

Master Thesis

Comparison between Four Chamber Flotation Unit and Dissolved Flotation Unit for the Treatment of Back-Produced Polymer Water

Experiments and Operational Challenges



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Kurzfassung

Produziertes Wasser ist der größte Abfallstrom der bei der Öl- und Gasproduktion anfällt und die heutigen konventionellen Wasseraufbereitungstechnologien sind mit den in der Ölindustrie verfügbaren Technologien der Gewinnung von Öl und Gas aus unkonventionellen Lagerstätten nicht länger problematisch.

Eine Möglichkeit aus diesen unkonventionellen Lagerstätten zu produzieren, ist die Nutzung der tertiären Rückgewinnung, die als "Enhanced Oil Recovery" bezeichnet wird. Diese Arbeit konzentriert sich auf eine chemische Rückgewinnungsmethode im Speziellen auf das Polymerfluten.

Die OMV Exploration & Produktion GmbH hat ein Polymerflutprojekt, bei dem Polymerlösungen für mehrere Jahre in zwei Bohrungen im Ölfeld Matzen injiziert werden. Dem Injektionswasser wird ein wasserlösliches Polymer zugesetzt, um die Viskosität zu erhöhen. Durch die Erhöhung der Viskosität wird der volumetrische Entölungsgrad der Erdöllagerstätte gesteigert. Nach einiger Zeit der kontinuierlichen Injektion enthält das erzeugte Formationswasser rückproduziertes Polymer.

Es ist wichtig das rückproduzierte Polymer zu behandeln und aus dem Wasser zu entfernen, um den Injektionsdruck so niedrig wie möglich zu halten. Tests haben gezeigt, dass das Polymer die Effizienz der Flotationseinheiten in der Wasseraufbereitungsanlage beeinflusst, die nur für die Aufbereitung von konventionellem Wasser derzeit verwendet wird.

In dieser Masterarbeit wurde eine sogenannte Mikroblasen-Flotations-Pilotanlage getestet. Die Anlage ist eine Kombination von gelöster Gasflotation als auch induktiver Gasflotation zur Behandlung von polymerhaltigen Wässern. Diese Anlage wird mit der gelösten Flotationseinheit, die in der OMV Wasseraufbereitungsanlage verwendet wird, verglichen.

Diese Pilotanlage kann bis zu einer Fließgeschwindigkeit von 33 m³/h betrieben werden. Die Tests wurden erstens bei einer niedrigeren Fließgeschwindigkeit von 1,2 m³/h durchgeführt, wo die Fließgeschwindigkeit die Sonde ist, die das Polymer zurückproduziert hat. Zweitens wurden auch Versuche mit höheren Fließgeschwindigkeit von 13 m³/h durchgeführt.

Verschiedene Mengen an Chemikalien wurden während dieser Versuchszeit getestet. Das Formationswasser, das in die Pilotanlage gelangt stammt aus neun verschiedenen Produktionssonden eines unterirdischen Ölreservoirs (8. Tortonian Horizont), welches sich im Ölfeld Matzen befindet.

Verschiedene Versuche wurden mit der Pilotanlage getestet: konventionelles Formationswasser & polymerhaltiges Wasser mit unterschiedlichen Flussraten sowie mit und ohne Chemikalien. Die Ergebnisse jedes Versuches wurden als Öl-in-Wassergehalt, Feststoffkonzentration, Polymerkonzentration und Trübung des Einlasses und aller Kammern der Flotationseinheit gezeigt.

Abstract

Produced water is the largest waste stream acquired in the recovery of oil and gas. Water treatment technologies have achieved significant progress in the treatment of conventional water in the recent years, hence reducing the burden of dealing with this byproduct and therefore removing the restriction of the production from unconventional resources.

In order to efficiently develop these unconventional reservoirs, the tertiary recovery method (Enhanced Oil Recovery) can be used. This thesis focuses on a chemical flooding method entitled polymer flooding.

The OMV Exploration & Production GmbH has a polymer flooding project, where a polymer solution is injected into two wells in the Matzen oilfield for several years. The water-soluble polymer is added to the injection water to increase the viscosity which leads to improving the volumetric sweep efficiency aspect of the oil recovery. After some time of continuous injection, the produced oilfield water contains back-produced polymer.

However, it is important to treat and remove the back-produced polymer from the water so that the injection pressure stays as low as possible. Tests have shown that the back-produced polymer affects the efficiency of the dissolved flotation units in the OMV water treatment plant, which is only designed to treat conventional water.

In this thesis, a pilot plant called micro-bubble flotation was tested. This pilot plant has a multi-chamber flotation unit with the combination of both dissolved gas flotation and induced gas flotation. This unit was tested for the treatment of back-produced polymer and compared with the dissolved flotation unit in the OMV water treatment plant.

The unit can operate up to a flow rate of 33 m³/h. The tests were done with a low flow rate of 1.2 m³/h, which is the rate of the well containing back-produced polymer. Additionally, a higher flow rate of 13 m³/h was done also, which reflects the conventional wells flow rate.

Different amounts of chemicals were analyzed during the time of this pilot plant trial. The pilot plant is supplied with formation water coming from nine different wells of an underground oil reservoir in the 8. TH Tortonian reservoir, which is located in the Matzen oilfield.

Different trials were tested within the pilot plant: conventional water with different flow rates with and without chemicals, back-produced polymer water with and without chemicals. Samples were taken on a daily basis from all sample points (inlet to outlet) and the water phase was analyzed according to different parameters such as oil-in-water concentration, total suspended solids, polymer content and turbidity.

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1 Introduction

Oil recovery is basically separated into three stages of recovery as shown in Figure 1-1: primary, secondary and tertiary. Primary recovery depends mainly on the natural energy available in the reservoir. This energy comes from various forces like expanding force of natural gas, gravitational force and buoyancy force of encroaching water. Secondary recovery can be done by using different techniques like artificial methods, water flooding and immiscible gas injection. Tertiary recovery, also known as enhanced oil recovery (EOR), is divided into three categories which are chemical flooding, gas injection and thermal processes. [1, p. 693]

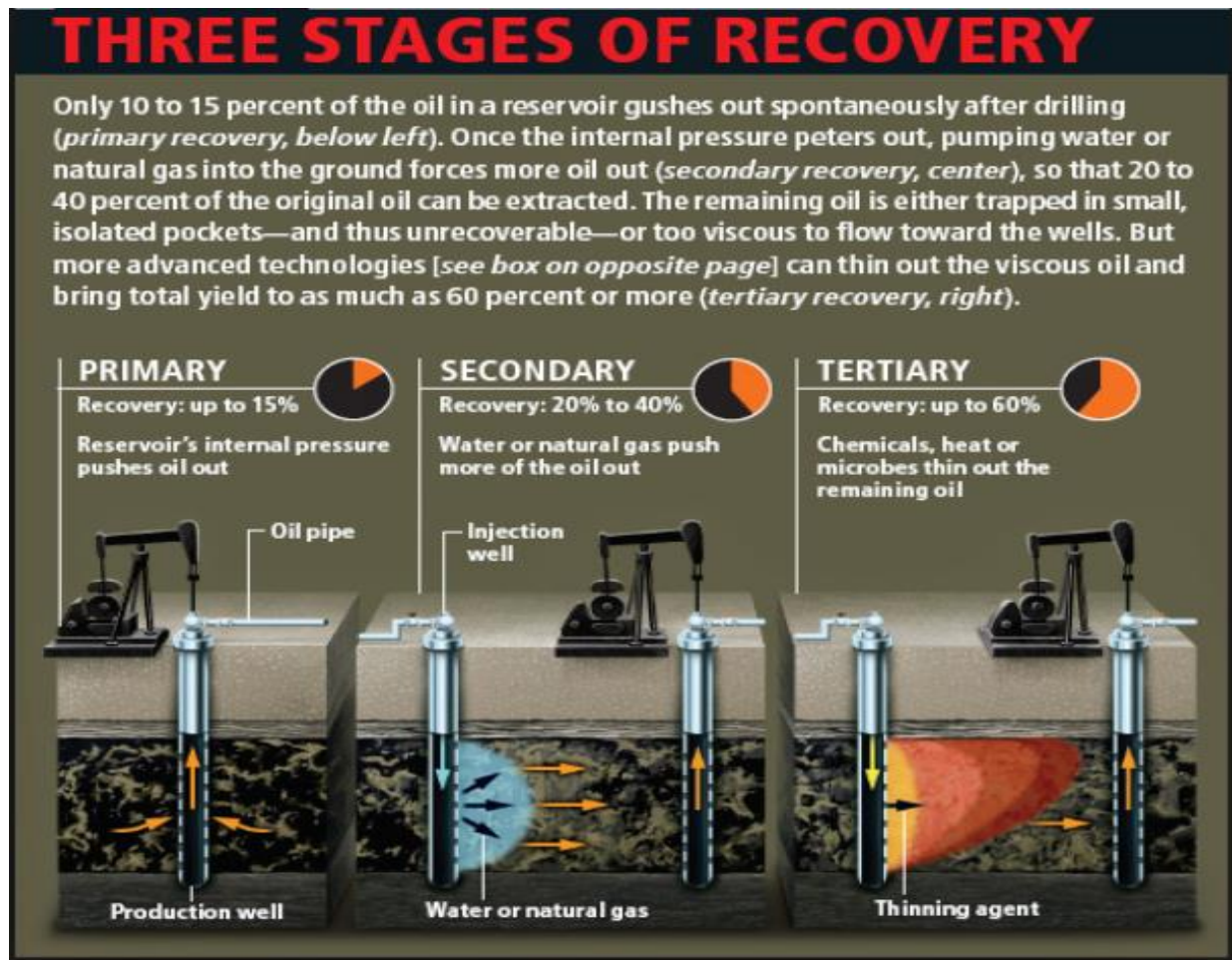


Figure 1-1: Stages of Recovery [2, p. 56]

The objective of EOR is to increase the recovery from reservoirs depleted by secondary recovery. Thermal processes are extensively used for the displacement of heavy oils, while chemical and gas injection displacement processes are employed for the recovery of light oils. [3, p. 14]

Based on an analysis of more than 1,500 test plants worldwide, the thermal and chemical methods were found to be more efficient in sandstone reservoirs compared to carbonates. The gas methods showed higher efficiency in carbonate reservoirs. [4, p. 1]

1.1 Polymer Flooding

Polymer flooding is considered as the most important type among other chemical EOR methods. It is the only approved chemical method that can be used in carbonates and in sandstone reservoirs. Polymer flooding has the potential to increase the recovery factor by 5-30% of the OOIP (Original Oil in Place). The polymer flooding process can be considered as an extension of the water-flooding process, which means that all ground facilities that are already available on site can be used in the polymer flooding process. [5, p. 157]

The mobility ratio plays an important role in deciding whether the polymer injection is successful or not. This depends on the fractional flow obtained from Darcy's law as it's shown in **Eq.1** [6, p. 12]:

$$f_o = \frac{1}{1 + M} = \frac{1}{1 + \mu_{oK_w} / \mu_{wK_o}} \quad (1)$$

The addition of polymers will lead to the increase of water viscosity, reduction in water relative permeability, increase in the fractional flow and hence an increase in the oil recovery by improvement in areal and vertical sweep efficiency. When the mobility ratio is less than one, the displacement of the oil by the water is efficient. However, mobility values greater than one will cause the water to push through the oil front and leave behind unswept oil. [7, p. 1504]

Generally, there are two types of polymers that are being used for EOR applications. The most frequently used one is the synthetic (hydrolyzed polyacrylamide – HPAM as shown in Figure 1-2) and the second is a biopolymer (Xanthan gum as shown in Figure 1-3). HPAM is used in about 92% of the EOR cases due to its low cost.

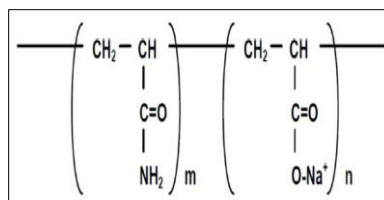


Figure 1-2: Molecular Structure of HPAM. [8, p. 3]

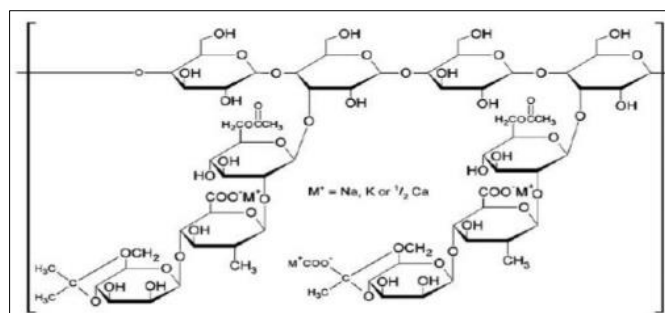


Figure 1-3: Molecular Structure of Xanthan Gum. [9, p. 2]

1.2 Problem Definition

The demand for chemical enhanced oil recovery methods in the oil and gas business is increasing. Polymer flooding is one of the most important chemical methods, which is done by adding polymer to the injection water, whereby the viscosity of the water increases and improves the mobility ratio. Yet, the difficult challenge for the oil and gas companies is to treat the back-produced polymer water. [10, p. 1]

OMV water treatment plant (WTP) is designed to treat produced water without polymer. Early investigations have shown that the treatment of the produced oilfield water containing back-produced polymer with state of the art in oil and water separation technology such as the dissolved gas flotation, which is used in the WTP have problems with polymer-containing water.

The polymer and specifically polyacrylamide leads to higher viscosity and strong chemical interactions. Whereby, the performance of the flotation unit suffers and the removal efficiency decreases dramatically. Researchers showed that the separation efficiency of the flotation units decreases with increasing viscosity. [11, p. 6]

There are three treatment stages in WTP: parallel plate separators, dissolved flotation tanks and filtration stage (nutshell filters). Tests with back-produced polymer were performed and showed that the polymer has no effect on parallel plate separators, but the polymers affect the dissolved flotation stages.

As a result of low removal efficiency by the flotation cells, nutshell filters cannot be used to treat back-produced polymer water because the oil-in-water content is going to be too high with the presence of polymer. [12, p. 11]

1.3 Research Objectives

The objectives of this thesis are to compare the dissolved gas flotation unit of the OMV water treatment plant with the new technology called micro-bubble flotation using four chambers. This new technology combines the process of dissolved gas flotation and induced gas flotation. A comparison of the important parameters for both flotation types was performed in order to evaluate if the new technology can treat back-produced polymer water.

However, the main objective is to explain the advantages regarding the four chamber unit in form of different operating parameters such as nitrogen consumption, chemical consumption, retention time, flow condition, micro-bubbles quality and high oil loading.

The scope of this thesis is to show the results of the four-chamber unit regarding oil-in-water content, total suspended solids, polymer concentration and turbidity for conventional water as well as for back-produced polymer water.

For the comparison laboratory analysis were used: sludge analysis, oil-in-water content, polymer content, the molecular weight distribution of the polymer, suspended solids and turbidity analysis.

2 Literature

2.1 Principles and Types of Flotation Technologies

The first references about flotation by buoyancy were introduced in 1860 by the British patent, the process involved the addition of oil to waste water where the lighter density of the oil surfaced impurities in the water itself. This concept took 44 years to improve until some scientists in the United States discovered the concept of introducing gas and creating gas bubbles by mechanical dispersion. In the 1920s the concept of dispersed air flotation and electrolytic flotation was introduced, while the dissolved air flotation came to the industry in the early of 1930s. [13, p. 699]

There are several factors that increased the demand for the new gas flotation technology such as:

- Increased use of EOR techniques
- Increased production from unconventional reserves (heavy oil and oil sands)
- More stringent environmental regulations throughout the world
- Increased volumes of produced water from older reservoirs

The first two points result in generating produced water, which is more difficult to treat. This indicates that the older flotation techniques are no longer meeting the industry requirements. [14, p. 1].

There are important flotation design parameters which are gas solubility, Stoke's law, bubble size, rise rate and air-to-solids ratio.

Separation technologies can be generally divided into two main types, gravity and non-gravity based separation technologies. Non-gravity technologies typically used worldwide are hydrocyclones and filtration technologies such as walnut shell filters, sand filters and multi-media filters. The gravity technologies focus on the fact that the specific gravity of oil is less than that of water. They are divided into two categories, those that operate with the assistance of gas which is the topic of this thesis and those that operate without gas in the flotation process. [15, p. 2]

The flotation process consists of several steps (1) generation of gas bubbles (2) contact between the gas bubbles and the particles to be removed, (3) flotation created through the buoyancy force and (4) removal by skimming. The dissolved gas can be either air, nitrogen or another type of gas. The bubbles attach to the particles and through the buoyancy force of the combined particle and the gas bubbles, they rise to the surface. Particles that have a higher density than the liquid can rise while the particles with a lower density than the liquid can be facilitated (suspension). In the end, the particles float to the surface and they can be collected through a skimmer. [16, p. 431]

Gas flotation technologies are subdivided into dissolved air flotation (DAF), induced (dispersed) air flotation (IAF) and electrolytic flotation (EF). The technologies differ by the method used to generate gas bubbles and the resulting bubble sizes. [16, p. 432]

In dissolved air flotation, the gas is released from a supersaturated solution as a result of the reduction of pressure. In induced air flotation, the gas and liquid are mechanically mixed to induce bubble formation in the liquid and in the electrolytic flotation, the bubbles are generated by electrolysis of the water. [16, p. 432]

2.1.1 Design Parameters of Flotation

There are several key parameters, which are commonly applied when considering and assessing the design of flotation system.

2.1.1.1 Gas Solubility

Gas solubility in fresh water depends mainly on temperature, type of gas and partial pressure of the gas. Additionally, the gas solubility of saline water depends also on the salinity. William Henry discovered in 1803 the relationship between the solubility of gas and the pressure of the gas. His law can be expressed as “at constant temperature, the amount of gas that dissolves in a given type and volume of liquid is directly proportional to the partial pressure of that gas in equilibrium with that liquid”, in other words, the higher the pressure the greater the solubility of the gas in the liquid as it's shown in **Eq.2** [17, p. 82].

$$C = K_H P \quad (2)$$

C reflects the solubility of a gas at a constant temperature in mg per liter in a particular solvent. K_H is Henry's law constant and P (atm) is the potential pressure of the gas. The constant K_H is different for each system and varies with temperature as the greater the pressure the more air can be absorbed into the water at a constant temperature. As an example, if we double the pressure of a liquid, the solubility of the solution is also going to double. [17, p. 82]

2.1.1.2 Stoke's Law

In 1851, British scientist Sir George G Stokes described the physical relationship that governs the settling of solid particles in liquids as it's shown in **Eq.3** [18, p. 54]. Flotation unit make use of the size and density parameters in Stoke's equation. The latter can be used to calculate the bubble rise rate or the falling rate of a spherical object in a fluid such as water. It states that the rise velocity is dependent on bubble/droplet diameter and density difference. Therefore oil droplet size is very important because the smaller the droplets the slower the rise velocity. Attaching gas to oil reduces the oil density, which increases the density difference between the oil agglomerates and the water. At the same time the diameter of the agglomerate is going to increase, producing a faster rise rate.

$$V_T = \frac{gD^2(d_W - d_B)}{18\nu} \quad (3)$$

In this expression V_T is the terminal settling velocity or particle settling velocity (m/s), g is the gravitational constant (9.81 m/sec^2), ν is the water viscosity (Pa-S), d_w is the water density (kg/m^3) and d_B is the density of the gas bubble (kg/m^3) and).

Stoke's law is a function based on the condition of the following assumptions:

1. Particles are spherical and have the same size
2. Flow is laminar in both directions, horizontally and vertically. [18, p. 54]

2.1.1.3 Bubble Size and Rise Rate

The rise rate of oil droplets is also governed by Stoke's Law. If the droplet size, the specific gravity and the viscosity of the continuous liquid are known, the rising rate can be calculated. The calculation using this method for all the droplets is impossible due to their size discrepancy and coalescence into larger droplets

Generally, the smaller the bubble size a better removal will be achieved because smaller bubble size would result in a rise at a slower rate. Bubbles larger than $150 \mu\text{m}$ called "macro-bubbles" and they will have a faster rise rate as predicted by Stoke's Law. These bubbles impair the flotation processes by hindering interactions between flocs and micro-bubbles, causing a turbulent flow because of their high rise rate. Figure 2-1 shows the difference between small and large bubbles, the left one is from the year 1979 by Ramirez, the right one is an extension for the large bubbles by Degremont. The graph on the left for the small bubble size by Ramirez showed that the equation of stoke's is valid until $150 \mu\text{m}$, the reason is that of the elliptical shape that offers less resistance to its rise. The dashed lines in the left graph show that the rise rate is greater than the theory predicts for diameter greater than $150 \mu\text{m}$. For the graph on the right side, Degremont extended the curve to illustrates the rise rates for larger-size bubbles which obviously are faster. [19, p. 13]

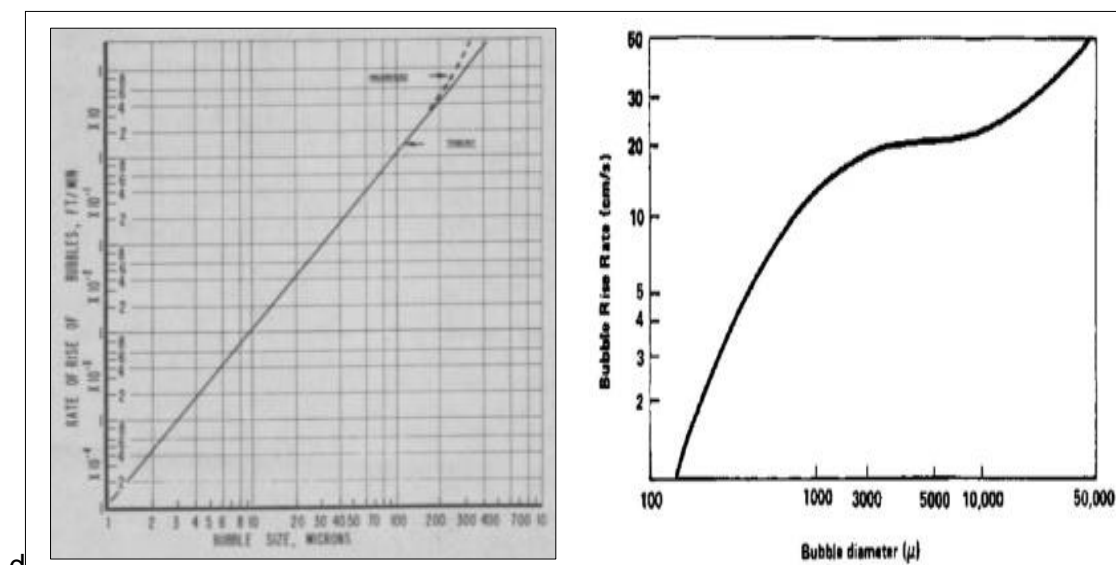


Figure 2-1: Bubble Rise Rate (cm/s) vs Bubble Diameter (μm) (Left: between 0-150 Micrometer [13, p. 701]) (Right: between 100-50,000 Micrometer [19, p. 13])

Figure 2-2 shows the bubble size distribution in micro-meter for the three flotation systems. As shown from the distribution, induced air (dispersed) is far away from normal distribution not like the case for electrolytic flotation and dissolved air flotation, which have a normal distribution. [13, p. 703]

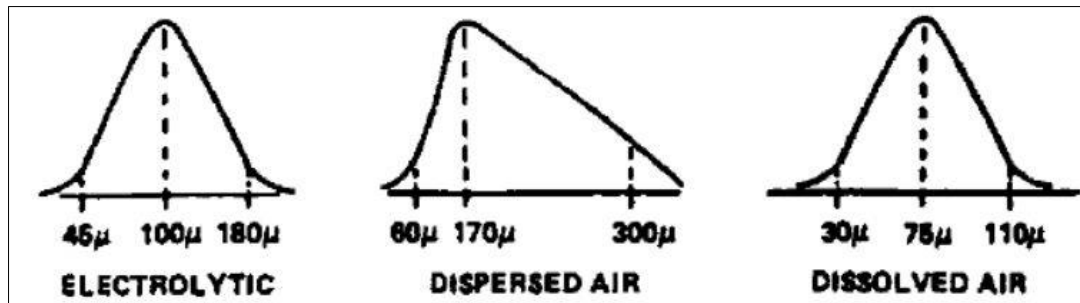


Figure 2-2: Bubble-Size Distribution of Three Different Flotation Systems [13, p. 703]

2.1.1.4 Air to Solid Ratio

There is an interaction of gas to solid (bubble-particle) and gas to liquid (gas dissolving into liquid and precipitated as fine size bubbles). The air to solids ratio (i.e., A/S) is an empirical parameter defined as the ratio of the mass fluxes of air and solids, A is the mass of air and S is the mass flux of solids. In a system containing oil, the term “solids” can be replaced by “oil and grease”. A/S ratio considers the most important parameter in designing flotation systems because if less amount of air than the optimum is used, the efficiency of solids removal is reduced or if the amount of air exceeds the optimum. In this situation the power is wasted in compressors. Typical A/S ratios needed in the process of thickening the sludge in waste water treatment plants range from 0.005 to 0.060 ml (air)/mg (solids). [17, p. 80]

The air/solids ratio is expressed in **Eq.4** [17, p. 80]

$$\frac{A}{S} = \frac{1.3 C_s (fP - 1)}{S_a} \quad (4)$$

Where A/S is the air/solids ratio (ml/mg), C_s is the air solubility, f is the fraction of gas dissolved at pressure P (typically f is 0.8) and S_a is the sludge solids concentration of total suspended solids, the factor 1.3 is the weight in milligrams of 1ml air.

In recycle pressurization systems, the whole amount of air is in the recycle stream and the suspended contaminants are in the raw waste stream. Therefore the equation must be modified as shown in **Eq.5** [17, p. 80]:

$$\frac{A}{S} = \frac{1.3 C_s (fP - 1)R}{S_a Q} \quad (5)$$

Where R is the pressurized recycle and Q is the influent flow rate. [17, p. 80]

2.1.2 Comparison between Flotation Types

The different methods of producing the gas bubbles lead to different types of flotation processes which are electrolytic flotation, dissolved air flotation and induced air flotation.

2.1.2.1 Electrolytic Flotation

The basis of EF is the generation of bubbles of hydrogen and oxygen in a dilute aqueous solution by passing a direct current between two electrodes. Electric power is supplied to the electrodes at a low voltage potential of 5 to 10 V by means of a transformer-rectifier resulting in an electric field between the cathode and the anode due to the conductivity of the liquid. The energy required for EF depends on the conductivity of the liquid and the distance between the electrodes. [20]

As a result of this reaction, hydrogen and oxygen are liberated at the cathode and the anode respectively in the form of gas bubbles that rise and produce a blanket effect. The bubbles carry the particles and the oil droplets to the surface where a floating sludge layer forms. This floating layer can be mechanically removed. [20]

There are several advantages of EF such as:

1. Uniform mixing of the gas bubbles is achieved because the electrode grids can be arranged to provide good coverage of the surface of the tank.
2. Very large numbers of small bubbles (20–40 μm) are formed with low turbulences.

Some of the problems are that the electrodes always require maintenance and periodic replacement, fouling of the electrodes is also a frequent problem.

The application of EF has been restricted mainly to sludge thickening, treatment of animal wastes and textile waste waters. A schematic diagram of an EF plant can be shown in Figure 2-3.

EF considered as the perfect system for a flow rate of 10 – 20 m^3/h . [21, p. 351]

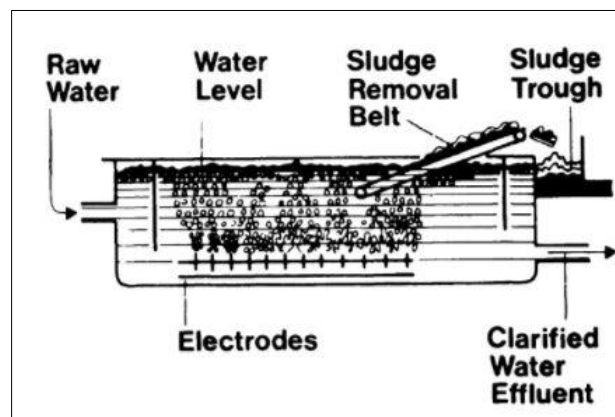


Figure 2-3: Schematic of an Electrolytic Flotation Plant [21, p. 351]

Generally for considering the bubble sizes, EF generates smaller bubbles in comparison to induced air flotation but larger bubbles than dissolved air flotation.

2.1.2.2 Dissolved Air Flotation

In DAF the bubbles are generated by pressure reduction of a water stream saturated with air.

A typical DAF system as shown in Figure 2-4 consists of:

1. Pressurizing pump
2. Air-injection system
3. Saturation vessel (Gas dissolving reactor)
4. Pressure relief valve
5. Flotation vessel
6. Chemical addition system

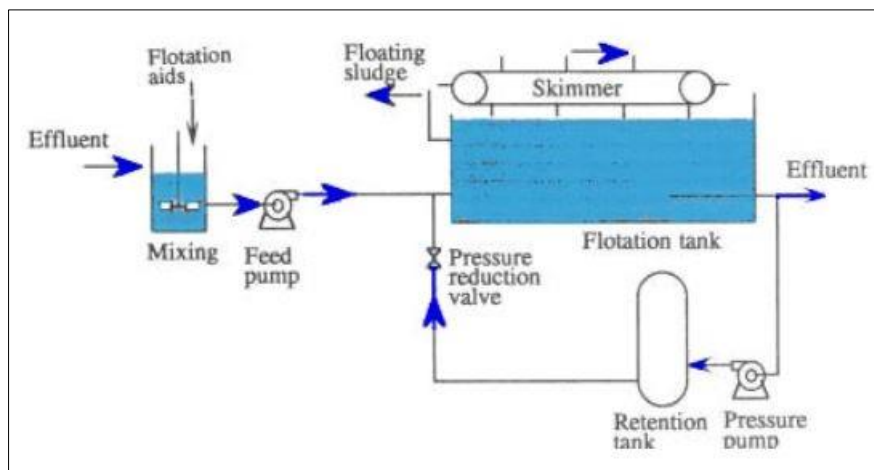


Figure 2-4: Schematic of a Typical DAF System [22]

There are two main types of DAF, which are vacuum flotation and pressure flotation.

a) Vacuum Flotation

The water, which needs to be treated is saturated with air at atmospheric pressure. A vacuum is then applied to the flotation tank that releases air as small bubbles. The attached solid particles and bubbles rise then to the surface to form a scum blanket which is removed by a skimmer. The amount of air available for the flotation is limited by the vacuum. Furthermore, this system is operated as a batch process, which means it requires sophisticated equipment to maintain the vacuum. This method is limited and replaced by the pressure flotation. [23, p. 420]

b) Pressure Flotation

In pressure flotation air is dissolved in water under pressure. There are three basic pressure DAF processes as shown in Figure 2-5: flow pressure flotation, split flow pressure flotation and recycle flow pressure flotation.

- Full flow pressure flotation: the entire inlet is pressurized and saturated with air. In this process most of the air is being dissolved that gives a high probability of particle-bubble contact. This process is cost-intensive because it needs a lot of space.
- Split flow pressure flotation: a fraction of the water is pressurized (less costs) because it requires smaller pressurizing pumps than full flow are needed. Due to the small equipment less air is provided, which means operation at high pressure is required to provide the same amount of air.
- Recycle flow pressure flotation: in this process the whole incoming water flows into the flotation tank after the water was treated. 20-50% of this water is recycled, pressurized and saturated with air. The pressurized water is transferred back into the flotation tank. The flotation tank has to be designed larger than the other two processes because the recycle flow is also added. [19, p. 21]

DAF generates smaller bubbles when compared with IAF and EF and it is the most suitable method for the operating in large plants.

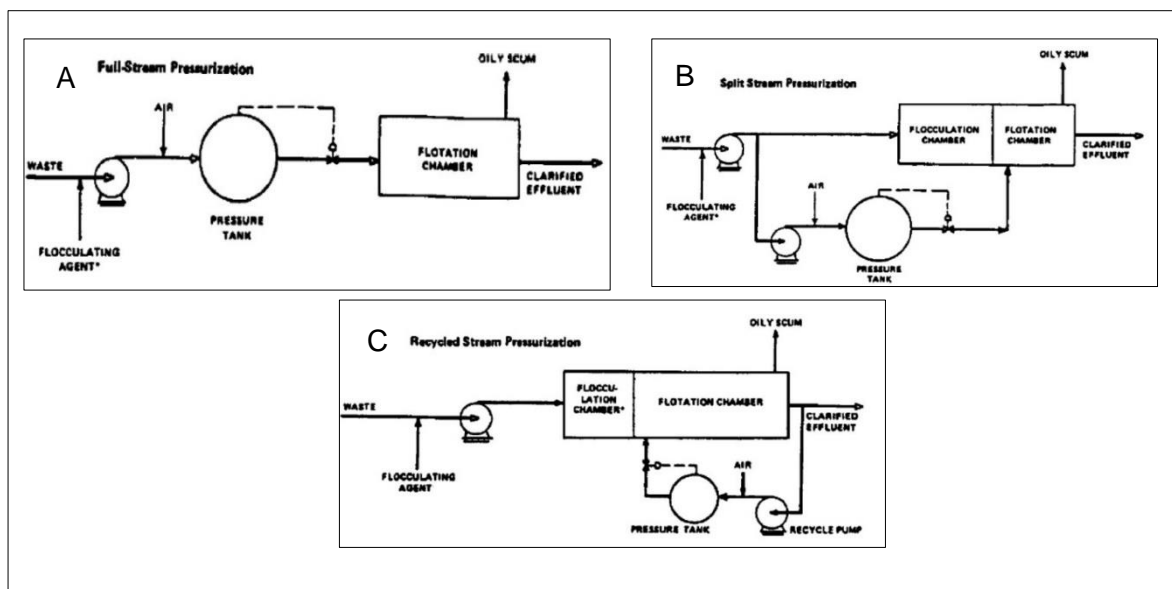


Figure 2-5: Flow Diagram of DAF Pressurization Systems [19, p. 22]

2.1.3 Induced Air Flotation

Induced air flotation introduces the air directly into the water. Therefore revolving impeller, a diffuser or an ejector are used at low pressure to generate larger air bubbles into the larger volume under turbulent conditions. The impeller acts as a pump forcing the fluid through dispersed openings and creating a vacuum in the standpipe as shown in Figure 2-6. The vacuum pulls air (or gas) into the standpipe and mixes it with the liquid, as the gas/liquid mixture travels through the disperser. A mixing force is created so that the gas creates very fine bubbles. The liquid moves through a series of cells before leaving the unit. Oil particles and suspended solids attached to the bubbles rise to the surface. The oil and suspended solids gather in a dense froth at the surface and are removed by skimming paddles. [23, p. 421]

Advantages of this process are compact size, lower capital costs and capacity to remove free oil as well as suspended solids. Disadvantages are that IAF generates bigger bubbles which rise faster than the bubbles of DAD and EF.

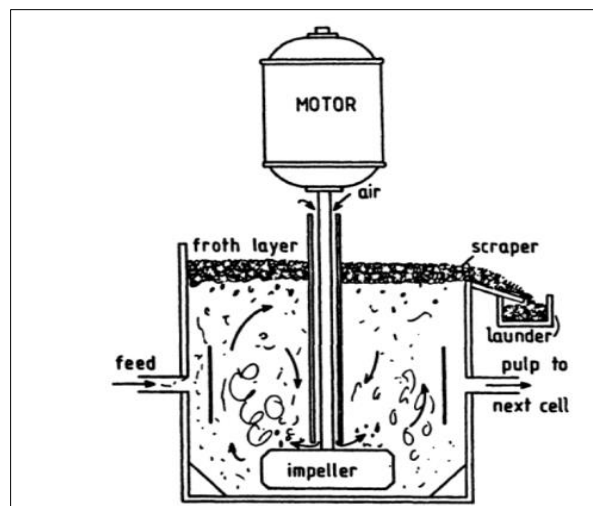


Figure 2-6: Schematic of Induced Gas Flotation. [21, p. 17]

Table 2-1 shows a comparison of the important parameters between flotation technologies:

- Dissolved air flotation produces the smallest bubble diameter.
- Due to small bubble diameter, the surface area of the bubbles for dissolved air flotation is the highest compared to the other two flotation technologies
- Dissolved air flotation has the highest amount of bubbles per cm^3
- The rise rate of the bubbles for dissolved air flotation is the slowest.

All the points indicate that DAF is the best flotation technology.

Table 2-1: Comparison of Flotation Technologies [13, p. 705]

Parameter	Electrolytic	Dissolved	Dispersed
Average bubble diameter (μm)	100	75	170
Bubble surface area (cm^2)	545	800	293
Number of bubbles/ cm^3	10^6	$3.6 \cdot 10^6$	$0.2 \cdot 10^6$
Bubble rise rate (in/min)	12	3.8	36

2.2 Coagulation and Flocculation Principles

Figure 2-7 shows the process of coagulation and flocculation. Those are the basic principles for understanding the chemical involvement in the water treatment process; these principles are typically applied prior to flotation or filtration processes to enhance the ability of these processes in removing oil particles.

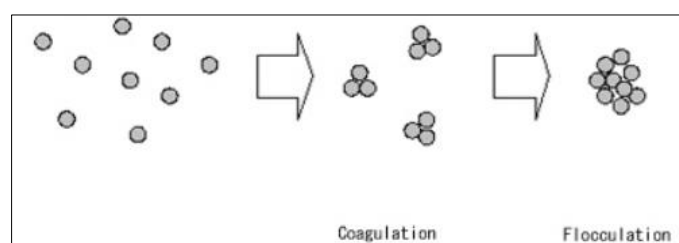


Figure 2-7: Process of Coagulation and Flocculation [24]

The coagulation process occurs when adding chemicals carrying opposite charges to that of the colloidal particles that carry a negative charge. In other words the coagulants are chemicals with a positive charge which are added to the water. They will help to overcome the repulsive charge and destabilize the suspension.

The coagulants that are used in the water treatment process can be divided into two groups, aluminum base and iron base. Iron base coagulants include ferrous sulfate, ferric sulfate and ferric chloride. Aluminum coagulants include aluminum sulfate, aluminum chloride and polyaluminum chloride. There are some other metals that can be used as coagulants like titanium and zirconium but they are significantly more costly and rare. [25, p. 55]

The next step is flocculation. Natural polymer is a typical flocculant in the water treatment industry. Flocculation occurs in two stages; the first stage is based on Brownian movement and named after “Perikinetic (Brownian) flocculation”. Flocculation during this stage commences immediately after destabilization and is completed within seconds since there is limiting floc size which was shown by the Brownian motion.

The second stage in the flocculation process is called orthokinetic flocculation and arises from the induced velocity gradients in the liquid. Such velocity gradients may be induced by setting the liquid in motion by mechanical agitation or passages around baffles. The effect of velocity gradients with a body of liquid is to set up relative velocities between particles that provide an

opportunity for contact. The greater the velocity gradients, the smaller the ultimate floc size would be due to the breakdown of the larger flocs and the lower the velocity gradients the longer will be the time needed to reach the optimum floc size which results in larger final floc size [26, p. 84].

Typically the number of chemicals that are needed for both coagulants and flocculants can be tested using jar tests. The purpose of the jar test is to select the right type of the chemicals, also to estimate the optimal dose of coagulants and flocculants that is needed to remove particles that occurred in water.

2.3 Treatment of Back-Produced Polymer Water

Nowadays the conventional water treatment is no longer representing an issue. The reason is due to the availability of modern technologies like flotation, hydrocyclones and filtration processes. In order to improve the productivity of a particular reservoir, tertiary recovery methods can be used, whereby one option is polymer flooding, which adds polymer to the conventional water. Problematic is the treatment of back-produced polymer-containing water streams and the reason is through the increased viscosity of the water which will affect all these new technologies as shown in Figure 2-8. The loss of efficiency can reach more than 50% for flotation devices and 90% for hydrocyclones. A combination of technologies or pre-treatment (viscosity reduction) are the solutions for treating the back-produced polymer water. [10, p. 2]

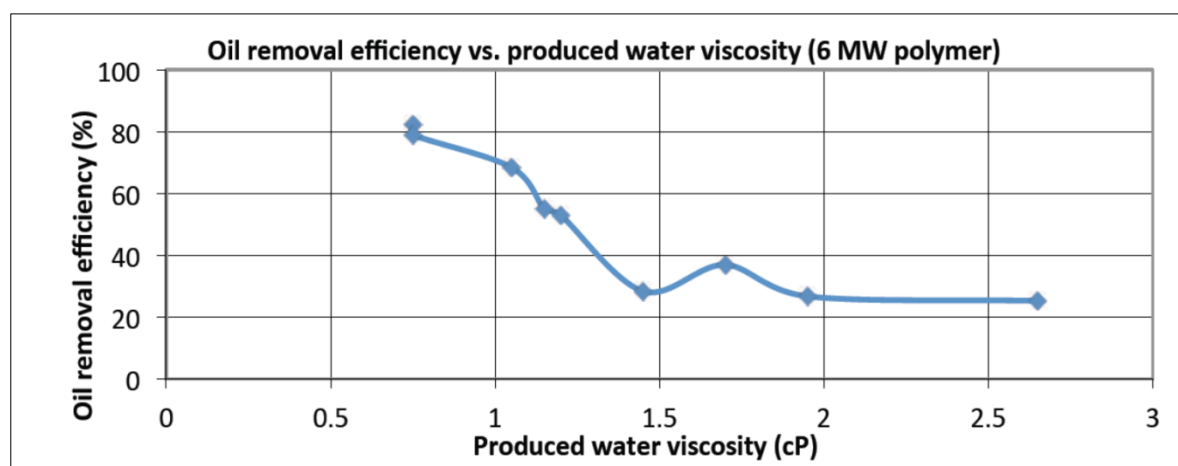


Figure 2-8: Oil Removal Efficiency vs. Produced Water Viscosity [10, p. 2]

As shown in Figure 2-8, the oil removal efficiency decreases to below 40% when the produced water viscosity increased to more than 1.5 cP.

2.3.1 Viscosity Reduction

Increasing water viscosity by injecting polymer is going to simultaneously increase the oil recovery but also decreases the efficiency of the treatment tool that is used for treating the conventional water. The decrease in the treatment efficiency is due to the high viscosity caused by the introduction of the polymer.

In order to improve the conventional treatment technology, the viscosity of the back-produced polymer water has to be reduced to an acceptable range so that the conventional treatment methods such as induced gas flotation can handle it.

Viscosity reduction is considered as a pre-treatment phase. The aim of the pre-treatment is to degrade the long chains of the polymer so that the conventional water treatment technologies can work with its maximum efficiency. Different degradation methods can be used for decreasing the viscosity of the polymer such as chemical, thermal and mechanical.

- **Chemical degradation:** decreasing the viscosity by using chemicals such as oxidizing agents. These agents generate radicals which break the polymer chains.
- **Thermal degradation:** the long chain backbone of the polymer can be broken and react with one another to change the properties of the polymer at high temperatures.
- **Mechanical degradation:** mechanical degradation of a polymer means shear degradation that occurs primarily in the surface injection equipment such as valves and pumps or in the downhole restrictions like tubing or perforations. Commonly pumps are used as shearing tools. Passing the polymer through pumps generates a viscosity drop of the back-produced polymer water. [27, p. 476]

Figure 2-9 shows the comparison between the results of shear degradation through a pump and through a valve.

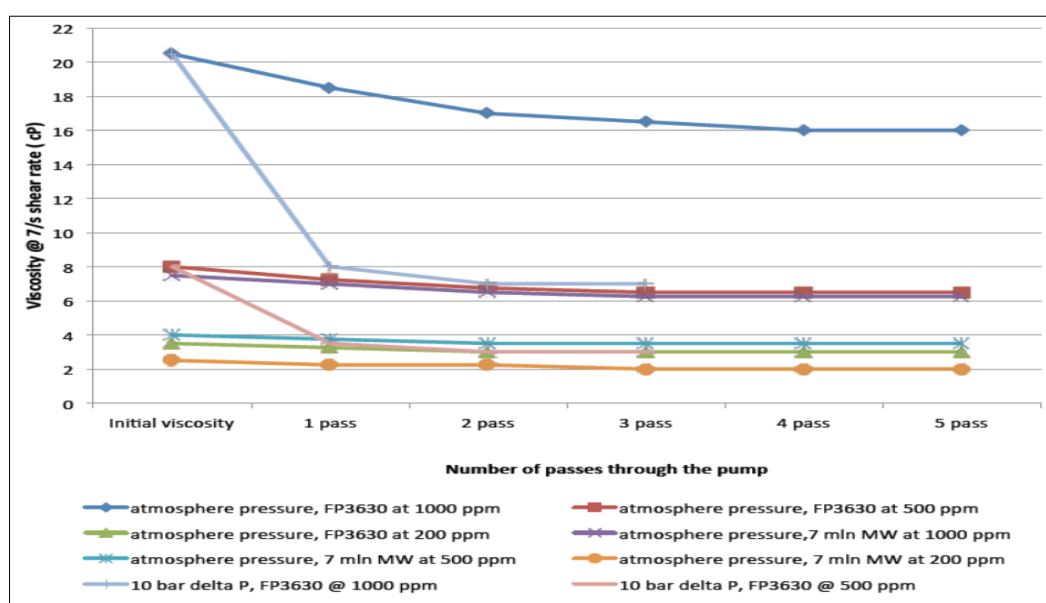


Figure 2-9: Viscosity Vs Number of Passes through Centrifugal Pump [10, p. 7]

As shown in Figure 2-9, the atmosphere pressure trial are ones with pumps which were six trials. The trials named 10 bar are those with valves which were just two.

From comparing two results which had similar viscosities at the start (pump trial “dark blue” and the valve trial “light blue”), the trial with the valve was decreased from 20.5 cP down to 8 cP. The trial with the pump decreased only down to 18.5 cP. This can clearly explain that degradation by valves is way better than with pumps.

2.3.2 Combine Two Flotation Technologies

One of the methods for treating back-produced polymer is to combine two flotation methods. The test was done with induced gas flotation and dissolved gas flotation which were explained in details in the sub-chapter (2.1.2).

Using induced gas (air) flotation produces macro-bubbles, which are bigger bubbles than 100 microns. Dissolved gas (air) flotation produces micro-bubbles, which are bubbles with a size of 10-100 microns. A combination of both processes increases the efficiency of treating back-produced polymer water. Micro-bubbles are first injected into the flotation tanks, attached to the oil droplets to create a layer around the oil droplets while the macro bubbles lift the oil droplets by attaching to this layer. As shown in Figure 2-10 the combination is going to increase the efficiency of treatment of the back-produced polymer of about 15% on average at different polymer concentrations.

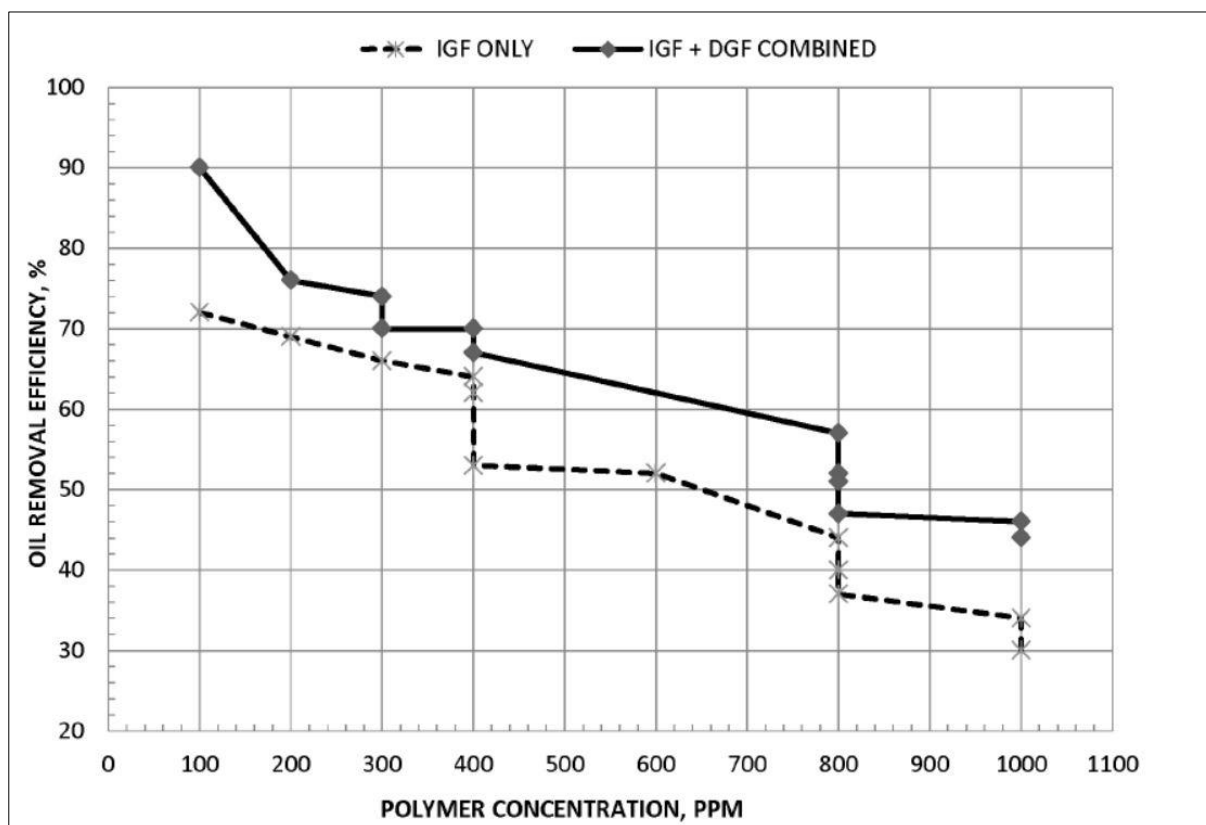


Figure 2-10: Efficiency of IGF vs. Combination of IGF&DGF for the Treatment of Polymer [28, p. 14]

2.4 Micro-Bubble Flotation Technology

There are a few technologies for the treatment of back-produced polymer water such as hydrocyclones or dissolved gas flotation. OMV ran a trial for the treatment of back-produced polymer water using a hydrocyclone in addition to a compact flotation unit and the results were not satisfactory. This trial pushed the company to look for a new technology in the treatment of back-produced polymer water. They found the technology of micro-bubble flotation which was developed by a company called GLR which is subsidiary of Exterran. The main design of this technology depends on the combination of two flotation principles that was shown in sub-chapter (2.3.2) which is found to be the best option.

GLR solution introduced the new technology of producing micro-bubbles with sizes between 5-50 microns. The micro-bubble flotation technology achieved such small sizes by combining the process of DGF and IGF. The technology has a DGF part as it has a gas and a liquid phase under pressure and IGF part because it will use shear as well as impact forces to create microbubbles.

The principle of this technology is that the flotation unit has four separated chambers instead of the normal flotation unit which is only one chamber. This four chamber system treats the water four times instead of one time and leads to a higher retention time so that the removal efficiency increases.

The microbubble flotation system consists of flotation tank, gas-liquid reactor, pumps and valves.

The gas liquid reactor is the heart of this system. Water and nitrogen enter from the top where the nitrogen gas dissolves into the water. By using shear and impact force, the mixture of the gas droplets and the water goes out from the gas liquid reactor in form of micro-bubbles, which make the water look milky. These micro-bubbles enter then the chambers in different amounts in order to adsorb to the oil droplets and take them to the surface of the chambers, where they will be skimmed and collected in the skim tank.

The advantage of this technology is that part of the outlet of the final chamber will mix with nitrogen which means that the nitrogen amount is not going to be changed when the flow rate is changed.

3 Plant Site

The plant site where the pilot unit is located, is called Schönkirchen V. This is the metering station used by OMV for testing different pilot units. All the units located here like separator, production wells, OMV water trial (OWT), four chamber flotation unit (FCFU), nitrogen tank and slope containers as shown in Figure 3-1.

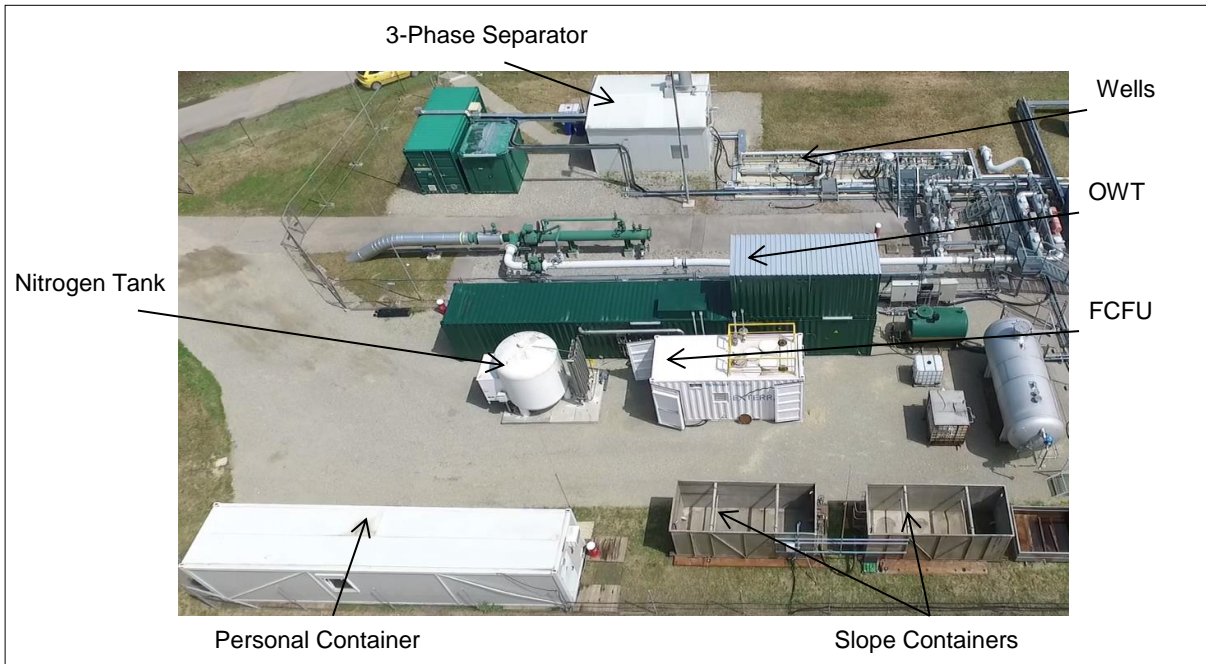


Figure 3-1: Schönkirchen V

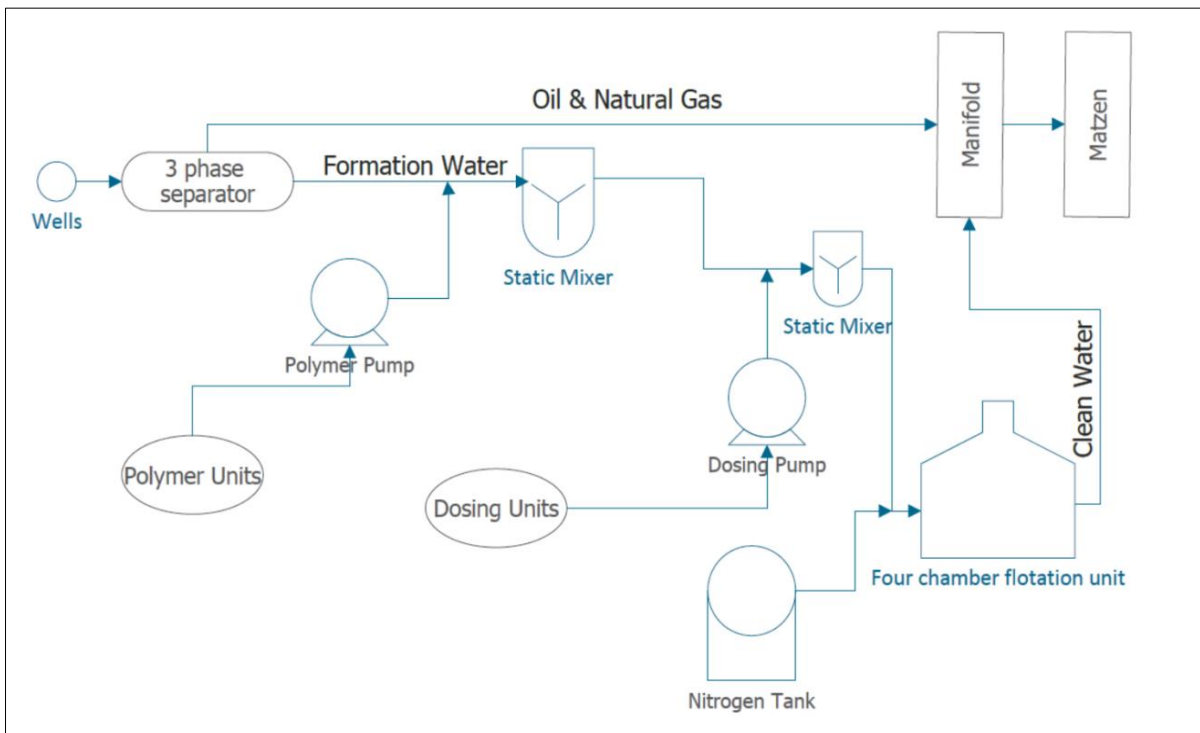


Figure 3-2: General Process Flow Diagram of the Plant Site

In Figure 3-2, a process flow diagram (PFD) of the plant site is shown. The formation water coming from the wells enters firstly into the three-phase separator, where oil and gas are separated and then transferred to the manifold, whereby the water goes through two static mixers. There it is mixed with the chemicals from the dosing pump. The water enters the FCFU and is mixed with nitrogen to produce the bubbles which will separate the oil from the water. In the end the clean water is pumped to the manifold.

3.1 Production Wells

Eight wells were connected to the pilot plant according to the different trials. All these wells are producing from an underground reservoir located in the 8 Tortonian Horizon.

All the production wells are conventional wells except well S66 which is a back-produced polymer well. The details of the production volumes of each well are stated in Table 3-1.

Table 3-1: Production Volumes of the Production Wells

Well No.	Gross Rate (m ³ /d), avg	Oil (m ³ /d), avg	Gas (Nm ³ /d), avg	Water (m ³ /d), avg	Water (m ³ /h), avg
Ma108	32.1	1.1	72	30.9	1.29
S66	42.8	3.1	116	39.3	1.64
S79	72.6	1.6	179	70.8	2.95
S96	21.5	1.7	260	19.6	0.82
S109	62.9	1.8	161	60.9	2.54
S110	54.8	1.7	146	52.9	2.21
S126	29.8	1.2	129	28.5	1.19
S135	83.1	1.4	128	81.6	3.40

3.2 Three-Phase Separator

The term three-phase separator (TPS) in oilfield terminology refers to a pressure vessel that separates fluids into three phases as shown in Figure 3-3. The TPS uses gravity to separate produced well fluids into gas, oil and water phases.

The fluid enters the vessel and hits an inlet separating element like a diverter which will cause a sudden change in momentum of the fluid that causes the initial gross separation of liquids and vapor. The gas flows horizontally and exits through a mist extractor to a pressure control valve which maintains constant pressure while the liquids go down into the liquid collection part of the vessel. The liquid collection part must provide sufficient time for oil and emulsion to form a layer above the free water. The baffle controls the oil level while the liquid level controller or interface controller maintains the water level. The oil spills over the top of the baffles into the oil accumulation area, the oil level in this area is controlled by a level controller. The interface controller sets the height of the water-oil interface by signaling the water dump valve to release as much water from the vessel as it's needed to maintain the interface at the designed height. [29, pp. 12-2]

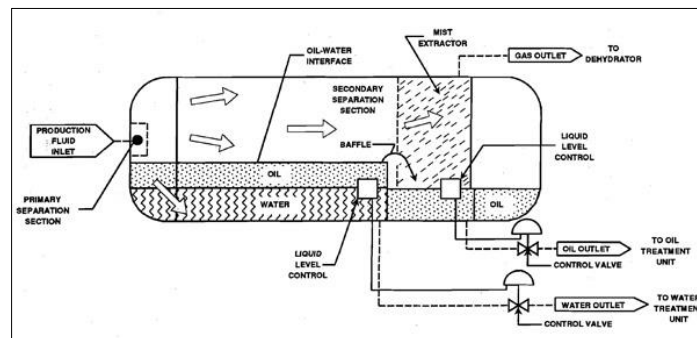


Figure 3-3: Schematic of Typical Horizontal Three-Phase Separator. [30]

Oil and gas phases are removed, transferred back to the metering station Schönkirchen V and then to the gathering station Matzen where a treatment plant is located. The water phase will be treated in the four chamber flotation unit. After the treatment, the water phase will be transferred back to Matzen treatment plant as shown in Figure 3-2.

3.3 Static Mixers

Static mixers are engineering motionless mixing devices that allow continuous blending of the fluids at the inlet.

Two static mixers were used during this trial: The first has a volume of 0.00104 m^3 , which was used to mix artificially added polymer with the water. The second static has a volume of 0.0004 m^3 and was used for mixing the chemicals.

3.4 Dosing Units

Two dosing units were used to inject different types of chemicals such as flocculent and coagulant into the line of the water using dosing motors at 1440 rpm. Different trials with different dosing amount of chemicals were performed.

3.5 Safety and Environment

Figure 3-4 shows the ex-zones of the site to show the hazardous environment of the plant.

There are three different explosion zones. Zone 0 shows the most dangerous zone where an explosive atmosphere is continuously present. Zone 1 has an explosive atmosphere which likely to occur in normal operation. All the area around zone 1 is considered to be zone 2, in this zone the explosive atmosphere is not likely to occur in normal operation.

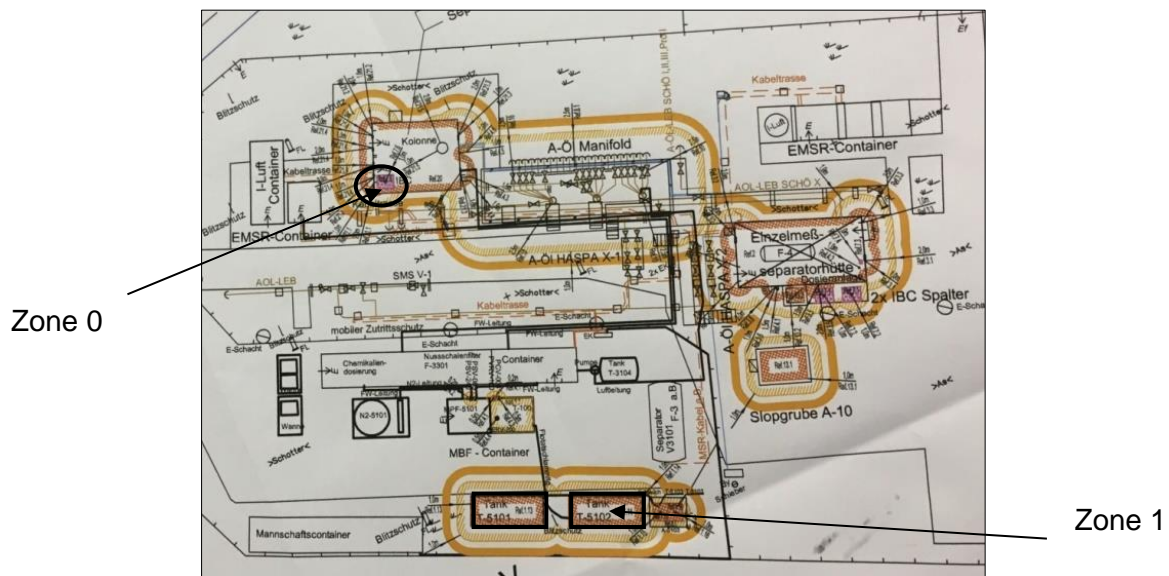


Figure 3-4: Ex-Zones of the Plant Site [31, p. 1]

4 Materials and Methods

4.1 Polymers and Chemicals

The polymers and chemicals which have been used will be addressed in the following subchapters.

4.1.1 Flopaam 3630S

The polymer Flopaam 3630S is from the company SNF FLOERGER and has been selected to be used for the polymer flood project in OMV. The structure of this HPAM was shown in Figure 1-2.

It's a copolymer of acrylic acid (AA) and acrylamide (AM) with a ratio of 30% molar (AA) and 70% molar (AM). The molecular weight of Flopaam is between 18-20 MDa. Its physical form is a powder, which indicates the S that stands for solid. [32, p. 5]

To get this polymer ready for testing, the powder has to be mixed with the injection water to bring it into a liquid form so that it can be easily injected.

Flopaam 3630 S is a straight chain polymer with a flexible structure that can coil up especially in solutions with divalent ions. In solution with water HPAM is a polyelectrolyte. [32, p. 5]

4.1.2 Chemical Set Vb

Set Vb consist of a coagulant (mix of 80% Floquat FL2949 & 20%Pluspac FD1465) and a flocculent (Chimec 5565). This chemical set was tested for WTP and also for FCFU.

Floquat FL2949

Floquat FL2949 is a cationic polymer with a pH range of 4-7 and has a specific gravity of 1.12-1.16.

Floquat FL2949 is a copolymer of epichlorohydrin and dimethylamine (EPI-DMA) and belongs to the chemical family of polyamines. It is supposed to react with Pluspac FD1465 to being used as a coagulant. [33, p. 2]

Pluspac FD1465

Pluspac FD1465 is an aqueous polyaluminiumchloride-hydroxide solution. It has pH values of 0.5-3.1 with a density range of 1.1-1.4 g/ml and has chemical stability at normal temperature (e.g. 25 °C). [34, p. 4]

Chimec 5565

Chimec 5565 belongs to the chemical family of anionic polymers and acts as a flocculent. Chimec 5565 has a physical form of viscous liquid at normal temperature and a pH value of 6-8 in distilled water.

It is recommended to use it as a flocculation aid with Floquat FL2949 and Pluspac FD1465. [35, p. 3]

4.1.3 Geochemical Set

The second chemical set which was used during the trial with FCFU is called PW1010C.

PW1010C is an inorganic metal salt in aqueous solution. It can be considered as coagulant/flocculant agent. It has a pH value of 0.5-1.5 and is a liquid with yellow color. It is soluble below a pH of 4. It has a specific gravity of 1.352-1.402 (-) and densities of 11.28-11.96 lb/gal. [36, pp. 5-6]

4.1.4 Drewfloc 285

Drewfloc 285 is a copolymer of acrylamide and acrylic acid. Drewfloc 285 was used as a flocculent aid in the process sludge treatment.

Drewfloc 285 is an anionic polyelectrolyte with medium molecular mass and high density. The Drewfloc 285 has a pH range of 6-10 and its physical state of solid.

4.2 Health and Safety

Flopaam 3630S

This chemical doesn't contain any hazardous substances. In case of skin contact a wash off with soap and plenty of water is needed but if irritation develops then a medical attention is needed.

There is a special hazard rise from the substance or mixture. Thermal decomposition may produce different components such as nitrogen oxides (NO_x), carbon oxides (CO_x). Hydrogen cyanide (hydrocyanic acid) may be produced in the event of combustion in an oxygen deficient atmosphere. [32, p. 2]

Chemical Set Vb

- **Floquat FL2949**

This chemical contains substances that are harmful to aquatic organisms and can cause long-term effects on the aquatic environment and spills produce extremely slippery surfaces. [33, p. 1]

- **Pluspac FD1465**

This chemical is an irritant hazard and can cause a serious damage to the eyes. It will form a corrosive media to metals in case of reaction with acids. [34, p. 1]

- **Chimec 5565**

This anionic polymer is an irritant hazard if it gets in contact with skin and eyes. In the case of skin contact all the contaminated clothes need be taken off. There is no fire explosion hazard; because this chemical is a water solution which means that fire is highly improbable. [35, p. 2]

Geochemical Set

- **PW1010C**

This chemical is considered hazardous to the skin and the eyes. It can cause severe skin burns and eye damage. It is considered as a corrosive agent to metals.

In case of a fire this product is a water solution and nonflammable. [36, p. 3]

Drewfloc 285

There is no available information about the hazardous situations regarding Drewfloc 285.

4.3 Analytical Methods

4.3.1 Determination of Oil-in-Water Content (OIW)

The evolution of the hydrocarbon content measurement started with infrared absorption, where oily water sample was first acidified and then extracted with a chlorofluorocarbon solvent. This followed by the separation of the extract, then the extract is removed and placed into an infrared instrument where the absorbance is measured. By comparing the absorbance obtained from an extract sample to sample with known concentration, the oil concentration in the extracted one can be calculated.

Infrared absorption is not a common method nowadays because of the problems related to solvents such as carcinogenicity like carbon tetrachloride or ozone-depleting substances like Freon.

There are two other methods that are dependent on solvents extraction which is gravimetric analysis and gas chromatography-flame ionization detection (GC-FID) but both of them are rarely used nowadays due to the same reasons mentioned above. [37, p. 3].

These problems with the solvents led to invent a new method using a solvent which has no effects on the environments. This new technology is based on quantum cascade laser infrared (QCL-IR) and on extraction by a cyclic hydrocarbon such as cyclohexane or cyclopentane. The evaluation is then carried out by measuring absorbance at a wavelength in the region of 1,350-1,500 cm^{-1} using mid-infrared spectroscopy that employs a quantum cascade laser as a light source. The company Analytics has a device called Erachek which measure oil-in-water using this method. [37, p. 4].

The term oil-in-water in this thesis refers to “Total Petroleum Hydrocarbons” (TPH) and is given in ppm (parts per million) referring to the mass of the sample.

Procedure of measurement with Erachek

500 ml sample bottles were used and weighted before and after the sampling. Figure 4-1 shows the samples of all sampling points from the inlet to the outlet. 5 ml of sulfuric acid was added to promote the dissolution of contained particles. Then 25 ml of cyclohexane was added as a solvent for the hydrocarbons. The bottles were then shaken for 10 minutes to extract the hydrocarbons from the water samples. Filtration step is the next where sodium sulfate is used to remove remaining water and aluminum oxide is for removing polar substances. Afterward the sample was measured with Eracheck, where the results are displayed in infrared light absorption unit (mAU). The hydrocarbon content is calculated according to **Eq.6 – 8** [38, p. 4].

There is a calibration factor K, which is dependent on the amount of OIW. This factor can be calculated as the amount of calibrations material divided over the average of different measurements. The calibration material can be any hydrocarbons such as Hexadecane, Tetradecane and etc... [38, p. 4].

$$\text{Oil in Solvent} = k (mAu) + d \quad (6)$$

$$k \text{ factor} = \frac{\text{Calibration Material}}{\text{Average of Different Reading Points}} \quad (6a)$$

$$\text{Enrichment} = \frac{\text{Sample Mass}}{\text{Solvent Mass}} \quad (7)$$

$$\text{Sample Mass} = \text{Sample Bottle Full} - \text{Sample Bottle Empty} \quad (7a)$$

$$\text{Solvent Mass} = \text{Mass of Sulfuric Acid} + \text{Mass of Cyclohexane} \quad (7b)$$

$$\text{Hydrocarbon Content} = \frac{\text{Oil in Solvent}}{\text{Enrichment}} \quad (8)$$



Figure 4-1: Samples of OIW from Different Sampling Points

4.3.2 Determination of Oil Droplet Size Distribution

Droplet size distribution explains the changes in the efficiency of any flotation or filtration unit. Vipa Jorin (Visual Process Analyser) was used to measure online and to provide valuable information about changes in the entire flow systems. Vipa can operate at high pressure and elevated temperatures. [39, p. 2]

Procedure

During the trial jorin analyzer was connected to the outlet and sometimes at the inlet of the four-chamber flotation unit.

During online measurements the droplet size and concentration of the oil and the solids are shown as a graph on the screen of Vipa. The results are measured at different points and showed up on the screen as an average every 30 minutes for all tests with FCFU. The time of the measurements can be adjusted before the test starts.

During the operation images of the fluid going through the flow cell are recorded with a high-speed camera and analyzed via particle recognition software. Jorin can understand the difference between oil droplets and particles by seeing their shape factor: oil droplets have the shape of a sphere which means that the oil droplets have a shape factor of one. While particles have an irregular shape meaning that their shape factor is less than one.

After a matter of time Jorin gives results which can be transformed into cumulative distributions and density functions curves. [40, pp. 1-4]

4.3.3 Determination of Polymer Concentration and Molecular Weight

Polymer Concentration

There are several methods in the literature for determination of polymer concentration.

1. Density: Through the relationship between the density and concentration it can be described as a linear function. The concentration of the polymer can be determined by density. One important advantage of density is its independence of molecular weight. [41, p. 127]
2. Ultraviolet-visible spectroscopy (UV-VIS): is the measurement of the attenuation of a beam of light after it passes through a sample or after reflection from a sample surface. This method was successfully used in the determination of polymer concentration in water samples. [42, p. 2]

Polymer Molecular weight

There are various methods for determining the molecular weight of the polymer. Viscosity can be considered as one of these methods as “Hermann Staudinger” reported in 1930. The relationship between polymer molecular weight and the relative magnitude of increase in viscosity. [43, p. 8]

OMV Tech Center & Lab uses a method called quantification of polyacrylamide in aqueous samples that so called SEC (Size Exclusion Chromatography). It determines the concentration and molecular weight of the polymer. The concentration of the polymer was measured by peak area and follow by a calibration of the diagram to have the concentration of the polymer. For the determination of the molecular weight the retention time of the maximum peaks is compared in order to measure the molecular weight. [44, p. 6]

4.3.4 Determination of Suspended Solids

Normally water contains a variety of solids or dissolved impurities. Total suspended solids (TSS) is the term used to describe particles which are large and unable to pass through the filter that is used to separate them from the water. These particles are larger than two microns, anything smaller than two microns is considered in the industry as a dissolved solid. When the oil is in the sample it leads to blockage the filter during a filtration test. [45]

Procedure

The procedure of testing the suspended solids concentration is described in the working procedure instruction AA D CHE 432 provided by OMV Tech Center & Lab.

The TSS was just measured at the samples of the inlet and outlet of FCFU as shown in Figure 4-2.

As shown in **Eq.9** [46, p. 3], the concentration of the suspended solids is calculated by the difference of the mass of the glass plus the membrane filter after the filtration & mass of the glass plus membrane filter before the filtration and the results divided by the volume that was filtered over it. The unit of the concentration is milligram per liter. If the sample has oil in it, the filter has to be cleaned from the oil by using isopropanol and naphtha as a solvent. After finishing the filtration, the filter was dried at 105°C for at least two hours.

$$\text{Concentration} = \frac{(\text{End Value} - \text{Start Value}) * 1000}{\text{Volume}} \quad (9)$$



Figure 4-2: Sample for the TSS measurement from the inlet & outlet

4.3.5 Determination of Turbidity

Turbidity is the reduction of the clarity in water due to the presence of suspended or colloidal particles. Suspended solids and dissolved colored material reduce water clarity by creating a muddy appearance. It can be also defined as an optical determination of water clarity. Turbidity can be measured by the amount of light which is reflected by the particles in the water. The more particles that are present, the more light that will be scattered. This means that turbidity and total suspended solids are related. [47, p. 4]

Turbidity is just a supplementary parameter for providing more information and confirming what other parameters like oil-in-water or suspended solids are demonstrating. Turbidity for the same sample can change with time due to the settlement of the particles on the ground of the sample.

Turbidity measurements are useful in determining the efficiency of filters or settling basins and are also useful in indicating the completeness of processes that utilize precipitations. [47, p. 4]

Procedure

For the testing procedure a nephelometer was used for turbidity measurements (Turbiquant 1000IR). The corvette which is the glass tube as shown in Figure 4-3 was filled with the sample and cleaned very well from the outside to avoid any mistakes during the measurements. The results are given in NTU (Nephelometric Turbidity Units).

The procedure of testing is based on a working procedure that was provided by OMV Tech Center & Lab. [48, p. 1]

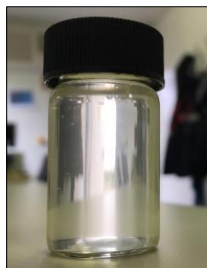


Figure 4-3: Glas Tube (Corvette) for Turbidity Measurements

4.3.6 Description of Sludge Analytical Method

Chemicals like coagulant and flocculent are added to the formation water that includes polymer for the purpose of increasing the amount of oil that is going to be extracted from the formation water and to increase the efficiency of the flotation units. These chemicals are going to form flocs which include oil as well as solids and the contaminations of these flocs called sludge. By injecting micro-bubbles that contains nitrogen, these flocs are going to connect with the nitrogen bubbles to form floatable flocs that accumulate on the surface of the water. This will then be removed by the water movement that lead to the oil tank then collected in the slope tank to be taken via special tanks to sludge treatment area. The slope tank is shown in the process flow diagram in Figure 5-1.

Procedure

In order to identify whether the sludge is treatable or not, laboratory measurements based on centrifugal forces were performed. Following steps explain the test procedure of the sludge evaluation:

1. After collecting the sludge from the chambers of the flotation unit three experiments with centrifugal tubes were performed in the laboratory. Their tubes have a volume of 100 ml. Experiment 1 shows the results without the use of any chemicals, in this experiment two tubes were filled with just sludge. In Experiment 2, the tubes were filled with two drops of demulsifier, 50 ml test benzene and 50 ml sludge. Tubes in experiment 3 were filled with 85ml sludge and 15 ml flocculent which is Drewfloc 285.
2. The temperature of the tubes was increased up to 60°C for experiment 2 due to the existence of benzene. For other experiments the temperature can be increased up to 80°C, which reflects the temperature of the tricanter. Tricanter are used to treat the sludge.
3. In the last step a centrifuge is used and run at a speed of 2,000 rpm for five minutes to separate the sludge into its components which are oil, water and solids. Due to the centrifugal movement the amounts of each component can be easily read as shown in Figure 4-4. [49, p. 17]

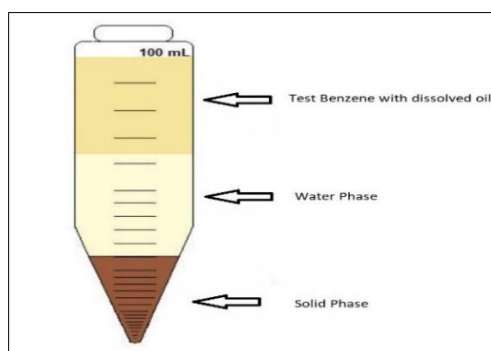


Figure 4-4: Centrifuge Tube Used for the Experiment [49, p. 18]

5 Comparison between Four Chamber Flotation Unit and WTP Flotation System

An introduction of each technology that defines the important components of their systems, their mechanical processes.

There are several parameters for comparing WTP flotation process and four chamber flotation unit (FCFU) process. These parameters are flotation principle, oil-in-water at the inlet/outlet, nitrogen consumption, chemical consumption, retention time, dissolving reactor efficiency and skimming principle.

5.1 Four Chamber Flotation Unit

Figure 5-1 shows the process flow diagram of the FCFU.

The four chamber flotation unit is a dissolved gas flotation unit combined together with an induced gas flotation unit. This unit works on the principle of the recycle flow pressure flotation.

The major components of the FCFU are as follows:

- Four chamber gas flotation Tank
- Gas dissolved reactor (GDR)
- GDR feed pump
- Sludge tank
- Skim oil discharge pump

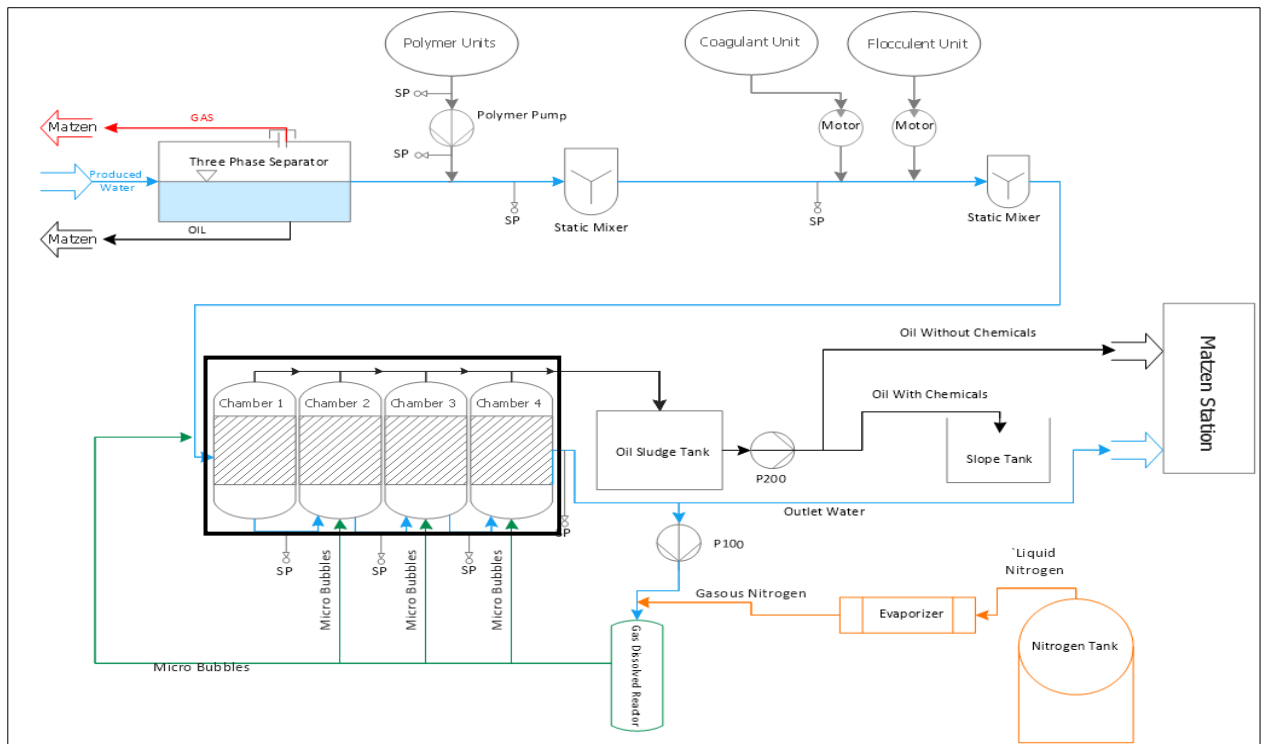


Figure 5-1: PFD of Four Chamber Flotation Unit

The tank is the most important part of the unit because the process of oil-water separation occurs in it. The tank is divided into four consecutively connected chambers which are all similarly designed for giving enough residence time for good oil-water separation efficiency.

GDR is responsible to produce micro-bubbles (size of 5 – 50 microns), approximately a third of the treated water is pumped through the feed pump P100 into the GDR. Nitrogen gas inlet is supplied to the discharge of the GDR, feed pump to generate a stream of water containing micro-bubbles (milky water).

The produced water entering the tank at the first chamber is mixed the micro-bubbles generated by the GDR. The mixture of oily water and gas bubbles causes the oil droplets to adhere, which makes the mixture floating up to the surface. After the bubbles reach the surface, the bubble burst leaves the oil on the surface of the water. The floating oil flows into the oil tank through notches at the end of the chamber by the hydraulic movement generated within the tank. The outlet water from chamber one flows from an opening into the next chamber and is mixed with another portion of the micro-bubbles to clean the remaining oil. A repetition of the same process and procedure occurs inside all other three chambers. A clean water exits the fourth chamber as an outlet.

The flow of the oil after the FCFU depends on whether chemicals were added or not. If chemicals were added and sludge is formed, oil pump P200 is going to pump the oil into the slop tank which is going to be taken by special trucks to be delivered to sludge treatment station. If no chemicals were added then P200 is going to pump the oil into the oil line which goes to the gathering station Matzen.

As was shown in Figure 5-1, there are different sampling points: at the polymer unit, chemical dosing unit and in addition there is also a sampling points at the inlet and outlet of each chamber.

5.2 WTP

The OMV water treatment plant (WTP) has three water treatment steps:

- Parallel plate separators (PPS)
- Flotation unit
- Nutshell filter

At the inlet the water contains 300 ppm residual OIW and is firstly going through five PPS. Due to the density difference the oil rises to the top of the surface. In each separator there are laminate plates which are closely packed that called Lamellae. These lamellae provide a surface on which the oil droplets can accumulate and increase in size then rise up to the surface. The accumulated oil is removed from the PPS and pumped through pipes for further treatments. The treated water has now approximately 150 ppm OIW and is transferred to the second stage of the treatment.

After the first treatment step the water is treated within the flotation unit which consists of a coagulation reactor, a flocculation vessel, a dissolved gas flotation cell and a gas dissolving reactor. Coagulant and flocculent are chemicals that are added to destabilize and agglomerate the particles into larger flocs. The purpose of the coagulation reactor is to give the coagulant enough time to react with the oil particles and to form micro-flocs. By adding the flocculation agent, the micro-flocs are agglomerated together to form floatable flocs that takes place in the flocculation unit. Mixing of water. The floatable flocs enter then the flotation cells where nitrogen is added to the mix so that the flakes are attached to the gas bubbles and float to the surface. Sludge is formed at the surface of each cell and is removed by a rotating and periodically controlled reamer and pumped into the sludge treatment plant section. The OIW after this stage is 20 ppm. [50, p. 5]

The third and the last stage is the filtration process. The water enters the nutshells filter vessel from the upper part to get in contact with walnut shells and flow into the walnut from up to downwards. The walnuts then absorb the remaining oil particles in the water and bind them. These walnuts can be washed out of the oil which gives them a long operational lifetime. The process of removing the oil out of the walnuts called back-wash process. This process can be done by closing the inlet and the outlet valve of the filter. The nutshell pump will force the remaining fluid to flow from down to upwards in closed circle movement inside the filter to separate the suspended oil from the walnuts. This procedure continues for several minutes depending on the oil-in-water concentration. The OIW after this stage is 2 ppm.

5.3 Flotation Cells Size and Principle

Flotation cells in the WTP work on the principle of DGF which is based on Henry's Law (2.1.1.1), where gas and liquid phase are under pressure. FCFU depends also on the same process and in addition also on IGF process which uses mechanical forces such as shear and impaction.

WTP: The flotation cells in WTP as shown in Figure 5-2 have U shaped plates which are the heart of the flotation. The spiral scoop is used for mechanical collection and removal of the sludge to transfer it to the sludge outlet pipe before sending it to the sludge treatment plant. The volume of each flotation cell is 80 m³.

The principle of the cells is, that the inlet water is mixed with the pressurized water containing the nitrogen micro-bubbles. This is uniformly distributed beneath the U-shaped elements. After mixing the water with flocculants and with the bubbles the water rises up between the U elements which cause the separation of the suspended matter from the water for the first time. This process depends on the principle of parallel flow lamella clarification. The water and the sludge rise together to the surface. After they reach the surface, the floated sludge layer is separated from the water layer and rises to the surface while the water flows down inside the shaped elements. The suspended water is separated again by the principle of counterflow lamellar clarification. The clarified water is collected at the bottom of the cell and the floated sludge is removed by the spiral scoop. [51, pp. 10-11]

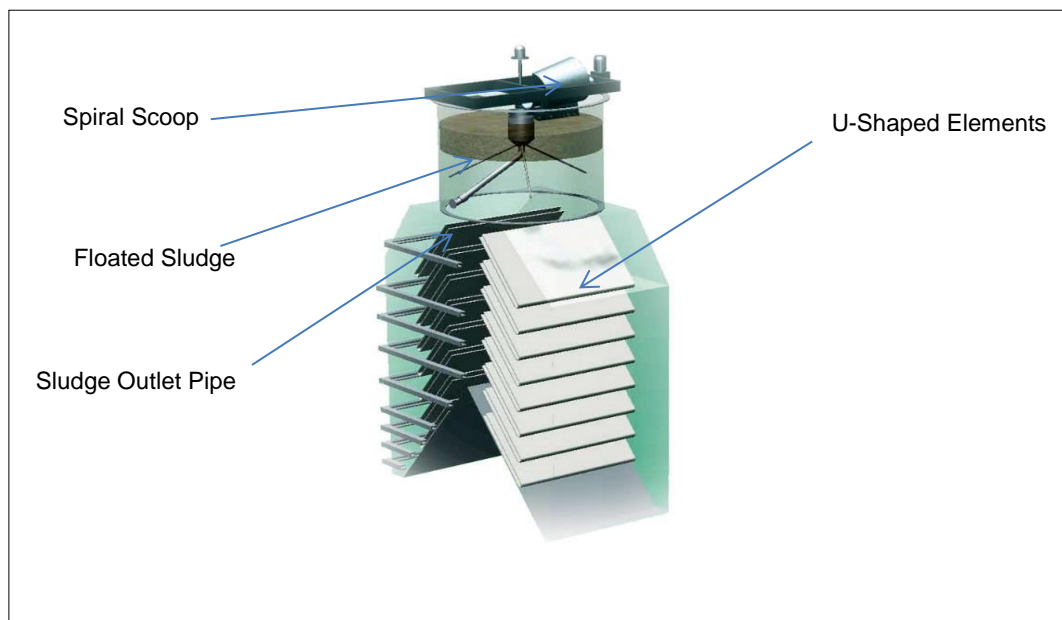


Figure 5-2: Schematic of WTP Flotation Cell [52]

FCFU: The computational fluid dynamics is a new technique for observing all the fluids within a vessel or tank. As shown in Figure 5-3, chamber one has the highest inlet in compared to the other chambers. The reason is that the first chamber is also the first inlet point into the tank. This means that most of the micro-bubbles are going to be injected into chamber 1, which leads to getting out most of the oil particles from the water in chamber 1.

The circular movement of the water in the chambers as shown in Figure 5-3 gives two advantages of FCFU to be considered compared to the DGF used in the WTP:

- FCFU doesn't need any coagulation tank because the circular movement gives enough time for the chemicals to react with the gas bubbles and the oil droplets.
- The hydraulic movement of the fluid to the front side of the chambers gives an automated system to remove the sludge and the accumulative oil out of the chamber into the sludge tank. This means FCFU doesn't need any mechanical equipment to remove the sludge. [53, p. 8]

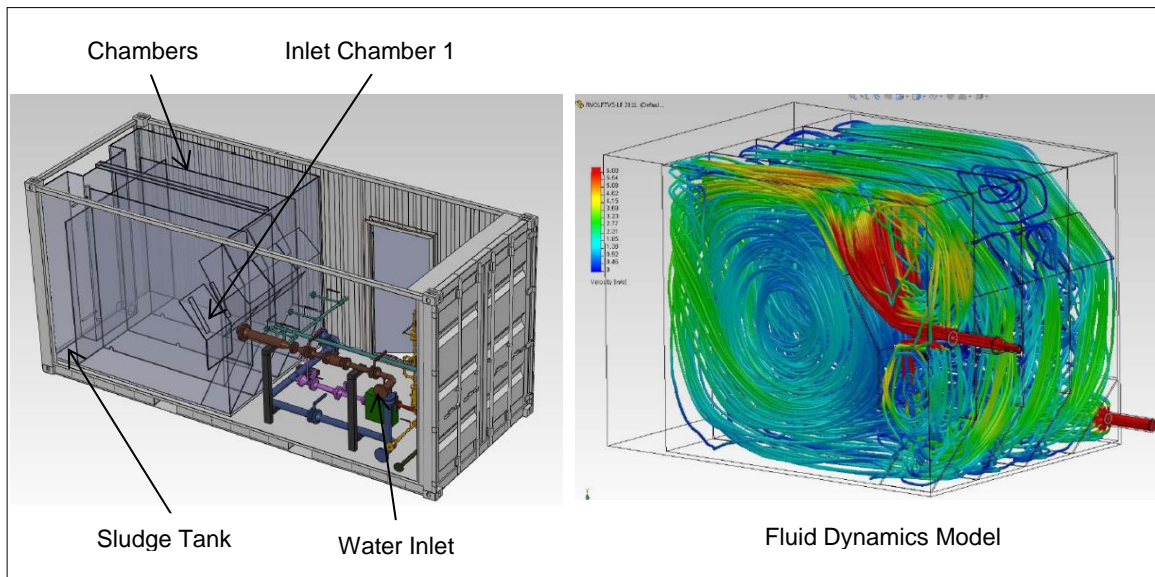


Figure 5-3: Schematic of the Equipment & Computational Fluid Dynamics of the Four-Chamber Flotation Unit [53, p. 8]

5.4 Gas Dissolving Reactor (GDR)

Gas dissolving reactor uses the principle of Henry's law which was explained in the sub-chapter (2.1.1.1), the law says that the gas solubility is proportional to the pressure, the higher the pressure the greater the solubility of air in water. GDR works on the same principle of recycle flow pressure which was explained in sub-chapter (2.1.2.2), part of the clarified water is recycled by means of the pressurization system. This water is pressurized with a sufficient volume of air; the air is dissolved in water under pressure and this saturated water is transferred back under atmospheric pressure via a pressure relief valve. According to Henry's law, the solubility of air in water is much lower at atmospheric pressure than at higher pressure. This leads that air is immediately released in the form of micro-bubbles. These micro-bubbles give a white appearance of the water (the so called "white water").

WTP has one gas dissolving reactor for each flotation cell. The reactor is called GDR120, whereby the number reflects the maximum flow rate in m³/h.

FCFU has one GDR which is a pressure vessel that uses DAF as well as IGF system by using hydraulic flow, shear, impact and pressure to create micro gas bubbles which are smaller bubbles in compare to the bubbles compared by the GDR in WTP

5.5 Sludge Removal and Skimming

The difference in sludge removal between the dissolved gas flotation used in the WTP and the four chamber flotation unit is the mechanism method. In WTP there is a mechanical device (Spiral Scoop) at the surface of each flotation cell, which removes the sludge from the flotation cell. In FCFU there are no mechanical devices inside the flotation unit. The sludge is removed through the water movement of each chamber.

The sludge is removed by one rotation of the mechanical device every 130 minutes in the WTP, each rotation of the device needs 3 minutes.

The skimming of the FCFU depends on the flow rate. The skimming duration for the conventional water with 13 m³/h is two minutes and five minutes for non-skimming. The skimming duration for the back-produced polymer water with 1.2 m³/h is six minutes and thirty minutes for non-skimming.

The details of the skimming optimization and calculation for FCFU can be found in subchapter (6.1).

5.6 Retention Time

Retention time gives the amount of time a liquid stays in a vessel. In other words, retention time is the time that chemicals need to react with oil and gas bubbles. Retention time can be calculated by dividing the liquid volume inside the vessel or the tank by the liquid flow rate as shown in **Eq.10**. [54]

$$\text{Retention time [min]} = \frac{\text{Volume [m}^3\text{]}}{\text{Flow rate [m}^3\text{/h]}} * 60 \quad (10)$$

WTP: retention time should be calculated as the sum of the time in the coagulation tank, flocculation tanks and in the flotation cells.

Five coagulation tanks: each tank has a liquid volume of 34 m³, the flow rate of 930 m³/h, three tanks are operated in the parallel mode for the coagulant and two tanks for the flocculent.

Each flotation cell has a liquid volume of 80 m³, the flow rate of 232 m³/h and in addition the recirculation rate of 80 m³/h from the GDR.

$$\text{Coagulant retention time [min]} = \frac{34 \text{ [m}^3\text{]} \times 3}{930 \text{ [m}^3\text{/h]}} * 60 = 6.5 \quad (10a)$$

$$\text{Flocculent retention time [min]} = \frac{34 \text{ [m}^3\text{]} \times 2}{930 \text{ [m}^3\text{/h]}} * 60 = 4.3 \quad (10b)$$

$$\text{Flotation cell retention time [min]} = \frac{80 \text{ [m}^3\text{]}}{(232 + 80) \text{ [m}^3\text{/h]}} * 60 = 15 \quad (10c)$$

OWT: due to the high difference in flow rate between WTP and any pilot units. A smaller scale pilot unit was built that the performance comparison with any other pilot plant with low flow rate can be easily done. This unit has the same treatment stages as WTP and it was built in the metering station Schönkirchen V. The unit called OWT (OMV Water-Treatment Trial) as was shown in Figure 3-1. The flow rate of the formation water of the OWT is 5 m³/h.

With a small flow rate of 5 m³/h the retention time inside the coagulant tank is 3.5 min and the retention time inside the flotation cell is 7.5 min. [50, p. 5]

FCFU: another advantage is the longer retention time because it is calculated as the total from all four chambers. This gives more time for the reaction of the bubbles with the oil droplets. Each chamber has a volume of 2.44 m³. The recirculating flow rate is divided into 50% into chamber 1, 25% into chamber 2, 15% chamber 3 and 10% into chamber 4. It is explained in details in the sub-chapter (6.2).

The flow rate is 13 m³/h in all chambers, recirculation rate is 30% of the whole rate which is in this case 3.9 m³/h. An important advantage of FCFU over WTP is that FCFU has a GDR that produces just after it reaches its operation point which is 12.5 m³/h or 55 gallons per minute (GPM). This means that GDR doesn't depend on the rate which it was the case in the WTP. As shown in Table 5-1, the total retention time in all chambers with 13 m³/h is almost 26 minutes.

Table 5-1: Retention Time Calculation of FCFU with a Flow Rate of 13 m³/h

Chamber	First	Second	Third	Fourth	Total
Main flow rate (m ³ /h)	13	13	13	13	13
Micro-bubble flow rate (m ³ /h)	6.2	3.1	1.9	1.2	12.5
Total flow (m ³ /h)	19.2	22.4	24.2	25.5	25.5
Volume (m ³)	2.4	2.4	2.4	2.4	2.4
Retention time (min)	7.5	6.5	6	5.7	25.7

The volume of the WTP flotation cell is 80 m³ and the flow rate is 232 m³/h. By dividing the volume by the rate, the result is one to third, this ratio gives a retention time of 15 minutes. This will makes the total retention time 26 minutes that includes the time in the flotation cells, coagulation tanks and flocculation tanks.

The ratio in the FCFU of the four chambers is 10:13 that gives a retention time of 25 minutes and without the need for a coagulation tank. If the flow rate was increased to 30 m³/h, the ratio of the volume over the rate becomes 10:30, which reflects the same ratio as in the WTP. The retention time is going to be 15 minutes as shown in Table 5-2.

Table 5-2: Retention Time Calculation of FCFU with a Flow Rate of 30 m³/h

Chamber	First	Second	Third	Fourth	Total
Main flow rate (m ³ /h)	30	30	30	30	30
Micro-bubble flow rate (m ³ /h)	6.2	3.1	1.9	1.2	12.5
Total flow (m ³ /h)	36.2	39.4	41.2	42.5	42.5
Volume (m ³)	2.4	2.4	2.4	2.4	2.4
Retention time (min)	4	3.7	3.5	3.4	14.6

Retention time only gives an important indication that the multi-chamber system is much better than single flotation cell. The first indication is with the same amount of retention time, the efficiency is higher. The second indication is that there is no need for any chemicals in the treatment of conventional water which is a cost saving issue.

Table 5-3: Retention Time Calculation of FCFU with a Flow Rate of 1.2 m³/h

Chamber	First	Second	Third	Fourth	Total
Main flow rate (m ³ /h)	1.2	1.2	1.2	1.2	1.2
Micro-bubble flow rate (m ³ /h)	6.2	3.1	1.9	1.2	12.5
Total flow (m ³ /h)	7.4	10.6	12.4	13.7	13.7
Volume (m ³)	2.4	2.4	2.4	2.4	2.4
Retention time (min)	19.5	13.7	11.7	10.6	14.6

Table 5-3 shows that with low flow rate the retention time in the FCFU is increased up to approximately 56 minutes which gives the reason why the FCFU works better with low flow rates.

5.7 Nitrogen Consumption

In the flotation processes nitrogen is used as flotation gas because the usage reduces corrosion problems and possible hydrocarbon degradation caused by the use of air.

WTP: the nitrogen consumption for the flow rate of 232 m³/h for each flotation cell is 2.1 m³/h. Each 1 m³/h water needs 0.0095 m³/h nitrogen which can be calculated by dividing the flow rates of water over the nitrogen. The nitrogen pressure is 7.8 bar.

FCFU: the multi-chamber flotation consumes the same amount of nitrogen and is not dependent on the water flow rate. Because the GDR in this system is dependent on the recycled flow from the outlet. The multi-chamber flotation nitrogen consumption is around 2.1 m³/h by a pressure of 10 bar.

5.8 Oil-In-Water Inlet and Outlet Conditions of the Flotation Cells

WTP: The WTP treats 930 m³/h in all treatment stages; whereby each flotation cell treats 232 m³/h. The flotation inlet has an OIW of 170 – 200 ppm. If the operation works properly the outlet has a quality of 30 ppm and if fluctuations come up to about 80-100 ppm. There are some reasons for the variation in performance which are internal influences like the difference in chemical concentration, high oil concentration at the inlet, problems with skimming operation, any mechanical failure in one of the flotation cells and the size of the flocs which says that the smaller the sizes of the flocs the worse is the removal efficiency which was confirmed by the lab results. There is also external influencer like corrosion inhibitors which can reduce the performance of the flotation cell.

FCFU: The OIW inlet in average is 960 ppm, which was with a flow rate of 13 m³/h. The OIW outlet is 1.8 ppm in its best performance which was with chemicals. The OIW outlet without chemicals is around 70 ppm. The result from FCFU cannot be directly compared with the one from WTP because of the high difference in the flow rate and the difference in volume of the cells. The results were therefore compared with the unit of OWT as it was explained in the sub-chapter (5.6).

5.9 Water Treatment Comparison

The comparison between both systems in terms of removal oil out of the water. Two different types of water were used, which are conventional water and back-produced polymer water. Both waters were treated with and without chemicals.

5.9.1 Conventional Water treatment without chemicals

The details of the wells are shown in the sub-chapter (3.1)

$$\text{Removal Efficiency [\%]} = \frac{\text{Inlet} - \text{outlet flotation}}{\text{Inlet}} \quad (11)$$

Eq.11 shows the calculation of the removal efficiency. This equation basically calculates the percentage of oil that has been removed from the water.

The results shown in Table 5-4 & Table 5-5 indicates that FCFU has three advantages compared to the OWT plant in the treatment of conventional water (CW):

1. OWT consists of two treatment stages (parallel plate separator and flotation unit) whereby the removal efficiency was lower than with FCFU. The reason is that OWT has one chamber whereby FCFU has four chambers that lead to increasing the efficiency. The other reason is also that the calculation of the removal efficiency dependent on the inlet quality as shown in **Eq.11**, which was in this case lower in OWT than in FCFU.
2. Although the flow rate in FCFU was double than the flow rate in OWT regarding the smaller volume of the flotation tanks in OWT but the removal efficiency was higher in FCFU.
3. The OIW at the inlet side of FCFU was more than the double of the OWT, but the outlet in FCFU is lower than OWT.

Table 5-4: OWT CW Treatment without Chemicals for a Flow Rate of 5 m³/h [12, p. 16]

Wells (S110, S135, Ma108)	Inlet Separator (ppm)	Inlet PPA (ppm)	Outlet PPA (ppm)	Outlet Flotation (ppm)	Outlet NSF (ppm)	Removal Efficiency (%)
	420	250	155	140	N/A	67

Table 5-5: FCFU CW Treatment without Chemicals for a Flow Rate of 13 m³/h

Wells (S79, S109, S110 S126, S135, Ma108)	Inlet (ppm)	Chamber 1 (ppm)	Chamber 2 (ppm)	Chamber 3 (ppm)	Outlet (ppm)	Removal Efficiency (%)
	961	340	133	113	96	90

Another important advantage about FCFU is the ability to treat high oil loads (> 3,000 ppm) from conventional water without chemicals as shown in Table 5-6.

Table 5-6: FCFU CW Treatment for High OIW without Chemicals for a Flow Rate of 13 m³/h

Wells (S66,S79, S96, S109, S126, S135, Ma108)	Inlet (ppm)	Chamber 1 (ppm)	Chamber 2 (ppm)	Chamber 3 (ppm)	Outlet (ppm)	Removal Efficiency (%)
	2120	280	142	114	92	96
	2823	244	134	96	80	97
	3155	228	131	103	98	97

5.9.2 Conventional Water Treatment with Set Vb

The amount of chemicals used for the OWT were 100 ppm coagulant (80% Pluspac and 20% Floquat) and 0.6 ppm flocculent (Chimec 5565) as shown in Table 5-7 with this amount of chemicals OWT unit achieved a removal efficiency of 95%.

Although just one test was done with set Vb for the treatment of conventional water for FCFU and the reason was that FCFU has already achieved a high removal efficiency without the need for chemicals as shown in

Table 5-5. Therefore just 15 ppm coagulant (100% Floquat) was used to treat the conventional water with FCFU as shown in Table 5-8. If the number of chemicals would be increased to 30 ppm or 40 ppm, the results at the outlet of FCFU might be below 5 ppm.

Table 5-7: OWT CW Treatment with Set Vb for a Flow Rate of 5 m³/h [12, p. 16]

Wells (S110, S135)	Inlet Separator (ppm)	Inlet PPA (ppm)	Outlet PPA (ppm)	Outlet Flotation (ppm)	Outlet NSF (ppm)	Removal Efficiency (%)
	490	280	140	20	1	96

Table 5-8: FCFU CW Treatment with Set Vb for a Flow Rate of 13 m³/h

Wells (S79, S109, S110, S126, S135, Ma108)	Inlet (ppm)	Chamber 1 (ppm)	Chamber 2 (ppm)	Chamber 3 (ppm)	Outlet (ppm)	Removal Efficiency (%)
	930	250	160	100	63	93

In order to test the above mentioned assumption of FCFU in the treatment of conventional water, another type of chemical was used for the treatment of conventional water which is so-called geochemical with a concentration of 30 ppm as shown in Table 5-9. The efficiency increased to almost 100% with a slight increase in chemical amount. This indicates that FCFU needs 30% of the chemicals that OWT needed to treat conventional water.

Table 5-9: FCFU CW Treatment with Geochemical for a Flow Rate of 13 m³/h

Wells (S79, S109, S110, S135, Ma108)	Inlet (ppm)	Chamber 1 (ppm)	Chamber 2 (ppm)	Chamber 3 (ppm)	Outlet (ppm)	Removal Efficiency (%)
	1150	100	25	5	1.8	99.8

FCFU shows better results than OWT after taking into consideration all these tests with conventional water. FCFU consumes 30% of the amount of coagulant that was used with OWT. FCFU doesn't use any flocculent in the treatment of conventional water which is not the case with OWT that consumes 0.6 ppm flocculent. The main reason that gives these advantages of FCFU over OWT is the retention time and the design of the four chambers. The design of the four chambers increased the probability of contact between oil droplets and gas bubbles which leads higher removal efficiency with fewer amounts of chemicals.

5.9.3 Treatment of Back-Produced Polymer Water (BPPW)

WTP: the current technology in the WTP cannot treat high amounts of back-produced polymer water (BPPW). The current amounts of the injected polymer are still low, this can be indicated by the amount of the back-produced polymer that comes from well S66. This means that the low concentration of the back-produced polymer will be almost zero comparing to the amount of the conventional water that is treated in WTP. This can give the reason why the back-produced polymer with such a low concentration compared to the conventional water has no influence on the treatment stages of WTP and can be treated with the current technology in WTP.

OWT and FCFU used Set Vb to treat different amounts of back-produced polymer (HPAM). The polymer which was injected is FS3630 (4.1.1).

5.9.3.1 Without Chemicals

- As was shown in Table 5-4, OWT cannot treat the conventional water without the use of chemicals, which points out that OWT cannot treat back-produced polymer water without chemicals. Therefore no tests were performed without chemicals in the treatment of BPPW.
- The back-produced polymer water used in the trial with FCFU was coming from the polymer well S66, the flow rate is 1.2 m³/h.

Table 5-10 shows different tests of the back-produced polymer well S66 with different amount of polymer concentration which was treated without chemicals. The results show that the removal efficiency decrease with increase in polymer concentration.

Table 5-10: FCFU BPPW Treatment without Chemicals for a Flow Rate of 1.2 m³/h

HPAM Content	Inlet (ppm)	Chamber 1 (ppm)	Chamber 2 (ppm)	Chamber 3 (ppm)	Outlet (ppm)	Removal Efficiency (%)
21 ppm (S66)	44	22	16	15	14	68
44 ppm (S66)	101	45	38	37	36	64
47 ppm (S66)	75	72	67	54	48	36

5.9.3.2 With Set Vb

The amount of chemicals used for the trials performed with OWT was 100 ppm coagulant (80% Pluspac and 20% Floquat) and 0.6 ppm flocculent (Chimec 5565). As shown in Table 5-11, with this amount of chemicals achieved the OWT unit a removal efficiency above 99% in the treatment of back-produced polymer water up to 30 ppm concentration of HPAM. [12, p. 16]

Table 5-12 summarizes the results of different tests for the treatment of BPPW with FCFU. Different polymer concentrations were tested with different amounts of chemicals. The first formation water which was with 16 ppm HPAM was treated with 25 ppm coagulant and 0.3 ppm flocculent. The second one which has 19 ppm HPAM was treated with 40 ppm coagulant and 0.4 ppm while the last one which has 20 ppm HPAM was tested with 50 ppm coagulant and 0.5 ppm flocculent.

From the results (Table 5-12) can be seen that the number of chemicals used for the trial with 20 ppm HPAM was too high because the results with 40 ppm coagulant and 0.4 ppm flocculent were satisfied where the outlet was 3.6 ppm OIW.

Table 5-11: OWT BPPW Treatment with Set Vb for a Flow Rate of 5 m³/h [12, p. 16]

HPAM Content	Inlet Separator (ppm)	Inlet PPA (ppm)	Outlet PPA (ppm)	Outlet Flotation (ppm)	Outlet NSF (ppm)	Removal Efficiency (%)
6 ppm (S95, S135)	540	190	90	25	3	99.4
20 ppm (S66, S110, S135)	520	180	100	2	1	99.6
30 ppm (S66, S95, S135)	500	230	110	2	1	99.6

Table 5-12: FCFU BPPW Treatment with Set Vb for a Flow Rate of 1.2 m³/h

HPAM Content	Inlet (ppm)	Chamber 1 (ppm)	Chamber 2 (ppm)	Chamber 3 (ppm)	Outlet (ppm)	Removal Efficiency (%)
16 ppm (S66)	51	15	10	4.8	4	92
19 ppm (S66)	55	40	20	4	3.6	93
20 ppm (S66)	33	5	4	2	2	94

6 Operational Challenges of the FCFU

There are different challenges while operating the FCFU. The first challenge is skim rate optimization for every single change that was made during the test like changing the flow rate, increasing the number of chemicals and introduce polymer into the system. The second challenge is to distribute the micro-bubbles in the best systematic way that the removal efficiency in all chambers is satisfied. The final challenge is to know the optimal amount of nitrogen.

6.1 Skim Rate Optimization and Calculation

Optimization

FCFU was operated in batch mode, which means that the batch parameters have to be set in the set-up page. There are different parameters which should be taken into consideration to optimize the skim rate and these parameters are shown in Figure 6-1. For example minimum level, maximum level, frequency timer (no skimming) and duration timer (skimming). The duration or the skimming (seconds) means that this is the time where the water level will rise until it reaches the set point of the maximum level where it stays constant. The frequency means that this is the time that the level will decrease until it reaches the minimum level and stays constant until the skimming starts. The duration and frequency together make up one skim cycle.

The challenge in the skim rate optimization is to find the optimum parameters for each flow rate. The optimum parameters are reached when first oil is skimmed into the skim tank and when no oil accumulation in any chambers. If there is any accumulation the outlet quality of the water will be affected.

The frequency and the duration are dependent in addition to the flow rate also on the amounts of chemicals and polymers that were used during the test.

FEED PUMP VALVE/VFD 4mA SETTING (MIN OUTPUT)	NNNN.N	BATCH MODE MINIMUM INLET WATER FLOW	NNNN.N
FEED PUMP VALVE/VFD 20mA SETTING (MAX OUTPUT)	NNNN.N	BATCH MODE ENABLE/DISABLE DELAY TIMER (SEC)	NNNN
NORMAL SKIM MAXIMUM WATER LEVEL (mm)	NNNN.N	BATCH MODE MINIMUM WATER LEVEL (mm)	NNNN.N
NORMAL SKIM FREQUENCY TIMER (SEC)	NNNN	BATCH MODE MAXIMUM WATER LEVEL (mm)	NNNN.N
NORMAL SKIM DURATION TIMER (SEC)	NNNN	BATCH MODE FREQUENCY TIMER (SEC)	NNNN
		BATCH MODE DURATION TIMER (SEC)	NNNN
BACK	PANELVIEW CONFIGURE		DEVICE BYPASS
MAIN	START SEQUENCE	INPUTS & OUTPUTS	PID CONTROL
			PROCESS OVERVIEW
			ACTIVE ALARMS

Figure 6-1: Screenshot of the Skim Parameters Set-up [55, p. 39]

In some cases there is good skimming in one chamber but the others are skimming badly. The reason behind these cases is the polymer and chemicals, which lead to form sludge that can be skimmed well from one or two chambers. Each chamber have a different amount of notches, chamber 1 has the high amount of notches in comparing to the other chambers. This situation leads us to the fact of using additional parts, especially in chamber 1 and 2 to ensure that all chambers have the good skimming. These additional parts are called caps and sit at the end of the chambers. Figure 6-2 shows two different styles of the caps, which were used to optimize the skim rate. Both of them can be used at the same time, one set at chamber 1 and the second one set at chamber 2 or vice versa, depending on the conditions of the water.

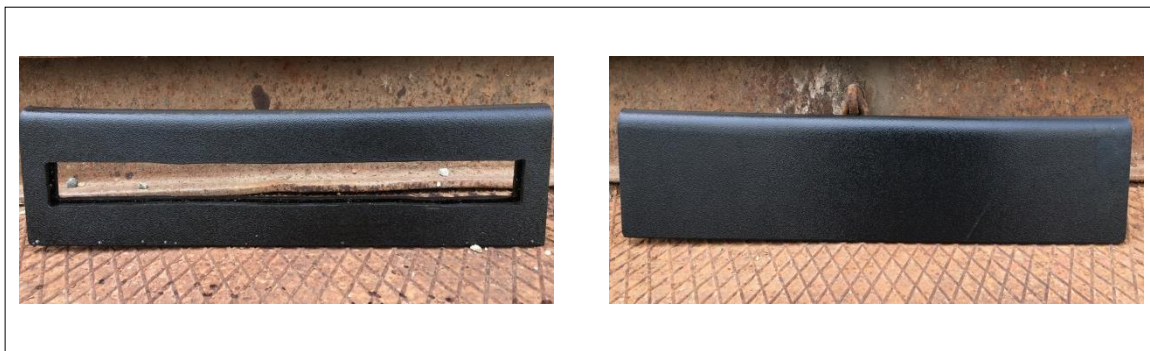


Figure 6-2: Caps used for Skim Rate Optimization

Calculation

The skim rate calculation is based on the volume of the skim tank and the flow rate of the formation water.

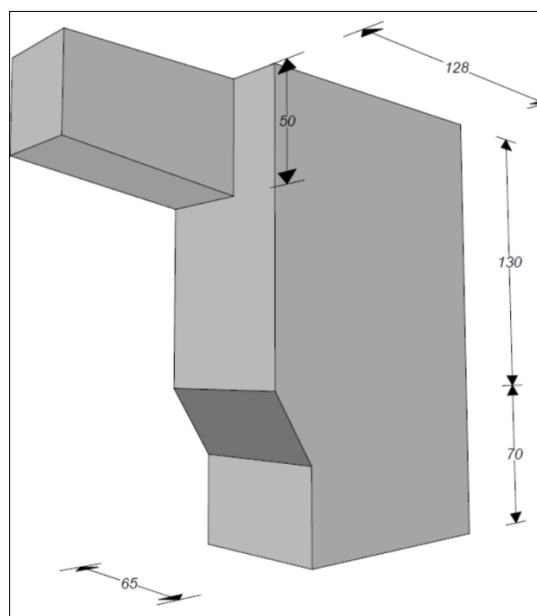


Figure 6-3: Schematic of the FCFU Sludge Tank; Dimensions are in Centimeters

To simplify the calculation of the skim rate, the straight section was used to calculate the skim volume. As shown in Figure 6-3, the straight section starts at 0.7 m from the bottom and ends at a height of 1.5 m.

The real dimensions of the straight section are 0.8 m (height), 1.28 m (width) and 0.65 m (depth). This makes the total volume of the straight section of the skim tank 0.66 m³.

Eq.12 shows the way to calculate the skim rate

$$\text{Actual Height}(AH)[m] = \text{End level}[m] - \text{start Level}[m] \quad (12)$$

$$\text{Skim Cycle}[h] = \text{Duration (Skimming)} + \text{Frequecny (Non Skimming)} \quad (12a)$$

$$\text{Skim Volume}[m^3] = AH * \text{Width}(1.28m) * \text{depth}(0.65m) \quad (12b)$$

$$\text{Skim rate}[\%] = \left(\frac{\left(\frac{\text{Skim Volume}[m^3]}{\text{Skim cycle}[h]} \right)}{\left(\text{Flowrate} \left[\frac{m^3}{h} \right] \right)} \right) * 100 \quad (12c)$$

6.2 Micro-Bubbles Distribution

The key part of this new technology is that the separation efficiency is mostly dependent on the size and the quantity of the micro-bubbles (MB). The MB is distributed in all chambers in different percentages, 50% go into chamber 1, where the most of the separation occurs. 25% go into chamber 2, 15% into chamber 3 and 10% into chamber 4 which is the outlet of the unit.

The optimal amount of flow inside the chambers according to the manufacturer is 12.5 m³/h inside the chambers. In the following this procedure for adjusting the MB distribution can be found:

1. The MB hand valve (HV) of chamber 1 should be open until the flow indicator shows 12.5 m³/h.
2. The amount of the flow rate into chamber 1 should be decreased down to 11.25 m³/h. The HV of chamber 4 is rotating in the open direction until the flow indicator shows 12.5 m³/h is reached. This means that 1.25 m³/h is injected into chamber 4 which is the 10%.
3. Slightly the HV of chamber 1 is rotating to the closing side, until the flow indicator shows 10.63 m³/h, then the HV of chamber 3 until 12.5 m³/h is shown on the flow indicator. This means that 1.87 m³/h which is 15% are injected into chamber 3.
4. Final rotation of the HV of chamber 1 to the closing side, until the flow indicator shows 9.37 m³/h. The HV of chamber 2 is open until 12.5 m³/h is shown on the flow indicator. This means that 3.13 m³/h, which is 25% is injected into chamber 2.

With this method the MB can be distributed at any percentages in the chambers.

6.3 Turbulence (Waves)

If the nitrogen amount is increased to a certain number it causes a turbulent flow or waves at the surface of the chambers, especially in chamber 1 and 2 due to the high amount of MB.

If waves occur it would affect the removing efficiency of the unit because the skimming principle is not going to work very well and will not remove oil.

The challenge is to find the optimum amount of nitrogen that would increase the outlet conditions and at the same time it should be no waves on the surface of the chambers.

7 Experiments: Results and Interpretations

Different types of tests were executed by the FCFU. All the results are explained and discussed in terms of oil-in-water content, total suspended solids and turbidity for the conventional water. In addition to the mentioned parameters, polymer concentration and molecular weight are explained for the back-produced polymer water. While the results regarding sludge analysis are discussed in percent of the separated water, oil, solids and floated sludge.

7.1 Trial 1: Conventional Water with 1.2 m³/h

Different flow rates of the conventional water were tested. The results showed at a lower flow rate, better outlet conditions can be achieved.

7.1.1 Oil-In-Water Content

As shown in Figure 7-1 with such a low flow rate (high retention time) independent how high the oil-in-water concentration was at the inlet, the outlet quality was below 20 ppm (no chemicals were required).

Figure 7-1 shows two different tests, the first one was with 500 ppm OIW and the second one was with 170 ppm OIW. More than 85 % of the OIW is removed by the first chamber for both tests. Other three chambers removed the rest of the OIW. Therefore there were no chemicals needed for the treatment with such a low flow rate. The reason is due to high retention time.

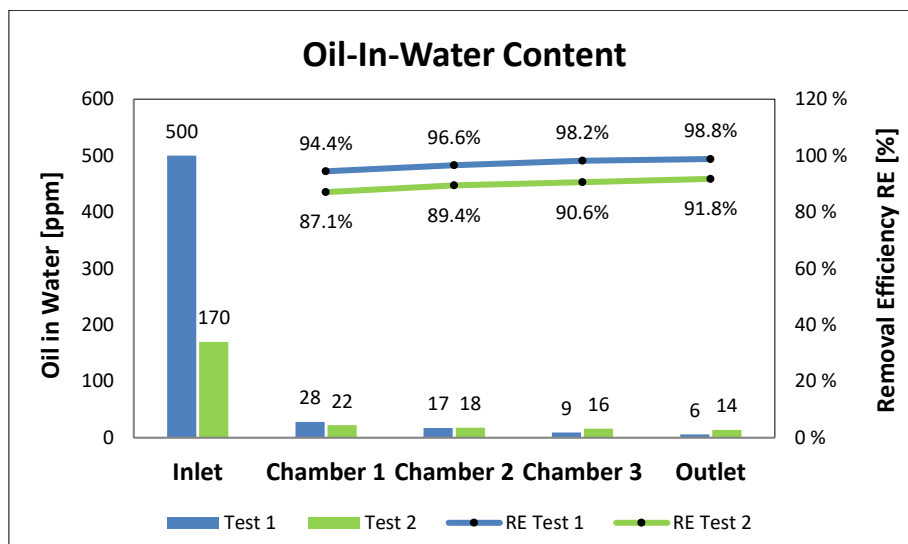


Figure 7-1: Oil-In-Water Concentration and Removal Efficiency at a Flow Rate of 1.2 m³/h. Graph Reflects Two Different Tests

7.1.2 Total Suspended Solids

Figure 7-2 shows that TSS depends on OIW: the higher the OIW the lower the TSS, which indicates why the TSS is almost zero at the outlet side. The trial with a high OIW has a high TSS at the outlet.

- Low OIW: at the inlet was an OIW of 44 ppm and at the outlet 6 ppm.
- High OIW: at the inlet was an OIW of 100 ppm and at the outlet 17 ppm.

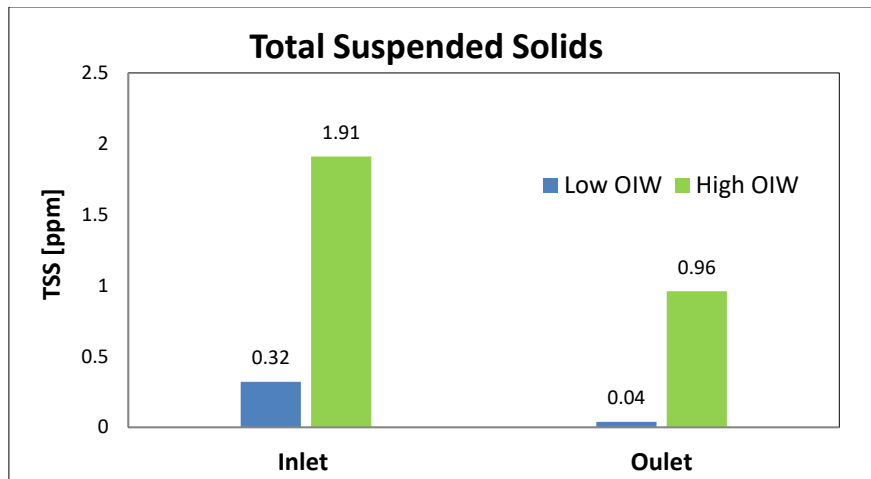


Figure 7-2: Total Suspended Solids at a Flow Rate of 1.2 m³/h comparing Different OIW Concentration

7.1.3 Turbidity

Figure 7-3 shows that turbidity is also dependent on OIW, the higher the OIW the higher the turbidity. The turbidity increased up to 70 NTU with the test of 170 ppm OIW, FCFU decreased the cloudiness of the fluid down to 32 NTU. This means that there is still a lot of individual particles that are invisible to the human eye. For the test with only 40 ppm OIW, the water still has 20 NTU that consider being high.

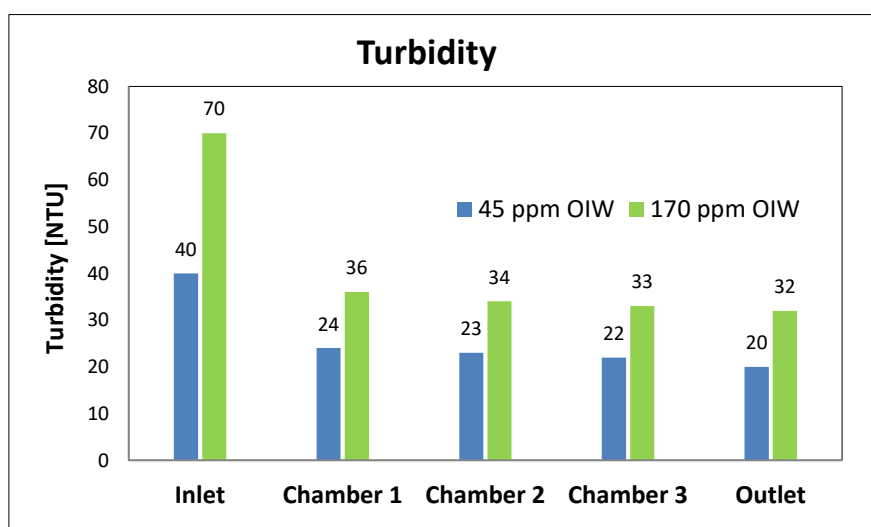


Figure 7-3: Turbidity at a Flow rate of 1.2 m³/h from the Inlet and All Four Chambers at Different OIW Concentration

7.2 Trial 2: Conventional Water with 13 m³/h

With such a flow rate the FCFU has to use chemicals to reach the proper concentration of oil-in-water at the outlet. The geochemical was used to test its removal efficiency of oil with conventional water.

7.2.1 Oil-In-Water Content

From Figure 7-4 two points are observed:

1. For the trial without chemicals at a flow rate of 13 m³/h the FCFU achieved 96 ppm at the outlet. This result consider to be high, therefore the use of chemicals was necessary to see the efficiency of FCFU with chemicals.
2. By adding 30 ppm of the geochemical, the removal efficiency increased to almost 100% and the outlet concentration decreased to below 2 ppm.

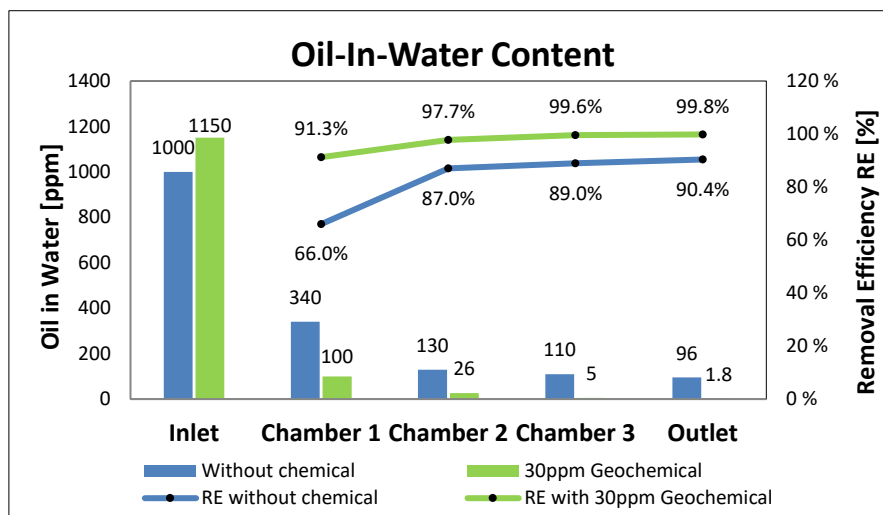


Figure 7-4: Oil-In-Water Concentration and Removal Efficiency at a Flow Rate of 13 m³/h comparing with and without the usage of the Chemicals

7.2.2 Turbidity

Figure 7-5 shows that turbidity decreased from 85 NTU without chemicals to 4 NTU by the use of just 30 ppm geochemical for the treatment of high flow rate. This low amount of chemicals decreased the number of turbidity down to 4 NTU which consider the water to be drinkable. The test without chemicals which has 96 ppm OIW shows a high turbidity of 85 NTU.

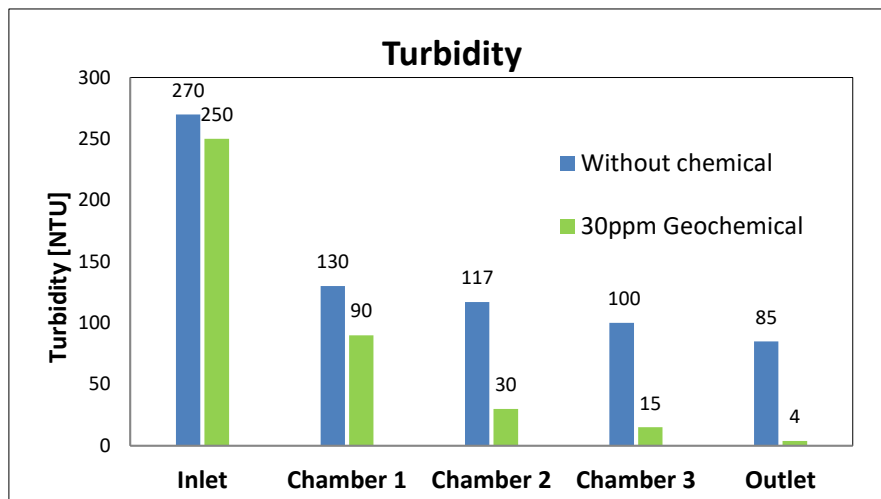


Figure 7-5: Turbidity at a Flow Rate of 13 m³/h from the Inlet and All Four Chambers for the Treatment with and Without Chemicals

7.3 Trial 3: Back-Produced Polymer Water without Chemicals

Two different scenarios were explained with back-produced polymer water, the treatment with and without the need of chemicals.

7.3.1 Oil-In-Water Content

As shown in Figure 7-6, two different HPAM concentrations were tested with almost the same OIW at the inlet. From the results it's obvious that the treatment of the lower HPAM content which is 20 ppm is better, FCFU can treat with such a low flow rate of 1.2 m³/h up to 45 ppm HPAM to remove oil from the water without the need of chemicals.

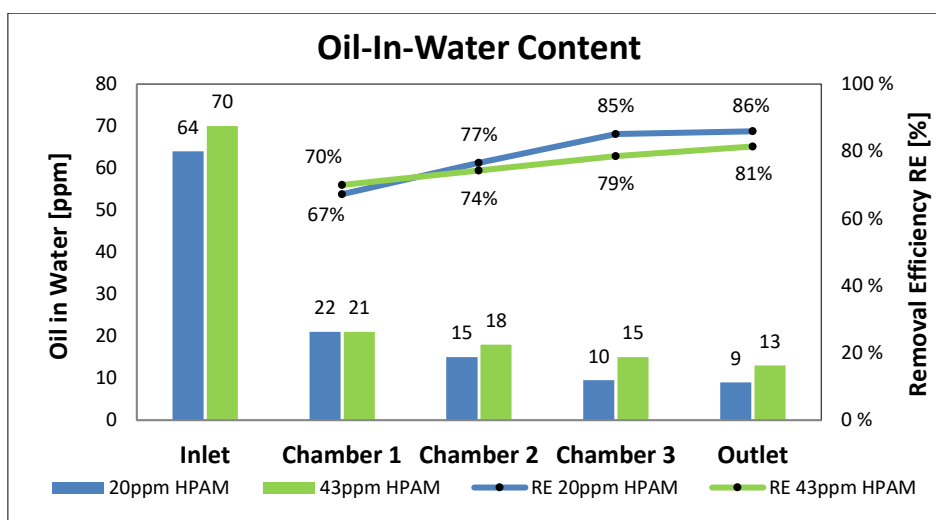


Figure 7-6: Oil-In-Water Concentration and Removal Efficiency with Back-Produced Polymer Water comparing various HPAM Concentrations without the Usage of the Chemicals.

7.4 Trial 4: Back-Produced Polymer Water with Set Vb

7.4.1 Oil-In-Water

Figure 7-7 shows two trials with different amounts of chemicals used for the treatment of the same amount of polymer which was 19 ppm HPAM. The first test was with 50 ppm coagulant and 0.5 ppm flocculent while the second test was with 40 ppm coagulant and 0.4 ppm flocculent. The results show that the outlet conditions are almost the same for the two tests indicating that 40 ppm coagulant and 0.4 ppm flocculent are enough.

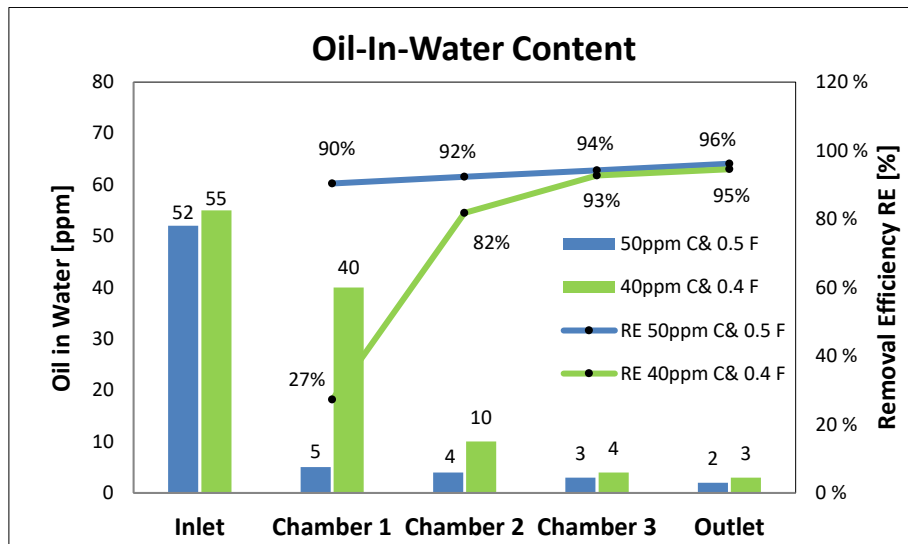


Figure 7-7: Oil-In-Water Concentration and Removal Efficiency with Back-Produced Polymer Water Comparing various HPAM Concentrations with the Usage of Chemicals

7.4.2 Turbidity

Figure 7-8 shows the turbidity results of the treatment of BPPW with and without chemicals. HPAM concentration for both tests was 20ppm.

- The turbidity of both tests was almost the same at the inlet.
- The results showing that chemicals didn't change much, it decreases the turbidity just down to 21 NTU. This result considers being high.

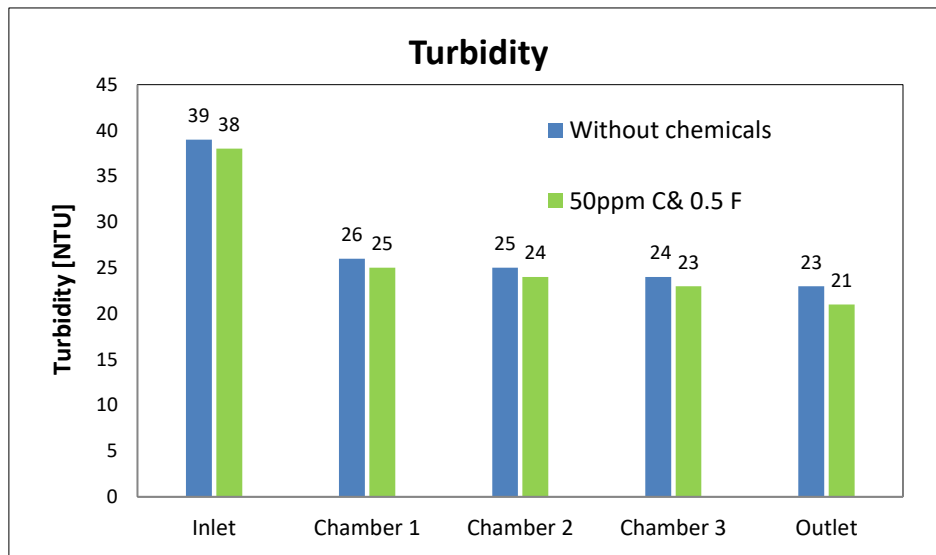


Figure 7-8: Turbidity of the Back-Produced Polymer Water to Treat the Same HPAM Concentration with and without Chemicals

7.5 Sludge Analysis and Interpretations

As shown in Table 7-1, three different samples were analyzed through different experiments. The details of each experiment were shown in (4.3.6).

Different polymer concentration and chemicals for each sample:

- **Sample 1:** the sludge contained 19 ppm polymer concentration (HPAM) and was treated with 50 ppm coagulant and 0.5 ppm flocculent.
- **Sample 2:** the sludge contained 19 ppm polymer concentration (HPAM) and was treated with 40 ppm coagulant and 0.4 ppm flocculent.
- **Sample 3:** the sludge contained 17 ppm polymer concentration (HPAM) and was treated with 25 ppm coagulant without any addition of flocculent.

An ideal sludge composes of water, oil and solids. In the reality there is always some percentage of floated sludge, the existence of the floated sludge has a negative effect on the tricanter which is the treatment unit for the sludge. The reason behind the occurrence of the floated sludge is the existence of polymer. [48, p. 34].

From Table 7-1, can be seen that sample 1 shows better results than sample 2. Although the difference of chemicals is not that much, the results showed that in sample 1 almost 90% of the floated sludge turns into solids while in sample 2 just 40 % did. Another important indication is that sample 3 was not treated with flocculent, which shows that almost none of the floating sludge turns into solids which indicates that the flocculent plays an important role.

Table 7-1: Sludge Results of Three Different Samples

Sample No.	Parameters	Experiment 1	Experiment 2	Experiment 3
1	Solids (Vol %)	1.7	0.2	41.2
	Water (Vol %)	43.5	44	50.6
	Floating Sludge (Vol %)	53	53.8	5.9
	Oil (Vol %)	1.8	2	2.4
	Total (Vol %)	100	100	100
2	Solids (Vol %)	1.7	0.2	35
	Water (Vol %)	43.5	44	44.1
	Floating Sludge (Vol %)	53	53.8	19
	Oil (Vol %)	1.8	2	1.9
	Total (Vol %)	100	100	100
3	Solids (Vol %)	0.2	0.1	1
	Water (Vol %)	48	44	49
	Floating Sludge (Vol %)	40.8	42	39
	Oil (Vol %)	11	13.9	11
	Total (Vol %)	100	100	100

8 Conclusion, Discussion and Future Work

The chapter summarizes the thesis and divided it into three sections: section 1 is a summary of the thesis and section 2 presents a discussion of the results and suggestions for future work. Final section 8.3 concludes the thesis.

Summary of the Thesis

This thesis represents the new technology in oil-water separation which is called four chamber flotation unit that allows a separation of oil from conventional water without the need of chemicals. Through this technology, a lot of costs for the chemicals can be saved. With the back-produced polymer water, this technology approved the use of 30% of chemicals that were used for the normal flotation unit.

Chapter 1 gives a small introduction about the chemical enhanced oil recovery methods. It explains the process of the polymer Flooding. The problem definition and the research objective of this thesis was explained in this chapter.

Chapter 2 gives an introduction to the principles of the flotation technologies and a comparison between the different types of the flotation technologies. The important principles for designing the flotation systems are gas solubility (relationship between solubility and pressure), Stoke's law (law of the settlement of solid particles in liquids), bubble size/rise rate (both dependent on Stoke's law) and air to solid ratio (mass fluxes ratio of air and solids). There are three different types of flotation technologies which are electrolytic flotation, dissolved air flotation and induced air flotation. Through the detailed comparison between all of them, it was approved that dissolved air flotation is the best technology.

Chapter 2 discusses the coagulation and flocculation principle. It discusses also the technologies available in the treatment of back-produced polymer water like viscosity reduction and the combination of flotation technologies. At the end of the chapter, there is an explanation of the four chamber flotation unit technology.

Chapter 3 discuss the components of the plant site. The water comes from the production wells through the three-phase separator to be separated from oil and gas. The water then goes through static mixers and mixed with chemicals if there was any to reach the end its final destination which is the flotation unit.

In Chapter 4, the materials and methods are discussed. Different chemicals were discussed in terms of physical and chemical properties as well as from health and safety point of view. Analytical methods of water and sludge were defined also in this chapter.

Chapter 5 which is the core of this thesis gives a comparison between the four chamber flotation unit and the dissolved flotation unit in terms of different parameters. The mentioned parameters are the flotation cell size, retention time, nitrogen consumption, chemicals consumption and the process of the gas dissolving reactor.

In chapter 6, operational challenges of the four chamber flotation unit were discussed. Three major challenges were dealt with like the skim rate optimization and calculation, the distribution of the micro-bubble and the turbulence flow.

Finally, chapter 7 shows different experiments and interpretations of different trials that depends on the composition of water. Conventional water results were divided into two parts which are the low and high flow rate. Back-produced polymer water results were divided according to the usage of chemicals. The results were shown and discussed in different ways depending on different parameters.

8.1 Discussion

Following points showing the facts in comparing both systems:

- The retention time for treating conventional water and back-produced polymer water in FCFU is longer than in WTP.
- WTP consumes 2.1 m³/h nitrogen for a flow rate of 232 m³/h. FCFU nitrogen consumption doesn't depend on the flow rate, the consumption is always constant and is because of the constant recirculation flow.
- The chemical demand is double as high for the WTP compared to FCFU.
- The Skimming in the WTP lasts three minutes and occurs after 130 min. The skimming for FCFU in the treatment of conventional water is an average two minutes every five minutes. For the treatment of back-produced polymer water the skimming depends on the conditions whether there is chemical involved or not.
- No mechanical units needed for skimming in FCFU, which is not the case with WTP.

These are some points explaining the suggestions for future work

- Test the efficiency of FCFU in the treatment of high concentrations of polymer.
- The size of the micro-bubbles should be tested.
- The results of the turbidity in the treatment of BPPW are not very well. The use of other chemicals set should be under consideration to decrease the turbidity.
- Test the FCFU with a higher flow rate to see its removing efficiency with a lower retention time.
- A coagulation tank and a flocculation tank should be installed before FCFU. This can increase the retention time which can lead to higher removal efficiency.
- Install filters after FCFU to increase the removing efficiency of the oil from the water.
- A shear degradation units like pump or valve will be an excellent pre-treatment phase to degrade the back-produced polymer. This can results also in an increase in the efficiency of FCFU to treat polymer water.

8.2 Conclusion

The achieved results show that FCFU is much better than WTP in the treatment of conventional and back-produced polymer water.

The technology of FCFU depends on the combination of induced gas flotation and dissolved gas flotation. This technology approved that the removal efficiency of oil-in-water increased in comparison with just dissolved gas flotation unit.

FCFU showed promising results in the treatment according to the chemical consumption, ability to treat high oil-in-water concentrations and high retention time. Its system operation is easy, it has an easy handling of the controlling and the operation costs of FCFU consider to be low due to the constant nitrogen consumption for all flow rates.

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Abbreviations

AA	Acrylic Acid
AM	Acryl Amide
BPPW	Back-Produced Polymer Water
CW	Conventional Water
DAF	Dissolved Air Flotation
EF	Electrolytic Flotation
EOR	Enhanced Oil Recovery
EPI-DMA	Epichlorohydrin-Dimethylamine
FCFU	Four Chambers Flotation Unit
GC-FID	Gas Chromatography-Flame ionization detection
GDR	Gas Dissolved Reactor
HPAM	Hydrolyzed Poly Acryl Amide
HV	Hand Valve
IAF	Induced Air Flotation
MB	Micro Bubbles
NTU	Nephelometric Turbidity Units
OIIP	Oil Initially in Place
OIW	Oil In Water
OMV	Österreichische Mineralölverwaltung
OWT	OMV Water Treatment Trial
PFD	Process Flow Diagram
PPM	Parts Per Million
PPS	Parallel Plate Separator
QCL-IR	Quantum Cascade Laser - Infrared
RPM	Revolution Per Minute
SEC	Size Exclusion Chromatography
TPH	Total Petroleum Hydrocarbon
TPS	Three Phase Separator
TSS	Total Suspended Solids
UV-VIS	Ultraviolet – Visible Spectroscopy
WTP	Water Treatment Plant

Appendix B: Back-Produced Polymer Water

Experimental data of the reference period for the treatment of back-produced polymer water with a flow rate of 1.2 m³/h.

Date	Time	Batch	Well Combination	Flow rate	Polymer	Chemical Dosing		Sample Points														
								Inlet				Chamber 1		Chamber 2		Chamber 3		Outlet				
								Turbidity (NTU)	OIW (ppm)	Polymer (ppm)	TSS (ppm)	Turbidity (NTU)	OIW (ppm)	Turbidity (NTU)	OIW (ppm)	Turbidity (NTU)	OIW (ppm)	Turbidity (NTU)	OIW (ppm)	Polymer (ppm)	TSS (ppm)	
19/07/17	8:15	<u>1</u>	S66	1.20	Y	--	--	--	143.8	40.4	0.63	--	47.1	--	45.1	--	40.5	--	33.9	39.7	0.14	
19/07/17	15:15	<u>2</u>	S66	1.20	Y	--	--	167	74.9	47.3	0.43	--	66.8	--	72.0	--	54.1	67	48.2	42.2	1.03	
20/07/17	14:00	<u>1</u>	S66	1.20	Y	--	--	57	101.2	43.6	1.81	55	37.9	55	36.0	55	38.1	55	45.1	42.7	1.90	
21/07/17	7:15	<u>1</u>	S66	1.20	Y	--	--	39	44.1	45.9	0.60	27	15.9	26.3	22.6	24.7	14.8	24	14.6	40.7	0.95	
21/07/17	11:20	<u>2</u>	S66	1.20	Y	--	--	40	40.1	--	--	25.31	20.0	24.46	16.1	23.5	15.9	23	13.9	--	--	
24/07/17	7:30	<u>1</u>	S66	1.20	Y	--	--	34.25	69.4	43.2	0.63	30.3	20.6	29.4	18.2	28.13	12.8	25.39	15.4	48.1	0.65	
25/07/17	7:30	<u>1</u>	S66	1.20	Y	--	--	--	50.7	45.2	--	--	42.1	--	16.9	--	17.4	--	14.7	45	--	
25/07/17	10:45	<u>2</u>	S66	1.20	Y	--	--	--	60.5	--	--	--	18.7	--	13.1	--	14.2	--	11.7	--	--	
26/07/17	7:30	<u>1</u>	S66	1.20	Y	--	--	36.22	39.5	43	0.32	32.32	22.6	31.24	20.6	29.33	22.8	26.57	20	45.5	0.66	
27/07/17	7:30	<u>1</u>	S66	1.20	Y	30 PW1010C	--	--	51.8	43.3	0.77	--	22.0	--	20.5	--	27.1	--	20.9	44.1	0.78	
27/07/17	11:30	<u>2</u>	S66	1.20	Y	30 PW1010C	--	--	35.35	44.1	43.3	--	30.95	32.5	29.85	23.9	29.25	21.2	27.45	21.2	43.1	--
28/07/17	7:30	<u>1</u>	S66	1.20	Y	120 PW1010C	--	--	49.27	46.2	44.2	3.59	46.31	26.1	45.24	23.8	44.39	23.5	43.62	21.6	47.9	1.10
31/07/17	7:30	<u>1</u>	S66	1.20	Y	160 PW1010C	--	--	32.2	43	3.52	--	13.6	--	12.1	--	6.6	--	9.3	19.7	--	
31/07/17	11:00	<u>2</u>	S66	1.20	Y	160 PW1010C	--	--	33.4	--	--	--	10.7	--	7.2	--	12.0	--	10	--	--	
04/08/17	7:00	<u>1</u>	S66	1.20	Y	140 PW1010C	--	--	61.76	79.0	1.3	--	48.56	153.8	40.52	25.3	38.54	18.2	37.94	14.7	22.7	--
07/08/17	7:30	<u>1</u>	S66	1.20	Y	--	--	38.65	63.9	2.2	0.36	24.87	21	24.31	9.4	23.56	9.3	22.76	15.5	22	0.79	
07/08/17	8:45	<u>2</u>	S66	1.20	Y	--	--	--	50.0	--	--	--	--	--	--	--	--	--	11.8	--	--	
07/08/17	13:45	<u>3</u>	S66	1.20	Y	--	--	--	58.9	--	--	--	--	--	--	--	--	--	13.7	--	--	
08/08/17	7:10	<u>1</u>	S66	1.20	Y	50 Floqu&Plus P	0.5 Chimec 5565	38.07	51.8	1.7	0.71	24.76	5.3	--	--	23.41	3.2	20.81	2.7	14.8	1.64	
08/08/17	11:30	<u>2</u>	S66	1.20	Y	50 Floqu&Plus P	0.5 Chimec 5565	--	32.2	1.3	--	--	--	--	--	--	--	--	1.8	13.2	--	
08/08/17	14:30	<u>3</u>	S66	1.20	Y	50 Floqu&Plus P	0.5 Chimec 5565	--	62.0	1.9	0.26	--	9.8	--	--	--	3.8	--	2.3	13.5	0.68	
09/08/17	7:00	<u>1</u>	S66	1.20	Y	40 Floqu&Plus P	0.4 Chimec 5565	29.26	54.5	2.1	0.58	28.35	39.8	--	--	27.95	4.1	27.74	3.6	13.3	0.39	
09/08/17	11:20	<u>2</u>	S66	1.20	Y	40 Floqu&Plus P	0.4 Chimec 5565	--	58.5	2.0	--	--	--	--	--	--	--	--	3.4	11.2	--	
09/08/17	14:30	<u>3</u>	S66	1.20	Y	40 Floqu&Plus P	0.4 Chimec 5565	--	59.0	1.8	0.24	--	23.3	--	--	--	4.4	--	2.5	11.2	2.32	
10/08/17	7:00	<u>1</u>	S66	1.20	Y	25 Floqu&Plus P	0.3 Chimec 5565	31.09	50.9	1.6	0.16	27.45	15.1	--	--	27.21	4.8	26.95	4.0	12.7	0.1	
10/08/17	10:30	<u>2</u>	S66	1.20	Y	25 Floqu&Plus P	0.3 Chimec 5565	--	37.7	2.0	--	--	--	--	--	--	--	--	3.8	12.7	--	
10/08/17	14:30	<u>3</u>	S66	1.20	Y	25 Floqu&Plus P	--	--	51.7	2.1	--	--	--	--	--	--	--	--	5.5	13	--	
11/08/17	7:00	<u>1</u>	S66	1.20	Y	25 Floqu&Plus P	--	--	43.2	2.1	0.27	--	9.6	--	--	--	6.5	--	4.3	13	0.6	
11/08/17	11:00	<u>2</u>	S66	1.20	Y	25 Floqu&Plus P	--	--	36.7	--	--	--	--	--	--	--	--	--	2.8	--	--	