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Abstract

Oil contaminated drill cuttings is considered as a significant waste stream in the oil and gas industry. Drill cuttings contain drilling fluids that interfere and destroy the life basis for animals in the sea. Various discharge limits for oil on cuttings is applied at different locations worldwide.

OSPAR have determined the discharge limit for its members to not exceed 1% OOC. The NCS is member of OSPAR but must also follow local regulations. The Norwegian Environment Agency (NEA) tend to practice stricter regulations as they follow BAT. Projects initiated by NEA have granted specific discharge limits at specific oil fields based on BAT.

The goal of the research carried out is to optimize a new treatment technology developed by Norwegian Technology AS. The optimization carried out was on basis of energy consumption and oil separation. The technology was also evaluated for various types of drill cuttings, from Canada and the NCS with the objective to investigate technology robustness. In addition to optimization of oil separation, energy consumption and technology robustness, a part of the process (condenser) was further investigated to determine its performance.

Three different drill cuttings were received from Conoco Phillips and Husky Energy and treated with the technology from Norwegian Technology AS. Testing of Conoco Phillips 17-inch section was performed in collaboration with a fellow student, Edward Reilly. The drill cuttings from Conoco Phillips 17-inch section was named CP17 and achieved an oil separation of 99.9% using 247 kWh/ton. Two different types were received from Husky Energy, which was named HE1 and HE2 achieved and oil separation of 81.7% and 96.3%, respectively. HE1 was centrifuged before treatment due to troublesome distribution of the drill cuttings in the sample holder during microwave treatment. The new treatment technology proved to increase oil separation and decrease energy consumption compared to microwave technology.

The robustness was also tested as the characteristics of the DC were variating. Treatment of CP17 and HE2 achieved OOC below 0.5%, while HE1 did not achieve OOC below 1%. However, results indicated that further optimization on HE1 could reach oil separation below OSPAR regulations.

Condensate from treatment was examined for product recovery. Oil in water was analyzed to be 0.55 mg/L, which is within OSPAR's regulations for discharge of dispersed oil in water of 30 mg/L. Separated oil can be reused as it did not show signs of cracking.

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Abbreviations

BAT	Best Available Technique
DC	Drill cuttings
GC	Gas chromatography
HPD	High power density
HSE	Health, Safety and Environment
LPD	Low power density
MEG	Monoethylene Glycol
NEA	The Norwegian Environmental Agency
NCS	Norwegian Continental Shelf
NTMW	Norwegian Technology AS microwave
OBM	Oil-based mud
OCDC	Oil contaminated drill cuttings
OOC	Oil on cuttings
OSPAR	Oslo Paris Convention
SBM	Synthetic-based mud
UON	The University of Nottingham
WBM	Water-based mud

1 Introduction

1.1 Drill Cuttings

Since the 1960's, offshore oil and gas activities have been established at the Norwegian Continental Shelf(NCS)(Bakke, Klungsøyr, & Sanni, 2013). A volume of 227 million Salable oil equivalents were produced at NCS in 2018. Norway produce 2% of all oil production, and 3% of all gas production in the world (Norsk Olje&Gass, 2019). Discharges to sea arises mainly from drilling operations and produced water.

Drill cuttings (DC) are produced through drilling activities in exploration and extraction of oil and natural gas. The solids consist of crushed rocks, clay and drilling fluids that is used in the drilling operation. The characteristics for each DC vary due to different rock and clay formations at each location, and what kind of drilling fluids that have been used (H. Shang, Snape, Kingman, & Robinson, 2005).

Drilling operations will become increasingly more difficult in the future as easily accessible oil and gas reserves are decreasing. The oil and gas reserves in the future will be more complex as drilling operations will take place at greater depths and in harsher environments. Complex drilling operations often require drilling fluids as oil-based muds (OBM). Leading to oil contaminated solids that cannot be directly discharged into the sea (Pereira, 2012). Laws and regulations are set by governments and agencies to preserve the environment and guide the industry.

1.2 Discharges and Regulations

Production of DC varies as it depends on how many wells that are being operated and explored. In 2017, 88,000 tons of oil contaminated drill cuttings (OCDC) were produced and handled at the NCS. In Norway, discharge limits related to offshore oil and gas activities are stated in The Activity Regulations (Aktivitetsforskriften). In § 68, it is stated that DC containing water-based mud (WBM) can be directly discharged to sea. This is because all chemicals in WBM are at PLONOR (Pose Little or No Risk to the Marine Environment) list. In 2017, 90,000 tons DC with WBM were discharged to the sea (Norsk Olje&Gass, 2018). Solid waste that is contaminated by formation oil, other oils or base-fluids containing organic drillings fluids is prohibited to discharge to the sea if oil concentration exceeds 1 % on dry weight(Petroleumstilsynet, 2019).

Typical North Sea DC usually contains around 5-15% oil(Bakke et al., 2013). The Oslo Paris Convention (OSPAR) is a commission that set the legislations in the north-west Europe, including the NCS. OSPAR legislations prohibit discharges of DC that exceeds 1% oil (OSPAR, 2000). This has been prohibited since 1993 in Norway, OCDC have been handled and treated in accordance to the regulations(Miljødirektoratet, 2016). Discharges of DC that exceeds 1% oil on cuttings (OOC) requires special authorization by The Norwegian Environment Agency (NEA) (Miljødirektoratet). OSPAR regions came around with the same demand as in Norway in 1996 and 2000.

Since 1993, OCDC have mainly been shipped and treated onshore or re-injected to the reservoir. For Re-injection, DC are crushed, and water is added to make slurries that are easier to inject. Offshore treatment of DC is an attractive option to offshore operators as this would be more efficient in terms of money. Offshore treatment that are in line with regulation can allow offshore operators to directly discharge DC to the sea. This is a strong economic incentive towards development of offshore treatment technologies.

In 2015, NEA permitted Total offshore treatment of DC at the Martin Linge field. This was a pilot project, and NEA set the OOC to 0.3%. Total achieved a separation of 0.38% oil, and the pilot project was canceled (Miljødirektoratet, 2016). Future discharge limits are expected to be stricter compared to OSPAR's 1 % OOC discharge limits. Specific discharge limits can be set by NEA, which can be below 0.5 % OOC.

1.3 Environmental Effects

OCDC piled up on the seabed around and beneath oilrigs until the regulations in 1993 and 1996 were decided. In the worst cases, OCDC were detected 5-10 km from the oilrigs, which damaged the sediments and the benthos (Bakke et al., 2013). Effects to the benthic macrofauna could be detected to the extent of 2-5 km or more.

Piles of DC on the NCS was accumulated before the regulations restricted oil contaminated discharges. Volumes of DC are estimated to be as large as 45,000 m^3 (Bakke et al., 2013). Hydrocarbon concentration in these piles have been measured to be in the range of 10,000 to 600,000 mg kg^{-1}. Hydrocarbon levels are presumed to be the main toxicity factor, but chemicals in drilling fluids and heavy metals may add to this.

Offshore discharge of WBM is allowed at NCS as it pose little threat to sediment macrofauna community, and in general have a low acute toxicity. Effect on fauna from WBM usage is oxygen deficiency in sediments. This is due to degradation of organic compounds in WBM,

such as Monoethylene Glycol (MEG). Furthermore, WBM cuttings only have a local and short term impact to the sediment fauna (Bakke et al., 2013).

Environmental monitoring of sites at the NCS have been performed to measure effects of OCDC on sediment macrofauna community structure. At these sites, macrofauna communities recovered after 4-10 years. After DC regulations, the extent of fauna effect from the discharge site have been reduced from up to 15 km^2 to less than 1 km^2 (Bakke et al., 2013). Studies have indicated that DC discharges in the past have not caused accumulative or long-lasting effects to macrofauna structure.

1.4 Problem Description

The limitations in terms of large footprints, safety, cost, treatment capacity and maintainability for offshore treatment of OCDC is the motivation for research of an alternative treatment technology. Norwegian Technology AS has developed a new microwave technology, which include the use of organic susceptors to enhance separation of oil from DC and reduce energy consumption. Organic susceptors have lower vaporization enthalpies than water, which increase energy efficiency. In addition, organic susceptors allow a higher process temperature, leading to enhanced oil separation. Higher oil separation, while simultaneously reducing energy consumption is achieved by dousing DC with the organic susceptor.

There are many parameters that need to be examined with this technology, such as: oil separation in correlation with energy input, parameters that effects oil separation and energy consumption, DC diversity, and recovery of susceptors and oil.

1.5 Thesis Objective

The aim of the thesis is to further optimize the new drill cuttings treatment technology developed by NT. This includes:

1. Finding important parameters that effects oil and energy consumption for the drill cuttings from Conoco Phillips 17-inch section. Testing of Conoco Phillips 17-inch section was performed in collaboration with a fellow student, Edward Reilly.
2. Apply the findings from the 17-inch section drill cuttings on cuttings from Canada to investigate the overall technology robustness with respect to oil separation for drill cuttings outside NCS.

3. Investigate the condensate for product recovery. This includes separated water, oil and MEG quality.

1.6 Collaboration with Industry

This thesis was initiated and performed with Norwegian Technology AS, which is a company located in Merkjavik, Stavanger. The company has specialized in treatment of water, mud and drill cuttings onsite and at waste reception facilities.

Norwegian Technology AS have a strong environmental focus and aim to develop more sustainable and greener technologies.

Norwegian Technology AS holds two patents for their drill cuttings treatment technology. The motivation is to further develop a drill cuttings treatment system that can treat the waste streams offshore.

Figure 1-1: The logo of Norwegian Technology AS(Norwegian Technology AS, n.d.)

1.7 Novelty of Research

The use of microwave radiation is implemented in various industries. A pilot scale unit for treatment of DC was tested by John Robinson at the University of Nottingham (UON), UK. According to test results from UON, OOC below 1% have been achieved.

Microwave treatment process uses water as microwave absorbing medium. The use of organic susceptor as microwave absorbing medium have been studied by Norwegian Technology AS.

The benefit of using an organic susceptor is that it requires less energy compared to water, it also provides lower vaporization enthalpy and high process temperatures. This translates to cost savings as energy consumption decreases and oil separation meets environmental requirements.

2 Drilling waste

2.1 Drilling fluids

Drilling fluids are special mixtures pumped into wells to improve drilling operations. The fluid is pumped down and through the drill bit. Then it returns to the surface via the annulus to be recirculated. Drilling fluids are often called muds, and has many functions; Cool and lubricate the drilling assembly, maintain formation pressure and well-bore stability, bring DC to the surface, corrosion control, etc.(American Society of Mechanical Engineers. Shale Shaker, 2005). Drilling fluids have different composition depending on reservoir characteristics.

The major ingredients in drilling fluids are liquid. The base is either water, oil or another organic fluid. Weighting material such as barite ($BaSO_4$) and additives which enhance technical performance are also added(Bakke et al., 2013). There are three main types of drilling fluids which are classified according to their base fluid: WBM, OBM and synthetic-based mud (SBM).

2.1.1 Water-based mud

WBM are aqueous drilling fluids and the continuous phase can be freshwater, seawater or brine. This mud is viewed as an environmentally friendly drilling fluid because of its low oil content and have earlier been directly discharged into the sea. Components in WBM are being increasingly restricted. This may lead to substitution of components or treatment of WBM in the future(American Society of Mechanical Engineers. Shale Shaker, 2005). WBM have proved to not be enough in non-vertical drilling operation, where OBM and SBM are more suitable.

2.1.2 Oil-based mud

OBM and SBM are both non-aqueous drilling fluids. OBM are distilled from crude oil and include diesel, mineral oils and refined linear paraffins. Until 1982, it was discharged directly into the North Sea, but the diesel oil was replaced with low-aromatic oils which are less

toxic(Bakke et al., 2013). Offshore usage of OBM are restricted and some operators must apply a zero-discharge policy.

2.1.3 Synthetic-based mud

SBM are known as pseudo-oil-based muds which include olefins, esters and synthetic linear paraffins. These muds are less toxic and more biodegradable under aerobic conditions. DC containing SBM was earlier viewed as environmental superior compared to OBM. This have proved not to be the case and initial cost of SBM is relatively high. SBM have a negative effect to oxygen conditions in the sediment. This have led to SBM being gradually phased out (Bakke et al., 2013).

3 Drill cuttings treatment technologies

DC are produced when a drill bit penetrates the seabed. These rock particles can have many different characterizations, depending on the type of mineral consisting in the seabed. Drilling fluids transport DC to the surface where solids are removed to maintain drilling efficiency. The DC are separated from the drilling fluids in shale shakers, which are recirculated and eventually sent back into the borehole.

OCDC must be treated on-site, sent onshore for treatment or reinjected to the reservoirs. Many factors related to handling of DC need to be considered:

- Handling and transportation costs
- Regulations for treatment and discharge
- Environmental impacts
- Capacity and facilities offshore and onshore
- Treatment technologies
- Distance to shore

There are many different treatment and disposal options for DC; non-biological, thermal technologies and biological.

3.1 Non-Biological treatment and disposal

3.1.1 Re-injection

Re-injection is an option at offshore rigs, which takes place on-site and saves transportation costs. DC are crushed and mixed with water, brine or seawater to make a slurry which are

pumped into the reservoir. To perform re-injection, it is important that the DC have properties that allow for pumping(Veritas, 2012).

Re-injection might not be the best option because fractures in reservoirs can leak harmful components to the environment (Norsk Olje&Gass, 2018). Leakages of re-injected DC may be a problem in the future, and reservoir may have to be controlled or monitored regularly to avoid environmental impacts.

3.1.2   Pit burials and landfills

Pit burials and landfills are onshore options for disposal of DC. The costs related to pit burials and landfills would mainly be transportation cost to shore, since the methods are relatively cheap. Pit burials and landfills are not optimal for drill cuttings with high concentration of oil and harmful metals(Ball, Stewart, & Schliephake, 2012). Oil contaminated solids can pose an environmental risk due to harmful components in leachate. This require monitoring of the sites if they pose any threat to groundwaters or rivers. Landfills are often equipped with geomembranes, often called liners, to prevent leachate reaching groundwaters.

3.1.3   Stabilization and solidification

Stabilization and solidification are processes which transform DC into less hazardous components. Solidification alter the physical structure by adding materials e.g. cement, which will encapsulate the DC. Stabilization alter the chemical structure of DC as chemicals are added to transform hazardous components into less soluble, mobile and toxic forms (Leonard & Stegemann, 2010).

The end-product from stabilization and solidification processes could be materials for road foundations, backfill for earthworks or construction of buildings. Another advantage in is that contaminants gets easier to handle, both in terms of mobility and safety (Ball et al., 2012).

3.2   Biological treatment and disposal

Biological treatment processes can be defined as a process where organism degrade organic material. This is a natural process which can turn hazardous contaminants into residues that are less harmful and toxic. There are different ways to utilize organism ability to degrade organic material, where some require larger investments than others. The less the process is controlled and enhanced, the less efficient it will be. Increased control goes hand in hand with costs as it requires more technology and personnel. Factors that can improve the degradability are temperature, aeration, water content and nutrients(Ball et al., 2012).

3.2.1 Land farming

Land farming is one technique that utilizes organisms to degrade hydrocarbons in DC. This technique utilizes the organisms that already are present in the soil. Drilling waste are spread over the soil in a 10-35 cm thick layer(Ball et al., 2012). This layer requirement restricts the application rate, and drilling waste might be spread in several applications. Nutrients such as fertilizers, manure and compost can be added to improve the process by facilitating the conditions for organisms. This technique is not fully controlled. Factors like temperature and water content may vary a lot in this process, which is a disadvantage that slows down the process. There is also a risk of hazardous leachate. If there is any risk of groundwater contamination, then the process should be monitored.

3.2.2 Land spreading

Land spreading is another technique of biological treatment. This technique is similar to land farming. The most important difference between these two techniques is that all drilling waste is spread over the soil in only one application. This demand a larger area compared to land farming.

3.2.3 Bioreactor

Bioreactors are more controlled and efficient way to degrade hydrocarbons. The process takes place in a container or tank with optimized conditions for organisms to thrive. Factors like temperature, water content and aeration are carefully controlled to enhance organism reproduction and degradation rate of hydrocarbons. The monitoring of the degradation process makes bioreactors to one of the more expensive biological methods. Another limiting factor for organisms is nutrients like nitrogen, phosphorous and potassium. These nutrients can also be added to stimulate degradation rate. However, bioreactors are more expensive option in terms of biological treatment, but it is also a quicker process compared to land farming and land spreading. After 10-12 days 99% of the drilling waste can be degraded(Ward, Singh, & Van Hamme, 2003).

3.3 Thermal treatment technologies

3.3.1 Incineration

Thermal treatment processes separate or destroy contaminants by using heat. These treatment technologies consume a lot of energy. Incineration is such a process that require a high energy input. This process degrades and destroy hydrocarbons due to high temperature, and

contaminants are transformed to inert residues. Incineration temperature usually vary between 820-1600°C(Ball et al., 2012).

Rotary kilns are the incineration technology which usually is used for incineration of drilling waste. Rotary kilns enhance the process by turning the drilling waste in a tumbler. This increase contact between drilling waste and hot burner gases. The combustion of drilling waste produce flue gases and residues containing heavy metals. These by-products need to be managed in an environmental responsible manner(Ball et al., 2012). Another disadvantage with incineration of drilling waste is its water content. Incineration of water is not efficient terms of energy. Energy spent to evaporate water is not favorable and adds to costs of running a rotary kiln.

3.3.2 Thermal desorption

Thermal desorption does not aim for destruction of hydrocarbons. Thermal desorption operates at lower temperature and is a separation process(Ball et al., 2012). This technology separate organics from drilling waste by volatilizing them. This process typically heats drilling waste to approx. 600°C. The volatilized part is sent to a separator due to a carrier gas or a vacuum system. Hydrocarbons and water are usually separated in scrubbers or filters.

3.3.3 Cuttings dryer

There are different cuttings dryer technologies. One type of cuttings dryer is based on centrifuges, which use centrifugal forces to dry DC. Another cuttings dryer dries cuttings using high-velocity air and differential pressure. These technologies cannot alone obtain the OOC requirement set by OSPAR (Billeaud & Morris, 2007). This restrict use on cuttings dryers at the NCS. However, a cuttings drier could be considered as pre-treatment for DC before further treatment.

In North-America, the discharge limit of DC is set to 6.9% OOC (National Energy Board, 2010). A cuttings drier can reduce OOC to 3-5%, depending on the DC. Therefore, cuttings driers are more attractive in North-America compared to at the NCS (Billeaud & Morris, 2007).

3.3.4 Thermomechanical cuttings cleaner

Thermomechanical cuttings cleaner (TCC) has the same principle as thermal desorption (Figure 3-1). The TCC is currently Best Available Technique (BAT) for both offshore and onshore treatment of DC.

Drilling waste is crushed to small particles by a hammermill. Friction between particles generate heat, and the process temperature in a TCC lie between 250-300 ˚C (Bytt, Vik, Stang, Henninge, & Kjønnø, 2014). Temperature raises due to increased thermal energy, which in turn evaporate both oil and water. This technology provides on-site treatment of DC and recovered oil can be reused (Ormeloh, 2014). TCC capacity depends on motor size and footprint is a factor of great importance at oil rigs. Therefore, the TCC facilities onshore have a better foundation for high treatment capacity.

Figure 3-1: TCC unit (Thermtech AS, n.d.)

Advantages:

- Meets OSPAR discharge limit of <1%OOC
- Hydrocarbons can be reused
- Lower process temperatures than incineration
- Eliminates transportation of DC to shore

Disadvantages:

- High investment costs
- Potential heavy metals in DC can be released
- Large footprint
- Noise emissions from hammer mill
- Processing requires constant supervision because of high temperatures

3.3.5   Microwave technology

Microwave is another option for thermal treatment of DC and will be extensively reviewed in this thesis. The main difference between the microwave and other thermal treatment technologies is that the microwave heats the drilling waste internally.

In contrast to conventional thermal technologies that heats material surfaces through conduction, convection and radiation(J. P. Robinson, et al, 2009). Electromagnetic waves cause friction as molecules alter position to match the charge in the electromagnetic waves. This friction evaporates water, and oil is separated from DC in an evaporative mass transfer process (Ogunniran, Binner, Sklavounos, & Robinson, 2017).
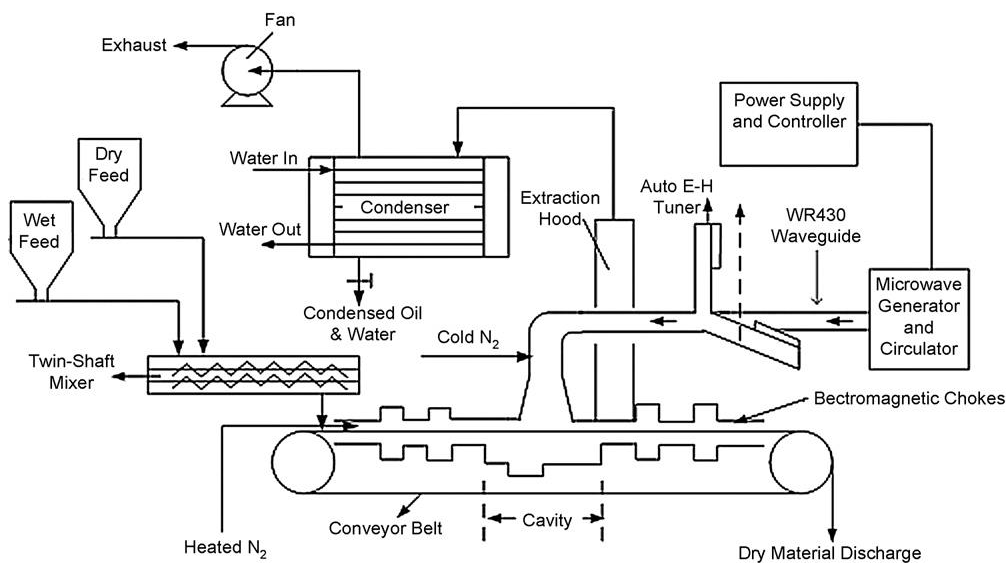
Figure 3-2: Schematic of pilot-scale unit for treatment of DC (J. P. Robinson et al., 2009)

A study at The University of Nottingham tested treatment of DC with a pilot-scale microwave unit (Figure 3-2). The unit had a maximum power output of 15 kWh and achieved OOC below 1 % using 90-100 kWh/ton.

Advantages:

- Low footprint
- Minimal Noise emissions
- No moving parts, except for a transportation belt of screw conveyor for transporting DC
- Instant startup of the unit and treatment
- High processing rates with good design

- Minimal downtime and good maintainability

Disadvantages:

- The robustness of microwave unit is not properly tested yet
- Treatment efficiency depend on distribution of microwaves in DC

4 Theory

Conventional thermal processing heats materials through convection, conduction and radiation. These processes transfer energy and heats the materials from its surface. Microwave heating is different from conventional thermal technologies. Unlike conventional thermal technology, microwaves make it possible to heat materials internally and are not limited by thermal conductivity(J. P. Robinson, Kingman, & Onobrakpeya, 2008).

Internally heating is more efficient than heating from a surface (Bhattacharya & Basak, 2016). A microwave generates electromagnetic and energy that is converted to thermal energy through molecular interactions. This occurs because the microwave emits electromagnetic waves which can penetrates materials. Penetration depth varies for different materials and temperatures. The electromagnetic field (Figure 4-1) created by microwaves enable the benefit of selective heating of specific phases in materials.
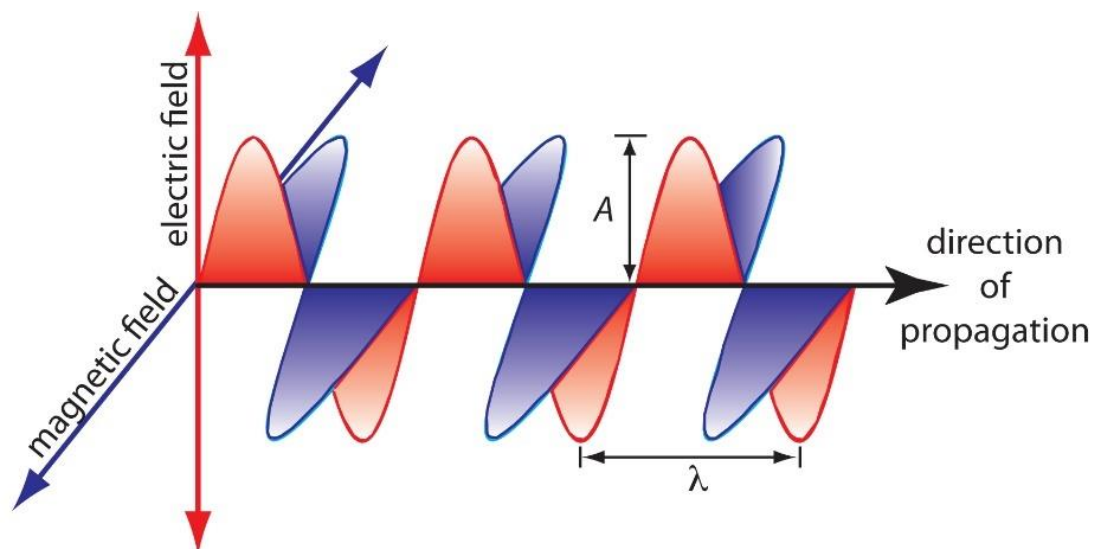
Figure 4-1: Electromagnetic field (Harvey, 2019)

4.1   Electromagnetic spectrum

The electromagnetic spectrum is broad and includes all frequencies from long radio waves to gamma rays. Microwave frequencies is commonly measured in Hertz (Hz). Number of Hz are equal the number cycles per seconds(Britannica, 2013).

Microwaves usually have a wavelength between 1 mm to 1 m. This corresponds to frequencies of 300 MHz to 300 GHz (Thostenson & Chou, 1999).The microwave spectrum covers frequencies that are used in radars, microwaves, cellphones and television satellite communication (Figure 4-2). The electromagnetic frequencies used for microwave heating are 0.915 and 2.45 GHz (Thostenson & Chou, 1999). Exact these two wavelengths were chosen in an international agreement to prevent interference with communication services(Meredith, 1998).
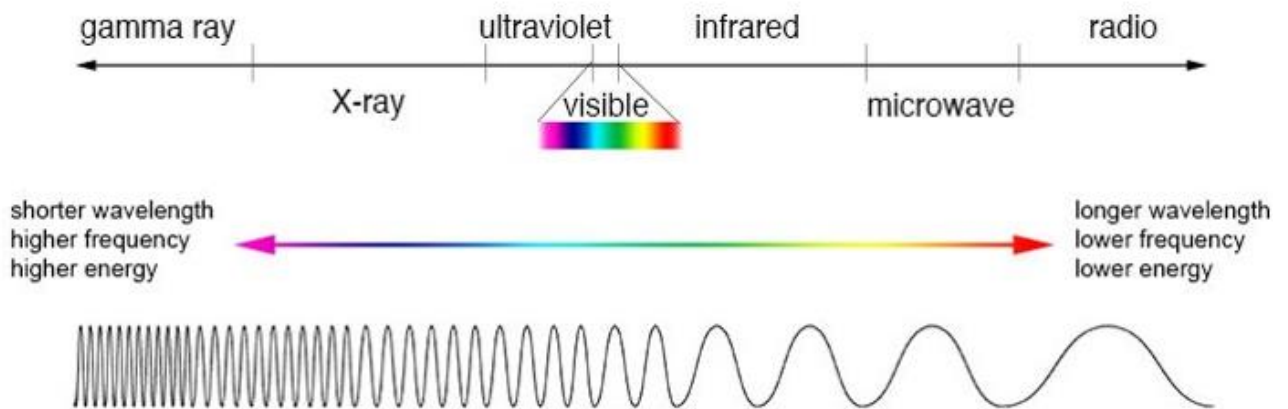
Figure 4-2: Electromagnetic spectrum (Humboldt State University, 2018)

4.2   Dielectric hysteresis

Microwaves heat materials due to dielectric hysteresis, often referred to as dielectric heating. As mentioned earlier, a microwave creates an electromagnetic field, which expose materials to microwaves and are continuously alternating direction in a very high frequency. Dipolar parts of a molecule will continuously try to align with the electrical waves (Figure4-3). Heat from friction is created because of these molecular interactions (Sumper & Baggini, 2012).
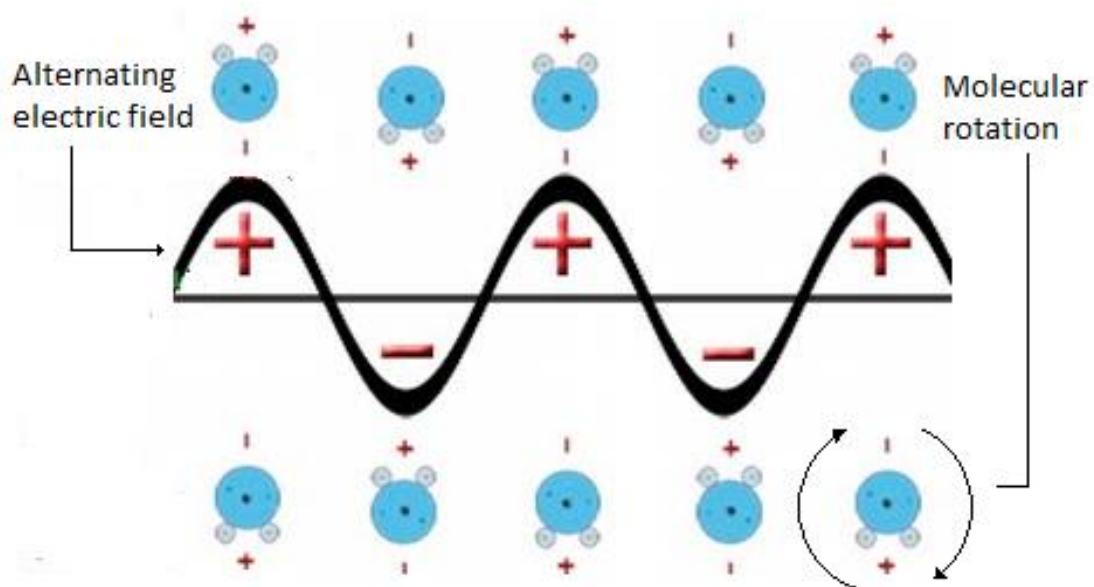
Figure 4-3: Electromagnetic energy converted to thermal heat due to rotation of molecules (Sites.google, 2018)

4.3 Dielectric properties

Heating abilities by a microwave depends on the treated materials dielectric properties. All materials respond differently to alternating electromagnetic fields. Two fundamental properties are used to measure materials response towards electromagnetic fields; dielectric constant and dielectric loss(Bhattacharya & Basak, 2016).

Dielectric constant is also called the permittivity. This property determines materials ability to store electric energy. Dielectric loss are materials ability to convert electric energy to heat. These properties refer to the electrical part of the electromagnetic field. Polarization of bound charges in molecules storage electrical energy. Conversion of electrical energy to thermal energy occurs through relaxation of polarized molecules (Bhattacharya & Basak, 2016).

As the electromagnetic field constitutes of both electrical and magnetically fields, there are properties that also covers the magnetic field as well; permeability and magnetic loss. Permeability determine materials ability to store magnetic energy, while magnetic loss determine their ability to convert magnetic energy to heat(Bhattacharya & Basak, 2016).

4.4 Penetration depth

Penetration depth describe how far microwaves penetrate the material. Microwaves do not have the same penetration depth for all materials. Materials can either reflect, transmit or

absorb microwaves (Shang, Snape, Kingman, & Robinson, 2005). How materials react to microwaves are classified as reflector, transparent and absorbing materials.

Reflectors are materials with very low penetration depth. This category are typically metals, and their penetration depth are usually around µm(Bhattacharya & Basak, 2016). Reflectors will completely reflect microwaves from their surface due to their low penetration depth. Hence, microwaves are not able to heat the material.

Transparent materials are opposite of reflectors, and their penetration depth are in magnitude of meters. Typical transparent materials are alumina, quartz and teflon, which can obtain penetration depth greater than 10m at a frequency of 2.45GHz (Shang et al., 2005). Microwaves pass right through these materials. Therefore, no significant absorption of microwaves occurs, and the materials will not be heated.

Absorbing materials will readily absorb microwaves (Figure 4-4). They usually have a penetration depth of the order of cm and can readily turn electromagnetic energy into heat. Typical microwave absorbing materials are water, carbon and silicon carbide(Bhattacharya & Basak, 2016). These materials contain molecules with charged dipoles which responds to electromagnetic fields by rotation, that again creates thermal heat.
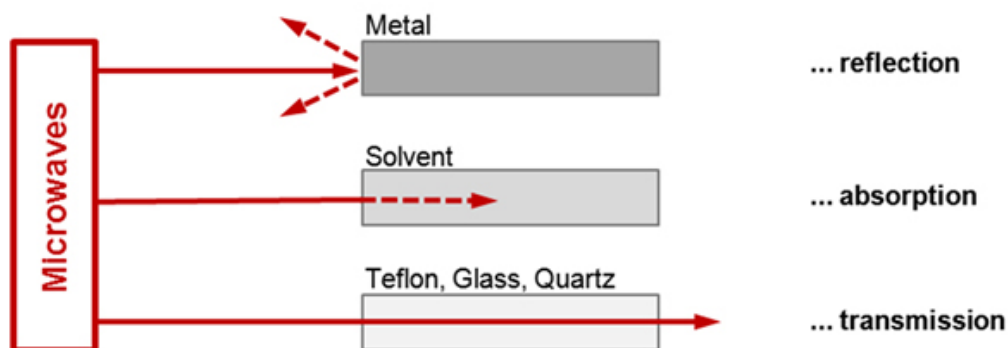
Figure 4-4: Penetration depth (Anton Paar, n.d.)

However, materials ability to absorb microwaves does not only depend on penetration depths. Factors like temperature, salinity and physicochemical properties can alter materials dielectric properties. Alumina is transparent to microwaves at 900 ˚C, but will become an absorbing material if the temperature is raised to 1000 ˚C(Bhattacharya & Basak, 2016).

Another important factor which impact on penetration depth is the microwave frequency. A lower frequency obtains a greater penetration depth. This result in better heating of materials, and larger mass transfer because a larger surface area is treated(Pereira, 2012). With other words, 0.915 GHz is more efficient than 2.45GHz.

4.5   Power density

Power density is power absorbed per unit volume(J. P. Robinson et al., 2010). High power densities yield higher heating rates, this results in a quicker conversion of water into steam and DC is treated more efficient.

Single mode cavities are often chosen over multimode cavities because of their ability to maximize power density. Using high power densities (HPD) will also push parameters of the process to its limits. This results in an increased probability of voltage breakdown, arcing and explosions, which can damage equipment(Meredith, 1998). These dangers can be restricted through proper design and usage of the microwave unit. Health, safety and environment (HSE) is the main reason why low power densities (LPD) are used in microwaves for commercial use. However, HPD are the most popular when considering DC treatment. This is because HPD is more efficient in terms of oil separation, time and energy input as HPD generate heat more efficient.

4.6   Arcing and thermal runoff

Arcing can occur in any microwave treatment processes but are most common in single mode cavities where HPD are applied. When materials that readily absorbs microwaves, such as water evaporates, more power will be reflected due to decreasing absorbing materials. Arcing occurs when reflective surfaces continue to be heated, which generate an intense local heating (Meredith, 1998). An example from daily life is a standard microwave in households (Figure 4-5). Sparks are generated inside a microwave when metals, such as aluminum foil, are put inside of this microwave.
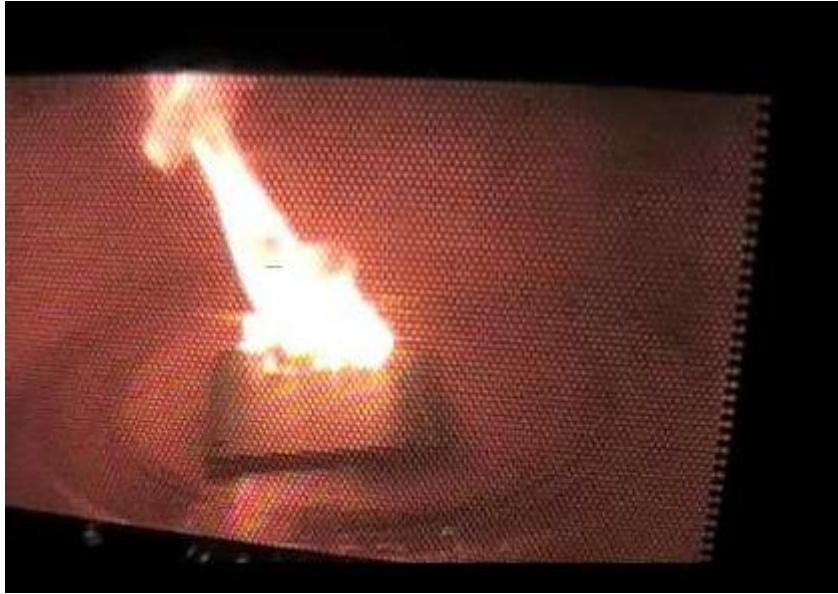
Figure 4-5: : Aluminum foil in a microwave resulting in arcing (InfoBarrel, 2010)

Thermal runaway occurs in a microwave process when applied power on a small part of a sample exceeds the rate of heat transmission to its surroundings. Hence, the increase of enthalpy is greater at one spot than the surroundings, the heat distribution is inhomogeneous. The temperature at this spot will increase at a faster rate until decomposition occurs(Meredith, 1998).

4.7 Microwave components

4.7.1 Microwave generators

Microwaves are generated in vacuum tubes which contain an anode and a cathode. These can generate 1-40 GHz microwaves(Bhattacharya & Basak, 2016). The anode is at a higher potential compared to the cathode. An electrical field is produced due to the potential difference between the anode and the cathode. Electrons are emitted from the cathode and accelerates to the anode due to the electrical field. Three different vacuum tubes are most commonly used; Klystrons, travelling-wave tubes (TWT) and magnetrons (Thostenson & Chou, 1999).

Magnetrons tend to be chosen over klystrons and TWTs, and they are common in industry and commercial use of microwaves. Mainly due to lower manufacturing costs and their high ability to convert electrical power to microwaves. Conversion efficiency of microwaves are usually between 30-70% (Pereira, 2012). Magnetrons are normally cylindrical, where the anode is the wall and contains resonant cavities in series. The cathode is positioned in the center (Figure 4-6).

30
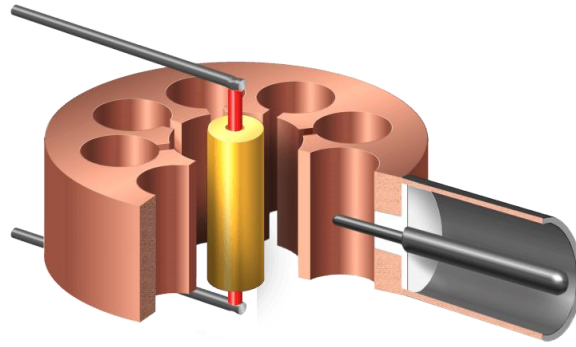
Figure 4-6: Magnetron (radartutorial.eu, n.d.)

4.7.2 Transmission lines/wave guides

Microwaves are transmitted from magnetrons or other vacuum tubes to a sample holder through transmission lines or wave guides. Transmission lines direct microwaves with rectangular, cylindrical wave guide or coaxial cables (Figure4-7). The Wave guide direct microwaves normally in a 90˚ bend between target and vacuum tube(Thostenson & Chou, 1999).
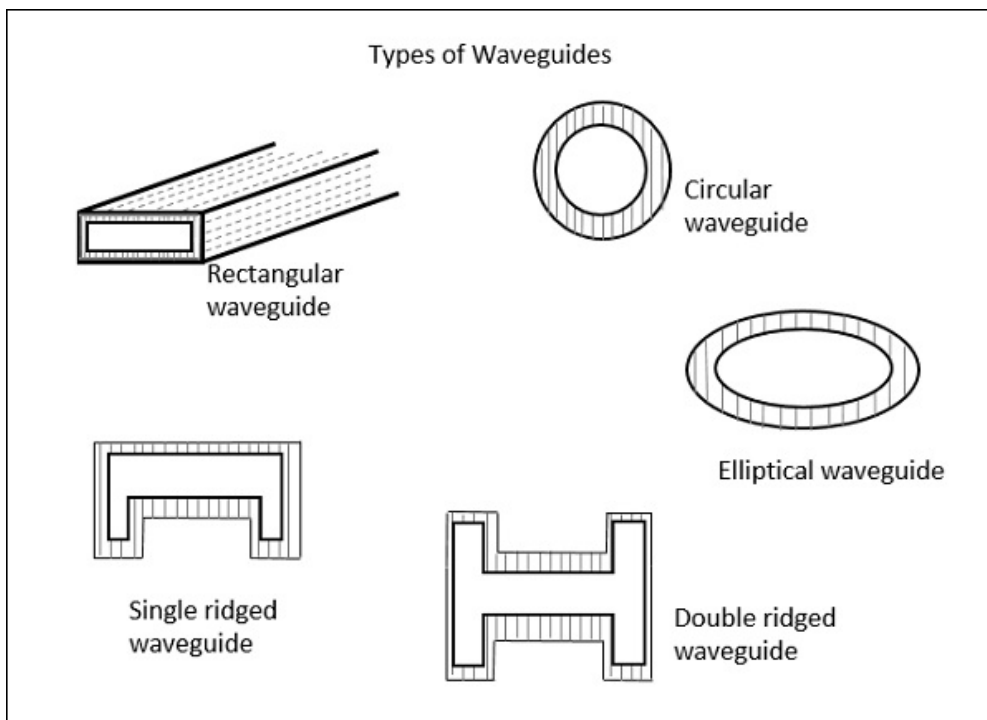
Figure 4-7: Different configurations of wave guides (tutorialspoint, n.d.).

4.7.3 Circulators

Materials can change their absorbing capabilities when temperature is raised. This can be due to evaporation of water or depletion of other microwave absorbing material. Materials will then start reflecting more and more microwaves back to the magnetron. Excessive reflected

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power can damage magnetrons. Circulators protect the magnetron by redirecting reflected microwaves back to the sample(Thostenson & Chou, 1999).

4.7.4   Applicators

The main chamber of a microwave is called the applicator, resonator or cavity. This is a cavity with metallic walls. The applicator plays a crucial role in microwave processing because microwave energy is transferred to the sample via the applicator. The applicators shape and size affect distribution and intensity of the electromagnetic field. There are different types of applicators (Figure 4-8), and the type used depends on which materials that are processed; single mode, multimode and various frequency multimode are all used in microwave processing(Thostenson & Chou, 1999).
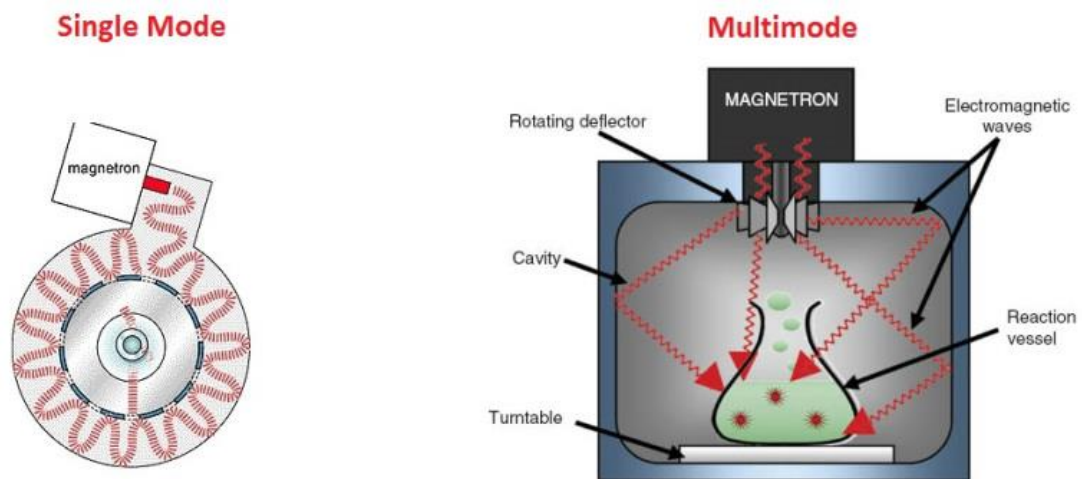
Figure 4-8: Single mode and multimode applicators (Hackaday, 2015)

4.7.4.1   Single mode applicator

Single mode applicators are usually a cylindrical cavity. The Radius of this cavity is restricted to approximately one wavelength, and they only support only one resonance mode. The electromagnetic field distribution of single mode applicators is non-uniform. However, the electromagnetic field can be predicted as a hot spot where microwave intensity is high(Pereira, 2012).

Correct design of a single mode applicator can enable to focus microwave intensity, and a high power density can be obtained at a desired location(J. P. Robinson et al., 2010). This is an ability which is useful in DC treatment, and single mode applicators are often chosen over other applicators because of this.

4.7.4.2 Multimode applicator

Multimode applicators are common in microwaves used in our daily life. This applicator is often a rectangular cavity that exists in various dimensions. Unlike a single mode applicator, the multimode can support many resonant modes and they are normally larger than one wavelength. As the size of the multimode applicator increases, the number of resonant modes increases as well (Thostenson & Chou, 1999).

Microwaves in a multimode applicator are considered random, and to target and heat a specific location can be difficult. The need for maximum power densities eliminates usage of multimode applicators for treatment of DC. For industrial applications as batch processing and largescale operations, multimode applicators are the most frequently used applicator, as they are more versatile than single mode applicators(Thostenson & Chou, 1999).

5 Materials and Methods

5.1 Microwave unit

The microwave used for the experiments in this thesis is a single-mode microwave which is constructed by Fricke und Mallah Microwave Technology GmbH. Operation frequency of the microwave is 2.45 GHz and have a maximum power output of 2 kW. Power loss in the system decreases operational treatment power to about 1.92 kW. Figure 5-1 illustrates the set-up and size of the microwave.
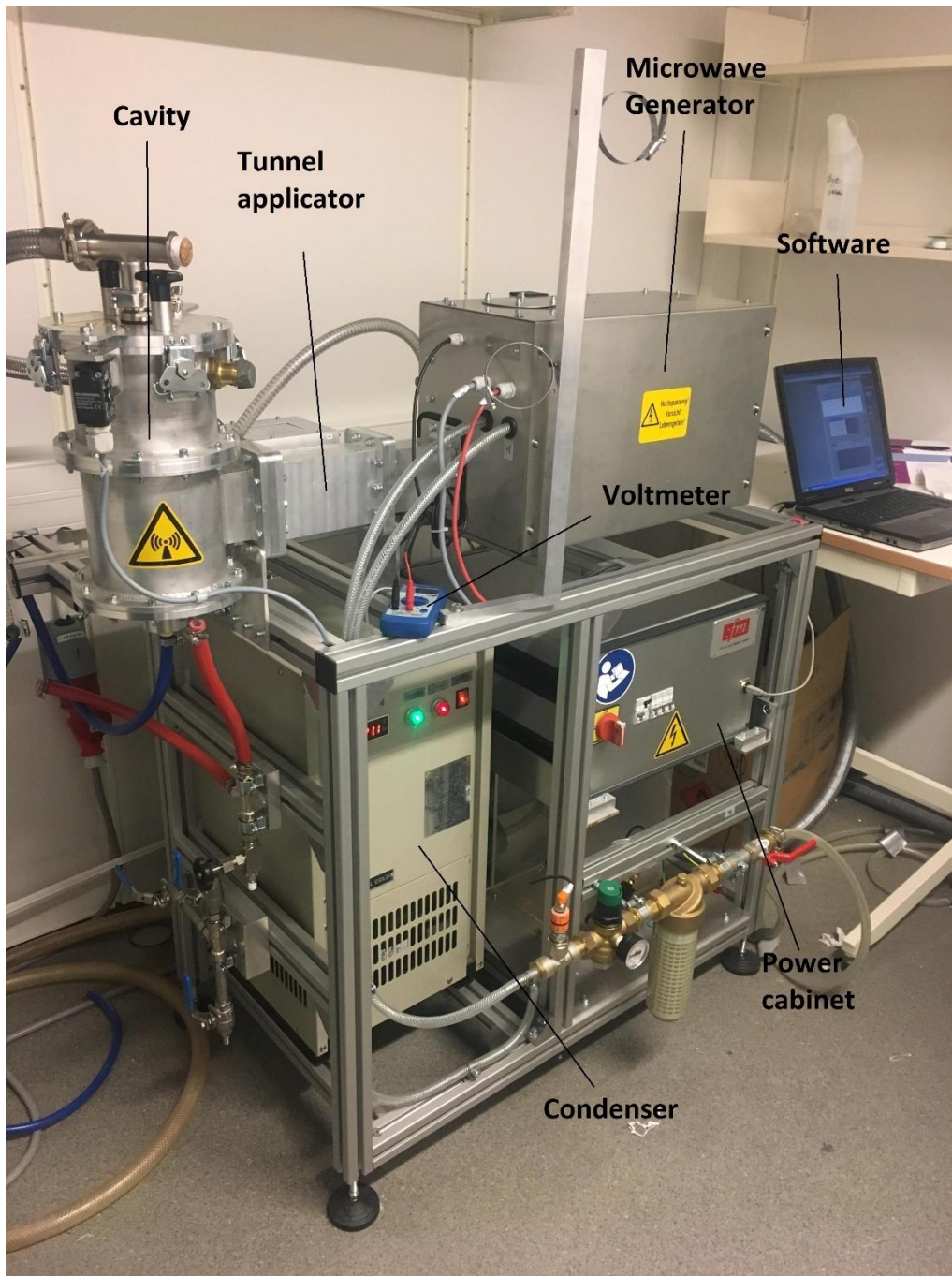
Figure 5-1: Microwave setup

All microwave tests were performed at the University of Stavanger. The materials used during microwave treatment are listed in Table 5-1.

Table 5-1: List of Equipment for microwave treatment

Equipment	Model	Manufacturer
Microwave	Version 1.03en	Fricke und Mallah Microwave Technology GmbH
Pressurized N_2 gas	Nitrogen 4.0	Yara Praxair
Voltmeter	1070 DMM	Peak Tech
Glassware	-	-
Weight	KERN440	KERN & SOHN GmbH
Centrifuge	Rotomix 46	Hettich
Magnetic stirrer	Hei-Standard	Heidolph
Susceptor	MEG	Sigma-Aldrich
Thread sealing tape	PTFE	Biltema
Twist	-	Biltema
Duct tape	-	Biltema
Vacuum pump	739003	-

5.1.1 Preparation of samples

All DC were stored in a cooling room at 4 ˚C to prevent bacterial growth and decay of properties. 150g of DC was weighed and placed in a sample cylinder. Twist were put on top and below of the sample to make sure correct position of the sample in the single mode cavity (Figure 5-2). The sample cylinder was then sealed with a top and bottom lid. The top lid has an extended tip and opening to readily lead away steam and oil. The bottom lid has a hole to ensure pressurized N_2 gas flowing through the sample cylinder and sample. The N_2 gas

substitute O_2 in the sample and created an inert environment while the microwave was running, which decreased arcing and explosion hazards. The two lids were properly sealed with thread sealing tape to prevent leakages. Duct tape was also used to make sure the bottom lid stays in place.

Figure 5-2: Sample cylinder with DC

The sample cylinder tube has two compartments which is separated by a glass weld. The bottom compartment contains the DC sample, while the top compartment ensures free outflow of steam and oil. The weld includes a glass tip with a bend, to prevent condensed water to enter the sample again.

5.1.1.1 Centrifugation of Husky drill cuttings

DC with high oil concentration and low water content need an additional pre-treatment step. For this thesis, centrifugation was chosen to prepare certain DC for microwave treatment. One of the DC from Husky Energy contained 9.81 % oil and 4.46 % water (HE1). As this sample

was difficult to position and clogged the sample cylinder, it was centrifuged before microwave treatment to remove liquids. Another advantage with centrifugation is that the samples become more homogenous, as oil and water composition can vary between each sample.

The centrifuge Rotomix 46 was used in this thesis. DC was weighed and divided in 4 bottles that fit in the centrifuge, each centrifuge bottle had exactly equal weight.

The DC was centrifuged for 3 minutes at 3000 rounds per minute (RPM). The samples were then placed upside-down at a desk overnight to properly remove additional liquids.

5.1.2   Operating the microwave unit

The DC is prepared as shown in Figure 5-2 and placed in the single mode cavity. Cooling water for the microwave must be running and regulated to a temperature between 18 to 25 ˚C. The microwave and software are then started.

Desired power input and treatment time is regulated with a software, which allows adjustments in power input by typing in percentages of maximum power. Treatment time is also decided by typing desired seconds into the program (Figure 5-3).
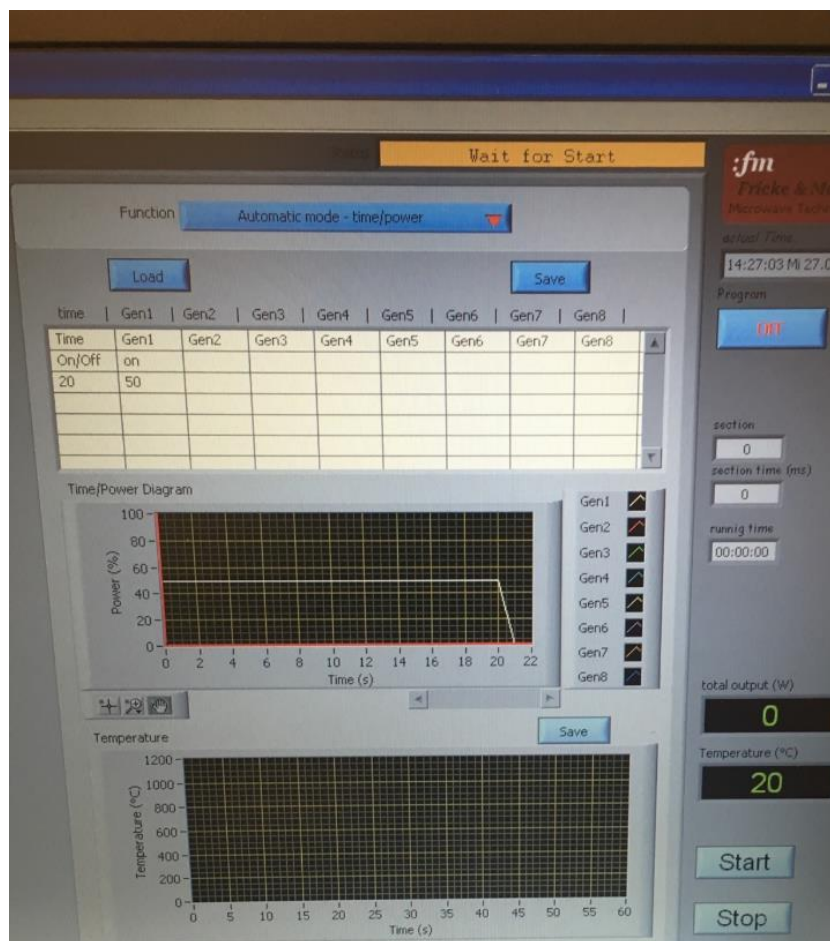
Figure 5-3: Microwave software

When the sample cylinder was placed in the single mode cavity it was sealed with a top lid. The microwave cannot start before the top lid is in correct position. Pressurized N_2 gas was set to a flow of 8 L/min through the cavity and sample. The microwave is an open system, which reduce pressure to build up. This again decreases the probability of explosions. A vacuum pump was applied to remove fumes and steams while treating DC.

5.1.3 Optimization of microwave

Optimization of microwave technology is important to become a competitive technology for treatment of DC.

5.1.3.1 Reflective power

Power consumption was measured in kWh/ton in this thesis. Microwaves are both being absorbed and reflected while treating DC. Microwaves are reflected when no materials have absorbed the microwaves. Reflected microwaves are energy that is recirculated, however some are lost in the system.

A voltmeter was used to measure the reflected microwaves. The Voltmeter was set to 200 mV and readings was voice recorded as the microwave was running. Recorded mV was converted to watts using Table 5-2.

Table 5-2: Relationship between millivolt and watt

Watt (W)	Millivolt (mV)
40	7,5
50	9,1
64	10,5
80	11,5
100	15
128	20
160	22
200	29
256	35
320	41
400	50
500	60
640	70
800	85
1000	100
1280	110
1600	125
2000	150

Absorbed power is the power consumption and was calculated by subtracting reflective power from input power (1.92kW/h). This had to be done for all time intervals that were recorded and summed up using equation 1. The sum was finally divided by the sample weight and converted to kWh/ton.

$$\sum \frac{Absorbed\ power}{Time\ interval} \times 3600 \qquad (1)$$

5.1.3.2 Mono ethylene Glycol

MEG was added to hot DC after microwave treatment to enhance oil separation, which will be explained in Chapter 6 as it is an important parameter of Norwegian Technology AS new technology.

A pipette was found to be the most efficient way to dose MEG to DC in the sample cylinder. Figure 5-4 show how MEG was dosed onto DC. The sample cylinder was turned as MEG was dosed to distribute MEG as good as possible in the DC.

Figure 5-4: Dosing of MEG onto DC

MEG was also heated before added to the DC to further optimize the microwave treatment. MEG was heated by a silicone oil bath in an Erlenmeyer flask, which was flushed with N_2 to prevent decomposition of the MEG, as shown in Figure 5-5. Addition of both cold and hot MEG was performed. MEG was added to the DC with a 25 ml pipette.
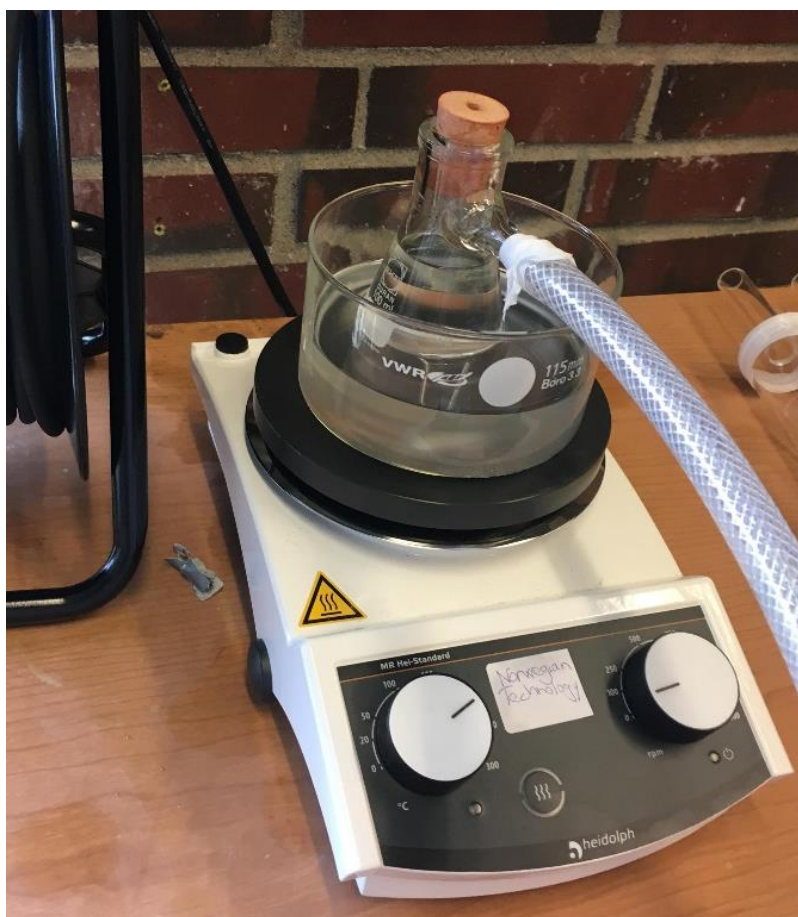
Figure 5-5: Heating of MEG

5.1.3.3 Activated carbon

AC was mixed with MEG to investigate further optimization for microwave treatment of DC. AC absorb microwaves and should in theory increase microwave absorption. A solution of 2% AC in MEG was created by mixing 2g granular AC with 100 ml MEG (Figure 5-6).

The granular AC clogged the pipette, and a 50 ml flask was used for dosing MEG and AC onto DC.

Figure 5-6: MEG mixed with AC

5.1.3.4 N_2 stripping

N_2 was applied during microwave treatment to create an inert atmosphere. Optimization using N_2 as stripping gas to remove condensate was examined. After treatment with MEG, the sample cylinder and DC was flushed with N_2 gas at 15 L/min for 2 minutes using N_2 gas from a pressurized tank.

5.1.3.5 Pre-heating of DC

DC were in some tests heated to approx. 70 C˚ to simulate offshore conditions. As DC is extracted from the borehole it holds a considerably higher temperature than the DC in this thesis, which was stored in a cooling room.

Samples of 150g DC was weighed and placed in waterproof plastic bags. It was important to prevent water from contaminating the sample, as it could alter energy consumption of the treatment.

Samples in plastic bags were put in a water bath and heated by a heating plate (Figure 5-7). Temperature was controlled using an electrode that measured temperature. Samples were moved to the sample cylindered and treated in the microwave when 70 C˚ were obtained.

Figure 5-7: Set-up for heating of DC

5.2   Soxtec Analysis

The Soxtec is a solvent extraction system, which extracts liquids from solids (Anderson, 2004). The method was used to determine OOC. The Soxtec can analyse six samples simultaneously. Each sample was analyzed in parallels to get as accurate and consistent results as possible. Equipment list for this method is in Table 5-3.

Table 5-3: Soxtec analysis equipment list

Equipment	Model	Manufacturer
Soxtec system	HT1043	Foss-Tectator
Thimble	-	-
Sample cups	-	-
Petroleum ether	-	Sigma-Aldrich
Heptane	-	Sigma-Aldrich
Analytical balances	Adventurer	Ohaus
Heating plate	Hei-Standard	Heidolph

The thimbles are made of cellulose and they hold the sample during the Soxtec analysis. The thimbles were weighed before 4-5g of sample was added. Then the thimbles were weighed again to find the exact sample weight. Cotton pads were placed in the thimble to make sure that sample stays in the thimbles during the analysis.

The extraction cups containing solvent was washed three times with heptane. The cups were dried and added 6-8 boiling stones. The cups were also weighed, and 50 ml of petroleum ether was added to each cup as solvent. Then thimbles and cups were placed in the Soxtec as shown in Figure 5-8.

The Soxtec has two modes; boiling and rinsing, which was applied for 50 minutes each. The boiling mode speeds up the extraction, whereas the thimbles are submerged into the petroleum ether. Rinsing mode is the second stage, and the thimbles are raised above the cups containing the solvent.

The apparatus to the right on Figure 5-8 heats silicone oil to 107.5 ˚C, which circulate into a heating plate below the cups. Any leakages were prevented by ensuring proper ceiling between cups and the Soxtec. Cooling water was applied for both modes. Gas raises to the top of the glass cylinders as petroleum ether vaporizes. This gas is condensed by a constant flow of cooling water on top of the glass cylinder and drips down into the thimbles.

Petroleum ether was chosen as solvent because oil have a higher solubility in this solvent than its original phase. Petroleum ether have a low boiling point, 30 to 40 ˚C, which vaporizes rapidly at 107.5 ˚C.

The extraction cups were removed from the Soxtec system after the two modes, boiling and rinsing, where completed. Petroleum ether remnants were carefully vaporized using a heating plate at 50 ˚C. Cups containing oil only were then weighed again to determine OOC. Equation 2 was used to calculate OOC.

$$OCC, wet(\%) = \frac{W_2 - W_1}{W} \times 100 \ \%$$
(2)

Where,

W_1 = Weight of cups before extraction

W_2 = Extraction cup weight after extraction

W = Weight of DC

5.3   Retort Analysis

A Retort is an instrument used to determine liquid and solid content in a sample. In this thesis, all samples were DC. The liquid and solid composition of DC can be determined gravimetrically or volumetric in the Retort. Oil and water are separated from rock and clay in a distillation process. Retort heats the sample and vaporizes the liquid components. Oil and water are then condensed and collected in a cylinder(ExpotechUSA, n.d.) Materials used in a Retort analysis is listed in table 5-4.

Table 5-4: Retort analysis equipment list

Equipment	Model	Manufacturer
Retort kit	165-14-3	OFITE
Weight	KERN440	KERN & SOHN GmbH
Measuring cylinders	-	-

The liquid components were determined gravimetrically in this thesis. Sample holder was weighed both with and without DC and placed in the Retort cell (Figure 5-9)

Figure 5-9: Retort sample holder

Temperature was set to 481 ˚C to vaporize all liquid components in the DC. Steam and oil were condensed in the Retort condenser and collected in a measuring cylinder as shown in Figure 5-10. The duration of the analysis was approximately 50 minutes, or until no more liquids dripped from the condenser. Measuring cylinder containing oil and water was weighed both before and after collection. As water and oil are immiscible liquids, each fraction can be calculated. Water content was calculated using equation 3. Dry OOC describes percentage of oil on dry DC and was calculated using equation 4. Equation 5 was used to calculate OOC_{wet} with oil and water.

$$Water\ (\%) = \frac{M_{water}}{M_{wet}} \times 100\% \qquad (3)$$

$$OOC_{dry}(\%) = \frac{M_{oil}}{M_{wet} - (M_{oil} + M_{water})} \times 100\% \qquad (4)$$

$$OOC_{wet}(\%) = \frac{M_{oil}}{M_{wet}} \times 100\% \qquad (5)$$

M_{oil}= mass of oil in cuttings

M_{wet}= Mass of wet cuttings

M_{water}= Mass of water in cuttings

The advantage with a Retort analysis is that it also finds water content, which a Soxtec analysis cannot do. On the other hand, the Retort is less accurate at lower concentrations compared to the Soxtec.

Figure 5-10: Retort apparatus

6 Microwave technology from Norwegian Technology – Enhanced treatment of drill cuttings

A new microwave technology using organic susceptors have been developed by Norwegian Technology AS. This technology can offer great benefits in treatment of DC in terms of enhanced oil separation using less energy. The new microwave technology is a robust system with good maintainability and can potentially offer less downtime.

Organic susceptors have a significantly lower vaporization enthalpy compared to water, which is the medium absorbing microwaves in microwave treatment. In addition, the use of organic susceptor allow high enough process temperatures. This offers high treatment capacity in a microwave unit using susceptors as the evaporative mass transfer occur at a faster rate (Egar, 2017).

6.1 Microwave technology for drill cuttings treatment

Microwave technology is a known technology that has been applied both for commercial and industrial use. A microwave can heat components in a bulk selectively, as various substances react differently to microwaves. Hydrocarbons are separated from DC through evaporative mass transfer process(Ogunniran et al., 2017). Removal of hydrocarbons from DC using a microwave have been extensively researched by UON, which have achieved results below OSPAR regulations of 1% OOC using 80-90kWh/ton(Pereira, 2012).

The treatment of DC using the microwave technology used by UON is restricted by DC characteristics. The composition of oil and water is crucial as oil is removed by evaporative mass transfer with water. The results referred to above was obtained with DC that contained of 7.8% oil and 10.6% water(Pereira, 2012). As water and oil composition varies a lot for each type of DC it limits the application of this microwave technology.

6.2 Mechanisms for oil removal in microwave treatment

The mechanism for oil removal in microwave treatment has been discussed in various researches (Ogunniran et al., 2017; Pereira, 2012). The three mechanisms; entrainment, steam distillation and steam stripping have been studied and argued to be among the important mechanisms for oil removal on DC. In steam distillation the water reduces boiling point of hydrocarbons, which lead to the hydrocarbon phase is boiled. Entrainment removes

hydrocarbons physically through rapid evaporation of water. Steam stripping removes hydrocarbons through evaporation into a stripping gas.

A clear correlation between oil removal and steam velocity have been found(Ogunniran et al., 2017), which indicate that steam stripping is the dominant mechanism for removal of hydrocarbons . System A in Figure 6-1 represents a 1-5 kW microwave unit and System B represents a 40-100 kW microwave unit. System B obtain higher steam velocity due to a more powerful unit that evaporate water faster. The relationship between removal of hydrocarbons and steam velocity seems to be linear, which indicate that steam stripping is the dominant mechanism for oil removal.
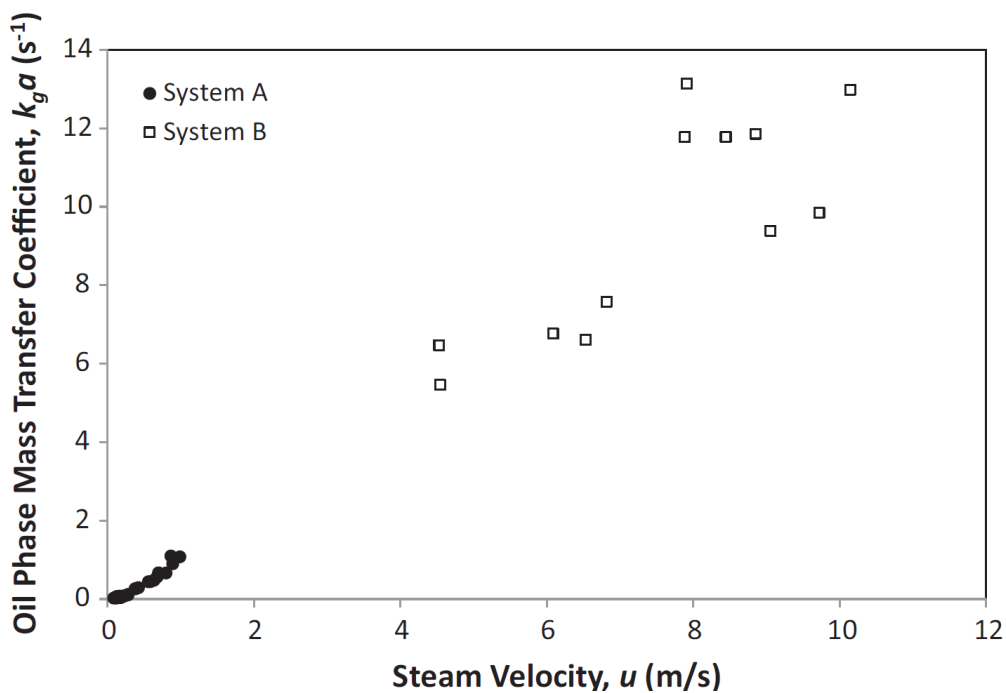

Figure 6-1: Correlation between steam velocity and oil removal (Ogunniran et al., 2017).

The high steam velocity is controlled by power density and particle size. Large particles give low steam velocity and the microwave technology would not perform efficiently in terms of oil separation This is due to low process temperature, which according to Dalton's law, yields limited oil separation(Egar, 2017).

Microwave generators capable of generating 55 kW or more each should be implemented in full-scale microwave treatment, based on a study from UON. The study tested microwave treatment of DC containing 7% OOC with a throughput of 300 kg/h and 800 kg/h at frequency of 0.896 GHz. To maintain a given energy input, a proportional energy increase was added when throughput of DC was increased. The increase of microwave energy resulted

in an exponential increase in oil separation, and 52% less energy was needed to achieve OOC below 1% (Pereira, 2012). Increased microwave power result in higher power densities and faster heating rates, which increase steam velocity and oil separation.

Powerful microwave generators do normally use a lower frequency such as 0.915 GHz. This frequency has a greater penetration depth compared to 2.45 GHz, which translates to greater treatment efficiency as a larger DC volume is treated simultaneously. The same study from UON tested the effects of two frequencies, 2.45 and 0.896 GHz. Testing of a DC with 7% OOC and a throughput of 300 kg/h for both frequencies. Lowering the frequency to 0.896 GHz reduced OOC below 1 % using 28% less energy than a frequency of 2.45 GHz(Pereira, 2012).

6.3   Norwegian Technology AS microwave technology

The treatment process of microwave radiation with susceptors (NTMW) is the brainchild of Norwegian Technology AS and consists of two treatment steps; microwave radiation (pre-treatment) and microwave radiation with a susceptor. The pre-treatment is based on normal microwave technology and is implemented to dewater the DC in order to add a susceptor. The susceptor treatment is the brainchild of Norwegian Technology AS, where chemicals are added to enhance oil separation. A schematic of the microwave unit is presented in Figure 6-2.
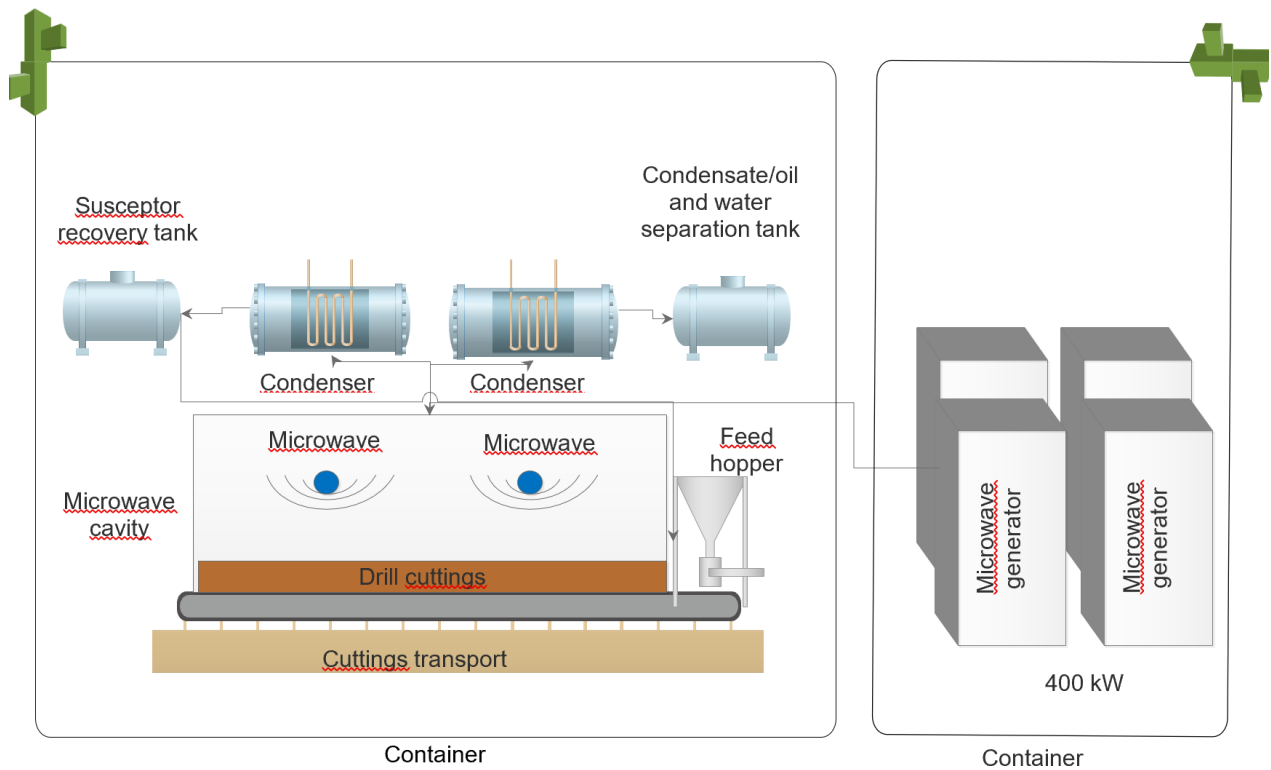
Figure 6-2: Schematics of Norwegian Technology microwave unit

The susceptor applied in the treatment must offer high process temperatures, recirculation and low impacts to the environment. MEG has been chosen as a suitable susceptor as it offers high enough process temperatures, little decomposition and are listed on OSPAR's PLONOR list (Rødne, 2018). Condensers can be used to remove water and oil in each treatment step. The condenser for susceptor treatment recovers evaporated MEG and recirculate it to a storage tank. Recirculation of MEG is of importance offshore as it can reduce footprints as less storage tanks are needed. In addition, recovery and reuse of MEG is of economic importance as it reduce the costs of running the unit over time.

MEG allows higher process temperatures than water as it has a boiling point between 196 to 198 C°(fisherscientific, n.d.). Higher process temperatures increase vapor pressure between MEG and oil which result in increased oil separation (Egar, 2017), and increases distillation rates of oil. The distillation rates of water and glycerol with oil are shown in Table 6-1 and Table 6-2, respectively.

Table 6-1: Boiling point, vapor pressure and distillation for oil distilled with water(Egar, 2017)

Distilled oil	Theoretical boiling point with water	Vapor pressure mmHg (oil/water)	Distillation ratio (ml oil /ml water)
Sipdrill (Tridecane)	100	7.3/758	1/9
Clarisol (Hexadecane)	100	0.57/758	1/90

Table 6-2: Boiling point, vapor pressure and distillation for oil distilled with glycerol (Egar, 2017)

Distilled oil	Theoretical boiling point with glycerol	Vapor pressure mmHg (oil/glycerol)	Distillation ratio (ml oil /ml glycerol)
Sipdrill (Tridecane)	228	636/125	17/1
Clarisol (Hexadecane)	265	457/300	6/1

The susceptor investigated in this thesis was MEG and offers a high treatment capacity. As ordinary microwave treatment can be limited by water content, NTMW can increase treatment capacity by substituting water with MEG. This is a very volatile chemical and has a significantly lower vaporization enthalpy compared to water (Figure 6-3) (Egar, 2017). Less energy is required to evaporate MEG on DC, leading to less energy consumed to achieve steam stripping and oil separation.
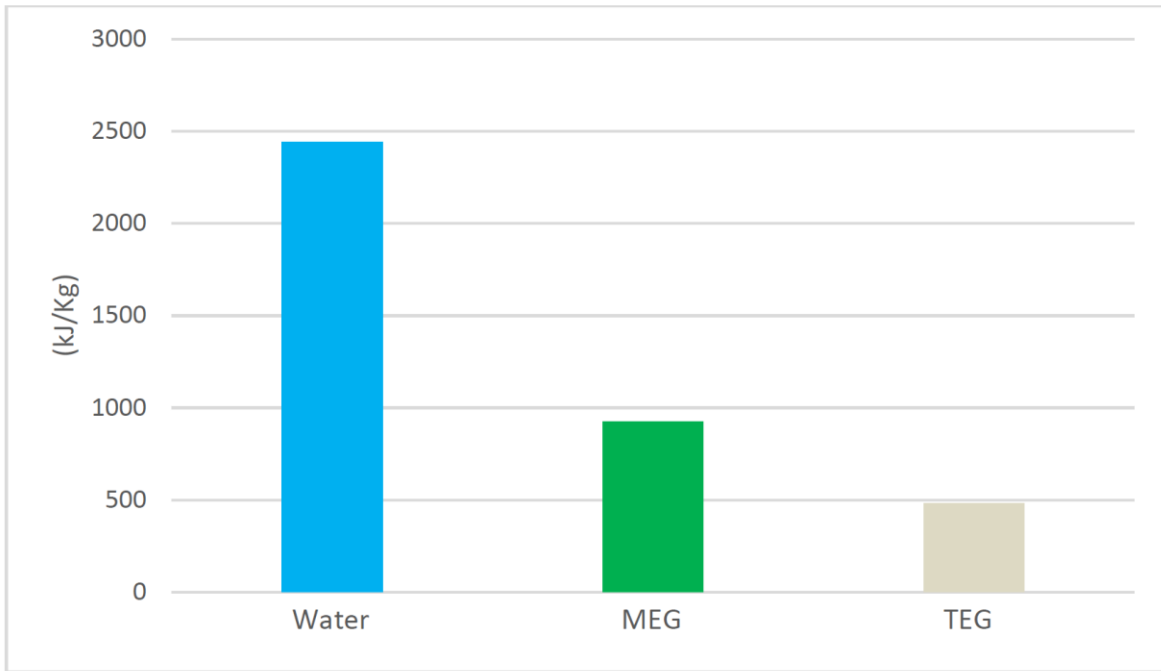
Figure 6-3: Enthalpies of water MEG and TEG(Rossi, 2016)

Solid susceptors have been used in industries to increase absorption of microwaves (Besson & Kappe, 2013). AC and silicon carbide (SiC) have been used as solid susceptor. Adding solid susceptors to MEG in susceptor treatment can enhance absorption of microwaves and heating of MEG. This can reduce energy consumption and treatment capacity because DC spend less time in the microwave unit. Granular AC mixed with MEG was investigated in this thesis.

Steam velocity is still of importance in NTMW, but the oil separation is less dependent on it as the process temperatures reaches 195C˚. Increased steam velocity is achieved as a result from MEG evaporating at a lower enthalpy. In addition, energy consumption is decreased using NTMW as microwave typically consume a large amount of energy to achieve an OOC below 1.5%.

7 Results & discussion

Chapter 7 presents results from several DC with different objectives in mind, the discussions are presented below results for each part objective. Important comments and parameters will also be discussed in the subchapters. Three different DC were treated in this thesis; one from Conoco Phillips 17-inch section from the NCS, and two from Husky Energy from Canada.

Since a lot of tests was performed, and each test have an abbreviation which explain what was done with the test. Table 7-1 explains the meaning of the abbreviations.

Table 7-1: Test abbreviations

Abbreviation	Meaning
CP	Conoco Phillips
HE	Husky Energy
G	MEG
H	Hot
AC	Activated carbon
N	N_2 stripping
P	Pre-heated DC

7.1   Drill Cuttings from Conoco Phillips

Norwegian Technology has developed the technology described in Chapter 6. The tested DC in this in chapter is from Conoco Phillips 17-inch section well (CP17), which is part of a joint industry project with Norwegian Technology. This DC wat thoroughly tested as it was the fundament for the understanding of the technology. Tests aimed to find possible optimizations of the technology.

Figure 7-1 is a visual representation of untreated CP17, and Table 7-2 show initial oil and water concentration of the DC.

Figure 7-1: Untreated CP17 DC.

The untreated DC consists of large agglomerates of particles. Oil and water surround the DC as a coat and the DC. The agglomerates are solid but is possible to deform by finger pressure.

Table 7-2: OOC and water values of CP17 from Retort analysis.

Parameters	Concentration [%]
$OOC_{Retort,dry}$	7.86
$OOC_{Retort,wet}$	6.21
Water	18.64

7.1.1   Treating drill cuttings from Conoco Phillips with microwave radiation

Raw DC was treated in the microwave unit. The microwave unit was set on full power (2kW), while variating treatment time. Therefore, the DC was exposed for variating energy inputs. The added power and treatment time do not correlate as reflective power vary at different treatment times.

Figure 7-2 is a visual representation of CP17 after microwave treatment, while Figure 7-3 is a visual representation of crushed CP17 after microwave treatment.

Figure 7-2: CP17 after microwave radiation.

The treated DC have the same characteristics before and after microwave treatment. The agglomerates maintain its form after treatment. There are some tendencies of pores from the water escaping from the agglomerates. The agglomerates are solid but possible to deform by finger pressure.

The large agglomerates of CP17 are crushed to its original size using finger pressure. The mineral composition and particle size were not measured accurately. Based on visual observation it seems to be graduated from clay to some larger particle that may be sandstone.

Table 7-3 shows water and oil separation from microwave radiation. Water separation in correlation with oil separation are plotted in Figure 7-4

Table 7-3: Pre-treatment results of CP17.

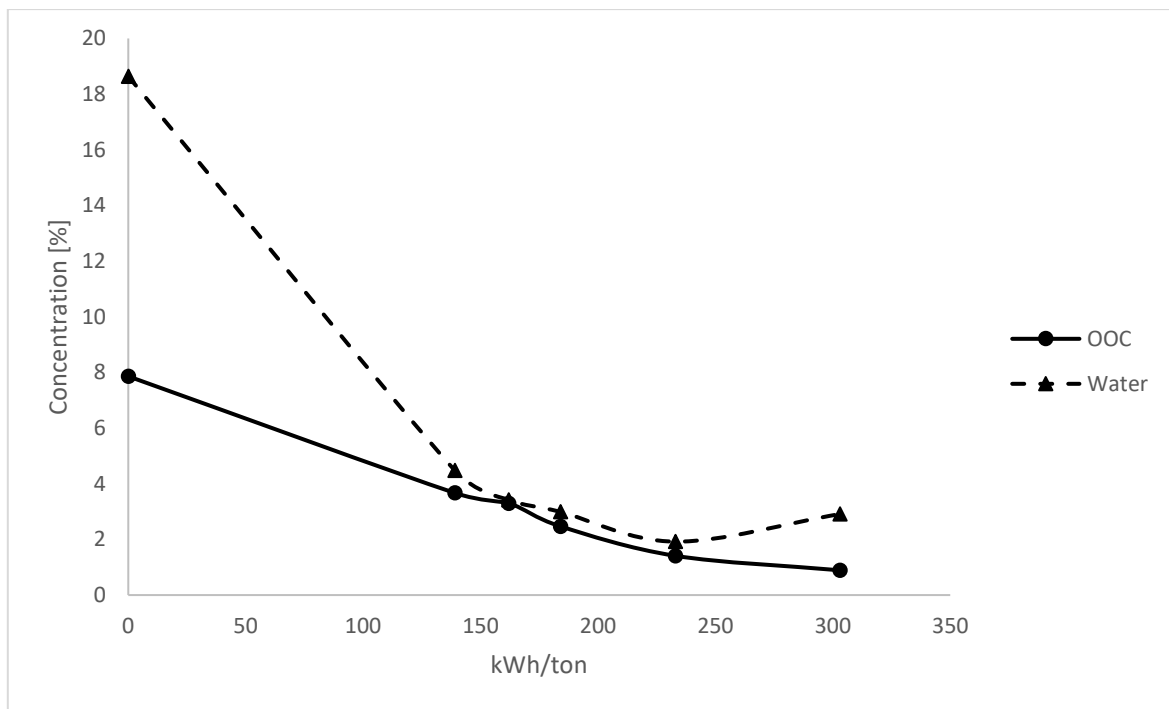| Sample	OOC$_{Soxtec}$ [%]	OOC$_{Retort, dry}$ [%]	Water [%]	kWh/ton	Time [s]
CP17.8	3.67	3.98	4.48	139	40
CP17.3	3.3	3.62	3.42	162	50
CP17.4	2.37	2.61	2.99	184	60
CP17.1	1.41	1.71	1.92	233	84
CP17.18	0.89	0.83	2.91	303	120

Figure 7-4: Correlation between water and oil separation after microwave radiation.

Figure 7-5 and Figure 7-6 show the reflected power of 40- and 84-seconds of microwave treatment, respectively.
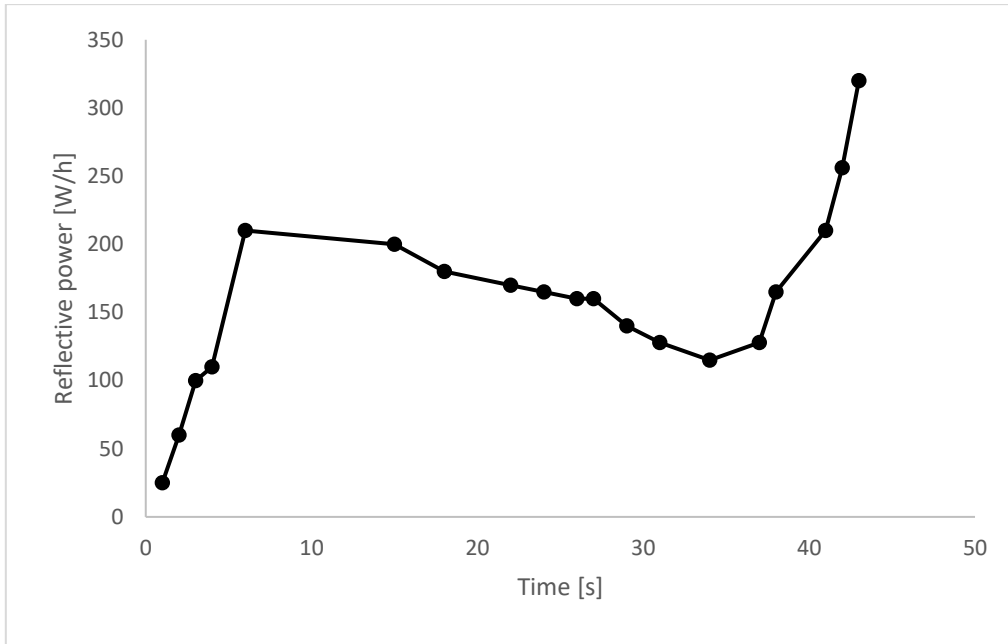
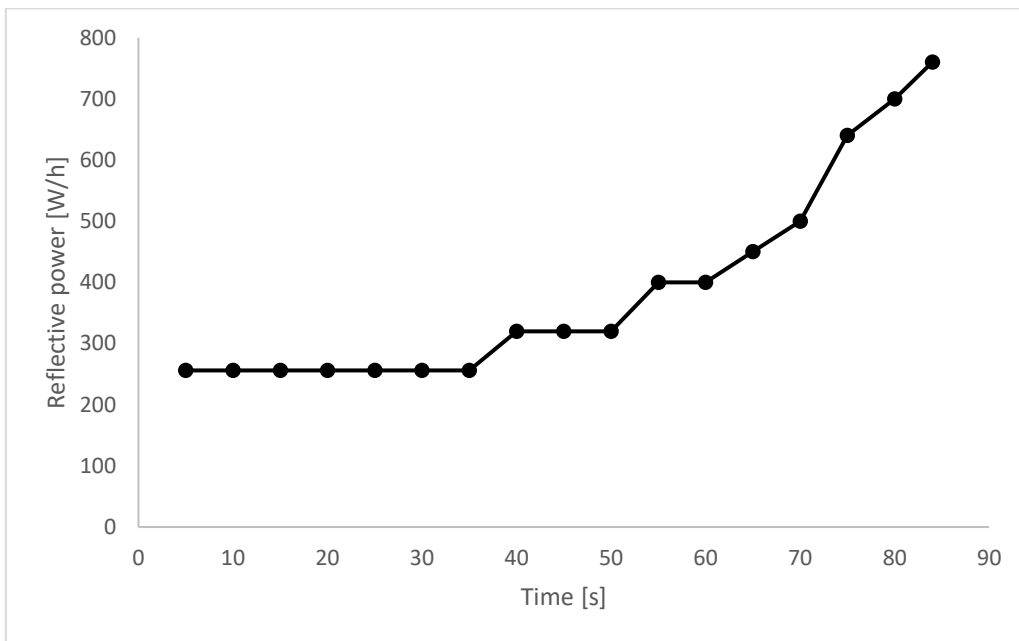*Figure 7-5: CP17 reflected power from 0 to 40 seconds.*

Figure 7-6: CP17 reflected power from 0 to 84 seconds.

Discussion

Figure 7-4 shows that CP17 seem to be relative linear with respect to oil separation. The energy consumption seems to be relatively high considering the decrease of OOC. This relates to high water concentration compared to relatively low oil concentration, as oil-to-water ratio is 0.42.

The curve for water separation in have an u-shaped form (Figure7-4). The curve is most likely to continue to decrease from 140 kWh/ton because of decreasing water concentration in the DC. Capillary forces in tight bound water in the DC do also influence energy consumption. Therefore, the remaining water can be more energy demanding to separate from the DC.

The water concentration starts to increase around 240 kWh/ton (Figure 7-4). As DC is treated and obtain relatively low water concentration, the DC absorbing water from the atmosphere after treatment. Therefore, the water curve is considered inaccurate after 150 kWh/ton. Improvements of water separation could be obtained using an exicator.

The water curve is of importance as it relates to separation of MEG. Dosing of MEG should be performed after dewatering, or when the microwave treatment of DC starts to work ineffectively and consume significant amounts of energy. The OOC curve in Figure 7-4 show a tendency to level out from 240 kWh/ton to 310 kWh/ton. Based on the OOC curve, a MEG dosing point might be detected. With respect to separating water from MEG as illustrated in the process drawing in Figure 6-2, the dosing of MEG might also take place around 170 kWh/ton. This is based on the motivation to separate MEG and water.

The reflective power curve from 0 to 40 seconds show an increase in reflective power from 0 to 8 seconds. This is most likely a result of the microwave unit startup as it takes approx. 7 seconds to reach 2 kW input power that was set in this test, which effects the reflective power curve as less energy is reflected (Figure 7-5).

After 40 seconds, the reflective power increases, which is most likely a result the DC heating up, which leads to less microwave energy absorbance. The fact that water starts to evaporate, and less water is present in the DC, also increase reflected power. The curve in Figure 7-6 indicate that water is evaporation consistently. However, this observation might not be true as there are several potential reasons for steadily increase of reflective power.

Considering microwave technology as a standalone technology for CP17, it shows that a significant increase in energy consumption is required to reduce OOC below 1%. Achieving OOC below 0.5% might not be possible for this DC. This might be due to particle distribution in the DC, as the stripping process depend on high steam velocity and large particle are unfavorable for this.

7.1.2 CP17 - Optimization of oil separation and energy consumption using susceptor

Based on the findings and discussion in Chapter 7.1.1, dosing of MEG should be performed in the energy consumption range of 170-240 kWh/ton. The various optimization parameters are investigated in this section.

Figure 7-7 shows CP17 reaction to MEG. Image "1" in Figure 7-7 show an agglomerate of CP17 where MEG is added, and Image "2" show the same agglomerate of CP17 that is split in half.

Figure 7-7: CP17 absorption of MEG.

MEG seems to surround the DC as a coat, and do not penetrate though the whole agglomerate but is adsorbed in some of the cavities.

7.1.2.1 Dosing Cold MEG on cold DC

CP17 was treated in the microwave as in Chapter 7.1.1. Then MEG at room temperature was added to the DC and mixed relatively homogeneously before the next treatment, susceptor treatment.

Table 7-4 represent the result from treatment with DC and MEG at room temperature.

Table 7-4: Results from cold DC treatment with cold MEG.

Sample	OOC$_{Soxtec}$ [%]	OOC$_{Retort,dry}$ [%]	Water/MEG [%]	kWh/ton	MEG [ml]	Time [s]
CP17.2G	0.67	0.96	5.97	233+143	20	84+48

* Pre-treatment is written before other treatment in kWh/ton and time

<u>Discussion</u>

The DC was cooled down after pre-treatment. Cold MEG was added under ideal conditions, which means MEG was mixed relatively homogeneously into the DC and mixed homogeneously. Added MEG indicate that 20 ml MEG was enough to achieve sufficient oil separation with the amount energy consumed.

In table 7-4, the MEG evaporated using 143 kWh/ton with a treatment time of 48 seconds in the microwave. The Retort analysis indicate that a significant amount of MEG might remain in the DC sample, and further treatment could potentially be performed.

Considering oil separation, 143 kWh/ton was used to reduce OOC from 1.41% to 0.67%. Remaining oil is challenging to remove, and a significant amount of energy is used in the second oil treatment step.

7.1.2.2   Dosing cold MEG on hot DC

To reduce energy consumption for oil separation with MEG, the susceptor was dosed when the DC remained hot from the microwave pre-treatment. Table 7-5 presents the results from cold MEG dosed on hot DC, and Figure 7-8 shows a comparison of cold and hot DC.

Table 7-5: Results from cold MEG dosed on hot DC.

Sample	OOC$_{Soxtec}$ [%]	OOC$_{Retort,dry}$ [%]	Water/MEG [%]	kWh/ton	MEG [ml]	Time [s]
CP17.5G	0.11	0.19	2.31	233+185	20	84+60
CP17.10G	0.69	0.87	4.80	139+149	20	40+48
CP17.11G	0.61	0.45	3.58	139+166	20	40+60
CP17.12G	0.52	0.43	2.56	162+166	20	50+60
CP17.17G	0.35	-	-	162+218	20	50+84

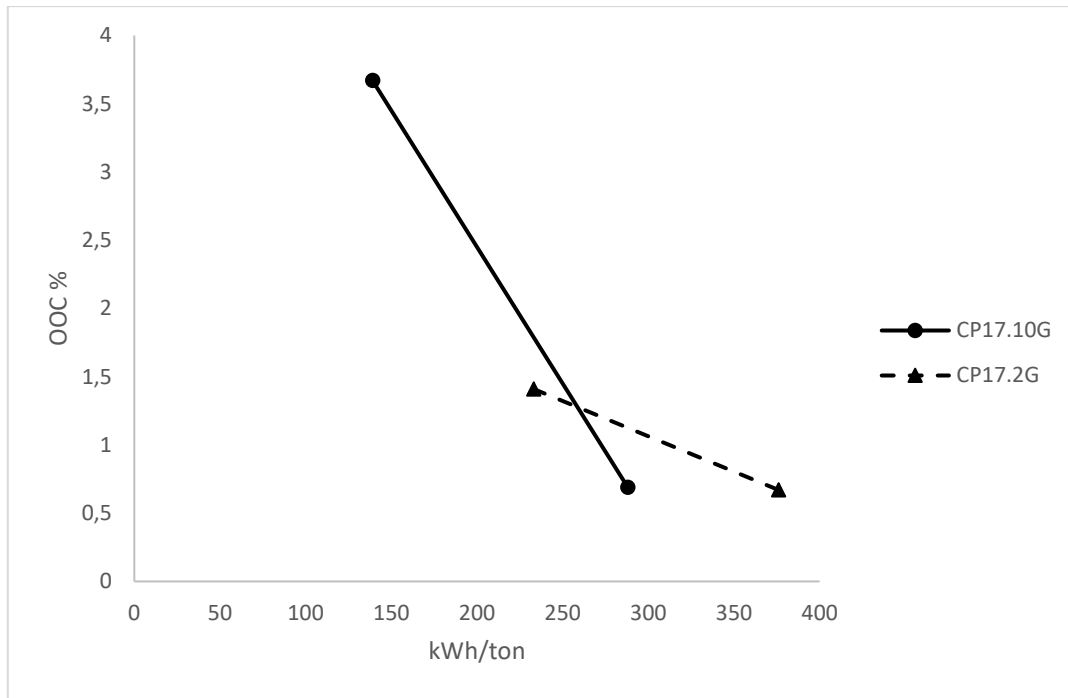* Pre-treatment is written before other treatment in kWh/ton and time

Figure 7-8: Comparison of hot and cold DC.

The best result from in 6-4 was sample CP17.5G and achieved an OOC of 0.11%, which is an oil separation of 98.7%.

Discussion

The sample achieved good results, reducing OOC from 1.41% to 0.11% using 185 kWh/ton. The oil concentration is relatively inhomogeneous, and each sample will slightly differ from each other. CP17.5G is most likely a sample fraction with low OOC, as it got very high oil separation. However, this is not necessarily true as the sample consumed a relatively large amount of energy.

Figure 7-8 shows the importance of DC temperature before susceptor treatment. Sample CP17.10G was added MEG and treated immediately after pre-treatment, while CP17.2G was cooled down to room temperature before MEG treatment.

CP17.2G and CP17.10G removed 52.4% and 81.1% of remaining OOC after pre-treatment, respectively. The samples with hot and cold DC from Figure 7-8 achieved approx. 0.7% OOC, but the sample with hot DC consumed less energy doing so. The slope gradient and oil separation of CP17.10G indicates a more efficient oil separation when MEG is added to hot DC. Hot DC is energetically favorable as cold DC demanded 23.5% more energy to reach as good OOC % as hot DC.

7.1.2.3   MEG volume

Finding a suitable volume of susceptor to dose on DC is important concerning treatment efficiency and costs. Susceptor volumes of 10,20,30 and 50 ml MEG was tested on the DC.

Table 7-6 presents the results from MEG volumes of 10-, 20-, 30- and 50-ml. Figure 7-9 shows reflective curves of 30- and 50-ml MEG, while Figure 7-10 presents reflective curves of 10- and 20-ml MEG.

Table 7-6: Results of various MEG volumes.

Sample	OOC$_{Soxtec}$ [%]	OOC$_{Retort,dry}$ [%]	Water/MEG [%]	kWh/ton	MEG [ml]	Time [s]
CP17.7G	1.01	1.28	6.39	196+127	50	63+39
CP17.6G	0.60	0.60	3.60	184+145	30	60+46
CP17.9G	1.06	1.46	3.34	139+160	10	40+60
CP17.11G	0.61	0.45	3.58	139+166	20	40+60

* Pre-treatment is written before other treatment in kWh/ton and time
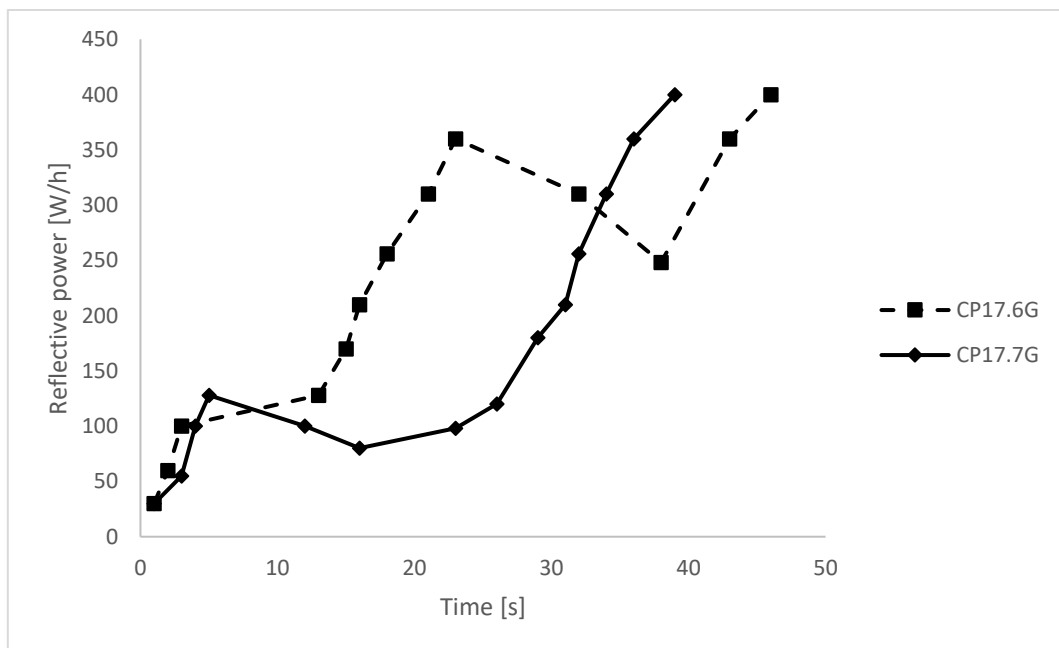
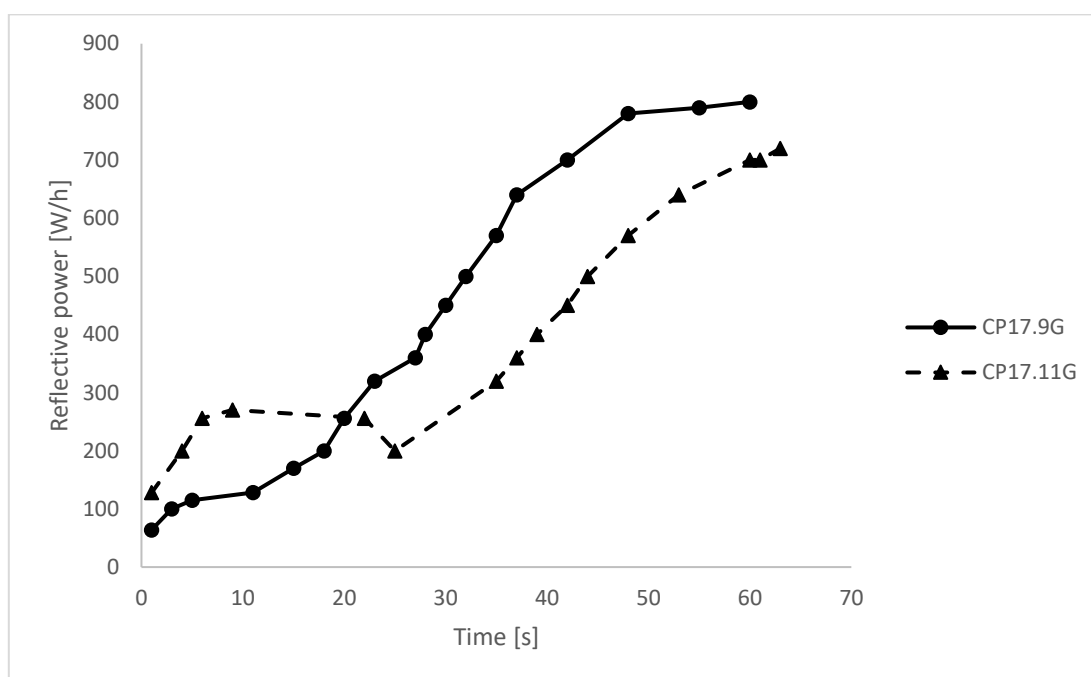*Figure 7-9: Comparison of 30- and 50-mL MEG.*

Figure 7-10: Comparison of 20- and 10-mL MEG.

Discussion

Figure 7-9 shows that CP17.7G absorbs more microwaves than CP17.6G from 0 to 30 seconds. This is most likely because more MEG is added which lead to more absorption of microwaves. The rapid increase in the curves from 0 to 8 seconds is due to microwave startup, as discussed in Chapter 7.1.1. Then the reflective curves level out as MEG is absorbing microwaves.

The curves start to increase at different times, most likely due to variating MEG volume (Figure7-9). Therefore, less added MEG result in a faster evaporation of the volume MEG added. Steadily increasing curves indicate that MEG is boiling on the DC[1]. CP17.7G and CP17.6G reduced oil to 1.01% and 0.60% OOC, respectively. CP17.7G needed more energy to boil MEG compared to CP17.6G. In addition, 50 ml demands more energy to achieve as good oil separation compared to 30 ml, this is most likely because more treatment time is needed to evaporate a larger volume of MEG. As both curves are still increasing at the end of the treatment, it is reasonable to believe that MEG remains in the DC after treatment and further treatment could have been applied.

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[1] From discussion with John Robinson (University of Nottingham) as he visited the University of Stavanger.

CP17.9G and CP17.11G reduced OOC from 2.37 % to 1.06% and 0.61%, using 160 kWh/ton and 166 kWh/ton respectively (Table 7-6). This indicate that 20 ml MEG was more efficient in oil separation as energy consumption was approx. the same. A volume of 10 ml MEG seems to be insufficient in high oil separation, this might be because smaller volume was more difficult to distribute onto the sample and less MEG soaked the DC.

A volume of 20- and 30-ml MEG seems to be to be the most successful tests. As CP17.6G and CP17.11G achieved oil separation below 1% OOC. Various treatment times was implemented, and CP17.7G would most likely achieved OOC below 1% if more treatment was applied.

7.1.2.4 MEG temperature

Based on results and discussion in section 7.1.2.3, MEG volume should be between 20 to 30 ml. Heating of MEG before treatment was investigated to increase treatment efficiency as it might reduce the time for MEG to evaporate in the sample.

Table 7-7 presents results from MEG pre-heated between 120 and 160 C˚. Figure 7-11 represents a comparison of treatment with hot and cold MEG.

Table 7-7: show samples with pre-heated MEG at different temperatures.

Sample	OOC$_{Soxtec}$ [%]	kWh/ton	MEG [ml]	MEG temp. [C˚]	Time [s]
CP17.14GH	0.63	162+110	20	120	50+40
CP17.15GH	0.31	162+154	20	120	50+60
CP17.16GH	0.27	233+87	20	120	84+30
CP17.19GH	0.52	233+90	25	160	84+30
CP17.23GH	0.37	233+153	25	130	85+50
CP17.12G	0.52	162+166	20	-	50+60

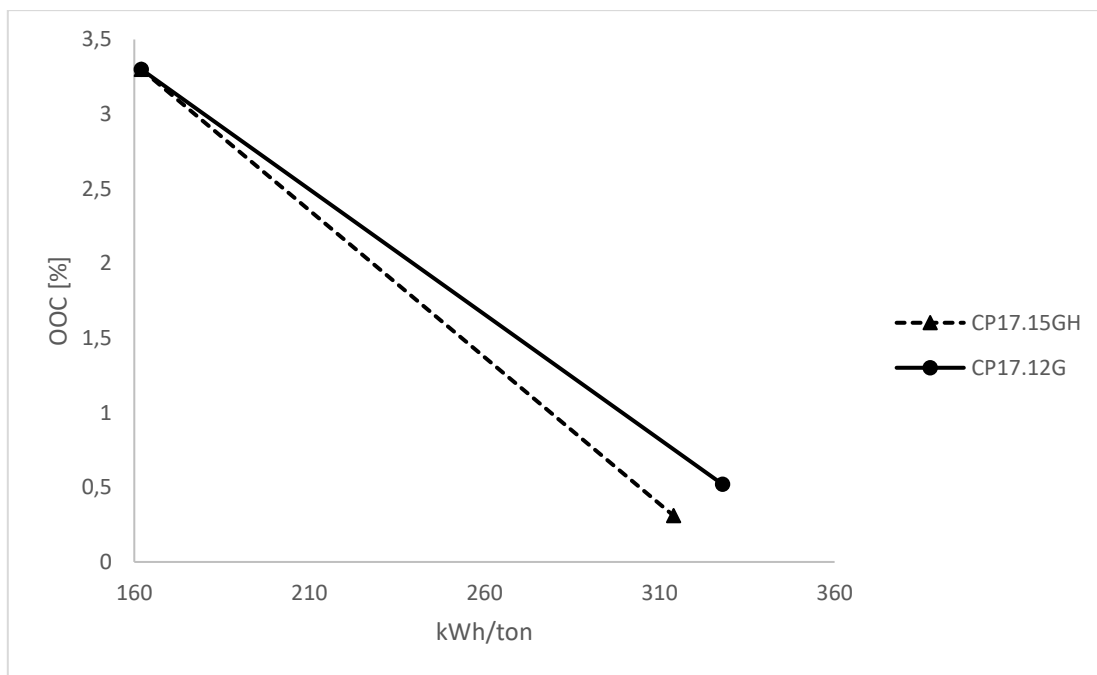*Pre-treatment is written before other treatment in kWh/ton and time

Figure 7-11: Comparison of hot and cold MEG.

Discussion

Figure 7-11 shows that heated MEG was an advantage for both separation of oil and energy efficiency. CP.15GH was compared to CP17.12G which obtained an OOC of 0.31% and 0.52%, respectively. The samples had equal treatment times, but heated MEG removed hydrocarbons more efficiently as it achieved lower OOC and energy consumption. This is most likely because heated MEG spends less time to reach high enough temperature to evaporate, which lead to stripping effect. However, cold MEG might achieve as low OOC as hot MEG but would most likely require more treatment time and energy. Also, the increased oil separation can be due to decrease in MEG viscosity when it is heated. Viscosity of MEG is reduced as it is heated, which enable MEG to be more homogeneously distributed in DC pores and surfaces.

In Table 7-7 CP17.16GH and CP17.15GH reduced OOC to 0.27% and 0.31%, respectively. The samples were treated with different setting in microwave and susceptor treatment, which indicate that different treatment combination can be applied.

Table 7-7 indicates that hot MEG is an advantage in terms of oil separation and energy consumption. Therefore, CP17.19GH was expected to be one of the best results as MEG was heated to 160 C˚. Results from Table 7-7 indicate that heating MEG from 120 C˚ to 160 C˚ did have a negative effect in oil separation. This is most likely because the tests were performed manually, and available equipment made it difficult to handle MEG at high

temperatures, leading to errors in sample CP17.19GH. Implications that might have reduced validity of CP17.19GH is inaccurate MEG dosing, and cooldown of DC and MEG as it took time to dose the MEG.

7.1.2.5 *Activated carbon with MEG*

Based on the results in section 7.1.2.4, MEG should be heated to 120 to 160 C° as it results in a more rapid evaporation of MEG. In order to achieve even faster evaporation of MEG, AC was added as a second susceptor.

Table 7-8 represents results of AC mixed with hot and cold MEG. Figure 7-12 presents a comparison of cold MEG with and without AC, and Figure 7-13 represents a comparison of hot MEG with and without AC.

Table 7-8: Testing of AC.

Sample	OOC$_{Soxtec}$ [%]	Water/MEG [%]	kWh/ton	MEG [ml]	MEG temp. [C°]	Time [s]
CP17.13GAC	0.45	3.54	162+142	20	-	50+40
CP17.21GHAC	0.29	-	233+102	25	120	84+30
CP17.12G	0.52	-	162+166	20	-	50+60
CP17.16GH	0.27	-	233+87	20	120	84+30

*Pre-treatment is written before other treatment in kWh/ton and time
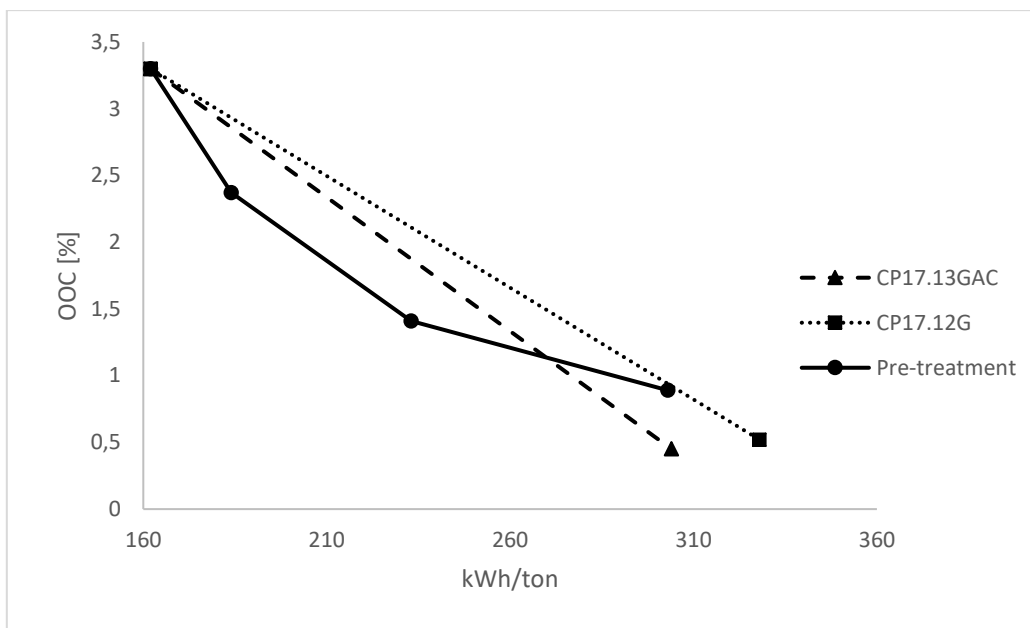
Figure 7-12: Effect of AC with cold MEG.
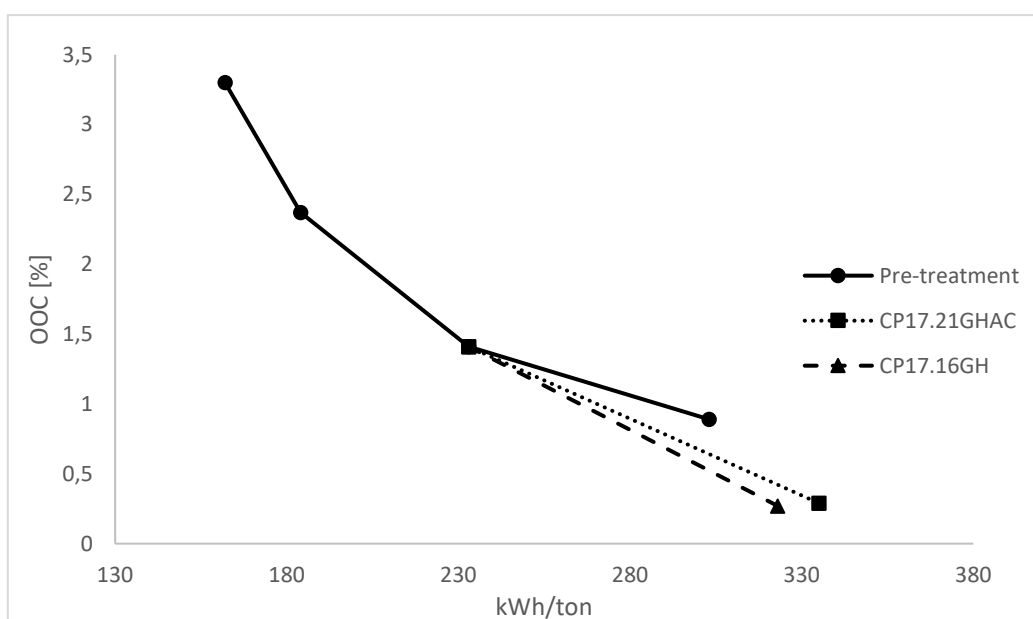
Figure 7-13: Effect of AC with hot MEG.

Discussion

Figure 7-12 shows that AC with cold MEG seems to increase oil separation and decrease energy consumption. CP17.13GAC was added MEG at room temperature with AC and achieved an OOC of 0.45%, while CP17.12G obtained an OOC of 0.52%. The DC with AC treatment also, spent less time in the microwave. The AS was most likely increasing microwave absorption and it seems like MEG evaporated at a faster rate. Therefore, obtaining a lower OOC%. CP17.12G separated less oil from DC using more energy in the second

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treatment step. Using AC in combination with cold MEG improved oil separation with 13.5%. CP17.13GAC and CP17.12G performed worse than microwave treatment from 0 to 270 kWh/ton, which indicate that MEG had to be heated and evaporate before improving oil separation and energy consumption.

Figure 7-13 indicates that AC was less effective in combination with hot MEG. MEG was heated before treatment, and MEG did not need much additional heating before evaporating from the DC. Therefore, the effect of AC was insignificant. CP17.16G was slightly performing better in terms of energy compared to CP17.21GHAC. This is most likely due to increased microwave absorption from AC and 5 ml extra MEG was added in CP17.21GHAC. AC did not prove to have a significant effect on heated MEG.

As hot MEG improved oil separation and energy consumption and AC showed little effects, it might be desirable to avoid AC at this type of DC. Adding AC to the process might be another factor to cause other problems, e.g. in a condenser. Also, evaporated MEG may also contain impurities of AC after treatment, and a special condenser would be needed in a pilot- and full-scale installation to preserve MEG for reuse.

7.1.2.6 N_2 stripping after microwave treatment

N_2 gas have been used in all microwave tests to create an inert environment. Potential increased oil separation with N_2 gas as sweeping gas was examined in this section.

Table 7-9 represents results with N_2 stripping, and Figure 7-14 is a visual comparison of the sample cylinder with DC with and without N_2 stripping. Figure 7-15 shows oil separation with and without N_2 stripping.

Table 7-9: Comparison of samples with and without N2 stripping

Sample	OOC$_{Soxtec}$ [%]	kWh/ton	MEG [ml]	MEG temp. [C°]	Time [s]	N$_2$-stripping [15L/min]
CP17.20GHN	0.35	233+86	18	160	84+30	2 min
CP17.22GHACN	0.40	233+95	20	160	84+30	2 min
CP17.19GH	0.52	233+90	25	160	84+30	-

*Pre-treatment is written before other treatment in kWh/ton and time

Figure 7-14: N2 stripped sample to the left

After visual control of treatment, it seems like evaporated liquids tend to condensate at surfaces of the sample cylinder and DC. The DC and sample cylinder that were stripped with 15 L/min N_2 gas for two minutes looked significantly drier (Figure 7-14).
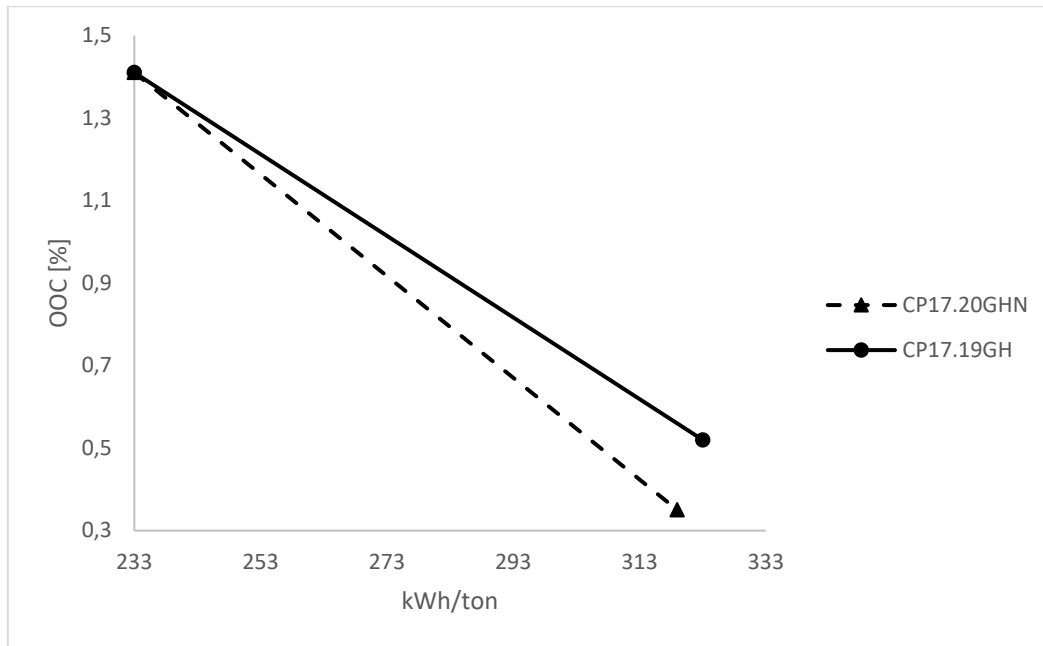
Figure 7-15: Effect of nitrogen gas

Discussion

Figure 7-15 shows that N_2 stripping increased oil separation in the DC. This might be because of the shape of sample cylinder, which enables N_2 gas to efficiently channel evaporated gases out of the DC. This might not be the case in full-scale installations as a different unit configuration would be used.

Figure 7-15 shows 32.7% better oil separation using N_2 as stripping gas in combination with heated MEG. However, energy consumption is not be affected by the stripping as N_2 gas as it was stored in a pressurized tank. To have enough pressurized N_2 gas available at an oil rig is another cost that would have to be considered before implementing N_2 stripping to a full-scale unit.

Inefficient dosing of MEG and undesirable cooling of DC lead to errors when N_2 stripping was combined with AC and heated MEG. N_2 stripping show a negative effect. However, small errors in tests can be significant to the results at this stage of the optimization.

7.1.2.7 Pre-heating of drill cuttings before treatment

In this section, the DC was heated to 70 C° to simulate offshore conditions. Raw DC was heated in a water bath before treatment. Table 7-10 shows the result of heating the DC, and Figure 7-16 represents a comparison of microwave treatment with cold and pre-heated DC.

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Table 7-10: result from pre-heating DC before microwave treatment.

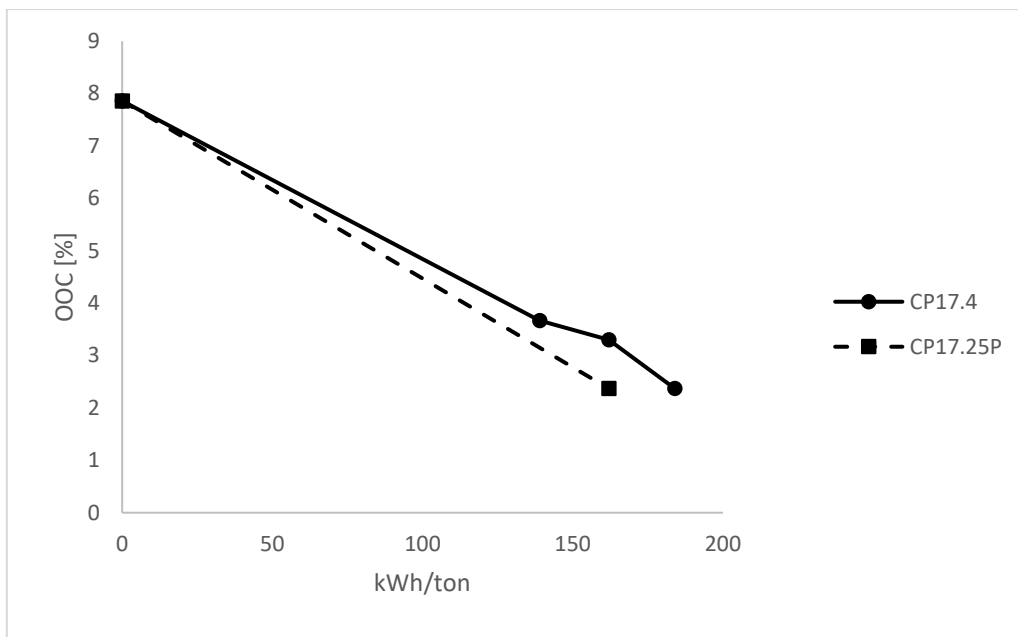| Sample	OOC$_{Soxtec}$ [%]	kWh/ton	Time [s]	DC temp. [C°]
CP17.25P	2.37	162	58	70
CP17.4	2.37	184	60	-

Figure 7-16: Comparison of cold and hot DC before microwave treatment

Discussion

The curve with pre-heated DC shown in Figure 7-16 to have a slightly steeper curve. This might be because water evaporate at a faster since it pre-heated to 70 C° and had a head start. The cold DC curve must be heated from room temperature, and evaporating the water takes longer. Hence, more energy is consumed. Pre-heated DC resulted in 4.1% better oil separation and 11.0% energy savings by comparing CP17.25P and CP17.4.

7.1.2.8 Best set-up for treatment of Conoco Phillips drill cuttings

Based on the optimization parameter discussed in this chapter, the best set-up was investigated implementing all the parameters found in this thesis. Table 7-11 represents the

best set-up found in this thesis for treatment of CP17. Figure 7-17 illustrates the difference between ordinary microwave treatment and susceptor treatment.

Table 7-11: Results from optimization of CP17.

Sample	OOC_{Soxtec} [%]	kWh/ton	MEG [ml]	MEG temp. [C°]	Time [s]	N₂-stripping [15L/min]	DC temp. [C°]
CP17.24PGHACN	0.55	162+85	25	130	58+30	2 min	70
CP17.16GH	0.27	233+87	20	120	84+30	-	-
CP17.18	0.89	303	-	-	120	-	-

*Pre-treatment is written before other treatment in kWh/ton and time
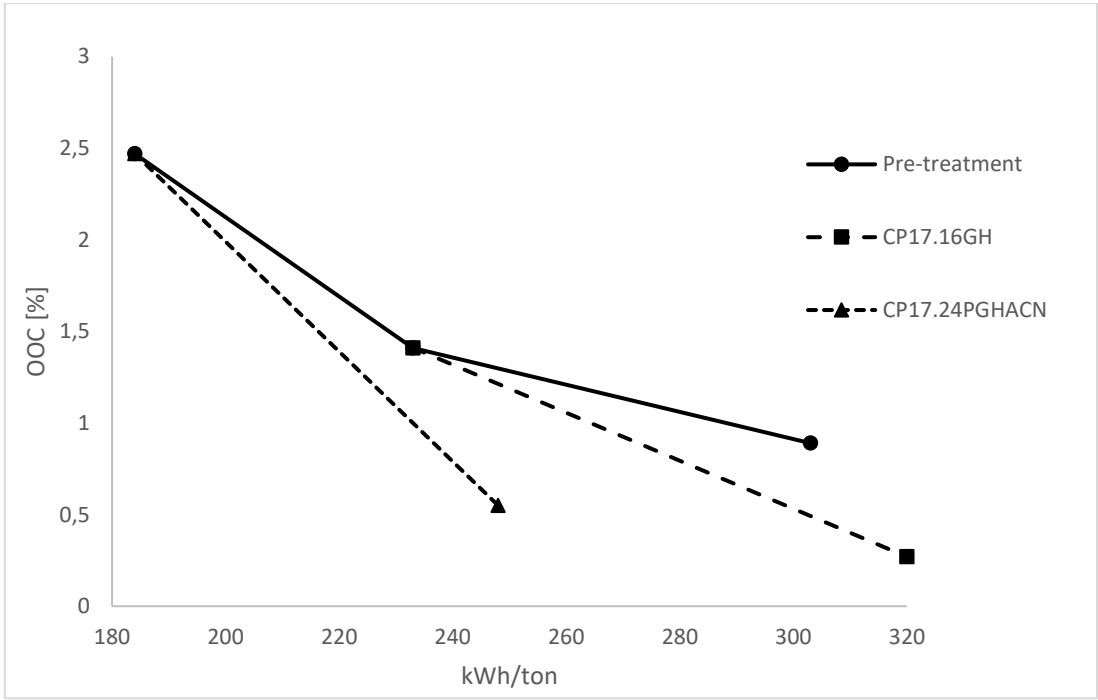
Figure 7-17: Comparison of microwave treatment and NTMW treatment

The best set-up of treatment with susceptor achieved 38.3% lower OOC, while simultaneously saving 18.2% energy consumption compared to pre-treatment or microwave treatment.

<u>Discussion</u>

The best set-up includes pre-heating of DC, which is not performed for the pre-treatment curve in Figure 7-17, which would adjust the curves in favor of microwave. However, pre-heated DC did not show significant improvements for oil separation in microwave treatment (Figure 7-16).

CP17.16GH is added to emphasize the advantage of treatment with susceptors, as this test also was run without pre-heated DC.

7.2   Treating drill cuttings world wide

DC extracted outside of NCS was treated with microwave and susceptor technology to investigate the technology's potential worldwide. Two DC from Husky Energy was tested and analyzed. The DC had different characteristics and was separated by the given names, Husky Energy 1 (HE1) and Husky Energy 2 (HE2). The experience from CP17 was applied for the DC from Husky Energy.

7.2.1   Husky Energy 1

Raw HE1 is visually presented in Figure 7-18, and initial oil and water concentrations are shown in Table 7-12.
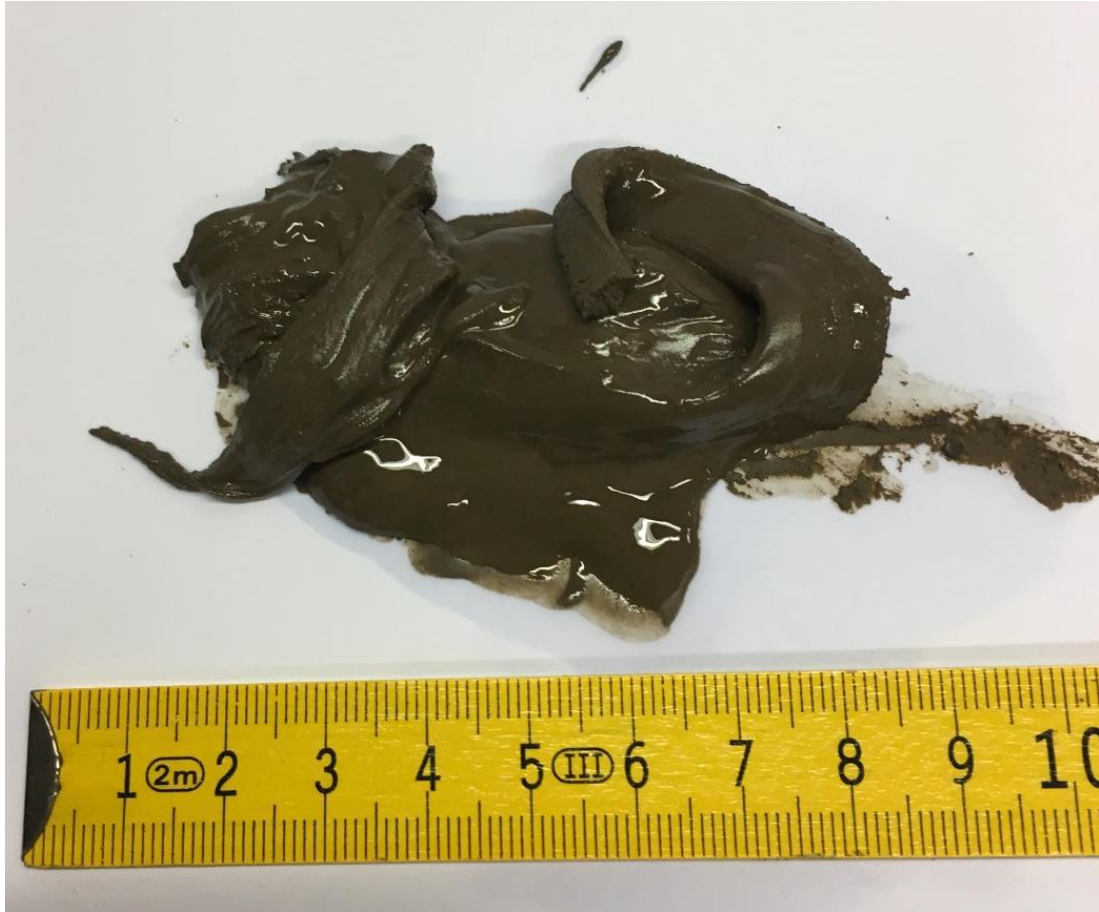
Figure 7-18: Untreated HE1

HE1 is like a sludge due to excessive concentrations of mud. The DC was soft and easy to shape and brake by finger pressure.

Table 7-12: OOC and water concentration of HE1

Parameter	HE1
$OOC_{Retort,dry}$ [%]	9.81
$OOC_{Retort,wet}$ [%]	8.59
Water [%]	4.46

7.2.1.1   Treating Husky Energy 1 with microwave radiation

HE1 was centrifuged before pre-treatment and was named HEC1. The characteristics of HE1 proved to be difficult to distribute in the sample cylinder, therefore the DC was centrifuged before microwave treatment.

Raw HE1 was treated in the microwave unit and was set on full power (2kW), while variating treatment time. Therefore, the DC was exposed to variating energy inputs. Table 7-13 represents water content and OOC after centrifugation and microwave treatment. Centrifuged and microwave treated HEC1 is visually presented in Figure 7-19. Figure 7-20 represents OOC and water content of the microwave treatment.

Figure 7-19: HEC1 after treatment

The treated HEC1 formed a hard and porous structure in the sample cylinder that had to be excavated. The treated DC consist of small particles and larger agglomerates. Pores in the agglomerates could be spotted, as water was evaporated.

Table 7-13: Centrifuged and microwave treated HE1.

Sample	OOC$_{Retort, dry}$ (%)	Water (%)	kWh/Ton	Time [s]
HEC1	8.01	4.05	-	0
HEC1.1	7.14	1.79	114	40
HEC1.2	5.52	1.66	160	60
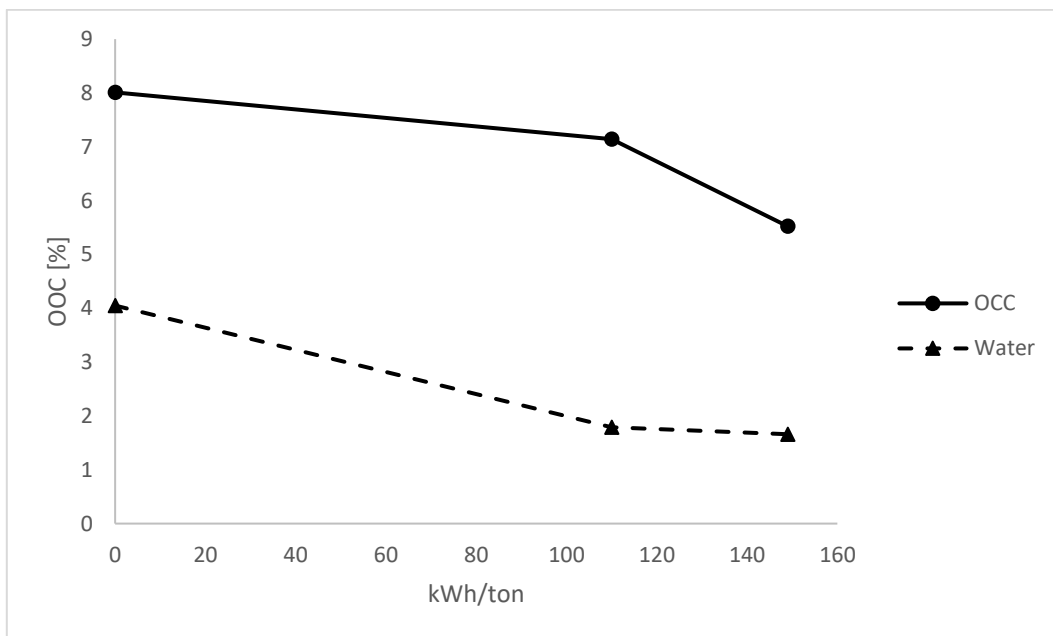
Figure 7-20: OOC and water content in HEC1

Pre-treatment reduced water below 2 % after 40 seconds.

Discussion

A MEG dosing point was spotted at 110 kWh/ton as the water curve levelled out. Indicating that further treatment would not reduce OOC significantly. The OOC was not significantly reduced, this is most likely due to a low water-to-oil ratio. Therefore, it was considered that further pre-treatment was not necessary.

 The oil separation was poor, this might be because of low stripping effect because of low water content. Therefore, MEG should be to increase potential stripping effect and oil separation.

Based on the findings and discussion in section 7.2.1.1, MEG should be dosed at 110 kWh/ton. Three optimized tests for HEC1 was performed and are shown in Table 7-14. A comparison of microwave treatment and susceptor treatment are presented in Figure 7-21. Pre-treatment of 40 seconds was applied, based on results in Table 7-11. As different volumes of MEG were applied, the reflective curves are compared in Figure 7-22.

Table 7-14: Results from optimization of HEC1

Sample	OOC$_{Soxtec}$ [%]	kWh/ton	MEG [mL]	MEG temp. [C°]	Time [s]	N$_2$-stripping [15L/min]
HEC1.4GHN	2.27	104+152	35	120	40+50	2 min
HEC1.5GHN	2.08	104+134	20	120	40+50	2 min
HEC1.6GHACN	2.26	104+119	20	120	40+50	2 min

*Pre-treatment is written before other treatment in kWh/ton and time
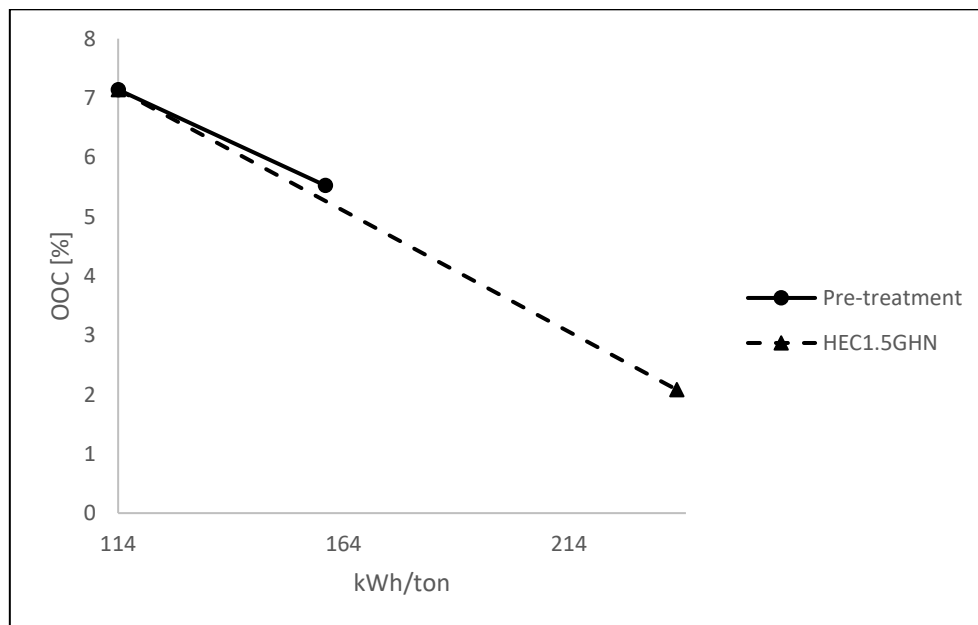
Figure 7-21:Best result of HEC1 against pre-treatment

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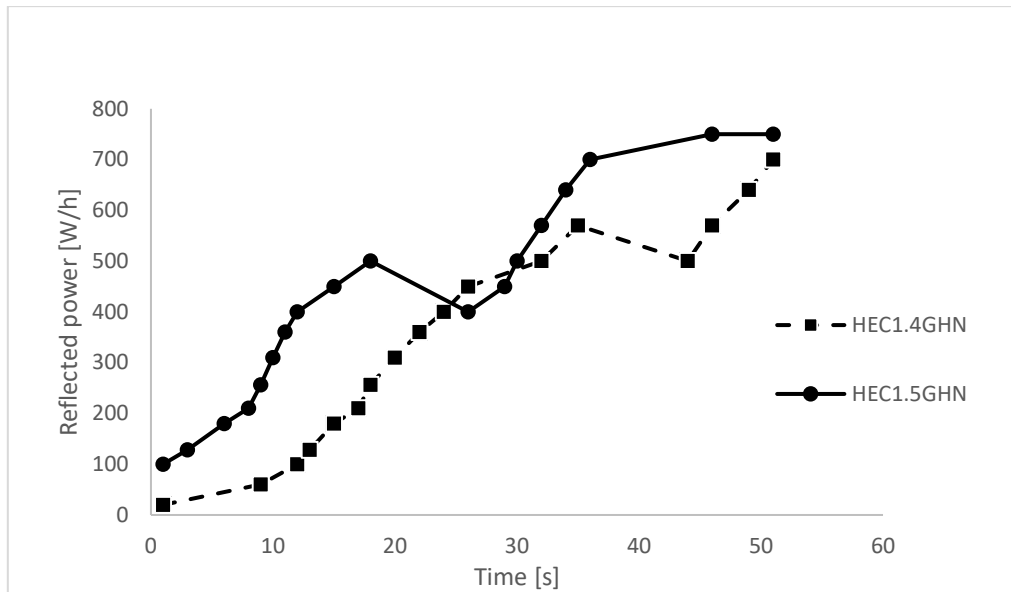
Figure 7-22: Comparison of reflective curve of HEC1.4GHN and HEC1.5GHN

Discussion

HEC1.5GHN was the best set-up and achieved an OOC of 2.08 using 238 kWh/ton (Figure 7-21). Comparing 40 seconds of pre-treatment and test HEC1.5GHN, indicating that treatment with MEG enable further oil separation. As the curves is made of only two points, it can be hard to judge if more pre-treatment would reduce OOC further. However, water content would be a limiting factor for oil separation for pre-treatment as effects of steam stripping would decrease. A volume of 35 ml MEG was added to HEC1.4GHN, which achieved 8.4% worse oil separation and consumed 11.8% more energy compared to HEC1.5GHN.

Figure 7-22 shows that larger volumes of MEG demand more time and energy to evaporate. The curve of HEC1.5GHN increases faster and levels out after 35 seconds. Indicating that MEG have been boiling and evaporated off the DC. The curve of HEC1.4GHN have a slower climb and do not level out after 50 seconds of treatment. Therefore, as more MEG is added a longer treatment is also needed to evaporate MEG. If HEC1.4GHN had been treated for a longer time, it is reasonable to believe this sample would have achieved OOC like HEC1.5GHN or lower.

AC was tested with HEC1 even if no promising results was found with CP17, but different DC might react differently. Table 7-14 also shows that AC did not improve oil separation.

Small variation in results in Table 7-14 could be because initial OOC in various sample fractions, and a 100 % consistency is seldom the case. Another reason could be due to varying distribution of MEG onto the DC.

A previous study of HE1 achieved 0.67% OOC using cold MEG. The result was achieved using MEG and the same microwave unit as in this thesis (Rødne, 2018). Therefore, it is reasonable to believe that the OOC curve would continue further down if more energy was applied with MEG.

7.2.2 Husky Energy 2

In this section the results from testing HE2 are presented. The DC characteristics of HE2 was quite different to HE1, which is visually represented in Figure 7-23. The initial water and OOC concentrations of HE2 are shown in Table 7-15.

Figure 7-23: Untreated HE2

Untreated HE2 consisted of agglomerates like CP17. The agglomerates were covered of mud and was hard to deform with finger pressure. Indicating a different mineral composition compared to HE1 and CP17.

Table 7-15: Water and oil content of HE2

Parameter	HE2
$OOC_{Retort,dry}$ [%]	6.78
$OOC_{Retort,wet}$ [%]	5.87
Water [%]	8.75

7.2.2.1 Treating Husky Energy 2 with microwave radiation

Raw DC was treated in the microwave unit, which was set on full power (2kW), while variating treatment time. Therefore, the DC was exposed for variating energy inputs. The added power and treatment time do not correlate as reflective power vary at different treatment times.

Results from pre-treatment of HE2 are presented in Table 7-16. A visual representation of pre-treated HE2 are shown in Figure 7-24 and a visual presentation of crushed DC is shown in Figure 7-25. Oil and water separation are shown in Figure 7-26.

Table 7-16: Pre-treatment of HE2

Sample	$OOC_{Retort, dry}$ (%)	Water (%)	kWh/Ton	Time [s]	DC temp. [C°]
HE2.1	2.62	2.02	121	40	-
HE2.2	2.24	2.03	168	60	-
HE2.3	1.37	1.07	220	84	-

Figure 7-24: HE2 after microwave radiation

The treated DC have the same characteristics before and after microwave treatment. The agglomerates maintain its form after treatment. There are some tendencies of pores from the water escaping from the agglomerates. The agglomerates are solid and difficult to crush by finger pressure.

Figure 7-25: Crushed HE2 after microwave radiation

The large agglomerates of HE2 was crushed to its original size. More pressure had to be applied to crush the agglomerates to its original size using finger pressure, than with CP17. This indicates a different mineral composition in this DC. The mineral composition and particle size were not measured accurately.
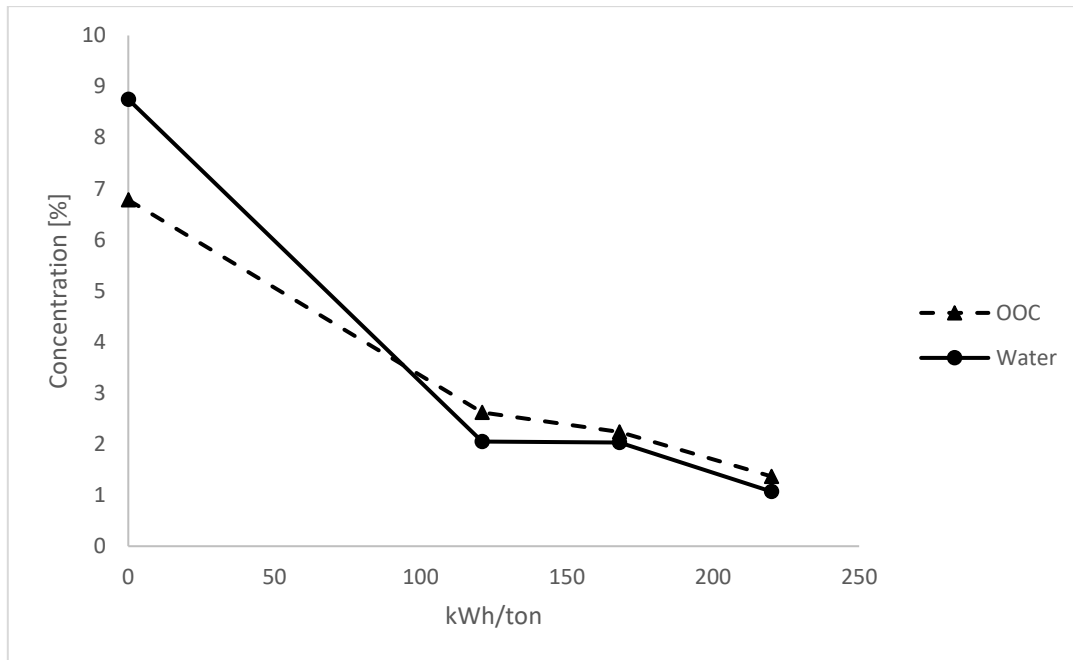
Figure 7-26: Pre-treatment of HE2

Discussion

HE2.3 was treated in the microwave for 84 seconds, which reduced OOC from 6.78% to 1.37% OOC using 220 kWh/ton (Figure 7-26). Water concentration results are misleading as it does not show a consistent decrease of water as it levels out before continuing down. This is errors mainly due to the DC soaking up humidity from the atmosphere which was discussed in Chapter 7.1.1. Therefore, it is reasonable to assume an even lower water concentration.

The water curve is crossing the OOC curve and leveling out, which indicate a potential dosing point for MEG at 120 kWh/ton. The OOC curve follows the water curve when it continues to decrease at 160 kWh/ton indicating that the stripping effect is still taking place. Therefore, MEG might be dosed at 230kWh/ton as well.

A slight decrease of the slope gradient in the OOC curve occur at 120 kWh/ton. This is most likely due to the water concentration decreasing and the stripping effect lose its effect as water evaporates. This might be another potential dosing point of MEG.

7.2.2.2 Husky Energy 2 - Optimization of oil separation and energy consumption using susceptor

Based on findings and discussion in section 7.2.21, MEG should be dosed in the energy range of 120 kWh/ton to 230 kWh/ton.

Hot MEG is dosed on hot DC in this section as it proved to be beneficial in Chapter 7.1. HE2 treatment is presented in Table 7-17. The tests were performed to find the best combination of pre-treatment and susceptor treatment. Figure 7-27 shows oil separation and water content from HE2 treatment.

Table 7-17: Results from treatment with MEG

Sample	OOC_{Soxtec} [%]	kWh/ton	MEG [mL]	MEG temp. [C°]	Time [s]
HE2.5GH	0.68	146+157	20	120	50+60
HE2.4GH	0.78	220+87	20	120	84+30
HE2.6GH	0.94	166+189	33	120	57+64

*Pre-treatment is written before other treatment in kW/ton and time
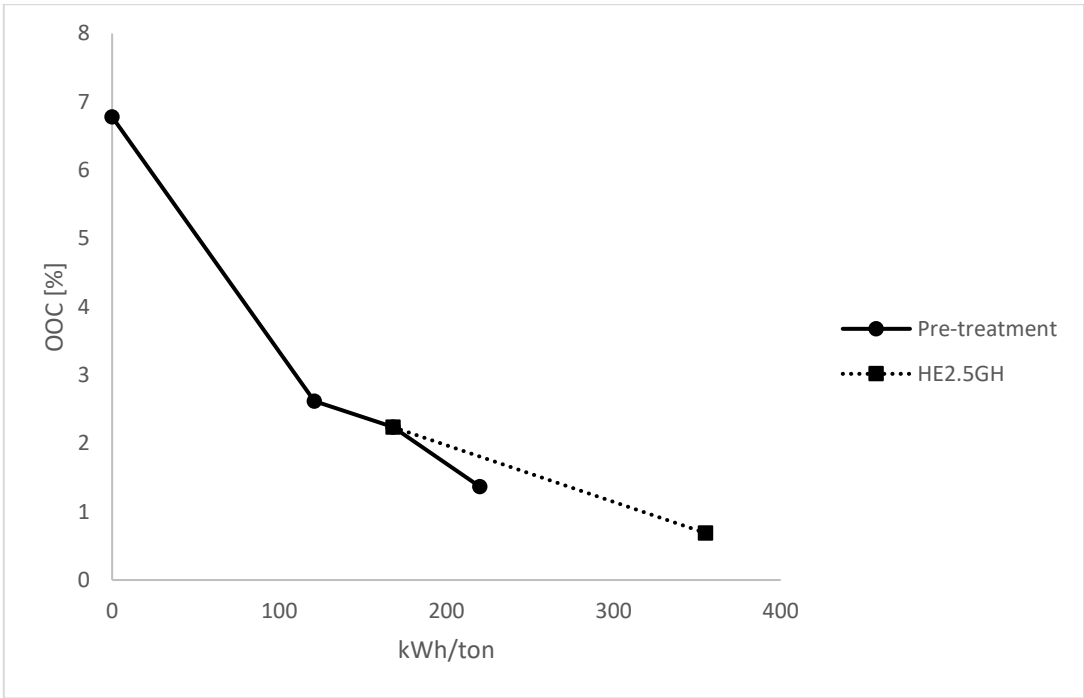
Figure 7-27: Oil separation and water content from pre-treatment of HE2

Discussion

HE2.5GH was the best oil separation of the samples with hot MEG. This reduced OOC to 0.68% using 303 kWh/ton. Figure 7-27 shows that the pre-treatment has a steeper curve than HE2.5GH, indicating more efficient treatment. Pre-treatment reduced water concentration to 1.07% and will most likely not be able to reduce OOC further because evaporation of water is needed to remove oil. Therefore, MEG was advantageous as it enabled to increase the oil separation and HE2.5GH achieved an OOC of 0.68%

Test HE2.4GH in Table 7-17 was pre-treated for 84 seconds and treated with MEG for 30 seconds. This treatment combination was performed as it gave good results with CP17. Results from HE2.4GH was significantly worse compared to similar test with CP17, and HE2.4GH resulted in an OOC that were double as high as CP17.16GH. This is most likely because different mineral composition and particle size which effects the stripping mechanism.

7.2.2.3 Pre-heating og HE2 before microwave treatment

In this section, the DC was heated to 70 C˚ to simulate offshore conditions. Raw DC was heated in a water bath before treatment. Table 7-18 show the result from pre-heated DC. Figure 7-28 represents a comparison of pre-heated microwave treatment and cold DC microwave treatment.

Table 7-18: Pre-heating of HE2.

Sample	OOC$_{Retort, dry}$ (%)	Water (%)	kWh/Ton	Time [s]	DC temp. [C˚]
HE2.8P	1.51	-	124	44	70
HE2.3	1.37	-	220	84	-
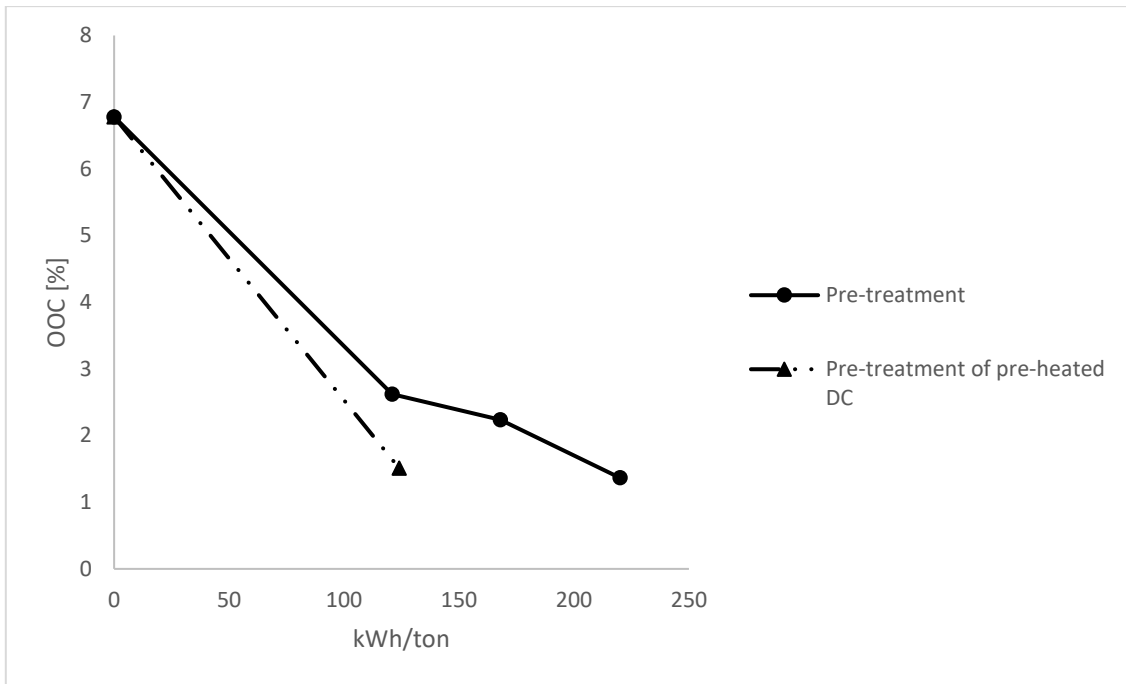
Figure 7-28: Comparison of microwave radiation with and without pre-heated HE2

Discussion

Figure 7-28 shows that H2.8P was pre-heated to 70 C° and reached 1.51 OOC % using 124 kWh/ton. This is a significant difference from HE2.1, that is the most comparable of the samples. Pre-heating of HE2 increased oil separation, which proved to be even more significant compared to pre-heating of CP17.

Pre-heating of HE2 managed 47.8% lower OOC compared cold samples with equal treatment time. This might be because water in HE2 are more available in the DC. As the DC was more compact, less water is trapped in pores, therefore less energy is consumed to evaporate the water tight bound water. CP17 had a different mineral composition and particle size, as it was softer, it most likely contains more clay. This allows several pores to trap the water, and more energy is required to evaporate the water.

7.2.2.4 Best set-up for treatment of HE2

Based on finding and discussion in Chapter 7.2, the best set-up of HE2 treatment was performed and the result are shown in Table 7-19. Figure 7-29 represents a comparison of microwave treatment and various optimization measures, as well as the best set-up.

Table 7-19: Best set-up of HE2 treatment.

Sample	OOC$_{Soxtec}$ [%]	kWh/ton	MEG [ml]	MEG temp. [C$^\circ$]	Time [s]	N$_2$-stripping [15L/min]	DC temp. [C$^\circ$]
HE2.7PGHACN	0.46	124+153	20	150	44+60	2 min	70
HE2.5GH	0.94	146+157	20	120	50+60	-	-
HE2.8P	1.51	124	-	-	44	-	70
HE2.3	1.37	220	-	-	84	-	-

*Pre-treatment is written before other treatment in kW/ton and time
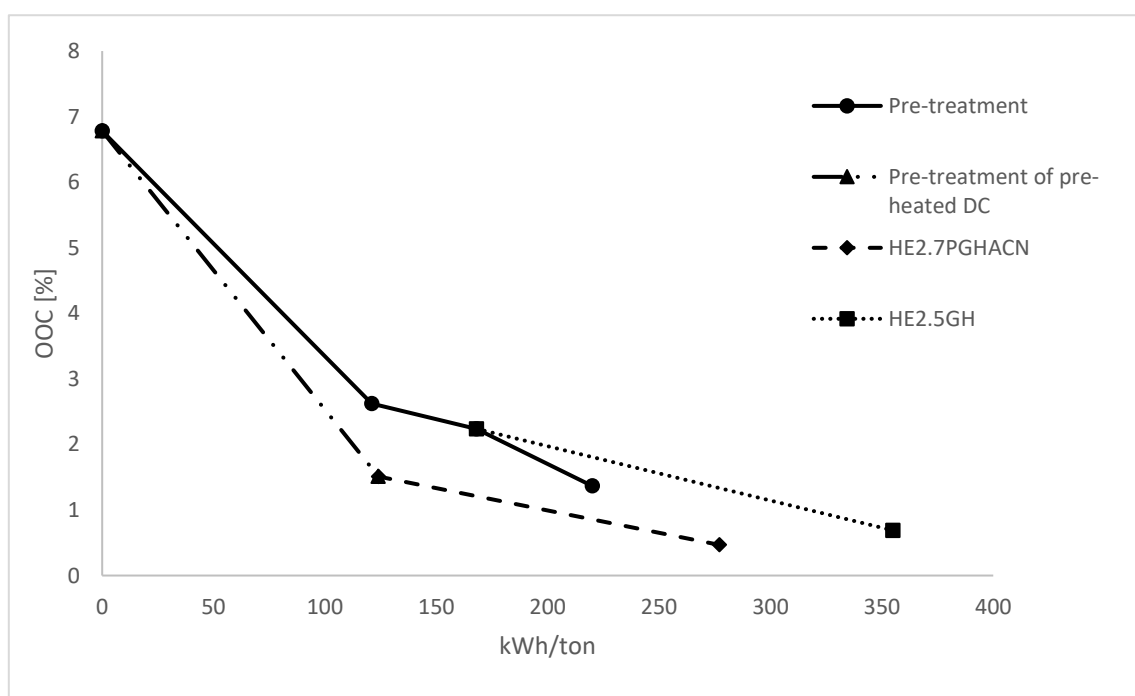
Figure 7-29: Comparison of microwave treatment, susceptor treatment and best set-up

Discussion

Treatment with pre-heated DC showed to be a significant advantage from Figure 7-29 as the curves are below curves from the other treatment curves. HE2.7PGHACN achieved an OOC of 0.46% using 277 kWh/ton, which was the best result with HE2. The technology from Norwegian Technology manage to treat HE2 below both 1% and 0.5% OOC.

7.3   Product recovery – Separated water, oil and MEG quality

The microwave technology presented in Chapter 6 include the use of condensers to recover oil, water and MEG from the DC. In this chapter, the life cycles of the liquid components retrieved from the treatment will be discussed. Table 7-20 presents measured oil in water, and Table 7-21 shows an estimate of MEG loss and costs.

Figure 7-30 show three different ampoules containing different liquids. From the left in Figure 7-30:

1) Cracked oil and MEG from Retort analysis
2) Recovered oil and MEG from microwave treatment
3) Recovered water from microwave treatment
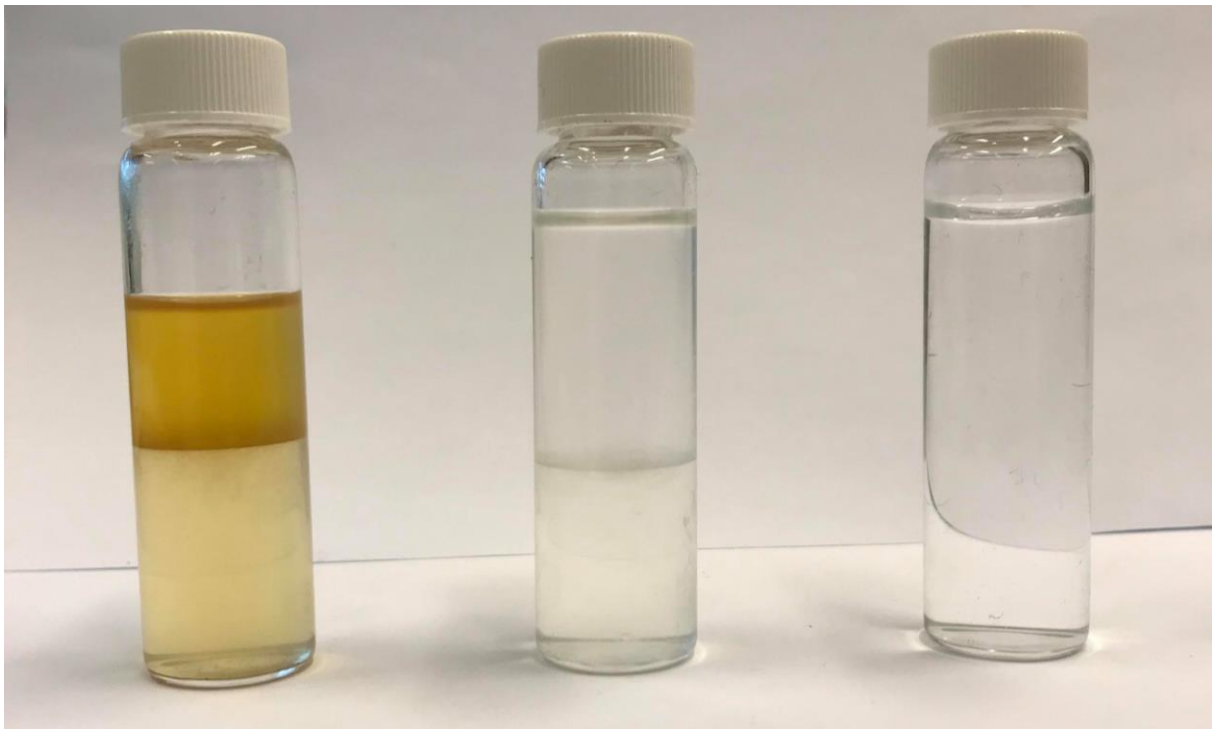
Figure 7-30: Cracked oil and recovered oil, MEG and water from treatment

Table 7-20: Oil concentration in recovered water

Oil in water [mg/L]	OSPAR discharge limit of oil in water[mg/L]
0.55	30

Table 7-21: Estimated costs from loss of MEG on DC

MEG loss [%]	MEG loss per ton DC	Economic loss [$/ton DC]
1	10	5
2	20	10
3	30	15
4	40	20
5	50	25
6	60	30
7	70	35
8	80	40
9	90	45
10	100	50

Discussion

The Retort analysis was performed with a maximum temperature of 481 C˚, the oil cracked at this temperature. This leads to coloring of the oil, which turns brown (Figure 7-30). The microwave treatment do not run at high enough temperatures in order to crack the oil. Therefore, oil from microwave treatment can be recoverd and reused as middle ampoule in Figure 7-30 shows no signs of color in the oil phase.

Oil and MEG are immicible liquids and drilling fluids contain emulsion breakers. Oil and MEG from the middle ampoule clearly show two liquid phases. This indicates that microwave treatment brake down eumusifiers in the mud, which enable easier and reuse separation of oil and MEG.

The ampoule th the right in Figure 7-30 shows that there are no discoloration of the water. The oil concentration in water recovered from microwave treatment was 0.55 mg/L [2]. This is well below OSPAR limits from discharge of dispersed oil in water, which is 30 mg/L (OSPAR Commission, 2001). Therefore, recovered water from microwave treatment do not need additional treatment and can be directly discharged to the sea.

MEG should be recoved in terms of costs and footprint concerning offshore treatment. Table 7-21 shows an estimate of the amount MEG lost in microwave treatment and the economic consequence. The ratio between water and MEG in treated DC was not determined in this thesis. Therefore, Table 7-21 is an estimated overview of lost MEG in DC. The costs per ton of MEG vary depending on suppliers, Purity and volume of purchase. The cost per ton MEG used in Table 7-21 was set at 500 $ per ton (Alibaba.com, n.d.).

If the case was that 3% MEG was lost during the treatment it would cost the operator 15,000 $ per 1000 ton DC treated. This is a cost that add up the more drilling activites that is performed. Another reason that recirculation of MEG is of best interest to the operators is the footprint. Recirculation of MEG reduce footprint as smaller storage tanks can be used.

Considering environmental impact, MEG is recognized at OSPAR's PLONOR list, and discharge of MEG to the sea should not be harmful to the environment.

7.4   Drill cuttings treatment – oil separation

Handpicked DC samples were sent for testing to Eurofins Environment Testing Norway AS. Eurofins' analysis is more accurate compared to the Retort and Soxtec analysis used in this thesis. Eurofins analyze hydrocarbon concentration using gas chromatography (GC). Results from Eurofins was received 13.06.19.

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[2] Oil concentration in water received from Eurofins.

Table 7-22: Test results from Eurofins

Sample	OOC$_{Soxtec}$ [%]	OOC$_{GC}$ [%]	Change in OOC [%]
CP17.1	1.41	0.93	33.8
CP17.5G	0.11	0.0017	98.4
HE2.7PGHACN	0.46	0.25	45.2
HEC1.5GHN	2.08	1.46	29.6
CP17.18	0.89	0.17	80.4
CP17.21GHAC	0.29	0.16	44.4

Discussion

OOC results from Eurofins was considerably lower than measured OOC in this thesis. This is mainly because Eurofins are selectively analyzing amount of specific hydrocarbon chains. As Eurofins used GC, they measured hydrocarbon chains from C_{10} to C_{40}. In addition, this is the specific hydrocarbon range used by NEA when discharge limits are determined.

All hydrocarbons in this thesis was measured with Retort and Soxtec analysis. These methods measure all hydrocarbon chains in the sample. Therefore, the hydrocarbon range that was measure was significantly larger, which resulted in significant difference between the test results.

The microwave technology from Norwegian Technology can be considered more efficient and the optimization process could have been different. Knowing this, considerable amounts of energy could have been saved to reduce OOC in the DC below 1% and 0.5%.

8 General discussion and Conclusion

The parameters that were investigated to optimize oil separation and energy consumption are presented below.

Dosing of MEG on hot drill cutting immediately after pre-treatment seemed to be beneficial. Energy consumption was reduced with 23.4%. This is most likely because less energy was spent heating the sample, as it already was heated from pre-treatment.

Susceptor volume proved to be of importance, and 20 to 30 ml MEG was found to be ideal for treatment of 150g samples. A volume of 10 ml MEG was considered as to little, while 50 ml MEG was too much. Oil separation was increased by 40% by dosing 20- or 30-ml MEG. However, 50 ml MEG would achieve better oil separation with more treatment time, but this would also increase energy consumption.

Heating of MEG was advantageous as it increased treatment efficiency, as treatment time was decreases due to a faster evaporation rate. Oil separation was increased with 40.3% when MEG was heated to 120 C°. Energy consumption was also reduced with 16.8%.

N_2 gas was applied to increase removal of condensate. Oil separation was increased with 32.7%. Energy consumption was not affected using N_2 gas as it was stored in a pressurized tank. Implementation of N_2 gas in offshore treatment must consider costs from transportation and storing gas tanks and compressors.

AC was added to achieve faster heating of MEG during treatment. This was intended to increase microwave absorption. AC in combination with cold MEG increased oil separation with 13.4% and decreased energy consumption with 20%. When AC was mixed with hot MEG, no significant advantage was detected. Hot MEG would most likely be applied in a full-scale unit, therefore AC should not be implemented.

Pre-heating of DC to 70 C° was intended to simulate offshore conditions. By stating treatment with a heated sample resulted in 11.9% less energy consumption. Microwave treatment should therefore be performed with hot DC.

Treatment of DC from NCS using microwave radiation with MEG proved to achieve oil separation down to 0.0017% OOC. Findings from Conoco Phillips 17-inch was applied to the treatment of DC outside of NCS.

The DC from Husky Energy was of different characteristics in terms of mineral composition, particle size, water content and OOC. HE2 consisted of agglomerates like CP17 and was successfully treated to less than OSPAR requirements. The best OOC value achieved with HE2 was 0.25% using 277 kWh/ton. Microwave treatment without susceptor had problems with oil separation on HE1. This is most likely because of high oil concentration compared to water. The sample had to be centrifuged to not block gas flowing through the sample cylinder. Microwave treatment with susceptor achieved an OOC of 1.46%. Further optimization might achieve OOC below 1%.

Recovered water was found to contain 0.55 mg/L and can be directly discharged to the sea as it is below OSPAR's regulations of 30 mg/L for dispersed oil in water. Process temperature in microwave treatment with MEG is around 195 C˚ and recovered oil after treatment did not show any signs of cracking. This indicate that reuse of oil is possible after treatment. It was difficult to determine loss of MEG as the concentration in treated DC is unknown. However, recovered MEG did not show signs of decomposition, and could be reused.

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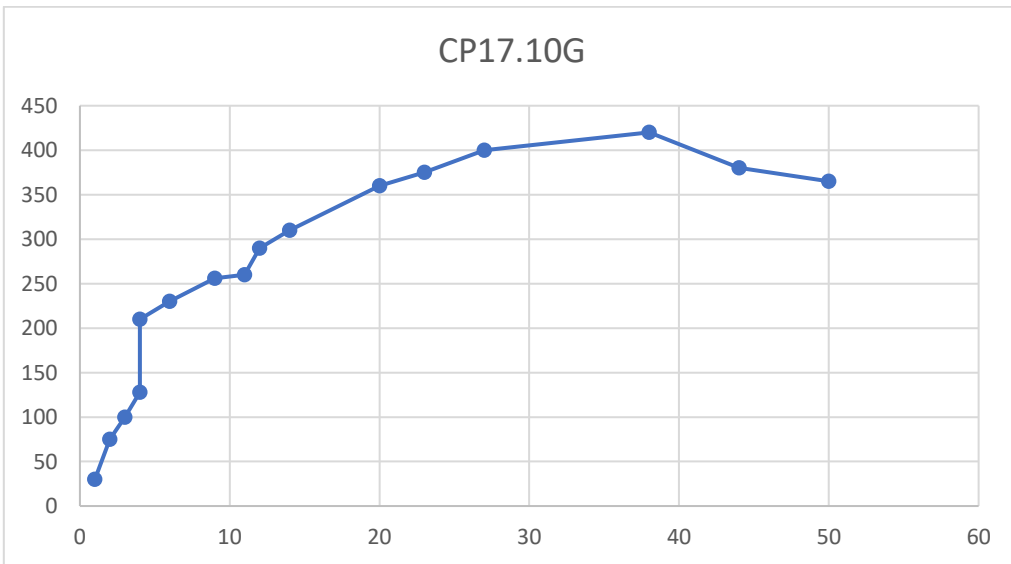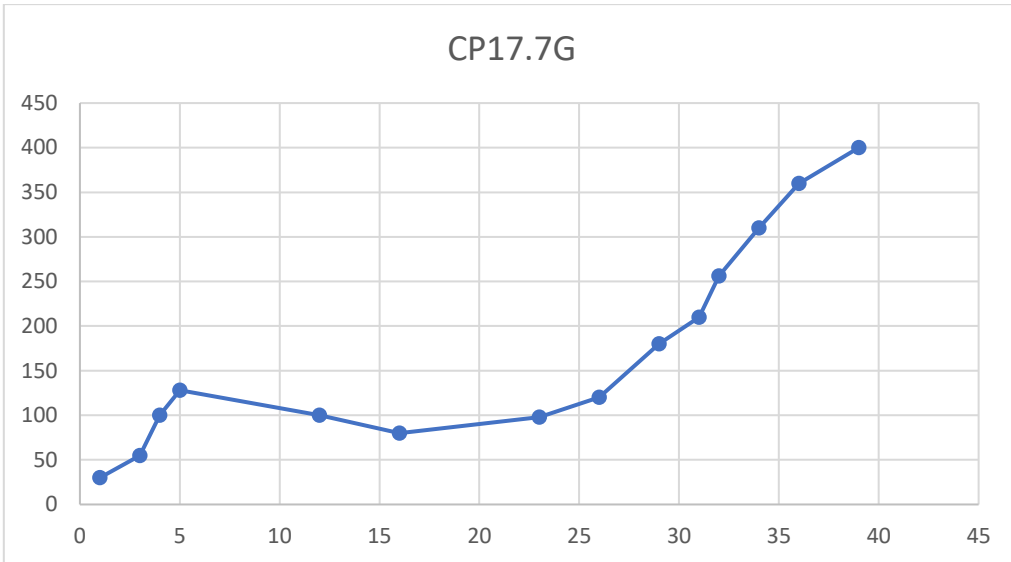
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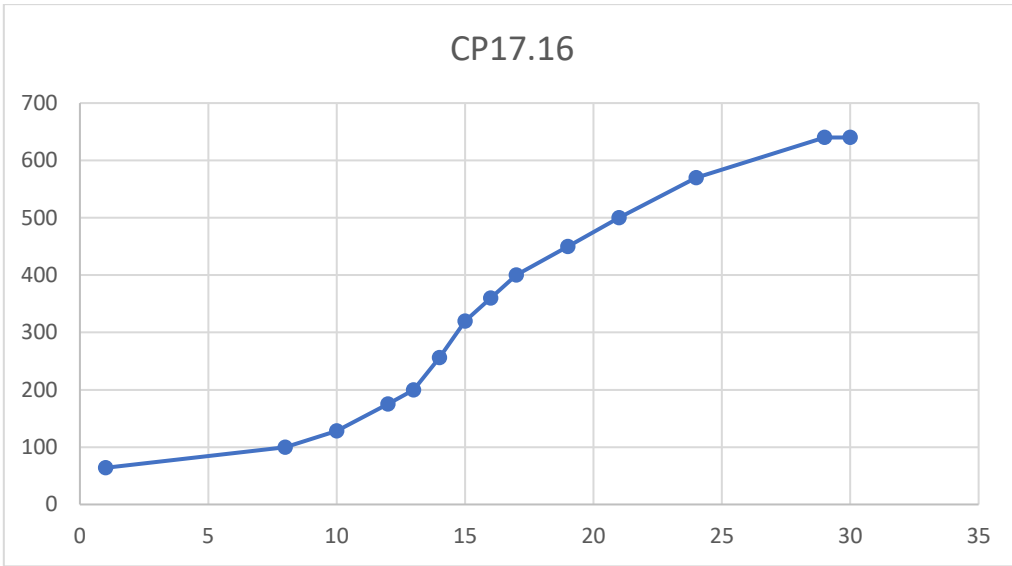   heating. In (pp. 458-463).

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Appendices

1) Reflective curves of Conoco Phillips 17-inch section. The Y-axis show reflected power (W/h) and the X-axis show time (s)
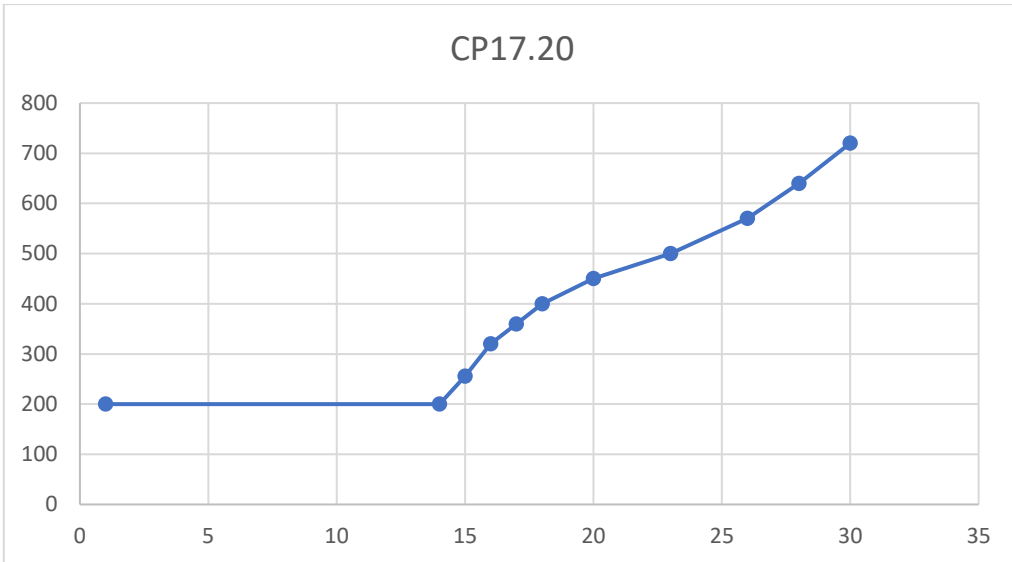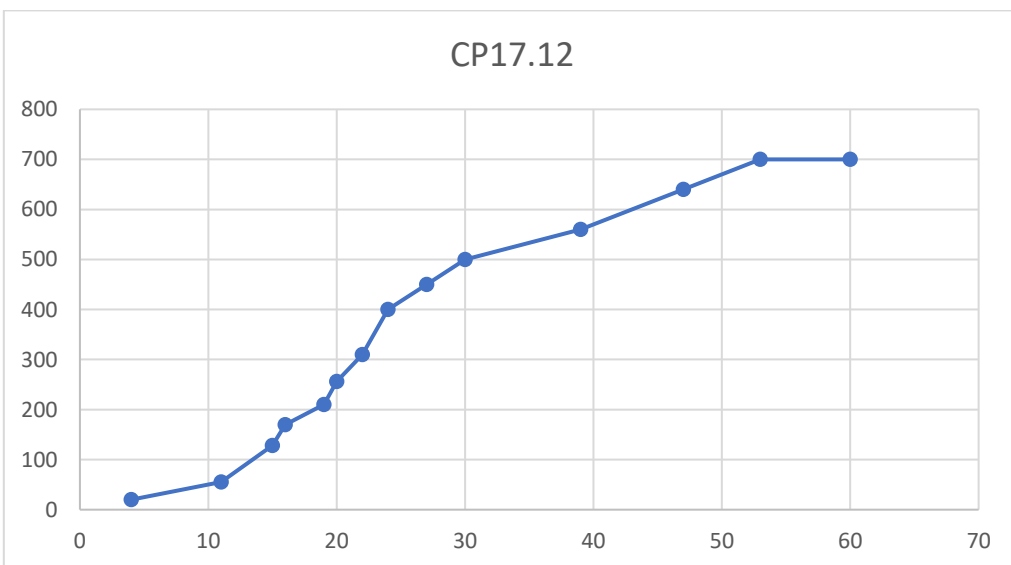

CP17.1

CP17.4

CP17.7G

CP17.10G

CP17.13

CP17.16

CP17.19

CP17.25

CP17.2G

CP17.5G

CP17.8

CP17.20

CP17.23

CP17.3

CP17.6G
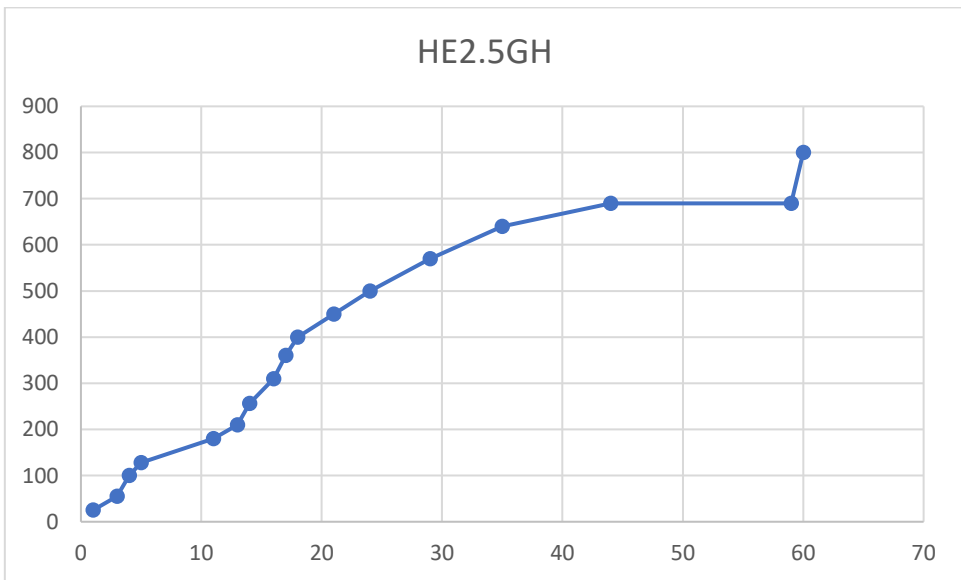

CP17.9G

CP17.12

CP17.15

CP17.18

CP17.21

CP17.24
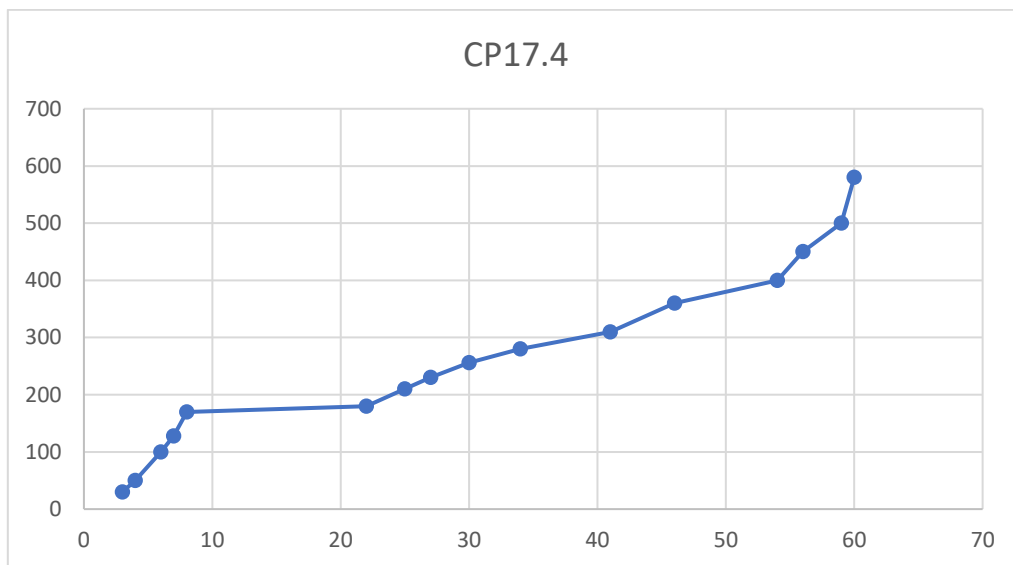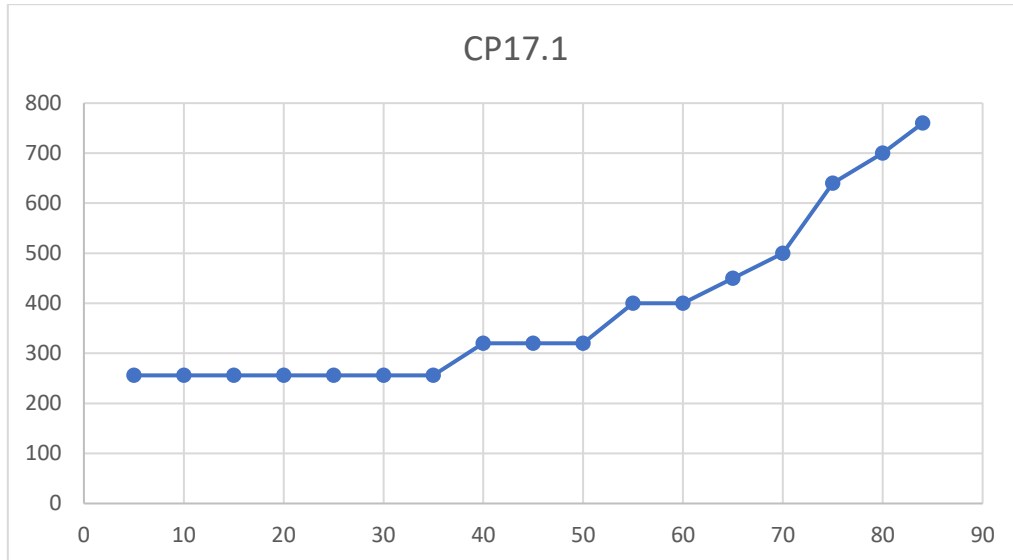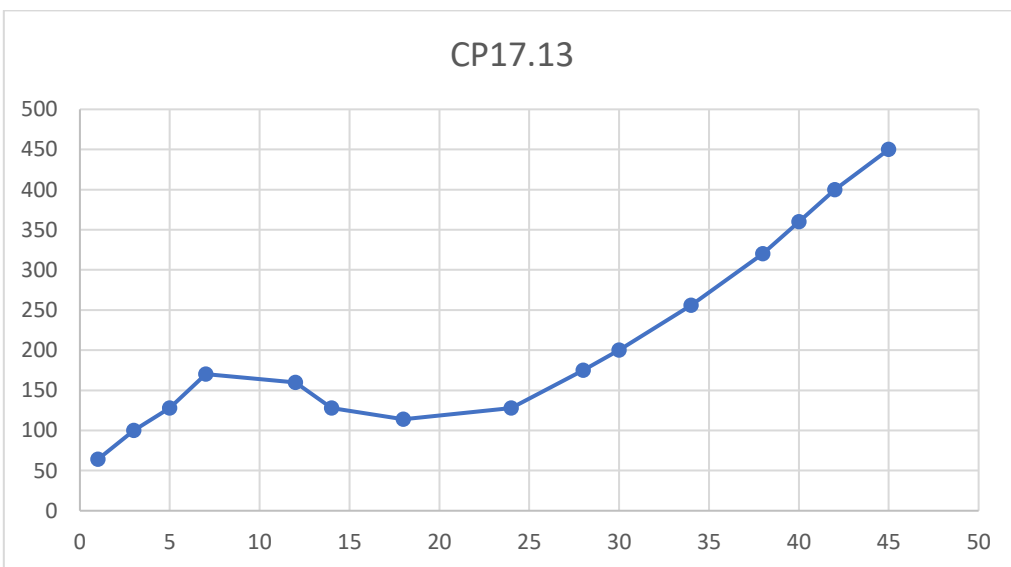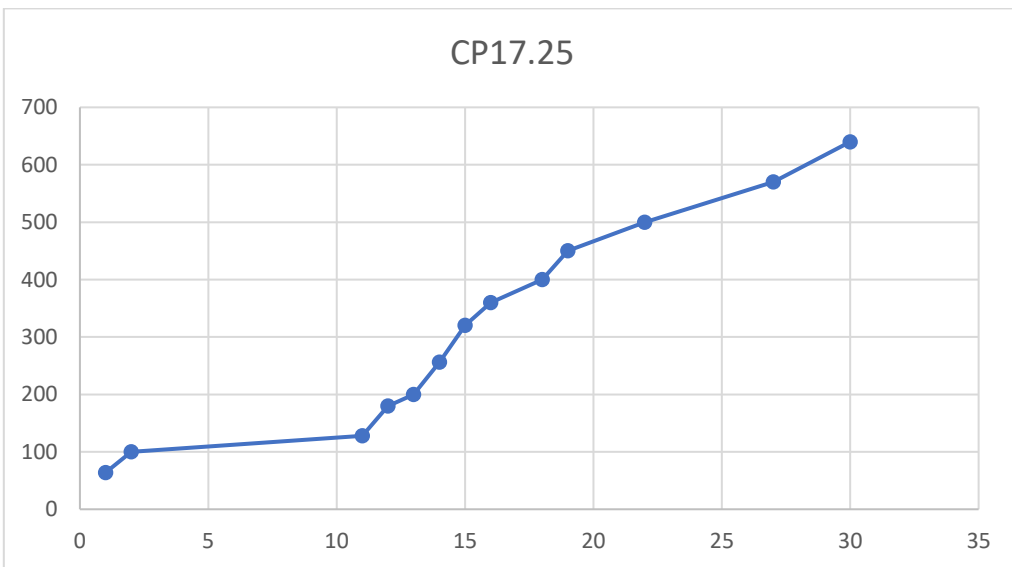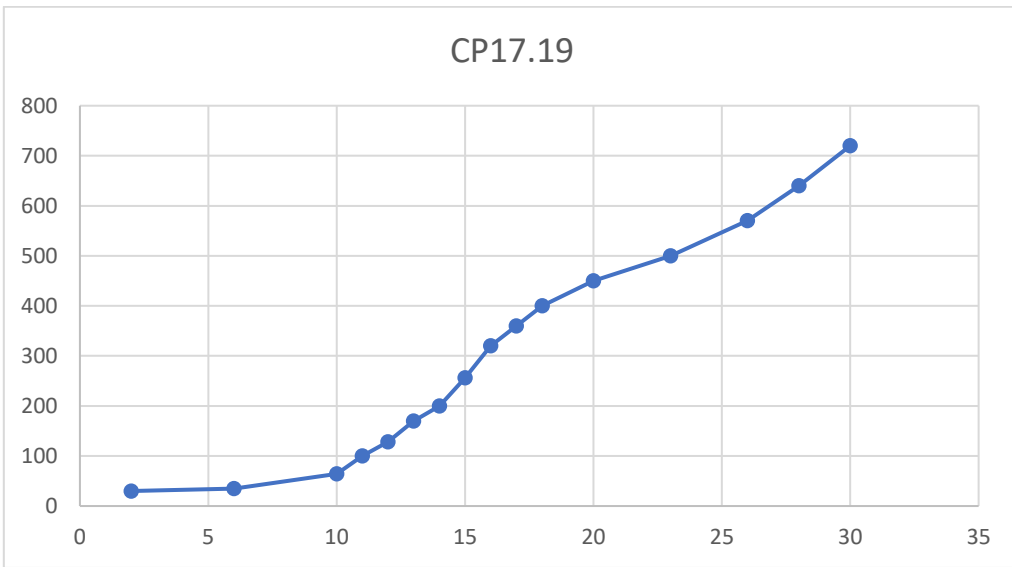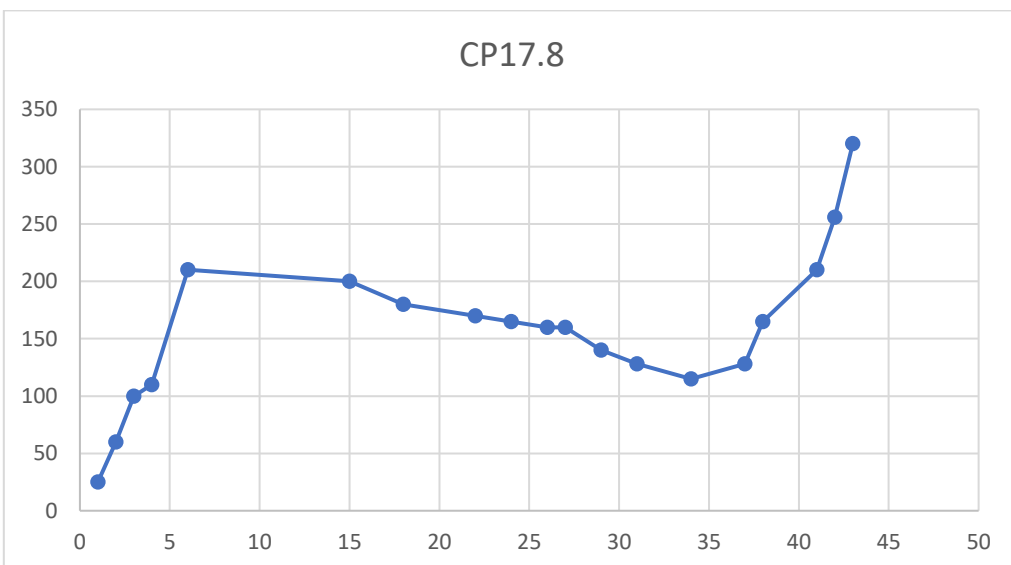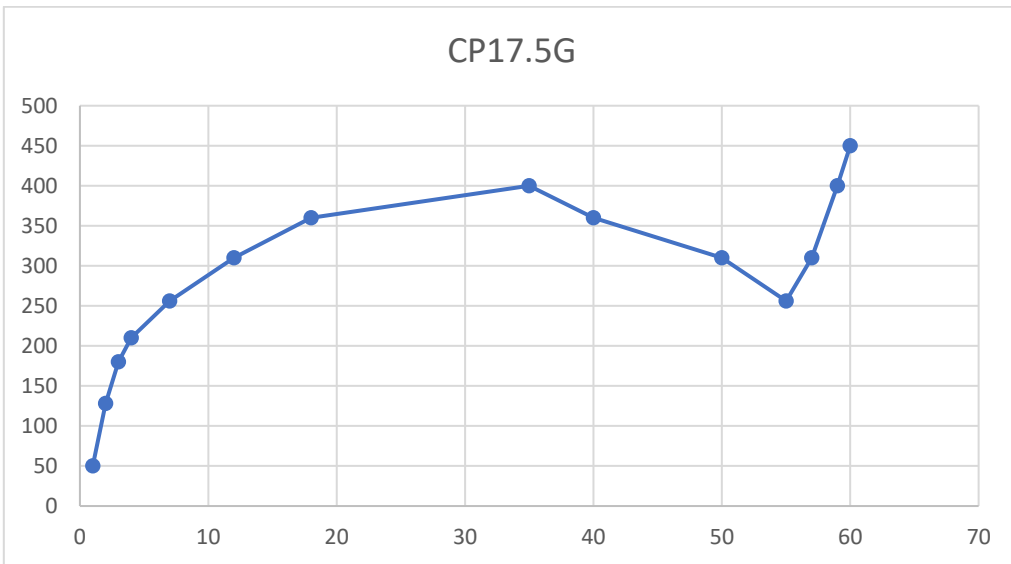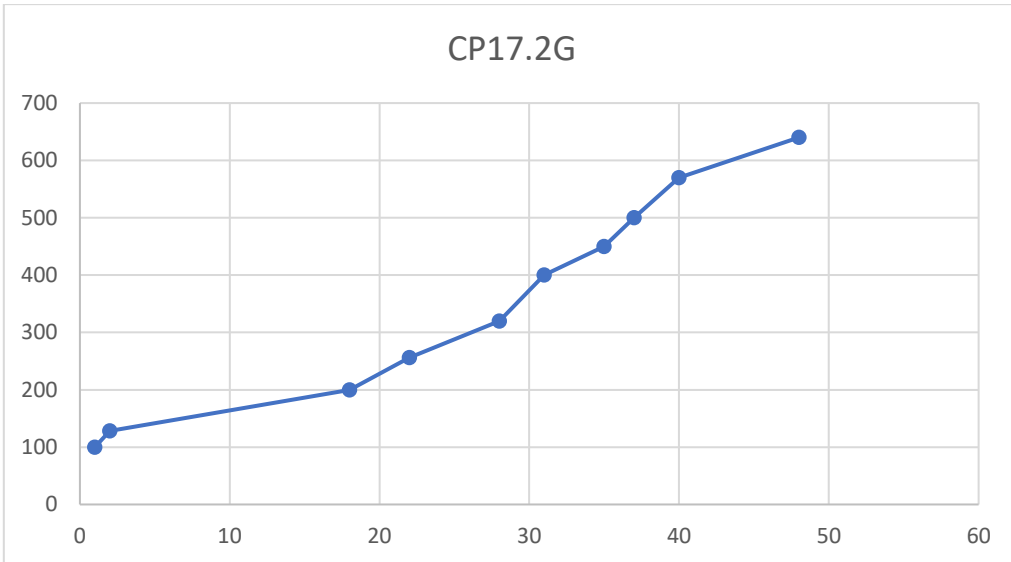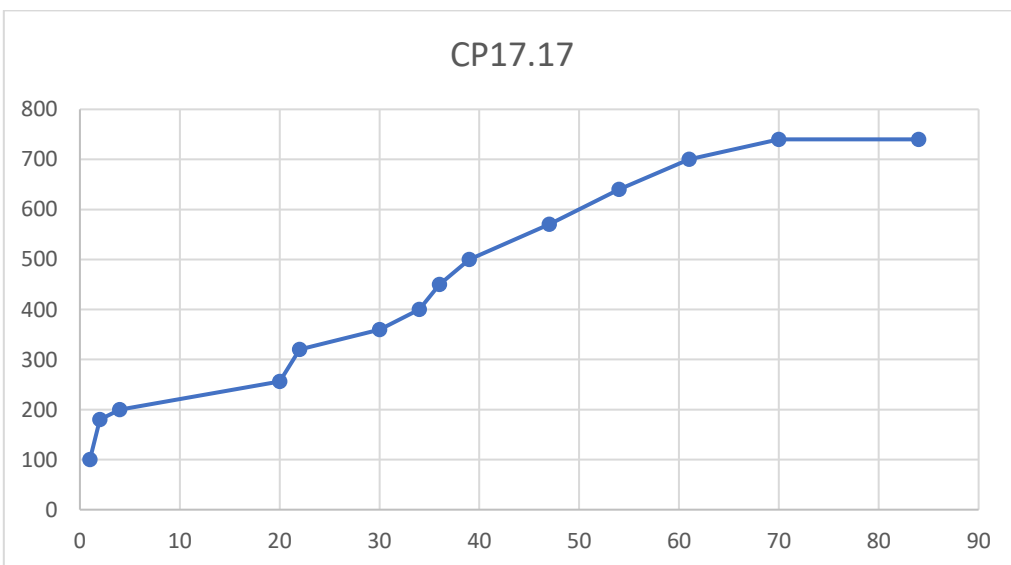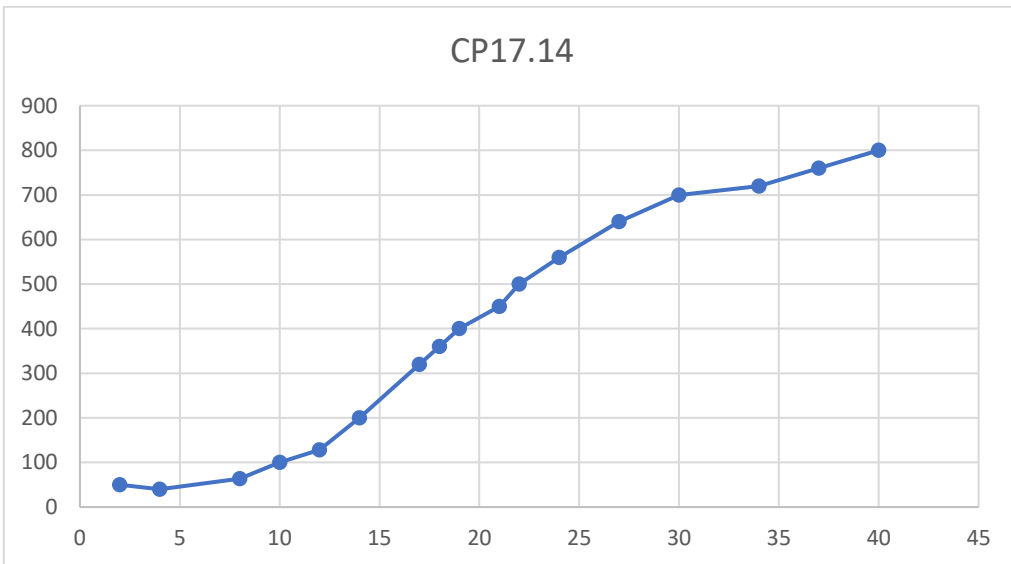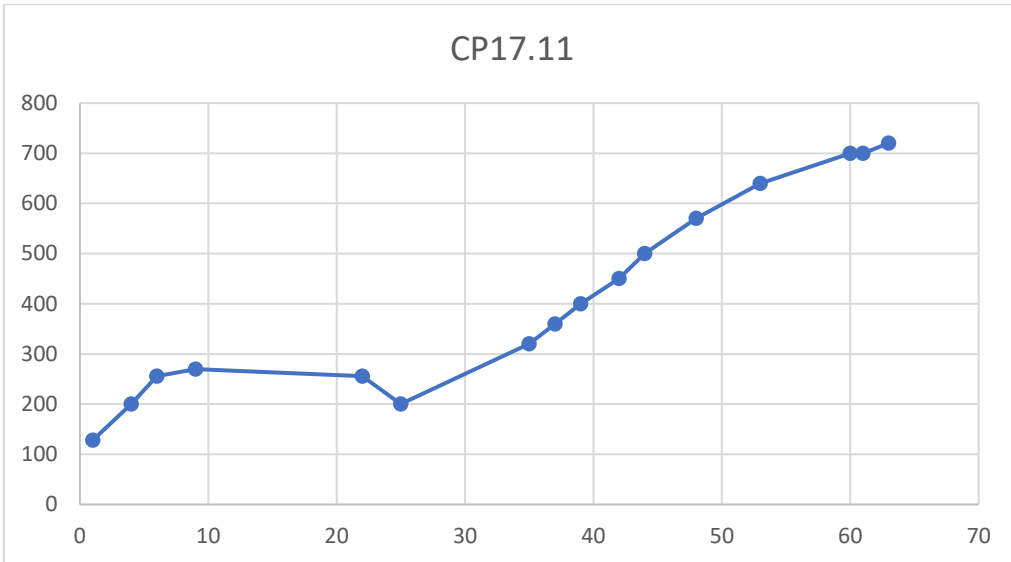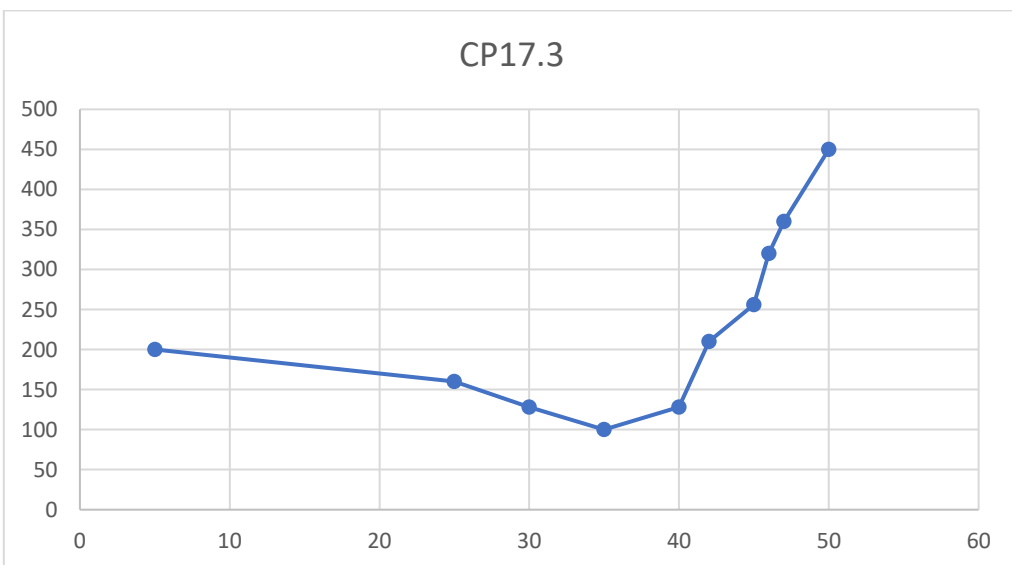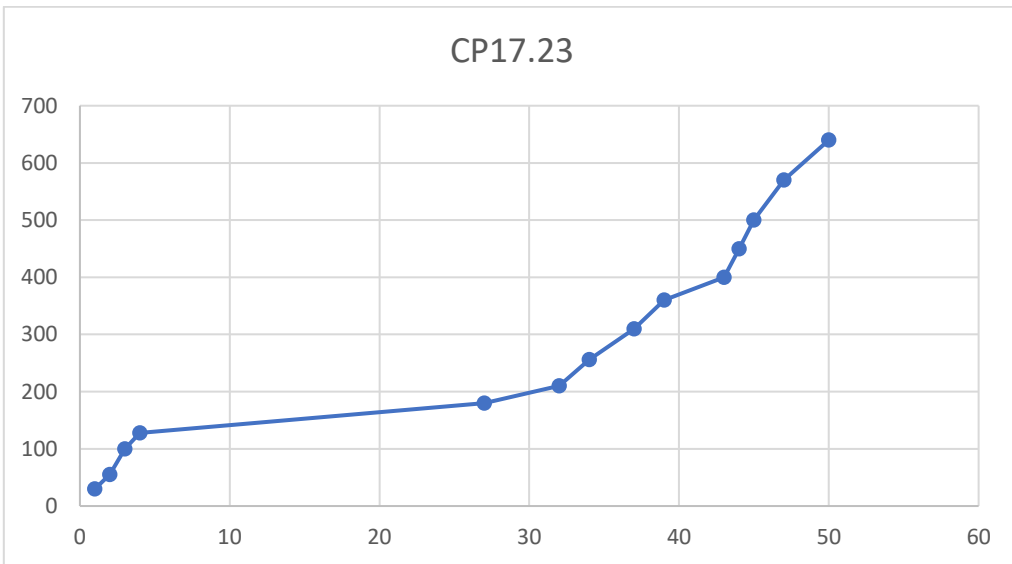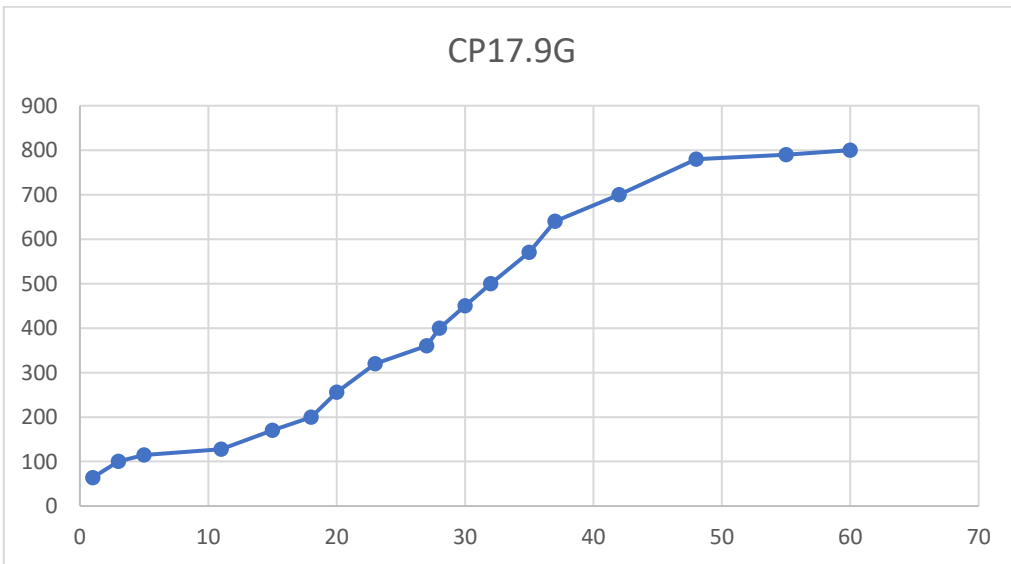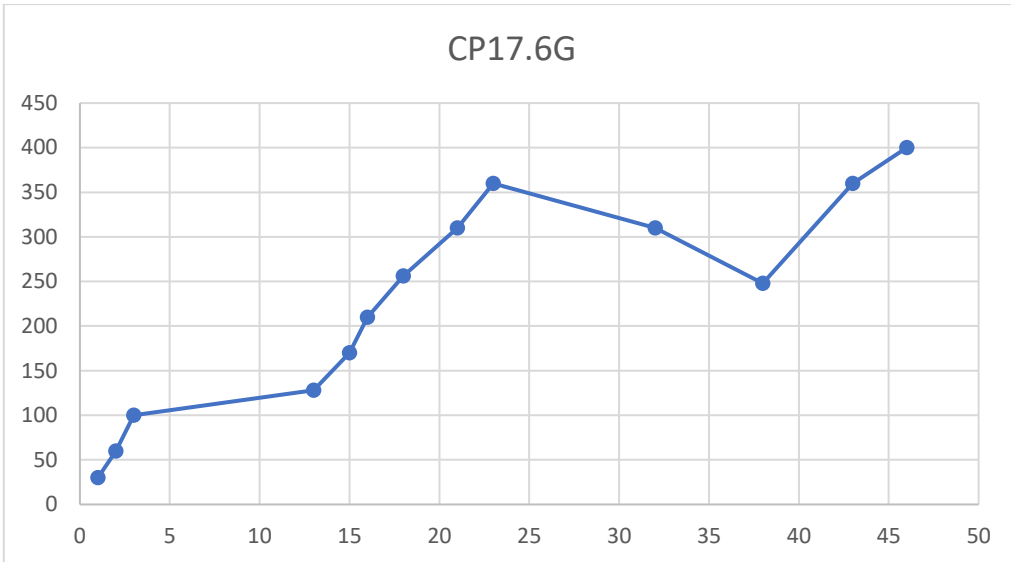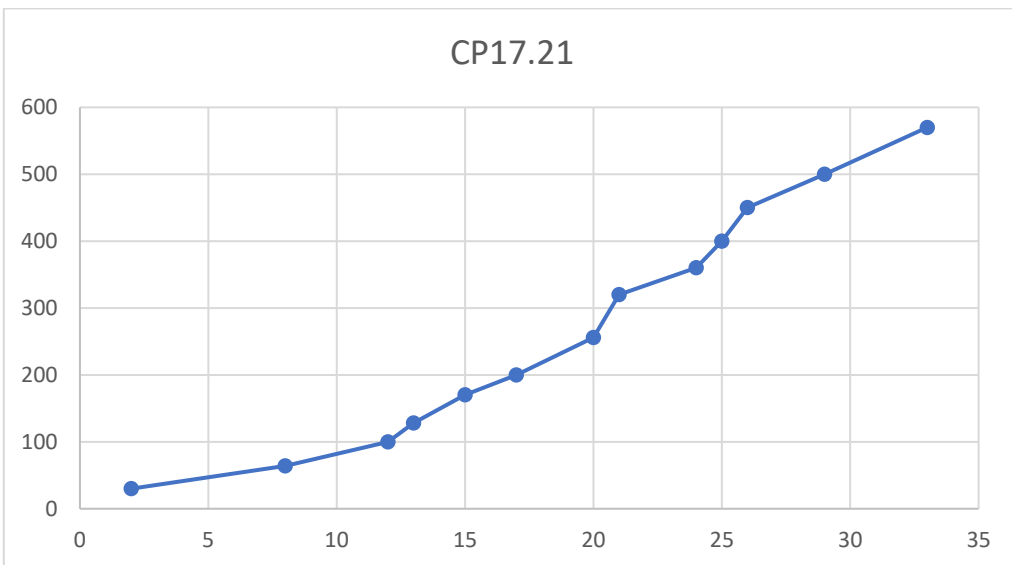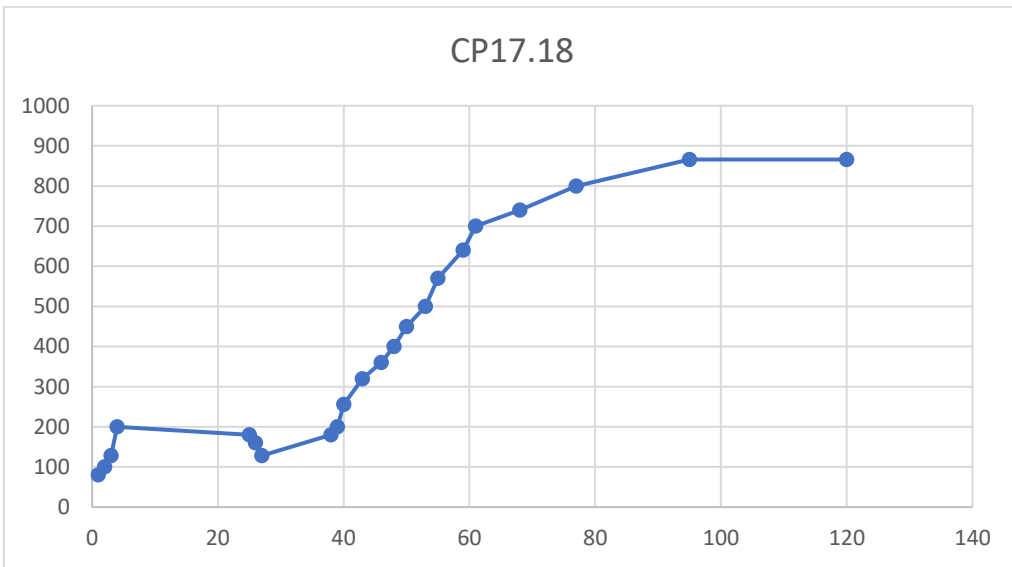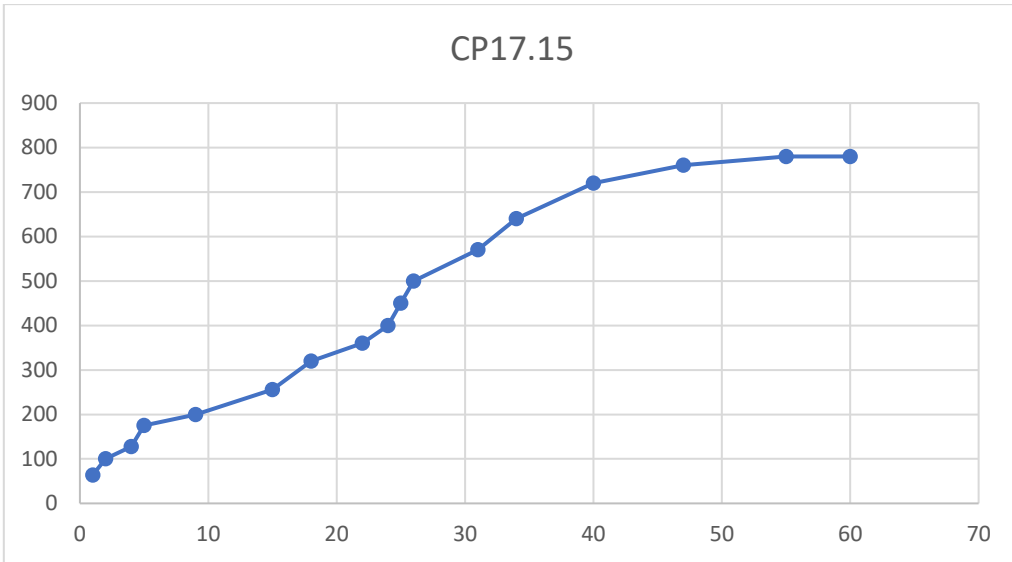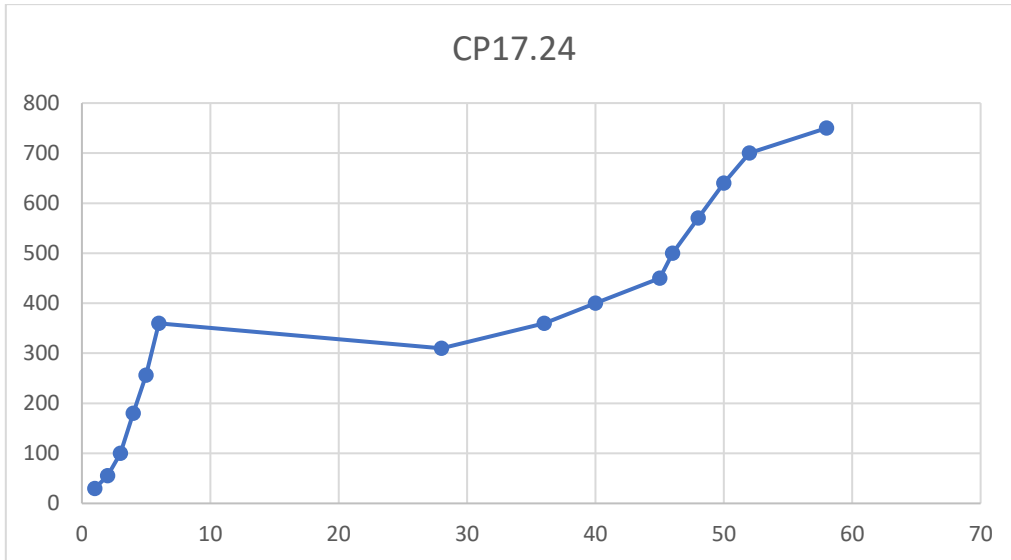
2) Reflective curves of HE2. The Y-axis show reflected power (W/h) and the X-axis show time (s)

HE2.2

HE2.3

HE2.1

HE2.6GH

HE2.8P

H2.7PGHACN

HE2.5GH

HE2.GH4
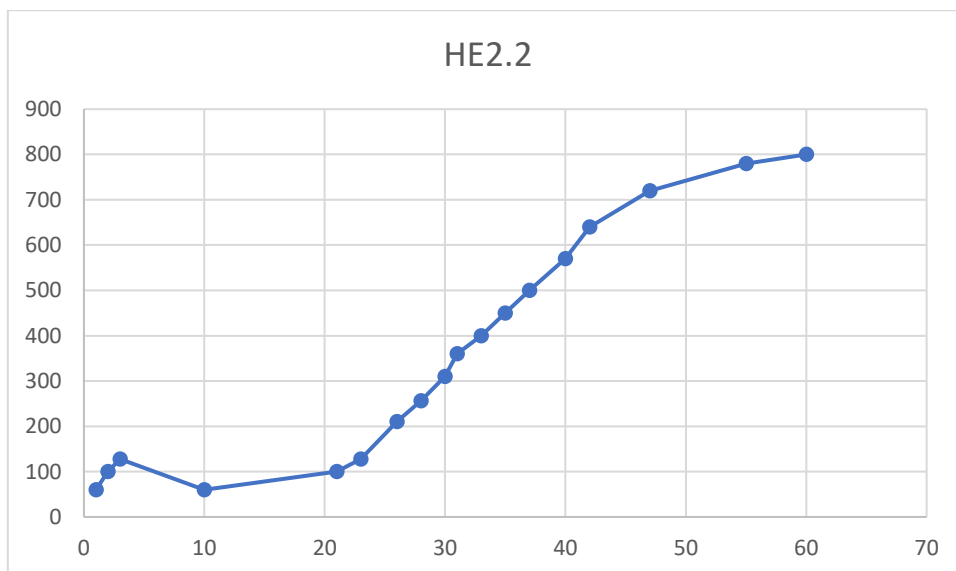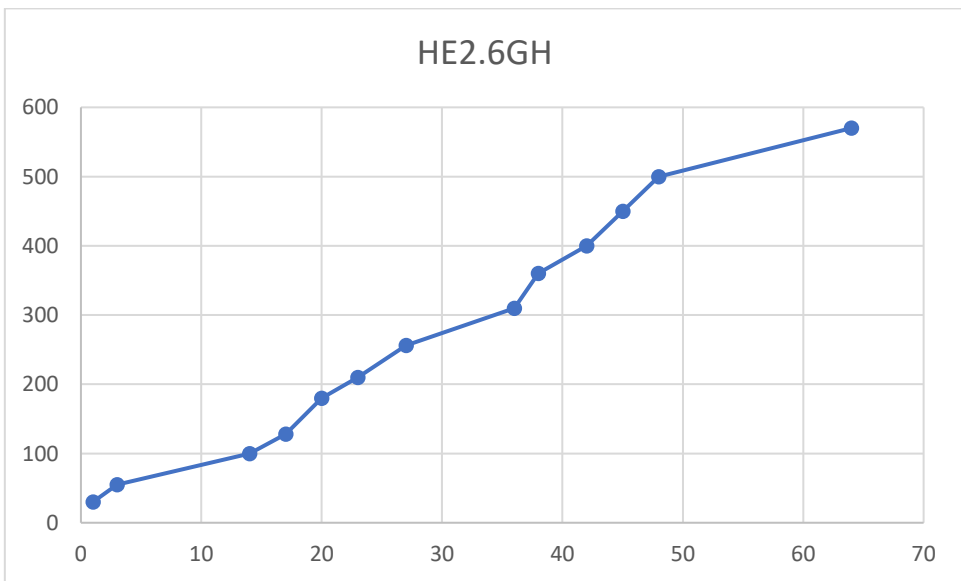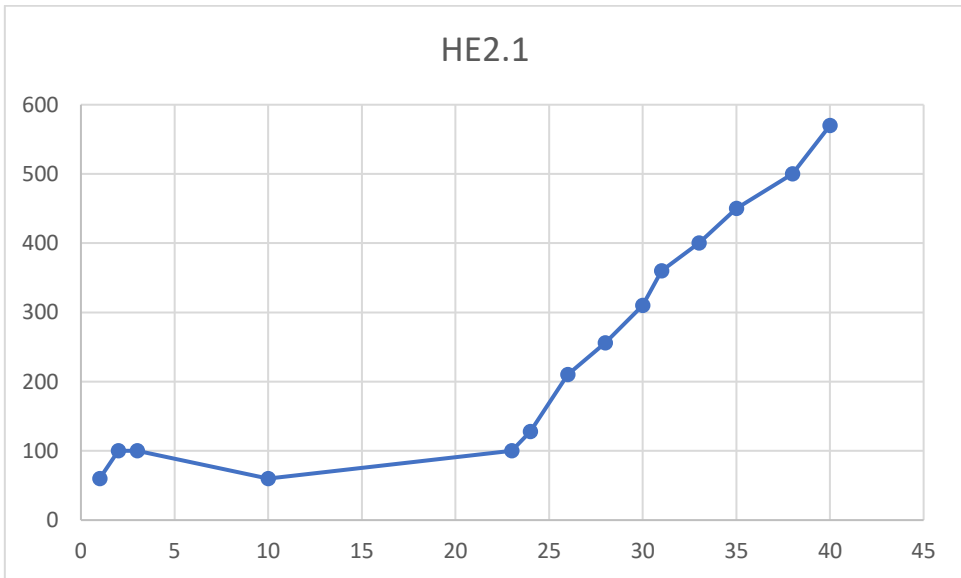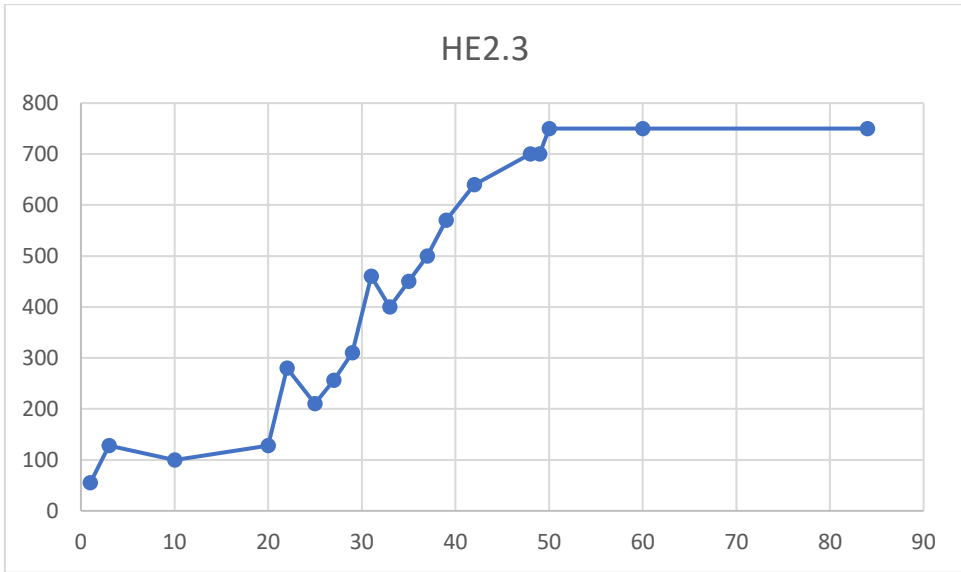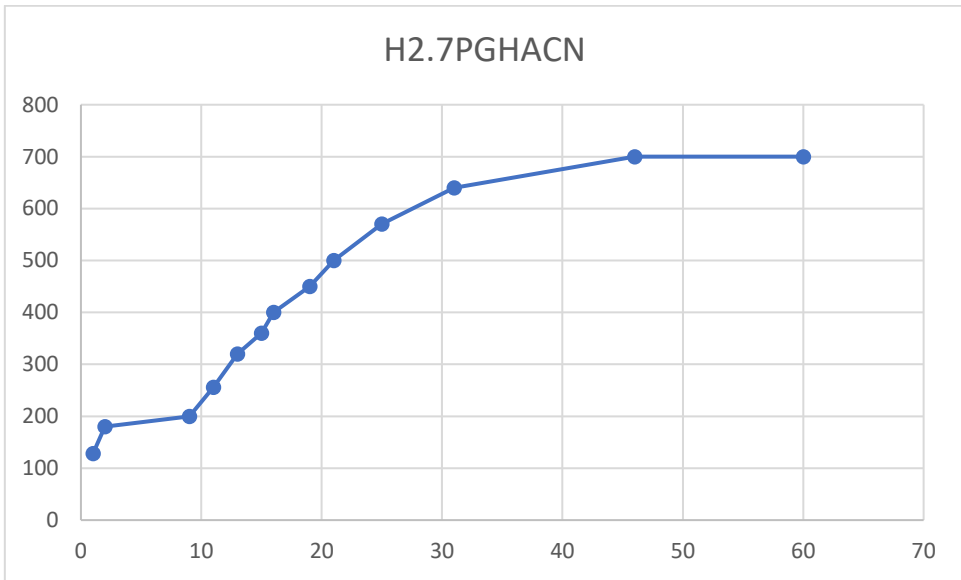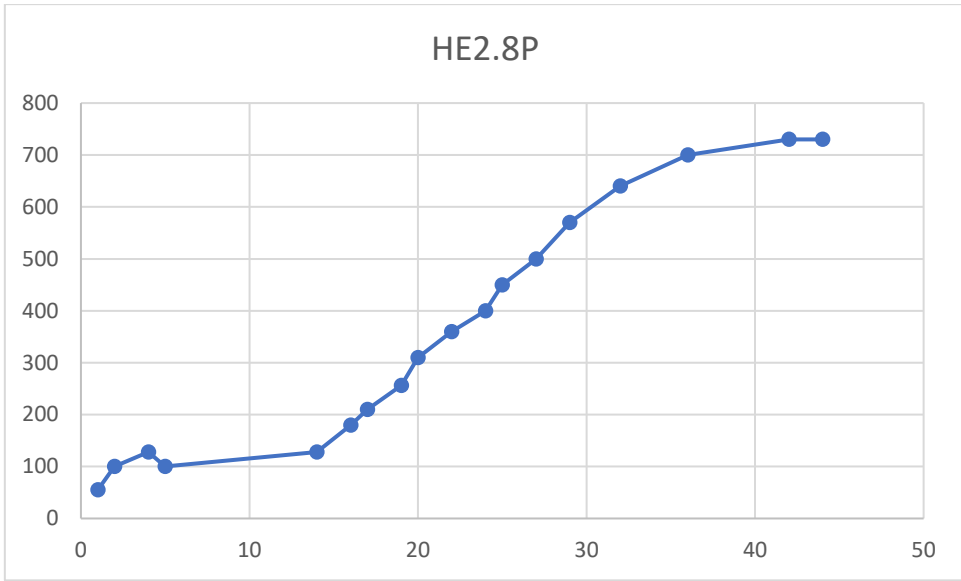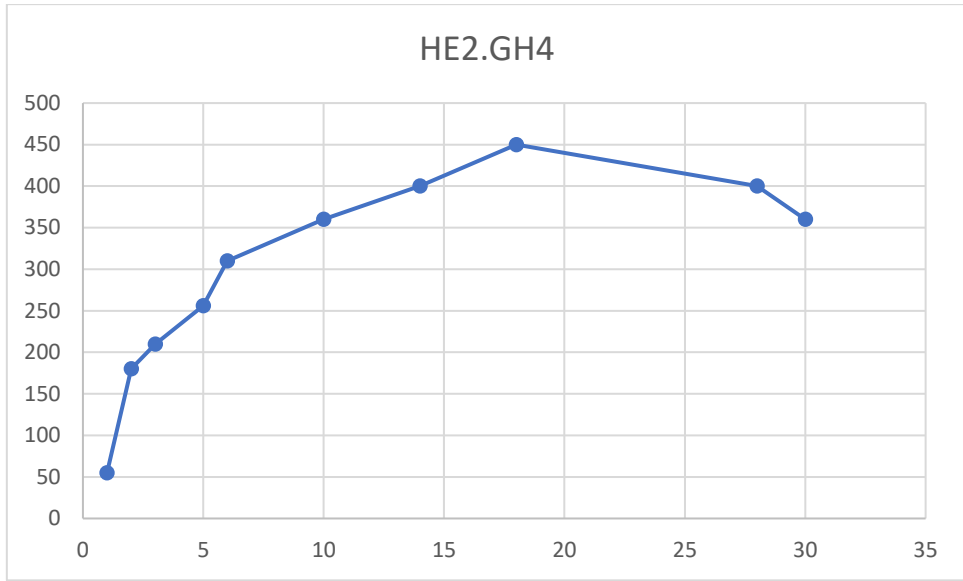
3) Reflective curves of HE1 The Y-axis show reflected power (W/h) and the X-axis show time (s)

HE1.3

HE1.2

HEC1.1

HEC1.2

HEC1.4GHN

HEC1.5GHN

HEC1.6GHACN