Universitetet
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**FACULTY OF SCIENCE AND TECHNOLOGY**

# MASTER'S THESIS

| Study programme/ Specialization<br><br>Environmental Engineering/ Offshore Environmental Engineering | Spring Semester 2018<br><br>Restricted Access |
| --- | --- |
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| Title of master´s thesis:<br><br>Treating Drill Cuttings with Susceptors in a Single-mode Cavity Microwave | |
| Credits (ECTS): 30 | |
| Keywords:<br><br>Drill cuttings, Drilling Mud,<br>Microwave, Single-mode Cavity,<br>High Power Density, Susceptor | Number of Pages: 113<br><br>+ Enclosure: 0<br><br>Stavanger, June 29, 2018 |

Submitted in partial fulfilment of the requirement for the degree of
Master *of* Science

# Treating Drill Cuttings with Susceptors in a Single-mode Cavity Microwave

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June, 2018

# Acknowledgements

First, I would like to thank my external supervisor Sindre Åse Lunde, for your guidance, patience and support throughout this project. I would also like to express my gratitude to Professor Torleiv Bilstad, for always having an open-door policy and giving me inspiration to reach to reach my goals. All lab personnel, especially Liv Margareth Aksland and Hans Kristian Brekken are also thanked for their support with the laboratory work.

Lastly, thank you all family, friends and my partner Ole Christian, for all of your support through thick and thin at my time at the University. Without your love and encouragement, I would not be where I am today.

# Abstract

The aim of the thesis was to evaluate the use of single-mode applicator in combination with susceptor technology for treatment of oil contaminated drill cuttings. Single-mode applicators allow samples to be exposed to high power density which increases the energy efficiency.

Oil separation was clearly enhanced with high power density for cuttings from Halliburton/North Sea. Less energy was used with high power density, where the result of OOC was determined to be 0,76%. For low power density, 2,24% OOC was achieved. This equals to a separation degree of 90,5 and 72,1, respectively, compared to initial OOC. The effect of susceptor was also tested on the Halliburton cuttings. The OOC was reduced to 0,16% after addition of MEG as susceptor and treatment in microwave.

More tests were conducted on a type of drill cuttings received from Canada. The effect of low and high power density, energy consumption, alternating cutting characteristics, susceptor quantity and dosing point was investigated in these experiments. The effect of high power density on oil separation was not evident without susceptor. The North Sea cuttings characteristics was to a high degree different from the Canadian cuttings. The best oil separation value for Canadian cuttings was 93,2%. This was an effect of using high power density, combined with dewatering in microwave before susceptor and salt was added. In some cases, 15% MEG showed to sufficient to remove oil below 1%, however some tests implied that increasing MEG concentration led to better oil separation.

The energy consumption was in general high for Canadian cuttings. Actions can be done to decrease the energy consumption, for example to pre-heat the susceptor and dose it on warm cuttings. Unfortunately, this was not included in this thesis and is still yet to prove.

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## List of Tables

## List of Equations

| | | |
|---|---|---|
| 1 | $\nabla \times \boldsymbol{E} = \frac{\partial \boldsymbol{B}}{\partial t}, \qquad \nabla.\mathbf{B} = 0$ | Maxwell Equations |
| 2 | $\nabla \times \mathbf{H} = \frac{\partial \boldsymbol{D}}{\partial t} + \boldsymbol{I}, \ \nabla.\mathbf{D} = \rho$ | Maxwell Equations |
| 3 | $\varepsilon' = 1 + \frac{N\alpha_p}{\varepsilon_0} = \frac{C}{C_0}$ | Dielectric Permittivity |
| 4 | $\varepsilon'' = \varepsilon''_d + \frac{\sigma}{\varepsilon_0 \omega}$ | Dielectric Loss Factor |
| 5 | $\dot{\varepsilon} = \varepsilon' - j\varepsilon''$ | The complex dielectric permittivity |
| 6 | $tan\delta = \frac{\varepsilon''}{\varepsilon'}$ | The Loss Tangent |
| 7 | $P_d = 2\pi f \varepsilon_0 \varepsilon'' E_i^2$ | Power Density |
| 8 | $D_p = \frac{\lambda\sqrt{\varepsilon'}}{2\Pi\varepsilon''}$ | The Pentration Depth |
| 9 | $A = \sum \frac{y_1 + y_2}{2} \times (x_2 - x_1)$ | Area for Trapezoid method |
| 10 | $Water\ content\ (\%) = \frac{M_{water}}{M_{wet}} \times 100\%$ | Water content |
| 11 | $OOC_{wet}\ (\%) = \frac{M_{oil}}{M_{wet}} \times 100\%$ | Retort, OOC wet |
| 12 | $OOC_{dry}\ (\%) = \frac{M_{oil}}{M_{wet} - M_{oil} M_{water}} \times 100\%$ | Retort, OOC dry |
| 13 | $OOC\ wet\ (\%) = \frac{W_2 - W_1}{W} \times 100\%$ | Soxtec, OOC wet |
| 14 | $A_2 = length \times width \times \#\ of\ blocks$ | Area for counting blocks |

## Abbreviations

| | |
|---|---|
| **AP** | Alkyl Phenols |
| **BAT** | Best Available Technique |
| **Bp** | Boiling point |
| **DEG** | Diethylene Glycol |
| **DPG** | Dipropylene Glycol |
| **EM** | Electromagnetic |
| **EMS** | Electromagnetic Spectrum |
| **IRIS** | International Research Institute of Stavanger |
| **MEG** | MonoethyleneGlycol |
| **MPG** | Monopropylene Glycol |
| **NEA** | Norwegian Environment Agency NOROG Norwegian Oil and Gas Association NCS Norwegian Con |
| **OBM** | Oil Based Mud |
| **OCDC** | Oil Contaminated Drill Cuttings |
| **OOC** | Oil on Cuttings |
| **OSPAR** | Convention for the Protection of the Marine Environment of the North East Atlantic PAH Polyar |
| **PAH** | Polycyclic Aromatic Hydrocarbons |
| **SBM** | Synthetic Based Mud |
| **TCC** | Themomechanical Cuttings Cleaner |
| **TEG** | Triethylene Glycol |
| **TWT** T | ravelling Wave Tube |
| **WBM** | Water Based Mud |
| **Wt%** | Percent by weight |

# PART 1- BACKGROUND

# 1. Introduction

## 1.1 Background

Drill cuttings are by-products from drilling activities and are made when formation is crushed by a drill bit. Drilling fluid is continuously added to the formation to remove the cuttings in order to drill the well further. North-Sea drill cuttings usually contain 5-15% oil from mud when they arrive topside and must be separated before discharge according to the Oslo Paris Convention (OSPAR) legislation (J. P. Robinson et al., 2009). OSPAR is the current legislation in the north-west Europe, including the Norwegian Continental Shelf (NCS). The commission seeks to protect the marine environment. OSPAR has set the limit for discharge of oil contaminated substances to be <1% (Norwegian Petroleum Directorate, 2012). Laws are different in other areas, such as the in Gulf of Mexico and North America, where the limit is set to 6,9% (Gerard and Antle, 2003). These laws are however changing, forcing North American platforms to reduce the discharges of hydrocarbons.

The oil and gas industry are facing multiple environmental challenges, one of them to handle contaminated drill cuttings. Oil contaminated drill cuttings were allowed to be discharged to sea until 1992, but accumulating cutting piles were observed around the platform. Regulations and pressure from governments are forcing companies to develop technologies that meet requirements. Common practices have been to reinject the cuttings or “skip to shore” for further treatment. Both of these methods are costly and cause negative environmental impacts.

Companies are competing to design a plant that can minimize environmental impacts, HSE risks and to meet limitations such as footprint and energy consumption. The ideal solution for separation of drill cuttings would be to do a complete separation process offshore, where oil is reused, while cuttings and water is discharged. In addition to reducing emissions from tankers and HSE benefits, this arrangement would reduce the space needed for storage of drill cuttings. An offshore solution would also make it easier for rigs in icy areas to have continuous treatment, preventing the platform to shut off drilling.

Current Best Available Technology (BAT) on the NCS for treatment of drill cuttings is the Thermomechanical Cuttings Cleaner (TCC). Total E&P tested it on the Martin Linge field but was denied permission after some time because the TCC plant did not reduce the OOC to 0,05% by weight, which was a limit determined by the Norwegian Environment Agency (NEA).

## 1.2 Problem description

Multiple sources have identified a pilot plant using microwave irradiation to treat the OOC to an acceptable amount (Perira, 2012, Robinson et al., 2010). Current technology developed by Norwegian Technology AS has proved to treat cuttings well below the requirement set by OSPAR. The new microwave pilot plant designed by NT allows samples to be exposed to high power density. The benefits of single-mode cavity are reduced footprint and treatment time, in addition to increasing the oil separation using less energy. The addition of an organic susceptor in the treatment step is brainchild of NT and has proven to increase the process temperature that enhances oil separation.

The combination of single-mode cavity and susceptor has not been investigated before by Norwegian Technology AS (NT). The effects of alternating cuttings characteristics with susceptor in regard to oil separation were examined in this project. The real energy input was also determined because of the software that followed the microwave and its ability to measure energy reflected.

## 1.3 Thesis Objectives

The aim of this thesis was to study the use of a single-mode cavity with susceptor for treatment of OCDC. Practical analysis´ in the laboratory was used to assess effects. The following tasks were conducted in order to achieve the goal.

I. Optimizing the unit
    i. Troubleshooting and optimizing were done to cancel challenges that occurs in microwave treatment, for example re-condensation of gas.

II. Determine the effect of high and low power density on two different types of drill cuttings:
    i. Two types of drill cuttings were exposed to both high and low power density to see the effect in a single-mode cavity
III. Alternating cuttings characteristics:
    i. Changing the cuttings characteristics could improve the oil separation
IV. Investigate susceptor inmixing point and dosing quantity
    i. The effect of pre-treatment with microwave before susceptor dosing was assessed
    ii. The susceptor quantity was alternated to see effect on oil separation

## 1.4 Collaboration with the Industry

This thesis was initiated and performed with help and support from *Norwegian Technology.*

The company specialize in treating and disposing any type of industrial water, onsite and offsite. They also offer chemical solutions to treat and destabilize completion and drilling fluids. The company has a strong environmental focus and aim to deliver solutions that are easy to operate with minimum moving parts and high treatment capacity. Furthermore, their technologies have low footprint and weight, enabling the technologies to be implemented both onshore and offshore.

NT was earlier this year awarded a patent on their latest concept that separate drill cuttings offshore. The oil from the mud will be re-used, while clean cuttings and water are allowed to be discharged to sea without environmental pollution. The patent is based on microwave irradiation combined with chemicals which minimizes energy consumption.

## 1.5 Novelty of Research

The use of microwave is used in many industries. A pilot plant was built on the initiative from John Robinson at the University of Nottingham. According to tests from Nottingham, oil levels less than 1 wt% have been obtained.

The use of organic susceptors in a multimode microwave treatment have been evaluated in earlier works for NT. Organic susceptors requires less energy compared to water and provide high process temperatures during treatment. Significant costs can be saved using this technology, due to high process temperature and because the oil contributes with a higher vapor pressure than water.

# 2. Process and waste related to drilling

## 2.1 The drilling process and use of drilling fluids

Oil is withdrawn from reservoirs through wells that are drilled in a process known as rotary drilling. The process starts with a large diameter drill bit crushing the formation. As the process advance, smaller diameter drill bits are used. Drill cuttings is formed when the formation is crushed. The drill bit is located at the tip of a drill string. Drilling fluids are circulated downwardly to the bottom of well through the drill string. The drilling fluids return to surface by circulating upwardly in the annulus between the drill string and the interior of the borehole (Figure 1) (Rigzone, 2018). The use of drilling fluids is a crucial part of offshore drilling activities. Drilling fluids, also called drilling muds, are used for multiple reasons. The main functions of the fluid are to add pressure to the well, remove cuttings from the wellbore and bring the cuttings to surface for treatment (El-sayed & El-Naga, 2001). Drilling fluids are also used to remove heat from the formation, to control corrosion, transmitting power to the drill bit and to act as a filter on the wall to prevent fluids from going into the formation.

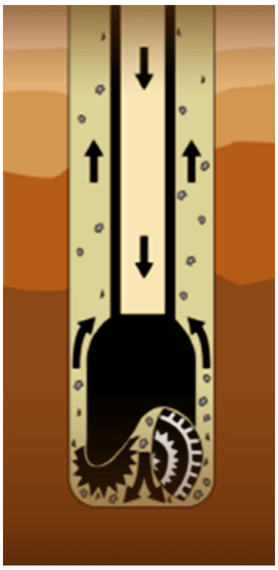


*Figure 1: Drill cuttings crushed by drill bit circulating in the drilling fluid flow*

The only way to transport the cuttings to the surface is to use drilling fluids. This an essential part in order to keep the borehole clean. Each fluid is specially designed to meet different criteria for each reservoir, and abandonment of the well can be the worst outcome if the drilling fluid has wrong properties (Bourgoyne & Bourgoyne, 1986).

Three types of fluids are available today; Water- based Mud (WBM), Oil-based Mud (OBM) and Synthetic-based Mud (SBM) (El-sayed & El-Naga, 2001). WBM can be discharged directly so sea because oil content is under 1%. OBM and SBM are considered as hazardous but these muds are used in environments where WBM is not suitable, for example in drilling for deep reservoirs. It is predicted that future oil and gas activities will occur in more difficult environments with more complex drilling operations, where WBM will not be able to perform its objectives. It is argued that OBM and SBM should be avoided due to environmental and economic reasons (Shah et al., 2010).

### 2.1.1 Water-based mud

Water-based muds is also referred to as aqueous drilling fluids. In WBM´s, water act as the continuous phase and it can be either fresh water, brine or seawater (El-sayed & El-Naga, 2001). WBS are attractive because of the constituents are less environmental damaging, which is an increasing concern for operators. It is therefore done much research to improve performance of WBS in order to compete with OBM and SBM (Williamson, 2013).

### 2.1.2 Oil-based mud

There are mainly three types of OBM used today. Common for OBM is that oil is the continuous phase. OBM are used in environments facing tough conditions, such as high temperature and pressure and difficult formations. The different types of OBM are:

<u>True oil base mud:</u> This mud contains less than 5% water and other substances such as oxidized asphalt and organic acids. True base oil mud is good for wells with high temperature, but the mud is toxic and can impact the environment (El-sayed & El-Naga, 2001). This type of mud is not used on the Norwegian Continental Shelf (NSC) today.

<u>Low aromatic nontoxic oil base mud:</u> Low aromatic nontoxic oil base mud acts the same way as the true oil base mud and has a low toxicity. The reason why this mud is not often used is due to insufficient amounts in the marked and high costs.

Inverted emulsion oil base mud: Oil is the continuous phase in the emulsion, but water content can be up to 50% in the form of dispersed droplets (El-sayed & El-Naga, 2001). This mud is also similar as the true base mud. The mud is easy to use, but contains up to 10% diesel, therefore special treatment is necessary to ensure no environmental damage.

### 2.1.3 Synthetic-based mud

In synthetic mud, a synthetic fluid is the external phase. They are more degradable and has lower toxicity than other OBM (Schlumberger, 2018), but can still be used in challenging environments found on the NSC. SBM can based on esters, ethers or olefin-based. The mud is very expensive but is currently just used most on the NCS.

## 2.2 Drilling waste

Cuttings, drilling fluids, slop and produced water coming up from the well are considered as drilling waste. Slop water is a mixture of waters that is collected onboard a rig, such as wash water containing soap and chemical residues, rain runoff and drilling muds. Produced water is an effect from the production of crude oil and gas. In 2016, nearly 530000 tonnes of drilling waste had to be treated (NOROG, 2017). Oil contaminated drill cuttings comprised of 259000 tonnes from the waste.

During the mid-1990´s, discharge of oil contaminated drill cuttings was the largest contributor to hydrocarbons entering marine environments on the NCS (Bakke, Klungsøyr, & Sanni, 2013). The cuttings size, shape and texture vary depending on reservoir rock. Cuttings go through primary separation in shakers, hydrocyclones and/or centrifuges. A secondary treatment step is necessary as some oil will still remain on the cuttings after primary separation. The usual practices for treating cuttings have been to slurry and reinject, or to transport the cuttings to shore. An increase in "skip to shore" is seen from 2009 due to problems with injection (Figure 2 and Figure 3) (NOROG, 2017). The major issues of reinjection are plugging of well, leakages and corrosion (Gumarov et al., 2014). The latter option is not without problems either. Skip to shore is quite expensive and the environmental impact of emissions from tankers are distinct. Furthermore, health, safety and environment (HSE) risks are associated with the transfer of the waste (Paulsen, Omland, Igeltjørn, Aas, & Solvang, 2003), for example issues concerning loading/offloading, crane lift, accidental oil spills and human accidents. This method also faces challenges during harsh weather.

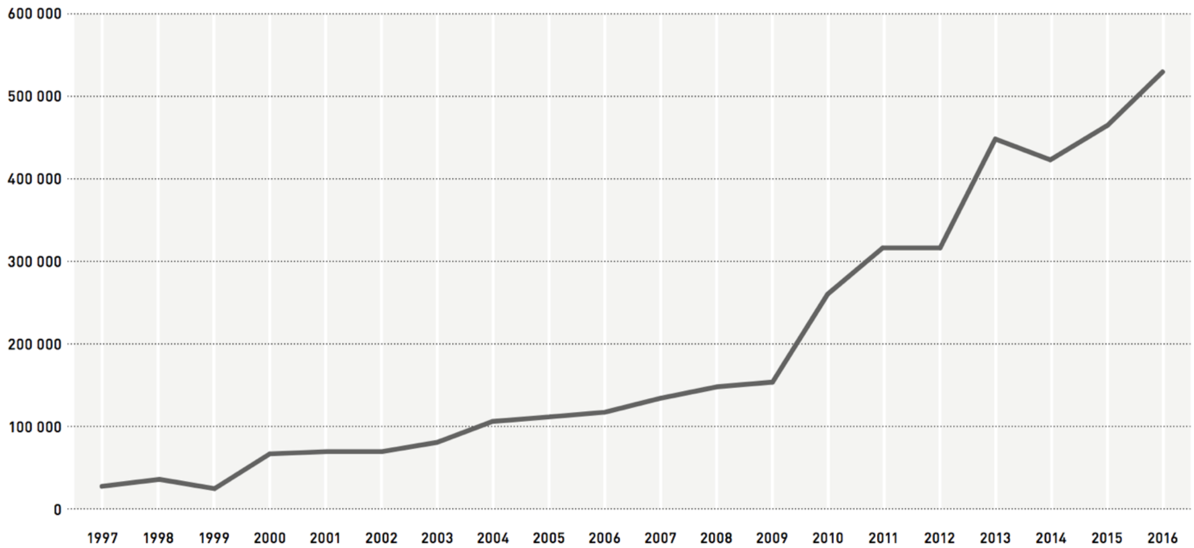



*Figure 2: Hazardous waste sent ashore from drilling rigs*

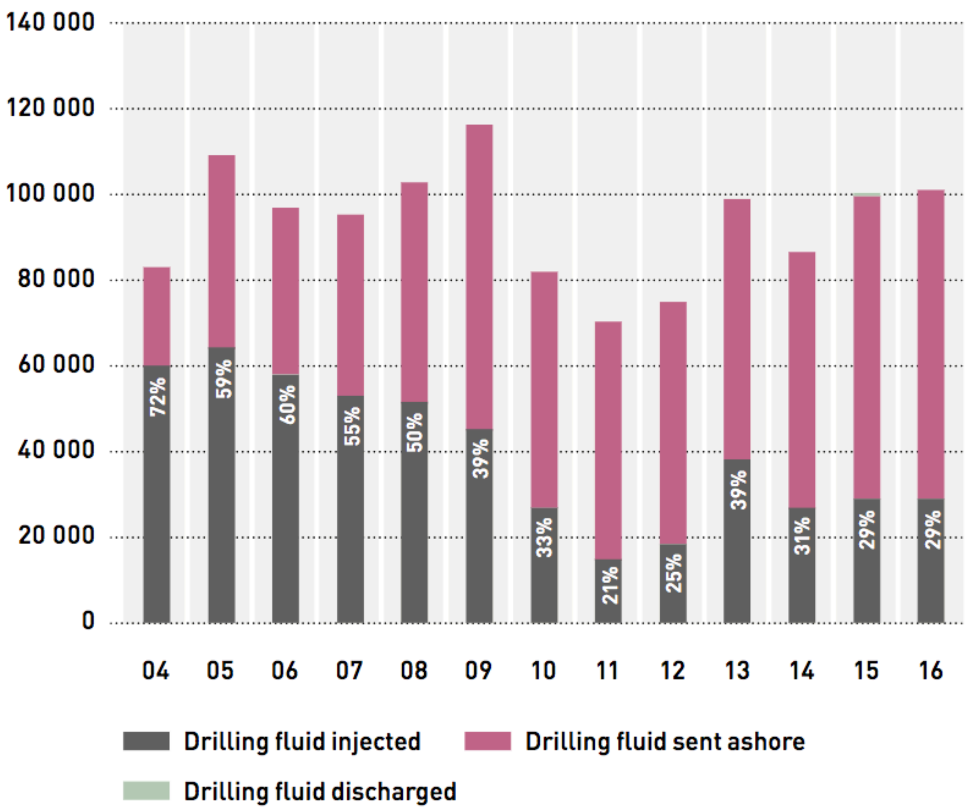



*Figure 3: Disposal of oil contaminated drill cuttings (tonnes)*

The Thermomechanical Cuttings Cleaner (TCC) is considered as BAT and has been tested on the at Martin Linge field. The operator, Total E&P, was permitted by the Norwegian Environmental Agency (NEA) to release 0,05% after treatment with the TCC. The limit for discharge was strict compared to the 1% rule in the OSPAR legislation. The TCC had shown good results on the UKCS. The technology was considered as a pilot project by NEA and was meant to give an indication of whether the TCC could be used on the NCS. The treatment process was stopped in 2015 due to several deviations revealed by NEA. For example, average oil on cuttings was determined to be 0,38%, exceeding the permitted levels given by NEA (Lie, 2017). However, the TCC will be used on Johan Sverdrup with permission to discharge 0,3% oil on cuttings (OOC). Marine Scientists argue that this will lead to toxic environments and a decrease in species richness (Lie, 2017).

## 2.3 Toxicity testing and environmental impact of oil contaminated drill cuttings

Every year, toxicity tests are conducted to examine influence of drilling waste on marine ecosystems. Impacts from discharge of drill cuttings was significant before the OSPAR legislation was implemented in 1993. Signs of fauna disturbance was evident more than 5 km from some platforms (Bakke et al., 2013). This has fortunately changed after 1993 and any effect of OOC is usually not detected beyond 500 meters. However, years after discharges of oil contaminated drill cuttings, high concentrations of oil and little indications of biodegradation are apparent in sediments, especially in the deep anaerobic sediment layers (Daan, Booij, Mulder, & Weerlee, 1996). Polycyclic aromatic hydrocarbons (PAH) and alkylphenols (AP) occurring from drilling waste have been detected in an accumulating amount in caged cod and blue mussels (Bakke et al., 2013). It is evident that PAH has carcinogenic potential via DNA adduct formation (Pampanin & Sydnes, 2013).

International Research Institute of Stavanger (IRIS) will carry out environmental tests throughout this project to determine the toxicity and environmental impact of cuttings after treatment.

## 2.4 Technologies used for drill cuttings available today

### 2.4.1 Cost related to injection, treatment and disposal of drill cuttings

For all types of treatment solutions, maintenance and operation costs are included. If cuttings were to be injected, a well must be drilled. The costs related to this depends on equipment available for drilling, the lifetime of the well and the well capacity. Costs related to "Skip to shore" will vary according to distance to shore, price, treatment equipment onshore and transport capacity and facility. Treating the cuttings offshore will only depend on the equipment. Karlsen (2012) estimated the total costs for the three options (Table 1). Significant costs can be saved utilizing an offshore solution.

*Table 1: Cost estimation of injection and treatment onshore and offshore*

| Cost | Re-injection | Onshore | Offshore |
|---|---|---|---|
| Total (NOK/ton) | 9600 | 9000 | 6500 |
| Total (EUR/ton)[1] | 1003 | 940 | 679 |

### 2.4.2 Comparison of technologies

Statoil is one of many companies that have implemented a waste minimization hierarchy, where the first rule is to prevent waste generation, the second to reduce waste and lastly to reuse and recycle the waste as much as possible (Jensen, Paulsen, Saasen, Prebensen, & Balzer, 2004). In order to treat and recycle waste offshore, better, more efficient and compact technologies are necessary to reach the zero-discharge goal. The ideal solution for separation of drill cuttings would be to do a complete separation process offshore, where oil is reused, and cuttings and water discharged. In addition to reducing emissions from tankers and HSE benefits, this arrangement would minimize space needed for storage.

When cuttings and mud arrive platform, they are separated in what is called the primary treatment step. Here, the largest pieces of cuttings are removed before smaller and finer

[1] 1 EUR= 9,57 NOK (Conversion rate 13.05.18)

pieces of cuttings are separated in shale shakers (Melton et al., 2004). A secondary treatment step is necessary in order to dispose the waste safely.

Many technologies have been suggested as secondary treatment, but requirements such as space, capacity, efficiency and cost are not met. Ormeloh (2014) did an evaluation of treatment technologies used for drill cuttings and his findings are summarized in table 2. A more detailed explanation is given below the table.

*Table 2: Overview over treatment solutions for drill cuttings*

| Method | Offshore Usage | Cleaning/Disposal Mechanism | Usable End Product |
|---|---|---|---|
| **Incineration** | No | Oxidation or combustion of organic components | No End Product |
| **Indirect Thermal Desorption** | No | Evaporation and Condensation of Oil and Water | Oil as fuel |
| **Thermal Treatment/ Thermomechanical Cuttings Cleaner** | Yes | Evaporation and Condensation of Oil and Water | Oil as new base oil/oil as fuel |
| **Bioremediation/ Land farming** | No | Biodegradation | No End Product |
| **Dispersion by chemical reaction** | No | Solidification, oil/metals stabilized in cuttings matrix | Construction Material |
| **Cuttings Dryer** | Yes | Centrifuge forces mud/ solids separation | Drilling Fluid |
| **Cutting Re-injection** | Yes | Injection of slurrified cuttings | No End Product |
| **Microwave Treatment** | Yes | Magnetic field transfers energy to water | Oil as New Base Oil |

| | | | |
|---|---|---|---|
| **Liquefied Gas Extraction** | No | Liquefied HC gases solve and remove oil | Oil as New Base Oil |

The process where waste is oxidized or combusted between 1200-1500 °C is called **incineration** (Ifeadi, 2004). Large volumes can be treated in the rotary kilns. Incineration has been used to treat drill cuttings, but is not ideal for inorganic components, because they can cause problems in the flue gas. It is very energy consuming, especially for waste with high water content. Large amounts of $CO_2$ and $NO_x$ are also produced.

As opposed to incineration, **thermal desorption** operates at lower temperatures (250-350 °C) in a distillation process (Ifeadi, 2004). Higher temperatures (520 °C) are needed if the oil contains heavy carbons. Water is vaporized first, and the formed steam lowers the boiling point for the oil, therefore, the temperature is lower. Both components condensates and can thereby be reused in drilling mud or as fuel. A second step is sometimes required if the cuttings are containing environmental hazardous components (Ifeadi, 2004).

Thermal treatment options heat the drill cuttings. The heat is sufficient so that the oil and water is vaporized from the solids, thereby leaving the cuttings “clean”. The quality of the remaining oil is not good enough for reuse (Vik, Blytt, Stang, Henninge, & Kjønnø, 2014). Examples that uses this kind of technology are the TCC, Solid recovery (SDR) and thermal phase separator (TPS) (Vik et al., 2014). These technologies require a large area, and as space is often limited on platforms, these are often not ideal for offshore use.

**The TCC** is the most promising technology of the thermal technologies. Frictional heat is generated by crushing rocks and when a sufficient temperature is reached, the cuttings are added. In addition to the motor size, the capacity and energy consumption depends on oil and water content. An offshore TCC was installed at the United Kingdom Continental Shelf (UKCS) and was considered as a proven technology (Norsk Olje og Gass, 2014). The TCC is mostly applied onshore in Norway (Ormeloh, 2014), but has been tested on the Martin Linge field.

*Figure 4: The TCC by Thermtech (Environmental Expert, 2018)*

Microbial degrading of hydrocarbons is called **bioremediation**. Under aerobic conditions, bacteria and fungi will use the hydrocarbons as energy source. These containers require large space. Cuttings can also be spread on land close to the source. As long as the cuttings are evenly spread out, organisms in the soil will degrade the hydrocarbons. This is called **land farming**. Bioremediation and land farming are both dependent on properties like temperature. Land farming has been questioned due to hazardous components in the cuttings and is currently not allowed in Norway.

**Dispersion by Chemical Reaction (DCR)** immobilize hydrocarbons by stabilize and solidify the cuttings (Ifeadi, 2004). The solidification will require large space area. It is a two-step process, where the first step is called pre-distribution step. Here, the components of dispersing chemical reaction are firstly charged with the substance to be dispersed. The second step is called the dispersing step, and here the actual chemical reaction occurs.

**The Cuttings Dryer** uses centrifugal forces to extract the fluids form the cuttings. This treatment method does not extract enough oil to meet the OSPAR requirements, hence further treatment is needed (Offshore, 2007).

**Microwave** pilot plants have been developed and have shown great ability to meet the requirements of <1% oil in the laboratory (J. Robinson et al., 2009). Energy in the form of an electric wave is dissipated to the absorbing field and molecular interactions occur. (J. Robinson et al., 2009). As opposed to conventional heating that takes very long time, microwaves heat individual molecules and therefore reduces time (Meredith, 1998). An offshore microwave will also have reduced foot print and weight due to shorter residence time. In addition to this, the microwave requires little utility and can function only on an electrical power source. Furthermore, selective heating is possible, where one phase is transparent and the other is absorbing. This will increase the energy efficiency. In the process of treating drill cuttings, the oil and solids acts as the transparent and a chemical/water is the absorbent.

# PART 2- THEORY

# 3. Microwave theory

In the electromagnetic spectrum (EMS), microwaves usually have frequencies between 300 to 30000 MHz, which equals about 1 mm to 1 m in wavelength (Figure 5) (Thostenson & Chou, 1999).

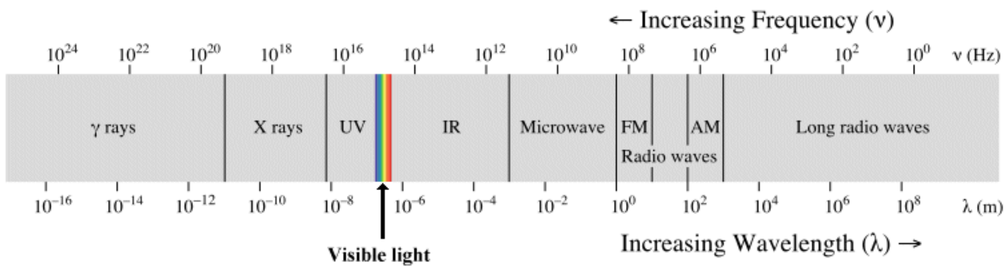



*Figure 5: The Electromagnetic Spectrum (Humboldt State University, 2018)*

Microwaves are common in ovens, radars and Wi-Fi. In ovens, microwaves are used to heat materials that have poor electric conductivity. Heat is according to Cambridge Dictionary a form of energy that a substance has due to increased motion of molecules at microscale level (Cambrigde Dictionary, 2018). Heat is transferred due to temperature differences, and temperature is the quantum size of heating. In conventional heating, conduction, convection and radiation transfers heat from the surface to the center of the material. Materials that are exposed to microwaves are heated throughout the material because of interactions between dielectric molecules (Meredith, 1998). Motion in the form of rotation of the dipoles will occur because molecules will try to align with the electric and magnetic fields that move very rapidly (Figure 6) (Sumper, Baggini, & Sumper, 2012). The interaction between molecules causes friction that produces heat.

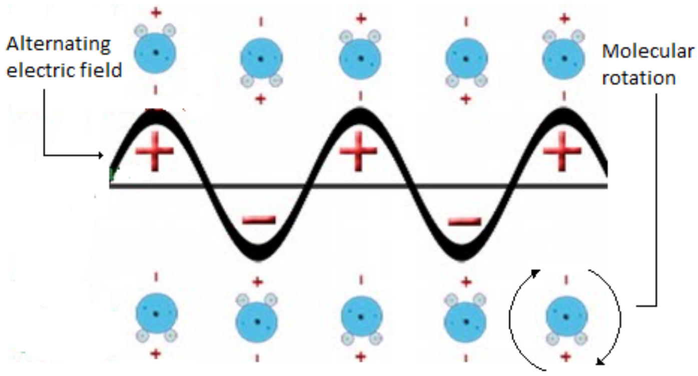



*Figure 6: Water molecules trying to align when exposed to electromagnetic energy (Sites.google, 2018)*

## 3.1 Electromagnetic Theory

Electromagnetic waves are according to Maxwell a combination of the magnetic and electric field. Maxwell also demonstrated that if the electric field is changing, the magnetic field will also change, and opposite; changing the magnetic field will change the electric field. Electric and magnetic fields are perpendicular to each other and the propagating wave (Figure 7) and the dependency of them forms the Maxwell equations (Equation 1 and 2) (Thostenson & Chou, 1999):

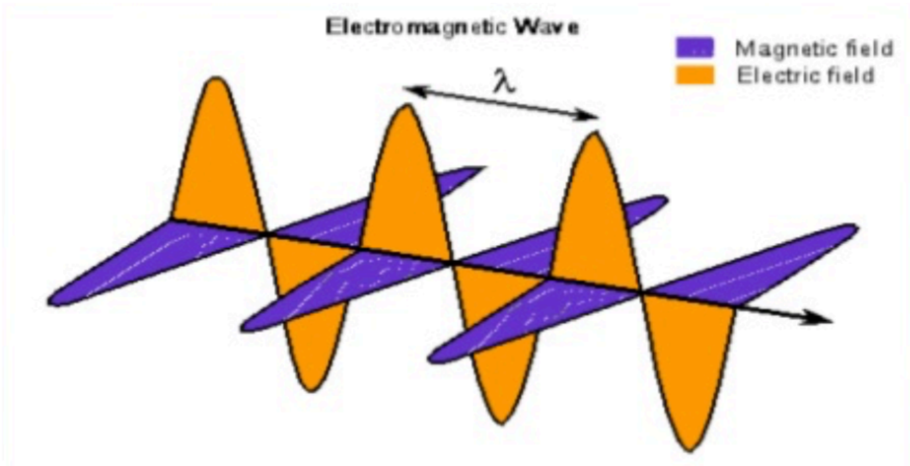



*Figure 7: Magnetic and electric field propagating with electromagnetic wave (Lembalemba, 2015)*

$$\nabla \times \boldsymbol{E} = \frac{\partial \boldsymbol{B}}{\partial t}, \qquad \nabla . \mathbf{B} = 0 \tag{1}$$

$$\nabla \times \mathbf{H} = \frac{\partial \boldsymbol{D}}{\partial t} + \boldsymbol{I}, \ \ \nabla . \mathbf{D} = \rho \tag{2}$$

Where $\boldsymbol{E}$ = electric field vector, $\boldsymbol{B}$ = magnetic flux vector, $\mathbf{H}$ = the magnetic field vector, $\mathbf{D}$ = the electric flux vector and lastly $\boldsymbol{I}$ = the current density vector. The charge density is $\rho$.

**Electric Permittivity**

There are two important properties in dielectric heating, $\varepsilon'$and $\varepsilon''$. A dielectric is polarized due to application of an external electric field. An internal field is created due to resistance within the dielectric when the external field is applied. The relationship between the permittivity of the dielectric (internal field) and vacuum (external field) is known as **the dielectric constant, $\boldsymbol{\varepsilon'}$**. It is also called the relative dielectric permittivity and describes the ability for a material to store energy (Equation 3).**Feil! Ingen sekvens spesifisert.**

$$\varepsilon' = 1 + \frac{N\alpha_p}{\varepsilon_0} = \frac{C}{C_0} \tag{3}$$

$\alpha_p$ = polarizability of the medium, N = particle concentration. C = internal field, $C_0$= external field in vacuum.

**The dielectric loss factor**, $\boldsymbol{\varepsilon''}$, is the result of ion-, dipolar- and electronic mechanisms that causes polarization (Equation 4) (Thostenson & Chou, 1999), in other words, how well the material convert electric energy to heat. ε" <0,005 is considered as transparent and will not dissipate any heat in an applied field. Materials with loss factor over 0,1 are accepted as good materials for microwave heating.

$$\varepsilon'' = \varepsilon''_d + \frac{\sigma}{\varepsilon_0 \omega} \tag{4}$$

$\varepsilon''_d$ = the energy that is dissipated, $\sigma$ = ionic conductivity

**The complex dielectric permittivity**, $\dot{\boldsymbol{\varepsilon}}$, (Eq 5) describes the dielectric properties of a material and is given as the relationship between the dielectric constant (Eq 4) and dielectric loss factor (Eq 5):

$$\dot{\varepsilon} = \varepsilon' - j\varepsilon'' \tag{5}$$

The loss tangent, $\delta$ (Eq 6), also describes the materials dielectric loss. For a material to be characterized as a good absorbent it must have tan$\delta \geq 1$. Materials with tan$\delta \leq 1$ will not absorb microwaves good.

$$tan\delta = \frac{\varepsilon''}{\varepsilon'} \tag{6}$$

The power density, $P_d$ (Eq 7), in a material refers to the amount of power per unit volume and is essential in this thesis to determine the effect of oil separation on high and low power density. Power density is further explained in section 4.1.

$$P_d = 2\pi f \varepsilon_0 \varepsilon'' E_i^2 \tag{7}$$

f= frequency (Hz), $\varepsilon_0$= permittivity in free space and $E_i$= internal voltage stress ($Vm^{-1}$).

The ability for microwaves to penetrate into a dielectric material is called the penetration depth, $D_p$ (Eq 8). A low $D_p$ is desirable in order for the microwaves to penetrate and heat the whole material, not only the surface (Pereira, 2012). A higher wavelength ($\lambda$) will increase the penetration depth. A high dielectric factor ($\varepsilon''$) will decrease $D_p$.

$$D_p = \frac{\lambda\sqrt{\varepsilon'}}{2\Pi\varepsilon''} \tag{8}$$

## 3.2 Set up in Microwaves

The main components in a microwave consists of a microwave generator that creates energy, a transmission component that transfer the energy to sample, power supply and heating applicators.

### 3.2.1 Generators

Microwaves are produced from power provided in vacuum tubes. The three types of vacuum tubes used today are travelling-wave tubes (TWT), klystrons and magnetrons. Magnetrons have a higher reliability and are cheaper to manufacture than the other two and is therefore often preferred. Another reason for choosing magnetrons over TWT and klystrons is because magnetrons convert the power to microwaves faster (Pereira, 2012). Vacuum tubes in the form of magnetrons are common in microwaves used in everyday life and is also the type of vacuum tube used in this thesis. Magnetrons only are therefore highlighted below.

The magnetron is usually a hollow cylindrical cavity composed of magnets and a vacuum tube. The cathode is placed in the center with a series of resonant cavities and anode around (Fig 8)(Webb, 2014). A potential difference is generated because the anode is placed at a higher potential. This difference equals the electric field.  Voltage makes electrons go from the cathode to towards the anode (Pereira, 2012). The magnetic field in microwaves is created by an external magnet.

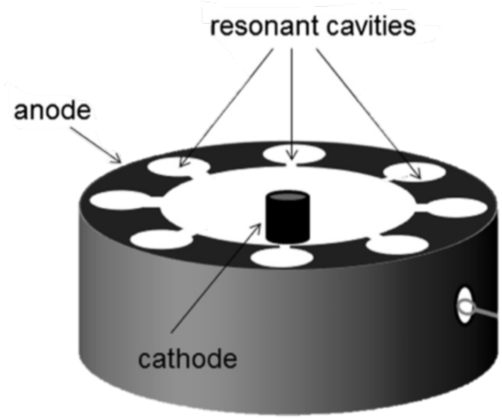



*Figure 8: Schematic figure of a magnetron cavity (Webb, 2014)*

### 3.2.2 Transmission lines/Wave guide

The microwaves that are generated in the magnetrons are transferred to the sample container in waveguides. The waveguides can be both cylindrical or rectangular, in straight and 90℃ bend sections (Thostenson & Chou, 1999).

### 3.2.3 Applicators

Applicators allow the microwaves to be transferred from the generator to the load. Applicators can be produced in different shapes, forms and for specific requirements, but since this thesis will be an investigation of single-mode and multimode mode applicators, these are the only ones that will be covered here:

#### *3.2.3.1 Single-mode applicators*

Microwaves in single-mode applicators are usually non-uniform distributed, the waves are however predictable, and it is therefore possible to place the material of interest where the field has highest intensity (Mehdizadeh, 2010). Some areas will experience overheating, while some areas will not be heated sufficiently. To get as even heating as possible, a tunnel applicator with self-cancelling reflection can be added (J. P. Robinson et al., 2010). The treatment time is shorter for single-mode. Because of the uneven heating in this type of applicator and geometry of materials , they are only designed for specific reasons (Thostenson & Chou, 1999).

#### *3.2.3.2 Multimode applicators*

The microwave used in daily routines at home is an example of a multi-mode applicator. In multimode applicators, distribution of electric waves is said to be random and less predictive compared to single-mode applicators (J. P. Robinson et al., 2010). This may result in overheating in some areas because of high power density, while other areas receive less power. Turntables are used to avoid this. This type of applicators is more flexible than single mode applicators for large scale operations, batch processing and objects with complicating geometry (Thostenson and Chou, 1999). High power density is hard to achieve in multimode cavities (H. Shang, Snape, Kingman, & Robinson, 2005), but this type of applicator can handle larger volumes compared to single mode.

# 4. Treating drill cuttings with microwave radiation

The use of microwave heating for oil separation of drill cuttings started testing in the beginning of the 21st century (H. Shang et al., 2005). Since then, there has been a great improvement of the technology, especially with respect to energy consumption (Pereira, 2012). This is mostly due to the use of a single-mode cavity, but other factors such as sweep gas, power density, particle size, cuttings characteristics and moisture content also impact the oil separation.

It has been shown that microwave heating can be more energy efficient than conventional heating and current BAT, which is the TCC. This is because microwaves are only absorbed by the water phase, while the oil phase is desorbed or stripped. Below are some advantages of microwave radiation, compared to BAT[2]

- Selective heating
- Homogenous heat distribution
- Proven technology
- Not crushing the particles
- No contact
- Increased capacity
- Smaller footprint and weight
- Energy efficient
- Robust
- HSE (noise emission and discharge control)
- Reduced loss operation
- Less downtime

[2] PowerPoint, 30.05.18: JIP presentation by Norwegian Technology

**Important treatment parameters and results obtained**

H. Shang et al. (2005) demonstrated that the oil content in drill cuttings was reduced to less than 1% after being exposed to over 3000 kWh/t (Figure 9). This was executed in a multi-mode applicator. Increasing the cavity power resulted in larger oil removal due to faster heating of materials. However, very high energy requirements are not competitive in today's industry. Single-mode applicators has been tested in the years after and proved to yield results under the OSPAR limit requiring only 110 kWh/t (Figure 10) (Pereira, 2012). Unpublished experiments[3] showed that OOC below 1% was possible with only 100 kWh/t. These tests however are conducted under ideal circumstances on full-scale equipment. Drill cuttings characteristics are specially selected with respect to oil and water content, porosity etc., to obtain the best results.  The development in energy efficiency of microwaves from 2005 to 2017 is illustrated in Figure 11.

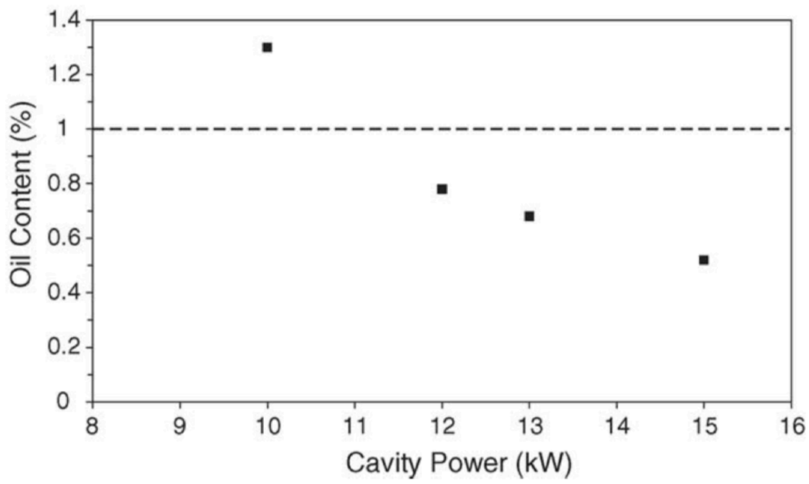



*Figure 9: Oil content after treatment with different cavity power in 2005*

[3] Sindre Åse Lunde, 28.05.18: Personal communication citing that results were achieved at the University of Nottingham/ J.P. Robinson.

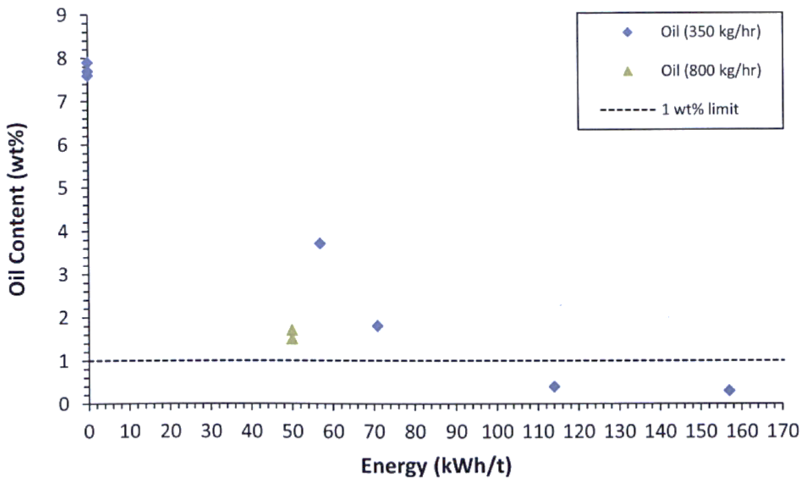



*Figure 10: Oil content after treatment with different energy input in 2012*

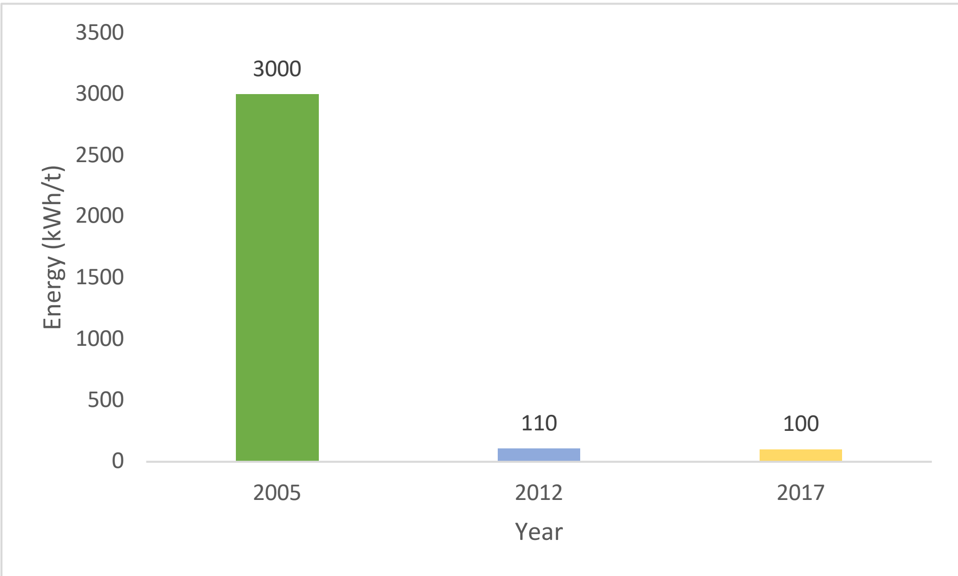



*Figure 11: Development in energy consumption from 2005 to 2017 using microwave for treatment of drill cuttings*

There are mainly three mechanisms used to explain how microwaves work on oil contaminated drill cuttings (J. P. Robinson et al., 2009). The first mechanism is through evaporation using a gas. The second one is entrainment, and the last mechanism is vaporization or steam distillation. Pereira (2012) identified the last mechanism as the best, while Ogunniran, Binner, Sklavounos, and Robinson (2017) argues that steam stripping is the dominant mechanism in microwave treatment of drill cuttings. The two mechanisms are often mistaken for being the same, however, they are based on two different physical principles (Figure 12). In steam distillation, the hydrocarbon phase is boiled. The water reduces the boiling point of the organic phase. In the process of steam stripping, transfers steam that is generated in-situ the hydrocarbon phase to the stripping gas.

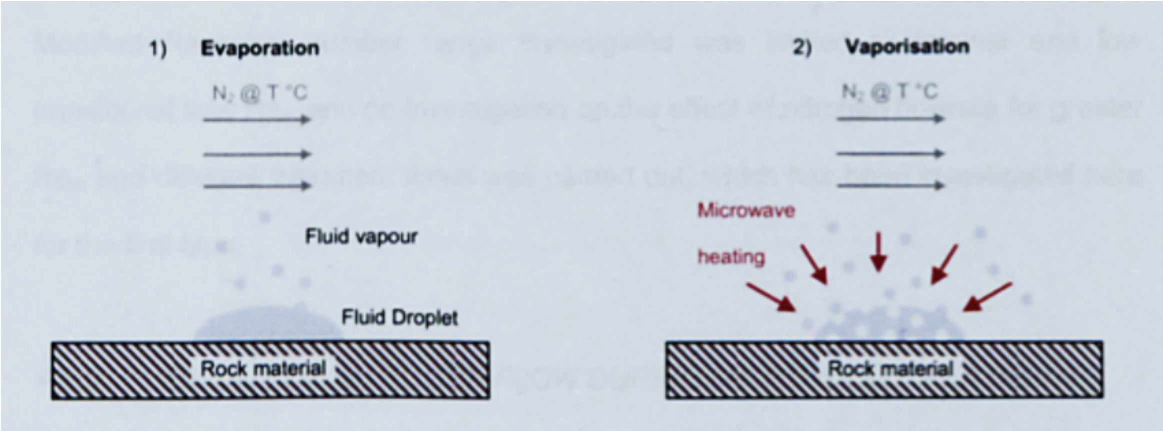


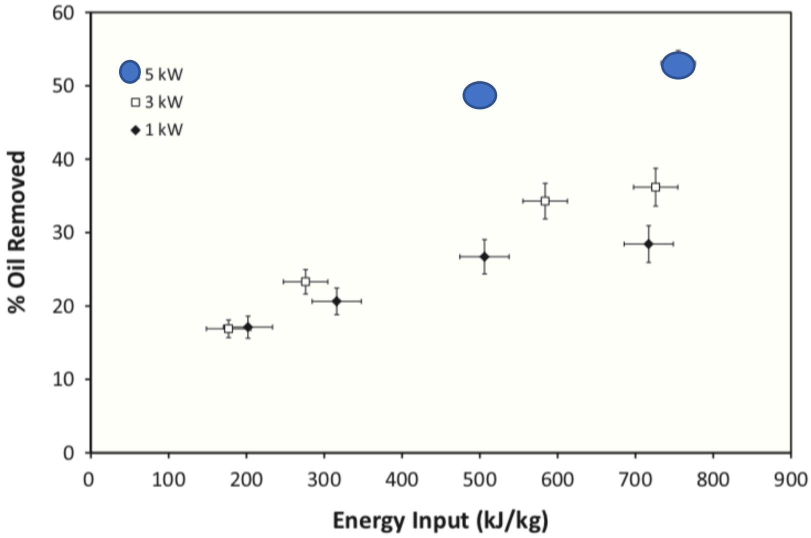

*Figure 12: Physical principle for steam stripping (evaporation) and steam distillation (vaporization) (Pereira, 2012)*

Ogunniran et al. (2017) claims that increasing the power density within the solid bed will increase the oil separation because steam is created faster with higher power density. Power density could also be changed if the geometry of the processing system is manipulated.

Power density is a measure of the amount of power that is transferred per unit volume. Haga (2017) investigated the effect of low power density. The major preference for using low density over high density equipment are HSE concerns. With high density equipment, the risk of breakdown voltage, arcing and explosions increases. However, from an energy and separation point of view, high density is better. High power is more efficient than low power to remove oil at equivalent energy input in microwave heating (Figure 13) (J. Robinson, Binner, Saeid, Al-Harahsheh, & Kingman, 2014). Note that the oil content was not reduced to under the OSPAR requirement in that experiment, the aim of the figures are only to show the effect of increasing power and heating time.

Haga (2017) demonstrated that pre-treatment of drill cuttings with low power density resulted in a very low oil separation, but a high-water separation. High power density increases the entrainment because the steam will be formed faster, and velocity will be high (Pereira, 2012). The entrainment effect will increase the oil recovery and decrease the retention time, using the same overall input.

a)




b)

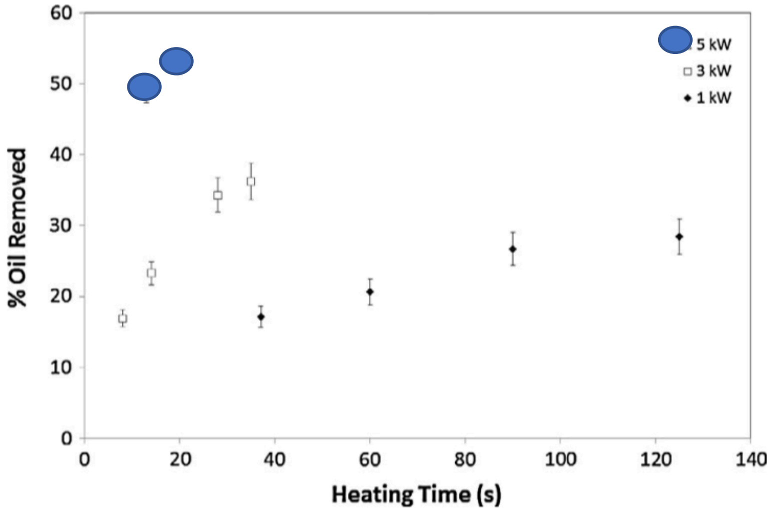



*Figure 13: Oil removal increases with a) increasing power and b) time*

As more and more water is removed in the process, the reflected power increases. This may lead to arcing and is more common for high power density processes compared to low power density processes. It occurs when microwaves strike a reflective surface. If a situation like this occurs (Figure 14), it can damage the equipment and lead to lost power (Meredith & Institution of Electrical, 1998). The chance of arcing to appear also increases if the cavity is built wrong.

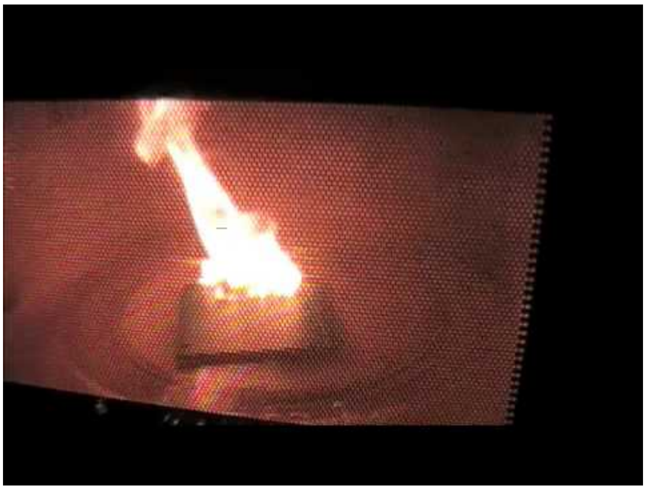


*Figure 14: Arcing in a Microwave(Sagar, 2012)*

The absorption of electromagnetic waves for dielectric materials usually increases with temperature. When the absorption is larger than the energy loss of the sample, an uncontrolled temperature growth will occur. This rapid, out of control increase in a materials temperature is known as thermal runoff or runaway (Semenov & Zharova, 2006). In situation were thermal runoff occurs, heat is distributed inhomogeneous in the sample, the result is areas that are heated more than others. These areas are known as hot spots (figure 15) and are often very rocky and rigid. More energy is absorbed when the material´s $\varepsilon''$ is higher, therefore the chance of thermal runoff increases with increasing $\varepsilon''$. It is often evident in multimode applicators because of the uneven distribution of energy.

*Figure 15: Hot spot as a result of thermal run-off (Egar, 2017)*

The addition of a susceptor will decrease the chance of arcing and thermal runoff, because there is a higher chance that there will be absorbing gas present. To maintain a safe temperature rise of a high density system, the amount of heat dissipated has to be decreased which means that the power supply´s efficiency must be increased (On semiconductor, 2017).

Using a sweep gas has shown to increase the oil separation (Pereira, 2012). An experiment by Pereira showed that when sample was exposed to 15 L/min of nitrogen for 2mins at 700W, decreased the weight of sample by 18 g (Figure 16). Sweep gas without radiation did not show any significant reduction.

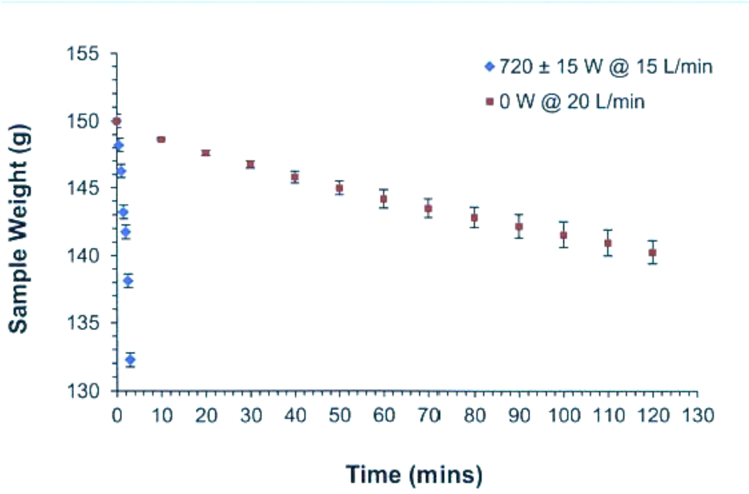



*Figure 16: Effect of nitrogen sweeping gas with and without microwave irradiation*

It has been shown that using nitrogen as sweep gas is important. Adding the gas has shown to increase the combined water and oil removal by sweeping the molecules out because it distributes the temperature in the sample, improves mass transfer and because re-condensation is less likely to occur (Pereira, 2012). Adding nitrogen as a sweep gas is not only necessary because of this, nitrogen gas is also necessary because it creates an inert environment and thus reduces the possibility for an explosion to occur. It has also proven that using nitrogen instead of air, will reduce the chance of susceptor to degrade (Mattikow & Cohen, 1943). The gas does not absorb microwaves and is not heated by the waves.

The liquids lay different in the cuttings (Figure 17). The hydrocarbons that lay near the surface are easy to remove. The hydrocarbon droplets that are trapped in small pores will be harder to remove. The phenomenon channelling occurs when the hydrocarbon droplets are trapped between pores (Ogunniran et al., 2017). The mass transfer of hydrocarbons in conventional heating can be limited even at very high gas velocities. Ogunniran et al. (2017) concluded that this phenomenon was reduced in microwave heating because steam is generated in-situ throughout the bed.

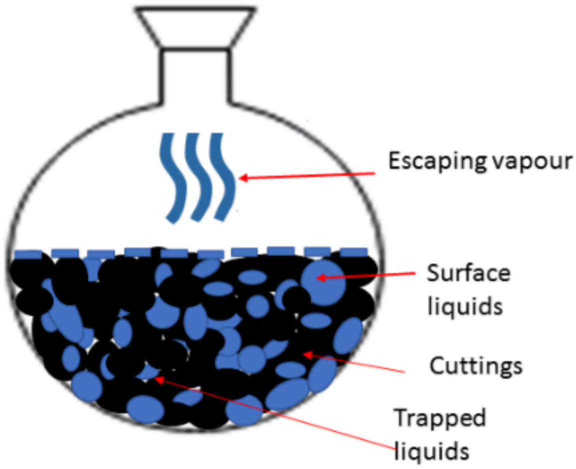



*Figure 17: Channelling of hydrocarbon droplets (Egar, 2017)*

Drill cuttings with low density is preferred because they often have larger surface areas, hence better contact for steam and oil droplets (Figure 18) (Pereira, 2012).

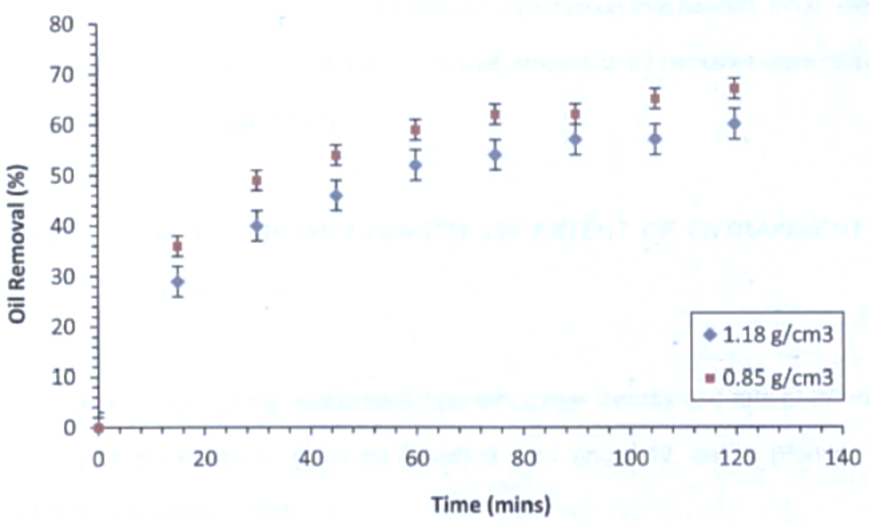



*Figure 18: The effect of density on oil removal*

Particle size will affect the radiation due to different electric field distribution in samples. Small particles have higher electric field strength than larger particles. It's easier to heat small particles because the heating path is smaller, and mass are easier transferred (Pereira et al., 2011). Pereira also showed that small particles contain more oil due to its larger surface area for more oil to adhere to (Figure 19).

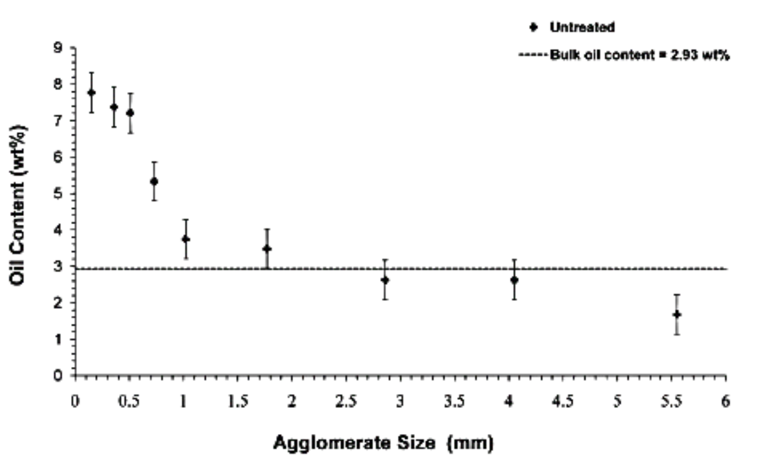



*Figure 19: Oil content decreases with increasing particle size*

Adding extra moisture has shown to have a positive impact on the oil separation (figure 20) (H. Shang et al., 2005). When moisture content was over 12%, oil levels were under OSPAR discharge limit of 1% in Shang´s experiments.

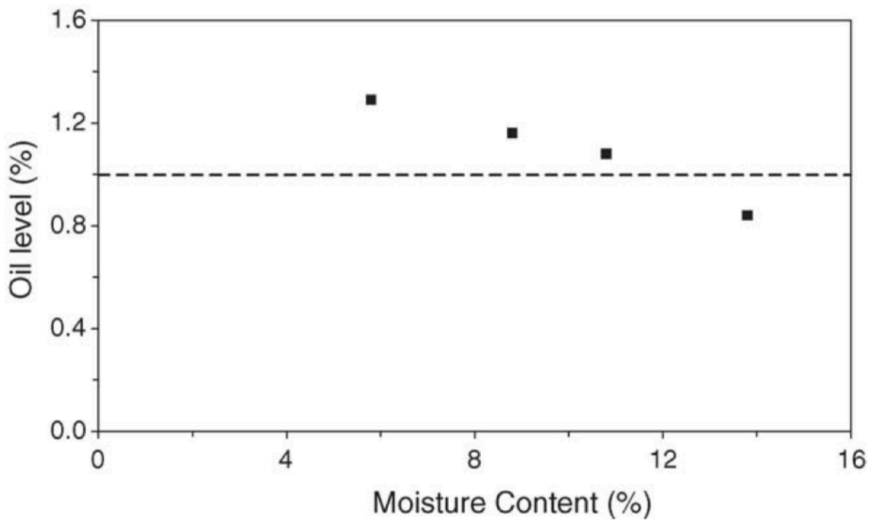



*Figure 20: Positive effect on oil levels of increasing moisture content*

Increasing the moisture level is supported by other authors. Microwave radiation together with MEG as susceptor has proven to decrease the oil concentration in cuttings (Egar, 2017; Haga, 2017).

Oil is considered as transparent and requires long heating time which reduces the energy efficiency of the treatment process. This is because the dielectric constant and loss factor is significantly smaller for oil than water (table 3) (Buttress et al., 2015). Oils with low boiling point need less energy and time compared to oils with high heavier fractions. Dosing the material with a susceptor will increase the dielectric properties of the sample and hence increase the energy efficiency.

*Table 3: Dielectric properties for water and fuel oil*

| Material | Dielectric constant, ε' | Dielectric loss factor, ε" |
|---|---|---|
| Water | 77 | 13 |
| Fuel oil | 2 | 0,002 |

**Evaluation of microwave technology for cuttings treatment**

Microwave technology has proven to treat oil contaminated drill cuttings to values less than the OSPAR regulation. The energy efficiency depends on factors such as degree of separation, particle size and cuttings characteristics. Cuttings with high viscosity oil is more challenging to remove in microwaves than low viscosity oil. Some oils with very high viscosity demonstrated to be unappropriated for microwave technology in experiments conducted by J. Robinson et al. (2014).

The oil separation increases with high power density. Arcing and thermal runoff are consequences that may occur with high power density. Cavity design is important in relation to arcing. Arcing can cause explosions and damage to microwave. With the correct design, the chance of arcing is neglected, however, it causes a risk element. It is also important to be aware of what one is doing when handling the equipment for safety reasons.

Microwave irradiation (100 kW/t) has lower energy consumption compared to BAT (200 kW/t) (Ormeloh, 2014), and there are therefore tremendous amounts of energy to save. It is necessary to mention that the cuttings characteristics are different in the tests comparing the microwave and TCC. Cuttings characteristics will affect the energy requirements. The trade-off occurs when the oil concentration becomes very low. The removal gets more challenging and requires more energy.

# 5. Materials and Methods

## 5.1 The microwave

The microwave was custom made for NT for this project. It is constructed by Fricke und Mallah (FUM) GmbH with 2,45 Gz with maximum output of 2000 kWh. This allows tests to be assessed with high power density. Figure 21 below illustrates the set-up in the microwave.

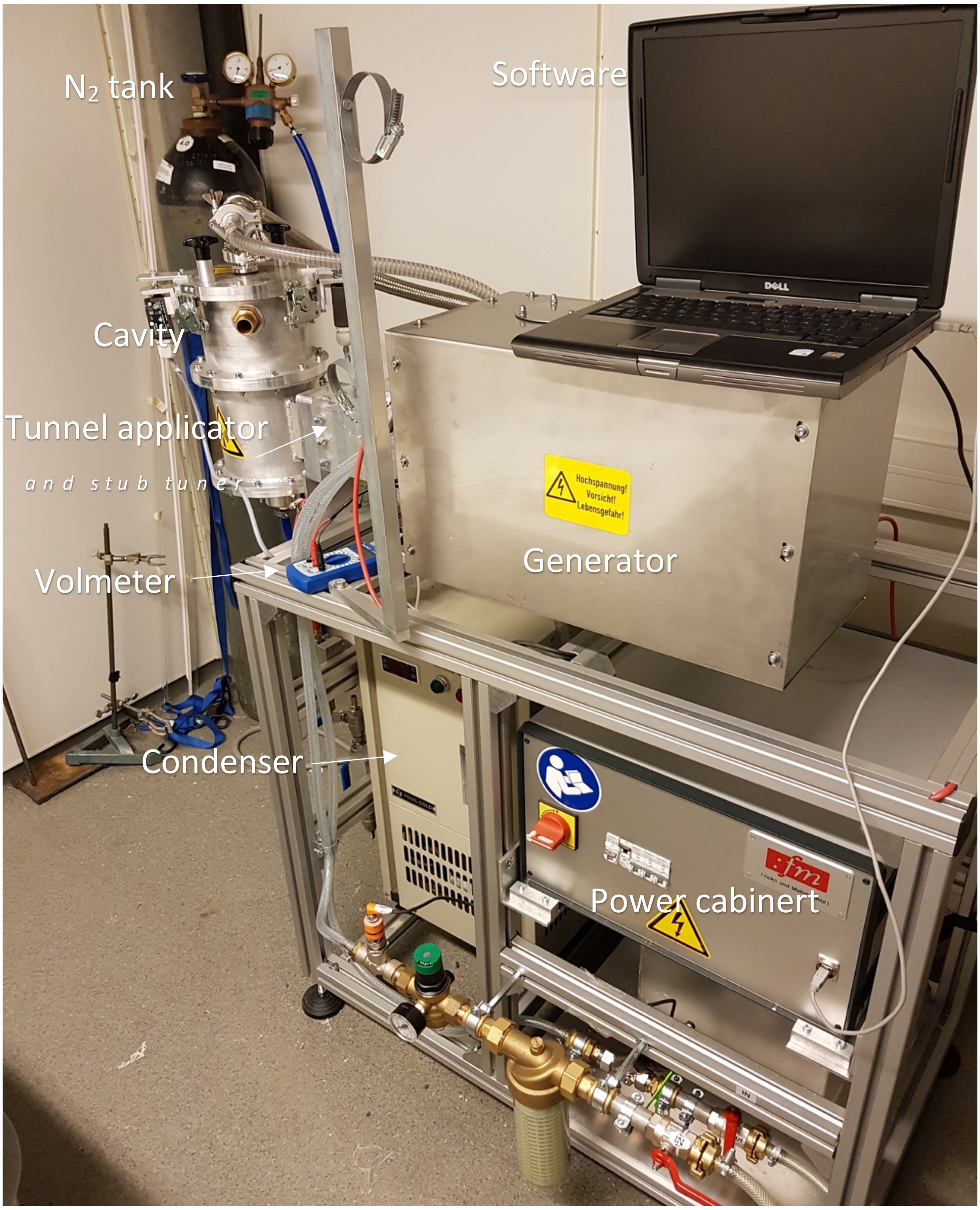



*Figure 21: Set-up in the microwave*

The power input and time for irradiation is adjusted using a software on the computer connected to the control unit/power cabinet (Figure 22). Time (seconds) and power (% of 2000 W) are entered in the table to adjust the power input. The software logs the power input during the process in the time/power diagram.

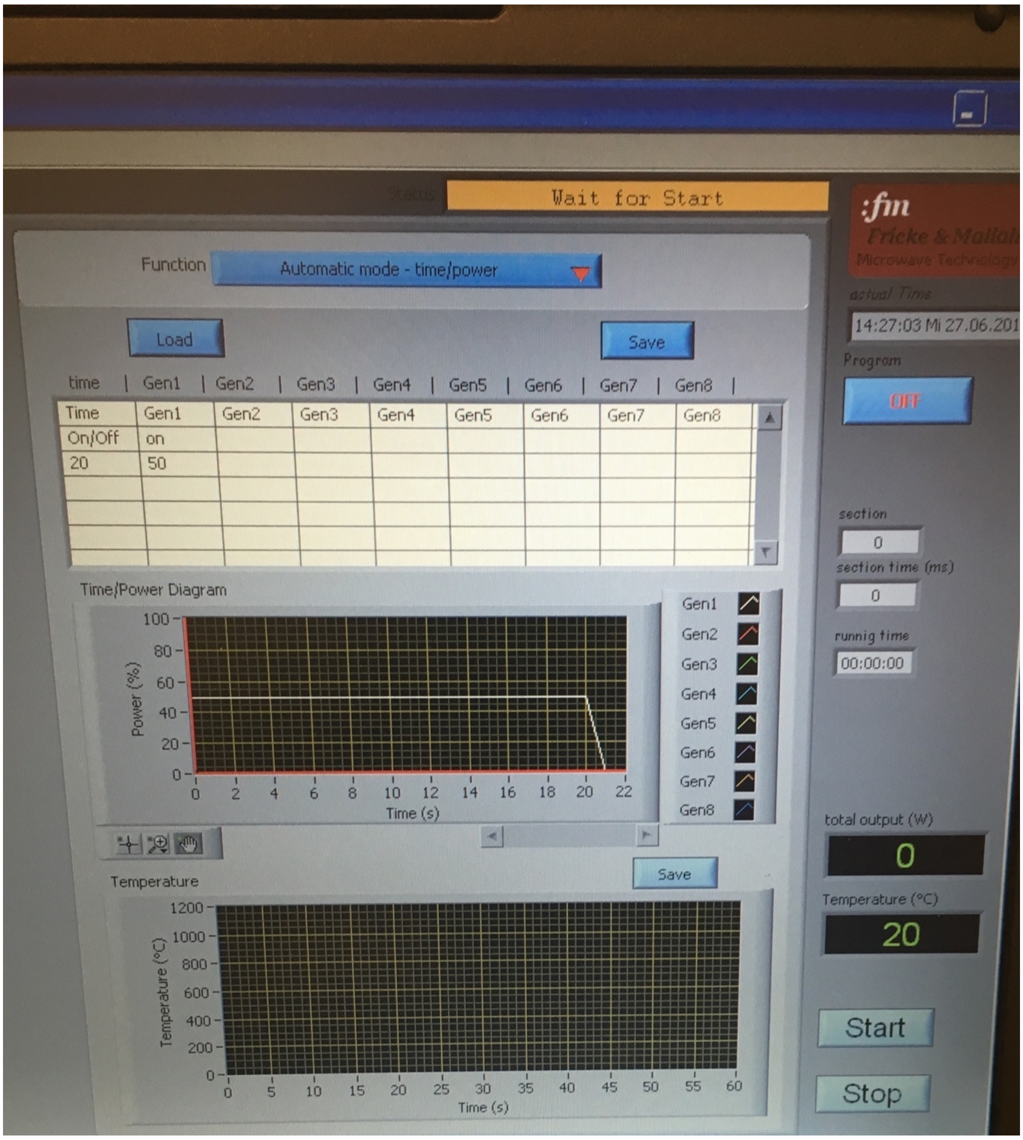



*Figure 22: Software controlling energy*

Reflection in the micro cavity was measured with a voltmeter but was not connected to the computer and had to be manually recorded. A method using voice recording was established during the experiments and showed to be the easiest way to log the reflected power. The reflected power was measured in millivolt and converted to watt using values from table 8.

The absorbed power was calculated from subtracting the reflected power from the power input.

*Table 4: The relationship between Watt and Volt*

| 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 |

A 3-pin stub tuner was used to adjust the power between the load and the generator by adjusting where the microwave strike. This makes it possible to minimize the reflected power. The stub tuner was used in the first experiments, however, it was determined to the let the tuning remain fixed on 23,3,3 throughout the rest of the experiments.

A teflon sample holder used in previously experiments for NT was intended to be used in these experiments as well (Figure 24. However, the teflon is assumed not to be able to withstand the temperatures in this microwave. Melted teflon is also not HSE friendly, so it was decided to design a new sample holder. Multiple sample containers were tested, more on this can be found in chapter 7.1. This modification of sample container is a 40 mm diameter custom made cylinder with a hole in the bottom to allow sweep gas to enter the sample. Two lids with glass cylinders were included; the upper lid is to prevent re-condensation of gas because of the metal element that connects the condenser. The lowest lid has a bent glass cylinder to trap any liquid that has re-condensed from re-entering the sample. The glass tube is bent in order to prevent the liquid that is re-condensing to fall from the upper glass tube through the lower glass tube and back to the sample. Twist was used in the bottom to lift the cuttings up to where the microwaves strikes and to absorb any excess liquid. Twist was also used the top to prevent solid entrainment and absorb any liquid that falls through the glass cylinders in case if the lids are not sealed enough. Nitrogen was supplied from the bottom.

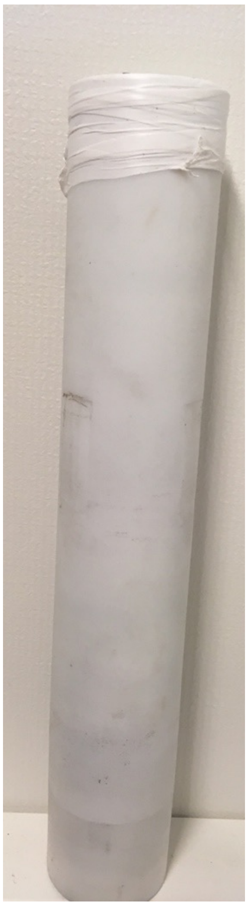


*Figure 23: Teflon sample holder*

A sketch of the whole set-up is demonstrated in figure 24.

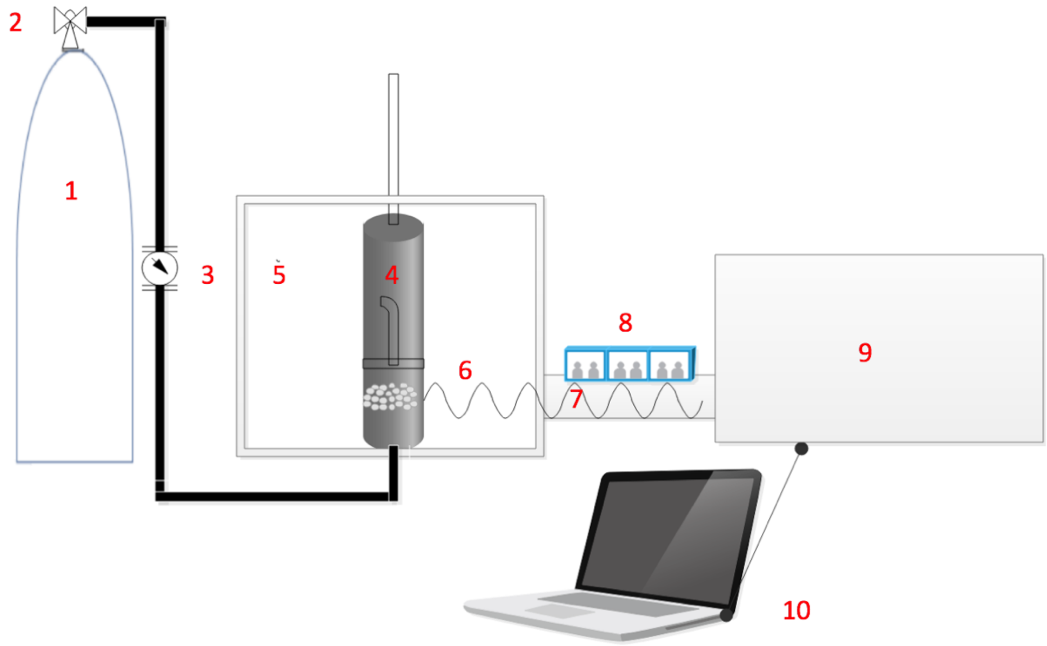



*Figure 24: Microwave set-up*

| | |
|---|---|
| 1. Nitrogen tank | 6. Microwaves |
| 2. Flow valve and display | 7. Tunnel applicator |
| 3. Flow meter | 8. Stubtuner |
| 4. Sample holder | 9. Generator |
| 5. Microwave Cavity | 10. Software |

In the microwave, energy is absorbed and reflected back. A voltmeter was used to measure the reflected power. It is therefore possible to quantify how much of the energy that is absorbed. When calculating the amount of energy absorbed, the reflected energy was subtracted from the input energy. Three methods have been evaluated to find the most accurate way to determine absorbed. Based on the results that is further described in chapter 7.2 "Troubleshooting and microwave optimization", the trapezoid method was evaluated as the best. An example for each method is found in chapter 7.2. The formula for calculating energy absorbed is seen in equation 9:

$$A = \sum \frac{y_1 + y_2}{2} \times (x_2 - x_1) \tag{9}$$

## 5.2 Preparation of Samples

Two types of cuttings were tested. All cuttings were stored in a 4 °C cooling room to preserve the optimum properties and to reduce biological growth. Before sampling, an amount of cuttings were homogenously mixed in the barrel and transferred to a plastic container. All experiments were executed at the University of Stavanger. The materials used during the experiments is shown in table 5.

*Table 5: Materials for experiments*

| Equipment | Model | Manufacturer |
|---|---|---|
| Microwave | Version 1.03en | Fricke und Mallah (FUM) GmbH |
| Voltmeter | 1070 DMM | Peak Tech |
| Pressurized $N_2$ gas | Nitrogen 4.0 | Yara Praxair |
| Glassware | - | - |
| Thread sealing tape | PTFE (Teflon) | - |
| Twist | - | Biltema |
| Susceptor | MEG | Sigma- Aldrich |
| Salt | NaCl | Sigma- Aldrich |
| Drill Cuttings | | Halliburton and Canadian cuttings |
| Centrifuge | Rotomix 46 | Hettich |
| Soxtec system | HT 1043 | Foss-Tectator |
| Retort kit | 165-14-3 | OFITE |

Cuttings were tested “raw” and centrifuged. Raw cuttings equal uncentrifuged cuttings. In centrifuges, substances are separated due to density differences. It is clear that the centrifuge alone cannot act as a pre-treatment step, but removing some liquid in the centrifuge may save some energy in the microwave. Samples of around 1,2 kg contaminated drill cuttings were centrifuged 2,5 minutes and 2500 rounds per minute (RPM). Two distinct layers with cuttings in one layer and water and oil in the second is clearly visible after the process. The excess liquid was poured out, while the rest of the sample was transferred to a larger container.

*Table 6: Equipment for centrifuging*

| Equipment | Model | Manufacturer |
|---|---|---|
| **Centrifuge** | Rotomix 46 | Hettich |
| **Plastic containers** | - | - |

Different methods were applied in the chapter 7.4-7.7, depending on whether the sample was pre-treated in microwave, if MEG and salt was added etc. The procedure for each experiment is explained in process diagrams in each sub-chapter to avoid any misunderstandings.

Retort and automated solvent extraction, using the Soxtec machine was used to determine the OOC. The Retort also measures water content. The Retort and Soxtec are described in described in the two following chapters.

## 5.2 Retort Analysis

Water and oil content can be determined gravimetrically or volumetric in the retort. The gravimetric approach was selected for this thesis, as it gives more accurate results. The analysis method is a distillation process executed according to Fann Instrument Company (2013).

A known mass of OBM is put in the retort cell and temperature was set to 480 °C. The liquids vaporize off from the cuttings and are collected in a cylinder after condensation. End-point for the test was when no liquid was seen dripping from the condenser, this was approximately after 45 minutes.

A set-back for the retort is its constraint with measuring low concentrations of oil and water. This is because it is hard to read the accurate volume of the oil and water in the cylinder due to capillary forces affecting the liquids.

The water content in the sample is calculated using equation 10. $OOC_{wet}$ is the percentage of oil on solids with oil and water and is calculated by Equation 11. $OOC_{dry}$ describes the percentage of oil on dry solids and the formula is given in equation 12.

$$Water\ content\ (\%) = \frac{M_{water}}{M_{wet}} \times 100\% \tag{10}$$

$$OOC_{wet}\ (\%) = \frac{M_{oil}}{M_{wet}} \times 100\% \tag{11}$$

$$OOC_{dry}\ (\%) = \frac{M_{oil}}{M_{wet} - M_{oil} M_{water}} \times 100\% \tag{12}$$

Where $M_{oil}$ is the mass of oil, $M_{wet}$ equals the weight of the wet cuttings and $M_{water}$ is the weight of the water.

## 5.3 Soxtec Analysis

The Soxtec method is based on the liquid-solid extraction principle (Anderson, 2004). Extraction utilizes the solubility to transfer a solute form one phase to another. The solute must have a higher solubility in the solvent than in the original phase. The cellulose thimble acts as a filter. The cellulose thimble is submerged over a boiling solvent, where the solvent condenses. The condensed solvent will return to the thimble and solubilize the oil in the sample. It usually requires many cycles for the solvent to fully solubilize the sample, however, when everything is finally solubilized, the extraction cup contains both solvent and oil.

Thimbles were weighed without and with sample. After the weighing, cotton pads where put over the cuttings to keep the particles in the sample in place during the condensation process. The extraction cups with boiling stones where weighted before 55 ml of Petroleum was added to each extraction cup and inserted in position in the Soxtec HT1043. To prevent leakage of solvent and oil, proper ceiling of the cups was ensured by twisting the cups until they were in a deadlock. The machine was switched on after the thimbles were submerged in the solvent and left to boil for 50 minutes. After the boiling, the thimbles were raised up for 40 minutes to rinse off the remaining oil and solvent from the sample. The machine was switched off after the rinsing process, and the extraction cups were cooled for about 5 minutes. To prevent rapid evaporation of oil and solvent, the cups should be lifted above the heating plate. The remaining solvent was evaporated off using a heating plate. The extraction cups were removed from the heater plate when a small amount of liquid was left. The rest will evaporate off after some time. This step is crucial in order for the oil not to evaporate with the solvent. Weighing the extraction cups after the evaporation and using Equation 13 will give the OCC.

$$OOC\ wet\ (\%) = \frac{W_2 - W_1}{W} \times 100\% \tag{13}$$

Where $W_1$= weight of extraction cup after evaporation, $W_2$ = weight of extraction cup plus extract and W= weight of cuttings

# 6. Norwegian Technology- Increase of Microwave Performance

Norwegian Technology AS (NT) has invented a technology to what could possibly be a game changer for treatment of oil contaminated drill cuttings (OCDC). The principle behind the technology is to the change material properties using chemical additives that will result in a significantly lower energy requirement, with the possibility to recycle the chemical.

The process suggested by Norwegian technology is depicted in figure 25. Cuttings containing approximately 15% oil and water are sent through a dewater system which reduces the liquid content. The dewater system in the figure is a vertical cuttings dryer from Elign Separation Systems (2018), but can be another type of mechanical dewater system. The following step is where NT´s invention will aid the process. The dewatered sample is dosed with MEG before treatment in the microwave. The susceptor will be liquefied and recycled in a condenser and ready for re-use. Cuttings will contain oil less than 0,1% after treatment.

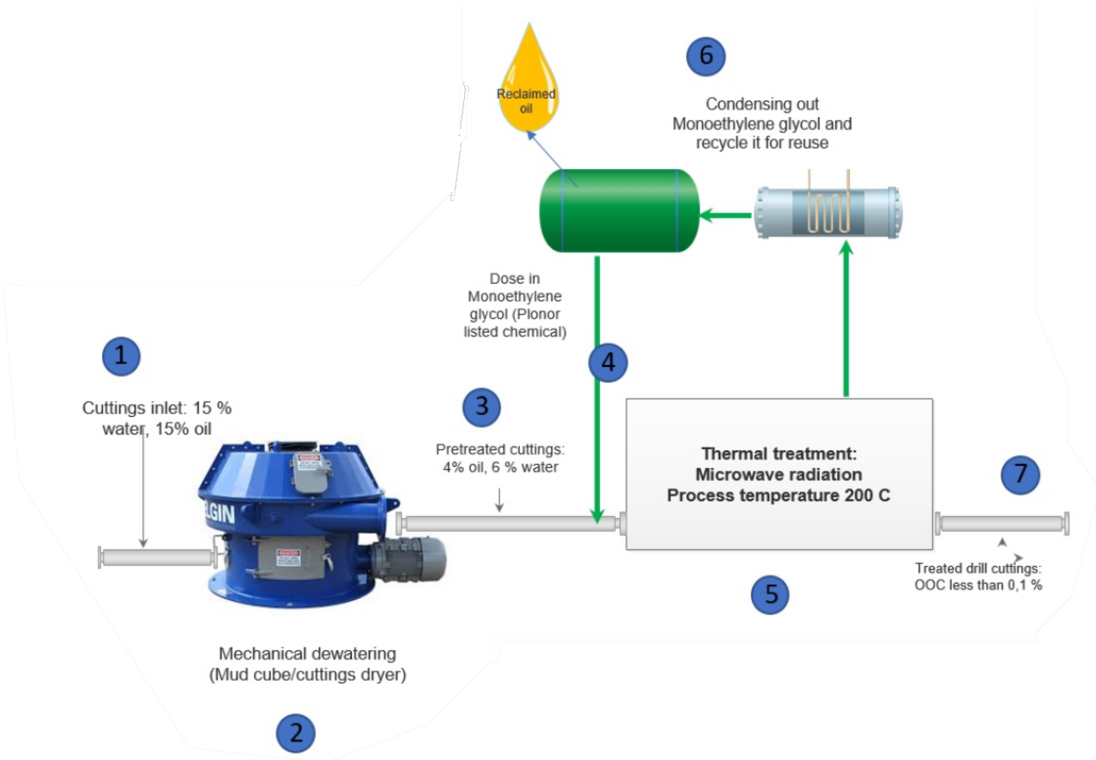



*Figure 25: Overall process for treating drill cuttings with susceptor*

The dotted “spots” in figure 26 is presented in Pereira (2012) and shows the trade-off between high separation degree and energy consumption. Three modifications are suggested by NT in order to cancel the trade-off and are estimates based on the theory of vaporization of glycol achieved in previously experiments (table 8 and 9) (Egar, 2017). The first modification is direct dosing of susceptor which could lower the energy requirement to 70 kWh/t. Increasing the power density will decrease the oil content to 1,5%, but to further increase the oil separation without susceptor requires significantly more energy. Modification 2 suggest to de-oil and dewater in microwave before susceptor dosing. This can reduce the energy consumption to 60 kWh/t. The third modification is assumed to use only 40 kWh/t, compared to 160 kWh/t without susceptor technology. Cuttings are pretreated in a cuttings dryer followed by susceptor dosing and microwave treatment.

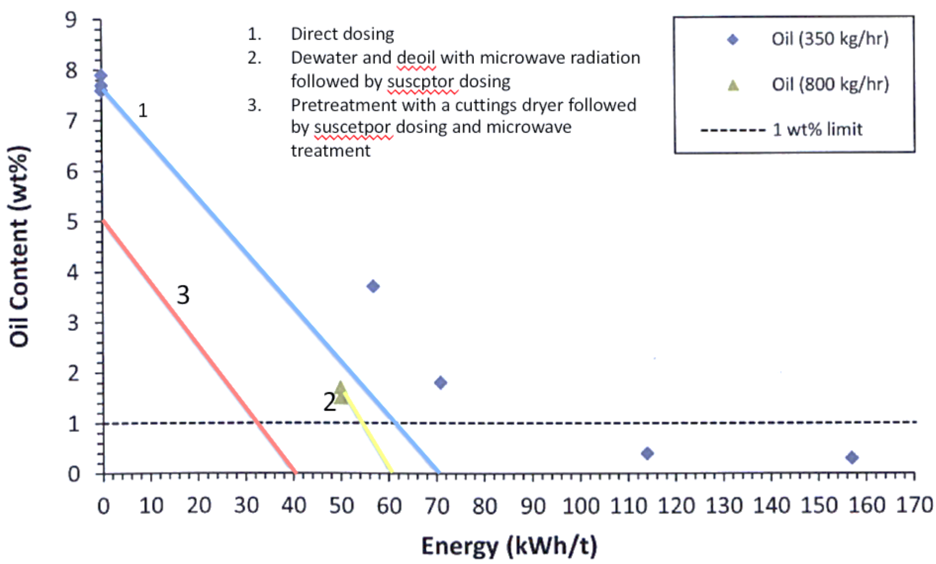



*Figure 26: Energy requirements with alternating modifications of NT susceptor technology*

A susceptor is necessary to heat transparent material and to prevent arcing in MW technology. Water is commonly used as susceptor, but the enthalpy of vaporization is very high, in other words, it requires much energy to vaporize it off. Substituting water as susceptor with a compound that has higher process temperature and lower enthalpy of vaporization would reduce the energy needed to treat OCDC to the value set by OSPAR. The oil contributes with high vapor pressure due to the high process temperature (Table 9).

Water (Table 8) and glycerol (Table 9) was distilled with two oils, Sipdrill and Clarisol. The high process temperature for the glycerol/oil distillation results in a high shared vapor pressure which leads to enhanced separation of oil and cuttings, demonstrated by the distillation ratios (Egar, 2017). Glycerol´s have high volatility, which translates to a notable energy efficiency (Figure 27). A higher distillation relationship equals less energy needed.

*Table 7: Boiling Point, Vapor Pressure and distillation for oils when distilled with water*

| 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 8: Boiling Point, Vapor Pressure and distillation for oils when distilled with glycerol*

| 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 |

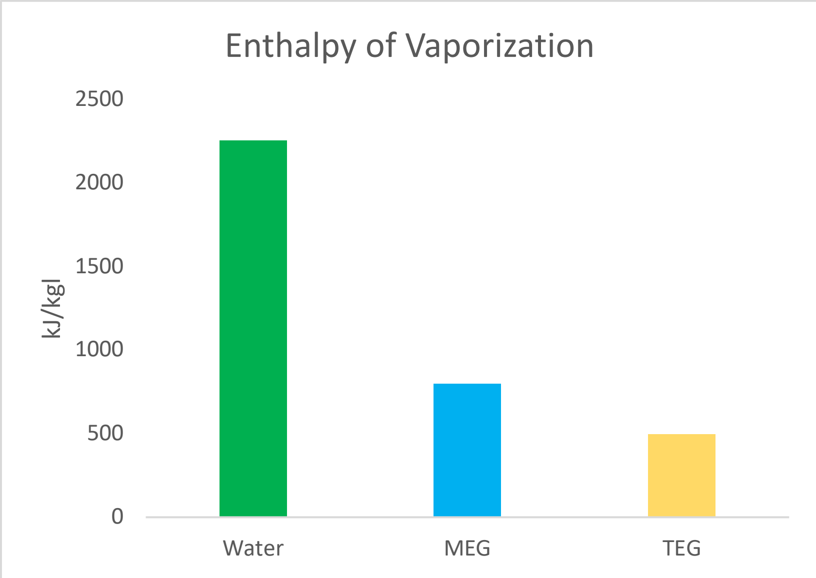



*Figure 27: Enthalpy for water, MEG and TEG (Rossi, 2016)*

Figure 27 demonstrates that approximately 5 times the energy can be saved when all the water is replaced with TEG. Applying the laws of Dalton and Raoult’s, the energy efficiency can be enhanced when combining oil and susceptor vapor pressures. The removal efficiency of oil depends on this. If only 50% of the water was replaced with TEG it could save 10 times the energy requirement (Rossi, 2016). The cost of energy savings, in addition to a small footprint, low weight and enhanced treatment capacity of this technology makes it superior compared to others. More about gas laws, vapor pressure and distillation is discussed in Rossi (2016) and Egar (2017)

## 6.1 Technology Summary

The work of investigating the principle of steam distillation and susceptors started with Rossi (2016) for NT. An evaluation of what type of dielectric heating source that was to be applied with susceptor was the first aim of his thesis. Microwave heating, with its ability to offer high power density, was the recommended technology in the works of Rossi (2016).

The main objective of Rossi´s work was to assess possible susceptors. It was concluded that glycols served best as susceptors in combination with microwave heating. MEG was pointed out as the most suitable, due to good decomposition results and its presence on OSPAR´s PLONOR (Pose Little Or No Risk) list. It was earlier thought that water was the limiting factor for oil removal. The evaluation of susceptors showed that water present had a negative effect on the treatment, because of waters ability to lower the process temperature. Rossi (2016) also saw the effect of high and low boiling point oils. When a low boiling point oil was treated, OOC levels was less than 0,05% after treatment. 0,15% OOC was achieved after treatment of a high boiling point oil.

Susceptor properties was further investigated in the works of Egar (2017), also in cooperation with NT. Both MEG and TEG showed signs of decomposition after microwave treatment, however, when drill cuttings was added to the treatment, only TEG decomposed. MEG could be recycled 10 times with cuttings before discoloration appeared. Using MEG in the experiments resulted in OOC values between 0,05-1,08 wt.%, depending on which parameters that was applied. Results also showed that increasing the dose of susceptor did not have a notable effect on OOC according to Egar. Therefore, dosing the right amount of susceptor with low water content is important in order to reduce the OOC to legal limit.

Together with NT, Haga (2017) examined the effect of low power density for dewatering of drill cuttings. This was done simulating Radio Frequencies (RF) in a microwave. With increasing retention time, sufficient amounts of water were removed, however, effects of oil separation were negligible. Haga concluded that low power density RF, in combination with high power input would be a good pre-treatment technology because of its small footprint, good efficiency in terms of energy and safety reasons.

The dielectric loss factor analyzed with the addition of salt was also investigated by Haga. Results showed that retention time and penetration depth was both reduced due to increase in the dielectric loss factor of the susceptor when salt was added. The distillation time was reduced to 30% for MEG and 25% for TEG. Oil separation was not evident when salt was added.

A high degree of oil separation was accomplished when TEG and salt was added, but problems such as decomposition reduce the incentive for applying this susceptor in treatment of drill cuttings. MEG showed little signs of decomposition and was able to treat cuttings to below 1%. It was also suggested that there should be more studies on how to remove susceptor from cuttings after treatment, as it was discovered that susceptor remained on cuttings (Haga, 2017).

Adding the sweep gas from below the cuttings bed was also investigated. This configuration lead to an increase in oil separation, but the same amount of susceptor was left in the sample container after treatment (Haga, 2017).

All of the authors mentioned above have managed to treat OOC to below 1%, however, none of them was able to measure the reflected energy. The real energy input is therefore not known.

## 6.2 Susceptors

A susceptor is a highly lossy material that is able to be rapidly heated by microwaves (Mehdizadeh, 2010). Activated carbon and silicon carbide are examples of solid susceptors used in industries for a variety of reasons (Besson & Kappe, 2013).

Pereira (2012) argued that water was the limiting factor in MW because high process temperature is hard to achieve. This is because water lowers the process temperature. Rossi (2016) suggested that the water should be replaced with an organic substance with high boiling point. The steam distillation will then occur at a higher temperature which according to Dalton´s law, causes the oil to act as the steam in the process. The oil will be the substance with highest vapor pressure, which leads to an increase in oil recovery.

The use of susceptor technology has proven in many cases to increase oil separation in microwave processing (Bhattacharya & Basak, 2016; Egar, 2017; Rossi, 2016; H. Shang et al., 2005). In addition to remove oil from cuttings, the susceptors function is to minimize the risk of breakdown voltage to occur.

The thermal energy is transferred from susceptor to material via normal heat transfer mechanisms. Depending on the type of susceptor, the energy needed to separate oil from cuttings is decreased by exploiting properties such as vaporization and boiling point. When the susceptor is vaporizing, it withdraws oil and water in the same process. A condenser can recover all the components which make it possible to reuse the susceptor. This makes susceptors highly attractive for use in microwave treatment of OCDC.

Rossi (2016) investigated possible susceptors in earlier works for NT. Boiling point, dielectric properties, melting point, decomposition temperature, recovery, price and environmental toxicity were important factors to consider when choosing the best fitted susceptor for oil separation. Both liquid and solid susceptors are available, however, liquid susceptors has proven to be most successful (Egar, 2017; Rossi, 2016).

Water alone is not able to remove high grade oils, but Hui Shang, Guo, Yang, and Zhang (2012) discovered that adding 5% activated carbon and 1 M NaCl increased the oil removal that lead to OOC values under 0,1%. Solid susceptors can be employed in different ways for example in powdered form or as granular (Besson & Kappe, 2013). Common for solid susceptors is that energy is only distributed on the surface of the cuttings, hence thermal runoff can occur. The heating mechanisms in solid susceptor is ohmic heating, not diploe polarization (referring to chapter 3). More on ohmic heating is described in Baggini and Sumper (2012). The loss of benefits from the steam distillation makes solids susceptor less desirable in the treatment of drill cuttings but can be a good option when limited amounts of liquid is present as microwave absorbers.

Liquid susceptor will be distributed all over the cuttings and in pores because of its wetting ability. The susceptor will increase the steam distillation temperature, sweep of the remaining oil fractions and have shorter re-condensation time than water. Rossi (2016) concluded that organic susceptors, in particular compounds with alcohol, ethers or ester as functional groups proved to be successful, as they will increase the process temperature and decrease the energy consumption. Based on theory, Rossi suggested 9 compounds to potentially be good susceptors: MEG, DEG, TEG, MPG, DPG, glycerol, anethole, dibutyl maleate and diethyl maleate. Only MEG, TEG, DEG, MPG and glycerol was available for experimental testing.

MEG has shown to be the best susceptor for treatment of drill cuttings according to several authors (Egar, 2017; Haga, 2017; Rossi, 2016). Rossi (2016) treated 200 g of centrifuged cuttings for 5 minutes in a 750 W microwave. The OOC after treatment was 3,8 wt%. The same experiment was repeated with the addition of 40 ml of susceptor. The result was then 0,5wt%. MEG is highly volatile and has significantly lower enthalpy than water (Figure 27). Replacing all the water with MEG would consume approximately 3 times less energy in theory, compared to using only water (table 8 and9 9).

The dielectric constants for MEG (90% wt.) is given in Table 10 and is a function of temperature and differs with percent weight in water (Figure 28). In chapter 3.1, the dielectric constant was described as the ability for a substance to store energy. A low ε' indicates that the substance has a low ability to store energy. MEG has a lower ε' than water (Table 3), and it is therefore expected to see more reflected power if the sample is dewatered. However, properties such as volatility and enthalpy of vaporization makes MEG better as dielectric for microwave. A smaller ε' is observed for increasing temperatures and decrease in % by weight in water. It is therefore also expected that more energy is reflected with increasing temperature.

*Table 9: Dielectric constant for MEG with different temperatures*

| Temperature (°C) | Dielectric constant, ε' |
|---|---|
| 100 | 24 |
| 80 | 32 |
| 60 | 36 |
| 40 | 40 |
| 20 | 45 |

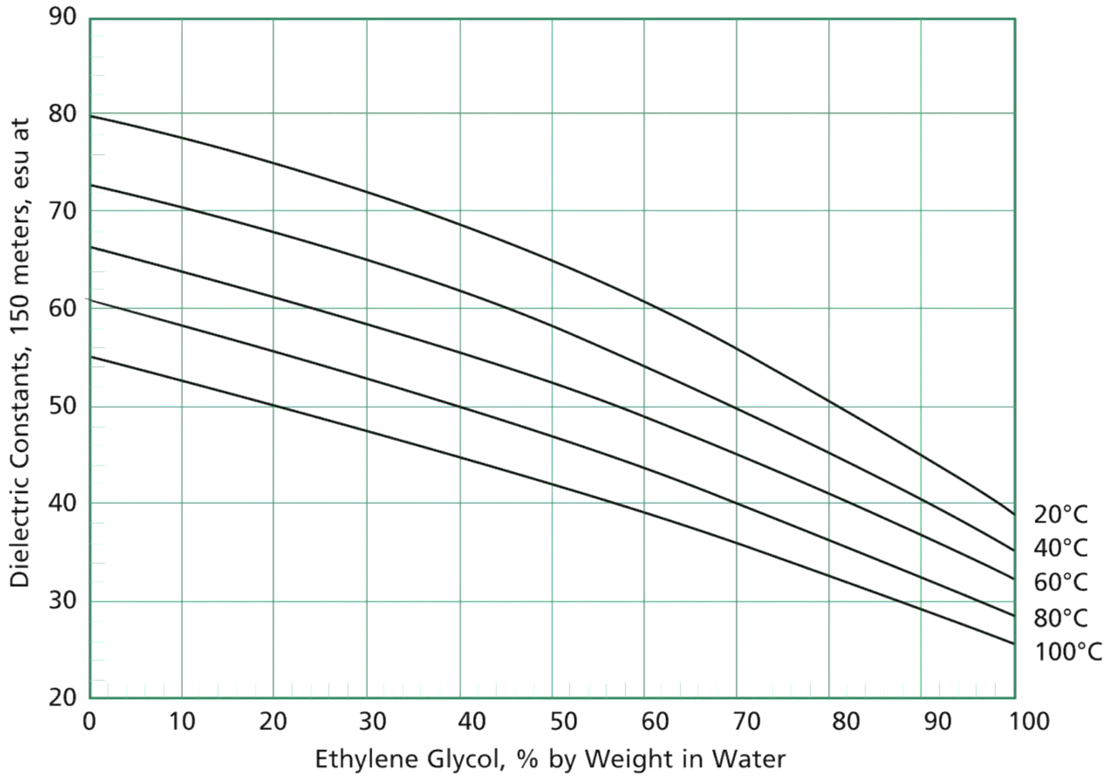



*Figure 28: Dielectric constant for MEG (MEGGlobal, 2018)*

MEG has the ability to produce higher process temperature than water, which lead to a high vapor pressure from the oil. By combining the vapor pressures of oil and susceptor, more energy could be saved (Rossi, 2016). In addition, MEG has demonstrated good ability to withstand degradation after treatment and the chemical is listed on the PLONOR list (Egar, 2017) This means that cuttings containing left overs from MEG will not damage the marine environment.

### 6.2.1 Susceptor Recovery

**Contamination of Susceptor**

MEG or other susceptors will be contaminated by water and lead to lower efficiency of susceptor. This can complicate the separation process (Egar, 2017). Egar did a theoretical evaluation of vapor pressure, because vapor pressure is an important feature in microwave steam distillation. He also studied the effects on the boiling point when mixing MEG and water. From his evaluation and studies, he created phase diagrams, which will give information about process temperature with various water/MEG ratios. This is crucial to know, because high process temperature will increase the oil separation, and water present will increase the energy demand. High process temperature may be difficult to achieve with water because water decreases the boiling point of susceptor, therefore pretreatment such as dewatering of cuttings is necessary. As mentioned, challenges with susceptor will occur when mixed with water. The ideal water concentration should be very close to 0 %, however, this is assumed to be very energy consuming as well. The phase diagrams are used to predict how much the cuttings must be dewatered. Dewatering of samples is also positive because it will also leave the sample more porous to absorb susceptor.

In the distillation process, susceptor will vaporize off with the water because they are miscible, thereby less susceptor will be available for dielectric heating and steam distillation removal of oil in the cuttings. This will result in a decrease in oil separation and recovery of MEG for reuse. The fact that water and MEG are miscible makes it also more challenging to separate later.

### 6.2.2 Available technologies for water and glycol separation

Two alternatives are possible in order to be able to reuse MEG, the first is to avoid contamination by water, the second possibility is to include a new separation technology for MEG and water.

1. **Avoiding glycol contamination**

The best option would be to avoid contamination. Pretreating the cuttings in a microwave can be used to do this. The chance of contamination to occur is assumed to decrease with decreasing water levels. The degree of contamination is also assumed to be smaller with decreasing water levels. Water levels under 0,5% was achieved in Haga (2017) with low power density. It is uncertain if the water will vaporize with the MEG when the water concentration is that low, because water condenses quicker than glycol.

An example of a set up used to avoid contamination is shown in figure 29. The cuttings are first sent through a centrifuge/cuttings dryer and then in a microwave to remove most of the water. After pretreatment, the susceptor is dosed before final treatment in microwave. (Haga,

2017) suggested to use the same technology for pretreatment and main treatment. This could save costs and operability problems.

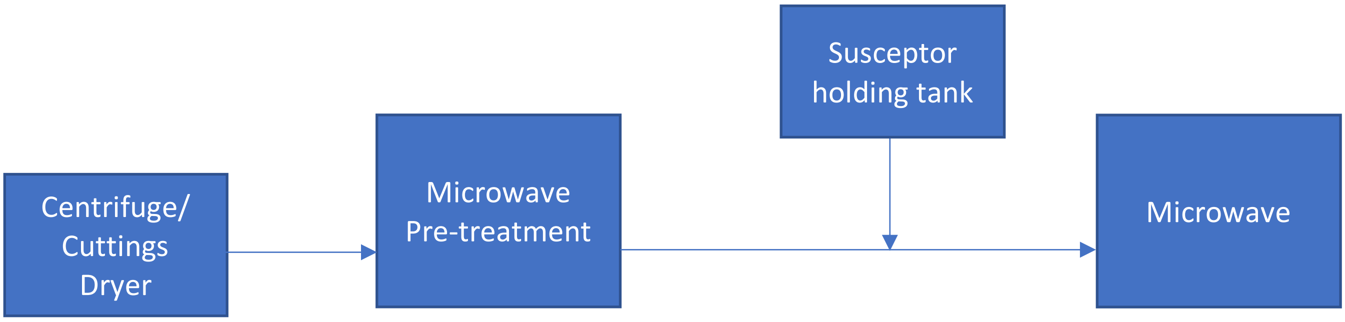



*Figure 29: Set-up with microwave as pretreatment technology*

### 2. Separation of contaminated glycol

If water is present, the goal is to be able to separate MEG and water after treatment so that the susceptor can be reused. Transportation of MEG to offshore platforms is then minimized. The susceptor must contain as little water as possible for it to able to be used over again. Footprint, efficiency and cost are among the factors that must be considered when choosing the right separation system. Some solutions are discussed below, but further research could reveal other technologies.

#### Flash distillation

Flash distillation is a relatively simple process that separates liquids. The feed stream is heated and pressurized before it is sent through a nozzle into a flash drum. The large pressure drop in the drum will result in some of the liquid to vaporize. The result is a vapor phase and a liquid phase. The vapor will be the most volatile, water, while the less volatile, MEG, will be in liquid phase. The water vapor escapes out from the top of the vessel and the liquid MEG will be withdrawn from the bottom. The dimensions of the drum will depend on flowrate. No costs was found for the flash drum. A drawing of a flash drum is shown below (Figure 30).

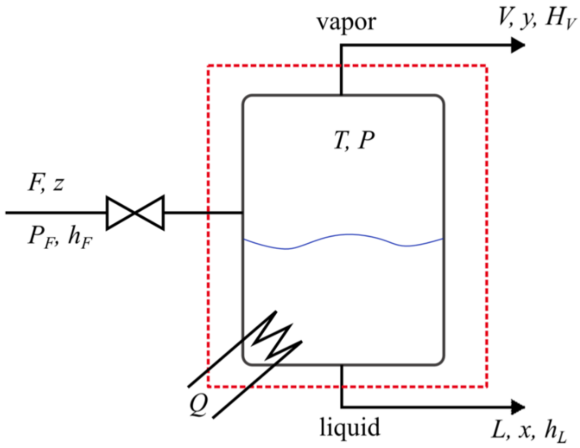



*Figure 30: Sketch of a flash distillation unit (Iggland & Mazzotti, 2015)*

Membrane

Membrane technology has also been suggested to purify MEG (Tomaszewska, Orecki, & Karakulski, 2005). Two spiral and one tubular membranes was tested with model solutions ranging from 1-20 wt.% glycol. Nano filtration is based on sieving or charge rejection. Uncharged molecules, such as glycol, are rejected due to sieving mechanism. Charged particles are rejected due to electrostatic interaction between solute and the membrane surface. The result showed that one of the spiral membranes rejected up to 70% of the glycol. However, the sample contained only 5 wt.% glycol and increasing the glycol concentration lead to concentration polarization. The pore size in Nano filtration may therefore be too large for glycol. Pervaporation is another membrane process used to dehydrate glycols. The fact that MEG is less volatile than water will affect the permeability behavior (Won Yim & Kong, 2013).

Distillation column

A distillation column is also a simple method to re-concentrate MEG. Lean MEG is formed by vaporizing the excess water off. The basis for distillation is explained above. This process is often used in the O&G industry when the salt concentration and chance of salt precipitation is low (Zaboon, Soames, Ghodkay, Gubner, & Barifcani, 2017). The distillation has an easy set-up and a small footprint (figure 31). This process can be either continuous or batch. Inserting a fractional distillation column increase the pureness of the substances, but it is energy intensive, and the process might have to be repeated to make it 100% pure. This can maybe decompose the susceptor.

*Figure 31: Distillation column used to separate MEG and water*

**Decomposition of Susceptor**

Decomposition of susceptors can occur in two cases (Kappe & Doris, 2005):

1. Thermal run off due to inhomogeneous heating
2. Temperatures leading to rapid chemical reactions

Decomposition of susceptors can, when subjected to high temperatures lead to compounds that are toxic and that can possibly give environmental impacts. The decomposed susceptor may also impact the oil separation and equipment, therefore it is undesirable for this to happen. The susceptor can also go back to its original building blocks after decomposition.

Egar (2017) examined decomposition of MEG, both with and without cuttings. Color change was used as an indication of susceptor degradation. Distilling MEG (85-95%) without cuttings showed signs of coloration when exposed to microwave radiation (Egar, 2017). A test with a mixture of MEG and TEG was also tried out, but with unsuccessful results to achieve no color change. The same experiment was performed with drill cuttings. 125 mL of MEG was added to 500 g of dewatered drill cuttings. The sample was distilled multiple times to determine the amount of cycles before decomposition occurred.  MEG was distilled 10 times before sign of decomposition occurred. The initial volume of MEG was reduced after each cycle due to evaporation when susceptor was added to hot cuttings in these experiments.

Glycolic acid is the main component that is created when MEG is degraded (The Dow Chemical Company, 2017). Oxalic and Formic acid are also formed. Generally, the degradation increases with increasing temperature, and the presence of metals or UV light can act as catalysts. Therefore, if the drill cuttings contain metals it can speed up the decomposition of MEG.

# PART 3- LABORATORY TESTING

# 7. Treating Drill Cuttings with Single-mode Microwave Unit

With the new single-mode microwave described in chapter 5.1, it is now possible to quantify the real energy input due to measurement of the reflected energy. This gives data that is crucial for scale-up. The power input is also automatically logged, and the microwaves can be adjusted. All these applications give data in regard to energy consumption. Use of single-mode unit allow high power densities which should in theory increase the oil separation and save energy.


*Figure 32: Single-mode cavity*

The treatment parameters in focus during the laboratory study were to

- Treatment unit trouble shooting and optimization
- Assess $N_2$ strip temperature
- Investigate treatment of drill cuttings with new characteristics
- Changing cuttings characteristics
- Assess the effect of high and low power density (with/without susceptor)
- Susceptor inmixing
- Susceptor dosing quantity

At the end of each subchapter there will be a discussion of the findings in addition to a discussion at the end to summarize.

## 7.1 Troubleshooting and microwave optimization

Many challenges occurred during testing. This chapter aims to optimize the microwave based on the following challenges.

**Re-condensation**

The teflon container described in chapter 5 was not used due to the risk of melting. A sample holder made of Pyrex glass was used instead.

After treatment when opening the cavity cover, re-condensation of vapor was detected at the glassware (Figure 33). The metal around the cavity caused the gas to re-condensate back into the sample container. It was clearly visible that the upper area of the treated sample was wet (Figure 34).

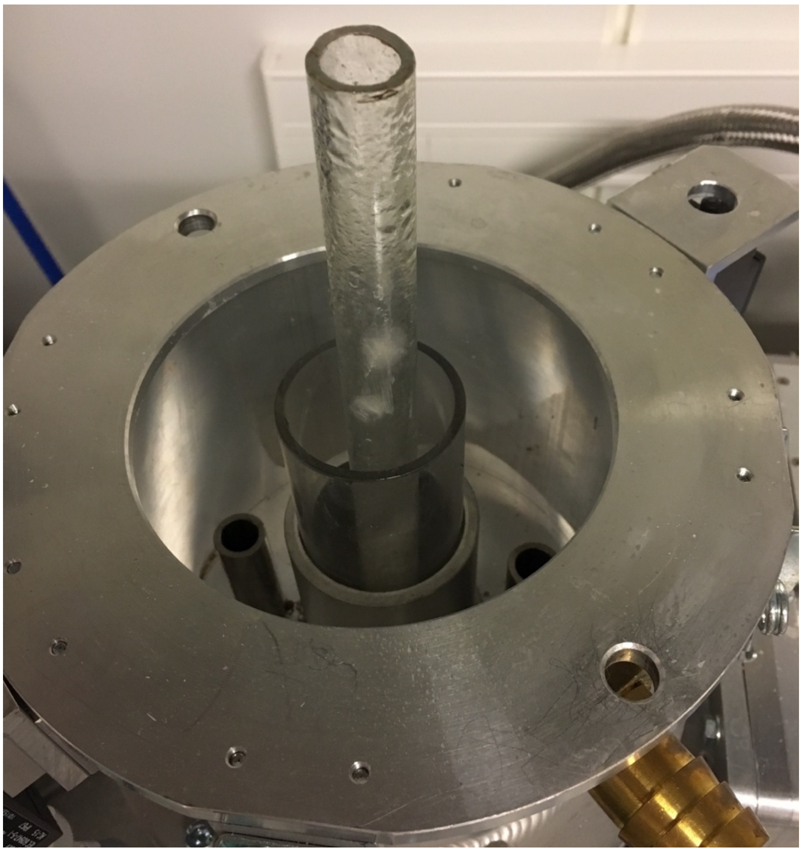


*Figure 33: Re-condensation of vapor dripping back to the sample*

*Figure 34: Top of sample wet after re-condensation*

To overcome the negative impacts and avoid re-condensation, a warm air gun was used. This modification was not sufficient, so different designs of the sample containers were tested. The first attempt involved cutting a glass tube long enough to avoid the vapor to come in contact with the metal. Vacuum was used to extract the vapor. This turned out not to be sufficient as well, as the top of the sample was still wet.

**Cuttings entrainment**

Problems with solid entrainment did also appear during testing. Solid entrainment occurs as a result of the sweep gas pushes the parts of, or the whole sample up in the sample container because it cannot find a way to entrain. The sample is then not exposed to microwave treatment when it is lifted up (Figure 35). Solid entrainment can also clog the glass tube.

*Figure 35: A part of the sample pushed up due to solid entrainment*

**Nitrogen Leakage**

Liquid dripping out from the bottom and a beep sound was detected during treatment. It was established that this was due to leakage of nitrogen from the sample holder and nitrogen inlet. After multiple testing, a teflon cap with a gap in the middle was drilled from a teflon plate.

The cap was added at the bottom of the sample holder with a thermally resistant adhesive. Teflon tape was used to ensure sealing around the bottom of the sample container.

Different designs were tested, but based on testing and calibration, the conclusion was that the last design cancelled all the effect mentioned above. It consists of a pyrex glass cylinder, with a cap in the bottom with teflon tape to seal the connection. A teflon lid and a bent glass tube was inserted in the middle. This prevents solid entrainment and liquid falling back to the sample due to re-condensation. The glass tube in the top was used to minimize re-condensation. Twist was used in the bottom to lift the cuttings up to where the microwaves strikes, and in the top, to prevent clogging of the glass tube if solid entrainment occurred. The twist also absorbs liquid. The final design is depicted in figure 36.

*Figure 36: Final design of sample container*

**Stub tuning to optimize reflective**

In the first experiments, 30 seconds was used to adjust the waves with 3 stub tuning pins. In the beginning of the treatment much of the energy was absorbed, while low reflection was measured. During the treatment when less and less energy absorbed, more energy was reflected. The stub-tuning was then adjusted, but in later experiments, stub-tuner values with little reflection was determined and stayed fixed for the rest of the experiments. The fixed values were 23, 3 and 3 (Figure 37).

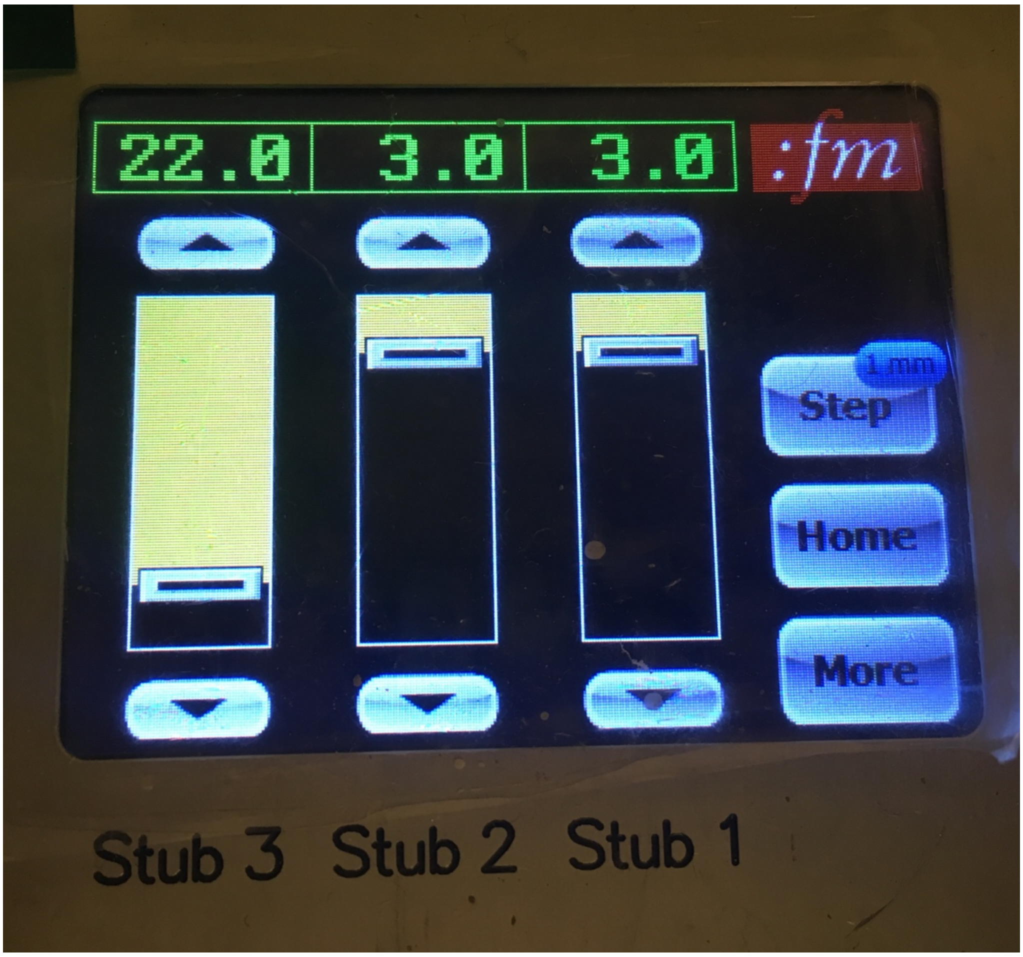



*Figure 37: Fixed stub tuning values*

**Determining Energy Input**

As described in material and methods, three methods were evaluated for determining energy absorbed. All methods are based on finding the integral. Results from sample 24 is used as an example in the calculations.

1. **Integrating using function of the graph.**

Plotting the values of time against energy absorbed will give a graph shown in figure 38. The function of the graph is determined by choosing a trend-line that fits the curve best. The function is displayed in the graph area.

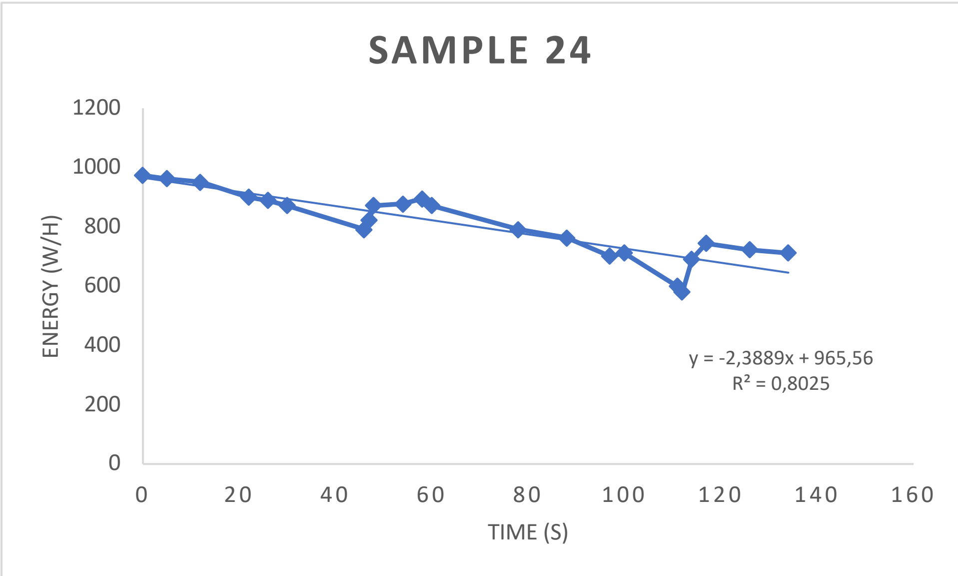



*Figure 38: Determining the function in Excel*

The area under the graph is determined by calculating the definite integral between two point, $t_{start}$ (0 s) and $t_{finish}$ (134).

$$y = -2{,}3889x + 956{,}56$$

$$A_1 = \int_0^{134} -2{,}3889x + 956{,}56 = [-1{,}995x^2 + 965{,}56x]_0^{134} = 108017{,}4$$

The correlation coefficient, $R^2$ explains how well the equation describes the data, in this case 80% fit. However, the curve is above and under the trendline, and it can be assumed that these areas will cancel each other. This cannot be assumed for all cases, therefore there are uncertainties related to this method.

## 2. Counting blocks

The second method is simple but time consuming. The method involves countingthe blocks under the curve and calculating the area using equation 14.

$$A_2 = length \times width \ \times \ \# \, of \ blocks \qquad (14)$$

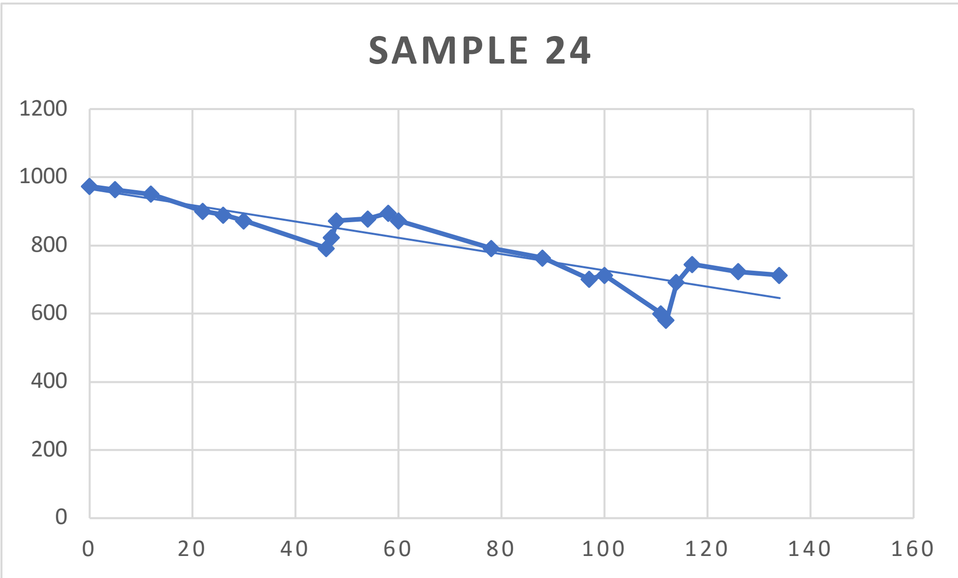



*Figure 39: Graph area divided into blocks*

The number of blocks under curve for sample 24 is roughly 26, the area will therefore be

$$A = 200 \, W \times 20s \ \times \ 26 = 104000$$

The uncertainty is high because it is difficult to find the exact number of blocks as the function is not a straight line.

## 3. Trapezoid method

The trapezoid method is much easier and does not depend on a function. It is based on dividing the area underneath the curve into rectangles, and then calculating the area for each one and lastly take the sum of the total. The more segments, the more accurate. The following formula was used:

$$A_3 = \sum \frac{y_1 + y_2}{2} \times (x_2 - x_1) \tag{9}$$

*Table 10: Data for sample 24*

| Time (s), x | Energy Absorbed per time interval (W/h), y | Area per time interval |
|---|---|---|
| 0 | 973,3 | 4840 |
| 5 | 963 | 6697 |
| 12 | 950,6 | 9253 |
| 22 | 900 | 3578 |
| 26 | 889 | 3522 |
| 30 | 872 | 13304 |
| 46 | 791 | 807 |
| 47 | 823 | 847 |
| 48 | 872 | 5248 |
| 54 | 877,6 | 3544 |
| 58 | 894,4 | 1766 |
| 60 | 872 | 14967 |
| 78 | 791 | 7770 |
| 88 | 763 | 6588 |
| 97 | 701 | 2119 |
| 100 | 712 | 7216 |
| 111 | 600 | 590 |
| 112 | 580 | 1271 |
| 114 | 691 | 2152 |
| 117 | 744 | 6601 |
| 126 | 723 | 5740 |
| 134 | 712 | - |

$$A_3 = \sum \frac{y_1 + y_2}{2} \times (x_2 - x_1) = 108424{,}5$$

*Table 11: Results the three methods given in Whs and W/kg*

| | Whs | W/kg |
|---|---|---|
| **$A_1$** | 1080017,4 | 137,0 |
| **$A_2$** | 104000 | 131,9 |
| **$A_3$** | 108424,5 | 137,5 |

The values in W/kg is quite similar for $A_1$ and $A_3$, but method 3 is chosen as it is a quick and easy method, in addition to less uncertainties linked to it.

## 7.2 $N_2$ strip temperature

H. Shang et al. (2005) demonstrated that supplying the sample with ambient temperature $N_2$ led to a lower treatment efficiency. This was due to the cooling effect of cold nitrogen that resulted in re-condensation of oil and water vapors.

In Egar (2017) work in determining boiling point of pure MEG and mixtures of MEG and water, the values obtained were lower than the theoretical values. Egar believed that this was either due to the fact that cold nitrogen was used to create an inert atmosphere, or a reduction of microwave power at the onset of boiling, or a combination of both. It was later confirmed that cold nitrogen did impact the results.

It is therefore suggested to use warm nitrogen in order to eliminate the cooling effect and hence increase the treatment efficiency. However, due to the modification of the sample holder, it is expected that the oil, water and MEG that is re-condensed will be trapped between the caps in the sample holder (Chapter 5). The following experiment aims to determine the effect of cold nitrogen.

**Experimental set-up and method**

Test 36 was conducted with the same set-up as described in chapter 5. For test 37, the gas was heated up to 200 °C in an inline heater (Figure 40). The temperature was measured in a temperature sensor which was placed right next to the inline heater. A short glass tube was used to connect the inline heater and the cavity. North Sea cuttings was used in this experiment. However, the cuttings had not been stored in a cooling room. It was therefore very dry, so 10% water was added. All parameters were fixed, except for nitrogen gas temperature (Table 11).

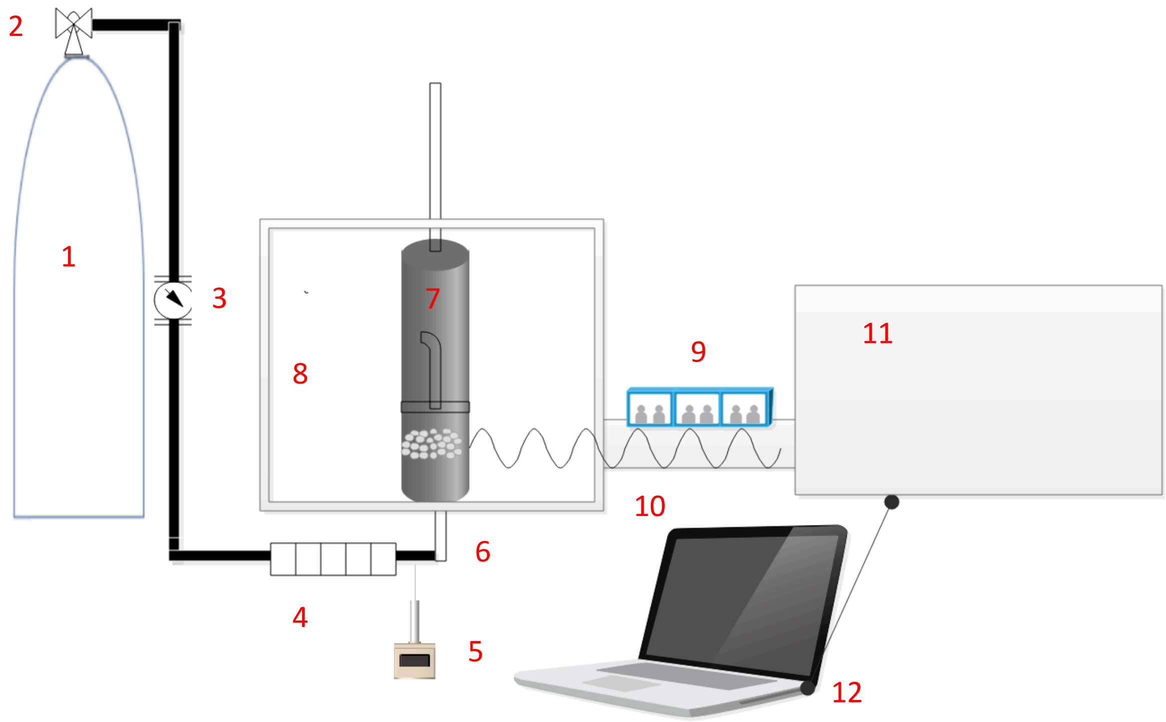



*Figure 40: Set-up for test 37 with warm nitrogen*

| | |
|---|---|
| 1. Nitrogen tank | 7. Sample holder |
| 2. Flow valve and display | 8. Microwave Cavity |
| 3. Flow meter | 9. Stub-tuner |
| 4. Inline heater | 10. Tunnel applicator and microwaves |
| 5. Temperature sensor | 11. Generator |
| 6. Glass tube | 12. Software |

The flowchart below explains the procedure (Figure 41)

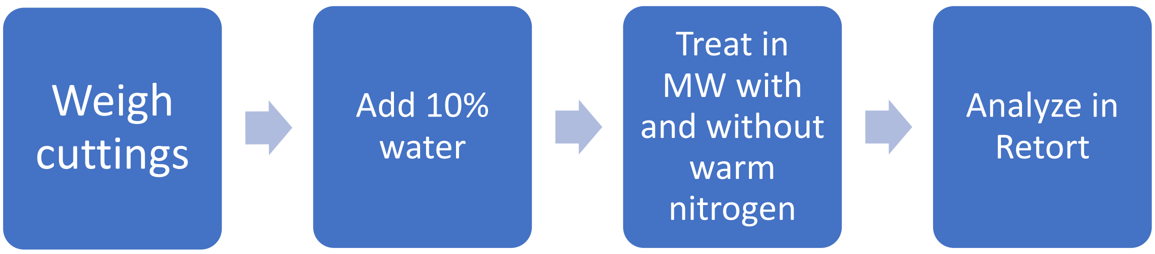



*Figure 41: Procedure for test 36 and 37*

*Table 12: Initial treatment parameters for test 36 and 37*

| | **Test 36** | **Test 37** |
|---|---|---|
| Nitrogen temperature (°C) | 25 | 200 |
| Flow rate of nitrogen (L/min) | 8 | 8 |
| Amount (g) | 185 | 185 |
| OOC dry (%) | 9,13 | 9,13 |
| OOC wet (%) | 9,8 | 9,8 |
| Water (%) | 13,2 | 13,2 |
| Exposure time (s) | 26 | 26 |
| Energy input (W/h) | 2000 | 2000 |
| Power Density (W/h*kg) | 10810 | 10810 |
| Input Power (W/kg) | 78 | 78 |

Results

The results for test 36 and 37 is seen in table 13. An insignificant difference was seen in the oil separation. More water was removed with warm nitrogen. Test 36 achieved 71,6% and 75,8% in oil separation with $OOC_{dry}$ and $OOC_{wet}$ values, respectively. Test 37 achieved 69,0% and 73,3. The structure after treatment is visualized in figure 42.

*Table 13: Results for test 36 and 37*

| | **Initial** | **Test 36** | **Sep. Degree (%)** | **Test 37** | **Sep. Degree (%)** |
|---|---|---|---|---|---|
| Nitrogen temperature (°C) | - | 25 | - | 200 | - |
| Time (s) | - | 26 | - | 26 | - |
| OOCdry (%) | 9,13 | 2,59 | 71,6 | 2,83 | 69,0 |
| OOCwet (%) | 9,8 | 2,37 | 75,8 | 2,62 | 73,3 |
| Water (%) | 13,2 | 5,94 | 54,7 | 4,71 | 64,3 |

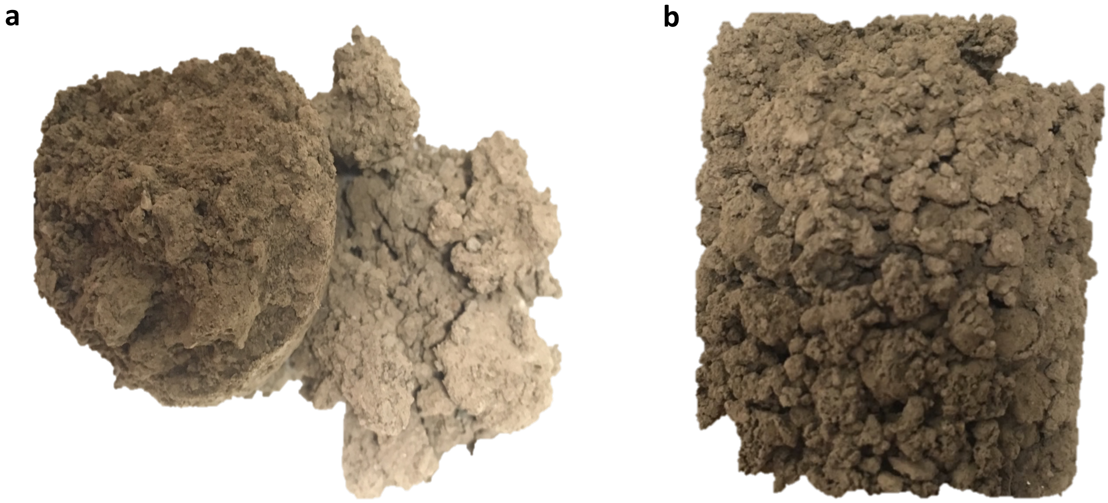



*Figure 42: Structure after treatment with a) cold nitrogen and b) warm nitrogen*

<u>Discussion:</u>
The tests had a similar consistency. Some clay was on both of the containers, this is likely to be due to the samples were only exposed to 78 W/kg. OOC is observed to be quite similar, it is therefore proved that with the modification of sample container, the effect of cold nitrogen is insignificant. Due to lack of cuttings material, no further tests were conducted on this type. It will require further study to clarify if it has a minor effect.

There are some errors in this experiment. Firstly, the fact that the cuttings had been stored in room temperature may have had impact on the results. Secondly, it is likely that there is heat loss from the inline heater to the cavity. The sensor was right next to the inline heater, so the temperature in the glass tube before the cavity may be different. However, col nitrogen was conducted for the rest of the experiments due to what the results implied and the simplicity in set-up.

## 7.3 Investigate treatment of two different drill cutting types

Halliburton cuttings and cuttings received from a field in Canada will now be presented. A smaller sample holder was used in the experiments with the Halliburton, therefore the power density is higher.

**Halliburton Cuttings**

Cuttings from the North Sea were used in the first experiments, however, testing was stopped on this type of cuttings due to lack of cuttings material for microwave optimization. Therefore, energy optimization was not the aim with the experiments conducted with this type of cuttings. Oil separation was the focus. The North Sea cuttings have been used in previously experiments by Egar (2017), Haga (2017) and Rossi (2016). Because the values have been stable during the experiments, it is assumed that the values are the same in these experiments. Values for centrifuged Halliburton cuttings is depicted in table 15.

*Table 14: OOCdry, OOCwet and water content for centrifuged North Sea Cuttings (Rossi, 2016)*

| Test # | OOCdry % | OOCwet % | Water % |
|---|---|---|---|
| **1** | 9,54 | 7,84 | 16,5 |
| **2** | 9,74 | 7,98 | 16,3 |
| **3** | 9,87 | 8,11 | 16,3 |
| **4** | 9,63 | 7,90 | 16,5 |
| **5** | 9,84 | 8,07 | 16,4 |
| **6** | 9,60 | 7,90 | 15,6 |
| **7** | 9,89 | 8,17 | 15,9 |
| **8** | 9,96 | 8,25 | 15,8 |
| **Average** | **9,76** | **8,03** | **16,2** |

Two experiments were conducted with the Halliburton cuttings. The first was to see the effect of single-mode applicator with and without MEG. The second experiment was conducted to see the effect of high and low power density in a single-mode applicator.

**Experiment 1: The effect of single-mode applicator with and without MEG**

Test 6 was conducted to see the effect of single-mode applicator and susceptor. The sample was first irradiated. After irradiation, approximately 15 g was sent for analyzing in the Soxtec. The rest of the sample was dosed with MEG and irradiated again. The procedure is seen in figure 43. The initial treatment parameters for test 6 is seen in table 16.

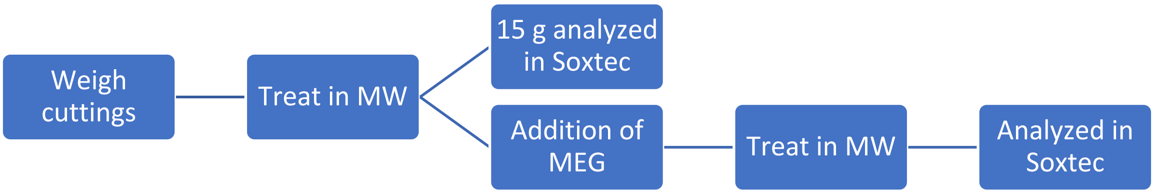



*Figure 43: Procedure for test 6 with and without MEG*

*Table 15: Initial treatment parameters for Experiment of test 6*

| **Test 6** | **Without MEG** | **With MEG** |
|---|---|---|
| Weight (g) | 134 | 118 |
| MEG (%) | 0 | 33 |
| Exposure time (s)* | 530 | 530 (160) |
| Energy input (W/h)* | 300 | 300 (300) |
| Power density (W/h*kg)* | 1103 | 1103(564) |
| Input power (W/kg)* | 272 | 272 (171) |

* values in parentheses are values after first round of irradiation

Results

Table 17 and figure 44 shows the results after treating sample 6 with and without MEG. Without MEG, 272 W/kg added to the test. This reduced the $OOC_{wet}$ from 8,03 % to 2,24%. Adding 33% of MEG and exposing the test for 171 W/kg reduced the $OOC_{wet}$ to 0,16%, which equals a separation degree of 98%.

*Table 16: Results for Test 6*

| OOC wet% Soxtec | Initial | Without MEG | With MEG |
|---|---|---|---|
| 1 | 8,03 | 2,14 | 0,06 |
| 2 | 8,03 | 2,32 | 0,09 |
| 3 | 8,03 | 2,47 | 0,33 |
| Average | 8,03 | 2,24 | 0,16 |
| Standard deviation | ∓ 0,2 | ∓ 0,09 | ∓ 0,15 |
| Separation Degree (%) | - | 72,1 | 98,0 |

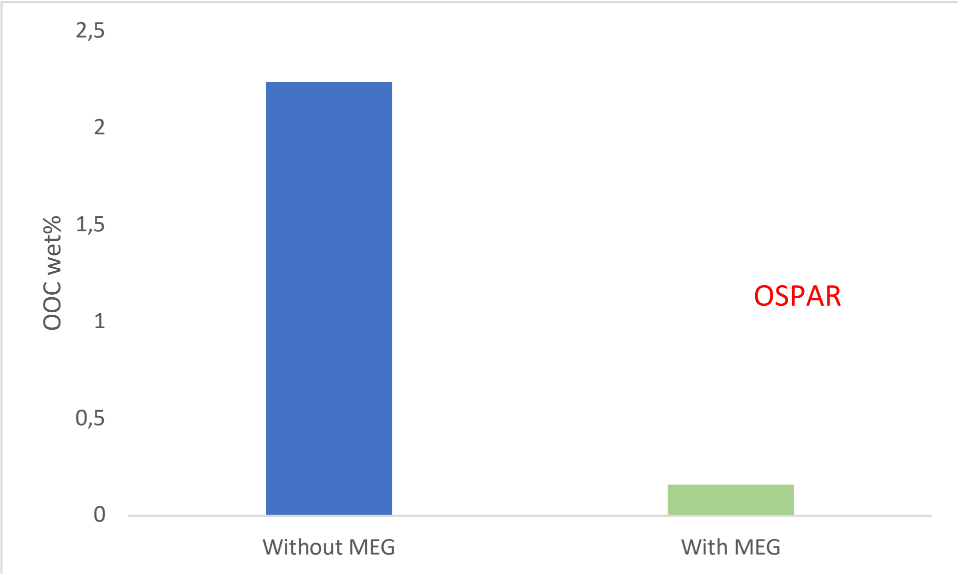



*Figure 44: OOC obtained for test 6*

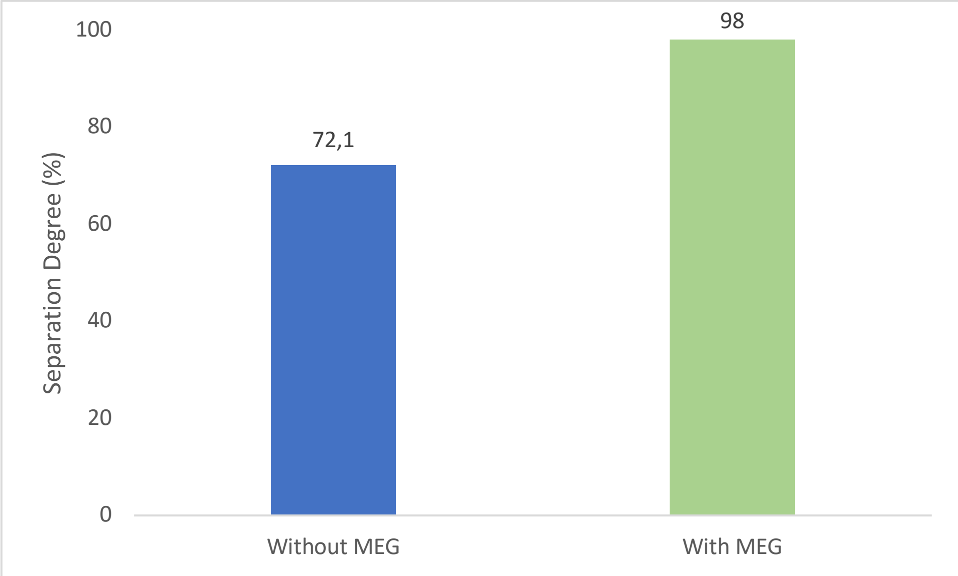



*Figure 45: Separation degree obtained for test 6*

**Experiment 2: The effect of high power density**

The power density was increased in test 12 to see the effect of high and low power density. No susceptor was added. The result was compared to test 6 that had been exposed to low power density. Procedure is depicted in figure 46 and initial treatment parameters is showed in table 18.


*Figure 46: Procedure for test 12*

*Table 17: Initial treatment parameters for test 6 and 12*

| | Test 6 | Test 12 |
|---|---|---|
| Power Density | Low | High |
| Weight (g) | 134 | 135 |
| MEG (%) | 0 | 0 |
| Exposure time (s) | 530 | 130 |
| Energy input (W/h) | 300 | 1600 |
| Power density (W/h*kg) | 1103 | 11851 |
| Input power (W/kg) | 272 | 245 |

<u>Results</u>

Treatment with high power density resulted in 90.5% separation degree in test 12, compared to 72,1% with low power density in test 6. Approximately the same energy was added to the tests (table 18). The results of OOC and separation degree are depicted in figure 47 and 48 respectively.

*Table 18: Results for test 6 and 12*

| **OOC wet% Soxtec, test #** | **Initial** | **Test 6** | **Test 12** |
|---|---|---|---|
| 1 | 8,03 | 2,14 | 0,77 |
| 2 | 8,03 | 2,32 | 0,78 |
| 3 | 8,03 | 2,47 | 0,76 |
| **Average** | **8,03** | **2,24** | **0,76** |
| Standard deviation | ∓ 0,2 | ∓0,09 | ∓0,006 |
| Separation Degree (%) | - | 72,1 | 90,5 |
| Power Density | - | 1103 | 11851 |
| Input Power | - | 272 | 245 |

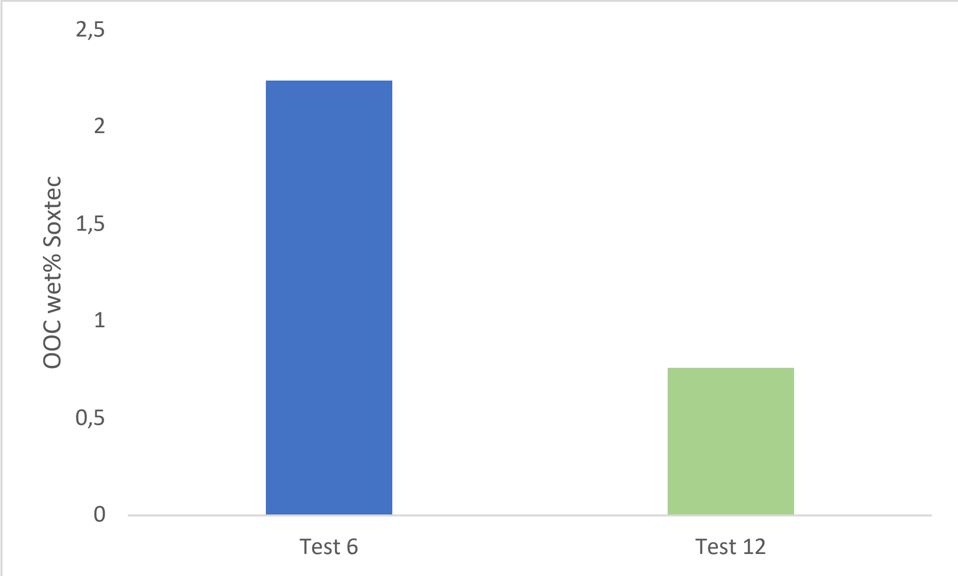



*Figure 47: OOC for test 6 and 12, alternating power density*

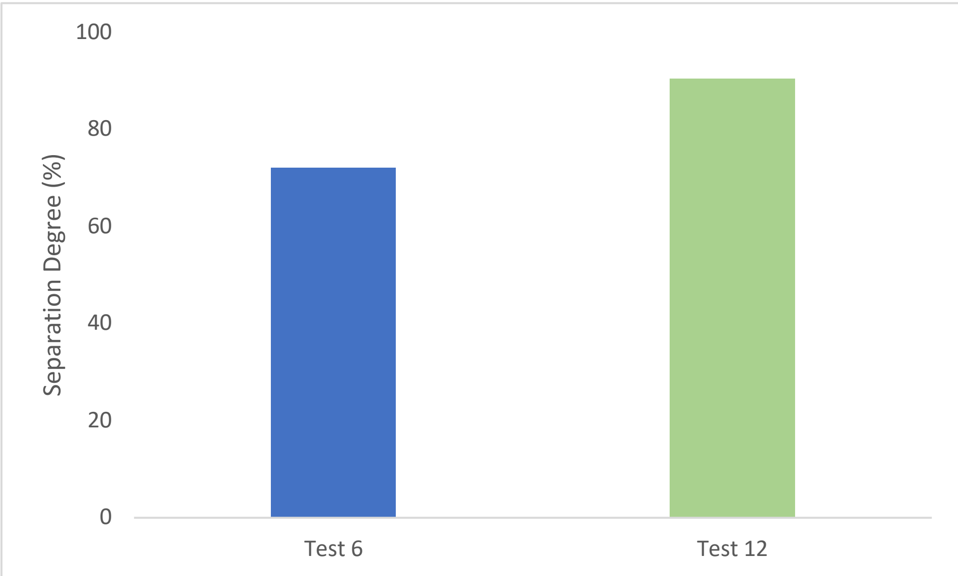



*Figure 48: Separation degree obtained for test 6 and 12*

Discussion:

A change in OOC was seen after the addition of MEG, where the oil separation degree increased from 72% to 98%. A lot of energy was used in this experiment, however, it is assumed that preheating the MEG and dosing it on hot cuttings, would reduce the energy consumption significantly. This is because the MEG is volatile once it is hot, and it will sweep the oil off.

The power density was increased 10 times in test 12, but still less power was put in to the test. Treatment with high power density resulted in $OOC_{wet}$ to 0,76, while low power density gave 2,24%. This equals an increase in oil separation from 72% to 90,5. This shows that increasing the power density will have a higher separation degree and require less energy input.

No further studies with high power density and MEG were conducted due to lack of cuttings material, but it is assumed that OOC would be reduced more, if MEG was added to cuttings in test 12. The biggest impact, with respect to both oil separation and energy consumption, will be when warm MEG is dosed on hot cuttings.

## New cuttings type received from an oil company in Canada

The cuttings received from this field will be referred to as "Canadian cuttings". This type of cuttings has never been tested by NT before. The drill cuttings come from a well that has been drilled with Oil based mud. More formation information and technical data has not been given. Two experiments without susceptor were conducted with low, medium and high power density to assess the effect of alternating power density on this type of cuttings.

### Experiment 3: The effect of low and medium power density with medium power input

For experiment 3, medium power input together with low and medium power density was investigated in this experiment. Haga (2017) studied the effect of low power density in microwave and found that low power density resulted in good water separation, but little oil separation. Procedure is depicted in figure 49 and initial treatment parameters is showed in table 20.


*Figure 49: Procedure for test 23 and 24 in experiment 3*

*Table 19: Initial treatment parameters for test 23 and 24 with low and medium power density*

| | **Test 23** | **Test 24** |
|---|---|---|
| Power Density | Low | Medium |
| Weight (g) | 227 | 219 |
| MEG (%) | 0 | 0 |
| Exposure time (s) | 200 | 134 |
| Energy input (W/h) | 600 | 1000 |
| Power density (W/h*kg) | 2643 | 4566 |
| Input power (W/kg) | 118 | 137 |

Results

The results for low and medium power density for Canadian cuttings is showed in table 21. Differences is seen in the values from the Retort and Soxtec. The initial $OOC_{dry}$ was 11,3 and $OOC_{wet}$ was 9,80 (Figure 50). Low power density resulted in 8,89 in $OOC_{dry}$, and the $OOC_{wet}$ was determined to be 8,35 and 6,47 in the Retort and Soxtec, respectively. A more significant reduction was seen in the water content, which decreased from 3,20% to 0,75%. Test 24 was exposed to most energy, 137 W/kg, but $OOC_{dry}$ was determined to be 9,15% and $OOC_{wet}$ was 8,05. In the Soxtec analysis, test 24 gave 4,25% in $OOC_{wet}$. Separation degree for experiment is shown in figure 51.

*Table 20: Results for low and medium power density in experiment 1*

| | **Initial** | **Test 23** | **Test 24** |
|---|---|---|---|
| Retort OOC dry (%) | 11,30 | 8,89 | 9,15 |
| Retort OOC wet (%) | 9,80 | 8,35 | 8,05 |
| Soxtec OOC wet (%) | - | 6,47 | 4,25 |
| Separation degree, wet (Retort) (%) | - | 14,8 | 17,8 |
| Separation degree, wet (Soxtec) (%) | - | 34,0 | 56,6 |
| Water | 3,20 | 0,75 | 1,39 |
| Power Density | - | 2643 | 4566 |
| Input Power | - | 118 | 137 |

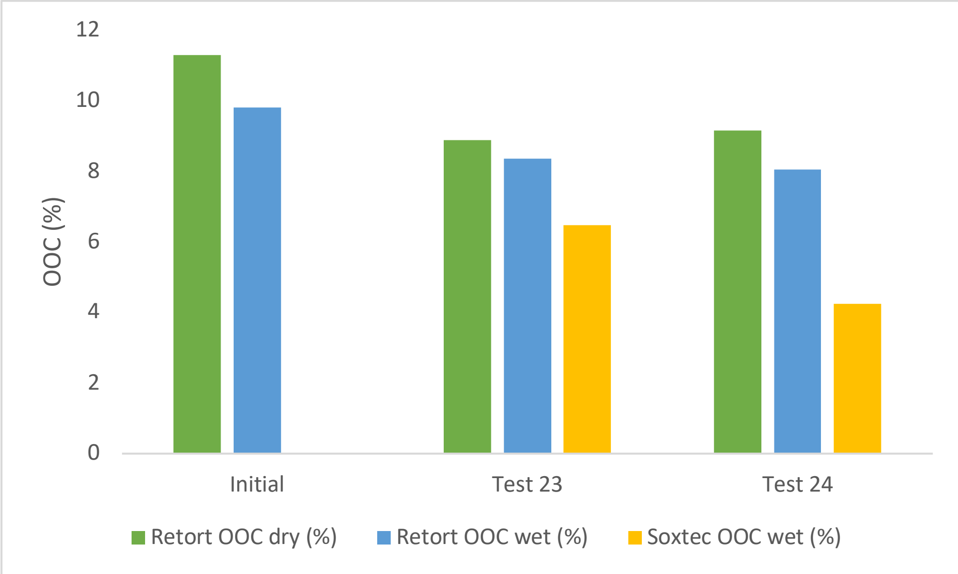



*Figure 50: OOC for test 23 and 24, with low an medium power density*

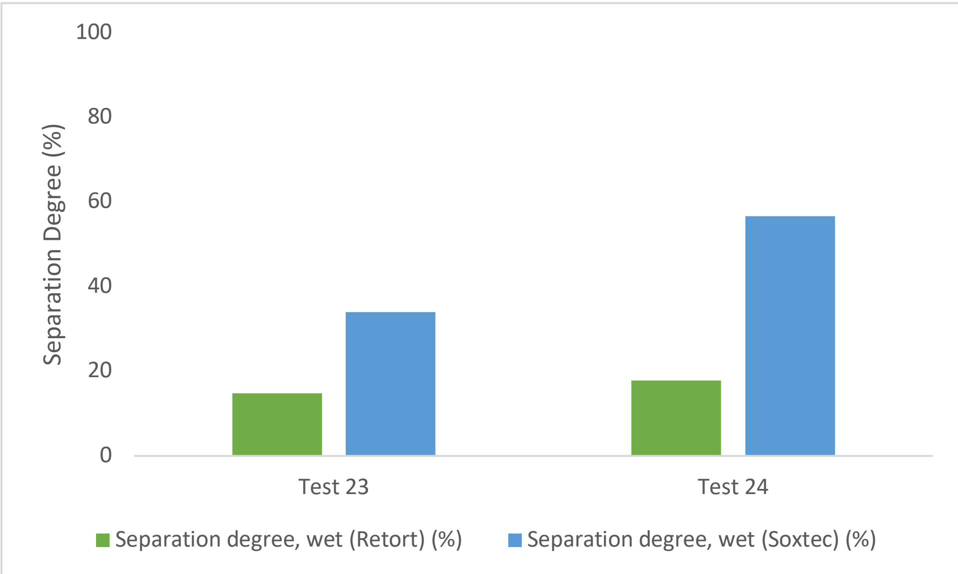



*Figure 51: Separation degree obtained for test 23 and 24*

**Experiment 4: The effect of medium and high power density with low power input**

Oil separation should in theory increase with increasing power density. This was tested in experiment 4. The procedure was the same as in Experiment 3 (Figure 52) and initial treatment parameters is showed in table 22.

*Table 21: Initial treatment parameters for experiment 4, with low and medium power density*

| | **Test 25** | **Test 26** |
|---|---|---|
| Power Density | Medium | High |
| Weight (g) | 268 | 229 |
| MEG (%) | 0 | 0 |
| Exposure time (s) | 55 | 36 |
| Energy input (W/h) | 1000 | 2000 |
| Power density (W/h*kg) | 3731 | 8733 |
| Input power (W/kg) | 50 | 56 |

<u>Results</u>

The results for medium and high power density is depicted in table 23. Differences in the values from the Retort and Soxtec was also seen in this experiment. The initial $OOC_{dry}$ was 11,3 and $OOC_{wet}$ was 9,80. Medium power density resulted in a separation degree of 14,7% and a reduction to 8,36 in $OOC_{wet}$, and the $OOC_{dry}$ was determined to be 9,27% (Figure 52 and 53). Water content was similar in the both of tests, 1,51 in test 25 and 1,59 in 1,59. The separation degree was less in test 26, only 9,1%. $OOC_{dry}$ was determined to be 9,99% and 8,90 in $OOC_{wet}$.

*Table 22: Results for Low and Medium Power Density for Canadian Cuttings without Susceptor*

| | **Initial** | **Test 25** | **Test 26** |
|---|---|---|---|
| Retort OOC dry (%) | 11,30 | 9,27 | 9,99 |
| Retort OOC wet (%) | 9,80 | 8,36 | 8,90 |
| Separation degree, wet (Retort) (%) | - | 14,7 | 9,1 |
| Water | 3,20 | 1,51 | 1,59 |
| Power Density | - | 3731 | 8733 |
| Input Power | - | 50 | 56 |

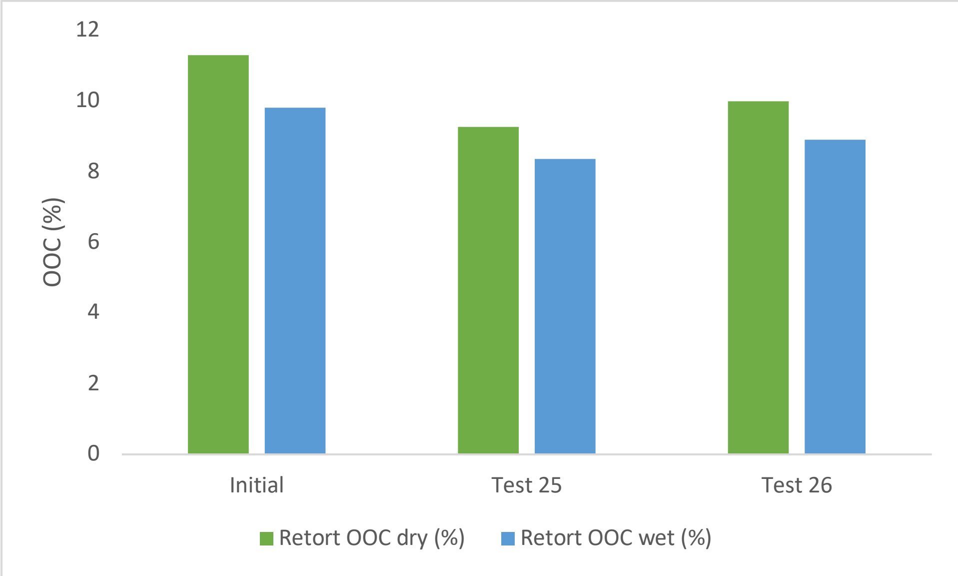



*Figure 52: OOC obtained in test 25 and 26*

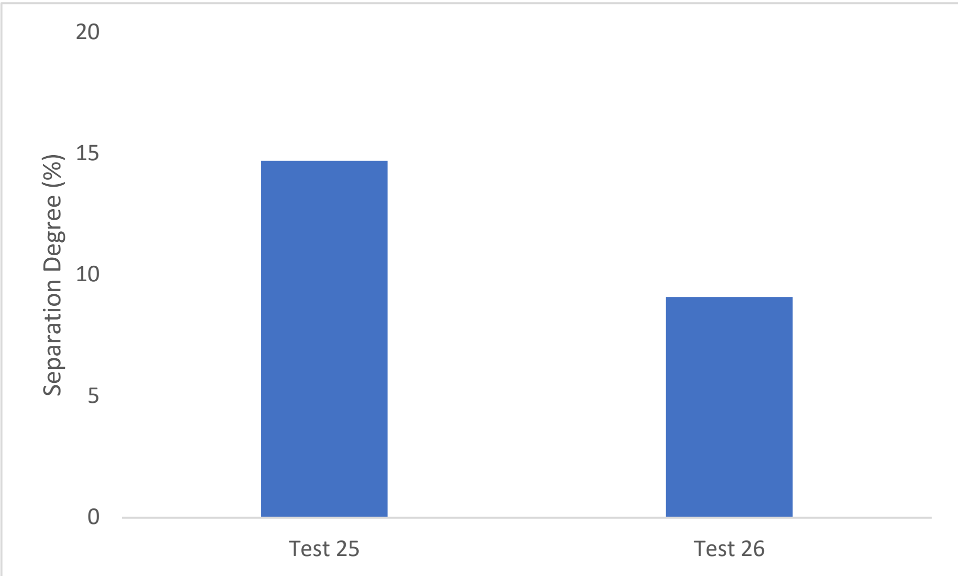



*Figure 53: Separation degree obtained in test 25 and 26*

<u>Discussion</u>

From the results it is clear that without susceptor, the oil levels are no near the limit required by OSPAR with the applied power in these experiments. The result in test 23 indicate that running the test on low power density with increasing treatment time result in good water separation. This was confirmed by Haga (2017).

Better oil separation was achieved with medium power density compared to low power density in experiment 3. This was controlled in the Soxtec, which is a more accurate test for low oil levels. The Soxtec demonstrated a much higher oil separation for both the test 23 and 24. The Soxtec did confirm that better oil separation was achieved with increasing power density. For high oil concentrations, retorte is the preferred analysis because it is considered as constant and accurate, it was therefore determined not to analyze test 25 and 26 in the Soxtec. The Retort is better on high oil concentrations than the Soxtec, because the solvent in the Soxtec is saturated when oil concentrations are high. However, the Soxtec is maybe more trustworthy as three parallel tests are conducted, while in the retort only 1 is conducted. The sample size in the Soxtec is also much smaller (5 grams), compared to the retorte cell which handles 60-90 grams of cuttings. Moreover, the mud contains polymers, such as xitanium which can decompose under high temperatures. If the polymers are decomposed in the Retort, it will be considered as oil. This could be confirmed if the Retort was conducted at low temperatures, because the polymers is likely to decompose at high temperatures.

The results from the Soxtec in experiment 1 confirms the theory about increased power density to increase the oil separation due to high entrainment. steam is generated quickly and sweeps off the oil.

In experiment 4, increased power density with that little amount of energy input does not seem to have an effect on oil separation. Reasons for poor oil separation might be due to water content, particle size, oil boiling point, but it was not conducted more studies without susceptor.

## 7.4 Assess the effect of high and low power density with susceptor

Two experiments with alternating characteristics were conducted to see the effect of high and low power density. Since the Soxtec is time-consuming, some samples were not analyzed in the Soxtec because of high oil content in the Retort.

**<u>Experiment 5: Uncentrifuged, pretreated with microwave, treat again with salt and MEG</u>**

In this experiment, the effect of uncentrifuged on high and low power density was evaluated. Both test was pretreated in microwave before 15% MEG and 4% salt was added (Figure 54). Initial treatment parameters are shown in 23.

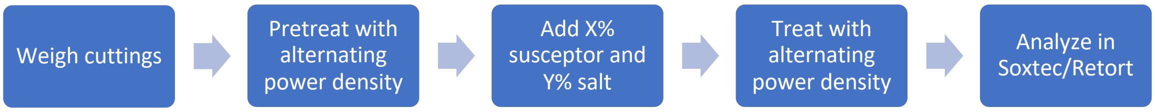



*Figure 54: Procedure for experiment 5*

*Table 23: Initial treatment parameters for experiment 5*

| | **Test 43** | **Test 50** |
|---|---|---|
| Low/High Power Density | Low | High |
| Dewatered | Yes | Yes |
| Centrifuged | No | No |
| Weight (g)* | 170 (188) | 160 (166) |
| MEG (%)* | 0 (15) | 0 (15) |
| Salt (%)* | 0 (4) | 0 (4) |
| Exposure time (s)* | 70 (168) | 30 (61) |
| Energy input (W/h)* | 600 | 2000 |
| Power density (W/h*kg)* | 3529 (3191) | 12500 (12048) |
| Input power (W/kg)* | 48 (111) | 74 (174) |
| Total input power (W/kg) | 159 | 248 |

* values in parentheses are after pretreatment in microwave.

Results

68% oil separation was achieved with high power density (test 50), while 56,4% was achieved with low power density (test 43), when looking at the Soxtec values. A larger difference in values were seen in the Retort. For high power density 77,5% was the result for $OOC_{dry}$ and 74,6% for $OOC_{wet}$ (Figure 56). Significant reductions in oil separation for the low power density test was observed with 41,6% in $OOC_{dry}$ and 56,4% in $OOC_{wet}$. In test 43, a reduction in water content was not evident. In test 50, the water content decreased by 29,7%.

*Table 24: Results for Experiment 5*

| | **Initial** | **Test 43** | **Sep. Degree (%)** | **Test 50** | **Sep. Degree (%)** |
|---|---|---|---|---|---|
| Retort OOC dry (%) | 15,8 | 9,22 | 41,6 | 3,55 | 77,5 |
| Retort OOC wet (%) | 13,1 | 8,13 | 37,9 | 3,33 | 74,6 |
| Soxtec OOC wet (%) | - | 5,71 | 56,4* | 4,19 | 68,0* |
| Water | 3,70 | 3,70 | 0,00 | 2,60 | 29,7 |
| Power Density | - | 3191 | - | 12048 | - |
| Input Power | - | 159 | - | 248 | - |

*Soxtec Separation degree is calculated based on initial value from Retort OOC wet

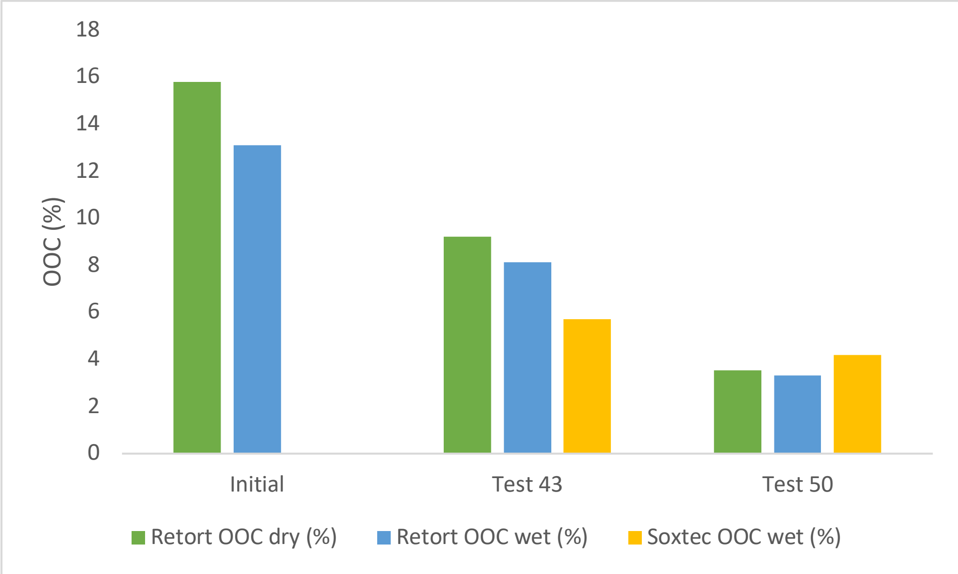


*Figure 55: OOC values obtained in Experiment 5, alternating power densities with susceptor and salt*

*Figure 56: Separation degree for uncentrifuged tested on high/low power density with susceptor and salt*

**Experiment 6: Centrifuged, direct dosed with MEG and salt.**

In this experiment, centrifuged cuttings were direct dosed with 15% MEG and 4% salt. The procedure is given in the flowchart in figure 57 and table 25 shows the initial treatment parameters for experiment 6.

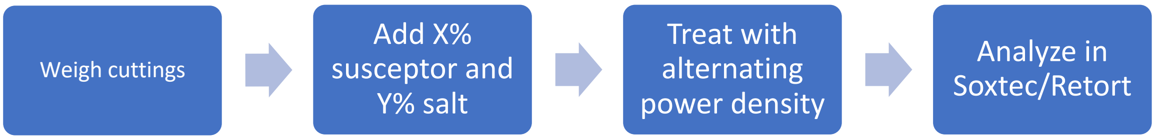



*Figure 57: Procedure for Experiment 6*

*Table 25: Initial treatment parameters for Experiment 6*

| | **Test 44** | **Test 51** |
|---|---|---|
| Low/High Power Density | Low | High |
| Dewatered | No | No |
| Centrifuged | Yes | Yes |
| Weight (g)* | 205 | 186 |
| MEG (%)* | 15 | 15 |
| Salt (%)* | 4 | 4 |
| Exposure time (s)* | 170 | 65 |
| Energy input (W/h)* | 600 | 2000 |
| Power density (W/h*kg)* | 2926 | 10752 |
| Input power (W/kg)* | 108 | 164 |

<u>Results</u>

A significant increase in oil separation was seen when applying high power density on centrifuged cuttings (Figure 59). 73,8% was achieved in test 51 with high power density, compared to test 44 with low power density, where separation degree was 21,2 when looking at $OOC_{wet}$. For $OOC_{dry}$, 68,6% was achieved with high power density, while 22,8 was the result with low power density.

*Table 26: Results obtained for Experiment 6*

| | Initial | Test 44 | Sep. Degree (%) | Test 51 | Sep. Degree (%) |
|---|---|---|---|---|---|
| Retort OOC dry (%) | 11,30 | 8,72 | 22,8 | 3,55 | 68,6 |
| Retort OOC wet (%) | 9,80 | 7,72 | 21,2 | 2,57 | 73,8 |
| Soxtec OOC wet (%) | - | - | - | 2,74 | 72,0* |
| Water | 3,20 | 3,74 | +16,8 | 2,43 | 24,1 |
| Power Density | - | 2926 | - | 10752 | - |
| Input Power | - | 108 | - | 164 | - |

*Soxtec Separation degree is calculated based on initial value from Retort OOC wet

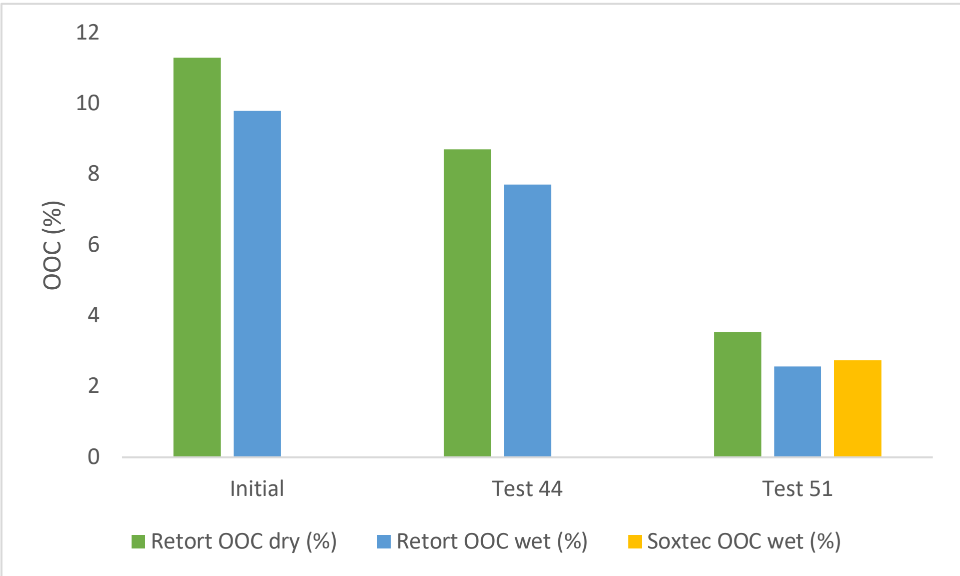



*Figure 58: OOC obtained in when alternating power density in Experiment 6*

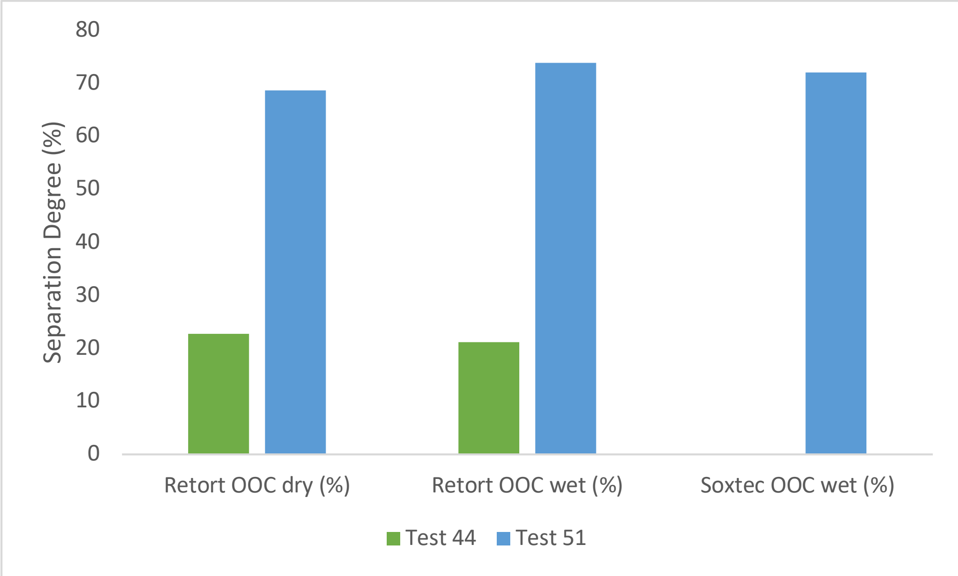



*Figure 59: Separation degree obtained in when alternating power density in Experiment 6*

Discussion

For the uncentrifuged cuttings, the Soxtec $OOC_{wet}$ was 5,71% for low power density and 4,19% for high power density. No OOC under 4 was measured in any of the uncentrifuged tests during this project. It is uncertain why it is not reduced more, but it can be assumed that too little susceptor has been dosed in the uncentrifuged samples. 15% susceptor might limit the oil separation for uncentrifuged samples. In experiment 6 conducted with centrifuged cuttings, the OOC reduced down to 2,74 with high power density. Comparing experiment 5 and 6 in this chapter, centrifuged cuttings contained less OOC after treatment. This was also achieved with less power than with uncentrifuged cuttings. 15% MEG indicates to be sufficient for centrifuged cuttings. This may be because oil and water have been reduced in the centrifuge. In addition, the cutting will be more compressed after the centrifuge. More compact cuttings allow for the MEG to flow through the sample easier. It is also beneficial with small particles (Chapter 4.1).

## 7.5 Changing cuttings characteristics with centrifuge

The effect of changing cuttings characteristics with a centrifuge was investigated. Centrifuging the cuttings were cuttings were conducted after the method described in chapter 5. The values for raw and centrifuged cuttings is given table 27. A separation degree of 28,5%, 25,2 and 13,6 was achieved for $OOC_{dry}$, $OOC_{wet}$ and water content. Figure 60 demonstrates the structure of raw and centrifuged cuttings. It is possible to see liquid in the raw cuttings, while the centrifuged cuttings appear to be more dry.

*Table 27: OOCdry, OOCwet and water content for raw and centrifuged cuttings*

| | Raw | | | Centrifuged | | |
|---|---|---|---|---|---|---|
| | **OOCdry %** | **OOCwet %** | **Water %** | **OOCdry %** | **OOCwet %** | **Water %** |
| **Test 1** | 17,9 | 14,6 | 3,9 | 12,6 | 10,8 | 3,4 |
| **Test 2** | 14,8 | 12,5 | 3,5 | 9,3 | 8,3 | 3,1 |
| **Test 3** | 14,7 | 12,4 | 3,6 | 12,0 | 10,4 | 3,1 |
| **Average** | **15,8** | **13,1** | **3,7** | **11,3** | **9,8** | **3,2** |
| **Standard deviation** | 2,13 | 1,26 | 0,2 | 1,71 | 1,34 | 0,2 |
| **Separation Degree (%)** | | | | 28,5 | 25,2 | 13,6 |

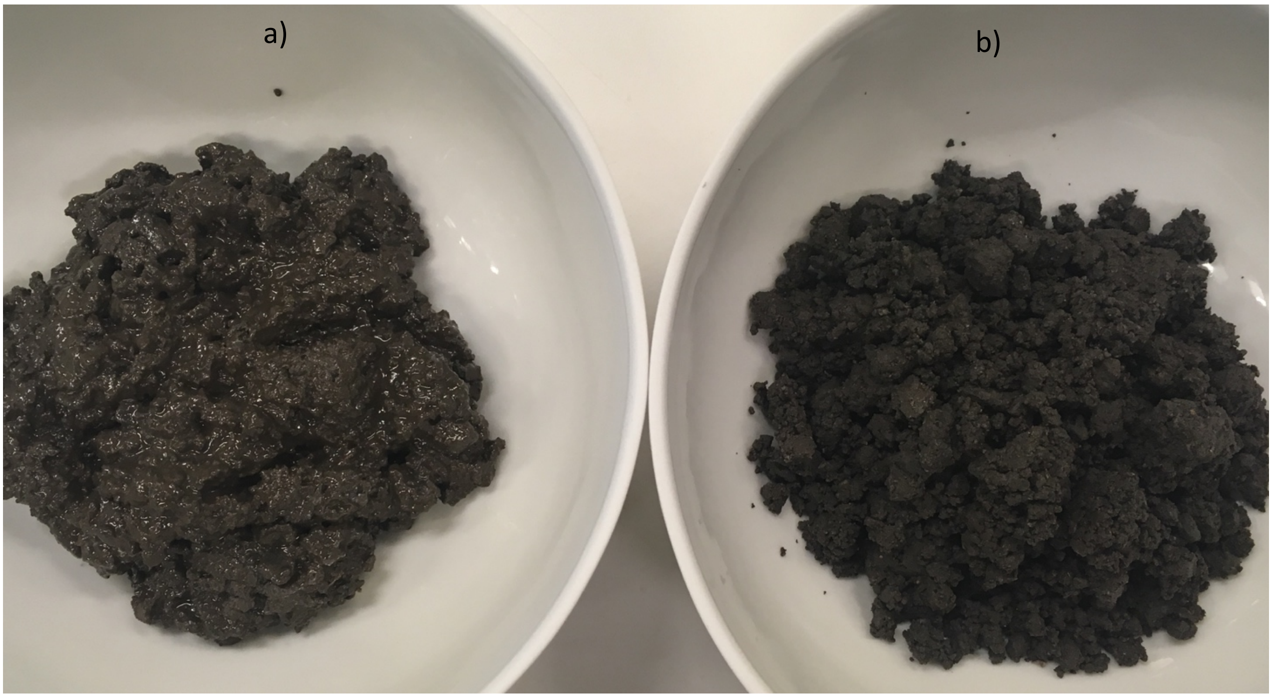



*Figure 60: Structure of raw and centrifuged cuttings*

**Experiment 7: The effect of centrifuging after treatment**

In this experiment, one sample was raw, and one samples was centrifuged, all other parameters were the same. Both of the samples were dewatered in microwave before 15% MEG and 4% salt was added to maximize oil separation (Figure 61). Both tests were conducted with high power density. Initial treatment parameters is given in table 28.

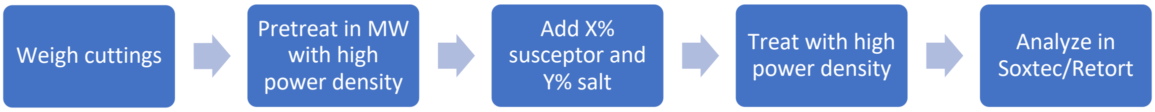



*Figure 61: Procedure for Experiment 7*

*Table 28: Initial treatment parameters for Experiment 7*

| | **Test 50** | **Test 52** |
|---|---|---|
| Centrifuged | No | Yes |
| Dewatered | Yes | Yes |
| Weight (g)* | 160 (166) | 160 (173) |
| MEG (%)* | 0 (15) | 0 (15) |
| Salt (%)* | 0 (4) | 0 (4) |
| Exposure time (s)* | 30 (61) | 30 (58) |
| Energy input (W/h)* | 2000 | 2000 |
| Power density (W/h*kg)* | 12500 (12048) | 12500 (11560) |
| Input power (W/kg)* | 74 (174) | 74 (156) |
| Total input power (W/kg) | 248 | 230 |

* values in parentheses are values added after dewatering in microwave.

Results

Test 52 was conducted with little less energy input than test 50, however a separation degree of 93,2 was achieved with centrifuged cuttings (Figure 63). Raw cuttings were reduced by 68% after treatment in microwave. The values in the Retort showed a smaller separation degree difference between the two tests, 81,7 in $OOC_{wet}$ and 83,5 for $OOC_{dry}$ for test 52 with centrifuged cuttings, while a separation degree of 74,5% in $OOC_{wet}$ and 77,5% in $OOC_{dry}$ was achieved after microwave treatment with raw cuttings. Test 52 was the only sample which was treated to under 1% with Canadian Cuttings (Figure 62).

*Table 29: Results obtained in Experiment 7*

| | Initial, raw cuttings | Test 50 | Sep. Degree (%) | Initial, centrifuged cuttings | Test 52 | Sep. Degree (%) |
|---|---|---|---|---|---|---|
| Retort OOC dry (%) | 15,8 | 3,55 | 77,5 | 11,30 | 1,87 | 83,5 |
| Retort OOC wet (%) | 13,1 | 3,33 | 74,5 | 9,80 | 1,79 | 81,7 |
| Soxtec OOC wet (%) | - | 4,19 | 68,0* | - | 0,67 | 93,2* |
| Water | 3,7 | 2,6 | 29,7 | 3,20 | 2,64 | 17,5 |
| Power Density | - | 12048 | | - | 11560 | - |
| Input Power | - | 248 | | - | 230 | - |

*Soxtec Separation degree is calculated based on initial value from Retort OOC wet

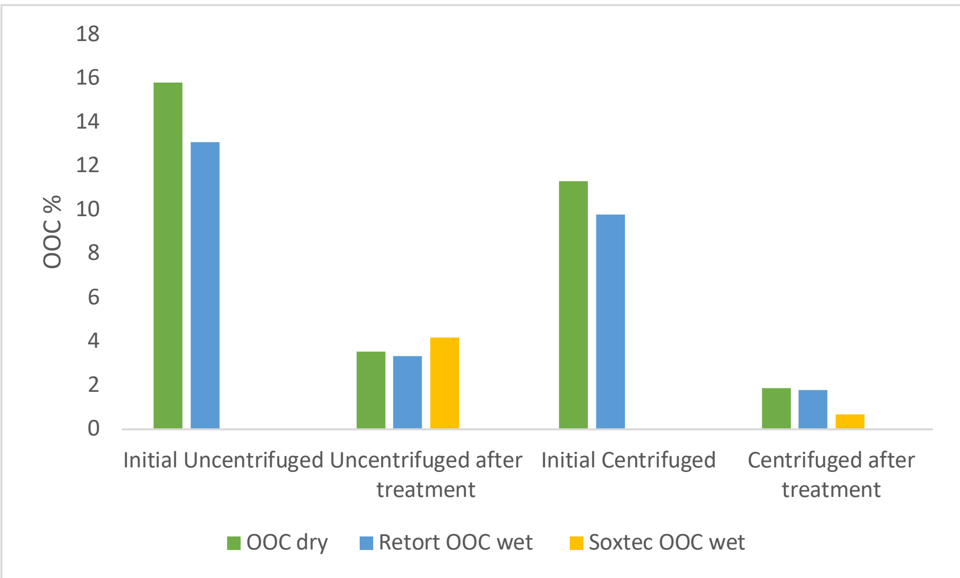



*Figure 62: OOC obtained in Experiment 7 with raw and centrifuged cuttings on high power density*

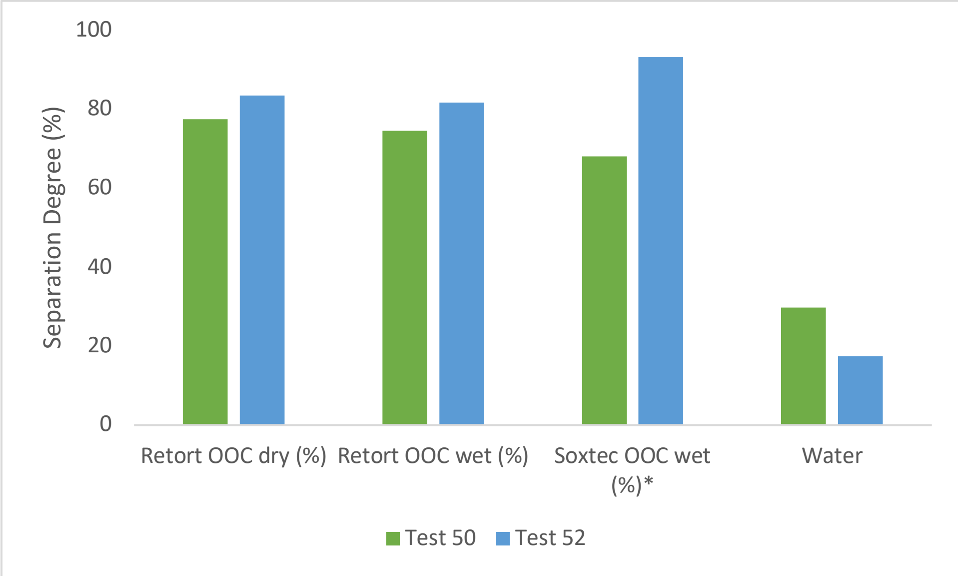



*Figure 63: Separation Degree for raw and centrifuged cuttings after treatment in microwave with high power density*

Discussion

A better separation degree for oil was obtained in all results with centrifuged cuttings. Higher water separation was achieved with raw cuttings. The difference between the cuttings before treatment was 3,3% in $OOC_{wet}$. After treatment in microwave, the difference was 3,5%. From these tests, centrifuged cuttings appear to be beneficial for oil separation. Some water is left in the test after treatment, and a part of the water content is assumed to be glycol. To reduce loss of glycol, it is suggested to put more energy into the sample. This is likely to lead to better oil separation and more glycol available for re-use.

## 7.6 Susceptor inmixing

The susceptor dosing point was investigated to see the effect on oil separation and susceptor when water was present compared to when it was absent. Direct dosing of susceptor could potentially save energy in the treatment step, but in order for MEG to be re-used, a separation system is then required to separate MEG and water. Moreover, water will decrease the process temperature which leads to reduced oil separation. To compare, experiments with direct dosing were also conducted. Figure 64 describes the procedure for the tests and initial treatment parameters is given in table 30.

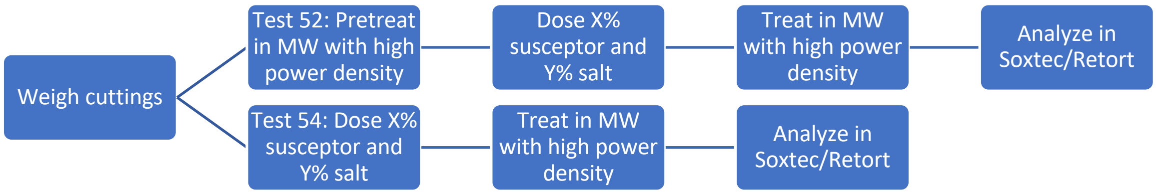



*Figure 64: Procedure for test 52 and 54*

*Table 30: Initial treatment parameters for tests 52 and 54*

| | **Test 52** | **Test 54** |
|---|---|---|
| Dewatered | Yes | No |
| Centrifuged | Yes | Yes |
| Weight (g)* | 160 (173) | 184 |
| MEG (%)* | 0 (15) | 15 |
| Salt (%)* | 0 (4) | 4 |
| Exposure time (s)* | 30 (58) | 77 |
| Energy input (W/h)* | 2000 | 2000 |
| Power density (W/h*kg)* | 12500 (11560) | 10869 |
| Input power (W/kg)* | 74 (156) | 207 |
| Total input power (W/kg) | 230 | 207 |

* values in parentheses are values added after dewatering in microwave.

Results

Up to 93,2% in oil separation was achieved with pretreating in test 52 with the Soxtec. The Retort showed 83,5% and 81,7% in oil separation for $OOC_{dry}$ and $OOC_{wet}$, respectively (Figure 66). Direct inmixing of MEG and salt in Test 54 also achieved a high oil separation, with 81,6% in the Soxtec. The results from the Retort gave 76,7% in $OOC_{dry}$ and 76,8% in $OOC_{wet}$. Again, test 52 achieved less than 1% with 0,67% in $OOC_{wet}$ , while test 54 achieved 1,80% (Figure65)

*Table 31: Results obtained for test 52 and 54*

| | Initial | Test 52 | Sep. Degree (%) | Test 54 | Sep. Degree (%) |
|---|---|---|---|---|---|
| Retort OOC dry (%) | 11,30 | 1,87 | 83,5 | 2,63 | 76,7 |
| Retort OOC wet (%) | 9,80 | 1,79 | 81,7 | 2,27 | 76,8 |
| Soxtec OOC wet (%) | - | 0,67 | 93,2 | 1,80 | 81,6 |
| Water (%) | 3,20 | 2,64 | 17,5 | 1,4 | 56,3 |
| Power Density | - | 11560 | - | 10869 | - |
| Input Power | - | 230 | - | 207 | - |

*Soxtec Separation degree is calculated based on initial value from Retort OOC wet

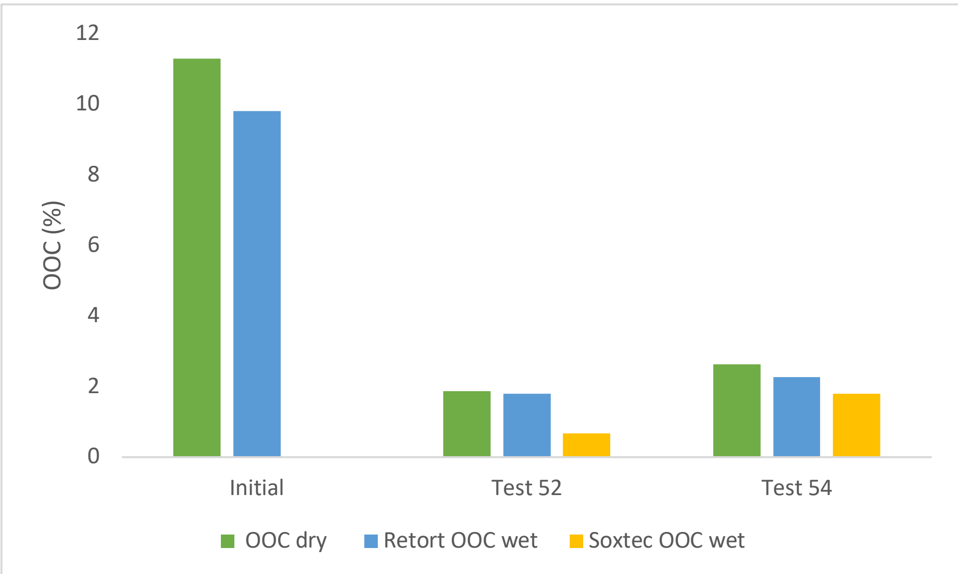



*Figure 65: OOC obtained test 52 and 54*


*Figure 66: Oil separation achieved in test 52 and 54*

<u>Discussion</u>

The tests indicate that pretreating the cuttings in microwave before adding MEG and salt is beneficial. Water present will reduce the process temperature which reduces the treatment efficiency, which is discussed in the works of Egar (2017). In these test, the MEG and salt was dosed on cold cuttings. This means that the cuttings need more heat for be warm again, and much energy is needed for MEG to heat. It is expected that dosing warm MEG on hot cuttings will have a distinct impact on the energy consumption.

It may also be an advantage to pretreat the samples in microwave with low power density as Haga (2017) recommended. This will increase the water separation.

## 7.7 Susceptor dosing quantity

The amount of susceptor was investigated in regard to oil separation. It is assumed that increasing amount of susceptor is advantageous for oil separation, despite previously findings by (Egar, 2017). If the cuttings are dosed with an insufficient amount, the susceptor is not able to wet all the pores, hence the MEG will not be able to catch all the oil and it will be left in the sample. Two experiments were conducted, one with salt and one without.

**Experiment 9: Without salt**

The procedure test without salt is depicted in figure 66 and initial treatment parameters is shown in table 32.


*Figure 67: Procedure for experiment 9, dosing quantity without salt*

*Table 32: Initial parameters for Experiment 9, dosing quantity without salt*

| | **Test 34** | **Test 35** |
|---|---|---|
| MEG (%)* | 0 (15) | 0 (20) |
| Dewatered | Yes | Yes |
| Centrifuged | Yes | Yes |
| Weight (g)* | 185 (134) | 185 (160) |
| Salt (%)* | 0 | 0 |
| Exposure time (s)* | 26 (22) | 26 (26) |
| Energy input (W/h)* | 2000 | 2000 |
| Power density (W/h*kg)* | 10810 (14925) | 10810 (12500) |
| Input power (W/kg)* | 48 (61) | 48 (60) |
| Total input power (W/kg) | 109 | 108 |

* values in parentheses are after dewatering in micro.

Results

The Soxtec illustrates that more oil is removed with 20% MEG than with 15%. Test 35 gave 72,6% in the Soxtec, while the Retort showed 42,4% for $OOC_{wet}$ and 39,1% $OOC_{dry}$ (Figure 68). Total liquid content increased for test 35 (Figure 69). The Retort demonstrated that more oil is separated with 15%. $OOC_{wet}$ was 45,5% and $OOC_{dry}$ was 46,23% for test 34. $OOC_{wet}$ in the Soxtec showed 62,7% for test 34.

*Table 33: Results obtained for Experiment 9*

| | **Initial** | **Test 34** | **Sep. Degree (%)** | **Test 35** | **Sep. Degree (%)** |
|---|---|---|---|---|---|
| Retort OOC dry (%) | 11,30 | 6,16 | 45,5 | 6,88 | 39,1 |
| Retort OOC wet (%) | 9,80 | 5,26 | 46,3 | 5,64 | 42,4 |
| Soxtec OOC wet (%) | - | 3,66 | 62,7 | 2,69 | 72,6 |
| Water (%) | 3,20 | 4,35 | +35,9 | 12,33 | +284,4 |
| Total liquid (%) | 14,50 | 10,51 | - | 19,21 | - |
| Power Density | - | 14925 | - | 12500 | - |
| Input Power | - | 109 | - | 108 | - |

*Soxtec Separation degree is calculated based on initial value from Retort OOC wet

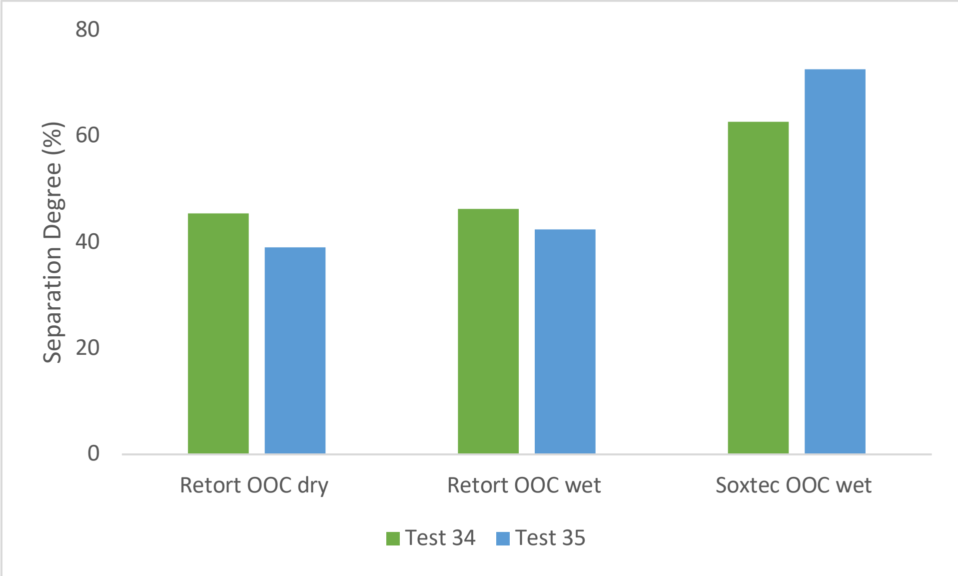



*Figure 68: Oil separation obtained for Experiment 9*

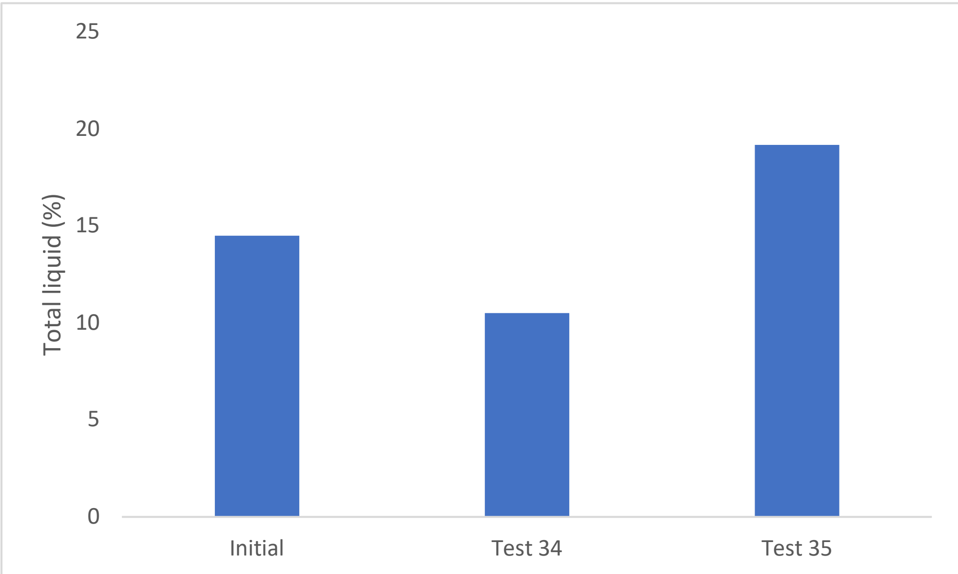



*Figure 69: Total liquid content increased with increasing MEG*

**Experiment 10: With salt**

In this experiment, salt was added due to findings by Haga (2017), which implied that salt would increase microwave absorbance. Input power was also increased due to very high liquid levels in test 35. Procedure for this experiment is given in figure 69 and initial parameters in table

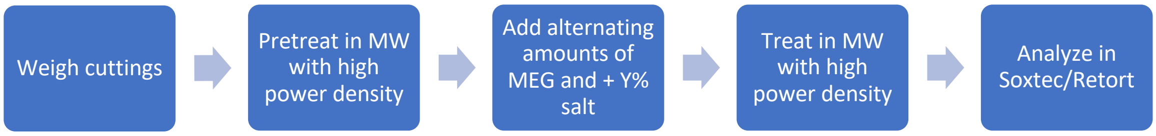



*Figure 70: Procedure for Experiment 10*

*Table 34: Initial treatment parameters for Experiment 10*

| | **Test 53** | **Test 55** |
|---|---|---|
| MEG (%)* | 0 (40) | 0 (30) |
| Dewatered | Yes | Yes |
| Centrifuged | Yes | Yes |
| Weight (g)* | 160 (202) | 160 (165) |
| Salt (%)* | 0 (4) | 0 (4) |
| Exposure time (s)* | 30 (58) | 30 (55) |
| Energy input (W/h)* | 2000 | 2000 |
| Power density (W/h*kg)* | 12500 (9900) | 12500 (12121) |
| Input power (W/kg)* | 104 (159) | 93 (160) |
| Total input power (W/kg) | 263 | 253 |

* values in parentheses are after dewatering in micro.

Results

In this experiment it was also observed a small increase in oil separation degree for the sample with largest amount of MEG (Test 53) (Figure 71). 89,5% was achieved with 40% MEG in the Soxtec. The Retort showed similar values, with 87,3% in $OOC_{dry}$ and 86,1% in $OOC_{dry}$. In test 55, which was added 30% MEG, the Soxtec value showed 85,7%. The Retort was determined to be 77,9% for $OOC_{dry}$ and 77,0 for OOCwet. In this experiment, total liquid content decreased with both of the test. The largest decrease in liquid content was achieved with the test that was added the most MEG.

*Table 35: Results obtained for Experiment 10*

| | Initial | Test 53 | Sep. Degree (%) | Test 55 | Sep. Degree (%) |
|---|---|---|---|---|---|
| Retort OOC dry (%) | 11,30 | 1,44 | 87,3 | 2,50 | 77,9 |
| Retort OOC wet (%) | 9,80 | 1,36 | 86,1 | 2,25 | 77,0 |
| Soxtec OOC wet (%) | - | 1,03 | 89,5 | 1,4 | 85,7 |
| Water (%) | 3,20 | 4,04 | +26,5 | 7,88 | 146,3 |
| Total liquid (%) | 14,50 | 5,48 | - | 10,38 | - |
| Power Density | - | 9900 | - | 12121 | - |
| Input Power | - | 263 | - | 253 | - |

*Soxtec Separation degree is calculated based on initial value from Retort OOC wet


*Figure 71: Separation degree obtained for experiment 10*

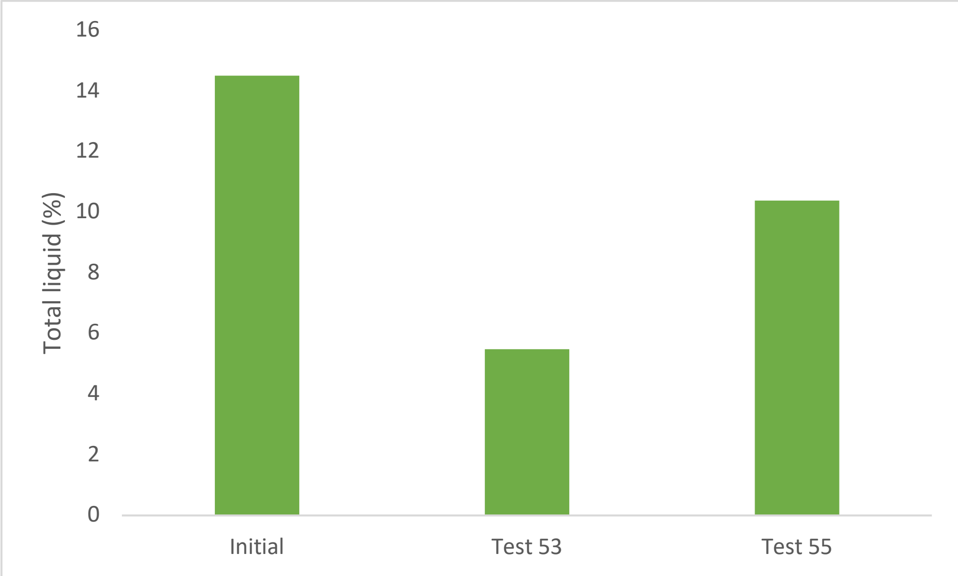



*Figure 72: Total liquid content achieved with 40% and 30% MEG*

Discussion

From experiment 9, oil separation is slightly larger in test 35 than 34, despite that very little energy that was used. This indicates that increasing dose of MEG is desired. Figure 68 shows that the total liquid increases. This imply that a significant amount of MEG has not vaporized off. More energy is needed to heat the susceptor.

In experiment 10, both the energy input and MEG concentration were increased. Test from experiments also indicates that increasing MEG enhances oil separation. What is notably is the difference in total liquid content in test 53 and 55. 53 was dosed with 10% more susceptor than 55, however the liquid content was half as much in test 53 compared to 55. 10 W/kg vaporized 15% more glycol off. This demonstrates that MEG need a lot of energy to be heated up to approximately 200℃, but when that temperature is reached, and the test is irradiated for a little longer of time, it will vaporize very quickly due to its volatility.

This implies that MEG should be pre-heated. If the MEG is pre-heated, the dose does imply to have insignificant meaning. From the experiments, it is demonstrated that more susceptor increases oil separation, and dosing the susceptor with 40% instead of 30% will only require 10 W/kg more.

Some samples was added salt based on Haga´s (2017) findings that more microwaves was absorbed when salt was present. The inmixing method varied, and it is therefore not possible to draw a conclusion of the results from this thesis. More research is needed to clarify the effect of salt. This was originally one of the thesis objectives, but other challenges had to be resolved before finding a good inmixing method.

# 8. Discussion

Re-condensation, solid entrainment and nitrogen leakage were challenges that occurred during testing. Modifications of the sample container were done to avoid the effects of the challenges. The result was a PYREX glass cylinder with two lids. The lids trapped the liquid between them, preventing the gas to re-condensate back on the cuttings. The lower lid also prevented the cuttings to be pushed up, thereby preventing solid entrainment. A cap was added to the bottom of the cylinder and sealed with Teflon tape to prevent nitrogen leakage. All the modifications were successful, and the effects of the challenges were cancelled.

The effect of nitrogen temperature was investigated with the new sample container. Egar (2017) argued that cold nitrogen was the reason for re-condensation of gas. The OOC values were similar in both of the test in this experiment, which indicated that the modifications of the new sample container trapped the re-condensate liquid between the lids. The rest of the test were conducted with cold nitrogen, based on the results, which simplifies the set-up. However, many uncertainties were related to this experiment. For example, cuttings characteristics were not optimal. Due to the uncertainties it is suggested to do more testing on this to clarify the effect of cold nitrogen.

Testing on Halliburton cuttings demonstrated that increasing power density led to a significantly decrease in OOC, at the same time as using less energy input compared to low power density. This correspond with the theory of high and low power density. The high power density increases the entrainment which is the reason of high oil removal. This is because steam is formed quickly, and the velocity of the steam is high. The effect of MEG was also tested on the Halliburton cuttings, and the results indicated a separation degree of 90,5%. This is due to the high process temperature for the glycerol/oil distillation which results in a high shared vapor pressure which leads to enhanced separation of oil and cuttings. An energy optimization was not conducted for the Halliburton cuttings due to lack of cuttings material. However, it is assumed that energy consumption could decrease significantly with pre-heated MEG dosed on hot cuttings.

Treatment of Canadian cuttings without susceptor appeared to be challenging, however the theory about increased separation was also demonstrated in these tests when sufficient energy is added. The importance of a susceptor is here demonstrated. Water requires a high amount of energy because of the high vaporization and distillation ratios (table 7). The poor oil separation that was achieved with little energy may be due to water content, particle size, oil boiling point, but it was not conducted more studies without susceptor.

Changing cuttings characteristics with respect to centrifuging demonstrated to have a good effect. Centrifuged cuttings were treated to <1%, in which uncentrifuged cuttings were no near. Uncentrifuged cuttings stagnated at 4% in all the tests. This may have been due to too little susceptor. However, uncentrifuged cuttings may have difficulties in absorbing the glycol because of the already high liquid content. The centrifuged cuttings absorb the susceptor better which allow the susceptor to cover more particles. Centrifuged cuttings also have smaller particles size which according to theory is good for enhanced oil removal.

Pre-treating the cuttings in microwave implied to have a good effect on the oil separation. Without pretreatment, more water will be present that reduces the oil separation because of reduced process temperature. Mixing water and MEG also decreases the chance of re-use of susceptor, because decomposition and decontamination.

The results for susceptor quantity supports the assumption that oil separation is increased if warm MEG is dosed on hot cuttings. Now, energy have been used on heating the cuttings and then for it to cool before susceptor is dosed. More energy is needed after the dosing to heat both the cuttings over again, but also the susceptor. The energy needed in this step is cancelled if the cuttings and susceptor were warm. The glycol need some energy to become warm, but vaporizes off very quickly when it is warm, and little energy is needed to further increase the separation.

# 9. Conclusions

The main goal of this project was to evaluate the use of a single-mode applicator when treating drill cuttings with susceptor. With this micro it is now possible to quantify the energy absorbed by calculating the energy input and reflected.

Modifications of the sample container had to be done due to challenges that was observed during testing. The modifications successfully cancelled the problems of re-condensation, cuttings entrainment and nitrogen leakage. A quick test on nitrogen gas temperature was conducted. The results demonstrated that the modification of sample holder also cancelled the effect of cold nitrogen. It is however recommended to do more research on this topic.

Halliburton cuttings was successfully treated to less than the OSPAR requirement with the single-mode unit. The enhanced effect of oil separation using high power density in a single-mode applicator was evident for the Halliburton cuttings, even without susceptor. The addition of susceptor enhanced the oil separation significantly.

Based on the tests with Canadian cuttings, the microwave had problems with removing oil without susceptor, even at high power density the cuttings with high power densities without susceptor. Cuttings characteristics was alternated using a centrifuge and it was demonstrated to be beneficial for oil separation. The best OOC value of 0,67% was achieved with centrifuged cuttings. OOC values below 4% was not achieved with uncentrifuged cuttings. Cuttings should therefore be centrifuged before treatment.

Pretreating the cuttings in a microwave before the addition of susceptor and salt also implied to be beneficial. However, the susceptor and cuttings should both be warm before being exposed to microwaves. This could save tremendously of energy but is still yet to be proved. The tests conducted on susceptor dosing quantity indicates that MEG need quite much energy to be warm. However, when the susceptor is warm, it does not require much energy to vaporize off and remove oil. In some cases 15% showed to decrease OOC below 1%.

Recommendations

- Investigate the energy savings on dosing warm MEG on hot cuttings. This have the potential to save a significant amount of energy and thereby cancelling the trade-off between oil separation and energy requirement.
- Determine water concentration in MEG after treatment to evaluate the decomposition degree
- Investigating the effect of decomposed MEG. More laboratory studies should be conducted to determine the degree of decomposition, acid generation etc.
- Determine the effect of cold nitrogen with optimal cutting characteristics

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