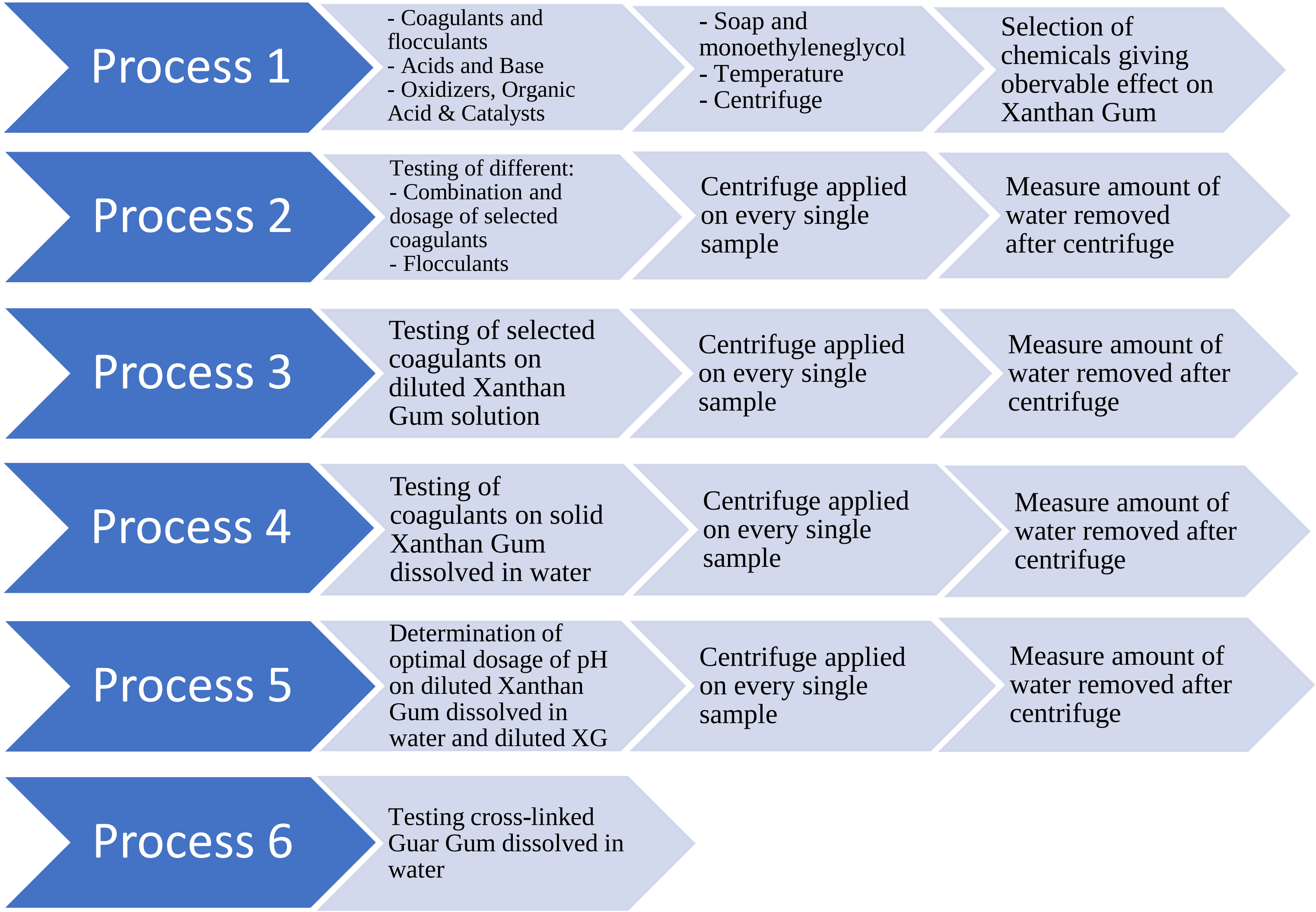

Figure 22: Overview of lab experiment

5.2.1.1 Process 1

First process executed in the lab was testing of different combinations of chemicals for treating XG dissolved in tap water (to simulate "drill water") to figure out which chemical or physical treatment had an effect on the viscosity of XG (desired effect is decrease in viscosity). This testing was conducted as a "screening", to determine the effect of different additive on the fluid.

XG 0.7 wt. % was poured in 1 L beakers for chemical testing experiments. Different dosage of chemicals were added to XG solution. Some of the chemicals added in these samples gave observable effect, meanwhile other chemicals and physical treatment, such as temperature and centrifuge, did not have any apparent effect. The most important was to determine the effects. Following chemicals were tested on XG $_{(aq)}$, *Table 4*:

- Nature NSC
- NaOH
- HCl
- Nature NIC
- Zirconium
- Nature NSP
- Vinegar Essence 35%
- Floc 3 (Flotreate D11506)

Table 4: Testing of different chemicals and physical treatment on xanthan dissolved in fresh water

Sample	**Oboy Standard**	**1**	**2**	**3**	**4**	**6**	**7**	**8**	**9**	**10**	**11**
Sample [ml]	1000	150	150	100	150	150	150	150	300	209 [g]	204 [g]
Temperature [°C]	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	100	N/A	N/A
Chemical Added	NSC pH$^+$	NaOH	HCl	NIC	Zirconium	NPS	Vinigar essence 35%	Floc 3	N/A	N/A	NIC
Volume chemical added [ml]	1,0 1,0	8,0	10	1,5	2,5	10	10	5	N/A	N/A	4
Centrifuge	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	3 min	3 min

By combining chemical precipitation (Nature NIC) with centrifuge less viscous solution was obtained and more water separated from xanthan floc was observed.

Untreated samples were highly viscous and air bubbles were observed, as shown in *Figure 25*. This untreated solution was viscous liquid, which adhered to the beaker without formation of flocs.

Testing of different Oxidizers, Organic Acid and Catalyst

The XG $_{(aq)}$ was precipitated with the use of coagulants and flocculants with varied results. Instead of precipitating the XG $_{(aq)}$ it was tried to break the viscosity in the lab. Therefore oxidizers, organic acid and catalyst, such as, Ammonium Peroxodisulfate $(NH_4)_2S_2O_8$, Sodium Percarbonate, Na_2CO_3, Citric Acid Monohydrate $C_6H_8O_7$, Ferrous Sulfate $FeSO_4{*}7H_2O$ and Ethylene Diamine Tetracetic Acid, EDTA were used to see the effect on XG $_{(aq)}$. Amount of chemicals added to XG is summarized in *Table 4*. These chemicals were added separately in different beakers and in the last beaker all chemicals were added together.

The first beaker was left blank and in second beaker $(NH_4)_2S_2O_8$ (0.7 g) was added and stirred for 20 minutes, following step was performed for $C_6H_8O_7$ (0.2 g)$_,$ $FeSO_4{*}7H_2O$ (0.5 g) and EDTA (0.6 g). In last beaker all these chemicals were added in same dosage as mentioned above. No effect on the XG were observed in beakers with individual chemicals, meanwhile some changes in viscosity were observed in beaker with all chemicals.

Testing of Soap and Monoethyleneglycol

“Masava Tank Cleaner” also used as soap to clean oil offshore was tested on XG $_{(aq)}$ solution. In addition, Monoethyleneglycol (MEG) was tested on XG $_{(aq)}$. The amount of substances added to the XG $_{(aq)}$ is summarized in *Table 6*.

In 1 L beakers, 500 ml and XG $_{(aq)}$ were added. In one of the beaker Masava Tank Cleaner (3 g) was added and in the other beaker Monoethyleneglycol (7,6 g) was added. Non-observable effects were detected in these beakers. The pH of XG $_{(aq)}$ containing Masava Tank Cleaner increased from 4.75 to 12.13 and decreased from 4.75 to 4.20 for the XG $_{(aq)}$ containing Monoethyleneglycol.

5.2.1.1.1 Conclusion from Process 1

It was concluded to use for further test Nature NIC and Nature NPC, since flocculation of XG $_{(aq)}$ was observed when these coagulants were added to XG $_{(aq)}$. Xanthan Gum behaves as a flocculants due to its polymeric properties, therefore additional polymer was not added in some samples.

5.2.1.2 Process 2

Testing different combination and dosage of coagulants on viscosity pills

In this section different combination of Nature NSC and Nature NIC were evaluated. The pH of the blank sample (no addition of chemicals) was measured to be 4.75. *Figure 23* shows an overview of how testing on Xanthan Gum was executed.

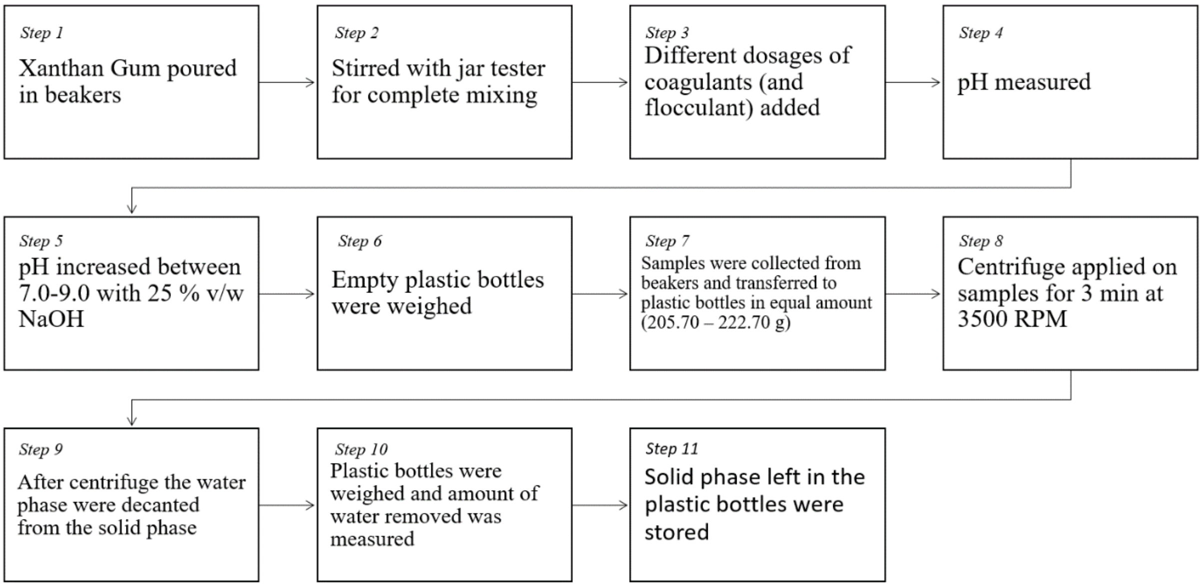

Figure 23: Overview of testing on Xanthan Gum

Testing of different combinations and dosages of coagulants on XG $_{(aq)}$ were performed in three different ways, as shown in *Table 7*. In section 1 from *Table 7*, XG $_{(aq)}$ was poured into five beakers, the dosage of Nature NSC varied from 0.5 ml to 2.5 ml and 0.5 ml Nature NIC was added in all five beakers and stirred for 10 minutes with a jar tester. The pH was not increased.

Stirring paddles were pulled up from the beaker and flocs were observed in the beaker surrounded around the paddles, some unreacted viscous XG $_{(aq)}$ were observed in the corner of the beakers. The flocs surrounded around the paddles were pulled off.

Samples were collected from the beakers and transferred to plastic bottles with spoon for centrifuge (Hettich ROTOFIX 46 benchtop). Approximately equal amount of samples from

205.7-206.30 were transferred to the plastic bottles before centrifugation. This was important for balance out the weight in the centrifuge. The centrifuge was applied on the sample for 3 minutes at 3500 RPM. *Figure 24* shows the centrifuge used and in the four buckets the plastic bottles with samples were placed.

Figure 24: Hettich ROTOFIX 46 Benchtop Centrifuge (Hettich Instruments, 2018)

Hard and big flocs were difficult to transfer due to small opening of the plastic bottle, therefore flocs were cut in smaller pieces with the spoon. In some plastic bottles more water was transferred from the beakers, and in others more flocs and viscous XG were transferred before centrifuge was applied. This dependent on the shape, size and resistance of the flocs, see *Figure 35*

In section 2 from *Table 7*, 0.5 ml of Nature NSC were added in all 5 samples and amount of Nature NIC added varied from 1.0 ml to 3.0 ml. The pH of the samples were increased between 7.0 and 9.0 with varying dosage (2.0 – 4.0 ml) with 25 % w/v NaOH and stirred for 10 minutes. It was observed clearly separation of water when increasing the pH with NaOH. Steps 6 to 11 shown in *Figure 23* were followed.

In sample 6 0.5 ml of Nature NSC and 1.0 ml of Nature NIC were added and the pH was increased by adding 4.0 ml of NaOH, see *Table 7***.** After centrifuge 55.50 g of water separated from XG $_{(aq)}$ and decanted from the sample. *Figure 34* shows sample 8 after chemically treated with coagulants and NaOH. Sample to left on *Figure 34* is before centrifuge and the sample to right is after centrifuge, which also shows the thick layer of water separated from XG $_{(aq)}$.

In section 3 from *Table 7*, the dosage of Nature NSC varied from 1.0 to 3.0 ml equivalent amount of Nature NIC were added. The pH was increased between 7.0 and 9.0 with 25 % w/v NaOH. Further, steps 6 to 11 shown in *Figure 23* were followed.

Similar observation was observed for these samples as for samples six to ten, immediately separation of water when addition of NaOH and thicker layer of water separated from XG $_{(aq)}$ after centrifugation.

The sample shown to left in *Figure 35* is an example of a hard and compact floc, and the sample in middle is an example of small and soft flocs. The last sample to the right in *Figure 35* is an example of XG $_{(aq)}$ that shows minimum reaction with the coagulants and it is highly viscous and sticky, which was easiest to pour over from the beaker to the plastic bottles. Due to hard and compact flocs less water was transferred from the beaker to the plastic bottles but more of the flocs were added by making the flocs smaller. More water was poured from the middle sample due to the soft flocs, which were well mixed with the water.

Testing of individual coagulants with and without flocculant

In this section individual coagulants were tested to see the effect on XG $_{(aq)}$. Amount of Nature NSC tested on XG $_{(aq)}$ was 2.0 and 2.5 ml, and amount of Nature NIC tested was 2.0 and 2.5 ml. In addition, the individual coagulants were combined with flocculant to see if bigger flocculants were obtained. Nature NPC was used as flocculant 1.0 ml was added in 2.0 ml of Nature NSC and 1.0 ml was added in 1.5 ml Nature NIC. The pH was increased between 7.0 and 9.0 with 25 % w/v NaOH. Further steps 6 to 11 were followed, as shown in *Figure 23*.

Before adding flocculant to the samples with coagulant some water was separated, but flocculated with XG immediately after addition of flocculant.

5.2.1.3 Process 3

In this section dilution of XG $_{(aq)}$ is discussed. XG $_{(aq)}$ was diluted by 50 and 75 % slop water.

Testing of coagulants on 50% diluted Xanthan Gum solution by Slop Water

In this step XG $_{(aq)}$ was diluted with 50% slop water given by Oseberg ØST. Slop water was poured into the container containing XG $_{(aq)}$ and the solution was well mixed before samples were collected for testing. Four tests were executed, where same amount of Nature NSC and Nature NIC were added in the first two samples (2.0 and 2.0 ml, 2.5 and 2.5 ml) and in the last two samples different amount of Nature NSC and Nature NIC were added (2.5 and 2.0, 2.0 and

2.5), see *Table 9*. The pH was increased between 7.0 and 9.0 with 1.5 – 2.0 25 % w/v NaOH. All steps mentioned in *Figure 23* were systematically followed until the end.

Testing of coagulants on 75 % diluted Xanthan Gum by Slop Water

XG $_{(aq)}$ was diluted with 75 % slop water in this step. Slop water was poured in container containing XG $_{(aq)}$ and diluted it with75 % slop water, the ratio was 25/75. One beaker was left blank, no chemical was added to the sample. Equal amount of Nature NSC and Nature NIC were added in the six beakers, e.g. 0.5 ml of Nature NSC and 0.5 ml of Nature NIC were added. The dosage of coagulants increased with 0.5 ml for each sample. The pH was increased between in same way as mentioned in the sections above. The pH was not increased for sample 32 and 33, only 2.0 ml and 2.5 ml of the coagulants were added in each. Steps mentioned in *Figure 23* were followed step by step.

5.2.1.4 Process 4

In this section Solid Xanthan Gum (Duo –TEC$^+$ NS) given by MI-Swaco (Schlumberger) was dissolved in warm spring water and effects of coagulants were tested. Solid XG was dissolved to compare its properties with XG solution. The XG solution was prepared by dissolving 16 g of solid XG in 1000 ml warm water and mixed with jar tester overnight. Equal amount of Nature NSC and Nature NIC (0.5-3.0 ml) were added in the sample, see *Table 11***.** Step summarized in *Figure 23* were systematically followed until last step.

Testing of coagulants on 50 % and 75 % diluted XG solution with seawater

In this section XG $_{(aq)}$ was diluted with 50 and 75 % seawater. See process 3 for the dilution steps. Equal amount of Nature NSC and Nature NIC (0.5-3.0 ml) were added to both dilutions, see Table 12. Further, steps summarized in *Figure 23* were performed. The optimal dosage of coagulant was also determined. Comparing all previous results 2.0 ml of coagulants was confirmed as optimal dosage.

5.2.1.5 Process 5

Determination of optimum pH dosage on 75 % diluted XG solution

In this section optimal dosage of pH was determined on 75 % diluted XG solution. The solid XG dissolved in warm water was in this section diluted with 75 % seawater. The main objective of this section was to determine the pH, which gave optimal coagulation and separation of water from XG $_{(aq)}$. The pH of diluted XG $_{(aq)}$ was measured to be 7.6. The coagulants were added in constant amount 2.0 ml to the samples. Different dosages of 25 % w/v NaOH (1.0-3.5 ml) were tested on the diluted XG, see *Table 13***.** Further, all steps mentioned in *Figure* 23 23 were followed step by step.

Storing Samples

All samples collected from process 2 to 5 were stored in refrigerator at 4 ^{0} C. Later, amount of water in the solid phase was measured by drying these samples in the oven at 130 ^{0}C for 24 hours.

Drying Samples

Samples were pulled out from the plastic bottles and transferred to porcelain bowls and put in the oven for drying. Amount of water in the solid phase was measured by drying these samples in the oven at 130 ^{0}C for 24 hours. The porcelain bowls were weighed before samples were added and they were weighed before and after drying, this method was done as described in standards methods for the Examination of Water and Wastewater section 2540 B. (American Public Health, Water Environment, & American Water Works, 2005). Each samples were divided in two different porcelain bowls approximately same amount due to limited capacity in porcelain bowls, see Appendix.

5.2.1.6 Process 6

In this section coagulants were tested on GG $_{(aq).}$ In a 1 L beaker 16 g of GG was added and dissolved in 500 ml water. This solution was tested with 2.0 ml Nature NIC and NSC.

GG $_{(aq)}$ was in addition cross-linked with borax. Borax, 1.0 g, was dissolved in 60 ml water and added in GG solution while continuously mixing with jar tester.

6. Results and Discussion

In this chapter, results from the experiments are presented and discussed. First, the results are presented as amount of water removed from the sample after treatment and in the end the results are concluded and discussed. Further, diluted XG $_{(aq)}$ is compared with non-diluted XG $_{(aq)}$, which have been the main focus in the lab. Finally, XG $_{(aq)}$ is compared with XG$_{(s)}$ dissolved in warm water.

6.1 Testing on Xanthan Gum

In this section different combination of chemicals and parameters were tested on Xanthan Gum. As mentioned in section 5.1.1, different processes were performed to find optimal treatment method and in addition, optimal coagulant and pH dosage were found.

6.1.1 Testing of different Coagulants and Flocculants on Viscosity Pills

The results from this testing were solely based on visual observation. The different combinations of chemicals and physical treatments applied are summarized in *Table 4***.** Oboy (drinking chocolate mixed with water) was used as a Standard solution, of a suspended solid in water. The main goal of this step was to examine which chemical had an effect on XG. This step was executed for screening. *Figure 25* shows untreated XG dissolved in drill water (fresh water). The air bubbles in untreated XG $_{(aq)}$ may have occurred while mixing.

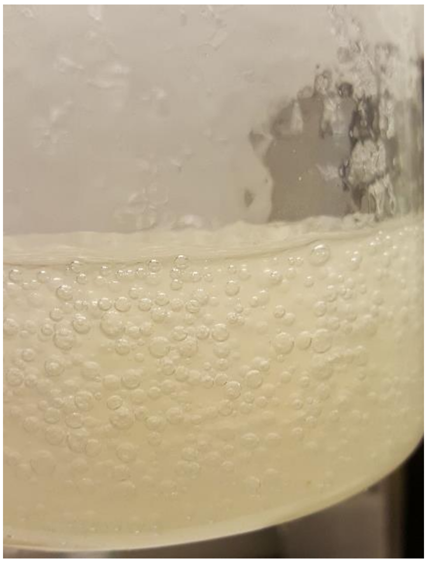

Figure 25: XG dissolved in drill water (fresh water) no treatment applied

6.1.1.1 Results

Addition of HCl in XG $_{(aq)}$ did not give any noticeable effect on the viscosity, neither increased amount of HCl had any effect. However, it was observed dissolution of the air bubbles with increased amount of HCl. *Figure 26* shows sample of XG $_{(aq)}$ with addition of 10 ml of HCl, where the bubbles dissolved from the bottom of the solution but foam was formed on the top. This is an example of a chemical that does not have any observable effect on the solutions viscosity. Reaction with the air bubbles when adding HCl may be the reason of the dissolution of air bubbles in XG $_{(aq)}$.

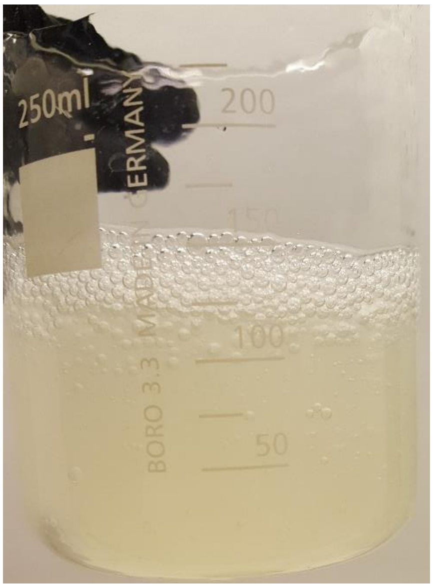

Figure 26: Addition of HCl in XG

Other chemicals tested in the screening and did not have any observable effect on the viscosity of XG solution were acetic acid and Poly-Fe-3. Acetic acid, pH^+ and Poly-Fe-3 neither had any observable effect on the viscosity or on the air bubbles. The solution was observed less viscous without any flocs formed when addition of Nature NPS and the air bubbles disappeared from the solution, as shown in *Figure 27*. Nature NPS containing acetic acid may be the reason for air bubbles dissolving in the XG solution, as seen in *Figure 27* air bubbles dissolved with increased acid, and the decrease in the viscosity may be due to NPS containing chitosan that is a polymer.

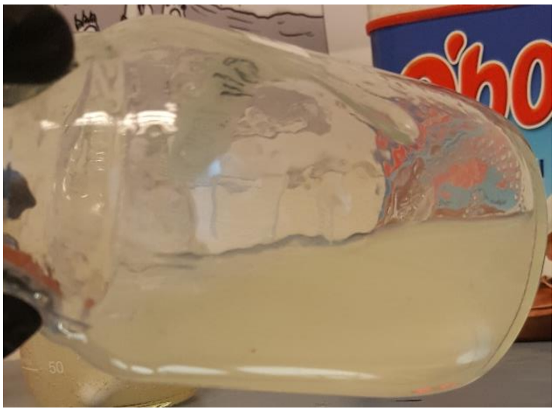

Figure 27: XG solution with addition of NPS

Figure 28: Addition of NIC in XG (aq)

Figure 28 shows XG $_{(aq)}$ with addition of Nature NIC which gave significantly good results. After addition of Nature NIC the viscous liquid bound to one big compact floc and a fraction of the water separated from the solution. Nature NIC was added to XG solution, and no excess viscous solution was observed with the floc. It was only observed viscous solution that did not mix with Nature NIC, but adhered to the upper side of the bottle which is marked in *Figure 28* with red circle. XG $_{(aq)}$ with Nature NIC was also centrifuged at 3500 rpm for 3 minutes. More separation of water was observed from the floc after the centrifuge.

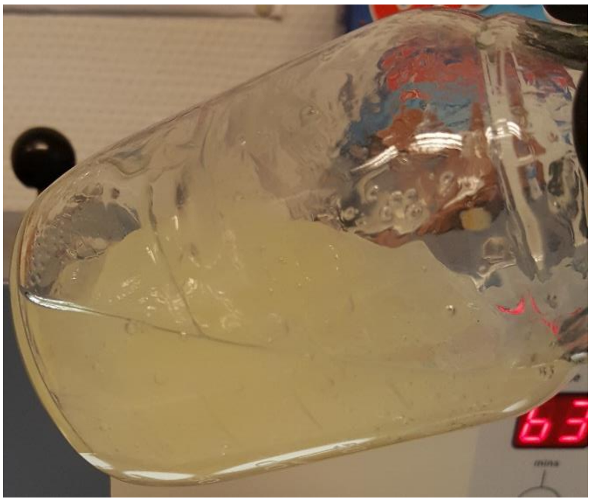

Figure 29: XG solution with addition of NSC

Nature NIC added to XG resulted in binding of XG and separation of water. Chemicals such as Zirconium Oxide and Nature NSC also gave binding of XG and a big floc was observed but no separation of water. The liquid observed in *Figure 29* is viscous XG which did not react with Nature NSC, and neither water separated from the floc was observed as it was observed when Nature NIC was added to XG $_{(aq)}$, same effect was observed when Zirconium was added to XG $_{(aq)}$.

6.1.1.2 Conclusion

The observations from this testing resulted in preference of Nature NSC and NIC for further testing and should be executed if other treatment method does not have an effect on the viscosity of XG solution. Addition of coagulants in XG solution decreased the viscosity but no effect on water separation. Nature NSC and NIC flocculated the XG $_{(aq)}$ in addition to some separation of water. XG hydrates rapidly water and have consistent water holding ability which may be the reason for less separation of water, which means the structure of XG have to be destroyed/break to separate water from XG $_{(aq)}$.

6.1.2 Testing of different Oxidizers, Organic Acid and Catalysts

In this section different oxidizers, organic acid and catalyst were tested. The aim of this testing was to break and dissolve the viscosity of XG $_{(aq)}$. The results from this section were completely based on visual observations. Different combinations of chemicals tested on XG $_{(aq)}$ are summarized in *Table 5*.

Table 5: Testing of different combinations of chemicals on XG $_{(aq)}$

Sample	**XG (ml)**	**$(NH_4)_2S_2O_8$ (g)**	**Na_2CO_3 (g)**	**$C_6H_8O_7$ (g)**	**$FeSO_4*7H_2O$ (g)**	**EDTA (g)**	**pH**
0	500	-	-	-	-	-	4,75
1	500	0,7	-	-	-	-	4,25
2	500	-	0,2	-	-	-	8,22
3	500	-	-	0,5	-	-	5,54
4	500	-	-	-	-	0,6	5,22
5	500	-	-	-	0,6	-	4,17
6	500	0,7	0,2	0,5	0,6	0,6	4,35

6.1.2.1 Results

In sample one to four different chemicals were added separately but no observable effect on the viscosity of XG $_{(aq)}$. However, it was observed foam on the top of some reactions, as shown in *Figure 30*. Sample zero was used to compare with the result of chemical treated samples. Some samples had clear reaction after addition of chemicals, such as formation of foam and dissolution of air bubbles, even though they had no effect on the viscosity of XG $_{(aq)}$, while other chemical did not have any remarkable effect on XG $_{(aq)}$.

Figure 30: Relative thick layer of foam on the top of the reaction after addition of $(NH_4)_2S_2O_8$ in XG (aq)

Best result was obtained when all chemicals, mentioned in *Table 4*, were added together in a beaker containing XG $_{(aq)}$. One big floc was formed in the center of the beaker and in the corners unreacted viscous XG was observed. In addition to the big floc small compact gel spheres were observed when adding $FeSO_4*7H_2O$, which is presented in *Figure 31***.** The reaction was allowed to react for 30 minutes with constant stirring. Centrifuge was applied to this solution. After three minutes in centrifuge at 3500 rpm, no effect on the viscous fluid was observed and neither separation of water from XG. No separation of water may be due to not enough mixing of chemicals, or to high viscosity of XG $_{(aq)}$ which did not allow the chemicals to reach and react with all XG $_{(aq)}$.

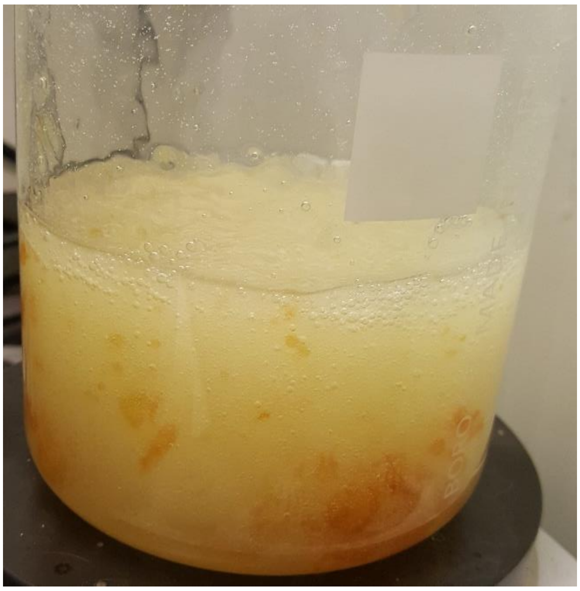

Figure 31: XG solution after addition of different chemicals

6.1.2.2 Conclusion

The observations from this testing resulted in the fact that long run use of all chemicals will not be economical efficient. In addition, there was no clear separation between the XG and water. It was also concluded not to test further these chemicals on XG $_{(aq)}$. Further testing with Soap and Monoethyleneglycol was done on the XG $_{(aq)}$.

6.1.3 Testing of Soap and Monoethyleneglycol

Soap and MEG were tested on XG $_{(aq)}$ in this section. The amount of these chemical added in XG $_{(aq)}$ are summed up in *Table 6*.

Table 6: Testing of Soap and Monoethyleneglycol on XG solution.

	Masava Tank Cleaner	Monoethyleneglycol
Xanthan Gum (ml)	500	500
Amount (g)	3	7,6
pH	12,13	4,20

6.1.3.1 Results

It was non-observable effect of these two substances on the viscosity of XG $_{(aq)}$. However, the XG solution with Masava Tank Cleaner had a layer of foam on the top, the same effect as shown in *Figure 30***,** and the pH increased from 4.75 for non-treated XG $_{(aq)}$ to 12.13 for treated XG $_{(aq)}$ with Masava Tank Cleaner. Meanwhile, non-remarkable observation was done after MEG was added to XG $_{(aq)}$. The pH dropped from 4.75 to 4.20 with addition of MEG. These chemicals are used to clean oily viscosity spills from deck, which may be laying in a thinner layer on the deck, but in the lab Soap and MEG were tested in a beaker with 500 ml XG $_{(aq)}$, which may not allow the chemicals to reach and react with XG $_{(aq)}$ properly.

6.1.3.2 Conclusion

It was concluded not to continue with MEG as treatment substance, since it did not have any remarkable effect on the XG solution. Addition of Masava Tank Cleaner gave foam on the top of the sample without having any effect on the viscosity of XG $_{(aq)}$. Nature NIC and Nature NSC coagulants tested first day in the lab gave good precipitation of XG $_{(aq)}$, therefore further testing with these two coagulants were decided to work with.

6.1.4 Testing of Coagulants on Viscosity Pills

In this section different combinations of coagulants were tested on XG $_{(aq)}$. In addition, the coagulants were tested separately and in combination with flocculant, as presented in *Table 7*.

6.1.4.1 Testing Different Combination and Dosage of Coagulants on Viscosity Pills

The pH of the blank sample (no addition of chemicals) was measured to be 4.67. Centrifuge applied on this sample did not have any effect on the viscosity of XG $_{(aq)}$, and neither water separated from XG $_{(aq)}$, but the small air bubbles observed before applying centrifuge dissolved. Total amount of water evaporated from this blank XG $_{(aq)}$ sample was measured to be 99.37 %.

Table 7: Result obtained from different combination of coagulants in XG $_{(aq)}$

	Sample	NSC (ml)	pH$^+$ (ml)	NIC (ml)	XG (ml)	Weight plastic bottles	Weight before centrifuge	Weight after centrifuge and water decanted	Amount of water separated from XG (g)
	0				500,00	24,40	205,70	205,70	0,00
1									
	1	0,50	-	0,50	500,00	23,80	206,30	198,40	7,90
	2	1,00	-	0,50	500,00	23,90	206,30	169,80	36,50
	3	1,50	-	0,50	500,00	23,90	206,20	138,70	67,50
	4	2,00	-	0,50	500,00	23,90	206,20	155,90	50,30
	5	2,50	-	0,50	500,00	23,80	206,40	173,50	32,90
									0,00
2	6	0,50	4,00	1,00	500,00	23,90	206,30	150,80	55,50
	7	0,50	2,00	1,50	500,00	23,90	206,40	156,50	49,90
	8	0,50	2,00	2,00	500,00	23,90	206,20	145,10	61,10
	9	0,50	2,00	2,50	500,00	23,80	206,30	135,10	71,20
	10	0,50	2,00	3,00	500,00	23,80	206,30	160,80	45,50
3	11	1,00	2,00	1,00	500,00	24,20	205,90	192,20	13,70
	12	1,50	2,00	1,50	500,00	24,20	206,00	190,90	15,10
	13	2,00	2,00	2,00	500,00	24,20	205,80	140,30	65,50
	14	2,50	2,00	2,50	500,00	24,20	205,90	122,30	83,60
	15	3,00	2,00	3,00	500,00	24,20	205,90	130,00	75,90

6.1.4.1.1 Results

Amount of water separated from XG $_{(aq)}$ after addition of coagulants and the centrifuge varied from 7.90 g to 67.50 g, where the dosage of Nature NIC was same for all 5 samples and NSC dosage varied from 0.5 to 2.5 ml. The best result was obtained when 1.5 ml of Nature NSC was added together with 0.5 Nature NIC, amount of water removed was measured to be 67.50 g. No chemicals were added for the change in pH. Formation of floc right after addition of Nature NIC and NSC was observed. The XG $_{(aq)}$ flocculated around the jar test paddles, which is very clear shown in *Figure 32*. It was also observed some small XG $_{(aq)}$ flocs in the beaker between the unreacted XG $_{(aq)}$.

Figure 32: XG flocculated around jar tester paddles

XG $_{(aq)}$ which reacted with Nature NIC and NSC were observed to create compact flocs **(***Figure 32***)**, and the part of the XG $_{(aq)}$ which did not react with the coagulants was highly viscous. It was not observed any separation of water from XG $_{(aq)}$ before centrifuge, but after the centrifuge some water separated from XG $_{(aq)}$ was observed, it is seen very clearly in the Figure 33 33. The treated XG $_{(aq)}$ sample released minimum amount of water, this was confirmed after drying the sample overnight in the oven at 130°C, the sample contained between 95.0 to 98.50 % water. The water separated from XG $_{(aq)}$ was decanted as much as possible before dried in oven. Reason for this high amount of water calculated in XG $_{(aq)}$ was due to the Xanthan gums water holding ability.

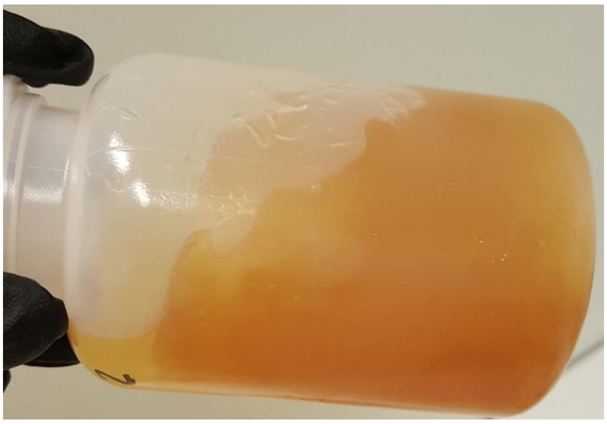

Figure 33: Sample of XG treated with coagulants and centrifuge

Immediately formation of floc after addition of coagulants may be due the polymeric property of XG. Biopolymers have no charge of their own and act on the principle of polymer bridging, and when addition of charged coagulants (such as Fe^{3+} and Al^{2+}) $XG_{(aq)}$ flocculated. Rapid flocculation of XG occurred after addition of coagulants as seen in *Figure 32***,** which indicate immediately change of XG properties, and decomposition of the structure.

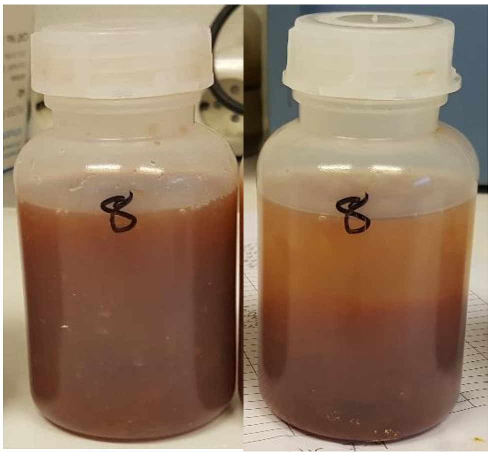

Figure 34: Treated XG (aq) with coagulants and NaOH before and after centrifuge

Different combination and amount of the coagulants and NaOH bonded the XG $_{(aq)}$ differently as shown in *Figure 35*.

Figure 35: Three different types of XG flocs

The sample shown to left had same amount of coagulants added, 2.0 ml and addition of 2.0 ml NaOH for increasing the pH. The sample in the middle, 2.0 ml of Nature NSC and NIC were added and 4.0 ml of NaOH was added for increasing the pH. In sample to right 0.5 ml of Nature NSC and NIC and no addition of NaOH. This indicates that water separates from the XG $_{(aq)}$ better with the increasing amount of NaOH. Shape and size of flocs changed with the amount coagulant and NaOH added.

6.1.4.1.2 Conclusion

The results from this test indicates that further testing with Nature NIC and NSC were required, with more focus on the addition of NaOH for increasing the pH. Since in this test XG was dissolved in fresh water before it is used for different purpose, it was decided to dilute the XG solution with slop in the first place and then see the effect of the coagulants applied.

6.1.4.2 Testing of individual coagulants with and without flocculant

In this section the coagulants were tested individually. In addition a flocculant was added to the coagulants. The combination and amount of coagulants and flocculant are summarized in *Table 8.*

Table 8: Results obtained from addition of individual coagulants and coagulants combined with flocculant

Sample	NSC (ml)	pH^+ (ml)	NIC (ml)	NPC (ml)	XG (ml)	Weight plastic bottles	Weight before centrifuge	Weight after centrifuge and water decanted	Amount of water separated from XG
16	2,00	2,00	-	-	500,00	24,20	206,10	193,60	12,50
17	2,50	1,00	-	-	500,00	24,20	206,00	149,00	57,00
18	-	1,50	2,00	-	500,00	24,20	206,10	144,60	61,50
19	-	2,00	2,50	-	500,00	24,20	206,10	139,50	66,60
20	2,00	1,00	-	1,00	500,00	24,20	206,10	201,10	5,00
21	-	1,50	1,00	1,00	500,00	24,50	205,80	161,00	44,80

6.1.4.2.1 Results

When only 2.0 ml of Nature NSC was added to XG $_{(aq)}$, 12.5 g of water was separated after centrifuge, the pH was increased with 2.0 ml of NaOH from 4.67 to 11.2. Coagulants are most efficient at pH 7-9. Separation of only 12.5 g of water may be due to destruction of the coagulant, because of this large amount of NaOH added. More water was removed from XG $_{(aq)}$ when 2.5 ml of Nature NSC was added to XG $_{(aq)}$ and the pH was increased with 1.0 ml of NaOH from 4.67 to 7.63. The pH was within the optimal range for a coagulant and good separation of water from XG $_{(aq)}$ was observed, also the measured water that separated from XG $_{(aq)}$ was 57.0 g, see *Table 8*.

The pH for sample with 2.0 ml Nature NIC increased to 11.32. It was observed darker colour of NIC with increased amount of NaOH, also more water separated from XG $_{(aq)}$ with increased addition of NaOH. Increased addition of NaOH moves the reaction to right and this cause increased amount of Iron (III) hydroxide formed, which may be the reason for the darker brown/red colour. Amount of water that separated from XG $_{(aq)}$ was measured to be 61.50 and 66.60 g, see *Table 8*.

Measured water separated from XG $_{(aq)}$ was 5.0 g, and can be seen in *Table 8*. Nature NIC, 2.0 ml was added to XG $_{(aq)}$ and addition of 1.5 ml of NaOH increased separation of water was observed. Addition of flocculant in XG $_{(aq)}$ containing Nature NIC and NaOH bound the already released water from XG $_{(aq)}$. Relative more water separated after addition of NaOH and NPC in NIC compared to addition of these in Nature NSC. It was measured 44.80 g of water separated after centrifuge, and is seen in *Table 8*.

6.1.4.2.2 Conclusion

Results from this test indicate that the use of flocculant such as NPC in further tests can be neglected. Addition of flocculant resulted in binding of the separated water to XG again. When coagulants were added to XG $_{(aq)}$ coagulation took place, this rapid coagulation may be due to XG acting as polymer in the solution. Addition of NaOH caused the release of water from XG $_{(aq),}$ some of this water got viscous and bound to XG when a flocculant, Nature NPS was added. The colour of Nature NIC increased with increased addition of NaOH, also increase in the separation of water. The release of water from XG $_{(aq)}$ can be explained with different reasons such as, destruction of the XG structure, reaction with pyruvic acid, acetyl group etc. which is out of the focus of this study. However, as a research it was needed to be mentioned. Nature NSC added to XG $_{(aq)}$ gave one big floc, but less water was separated when increased amount of NaOH was added and rather size of the floc increased. Compering the water separated from XG $_{(aq)}$ with the results obtained in *Table 8* it was concluded the need of further testing with both coagulants together than individually.

6.1.5 Testing of coagulants on diluted Xanthan Gum

XG $_{(aq)}$ in this section have been tested with coagulants after dilution. The dilution has varied between 50 and 75% of slop water and seawater. Both water types used for dilution contains high concentration of salts.

6.1.5.1 Testing of Coagulants on 50% Diluted Xanthan Gum Solution

The pH of this solution was measured to be 5.17. The viscosity of XG $_{(aq)}$ decreased after diluting with slop water. The small air bubbles in XG $_{(aq)}$ dissolved after diluting with slop water.

Table 9: Results obtained after diluting XG (aq) with 50 % slop water

Sample	NSC (ml)	pH^+ (ml)	NIC (ml)	XG (ml)	Weight plastic bottles	Weight before centrifuge	Weight after centrifuge and water decanted	Amount of water separated from XG
22	2,00	2,00	2,00	500,00	23,70	210,20	113,50	96,70
23	2,50	2,00	2,50	500,00	24,70	209,10	97,50	111,60
24	2,50	1,50	2,00	500,00	24,20	209,70	104,90	104,80
25	2,00	2,00	2,50	500,00	24,50	209,30	107,50	101,80

6.1.5.1.1 Results

Separation of water increased exceptionally after diluting XG $_{(aq)}$ with slop water. In the first two samples Nature NSC and NIC were added in same amount in XG $_{(aq)}$ and the pH was increased with 2.0 ml NaOH. Water separated from XG after centrifuge was measured to be 96.70 g and 111.60 g, see *Table 9.* It was observed a clear distinction between water and XG flocs. The huge difference of water separation from XG $_{(aq)}$ before and after centrifuge is clearly shown in *Figure 36***.**

Figure 36: Samples before and after centrifuge, treated with chemicals

The Sample to left in *Figure 36* show XG $_{(aq)}$ treated with chemicals before the centrifuge and the sample to right show after centrifuge. These two plastic bottle samples in this image show huge difference in water phase. Approximately half of the plastic bottle with sample contained water and the rest half was flocculated XG. It was observed huge difference between the flocs before and after centrifuge. The flocs before centrifuge were observed small, long and soft but after centrifuge they were observed hard and compact.

These samples were dried in oven and total amount of water evaporated was calculated to be approximately 96 %, see Appendix**.** Even though some samples dried in oven were hard and compact and others were smooth and jelly, both types contained a lot of water. This can be explained by XG having consistent water holding ability. The left over in porcelain bowl after heating was dried XG and samples diluted with slop and seawater contained in addition suspended particles.

6.1.5.1.2 Conclusion

Huge separation of water from XG $_{(aq)}$ was obtained after 50 % dilution compared to non-diluted XG $_{(aq)}$. The flocs observed were compact and small which may indicate that they contained less water compared to the soft and small flocs that may contain more water. This can be explained with different reasons such as, change of XG property when diluting with slop water due to high salt concentration and different components in slop water. Results from these tests indicated the need to increase the dilution of XG $_{(aq)}$ with 75 %, which is discussed in next section.

6.1.5.2 Testing of Coagulants on 75% Diluted Xanthan Gum

The pH was measured to be 4.70. The 75 % diluted XG $_{(aq)}$ with slop water was less viscous compared to the non-diluted and 50% diluted XG $_{(aq)}$.

Table 10: Results obtained after diluting XG $_{(aq)}$ with 75% slop water

Sample	NSC (ml)	pH^+ (ml)	NIC (ml)	XG (ml)	Weight plastic bottles	Weight before centrifuge	Weight after centrifuge and water decanted	Amount of water separated from XG
26	0,50	1,00	0,50	500,00	23,80	213,10	101,20	111,90
27	1,00	1,00	1,00	500,00	23,80	213,10	102,20	110,90
28	1,50	0,50	1,50	500,00	23,80	213,00	94,70	118,30
29	2,00	1,50	2,00	500,00	23,70	213,20	98,50	114,70
30	2,50	2,00	2,50	500,00	23,80	213,00	98,20	114,80
31	3,00	2,50	3,00	500,00	23,70	213,20	100,20	113,00
32	2,00	-	2,00	500,00	23,70	213,10	117,20	95,90
33	2,50	-	2,50	500,00	23,70	213,20	108,70	104,50
34	-	-	-	500,00	23,70	213,20	213,20	0,00

6.1.5.2.1 Results

Separation of water from XG $_{(aq)}$ was observed to be exceptionally high at low dosage of coagulants, see *Table 10*. Separation of water increased with increased NaOH dosage. For example in sample 29, 2.0 ml of the coagulants were added and the pH was increased with 1.50 ml NaOH and water separated from XG $_{(aq)}$ was calculated to be 114.70 g. Compared to sample 32, 2.0 ml of coagulants were added but the pH was not increased and the water separated from XG $_{(aq)}$ was calculated to be 95.90 g. This may indicate that the pH affects the degree of the separation of water from XG $_{(aq)}$. A more clear effect of NaOH on coagulant Nature NIC was observed. The increasing red colour of Nature NIC with Increased addition of NaOH was remarkable.

Comparing amount of water separated from XG $_{(aq)}$ in sample 26 with sample 30, the main difference the dosage of coagulant and NaOH added. The difference in amount of water separated from XG $_{(aq)}$ was minimal. This minimum difference in separation of water from XG $_{(aq)}$ may be due the way the samples are poured from the beaker to the plastic bottles. A spoon was used to pour the flocs and water to the plastic bottles. In some plastic bottles more water than flocs were transferred and vice versa.

6.1.5.2.2 Conclusion

Results from this testing confirmed that dilution decreased the viscosity property of xanthan gum and increased amount of separated from XG $_{(aq)}$. It was further decided to dissolve the powdered XG $_{(s)}$ delivered by MI Swaco for comparison with the XG solution, and to see if both had the same properties.

6.1.5.3 Testing of Coagulants on Solid Xanthan Gum dissolved in water

The main objective of this section was to see if the result obtained from the dissolved XG $_{(s)}$ gave same result as the XG $_{(aq)}$. XG $_{(s)}$ dissolved in water was observed very viscous and non-air bubbles were observed in the solution, compared to the XG $_{(aq)}$ given by MI-Swaco (Schlumberger). The pH was measured to be 5.1 which was higher than XG $_{(aq)}$ that was measured to be 4.65. Different dosage of coagulants were added in dissolved XG, the results obtained are presented in the *Table 11*.

Table 11: Results obtained after adding coagulants to solid XG dissolved in warm spring water

Sample	NSC (ml)	pH^+ (ml)	NIC (ml)	XG (m)	Weight plastic bottles	Weight before centrifuge	Weight after centrifuge and water decanted	Amount of water separated from XG
35	0,50	1,20	0,50	500,00	23,80	217,00	213,10	3,90
36	1,00	1,00	1,00	500,00	23,70	217,00	211,60	5,40
37	1,50	0,80	1,50	500,00	24,40	216,30	207,10	9,20
38	2,00	2,00	2,00	500,00	23,80	216,90	216,40	0,50
39	2,50	1,00	2,50	500,00	23,80	217,00	208,70	8,30
40	3,00	1,50	3,00	500,00	23,70	217,00	154,30	62,70

6.1.5.3.1 Results

Sample 35, 36 and 37 separated minimum water from the XG solution. Amount of water separated from these solutions were calculated to be 3.90, 5.40, 9.40 g. Sample 38 separated approximately no water and in this sample 2.0 ml of Nature NIC and NSC were added and the pH was increased to 7.71 with 1.0 ml NaOH. The floc was observed very strong, hard and big as shown in *Figure 37*. In sample 40 more water separated from the XG $_{(aq)}$ was observed. In this sample 3.0 ml of Nature NIC and NSC were added and the pH was increased to 8.0 with 3.0 ml NaOH, *Table11*. Water separated from sample 40 was calculated to be 63.70 g, *Table 11*, and big floc was observed.

Figure 37: A strong, hard and big floc of chemical treated XG solution.

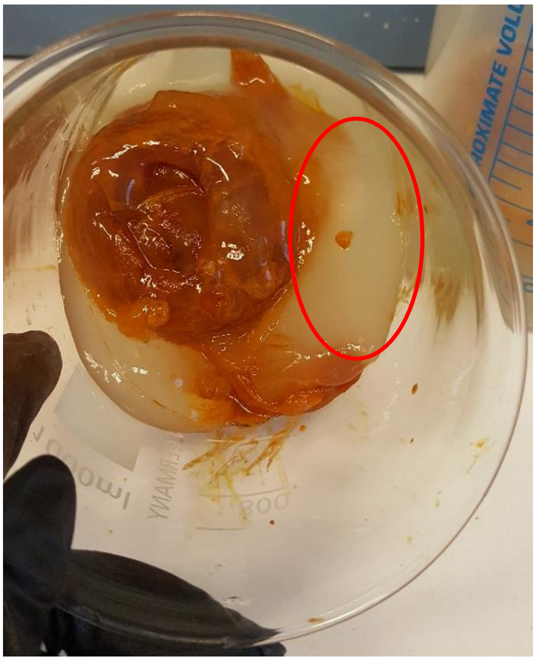

Figure 38: XG solution treated with coagulants. XG in the corners of the beaker did not reacted with the coagulants

Figure 38 shows a big hard floc of XG $_{(aq)}$. Unreacted XG $_{(aq)}$ was observed in the corner of the beaker, marked with red circle on the *Figure 38*. Compared to the floc in sample 38 shown in *Figure 37*, the floc in sample 40 *Figure 38* was observed very hard and compact.

6.1.5.3.2 Conclusion

The results obtained from the testing on XG $_{(s)}$ dissolved in warm water were same as the results obtained from testing on XG $_{(aq)}$. It was concluded, to dissolve the XG $_{(s)}$ dissolved in warm water for further testing. Due to same results were obtained for testing of coagulants on XG $_{(aq)}$ and XG $_{(s)}$ it was concluded to dilute the XG $_{(s)}$ with seawater.

6.1.5.4 Testing of coagulants on 50 % and 75 % diluted XG $_{(aq)}$ with seawater

The aim of this section was to see the effect of coagulants on XG $_{(aq)}$ diluted with seawater and in additional find the optimal dosage of the coagulants. The pH measured for 50 % diluted XG $_{(aq)}$ with seawater was 7.38 and the pH was measured to be 7.65 for 75 % diluted XG $_{(aq)}$. The results obtained are presented in *Table 12*.

Table 12: Results obtained from diluting XG (aq) with 50 and 75 % seawater

Dilution	Sample	NSC (ml)	pH$^+$ (ml)	NIC/JKL (ml)	NPS (ml)	XG (ml)	Weight plastic bottles	Weight before centrifuge	Weight after centrifuge and water decanted	Amount of water separated from XG
50 %	45	0,50	2,50	0,50	-	500	26,00	210,70	168,70	42,00
	46	1,00	1,50	1,00	-	500	24,70	212,00	177,70	34,30
	47	1,50	1,50	1,50	-	500	25,90	210,80	127,30	83,50
	48	2,00	1,50	2,00	-	500	26,70	210,00	98,00	112,00
	49	2,50	1,50	2,50	-	600	25,90	211,00	132,10	78,90
	50	3,00	2,00	3,00	-	600	24,80	211,90	120,50	91,40
75 %	53	0,50	0,50	0,50	-	500	24,20	207,40	191,90	15,50
	54	1,00	1,00	1,00	-	500	24,60	206,80	125,90	80,90
	55	1,50	1,50	1,50	-	500	24,40	207,40	144,80	62,60
	56	2,00	2,00	2,00	-	500	24,30	207,10	115,80	91,30
	57	2,50	2,50	2,50	-	500	24,70	206,60	118,60	88,00
	58	3,00	3,00	3,00	-	500	24,40	207,00	119,60	87,40

6.1.5.4.1 Results

The XG $_{(aq)}$ was observed less viscous after the dilution. For both dilutions the optimal dosage of coagulants were measured to be 2.0 ml, as seen in *Table 12* sample 48 and 56.

Highest amount of water separated from XG $_{(aq)}$ was measured to be 112.0 ml for sample 48 and 91.3 ml for sample 56. In sample 48 the pH was increased to 8.92 with 1.5 ml NaOH, and the pH in sample 56 was measured to be 7.03 increased with 2.0 ml NaOH. This number of NaOH indicates that the pH increased easily by adding less amount of NaOH.

Largest amount of water separated from XG $_{(aq)}$ was observed in beakers were the NaOH was added slowly in order to let the chemicals and XG react. By adding the NaOH slowly to the beakers the flocs appeared smaller and looser as seen in *Figure 39*. The separation of water from XG $_{(aq)}$ increased with increased mixing. Figure below shows two different types of flocs one (to left) after rapid addition of NaOH and mixing for some few minutes, and the other one (to right) after slowly addition of NaOH and mixing for 3-4 minutes. The difference in size and hardness appears clearly in *Figure 39*. The mixing rate for both samples shown in the figure below was preserved constant.

Figure 39: Samples showing different type of flocs

Flocs presented to left in figure above were observed hard and compact. The light colour of the sample to left was due to less amount, 1.0 ml of Nature NIC, added and in the sample to right 2.0 ml Nature NIC was added.

6.1.5.4.2 Conclusion

The main objective of this section was to determine optimal dosage of the coagulants. Optimal dosage was measured to be 4 ml of coagulants per liter of 0.7 wt. % XG $_{(aq)}$. Results from these tests indicate the necessity to determine optimal dosage of NaOH for optimal coagulation and this is discussed in next section.

6.1.5.5 Determination of optimum pH dosage on 75 % diluted XG $_{(aq)}$ with seawater

Results achieved from testing of coagulants and NaOH on 75 % diluted XG $_{(s)}$ (dissolved in warm water) with seawater are shown in *Table 13.* After dilution the XG $_{(aq)}$ was observed less viscous compared to non-diluted XG $_{(aq)}$.

Table 13: Results obtained from testing of coagulants on 75 % diluted XG $_{(s)}$ dissolved in warm water

Sample	NSC (ml)	pH⁺ (ml)	NIC/JKL (ml)	XG (ml)	Weight plastic bottles	Weight before centrifuge	Weight after centrifuge and water decanted	Amount of water separated from XG
59	2,00	1,00	2,00	500	26,10	220,90	104,50	116,40
60	2,00	1,50	2,00	500	26,00	220,90	113,10	107,80
61	2,00	2,00	2,00	500	25,40	221,40	120,20	101,20
62	2,00	2,50	2,00	500	25,00	221,80	125,30	96,50
63	2,00	3,00	2,00	500	25,90	221,30	116,20	105,10
64	2,00	3,50	2,00	500	24,40	222,70	147,60	75,10

6.1.5.5.1 Results

From previous observation and calculation optimum dosage of coagulants were measured to be 2.0 ml in 500 ml of XG $_{(aq)}$. The pH of XG $_{(aq)}$ increased after dilution with 75 % seawater. A decrease in the pH was measured after addition of Nature NIC in XG $_{(aq)}$. This may be due to the sulfuric acid in JKL (Nature NIC was not available, JKL was used instead of Nature NIC).

The optimum pH dosage was adjusted between 1.0-3.50 ml. Optimum solution of pH was measured to be 1.0 ml combined with 2.0 ml of the coagulants, see *Table 13*. The pH was measured to be 8.0. In sample 64 the pH was measured to be 11.3, which was not in the range of the pH of coagulants (7.0-9.0). This may be the reason why only 75.10 ml of water separated from XG $_{(aq)}$.

The water separated from XG $_{(aq)}$ immediately after addition of NaOH. The small and loose flocs were remarkable. Something that was very clear during optimization of dosing was that less NaOH was needed when separating water from XG $_{(aq)}$. This may be due to higher of the pH after dilution of XG $_{(aq)}$ with seawater compared with slop water. Diluting the XG $_{(aq)}$ with slop water decreased the pH, and therefore increased addition of NaOH was needed. In addition the composition of slop water varies widely and containing different composition of chemicals, which may work as buffer in the solution. The composition of slop water was unknown.

6.1.5.5.2 Conclusion

The aim of this section was to determine optimal dosage of NaOH for optimal coagulation. The results obtained from this section show that 1.0 ml was the optimal dosage of NaOH for the increase of the pH combined with 2.0 ml coagulants.

6.1.6 Comparison

The main objective of the experiments was to determine the best solution to reduce the viscosity of XG $_{(aq)}$. Different treatment methods and chemicals, such as oxidation, coagulants, heating and gravity force were applied to figure out which method gave the highest effect on the viscosity of XG $_{(aq)}$. The different treatment methods applied and the effect observed are summarized in the *Table 14*. Satisfying results are marked in colour.

6.2 Testing on Guar Gum

The main objective on this section was to see the effect of coagulants on GG and if the results obtained were the same as for XG. The pH of GG $_{(aq)}$ was measured to be 6.17. GG $_{(aq)}$ was in addition cross-linked with Borax.

6.2.1 Results

Addition of coagulants in GG $_{(aq)}$ gave minimal precipitation of GG $_{(aq)}$. GG $_{(aq)}$ viscosity increased exceptionally with addition of Borax. Cross-linked GG $_{(aq)}$ with borax flocculated to one big hydrogel. No excess water or viscous solution was observed in the corners of the beaker, see *Figure 40*.

Figure 40: GG cross-linked with Borax

6.2.2 Conclusion

The main objective of this section was to compare if GG had same effect as XG. The result from this testing was concluded with that GG $_{(aq)}$ with addition of coagulants did not have same properties as XG. GG $_{(aq)}$ viscosity increased exceptionally by cross-linking with Borax. A big hydrogel was formed in a single step, see *Figure 40*. After cross-linking GG $_{(aq)}$ with Borax coagulants were not added, due to the surface layer formed by the cross-linking.

Table 14: Comparison of the effect of treatment methods applied on Xanthan Gum solution

Xanthan Gum Solution									
Observations									
Treatment		**Air bubbles**	**Foam on the top layer**	**Viscosity**	**Flocs**	**Flocs Size and Form**	**Addition of NaOH**	**Centrifuge**	**Separated Water**
Temperature 100 °C	**No addition of chemicals**	Some dissolved	Not observed	No change	Not formed	-	-	Not applied	No separation
Centrifuge	**No addition of chemicals**	Dissolved	Not observed	No change	Not formed	-	-	No effect	No separation
Nature NSC		Dissolved	Not observed	Decreased	Formed	One big and hard	-	Not applied	No separation
Nature NIC		Dissolved	Not observed	Decreased	Formed	One big and hard	-	Not applied	Small amount
Nature NPS		Slightly dissolved	Some observed	Decreased slightly	Not formed	-	-	Not applied	No separation
HCl		Dissolved	Large layer	No change	Not formed	-	-	Not applied	No separation
Acetid Acid		No change	Not observed	No change	Not formed	-	-	Not applied	No separation
Zirconium Oxyde		Dissolved	Not observed	Decreased	Formed	One Big and hard	-	Not applied	No separation
Poly-Fe-3		No change	Not observed	No change	Not formed	-	-	Not applied	No separation
NaOH		No change	Not observed	No change	Not formed	-	-	Not applied	No separation

Oxidizers		Slightly dissolved	Large layer	No change	Not formed	-	-	Not applied	No separation
Organic Acid		Slightly dissolved	Large layer	No change	Not formed	-	-	Not applied	No separation
Catalyst		No change	Not observed	No change	Not formed	-	-	Not applied	No separation
Oxidizers **Organic Acid** **Catalyst**	**Centrifuge**	Slightly dissolved	Small layer	Decreased slightly	Formed	One big and Few small jelly sphere formed all over the beaker	-	No effect	No separation before or after centrifuge
Soap		Slightly dissolved	Small layer	No change	Not formed	-	-	Not applied	No separation
Monoethyleneglycol		No change	Not observed	No change	Not formed	-	-	Not applied	No separation
Nature NSC **Nature NIC** **NaOH**	**50% and 75 % dilution with slop water**	Dissolved	Not observed	Decreased exceptionally	Formed	Many small, loose, thin, smooth and long	Varied from 0.5 ml to 4.0 ml	Flocs compact and hard	Exceptionally separation of water
Nature NSC **Nature NIC** **NaOH**	**50 % and 75 % dilution with seawater**	Dissolved	Not observed	Decreased exceptionally	Formed	Many small, loose, thin, smooth and long	Varied from 0.5 ml to 3.0 ml	Flocs compact and hard	A lot of water separated

Different combination of Nature NIC and NSC were tested combined with adjustment in the pH. A better separation of water from XG $_{(aq)}$ was obtained after diluting the XG $_{(aq)}$ with 50 and 75 % slop and seawater. Before diluting the XG $_{(aq)}$ the flocs were observed very big and compact and less separation of water was observed. After dilution smaller and loose flocs were observed and a lot more water separated from the XG $_{(aq)}$, appears clearly in *Figure 41*. In addition, the separation of water increased with centrifuge, appears clearly in *Figure 36*. The sample to left show treated XG $_{(aq)}$ before centrifuge, minimal water separated is seen in *Figure 41*. A lot more water is observed in the sample to right after centrifuge. Huge effect of centrifuge was observed on the diluted XG $_{(aq)}$ compared to the non-diluted XG $_{(aq)}$ see *Figure 41*.

Figure 41: Diluted and non-diluted XG solution after centrifuge. From left to right non-diluted and diluted sample.

The non-diluted XG sample shown to left on *Figure 41* above was chemically treated before centrifuge was applied, minimum separated water observed from the XG $_{(aq)}$, appears clearly on *Figure 41***.** However, the diluted XG sample to right was also chemically treated before centrifuge was applied, a lot more of water was observed separated from the XG $_{(aq)}$ was observed**.** The big floc in the non-diluted sample hold a lot more water and the floc was observed more compact but smooth before and after centrifuge. In the diluted sample the flocs were observed smaller, thinner and looser before centrifuge. After centrifuge some small and long flocs were observed in the water phase but the flocs of XG were compact and relative hard and increased amount of water was remarkable, which appears clearly in the *Figure 42***.**

Figure 42: XG samples before dried in oven. From left to right: Diluted sample, non-diluted sample

The chemical and centrifuge treated XG $_{(aq)}$ samples were stored for 2 weeks before drying. The samples were poured in porcelain bowls from plastic bottles before they were dried. Sample to left *Figure 42* was diluted with slop. It was approximately no separated water left before the sample was dried see in *Figure 42*. Meanwhile, sample to right had a lot more separated water, and this sample was not diluted. Before the samples were stored all separated water was decanted, but some more water separated from XG was observed before drying. This may be due to the loose and smooth flocs released water over time.

Amount of water contained in samples were measured to be over 96 %, see Appendix. Approximately the same amount of samples were poured into the plastic bottles from the beaker before centrifuge, but amount of water decanted from the plastic bottles after centrifuge varied widely depending on how much water separated from XG flocs. Drying the samples in the oven confirmed that the XG flocs contained a lot of water, even though some of the flocs were compact and hard, and no additional separated water was observed right before drying, as appearing in *Figure 42* sample to left.

Some flocs were hard and big which were difficult to pour in small plastic bottles, other were small and smooth and they were easily poured to the plastic bottles. This led to the fact that, in some bottles, more water was transferred while in others more flocs, which may have affected the separation of water during the centrifuge.

Further research on new treatment methods and combination of different treatment methods should be executed. Such new treatment technologies applied on high viscous slop water may be microwaves d UV-light combined with chemicals. Although there is research on UV lamps used for Wastewater disinfection, which can be further researched for the application on high viscous slop water (Peng, Qiu, & Gehr, 2005). Panday and Ramontja, 2016, study on microwave-assisted synthesis of xanthan gum grafted polyaniline for chemical sensor (Pandey & Ramontja, 2016). This study show how viscosity of XG can be increased rapid, but microwave can be researched on non-treated and treated high viscous slop.

7. Conclusions

The aim of this thesis was to contribute to the understanding of the problem of complex slop water and high viscous slop, generated from drilling operations, as well as test and evaluate optimal treatment parameters. The focus was especially on Xanthan Gum, which is increasingly used in the offshore operations due to its varying rheological properties. A range of slop water treatment technology options used offshore and onshore was figured out.

Different treatment methods, such as chemicals, centrifuge and temperature, were applied on Xanthan Gum solutions to find best treatment method.

Chemical precipitation with coagulants and centrifuge were concluded as potential solution to the problem of high viscosity of slop water. Since this method is highly effective and easily available.

Different combination of Nature NIC and NSC with increased pH have flocculated the $XG_{(aq)}$. However, a big floc was obtained and no separation of water. $XG_{(aq)}$ was diluted with slop and seawater and smaller flocs were achieved, in addition to large amount of water separated. Dilution of $XG_{(aq)}$ with 75 % sea water was chosen as potential solution to the high viscosity of slop water.

Different dosages of Nature NIC and NSC were tested on diluted $XG_{(aq)}$ with 75 % seawater for determination of optimizing dosage. Amount of water separated from $XG_{(aq)}$ was 116.40 g with 2.0 ml/L of NaOH combined with 4.0 ml/L coagulants. It gave thinner layer of XG after centrifugation and increased separation of water.

Amount of water separated from $XG_{(aq)}$ increased a bit with increased NaOH addition, and increased remarkably with dilution of $XG_{(aq)}$. Addition of NaOH decreased after diluting $XG_{(aq)}$ with 75% seawater and centrifugation.

Separated water from $XG_{(aq)}$ flocculated with XG when a flocculant was added. XG worked as a flocculant after addition of coagulants due the polymeric property, and therefore additional flocculant was not needed.

Based on research done in this thesis and observations in lab, it is concluded that XG viscosity decreased by coagulation and separates water by diluting with seawater and addition of NaOH.

Change in temperature and addition of other chemicals played minor role on the decrease of the viscosity of XG. With addition of these chemicals only dissolution of air bubbles were observed.

After decanting water from beaker with XG $_{(aq)}$, samples were dried in oven, which showed 96% water in Xanthan Gum floc. This indicated that XG floc still contained water.

Results obtained from these research may be applied in Nature Oil and Gas AS treatment technologies when facing high viscosity of slop water caused by XG. These results are also applicable in other technologies and areas where high viscosity caused by XG is a problem.

8. Further Research

Areas relevant to a better understanding of high viscous slop water not covered in this thesis but should be the focus of future study are:

- A better understanding and modeling of the composition of slop water at different drilling operation times. This would increase the knowledge on how to treat slop water with different composition.

- More detailed research on composition of XG and its functionality under different conditions.

- A better description and understanding on how different chemicals applied to XG affect the structure of XG with use of Infrared (IR) spectroscopy and Nuclear Magnetic Resonance (NMR).

- Improve the uncertainty associated with water separated from XG. Determine a treatment method to increase the separation of water from XG.

- Research on new methods and combination of methods can be done in future with special attention to such methods as UV-light and microwave technology.

- Further work should be on sludge wastes generated from treated high viscous slop.

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Appendix

Table A 1: Results obtained from drying the samples

Sample	Weight porcelain bowl	Weight sample	Weight porcelain bowl after drying	Weight of dried solids	% removal
0_1	116,62	95,61	117,23	0,61	99,36
0_2	155,82	97,89	156,42	0,60	99,39
1_2	143,29	48,80	144,80	1,51	96,91
2_1	158,82	25,1	159,55	0,73	97,09
2_2	166,35	48,4	167,33	0,98	97,98
3_1	150,59	21,40	151,33	0,74	96,54
3_2	164,06	36,20	164,91	0,85	97,65
4_1	166,08	31,8	167,66	1,58	95,03
4_2	149,57	36,8	150,51	0,94	97,45
5_1	151,88	37,10	152,66	0,78	97,90
5_2	116,61	41,40	117,76	1,15	97,22
6_1	155,81	37,50	157,07	1,26	96,64
6_2	127,29	42,10	128,95	1,66	96,06
7_1	164,80	62,50	165,66	0,86	98,62
7_2	154,35	91,20	155,91	1,56	98,29
8_1	160,24	55,56	161,17	0,93	98,33
8_2	155,14	87,08	156,81	1,67	98,08
9_1	180,16	65,35	181,40	1,24	98,10
9_2	166,65	66,67	168,14	1,49	97,77
10_1	163,66	80,66	165,06	1,40	98,26
10_2	160,76	78,38	162,39	1,63	97,92
11_1	169,18	90,42	170,28	1,10	98,78
11_2	161,75	93,44	163,01	1,26	98,65
12_1	162,50	97,04	163,66	1,16	98,80
12_2	153,94	91,35	155,55	1,61	98,24
13_1	161,54	65,84	162,74	1,20	98,18
13_2	177,98	73,68	179,55	1,57	97,87
14_1	157,64	57,00	159,08	1,44	97,47
14_2	162,57	62,83	164,16	1,59	97,47
15_1	110,02	70,20	111,57	1,55	97,79
15_2	101,48	58,35	103,05	1,57	97,31
16_1	90,91	81,32	100,84	9,93	87,79

16_2	96,62	106,51	98,20	1,58	98,52
17_1	83,52	78,73	84,47	0,95	98,79
17_2	109,80	66,32	110,69	0,89	98,66
18_1	100,22	82,34	101,52	1,30	98,42
18_2	106,56	60,04	107,68	1,12	98,13
19_1	161,63	74,91	163,05	1,42	98,10
19_2	143,29	63,61	144,53	1,24	98,05
20_1	158,82	101,82	159,80	0,98	99,04
20_2	166,35	94,45	167,30	0,95	98,99
21_1	150,59	74,50	151,64	1,05	98,59
21_2	164,06	85,15	165,29	1,23	98,56
22_1	102,08	45,30	103,52	1,44	96,82
22_2	149,57	66,35	151,68	2,11	96,82
23_1	151,88	43,45	153,49	1,61	96,29
23_2	116,61	51,61	118,50	1,89	96,34
24_1	155,81	41,44	157,12	1,31	96,84
24_2	127,29	62,65	129,29	2,00	96,81
25_1	164,86	41,50	166,24	1,38	96,67
25_2	154,35	63,88	156,53	2,18	96,59
26_1	160,22	43,77	161,61	1,39	96,82
26_2	155,15	56,01	156,83	1,68	97,00
27_1	180,13	59,45	182,01	1,88	96,84
27_2	166,63	41,72	167,90	1,27	96,96
28_1	163,66	47,92	165,15	1,49	96,89
28_2	160,80	45,70	162,23	1,43	96,87
29_1	169,48	37,66	170,50	1,02	97,29
29_2	161,75	59,81	163,93	2,18	96,36
30_1	162,38	44,80	164,20	1,82	95,94
30_2	153,92	52,34	156,03	2,11	95,97
31_1	161,54	39,80	163,03	1,49	96,26
31_2	177,96	58,92	180,32	2,36	95,99
32_1	157,65	61,35	159,53	1,88	96,94
32_2	162,49	54,93	164,15	1,66	96,98
33_1	110,02	50,71	111,57	1,55	96,94
33_2	101,48	57,09	103,27	1,79	96,86
34_1	99,88	83,38	101,53	1,65	98,02
34_2	96,62	122,38	99,04	2,42	98,02
35_1	83,53	101,59	86,00	2,47	97,57
35_2	109,80	108,97	112,35	2,55	97,66
36_1	100,22	101,33	102,68	2,46	97,57
36_2	106,57	104,95	109,50	2,93	97,21
37_1	161,63	88,79	163,63	2,00	97,75

37_2	143,27	112,27	146,00	2,73	97,57
38_1	158,81	98,29	161,10	2,29	97,67
38_2	166,35	112,23	169,52	3,17	97,18
39_1	150,60	84,77	152,54	1,94	97,71
39_2	164,07	118,82	167,35	3,28	97,24
40_1	149,58	74,02	152,03	2,45	96,69
40_2	151,89	79,34	154,98	3,09	96,11
41_1	86,81	76,28	88,55	1,74	97,72
41_2	103,05	108,85	106,37	3,32	96,95
42_1	90,28	77,66	92,04	1,76	97,73
42_2	47,71	99,53	50,19	2,48	97,51
43_1	107,06	70,76	107,99	0,93	98,69
43_2	101,90	122,18	104,47	2,57	97,90
44_1	105,60	90,56	107,59	1,99	97,80
44_2	95,04	103,15	97,56	2,52	97,56
45_1	92,04	76,17	94,16	2,12	97,22
45_2	93,96	91,75	96,76	2,80	96,95
46_1	102,78	85,23	104,92	2,14	97,49
46_2	116,63	91,08	119,26	2,63	97,11
47_1	164,86	61,77	166,78	1,92	96,89
47_2	154,35	65,46	156,40	2,05	96,87
48_1	160,23	45,87	161,78	1,55	96,62
48_2	155,41	53,60	157,06	1,65	96,92
49_1	180,33	65,57	182,22	1,89	97,12
49_2	166,66	66,57	168,89	2,23	96,65
50_1	163,58	67,09	165,95	2,37	96,47
50_2	160,75	53,15	162,69	1,94	96,35
51_1	169,18	105,04	173,48	4,30	95,91
51_2	161,75	106,36	166,12	4,37	95,89
52_1	102,09	13,60	103,08	0,99	92,72
52_2	-	-	-	-	-
53_1	161,52	84,02	163,49	1,97	97,66
53_2	178,01	104,36	180,80	2,79	97,33
54_1	157,65	57,04	159,36	1,71	97,00
54_2	162,50	68,47	164,70	2,20	96,79
55_1	110,15	57,81	111,59	1,44	97,51
55_2	101,70	83,77	104,02	2,32	97,23
56_1	99,89	57,48	102,08	2,19	96,19
56_2	96,62	57,15	98,88	2,26	96,05
57_1	83,51	54,02	85,37	1,86	96,56
57_2	109,79	62,90	112,20	2,41	96,17
58_1	100,22	56,21	102,06	1,84	96,73

58_2	93,19	62,35	95,46	2,27	96,36
59_1	160,25	53,52	162,11	1,86	96,52
59_2	155,15	51,13	156,93	1,78	96,52
60_1	180,14	46,93	181,83	1,69	96,40
60_2	166,65	66,65	169,07	2,42	96,37
61_1	163,65	66,85	165,96	2,31	96,54
61_2	160,76	54,03	162,70	1,94	96,41
62_1	169,17	55,29	171,11	1,94	96,49
62_2	161,76	69,10	164,27	2,51	96,37
63_1	161,54	56,08	163,69	2,15	96,17
63_2	177,96	59,39	180,15	2,19	96,31
64_1	157,66	68,17	159,83	2,17	96,82
64_2	162,51	78,18	165,18	2,67	96,58