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Abstract

The need for more environmental and economical solutions to treat drilling waste offshore led Norwegian-Group AS to develop a novel technology. This technology is based on microwave processing of drilling waste. The efficiency and the capacity of microwave treatment is, however, limited by susceptor availability. To overcome this limitation, environmentally friendly chemicals, which have lower enthalpy of vaporization than water, are used. Proper pre-treatment of drill cuttings, which is the main subject of this thesis, is also required for this technology.

The aim of this thesis is to elaborate potential methods for proper pre-treatment of drill cuttings. To achieve this objective, the drilling wastes are first reviewed to understand their composition, the bonding system between oil, water and cuttings, the magnitude of the produced waste as well as the disposal options available for waste handling. The choice of the potential method relies on these parameters since they influence the efficiency of a treatment method. The existing regulations as well as the technologies currently available for offshore treatment of drill cuttings are also briefly discussed. Some potential drying methods for cuttings are presented, and techniques based on convective thermal drying are further evaluated. A pre-treatment method based on chemical and centrifugal separation was also tested in the laboratory to evaluate its feasibility. The experiment was carried out in two phases. The first phase was used to verify the effect of MudSplit chemicals on drill cuttings and the optimal conditions of their use. The second phase was dedicated to further testing the effect of MudSplit chemical by combining it with solvent extraction.

The evaluation of the convective drying methods indicated that high separation efficiency, very low energy consumption and high treatment capacity can be achieved with these methods.

The laboratory testing demonstrated that MudSplit 02 chemical followed by centrifugal separation is efficient in dewatering drill cuttings. However, it was ineffective in terms of oil separation and did not enhance the oil extraction when combined with solvents.

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Abbreviations

WER	World Energy Resources
OGP	International Association of Oil & Gas Producers
JWEL	Jacques Whitford Environment Limited (Canada)
WBM	Water based mud
OBM	Oil based mud
SBM	Synthetic based drilling mud
IOGP	International Association of Oil & Gas Producers
PAH	Polycyclic aromatic hydrocarbons
EPA	US Environmental Protection Agency
CMO	Conventional Mineral Oil
TAH	Total aromatic hydrocarbons
LTMO	Low toxicity mineral oil
ATSDR	Agency for Toxic Substances and Disease Registry
NCS	Norwegian Continental Shelf
ROC dry	Oil retention on dry cuttings
NOROG	The Norwegian Oil and Gas Association
DNV	Det Norske Veritas
PLONOR	Pose little or no risk to the environment
NEA	Norwegian Environment Agency
OSPAR commission	Oslo and Paris commission
OSCOM	Oslo Commission
PARCOM	Paris Commission
TCC	Thermo-mechanical Cuttings Cleaner
BAT	Best available technology
BEP	Best environmental practice
HMCS	Harmonized Mandatory Control System
OPF	Organic phase drilling fluid
HSE	Health, Safety and Environment
NPD	Norwegian Petroleum Directorate
SBM	Synthetic based mud
SSD	Superheated steam drying
SHS	Superheated steam
BMA	Braunschweigische Maschinenbauanstalt AG Germany

Chapter 1: Introduction

Pre-treatment of contaminated drill cuttings is a crucial step to provide efficient cuttings cleaning. In this thesis, potential methods for pre-treating drill cuttings prior to microwave treatment are evaluated.

1.1 Problem Description

Oil and natural gas consist of the second and third primary source of energy in the world after coal, according to World Energy Resources, WER (2013). Oil and natural gas production activities continuously rise. For instance, a growth rate of 25% and 62% was observed in the years 1993 to 2011 for oil and natural gas respectively (WER, 2013). The exploration and production of these resources involve drilling operation. Due to several factors including among others the increase in energy demand, technological development, resources depletion, the drilling activities become more and more advanced. If vertical drilling was first the most common technique, currently operators use more complex practice such as directional and extended-reach drilling to improve the productivity and explore more challenging reservoirs (Chevron Corporation, 2013). This leads to an increasing technical challenge, which requires the elaboration of more complex and refined drilling fluid to reach higher performance and production (OGP, 2003).

However, the resulting high cost and environmental concerns, which rise stricter environmental regulations, make drilling waste management an important key solution for a viable and sustainable productivity in the oil and gas industry (Geehan and McKee, 1989; Bloys et al., 1994; OGP, 2003). *The purpose is to reduce the operational cost by recovering as much used drilling mud as possible and to reduce the environmental impacts of drilling activities* (Paulsen et al., 2000). Several techniques have been adopted to treat and dispose drilling waste in order to achieve the objectives stated above. Offshore treatment and discharge were demonstrated to be the simplest and most economically feasible option for waste handling. However, few technologies have the capacity to reach the requirement in environmental regulations when operated offshore.

The elaboration of new drilling waste treatment methods suitable for offshore use is then necessary. Technologies which fulfill such requirements as robustness, treatment capacity higher than 5 t/h, small footprint, low energy consumption, suitable for offshore use, easy to operate, health, safety and environment friendly, suitable for any type of drill cuttings, are of high priority for offshore waste treatment.

1.2. Norwegian-Group AS Activities

Founded in 2012, Norwegian-Group AS is a company which provides environmental solutions to the oil and gas industry. The company has developed a novel technology to treat drill cuttings offshore. The technology consists of thermally separating the oil from drill cuttings using microwave processing. Microwave treatment of drill cuttings is an emerging and well-known method. It reduces the energy consumption by selectively heating the materials present in the waste. However, the efficiency of microwave treatment is limited by the availability of susceptor such as water which receives the energy. To achieve higher treatment capacity and high oil separation degree, large amount of water is necessary. Since drill cuttings generally contain less water (about 15 %) after the primary treatment, microwave treatment is not efficient to reach the low oil on cuttings level required. Additional water will be needed, which will result in higher energy consumption.

To overcome this challenge, the technology developed by Norwegian-Group AS is based on replacing the water on cuttings by environmentally friendly chemicals with low enthalpy of vaporization which can act as a susceptor. The enthalpy of vaporization of water is for instance three times and five times higher than that of ethylene glycol and tri-ethylene glycol respectively (Keller, 2015). Therefore, the separation of water from cuttings, as a pre-treatment, is of high importance prior to susceptor addition and microwave processing.

1.3 Objectives of the Thesis

This thesis was carried out in order to elaborate potential pre-treatment methods of contaminated drill cuttings. The objectives of this thesis are: (1) to evaluate the efficiency of using existing drying technologies to pre-treat drill cuttings offshore and (2) to assess the feasibility of using MudSplit chemicals for drill cuttings dewatering and washing.

1.4 Thesis Outline

To achieve these objectives the following topics are presented and evaluated in this thesis:

- Chapter 1 introduces the background and the objectives of this thesis.
- Chapter 2 provides a literature review on drilling waste including the types, composition, bonding system present in drilling waste as well as their magnitude.
- Chapter 3 summarizes the existing regulations on drilling waste and the offshore treatment methods available.
- Chapter 4 gives an overview of the potential methods to pre-treat drill cuttings.
- Chapter 5 evaluates the feasibility of using convective thermal drying as pre-treatment methods. The discussion is presented at the end of the chapter.
- Chapter 6 describes the materials and methods used to conduct a laboratory testing of using chemical destabilization followed by mechanical separation as a potential pre-treatment method.
- Chapter 7 presents the results obtained from the laboratory testing as well as the corresponding discussion.
- Chapter 8 provides Conclusions for the experiments performed and Recommendations for further research.

Chapter 2: Drilling Wastes

Petroleum production activities involve the extraction of hazardous substances, such as oil and natural gas, from the subsurface environment leading to contaminated waste generation. According to Wojtanowicz (2015), the broadest classification of the generated waste includes primary waste and associated waste. This classification is based on their origin and volume. Drilling waste with low toxicity but very large volume constitutes primary waste. This category comprises drilling muds and drill cuttings. Drilling waste with high toxicity but small volume is called associated waste. It may include rig wash, service company wastes such as empty drums, drum rancid, spilled chemicals, workover, swabbing, unloading, completion fluids and spent acid (Wojtanowicz, 2015). This thesis will mainly focus on the potential pre-treatment methods of primary waste. Hence, in this work, the term drilling waste will be used to designate primary waste only.

2.1. Drilling Waste Characteristics

In order to provide efficient treatment of the drilling waste, it is important to know their components as well as their respective characteristics. These topics are discussed in this section.

2.1.1. Drilling Mud

Drilling fluid or mud is a mixture of solids and other constituents in suspension within a liquid base (Wojtanowicz, 2008). It is used to aid the drilling of a borehole for oil and natural gas exploration and production (Schlumberger). Drilling fluid plays several important functions during the drilling process. It lubricates and cools the drill bit promoting more effective drilling operation. A pressure exerted by the mud supports the sides of the hole, maintaining downhole hydrostatic pressure and preventing collapse (Harispure et al., 2004). Drilling fluid also removes the crushed formation rock or drill cuttings from the borehole to the surface (Pantet et al., 2006).

As aforementioned, mud is composed of different kinds of components mixed with a fluid base. The former consist of weighting agents to increase the density of the mud. Examples of weighting agents used in mud formulation are barium sulfate or barite and iron (III) oxide. Clays and polymers are also used to adjust the viscosity of the mud. Chemicals are used to increase its lubricity and several minor additives to control the mud properties. The fluid base determines the type of the drilling mud. There are three main classes of drilling fluid: water based mud (WBM), oil based mud (OBM) and synthetic based mud (SBM) (El-sayed and El-Naga, 2001).

2.1.1.1. Water Based Mud

In WBM, fresh water, seawater, brine, saturated brine, or formate brine constitute the continuous phase and suspending medium for the additives compounds. WBM can be further classified into non-dispersed and dispersed (IOGP, 2016). It is based on whether the mud contains chemical dispersants or not.

This type of mud is relatively inexpensive and its formulations are generally non-toxic to the marine environment. An example of WBM formulation is presented in Figure 2.1.

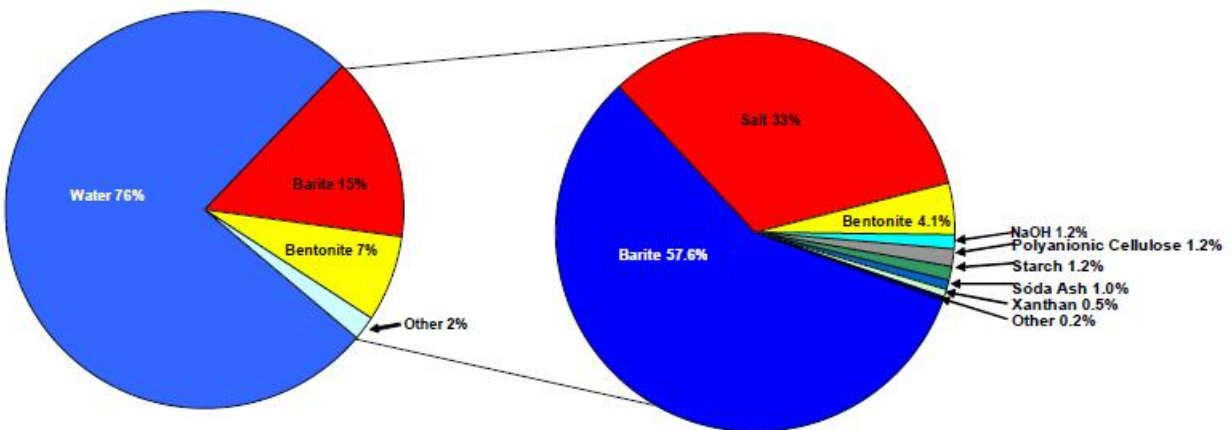


Figure 2.1. Water based mud composition (Neff, 2005)

2.1.1.2. Oil Based Mud

OBM is more expensive to use compared to WBM. It generally contains high level of toxic compounds such as Polycyclic Aromatic Hydrocarbons (PAH) and thus harmful to the environment. However, one or more of the following reasons justify its use (Lyons and Plisga, 2005; Doyle et al., 2008):

- Drilling deep and high-temperature well in which thermal stability of the mud is necessary.
- Drilling deviated wells which require specific lubricating characteristics of the mud.
- Drilling water-sensitive or soluble formations such as shales and salt. OBM reduces stuck-pipe and hole swelling or wash-out problems.

a. Composition of OBM

For OBM, the continuous phase consists of oil (Figure 2.2). This latter can be diesel or mineral oil (United States Environmental Protection Agency, EPA, 1993 as cited Doyle et al., 2008). OBM is such a WBM dispersed in oil and forming water-in-oil emulsion (Doyle et al., 2008). The emulsion is stabilized by surfactants, like emulsifiers and oil wetting agents (Wärnheim and Sjöblom, 1985; Neff, 2005). The small emulsified water droplets also contribute to the emulsion stabilization. These droplets can be as small as less than 1 μm in diameter (Hudgins, 1991; Norwegian Oil Industry Association Working Group, 1996 as cited in Neff, 2005). The water-in-oil emulsion provides viscosity to the mud system in addition to clay (Doyle et al., 2008).

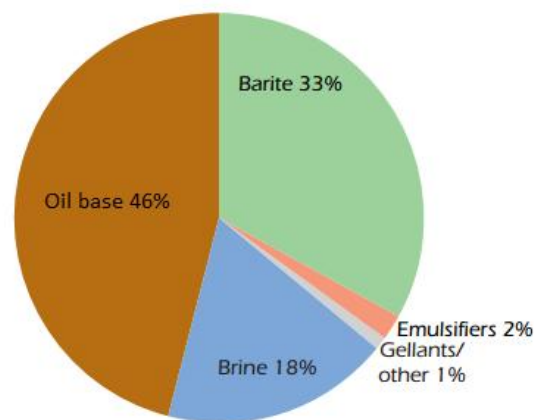


Figure 2.2: OBM composition (Melton et al., 2000 as cited in OGP, 2003)

b. Types of OBM

This class can be further divided into two groups. Group I consists of the first type of OBM used in drilling operation. The continuous phase of this group is constituted of either diesel or conventional mineral oil (CMO) which are both obtained from the distillation of crude oil. Diesel oil is produced without further specific treatment whereas CMO is manufactured by controlling the distillation process to reduce its total aromatic hydrocarbons (TAH) content. CMO was preferred over diesel oil because of the high potential toxicity of this latter. However, both diesel oil and CMO contain high aromatic content and a polycyclic aromatic hydrocarbons (PAH) greater than 0.35% (OGP, 2003).

The base oil of Group II OBM is made of low toxicity mineral oil (LTMO). As the two types of oil aforementioned, LTMO is also produced by distillation of crude oil. However the treatment process is controlled in order to make its TAH and PAH content less than that of diesel oil and CMO (Table 2.1). Several types or series of techniques are used for this purpose, namely, vacuum, solvent, acid or hydro-treatment (Agency for Toxic Substances and Disease Registry, 1999). LTMO is then a further development towards more environmentally friendly base fluid (OGP, 2003).

Together with CMO, LTMO is produced from the heavy-end of the crude oil distillates making them heavier than diesel oil. Their hydrocarbon numbers range from C15 to C50 (ABB Environmental, 1990 as cited in Agency for Toxic Substances and Disease Registry, 1999).

Table 2.1: TAH and PAH content of Diesel oil, CMO and LTMO (OGP, 2003)

Types of base oil	Diesel oil	CMO	LTMO
TAH content (%)	15-25*	About half the TAH content of diesel oil	0.5-5
PAH content (%)	2-4	1-2	0.001- 0.35

* Lee, 1980 as cited in Høiland et al. (1986).

2.1.1.3. Synthetic Based Mud

SBM contains petro-free synthetic organic compounds such as esters, ethers, acetyl or olefin as base fluids instead of water or oil (Neff, 2005; Hart et al., 2007). These compounds are produced by chemical reactions of relatively pure compounds (OGP, 2003). Mineral oils also can be considered as base fluid after special refining and/or separation processes (OGP, 2003). Comparable to OBM, SBM contains several additives and their general compositions are similar.

SBM is more environmentally benign while also able to perform the same tasks as OBM (Neff et al., 2000 as cited in OGP, 2003, Wotjanowicz, 2008). It has low toxicity, high biodegradability, and low bioaccumulation potential. The TAH and PAH content of SBM are less than 0.5% and 0.001% respectively (OGP, 2003). However SBM is in general characterized as OBM and subject to the same regulations as this latter. The environmental regulations of drilling waste will be discussed later in Chapter 3. Furthermore, SBM is more expensive than OBM and consequently its use is very occasional and decreased over the past years (Norwegian Oil and Gas Association, NOROG, 2015, 2016). Figure 2.3 gives an overview of mud consumption in the Norwegian Continental Shelf (NCS) from 2005 to 2015.

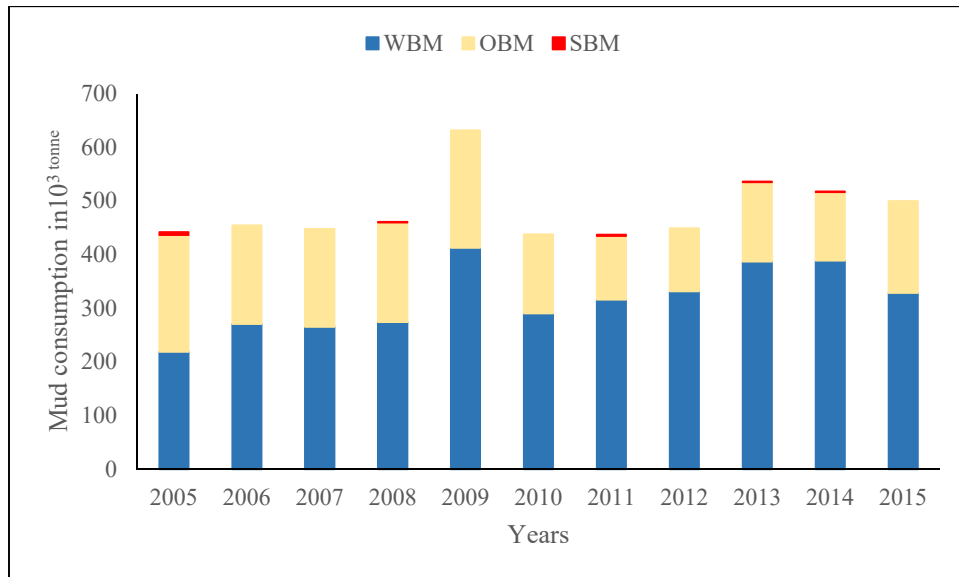


Figure 2.3: Mud consumption in the Norwegian Continental Shelf (NOROG, 2016)

2.1.2. Drill Cuttings

Drill cuttings are small pieces of formation rock generated during the drilling activity (Neff et al., 1987 as cited in Neff, 2005). Their size ranges from less than 2 µm for clay-like particles to more than 30 mm for coarse gravel. According to Neff (2005), drill cuttings have an angular shape.

Their chemical and mineralogical composition reflect the geological formation penetrated during the drilling process (Neff, 2005; Jacques Whitford Environment Limited, JWEL, 2009). Drill cuttings composition varies depending on the well location and depth.

Table 2.2 presents an example of the variation in drill cuttings composition with respect to the drilling location. Sandstone and shale are typical formation drilled from the North Sea (Gerrard et al., 1999 as cited in Neff, 2005) while clays predominate in Mid- and North-Atlantic. A study conducted by Westerlund et al. (Neff, 2005) showed that quartz (from sandstone) and barite (from the mud) are the two major minerals present in cuttings from the Beryl A and Ekofisk 2/4A platforms.

Table 2.2: Drill cuttings minerals vs. drilling area

Minerals		North Sea	Mid-Atlantic	North-Atlantic
	Montmorillonite		X	
	Chlorite		X	
Clays	Illite	S	X	X
	Kaolinite	S		X
Iron carbonate	Dolomite		S	
	Siderite		S	
Sandstone	Quartz	X	X	
Shale		X		
Iron sulfide	Pyrite	X		

X: major constituents. S: minerals present at smaller amount.

In Table 2.3, the variation of drill cuttings composition with respect to depth is illustrated. The example was taken from a study conducted by (Høiland et al., 1986) on drill cuttings samples from Statfjord wells in the North Sea.

Table 2.3: Drill cuttings minerals vs. true vertical depth (TVD)

	Sample A (1386 m TVD)	Sample B (1490 m TVD)
Smectite or Vermiculite (mixed layer)	(60 ± 7) % with traces of kaolinite	(18 ± 5) %
Kaolinite	-	Approximately 2 %
Barite and portlandite	(30 ± 7) %	(10 ± 5) %
Quartz and feldspar	(10 ± 5) %	(70 ± 10) %
Calcite, pyrite, haematite	traces	-

Based on the work of van Brackel (as cited in Mujumdar et al., 2006), drill cuttings can be classified into two main categories as presented in table 2.4.

Table 2.4: Categories of drill cuttings minerals

	Non-hygroscopic capillary-porous media	Hygroscopic-porous media
Pore space	Present	Present
Physically bound liquid	Negligible	Large amount
Shrinkage	No	Occurs in the initial stages of drying
Examples	Sandstone	Clay

The hygroscopic-porous media can be classified into two sub-categories: strictly hygroscopic media and hygroscopic capillary-porous or bidisperse media. The former comprises of micro-pores only while the latter presents both micro-pores and macro-pores. According to Chen et al., bidisperse porous media is represented as clusters of large particles formed by the agglomeration of small particles (Nield and Bejan, 2006).

The macro-pores are located between the clusters and the micro-pores within them. Clays are the main example of this type of solid. The structure of the solid wastes has a strong impact on the treatment efficiency.

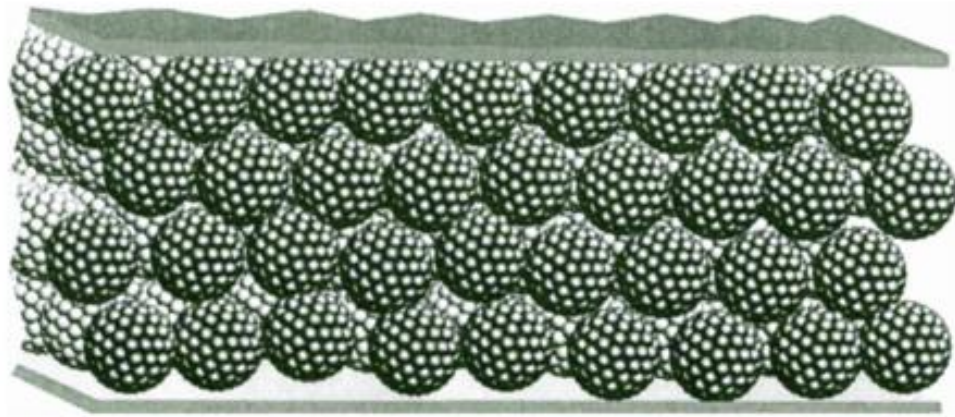


Figure 2.4: General structure of bidisperse porous media (Nield and Kuznetsov, 2005).

2.1.3. Contaminated Drill Cuttings

Drill cuttings alone are inert solids from the drilled formation. However they are contaminated by the drilling mud used to remove them up to the surface during the drilling operation. They may also contain crude oil and gas condensate from the formation such as the case for cuttings drilled from fossil fuel-bearing intervals (Neff, 2005). These cuttings can be either harmful or benign to the aquatic environment depending on the type of mud used. As discussed earlier, WBM cuttings are for instance non-toxic compared to SBM and OBM cuttings, and SBM cuttings are less toxic than OBM cuttings. The choice of mud depends on the conditions encountered during the drilling operation. Therefore, in a single well, more than one type of mud are used (Doyle et al., 2008).

The amount of mud coating the drill cuttings surface varies based on the type of mud used, the formation drilled and the cuttings particle size distribution (EPA, 1993 as cited in Doyle et al., 2008). The surfactants present in OBM for instance modify the surfaces of the drill cuttings and help the mud to be adsorbed on the drill cuttings and produce a stable system (Wärnheim and Sjöblom, 1985). Due to their structure, bidisperse-porous media such as clay can absorb and retain more mud than hard surface minerals like quartz (sandstone). Clay sized cuttings retain more mud than coarse gravel cuttings because the small size permits higher surface area available for the mud to adhere into.

These parameters determine also the efficiency of drill cuttings cleaning system used. Mineral oil based mud retention on cuttings is lower than that of diesel-oil based mud. Table 2.5 illustrates a comparison between the oil content on a dry weight basis (ROCdry) of diesel and mineral OBM cuttings after treatment with shale shaker. The data in table 2.5 was compiled by Wotjanowicz (2008) from Bennet (1983), Boyd et al. (1983), Høiland et al. (1986) and Cline et al. (1989) unless stated by asterisk.

Due to their structure and size, clay cuttings are difficult to separate from the mud compared to sandstone (Wiig 1984 as cited in Wärnheim and Sjöblom, 1985; Neff, 2005; Buddhadasa, 2008).

Table 2.5: ROCdry of cuttings discharged from shale shaker versus the type of mud

	Well number				
Drilling fluid	1	2	3	4	5*
Diesel OBM	20	13-16	9.8	10.8	16
Mineral OBM	7.9	10.3			5.3

* Bennet, 1983 as cited in Høiland et al. 1986.

As a general rule of thumb, drilling mud represents 5 to 25% of the waste total volume after solid control (primary treatment) (Ray, 1979 as cited in Pappworth and Caudle 2016; Neff, 2005). OBM consists of approximately 30% by volume of the drilling waste (Bilstad et al., 2013). Table 2.6 presents an example of the composition of OBM cuttings samples from the North Sea.

Table 2.6: Composition of OBM cuttings samples from the North Sea (Young et al., 1991)

Depth (m)	Mineralogy	Weight (%)			Ratio % by weight	
		Oil	Water	Solid	Oil/Solid	Water/Solid
1382	Shale	5.7	11.4	82.9	6.8	13.8
2352	Shale	9.6	13.9	76.5	12.6	18.2
2564	Chalk	17.5	6.5	76.0	23.0	8.6
2613	Limestone	16.0	4.7	79.3	20.2	6.0

2.1.4. Drilling Mud and Drill Cuttings Bonding System

The bonding nature of mud to the cuttings determines the choice and efficiency of a treatment used. As reported by Høiland et al. (1985) and Wotjanowicz (2008), oil on cuttings are bonded through the following mechanisms:

- Adhesive and capillary forces which represent 50 to 51% of the bonding system. Oil separated by centrifugal filtration.
- Weak adsorption consisting of 29 to 30%. Oil extracted with n-pentane.
- Strong adsorption representing 20%. Oil separated using thermal vaporization.

Oil and water which represent the major constituents in OBM are bonded into the cuttings solids as presented in Figure 2.5. The bonding system is important with respect to oil and water separation.

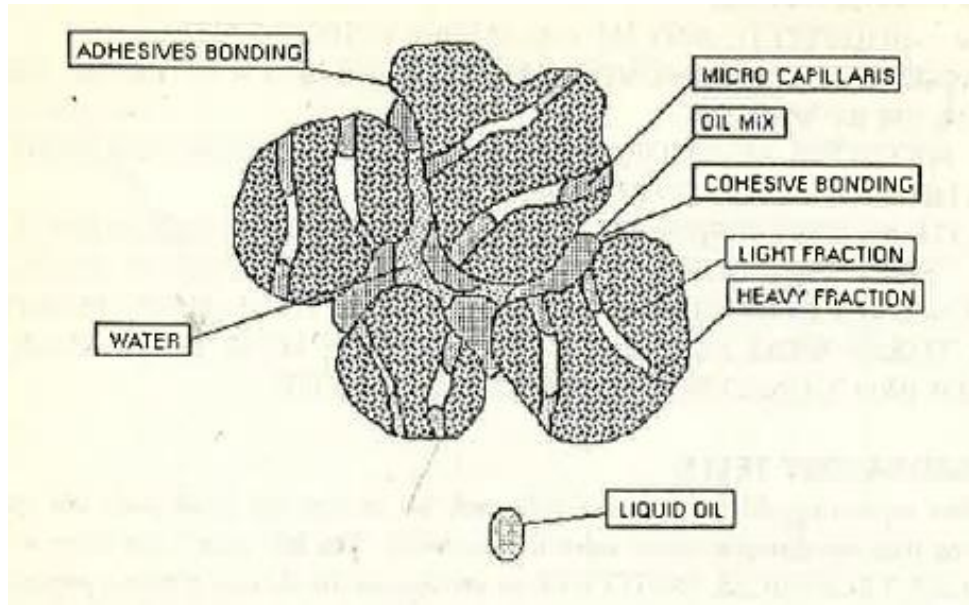


Figure 2.5: Bonding system in oil contaminated cuttings (Ellingsen, 1991).

For a formation rock made of water-saturated hygroscopic-porous minerals such as clays, the structure may differ and water may predominate. Based on the work of (Mok, 2006; Mujumdar, 2006; van't Land, 2012), water is present in clay solids with four different physical forms:

- Free water in solution with solid but not associated with the solid particles. It is also called unbound moisture and corresponds to the saturation humidity.
- Surface or vicinal water is chemically or physically adsorbed on the surface of the solid.
- Capillary or interstitial water is mechanically retained in the pores or interstices of the solids.
- Water of hydration is chemically bound or in solution in cellular structures.

These three last forms of water are called bound moisture. They exert a vapor pressure less than that of pure water and therefore require more energy to remove (Mujumdar, 2006). For non-hygroscopic media, all the moisture content is unbound.

2.2. Waste Generation

In general, drilling waste represents only few parts of the overall waste generated from the oil and gas operations, onshore and offshore. Produced water forms the majority of the waste (Ahnell and Evans, 2016; Pappworth and Caudle, 2016). Figure 2.6 illustrates the amount of drill cuttings generated on the NCS for the years 2005 to 2015 (NOROG, 2016).

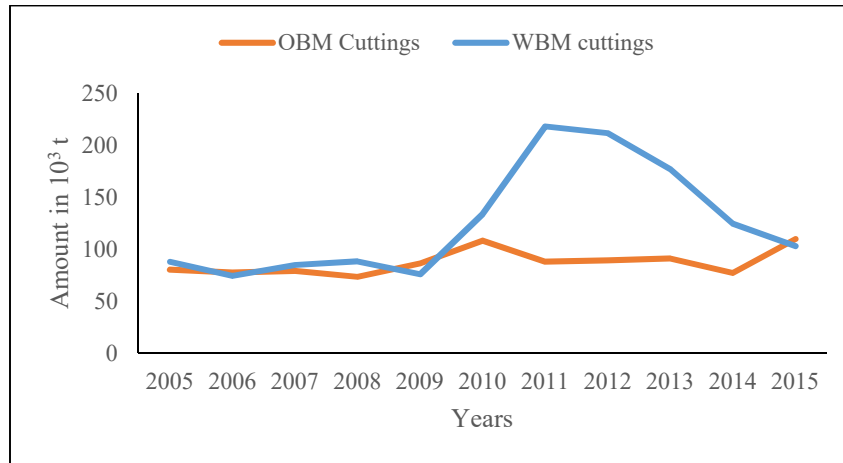


Figure 2.6: Cuttings generated from the NCS from 2005 to 2015

On a single well basis, the amount of drilling waste generated depends on the type of the drilled rock formation, the depth and the drilling fluid used. Since the borehole diameter decreases with depth (OGP, 2003; Paulsen et al., 2003), the higher quantities of waste are generated from the first few hundred meters of the well (OGP, 2003). Table 2.7 gives an estimated amount of drilling waste produced from an average well of 5169 m (Paulsen et al., 2003).

Table 2.7: Estimated amount of mud and cuttings waste generated from a 5169 m depth well.

Hole size (in)	36	26	17½	17 ½	12 ¼	8½	8½
Section length (m)	92	938	1288	1288	2458	393	393
Type of mud used			KCI P Glycol	OBM 2	OBM 1	Cesium formate	OBM 1
Mud waste (m³)	189	1175	771	231	319	79	40
Cuttings (tonne)	133	707	484	453	443	34	34

2.3. Waste Management and Handling

Once generated, the base fluid of the mud used determines the fate of the drilling waste. WBM contaminated cuttings can be discharged offshore whereas OBM and SBM cuttings are re-injected or shipped to shore. The discharge of OBM and SBM cuttings is restricted and subject to several requirements which vary from one country to another. These regulations are discussed in Chapter 3 along with the techniques used to comply with them.

2.3.1. Offshore Re-injection

The cuttings to be injected are grinded into smaller size particles and mixed with sea water to create a stable suspension. The produced slurry is either injected into a subsurface geological formation, into the annulus of the producing well, into a depleted well or into a dedicated or dual-use disposal well (OGP, 2003; JWEL, 2009). Many factors limit the possibility of this process:

- Absence of a suitable geological formation capable of accepting and containing the waste on a long-term basis.
- Logistical implications to handle storage and additional equipment.
- Loss of formation integrity (Bilstad et al., 2012). This latter leads to leaks and escape of the waste to sea bed. Fracturing and leaks from injection wells were discovered in old fields located on the NCS in 2007- 2009 (NOROG, 2016). Therefore this practice has decreased and resulting to the increase of shipment ashore (NOROG, 2015).

2.3.2. Skip and Ship-to-Shore

Skip and ship-to-shore consists of collecting and transporting the drill cuttings to shore for treatment or disposal. The cuttings are transferred into vessels using skippers. Another possibility is to slurry the cuttings before pumping them into the vessels and then deliver to shore (Det Norske Veritas, DNV, 2013; NOROG, 2015). This practice presents several limitations such as safety issues and use of huge amounts of energy for transport and processing. The use of large number of crane lifts is the main safety concern for operators (OGP, 2003; JWEL, 2009).

Furthermore, the operation is highly dependent on weather. Cuttings handling and transport pose logistical challenges because of the limited storage space available on offshore drilling rigs. Other issues such as air emissions, potential for spills in sensitive areas and onshore treatment, storage and disposal impacts are also involved in this method (JWEL, 2009). However, due to the high cost and all the challenges faced with re-injection cited earlier, the majority of drilling waste produced from the Norwegian offshore petroleum sector are shipped and treated ashore (NOROG, 2015).

Figure 2.7 gives an overview of the amount of OBM cuttings disposed of from the NCS with respect to the trends of disposal method used.

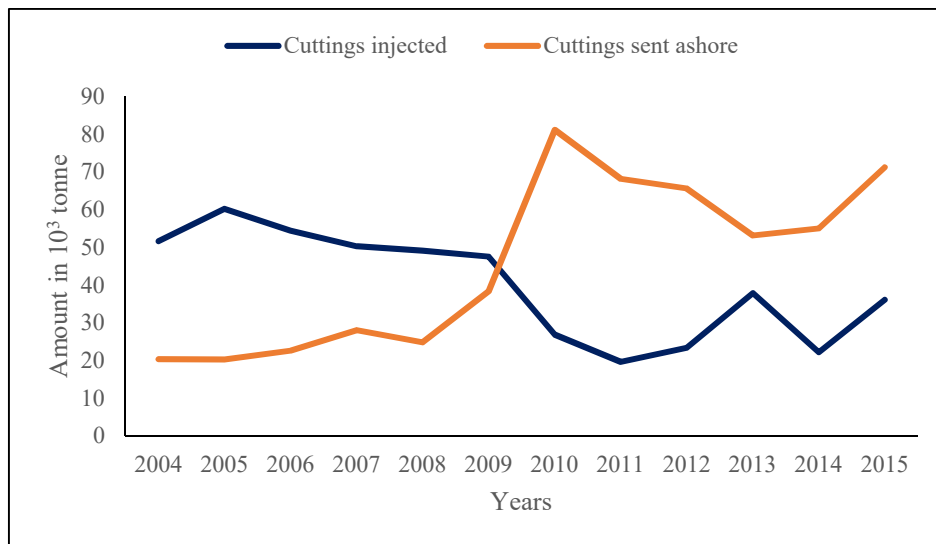


Figure 2.7. OBM cuttings sent to shore and re-injected on the NCS

2.3.3. Offshore Discharge

As stated earlier, this technique is primarily dedicated to WBM contaminated cuttings. WBM mud contains natural components and characterized as green chemicals or PLONOR (Pose Little Or No Risk) according to the Norwegian Environment Agency (NEA) and OSPAR Commission respectively (NOROG, 2015). OBM and SBM cuttings require further solids control and have to comply with the environmental regulations to be allowed for discharge.

After recovery of some part of the WBM, the spent mud and WBM cuttings are mixed with sea water and discharged through a pipe known as a “downcomer” into the local environment, at few

meters below the water surface (OGP, 2003). This technique is operationally simple and requires no temporary storage for cuttings nor additional equipment (OGP, 2003). When applicable, this method is the simplest and the most economical option. Figure 2.8 presents the amount of WBM cuttings discharged offshore on the NCS from 2005 to 2015.

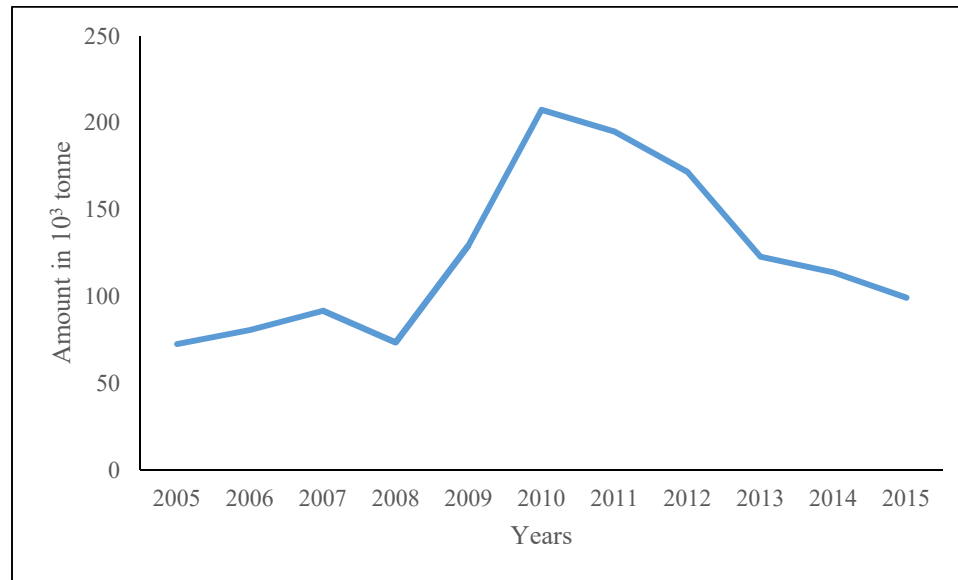


Figure 2.8: WBM cuttings discharged offshore on the NCS (NOROG, 2015)

An amount of 2 640 t of OBM cuttings was discharged offshore in 2015 due the adoption of Thermo-mechanical Cuttings Cleaner (TCC) technology on one platform on the NCS (NOROG, 2016). A brief description of this technology is given in Chapter 3.

2.4. Waste Handling Cost

The cost related to each type of the drilling waste disposal methods is highly variable and dependent on several parameters including the costs of drilling rigs, drilling time, drilling fluids cost and consumption, the costs of solids control equipment and transportation (OGP, 2003). A cost analysis conducted by OGP (2003) demonstrated that offshore discharge of drilling waste is the least expensive disposal option. This analysis is presented in Figure 2.9.

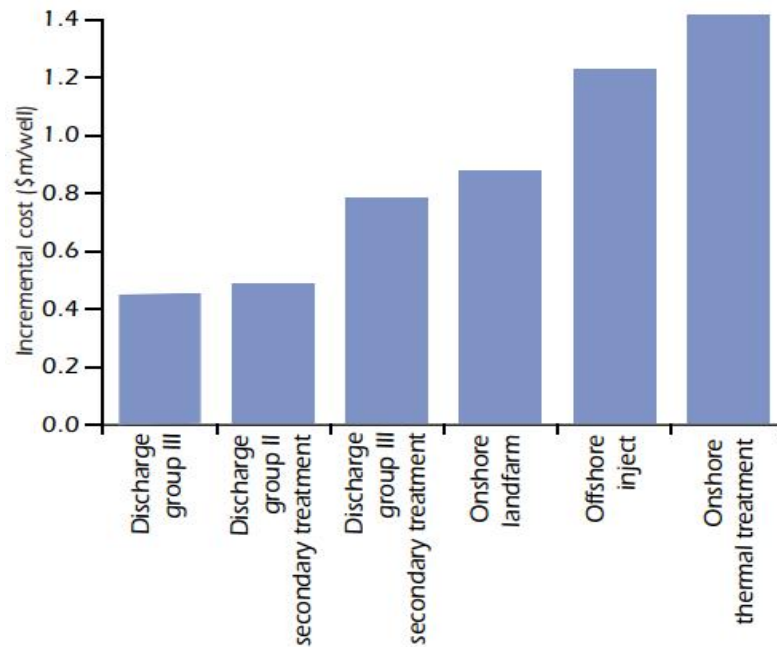


Figure 2.9: Waste handling options and associated costs (OGP, 2003)

In Figure 2.19, group II corresponds to the low toxicity mineral oil based mud and group III designates SBM. Example: discharge group III means discharge of SBM contaminated cuttings and costs \$450 000 (OGP, 2003).

For drilling and production activities in the Norwegian offshore petroleum sector, DNV (2013) found that offshore discharge of drilling waste is also the most economic option. A proper primary solid control followed by treatment of the drill cuttings with TCC cost approximately 6500 NOK per ton of cuttings. This value is comparable to the estimation made by Paulsen et al. (2003) for cuttings discharge using the same treatment method (TCC). According to DNV (2013), the costs per ton of cuttings for re-injection and transport and treatment onshore are respectively 9600 NOK and 9000 NOK.

Even though offshore treatment and discharge is the most economic option, this method is not yet well implemented in Norway (DNV, 2013). Stringent regulations oblige the operators to either re-inject or ship and treat the drilling waste onshore.

Chapter 3: Environmental Regulations and Drilling Waste Treatment Methods

In this chapter, the regulations concerning drilling waste and some of the treatment methods adopted to achieve the requirements in these environmental regulations are discussed.

3.1. Regulations for Drilling Waste Discharge

As argued in Chapter 2, offshore discharge is the least expensive disposal option for waste generated during drilling activities. However, due to the toxic nature of some drilling fluids used, the wastes may cause significant damage to the marine environment. Regulations were established in order to balance the economic development and the environmental protection. These regulations are developed through the work of regulatory authorities, industries and environmental groups (Doyle et al., 2008). According to the same authors, the development of regulations went through the following steps. The wastes are first identified. Then their volume, properties, potential impacts and the sensitivity of the receiving environment are assessed. When all these data are collected, control and monitoring strategies are determined and implemented.

The regulatory schemes for drilling waste discharge vary depending on the geological areas and countries. Some areas focus on potential toxicity of the inlet products to be used, the resulting waste is definitively discharged. But others use the “end of the pipe” system where the control is practiced on the volume and content of the outlet or effluent (Pappworth and Caudle, 2016). According to the same authors, there are three major regulatory systems used: the Russian and former Soviet Republic regulations, the United States regulations and the OSPAR agreements and national regulations in the OSPAR area. The other regional and national regulations are mainly based on the two last but with local modifications.

3.1.1. Russian and Former Soviet Republic Regulations

The regulations consist of a general prohibition discharge of effluents in the marine environment. But depending on the activities and the situations, certain materials, prohibited in the general regulations, can be allowed to be discharged under specific limits, after approval. The approval is gained if the materials pass toxicity and potential impact test, and the operator pays a compensation payment (Pappworth and Caudle, 2016).

3.1.2. United States regulations

For offshore drilling activities, these regulations are primarily developed by EPA and apply to all the United States waters. EPA together with numerous companies and industry associations identify and classify waste discharges and develop guidelines for permits issuing (IOGP, 2016). The main concerns in the environmental impacts are toxicity and oxygen depletion. Waters are divided into several categories and each category has its own specific limits of waste discharges (Pappworth and Caudle, 2016). For drilling waste discharges, WBM cuttings and excess WBM can be discharged if the toxicity limit is achieved (IOGP, 2016; Pappworth and Caudle, 2016). Discharge of OBM and OBM cuttings is prohibited. While the discharge of SBM themselves is banned, SBM cuttings can be discharged if the base fluid retention on cuttings is 6.9% for internal olefins and 9.4% for esters (IOGP, 2016). Furthermore, the trace amounts of cadmium and mercury in the barite used as weighting agent for mud should meet the limits imposed by EPA for WBM, WBM cuttings and SBM cuttings (IOGP, 2016; Pappworth and Caudle, 2016).

3.1.3. OSPAR Agreements and National Regulations in the OSPAR Area

OSPAR Commission is a treaty organization between the European Union and 15 countries bordering the North Sea, the Baltic Sea and the Northeast Atlantic including Norway. Negotiated in 1992, the OSPAR convention entered into force on 25 March 1998 (OSPAR Commission). It is a merger between the “Oslo Convention of 1972 for prevention of marine pollution by dumping from ships and aircraft” and the “Paris Convention of 1974 concerning prevention of land-based sources of marine pollution” (CAPP, 2001; OSPAR Commission, 2015).

According to the OSPAR Commission, the decisions, recommendations and agreements agreed under the OSCOM and PARCOM are still valid unless new measures adopted under the OSPAR Convention amend or terminate them.

3.1.3.1. OSPAR: An International Framework

OSPAR commission regulates international cooperation on the protection of the marine environment of the waters cited above (Pappworth and Caudle, 2016). The role of the OSPAR Commission is to identify issues, assess impacts and set goals for controlling pollution of the marine environment from several sources such as the oil and gas industry (Pappworth and Caudle, 2016). The member countries implement these goals through national regulations.

OSPAR commission uses both of the two approaches: control at source and control at “the end of the pipe”. But the primary emphasis is on the control at source approach (Pappworth and Caudle, 2016). Substances and preparations, to be used in offshore activities, are controlled by the authorities in order to avoid or minimize discharge of hazardous materials into the marine environment. For the end of the pipe approach, limits are set on individual waste discharge. Among other provisions, the OSPAR convention (Art. 2) obliges the contracting parties, while controlling or taking measures to prevent and eliminate pollution, to apply:

- The precautionary principle. Preventive measures are to be taken to prevent the potential impacts of the activities operated in the marine environment.
- “Polluter pays” principle. Polluters pay the costs of pollution prevention, control, and reduction measures.
- Use the best available techniques (BAT) and the best environmental practice (BEP).

3.1.3.2. Regulations on Drilling Waste

Chemicals, used in drilling fluid formulation, are regulated by the OSPAR Decision 2000/2 on a Harmonized Mandatory Control System (HMCS) for the Use and Reduction of the Discharge of Offshore Chemicals (as amended by Decision 2005/1). In this decision, minimization or substitution of hazardous substances by less or non-hazardous ones are encouraged. Regarding the base fluid, the use of diesel oil in mud formulation is prohibited since 1984 (Frost et al., 2006).

Mineral oil and then synthetic oil have been developed and are still allowed to be used after authorization by the competent national authority as stated in the OSPAR Decision 2000/3 on the use of organic-phase drilling fluids (OPF). The use of these OPFs (OBM and SBM) are also restricted to be only in the lower sections of the well.

For drilling waste discharge, the PARCOM Decision 92/2 on the use of oil-based muds banned the discharge of untreated OBM cuttings into the marine environment and resulted to the complete cessation of whole OBM discharge in 1996-1997 (Garland, 2005; Frost et al., 2006). After the OSPAR Decision 2000/3, OBM cuttings are prohibited for discharge unless the oil content is less than 1% by weight on dry cuttings. SBM and SBM cuttings are also subject to the same regulations. The discharge of whole SBM is prohibited and SBM cuttings can be discharged if only ROCdry is less than 1%. Here whole OBM or whole SBM designates drilling fluid not adhering to or mixed with cuttings (i.e. excess mud). However, in exceptional circumstances, SBM cuttings can be discharged after authorization based on the application of BAT and BEP. WBM and WBM cuttings can be discharged as long as the oil content is less than 1% by dry weight (JWEL, 2009).

3.1.3.3. Norway

The oil exploration and production in Norway are practiced along the Norwegian Continental Shelf (NCS), which includes the North Sea, the Norwegian Sea and a portion of the Barents Sea (IOGP, 2016). Disposal of waste, produced from these activities, is regulated by the Norwegian Pollution Control Act (Svensen et al., 2014). This latter is managed by NEA which regulates the waste discharge through issue of permits (Wills, 2000). As a contracting party in the OSPAR Commission, regulations in Norway are highly influenced by the OSPAR convention but the requirements are more stringent and the enforcement more rigorous than that of the other members (CAPP, 2001). However, there is also considerable reliance on self-regulation and self-reporting by the operators (Wills, 2000).

Drilling fluids formulations are controlled by the OSPAR decision 2000/2. WBM are permitted to be used and discharged if drilling fluid chemicals are approved after toxicity testing according to OSPAR protocols. The testing consists of bio-accumulation potential and bio-degradability of the chemicals (Wills, 2000).

Whole OPFs (i.e. OBM and SBM) are banned to be discharged offshore and therefore injected or shipped to shore for treatment (Frost et al., 2006).

Since 1993, OBM cuttings discharge is prohibited if the cuttings contain more than 1% oil on a dry weight basis. According to the Norwegian Petroleum Directorate (NPD) SBM cuttings were subject to the same requirement from 1995. Currently, cuttings contaminated by WBM, OBM and SBM are prohibited from discharge unless the ROCdry < 1%. According to NOROG (2010), Norway has adopted a zero discharge policy on the Norwegian section of the Barents Sea. Drill cuttings are re-injected after permit has been acquired or sent to shore if the onshore disposal facility disposes a license to receive the waste (Svensen et al., 2014).

3.2. Drilling Waste Treatment

In order to reduce the overall well cost and comply with the environmental regulations discussed earlier, several technologies are used to treat drilling waste. The treatment aims primarily at separating the individual components of the waste, such as oil and water from solids, to get the desired properties of the final product. This process can be achieved by means of a series of methods. Drill cuttings are subject to primary treatment to recover the drilling fluid. The recovered drilling mud is re-used and the remaining cuttings are transferred to secondary treatment prior to offshore discharge, re-injection or transport and treatment ashore (OGP, 2003). The second phase of the treatment is then dedicated to lowering potential hazards associated with the waste. This is done by reducing its toxicity and minimizing its volume to render it suitable for a particular disposal option. Figure 3.1 summarizes the treatment processes and disposal options of mud contaminated cuttings.

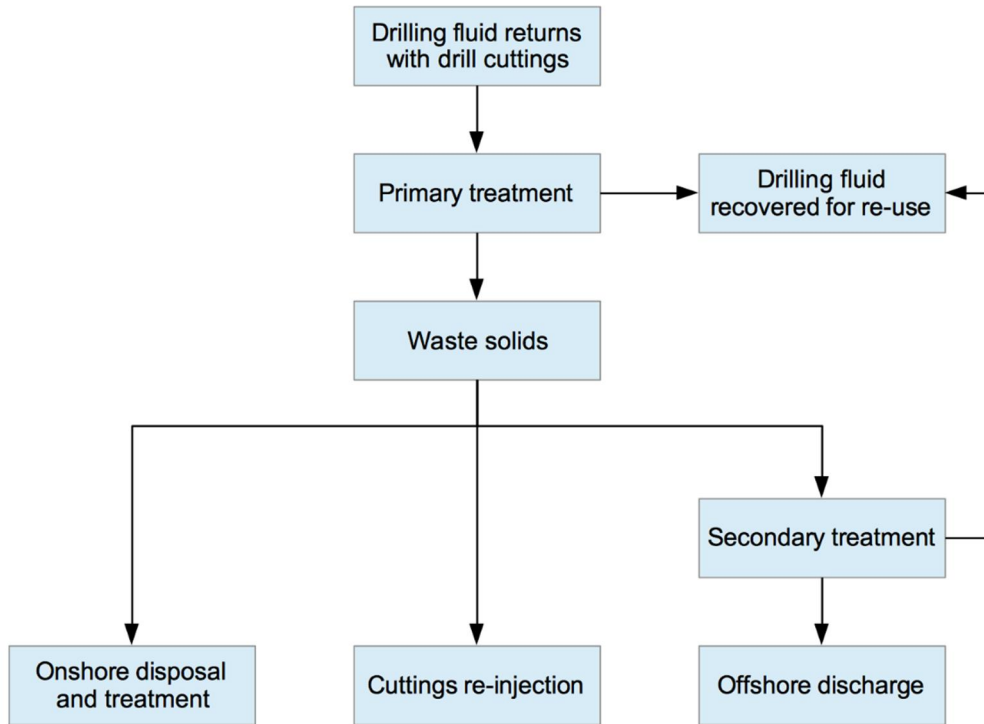


Figure 3.1: Treatment processes and disposal options of contaminated drill cuttings (OGP, 2003)

3.2.1. Treatment Methods for Mud Recovery

The aim of this step is to reduce mud consumption and waste production by recovering and recycling the drilling mud recovered (Pantet et al., 2006). There is no defined specific treatment process for solids-control applications. The selection of components to be used depends on the needs on the drilling site (JWEL, 2009). Commonly used primary solids-control equipment include shale shakers, hydrocyclones and centrifuges.

3.2.1.1. Shale Shakers

Shale shakers are the primary devices used for solids control (Geehan and McKee, 1989; OGP, 2003). They are made of a series of vibrating screens that sieve coarse particles. The size of the screen openings ranges from #200 to #10 and the finest (#200) can discard particles larger than 75 μm in diameter. The drilling fluids carrying drill cuttings from the well are directed to the shale shakers.

As shown in Figure 3.2, liquid mud and small particles pass through the holes of the screen to the next step of mud recovery system while coarser particles are collected for secondary treatment or disposed of as a final waste (JWEL, 2009). The efficiency of shale shakers to remove drill cuttings varies depending on the rock formation, the size of cuttings particle, the formulation of mud and other factors (Neff, 2005). According to Hou and Luo (1986) (as cited in Wojtanowicz, 2008), typical oil retention on cuttings discarded from shale shakers ranges from 11.1 to 16.5%.

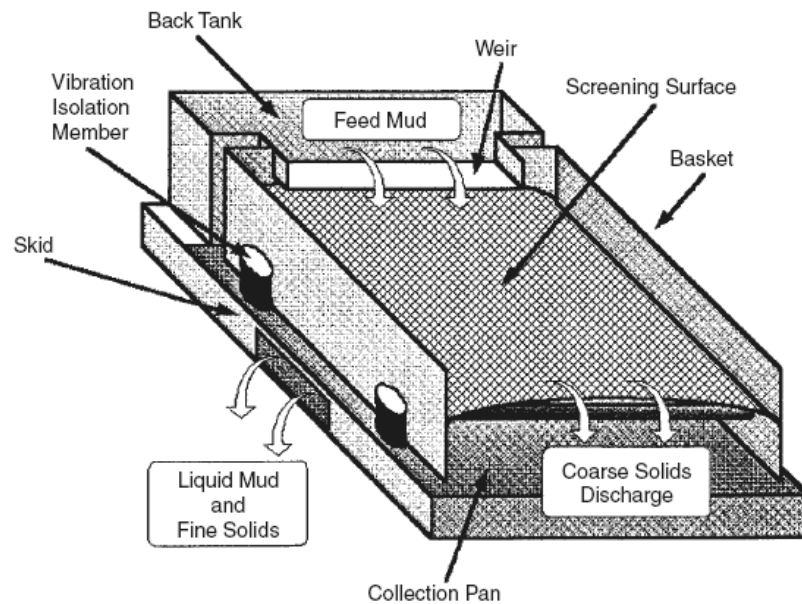


Figure 3.2: Shale shaker (Engineering 360)

3.2.1.2. Hydrocyclones

Hydrocyclones are used to remove fine solid particles in the drilling fluid recovered from shale shakers (JWEL, 2009). They are composed of a cylindrical top section and an inverted conical base which ranges from 2” to 12” in diameter (CAPP, 2001; JWEL, 2009). The mud is pumped tangentially at high speed at the top section of the hydrocyclone to generate a rotational fluid motion and produce high centrifugal force. The denser materials are discarded by gravity through the conical base on the bottom, while liquid and less dense particles are recovered from the top of the hydrocyclone.

According to Geehan and McKee (1989) and JWEL (2009), hydrocyclones are used as:

- Desander made of one or two units of large-diameter (generally larger than 5”) hydrocyclones working in tandem and used to remove sand-size particles (Figure 3.3).
- Desilter with several units of 4” hydrocyclones, used to remove silt-size particles.
- Mud cleaner composed of eight to ten 4” hydrocyclones arranged above a container which collects the small particles and sieves them through a very fine screen.

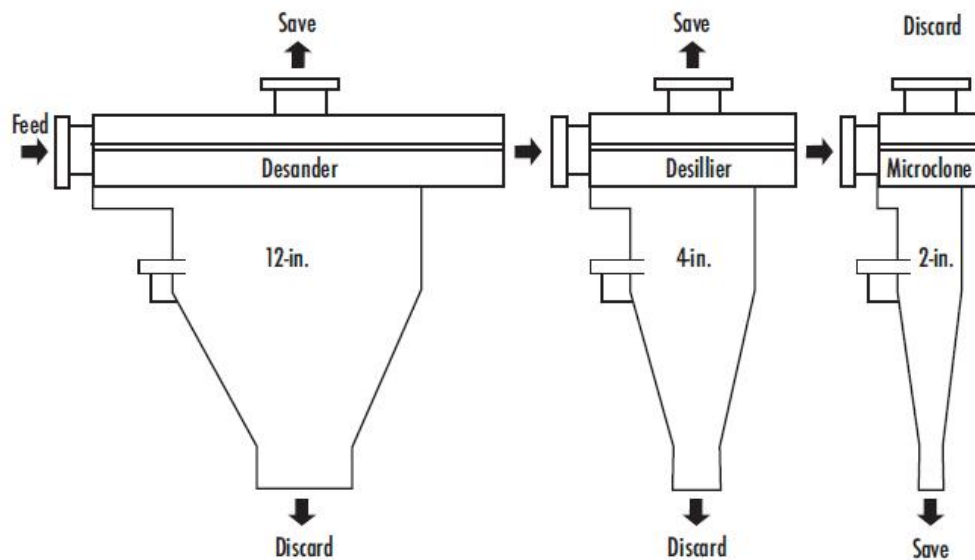


Figure 3.3: Different types of hydrocyclones (CAPP, 2001)

3.2.1.3. Decanter Centrifuges

Centrifuges are used to further process drilling fluids after treatment with hydrocyclones and prior to recycling (JWEL, 2009). They comprise an Archimedes’ screw that rotates slowly inside a conical tube, which itself rotates at high speed in the same direction as the Archimedes’ screw (Geehan and McKee, 1989). As illustrated in Figure 3.4, the mud is introduced in the middle of the screw and centrifugal forces separate solids from liquids.

Lighter materials such as colloidal particles, which could not be discarded by hydrocyclones, are separated using centrifuges. These particles create high undesirable viscosity to the mud (Geehan and McKee, 1989).

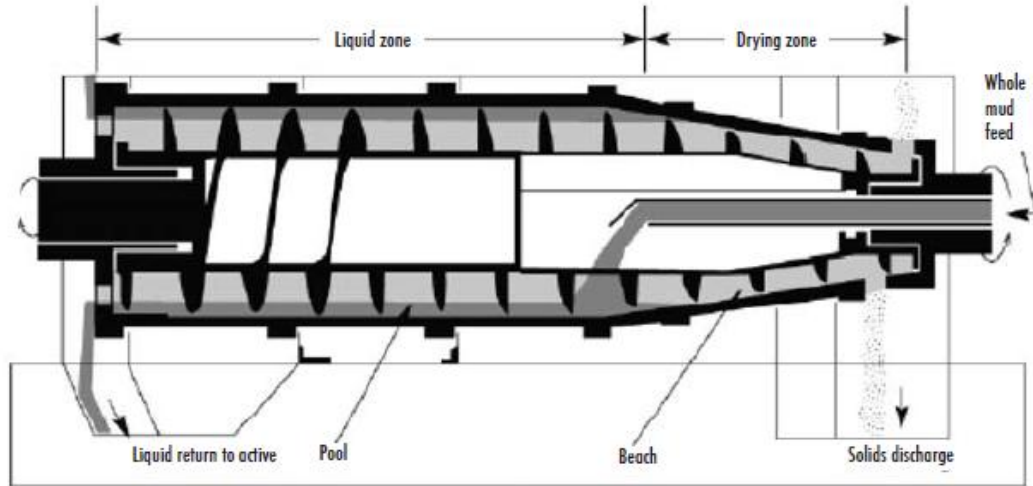


Figure 3.4: Cross-section of a decanter centrifuge (CAPP, 2001)

3.2.2. Treatment Methods to Reach Legislation Requirements for Offshore Discharge

In this section, the methods used for secondary treatment for offshore application are briefly revised. As discussed in Chapter 2, offshore discharge is the simplest and economic option to dispose drill cuttings. However, due to potential environmental impacts of drilling waste and the resulting strict regulations, several methods have to be adopted to further clean the cuttings from solids control equipment before discharge. Currently, two methods are available for this purpose, namely, cuttings dryers and TCC (MI-SWACO, 2008 as cited in JWEL, 2009).

a. Cuttings Dryers

A cuttings dryer consists of a fine mesh screen installed on a rotating basket. The rotation creates centrifugal forces used to separate drilling mud from drill cuttings (JWEL, 2009). The design of cuttings dryer was adapted from the coal industry where it is commonly used to dewater slurries of coal (CAPP, 2001; Lunde, 2014). Use of cuttings dryers to treat SBM cuttings is a common practice in the Gulf of Mexico (JWEL, 2009). However, they are not able to achieve the 1% ROCdry required by OSPAR decision 2000/3. A ROC reduction from 11.47% to 3.99 % and 11.8% to 2.1% was reported by Cannon and Martin (2001) and Melton et al. (2004) respectively (as cited in JWEL, 2009).

Johnston et al. (2004) as cited in JWEL (2009) found an average ROC of 4.39% from 72 wells drilled with SBM in the Gulf of Mexico.

Figure 3.5 illustrates a primary treatment system followed by cuttings dryer as a secondary treatment unit.

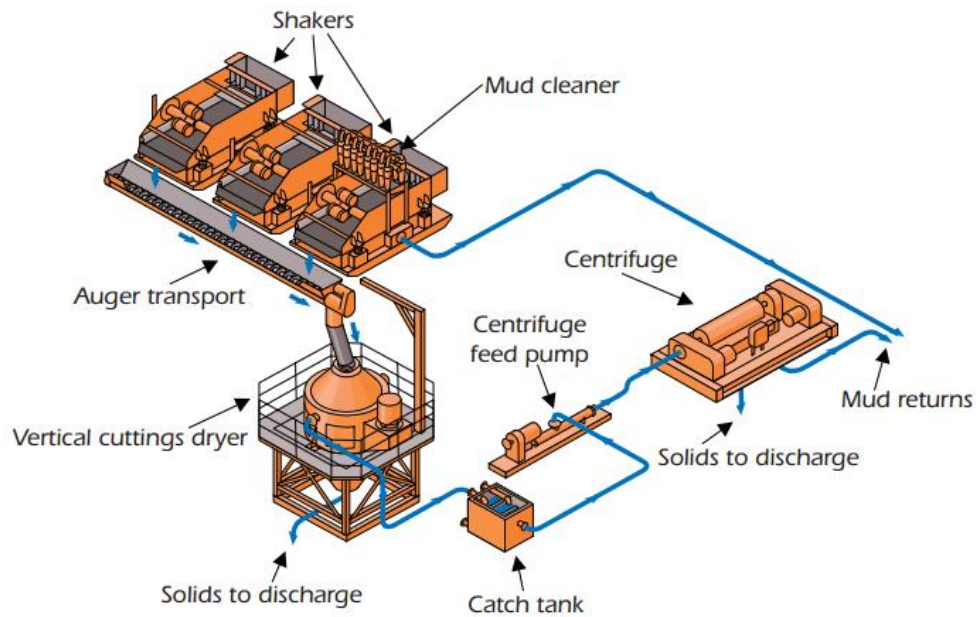


Figure 3.5: Offshore treatment of SBM cuttings with vertical cuttings dryer (OGP, 2003)

b. Thermal Desorption Treatment

TCC or hammermill system is a thermal desorption treatment method based on distilling water and oil with friction heat as a heat source (Murray et al., 2008; JWEL, 2009; Ormeloh, 2014). The heat is generated by friction between the cuttings materials and a series of hammer arms rotating within a process chamber as presented in Figure 3.6 (JWEL, 2009). The evaporated gases (oil and steam) are condensed through an oil and water condenser respectively and then recovered (Murray et al., 2008). The crushed dry cuttings are cooled down and rehydrated to eliminate dust contamination and facilitate cuttings handling (Halliburton).

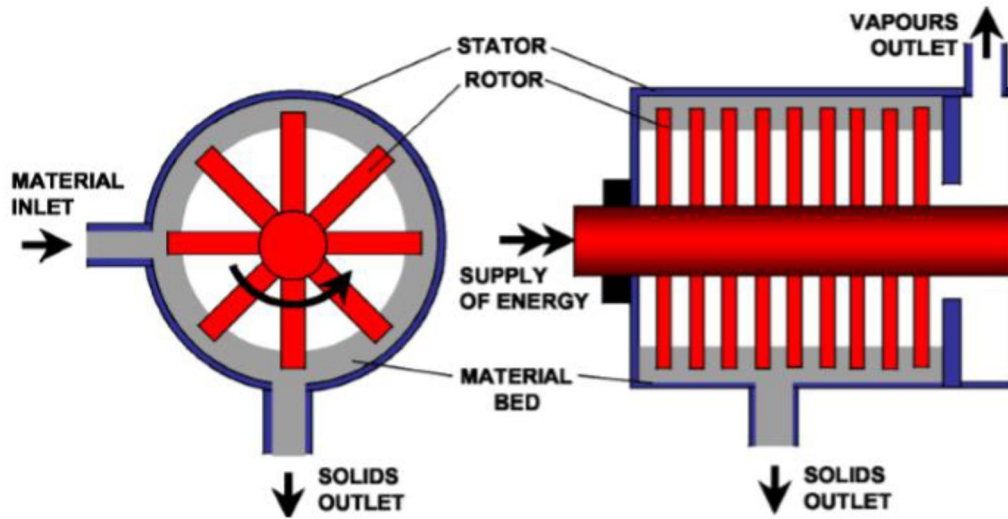


Figure 3.6: TCC working principle (Murray et al., 2008)

According to JWEL (2009), an offshore use of this system was successful and resulted in a ROCdry of less than 0.1%. Furthermore, it has the advantages of operating at smaller space, lower temperature and shorter retention time compared to traditional thermal treatments (Murray et al., 2008). According to Aquateam COWI (2014), TCC has a process temperature ranging from 250°C to 300°C. Therefore, the recovered oil is not degraded by the heat and has similar composition as the initial contaminating oil base and can be re-used in the mud system (JWEL, 2009).

Figure 3.7 illustrates the similarity between the oil in feed and the recovered oil after TCC treatment (Bilstad et al., 2014).

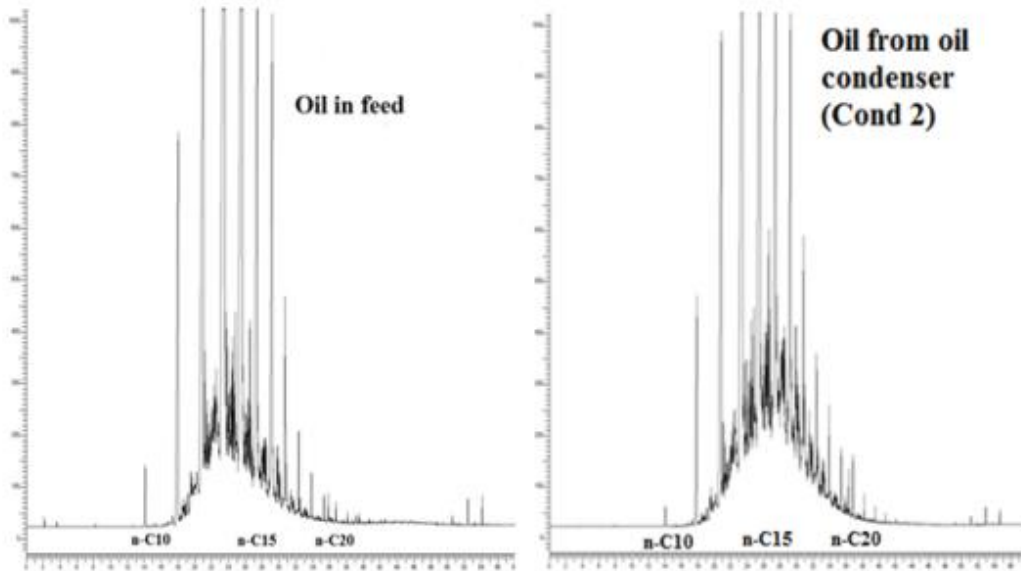


Figure 3.7. Virgin oil base versus oil recovered from TCC for reuse (Bilstad et al., 2014).

For Norway, TCC was recently approved by the NEA for offshore use but under stricter regulations. If the OSPAR decision 2000/3 stated a requirement of $ROC_{dry} < 1\%$, NEA specified an oil content of less than 0.05% for TCC treated cuttings to be discharged offshore (NOROG, 2016). Trials conducted on one offshore platform on the NCS in 2015, however, resulted in an average ROC_{dry} of 0.38% which exceeded the 0.05% requirement.

Chapter 4: Evaluation of Potential Methods for Pre-treatment of Drill cuttings

Since this thesis aims primarily to elaborate a suitable method to reduce water content of drill cuttings, this chapter is dedicated to evaluating some methods potential for this purpose. The ability of the method to reduce ROC is considered as an advantage.

Depending on the final product desired, several techniques can be used to remove water from drill cuttings. These techniques can be classified into two main groups: dewatering and thermal drying (Perazzini et al., 2016). As illustrated in Figure 4.1, dewatering or dehydration consists of separating the liquid and solid phases by means of mechanical expression, application of electric field or use of chemicals. This technique provides low water separation rate because it can generally remove free water and vicinal water for the case of electro-dewatering (Mok, 2006). Mechanical and electro-dewatering are discussed in Section 4.1 and thermal drying is reviewed in Section 4.2.

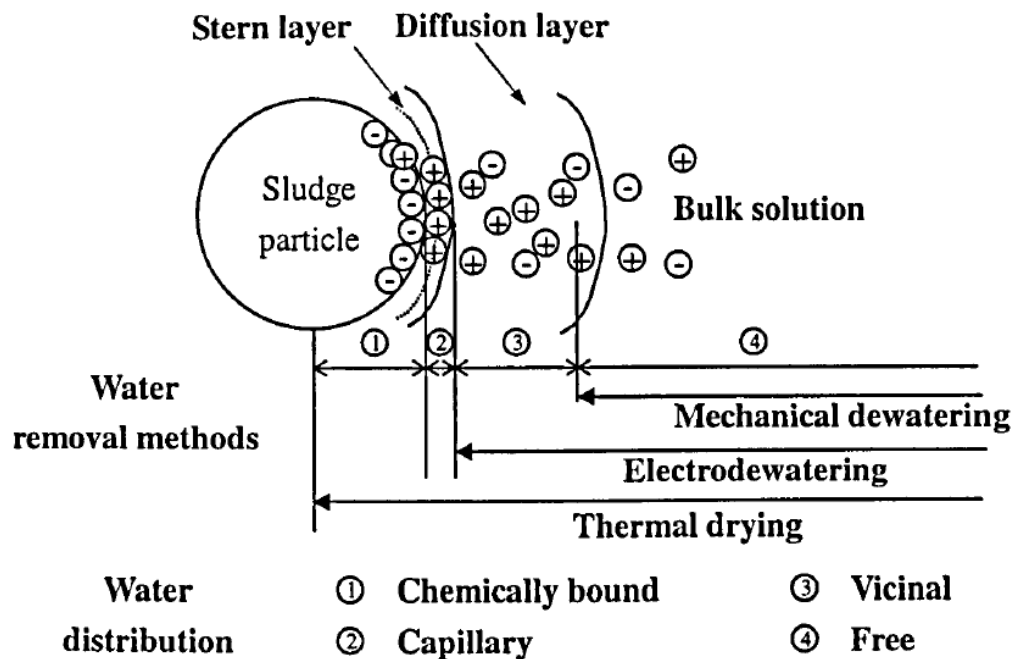


Figure 4.1. Water distribution in sludge and corresponding dewatering methods (Zhou et al., 2001 as cited in Mok, 2006)

4.1. Mechanical and Electro-Dewatering

The feasibility of using electric field and mechanical separation equipment such as centrifuges and cuttings dryer to separate water and oil from drill cuttings is discussed in this section.

4.1.1. Electro-Dewatering

Electrokinetic dewatering consists of two phenomena: electroosmosis and electrophoresis. Electroosmosis is the passage of water across a porous media induced by an applied electric field. When solids particles are entrained by an electric field within a liquid phase, it is called Electrophoresis (Raats et al., 2002; Iwata et al., 2013). Solid particles such as clay (Lajos, 2008) and sludge particles (Smollen and Kafaar, 1994 as cited in Mok, 2006) are negatively charged and surrounded by cations from the bulk solution to balance the charge. Under the influence of an electric field, the cations are attracted by the cathode and solid particles migrate towards the anode. While moving, the cations drag water into the cathode. Figure 4.2 presents the general principle of electro-dewatering.

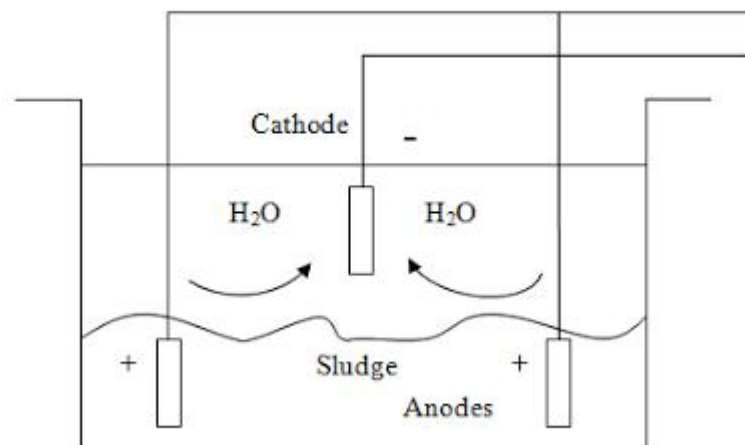


Figure 4.2. Electro-dewatering principle (Wastewater System)

Researches and laboratory studies have shown that this technique can be used to dewater a wide range of solid waste. This includes railway cuttings (Casagrande, 1952 as cited in Mok, 2006), sewage sludge (van Diemen et al., 1989 as cited in Iwata, 2013; Raats et al., 2002; Mok, 2006), pig manure (van Diemen et al., 1989), cement slurries (Kuin, 1985 as cited in Raats et al., 2002) and other fine particulate materials (Mok, 2006). One of the advantage of this technique is that the water flow rate is independent to the pore diameter or the hydraulic conductivity of the solids (Mok, 2006). It is therefore effective even for small size solids particles (Mitchell, 1991 as cited in Mok, 2006).

This method is capable of reducing sludge water content at low energy consumption. Alone, it can reduce water content up to 10-20%. If combined with mechanical filtration such as filter belt press, the performance is improved. Addition of cationic coagulant is also another option to improve its efficiency (Wastewater System). According to Raats et al. (2002), an energy consumption of 60 kWh per ton dry solids is required to treat sludge from a drinking water company in the Netherlands with a production of 4 m³ per hour. The same authors insist that the energy efficiency increases when the throughput is increased. The performance of this dewatering technique is, however, limited by the sludge conductivity, the strength of electric field and the zeta potential (Mok, 2006). Although electrokinetic dewatering is proven to be more efficient than the other conventional methods such as centrifuges or vacuum filters, this technology has not been successfully applied in industry or in large scale yet. According to Mok (2006), one of the reason is the lack of scientifically robust design methodology.

4.1.2. Mechanical Dewatering

Decanter centrifuges and cuttings dryers are also considered to be potential pre-treatment method. These methods are briefly discussed in Chapter 3. Work by Lunde (2014) indicated that if steam is provided into a cuttings dryer, its performance in separating oil and water is enhanced. However, experiments conducted by Norwegian-Group AS on clay cuttings resulted in ineffectiveness of cuttings dryer to treat these materials. Therefore, this method may be potential for other types of cuttings materials such as sandstone but not clays.

4.2. Thermal Drying

Thermal drying consists of vaporizing the moisture content of a product. This technique involves a heating source. When the heat is transferred into the wet solid, a temperature gradient develops within the inner part of the solid while the surface water gradually evaporates. This mechanism causes the internal moisture to migrate towards the surface and evaporates. The migration can be through diffusion, capillary flow or internal pressures (Mujumdar, 2006). Thermal drying is able to evaporate all forms of water even the chemically bound ones (Mok, 2006). It allows high or complete separation of water. However, this technique consumes 100 to 1000 times more energy than dewatering methods (Kudra and Hashmi, 2010). High energy is required to vaporize water (latent heat of vaporization). Thermal drying requires high energy to vaporize (latent heat of vaporization) water:

- For free water alone, 0.69 kWh/kg is required at 0°C.
- For chemically bound liquid (water or oil), which has low vapor pressure than pure liquid, additional energy equivalent to the bonding energy is required.
- For capillary trapped oil with boiling point of 300°C, a drying temperature of 560°C is required to create a gas pressure which is able to release the oil from the capillaries (Ellingsen, 1991).

Thermal drying comprises several methods. Some are operating in batch process and others in continuous. Different heating sources can be used such as dielectric heating, conductive heating and convective heating.

4.2.1. Dielectric Heating

In Dielectric drying, electromagnetic fields are used as heating source. Microwave drying is one of the major types of this drying method (van't Land, 2012). Dielectric heating has the unique ability to generate heat within the product. Microwave drying is the main subject in other thesis conducted in parallel with this thesis. Therefore, less focus will be given to this method.

4.2.2. Conductive Heating

The principle of conductive drying is to supply the heat required for drying into the surfaces of a drying chamber which is in contact with the materials. The drying media and the products are not in direct contact. According to the Mujumdar (2006), the evaporated moisture can be removed from the dryer by vacuum operation or stream of gas. For conductive drying, the enthalpy loss is very low resulting in high thermal efficiency (Mujumdar, 2006). However the drying process is very slow and takes up to several hours (van't Land, 2012). Typical examples of convective dryers are paddle dryers, indirect-heat rotary dryers and drum dryers.

4.2.3. Convective Heating

In convective drying, the wet materials and the heating media are in direct contact. The moisture content of the materials is separated by means of distillation due to heat transferred into the material surface. Typical examples of convective dryers are flash dryer, spray dryer and direct-heat rotary dryer (van't Land, 2012). The drying media consist of air, inert gas (N_2 for instance), superheated steam or solvent vapor (Mujumdar, 2006). According to van't Land et al. (2012), the drying gases and the wet materials can be fed into the dryer in four different flow modes depending on type of the dryer used: cross-flow, countercurrent flow, concurrent or parallel flow and mixed flow (mixture of countercurrent and concurrent and cross-flow). Cross-flow is mostly used in fluidized-bed dryers while parallel flow is used in flash dryers. Spray dryers and rotary dryers can be used under countercurrent flow, concurrent flow and mixed flow. Figure 4.3 illustrates the different flow modes used in a spray dryer (van't Land, 2012).

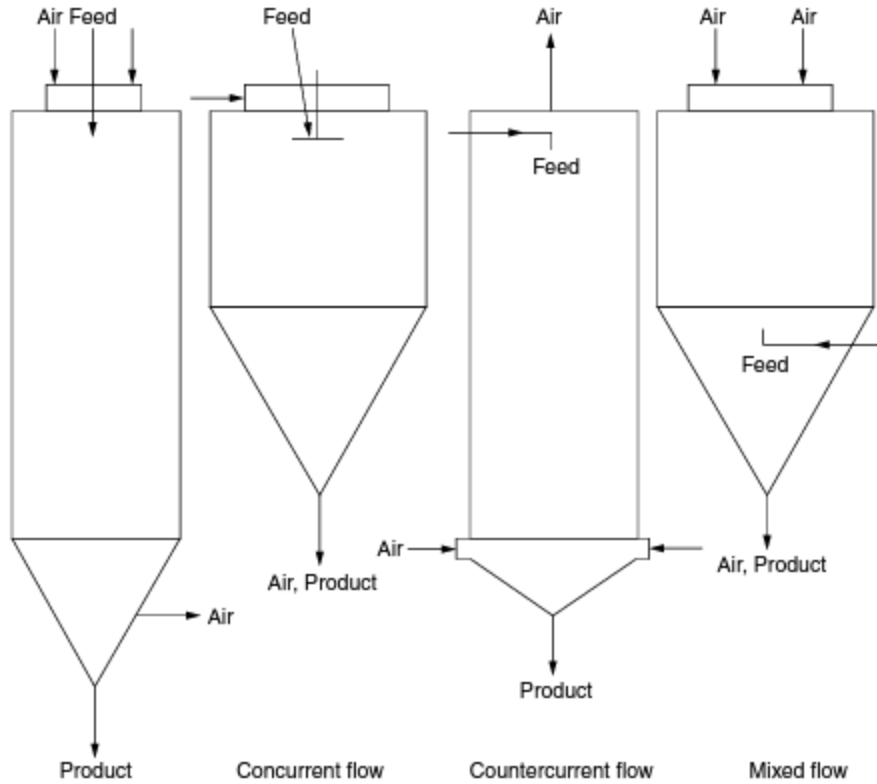


Figure 4.3: Air flow modes.

4.2.4. Choice of the method

Techniques based on thermal drying were chosen to be potential pre-treatment of drill cuttings in this thesis. Not only they can provide higher water (and oil) separation but may also satisfy most of the criteria for offshore use. According to Perez-Cordova (2010), these criteria are robustness and reliability of the equipment and processes, low space requirement, high capacity (process speed able to afford cuttings generation speed), low operating costs, small footprint.

van't Land (2012) stipulates that a continuous dryer is beneficial for a production capacity exceeding 100 kg/h, which is the case in this study. The same author suggested a decision tree for selection of suitable dryer type depending on the material to be dried and the desired final product (Appendix A1). According to (Perazzini et al., 2016), the choice of a drying method relies also on the ability of the method to provide better contact and degree of mixing between the solid surface and the gas phase. Therefore, the chosen methods will be based on convective heating. Compared to conductive heating, the areas and coefficients of heat transfer are much larger (van't Land, 2012). It ensures better contact between the gas and the product (Figure 4.4). Hence the methods chosen to be potential for cuttings pretreatment are based on convective heat transfer and operating in continuous process.

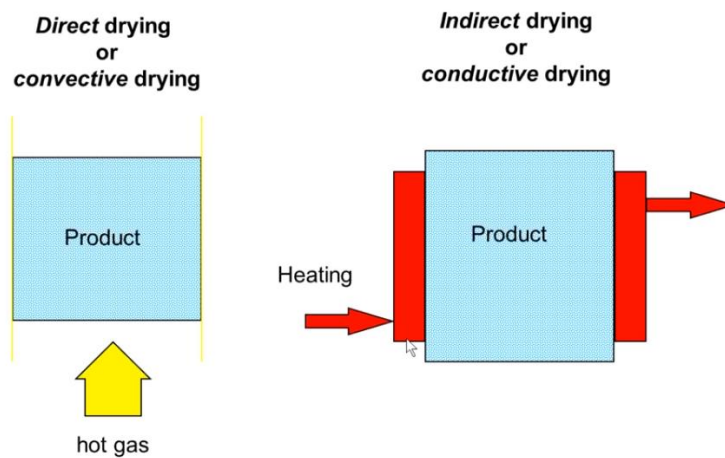


Figure 4.4. Convective vs. conductive drying mechanisms (Hosokawa Micron B. V.)

Chapter 5: Convective heating

Based on van't Land's decision tree in Appendix A1, flash dryer and fluidized-bed dryer were considered to be the potential methods for treating clay cuttings which are the most challenging to treat. These methods were chosen over the other types of convective dryers such as rotary dryer and spray dryer for the following reasons:

- suitability to treat clay-size materials
- short residence time
- robustness and simplicity of the equipment

Table 5.1 gives an overview of some convective dryers commonly used in industrial drying.

5.1. Drying Parameters

Some important parameters for convective drying are presented in this section. Section 5.1.1 deals with the types of drying media that can be used in flash and fluidized-bed dryers. The heating systems are discussed in section 5.1.2 and the design of product feeder in section 5.1.3.

5.1.1. Drying Media

Several types of heating media can be used in a convective dryer. Examples are air, combustion gases (natural gas for instance), superheated steam (SHS) or inert gas (Mujumdar, 2006; Febo, 2015). Since the drill cuttings to be treated contain hydrocarbons, presence of oxygen in the air makes this latter inappropriate for flash or fluidized-bed drying of drill cuttings. At high temperature, the oxygen causes oil cracking which may result to the formation of undesirable or carcinogenic compounds (Piper et al., 2005) and the oil recovered will not be suitable for re-use. Moreover, there is possibility of fire or explosion of the vapor or dust in the air. Therefore air is not suitable for use. Inert gas such as nitrogen gas or superheated steam (SHS) are recommended (Fraunhofer IGB; Young et al., 1991). SHS will be considered as potential drying medium because it is cost-effective compared to nitrogen gas (Romdhana et al., 2015).

Table 5.1. Dryers commonly used in industrial processes (Frosterud et al., 2011)

Type of dryer		Typical feed	Typical PSD* dried product	Comments	Retention time
Rotary dryer		Coarse particles, lumps, chips	As in the feed or reduced to >1mm		Several minutes 10-60 min**
Flash dryer	Conventional type	Friable solids or press cakes	0.01-10mm	Lump degradation by flashing	
	Ring dryer	Friable solids or press cakes	0.01-10mm	Internal milling and product recirculation	10-30 sec 1-10 sec**
	Ring dryer with back-mixing	High DS liquid	0.001-1mm	Internal milling and product recirculation	30 sec-3-5 min
	Superheated Steam Dryer	0.1-10	As in the feed	Only cyclone possible	20-60 sec 5-60 sec***
Fluid-bed		0.5-10 mm sticky	As in the feed	Sectioned for drying/cooling	1-10 min 10-60 min**
Swirl Fluidizer		Cakes or pastes	<50 μm	Internal milling	0.5-10 min
Spray dryer		Pumpable liquid	10-100 μm		10-30 sec
Fluidized Spray dryer		Pumpable liquid	50-250 μm Agglomerates	Fines recirculated	10-20 sec 1-5 min

*PSD: Particles Size Distribution/ **van't Land (2012)/ ***Frosterud et al. (2011) and van't Land (2012)

a. Advantages and Disadvantages of Using SHS

Superheated steam drying (SSD) is economically advantageous because it has low energy consumption compared to air drying. Recycling and reuse of exhaust steam from the dryer reduces the energy consumption (Mujumdar, 1991; Amos, 1998; Lunde, 2014; Romdhana et al., 2015). Pilot tests have shown that it is possible to save up to 85% (Worley, 2011) of the energy input by recovering the latent heat. This results in a net heat requirement of as low as 20% of that used in conventional air drying if the heat recovery is efficient (Mercer, 1994; Wardrop Engineering, Inc. 1990 as cited in Amos 1998).

Other advantages of using SHS are that it allows treatment at relatively high temperature (up to 600°C) without cracking the oil (Robinson et al., 2008 as cited in Lunde, 2014). There is no risk of fire and explosion because the SHS is oxygen-free. SHS has higher thermal conductivity (heat transfer), specific heat and low density than air. All these properties contribute to the high drying rate achieved with SSD (Amos, 1998). The low density ensures in one hand a better penetration of the condensed steam into the material to be dried and in the other hand a lower diffusion resistance of the evaporated moisture from the material to the SHS atmosphere (Fraunhofer IGB). This result in a low energy consumption in SSD.

If 1.10 to 1.70 kWh is required to evaporate 1 kg of water with hot air, it is 0.75 to 0.90 kWh with SHS (Desai and Hoadley, 2009 as cited in Fraunhofer IGB). Use of SHS provides better pollution control because the exhaust gas can be condensed to reduce emissions. SSD also provides higher drying rates for longer period of time compared to air drying. Furthermore, SSD allows organic solvents removal at relatively low temperature. Nitrobenzene for example has a boiling point of 211°C at atmospheric pressure, but it can be distilled at 99°C with SHS at atmospheric pressure (van't Land, 2012). However, the system and its operation, the start-up and shutdown processes are more complex for SHS dryers than for air dryers. Leaks are prohibited and the feeding and discharge process must not allow infiltration of air. The particles size must be small enough to be entrained and well-mixed with the steam (Amos, 1998).

b. Drying Mechanism

When the SHS is in direct contact with wet and colder materials, its internal energy (heat) is transferred to the materials. This causes the drying medium to cool down. If the SHS is slightly above its vaporization point at a given pressure, it may condensate on the materials' surface when they are in contact. Experiments conducted by Shi et al. (2012) on lignite drying with SHS resulted in high moisture content measured on the material at the initial step of the drying process when the temperature was ranging from 120°C to 200°C. Stokie et al. (2013) also found the same results while drying Victorian brown coals with SHS at the same range of temperature.

However, when the temperature was about 350°C in the case of Shi et al. (2012), the moisture content was equal to the raw material. In this case, the high temperature results in high heat transfer to the material's surface which heats up faster and do not allow significant condensation of the SHS (Shi et al. (2012). It has been proven also that when the SHS temperature increases the drying rate increases, the final moisture decreases (Shi et al. 2012) and the drying time decreases (Stokie et al, 2013). For drill cuttings, Young et al. (1991) have found a maximum water and oil removal when the temperature of cuttings bed treated in a rotary retort was approximately 427°C. Therefore, to provide better treatment of drill cuttings, the superheated steam should be at this range of temperature or higher.

The liquid form such as water and oil in the solid materials evaporates due to the sensible heat from the drying media converted into latent heat of vaporization (SUWIC Sheffield University, 2010; van't Land, 2012). The surface or trapped water is heated to its saturation temperature and then evaporates. The vapor pressure of the trapped water such as capillary water causes the moisture to flow out of the material to the surface by pressure gradient. The same mechanism occurs with oil, the light oil trapped within the capillaries boils and vaporizes first and its vapor pressure force the heavier oil at the periphery of the capillaries to flow out of the cuttings to the surface and then evaporates (Ellingsen, 1991).

The temperature of solids will remain at saturation temperature until all water and oil are removed, and once completely dry, the temperature will start to increase. Addition of evaporated water into the bulk steam causes the exit SHS to become saturated, in larger volume but lower temperature that when it entered the drying chamber. SHS is then recycled and reheated. Excess steam, slightly

superheated, is bled from the loop and used in other processes or condensed within a heat exchanger to recover its energy. It can also be used to heat the inlet steam. Figure 5.1 gives an indication of the drying process in a SSD.

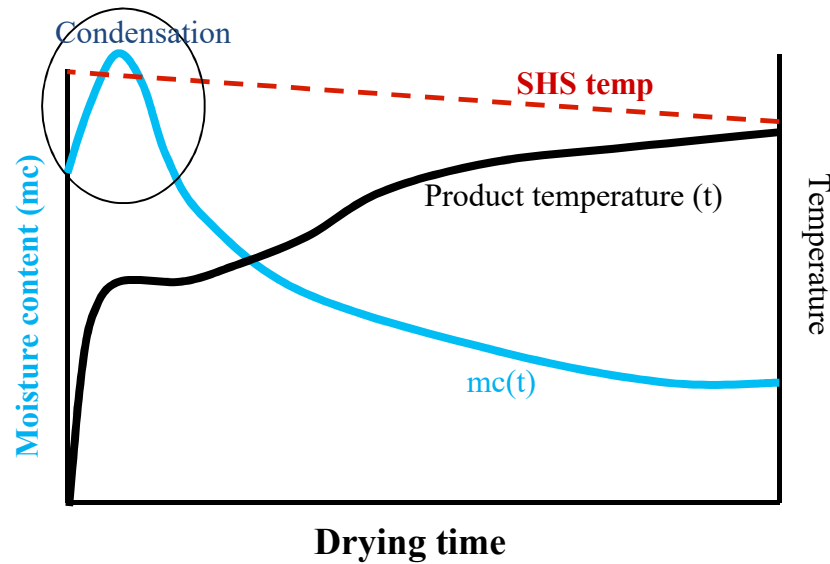


Figure 5.1. Drying process in a superheated steam dryer (Cenkowski, 2014).

c. Drying Gas Parameters

The required velocity, flow rate and pressure of the drying gas are discussed in this section.

To entrain or keep the solid particles in fluidized state, the drying gas velocity must be greater than the free fall velocity of the largest particle to be dried (Stokie et al., 2013). For the case of a fluidized-bed dryer using SHS, Equations (5.1 to 5.3), also called Wen and Yu correlation (Stokie et al., 2013) can be used to calculate the minimum fluidization velocity (u_{mf}) in function of particles size.

$$Re = \frac{u_{mf} d_p \rho_g}{\mu} \quad (5.1)$$

$$Ar = \frac{d_p^3 \rho_g (\rho_s - \rho_g) g}{\mu^2} \quad (5.2)$$

$$Re = [33.7^2 + 0.0408 Ar]^{1/2} - 33.7 \quad (5.3)$$

Here Re is the Reynolds number; d_p is the particle diameter (m); u_{mf} is the minimum velocity (m/s); ρ_g is the gas density (kg/m^3); μ is the kinematic viscosity (kg/m.s); Ar is the Archimedes number; ρ_s is the particle density (kg/m^3) and g the gravity (m/s^2).

For an average coal particle size of 1.2–1.7 mm for example, a minimum gas velocity of 0.23 m/s was obtained from these equations (Stokie et al., 2013). van't Land (2012) suggests typical air velocities of 0.25 to 1 m/s for fluidized-bed drying with air. However, for flash drying, the necessity to entrain all the solids should be taken into account. Typical gas drying velocities in flash drying range from 20 to 40 m/s if drying with SHS and from 10 to 30 m/s with air (van't Land, 2012). It is also worth noting that increase of velocity results in a decreased drying time, in both air and steam. The gas flow should be maintained constant to not affect the transport function. This allows a short residence time of the process. According to van't Land (2012) a solids/airmass ratio of 1 is good practice. Detailed steps for calculation of the drying gas (air) flow can be found in van't Land (2012).

The pressure of steam is an important parameter in a SSD. High pressure increases the condensation temperature of the SHS and improves the drying process. However, high pressure complicates the operation because it increases the possibility of leak from the feeder or the discharge locks. Therefore, these equipment need to be carefully designed to avoid steam escape.

c. Energy Recovery

In SSD, the latent heat of vaporization is easy to recover by condensing the steam. SHS can also be reused by compressing the exhaust steam to increase its condensation temperature and it can be used directly (Amos, 1998). For SHS used to dry oil contaminated cuttings for instance, it is possible to recover the oil by condensing the steam, separating the oil from the steam and then recirculating the oil-free steam.

5.1.2. Heating Systems for the Drying Media

For convective dryers, heating system comprises of two components: a water source or a steam generator and a heater. The heater can be direct or indirect. A filter can also be placed upstream the heater to provide clean heating media when it is required. In direct heaters, the drying gas is in contact with the heat source. The heat is provided by burning of gas or oil, electric heater or hot gas from other sources. In indirect heaters, the heat source and the drying medium are not in contact. The latter is indirectly heated by the heat sources cited above via heat transfer tubes or heat exchanger (Frosterud et al., 2011). Thermo-oil heaters are also used in indirect heating of drying gas. Mechanical vapor compressors are used for the case of recirculated steam (Worley, 2011). The choice of a type of heating system relies on the site utilities or the location of the drying operation. The heating system suitable for offshore use, for instance, differs from those used onshore.

Direct and indirect heaters can provide inlet gas temperature up to 650°C and 450°C respectively (Hosokawa micron). To regulate and maintain a constant temperature of the gas and the product outlet, the dryer should be equipped with a temperature control system. To control the drying operation, the gas or steam flow should be controlled on the basis of the gas-outlet temperature. van't Land (2012) proposed an example of relationship between the temperatures inlet (TA_{in}) and outlet (TA_{out}) of the drying gas for the case of a flash drying with air:

$$TA_{out} = 0.1875 TA_{in} + 35^{\circ}\text{C} \quad (5.4)$$

According to the same author (van't Land, 2012), the temperature of the product material (TP_{out}) should be 10 to 30 °C lower than the air outlet:

$$TP_{out} = TA_{out} - (10^{\circ}C \text{ to } 30^{\circ}C) \quad (5.5)$$

Zlobin (1985) obtained a difference of 30 to 40°C between flue gas as drying medium and the dry product using a ring flash dryer.

Steam generation with natural gas burning

Natural gas is one of the major combustion fuels currently used. It contains high amount of methane and varying amounts of ethane, propane, butane, and inert gases such as nitrogen, carbon dioxide and helium ((EPA, 1998). In general methane represents 85% or more of the constituents of natural gas (EPA, 1998; van't Land, 2012). The heat of combustion of methane is 804 kJ/mol (van't Land, 2012) and natural gas has an average gross heating value of approximately 1 020 Btu/scf (EPA, 2015) or 853 kJ/mol. The conversion is based on Morris et al. (2011).

As presented in Table 5.2, there are three major types of boilers used for natural gas combustion in commercial, industrial, and utility applications: watertube, firetube, and cast iron. Once generated, the steam is heated with a super-heater to get super-heated steam before being used.

Table 5.2. Natural gas boilers types (EPA, 1998)

	Watertube		Firetube	Cast iron
Principle	Water is passed through the inside of heat transfer tubes and hot combustion gases are placed outside of the tubes.		Hot combustion gases flow through the inside of the tubes and the water circulates outside of the tubes.	Same principle as firetube but units are made of cast iron rather than steel
Types	Field erected units	Packaged units	Packaged units	Packaged units
Construction	Large sized boilers equipped with multiple burners constructed on-site	Equipped with one or more individual burners. Constructed off-site and shipped to the location where they are needed		Constructed to produce low-pressure steam in small commercial applications
Heat input level	More than 100 MMBtu/hr (≈30 000 Kw)	Less than 100 MMBtu/hr (≈ 30,000 Kw)		

5.1.3. Product Feeder

The feed system plays a key role in terms of dryer feeding and steam leakage prevention. It must be carefully chosen and designed (Borde and Levy, 2006). The wet solids are fed into the dryer through feed elements which consist, in general, of a hopper or wet product bin and a pump. The wet product bin receives the materials to be treated and the pump drives them into the drying unit. The pump can be an auger, a screw, a diaphragm or a hose depending on the type of the feed materials. A hopper and an auger have been successfully used by Young et al. (1991) to feed OBM cuttings into a rotary dryer. The system is sealed by the cuttings in the feeder excluding oxygen (Young et al., 1991).

For pasty and sticky materials, the hopper can be equipped with agitator or kicker mill to disperse the wet material, to prevent incrustations at the feed point, and to provide constant feeding. Mixing a part of the dried product with the inlet materials improves also the feeding process. Model (a) and (b) in Figure 5.2 are typical feeding systems for pasty and sticky materials. For free-flowing and powdery feed materials, static hopper is efficient.

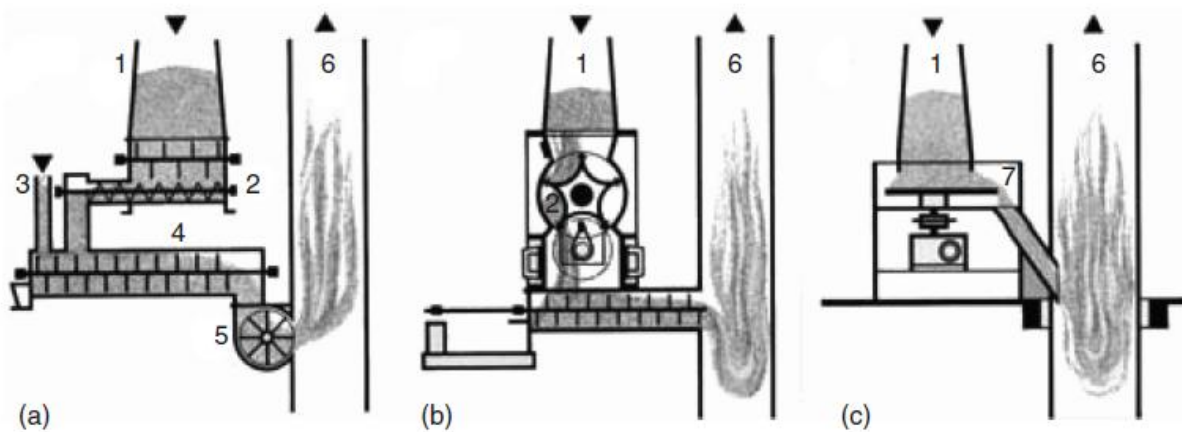


Figure 5.2. Typical feed systems (Borde and Levy, 2006)

Wet product bin (1); metering (2); recirculated product (3); mixer (4); sling (5); flash dryer tube (6); disc feeder (7).

5.2. Flash Drying or Pneumatic Drying

This technique consists of transporting the material to be dried through a vertical, horizontal or circular (ring) tube or duct by a continuous hot gas flow at high velocity (van't Land, 2012; Perazzini et al., 2016). While conveyed, the material dries using heat from the gas stream. The outlet mixture of product and drying gas is separated using a cyclone and/or bag filters. A flash dryer generally comprises a gas heater, a feeder, a drying column, a cyclone for gas/solids separation, a dried product collector, an exhaust fan, and a pneumatic transport for product cooling or transport to further treatment.

Flash dryer is suitable to treat paste-like and friable solid waste with surface moisture and particles size not exceeding 1 to 2 mm such as clay cuttings. According to van't Land (2012), if the particles are small enough, thermal conductivity does not control the heating process and thus providing better heat transfer. Furthermore, small solid particles can be easily entrained by the drying gas. As a technique based on convective heat transfer, flash drying provides good gas-solids contact and promotes high mass transfer and drying rate.

The residence time of flash drying is shorter than that of the other types of convective dryers (Table 5.1). It ranges from 30 s (Amos, 1998; SUWIC Sheffield University, 2010) to as low as 1 s or lower (van't Land, 2012). Flash dryer is more compact and requires lower space than rotary dryer for instance (Amos, 1998), making it suitable for offshore use. Other advantage of this technique is the simplicity of its equipment. Flash dryer has only few or no moving parts (van't Land, 2012). It also presents lower fire risks (Amos, 1998).

However, flash dryers present high installation costs (Fredrikson, 1984 as cited in Amos, 1998), high blower power costs in addition to the heat requirements for drying. It is inappropriate for drying large solid particles.

5.2.1. Drying Column in Flash Dryers

The drying column and the dryer in general must be internally smooth and made of insulating material to prevent incrustations and condensation of the SHS, and to lower heat loss (van't Land, 2012). The drying tube can also be equipped of classifier to separate larger particles from smaller ones which dry faster. To separate the solid product from the gas phase, cyclone is the cheapest option to achieve this purpose. However, if maximum separation is required, bag collector and wet scrubber can also be used downstream the cyclone.

In ordinary flash dryers (Figure 5.3), in order to acquire better solid handling, a part of the dried product is recycled into the feed and a cage-mill is installed to disintegrate the falling wet feed. For an industrial use, the diameter of the drying tube in this type of flash dryer can be up to one meter and the length between 10 to 30 m. However it can vary depending on the design and the material to be treated.

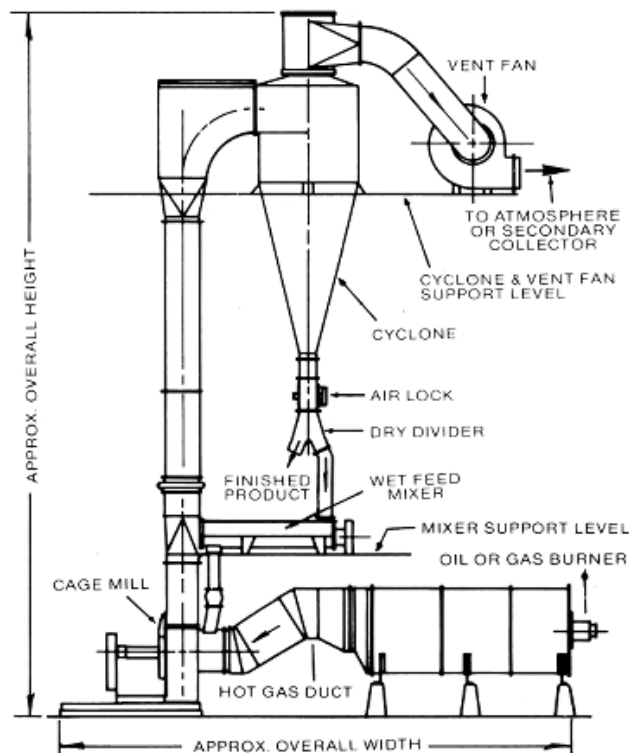


Figure 5.3. Conventional flash drying process and components (van't Land, 2012).

For more advanced ones, appropriate equipment such as rolling hammer or dispersion rotor are installed at the bottom of the dryer to de-agglomerate and pulverize the solid material. Blade or rotor classifiers are located at the top to classify the dried product. Fine particles are carried by the drying gas out of the drying chamber while coarser wet particles return to the lower section for further drying (Figure 5.4)

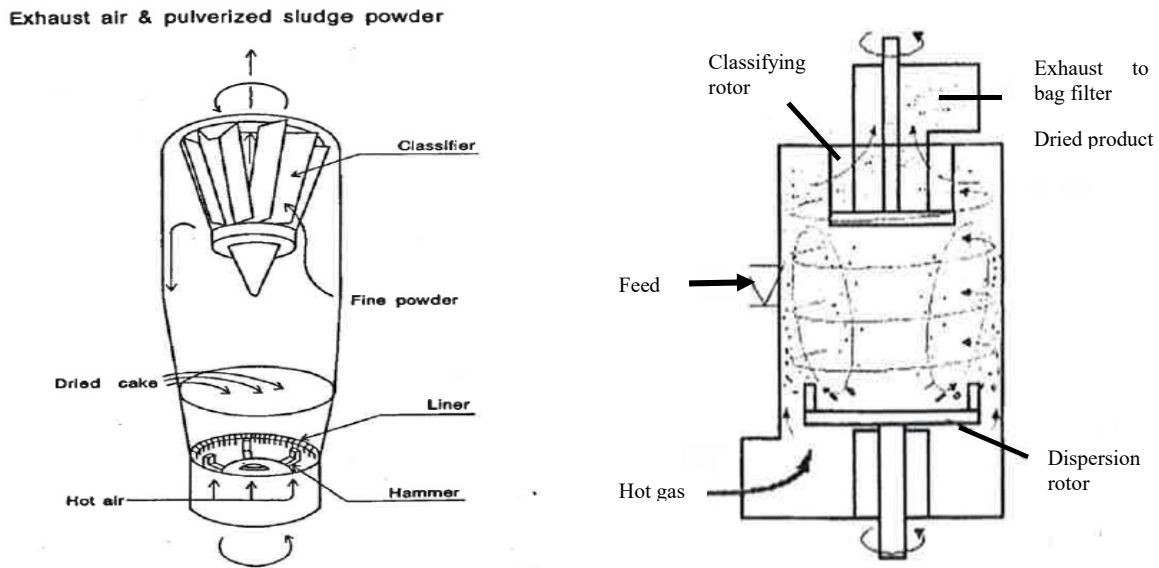


Figure 5.4. Flash dryers with internal disperser and classifier (Chen et al., 2002)

Ring dryer is another advanced type of flash dryer. It was developed to increase the applicability of flash drying technology. Ring dryer is also equipped with classifier, which allows fine particles to leave while retaining larger particles for longer drying time. The use of this last type in offshore application was considered to be feasible and will be discussed further in detail. Not only ring dryer requires lower space compared to the other types aforementioned, but it also presents the advantage of having more simple equipment with no moving parts.

5.2.2. Ring Dryer

Ring dryers have the same working principle as every type of flash dryers. The solid materials are conveyed by the drying medium within the ring drying duct (Figure 5.5). The particles are classified when entering the static classification zone. Larger and wetter particles are recirculated into the drying chamber while lighter and dryer particles are carried by the gas into the gas/solids separator (cyclone). This is important to provide better handling of the solids. However Zlobin (1985) argues that this practice complicates the dryer operation. Direct processing allows higher advantages than return processing from a technological point of view. According to Zlobin 1985, ring drying has been extensively used for drying slurries.

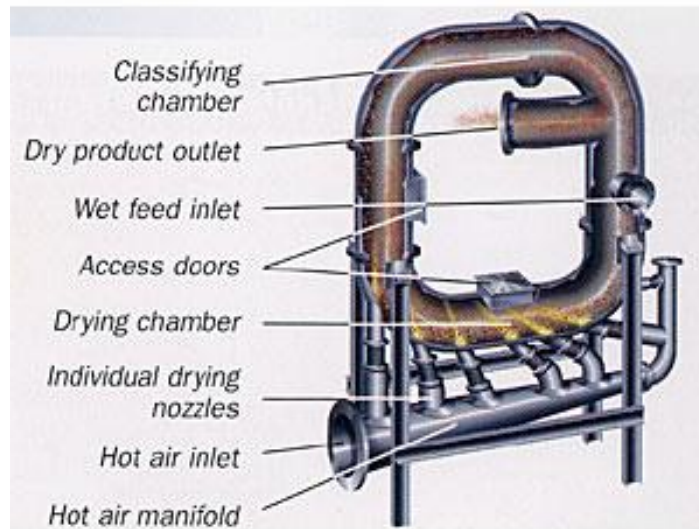


Figure 5.5. Working principle and equipment of a ring dryer (Fluid Energy Processing and Equipment Company).

Fluid Energy Processing and Equipment Company has manufactured ring dryers called ThermaJet. They can operate in laboratory, pilot and full-scale production with treatment capacity ranging from 50 g to 45 t/h. ThermaJet can work at high temperature, pressure and different with type of drying media. The inlet gas temperature can, for instance, range from 90 °C to 700 °C.

According to the same company, their ThermaJet ring dryers can be run continuously 24 hours a day and 365 days a year. Table 5.3 gives an overview of ring dryer models and their respective capacities based on 25% moisture content in the feed (Fluid Energy Processing and Equipment Company).

Table 5.3. Models and capacities of ThermaJet depending on the gas inlet temperature.

650 °C INLET / 80 °C OUTLET				
Model	Power kW	Feed rate tonnes/hr	Evaporation rate tonnes/hr	Total input kW
1	0.75	23 10 ⁻³	5 10 ⁻³	5
10	22	2.27	0.54	500
25	138	11.34	2.72	2 510
50	597	56.70	13.61	12 550
260 °C INLET / 70 °C OUTLET				
Model	Power kW	Feed rate tonnes/hr	Evaporation rate tonnes/hr	Total input kW
1	0.75	9 10 ⁻³	2 10 ⁻³	2
10	26	0.91	0.23	220
25	149	5.44	1.36	1 325
50	634	27.22	6.80	6 630

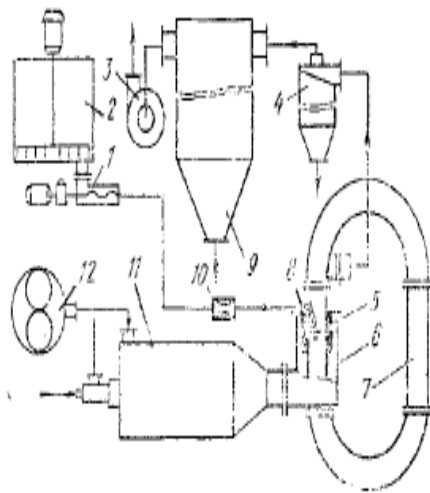
Figure 5.6 illustrates a Model 26 ThermaJet. This model was used to dry zeolite filter cake. A final water content of 10% was obtained after treating raw materials with initial water content of 40% (i.e. 75% water reduction). The treatment capacity was approximately 3.63 t/h (Fluid Energy Processing and Equipment Company).



Figure 5.6. ThermaJet Model 26

5.2.3. Design Example of a Ring Dryer

In this section, experiments conducted at the Institute of Silicate-Concrete Research and Design to treat slurries of naturally occurring chalk are discussed (Zlobin, 1985). The moisture content of the feed material was ranging from 36 to 54%. The drying process was carried out to produce more or less completely dry product using a ring dryer. The evaporation capacity of this latter was about 90 kg/h. The sketch of the experimental unit used in this study is shown in Figure 5.7.



Scheme of the experimental unit with ring dryer: 1) screw pump; 2) container with slurry mixing device; 3) exhaust fan; 4) block of cyclones; 5) annular jet; 6) housing; 7) ring dryer; 8) sprayer; 9) bag filter; 10) slurry flow meter; 11) burner; 12) blower.

Figure 5.7: Experimental set-up of ring drying process (Zlobin, 1985)

Slurries of naturally occurring chalk present drying difficulties because of their high viscosity and stickiness. The viscosity of the slurries treated range from $5 \cdot 10^{-2}$ to 10^2 Pa.s. Diluted flue gases having a temperature up to 600°C and a pressure of 0.02 to 0.03 MPa were used as drying medium. Several temperature and pressure conditions were tested at a gas flow of 200 to 300 m/s. This high gas velocity was used in order to provide a circulation of the material and drying medium up to 10 times within the dryer. The results obtained from the experiments are presented in Table 5.4.

Table 5.5 illustrates a design example of industrial ring dryer and conventional flash dryer. The ring dryer was designed by Zlobin (1985) based on data from the aforementioned experiments. The conventional flash dryer design data was taken from van't Land (2012). The flash dryer was designed to treat inorganic material of $600 \mu\text{m}$ average diameter and insoluble in water.

The ring dryer capacity is also compared with spray dryer capacity at the bottom of Table 5.5. The data about spray dryer was taken from Zlobin (1985), in which it was designed to treat the same material (chalk slurries) discussed earlier.

Table 5.4. Results from the experiments (Zlobin, 1985)

		1	2	3	4	5	6	7
Moisture (%)	slurry	34.6	36	38.6	40	47.1	47.1	54.1
	product	0.1	0	0.1	0.09	1.8	0	0.03
Air flow (m³/h)		545	541	552	556	489	492	510
Specific fuel consumption (kg tentative fuel/ton product)		80.2	98	104	123	172	209	143
Gas pressure at dryer inlet (MPa)		0.026	0.023	0.022	0.027	0.022	0.022	0.028
Gas temperature (°C)	inlet	550	490	470	540	392	400	550
	exit	153	155	150	198	150	195	145
Throughput (kg/h), with respect to	moisture evaporated	91.2	85	75	57	50	42	88
	dry product	173	151	120	86	59	47	75
Volume loading (kg/m³.h) with respect to moisture evaporated		1899	1770	1563	1188	1041	979	1816
Residue on sieve 40 µm (%)	slurry	4.9	5.1	4.9	5.05	4.71	4.77	5.1
	product	0.3	0.4	0.84	0.92	2.98	1.64	0.2

Table 5.5. Industrial design of flash and ring dryers

Technical Features of Industrial Unit		Ring Dryer	Conventional flash dryer
Capacity (kg/h) with respect to	moisture evaporated	2 000	876
	material feed inlet	5 500	5 876
	dry product outlet	3 500	5 000
Moisture content (%)	inlet	36	15
	product outlet	0.1	0.1
Energy required (kJ/h)			4 308 353
Flow (m ³ /h)	air	12 000	5 563.7
	fuel (natural gas)	230	211.82 nm ³ /h
Combustion chamber required in kW			1 400
Energy consumption by use of natural gas in kJ (kWh) per kg of evaporated water	Design		4 918 (1.37)
	Long-term		7 377 (2.05)
Gas pressure (kPa)		30	
Temperature of gases (°C)	inlet	550	600
	exit	150	147.50
Long-term electricity consumption (kWh per ton of evaporated water)			63.9
Dryer dimensions	weight (kg)	3 500	
	diameter (m)		0.45
	height (m)		12
Specific Indices (per ton product) of Dryers			
Unit		Ring dryer	Spray Dryer
Fuel consumption, kg tentative fuel		80	450
Electricity consumption (kWh)		100	150
Metal utilization (kg)	dryers	12 00	15 000
	units	12 000	40 000

Discussion

The high fuel consumption (natural gas) encountered with ring dryer (230 m³/h versus 211.82 nm³/h for flash dryer) in Table 5.5 may be due to the high moisture content of the chalk slurries treated. This product may present higher difficulties to treat than the product treated with flash dryer. Another possibility also is the type of natural gas used. In van't Land (2012), methane which is the main component of natural gas was assumed to be 85%. The gas and product recirculation and high gas velocities require higher amount of fan power (electricity).

The comparison between spray and ring dryers designs to treat the same material at the bottom section of the table shows that ring dryer is more cost-effective in terms of energy consumption than spray dryer.

The estimated energy requirement for flash dryer operating with air was 2.05 kWh per kg water removed in a long-term basis (van't Land, 2012). This means approximately 307.5 kWh per t of cuttings is required assuming 15% water content and complete water removal. As mentioned in Section 5.1.1.a Advantages and Disadvantages of Using SHS, the energy requirement is reduced to 0.75-0.9 kWh per kg of evaporated water while drying with SHS. An energy consumption of 112.5 to 135 kWh per t of cuttings is then required if SSD is used. If the heat recovery is efficient, the final energy consumption ranges from 22.5 kWh to 27 kWh/t of cuttings. Taking the same design example given on flash dryer in Table 5.5, the long-term energy (as natural gas and electricity) consumption for treating drill cuttings with 15% water by weight would be 32.09 to 36.59 kWh/t of cuttings while using SHS. The calculation is also based on the long-term electricity consumption given in Table 5.5 which is 63.9 kWh/t of evaporated water.

5.3. Fluidized-Bed Drying

This technique is discussed in order to evaluate its potential to treat drill cuttings with larger particles size. It consists of supplying the drying gas into a rectangular or cylindrical bed containing solids waste (Perazzini et al., 2016). The dryer bed is divided into several cells or sections and the wet material is fed in the first cell. The ascending hot gas flows through a special perforated distributor plate and keeps the solids in a fluidized state.

As in flash drying, the gas velocity should be greater than the free fall velocity of the largest particle to be dried and sufficient to keep the solids particles in a fluidized state (Frosterud et al., 2011). But contrary to flash dryer, the value is relatively low. Typical values are from 0.25 to 1 m/s while drying with air (van't Land, 2012).

This process provides very high heat and mass transfer values (Mujumdar, 2006). To improve the drying process, contact heating panels or heating tubes can be immersed within the bed of the dryer. This practice reduces the size of the dryer by up to two third while having the same performance as a purely convective or standard fluidized-bed dryer. It also reduces the gas flow required for the process. Another method to improve the efficiency of the process is to vibrate the dryer bed. It helps to de-agglomerate sticky materials and resulting in better fluidization and drying rate (Frosterud et al., 2011). Other possibility is to incorporate a slow-moving rotating agitator within the first section of the dryer. It serves to agitate gently the wet feed material. This provides disintegration of the material and better fluidization without causing particle degradation. Furthermore, the agitation helps the drying gas to break up into bubbles which improves the heat and mass transfer (SUWIC Sheffield University, 2010).

All the equipment used in flash dryers are also used in fluidized-bed dryers. The only difference is the drying chamber. The feed materials enter a feeder such as hopper and auger as discussed in section 5.1.3, and then fluidized by the drying gas within the drying chamber. The drying gas and lighter particles pass through a gas/solids separator such as cyclone while the larger ones are recovered by a discharge system composed of screw conveyor and a discharge lock. The particles separated from the cyclone also are directed to the discharge system (Frosterud et al., 2011). Figure 5.8 describes the general process of a fluidized-bed drying.

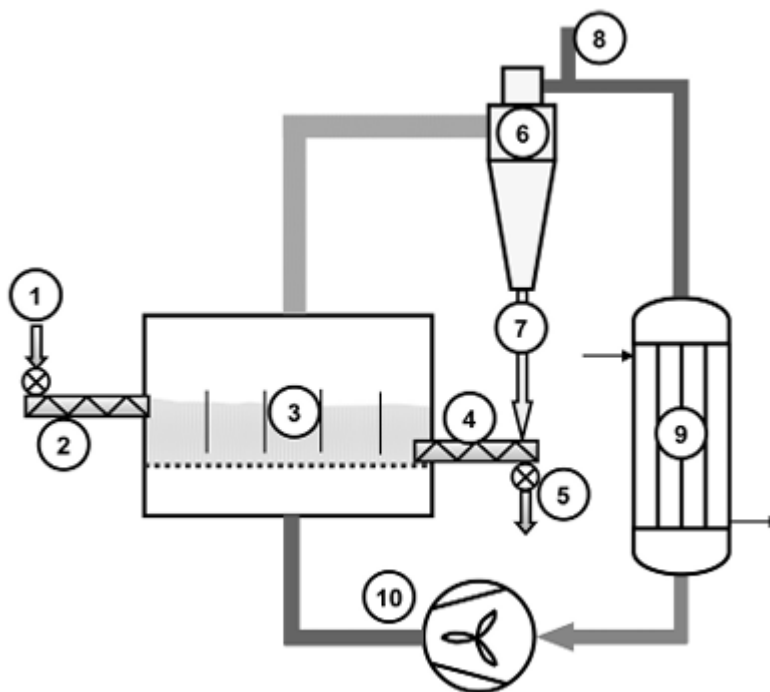


Figure 5.8: General principle and components of fluid-bed steam dryer (van't Land, 2012)
 Feed lock (1); screw conveyor (2); drying chamber (3); discharge screw conveyor (4); discharge lock (5); vaned ring (6); ejector (7); vapor pipe (8); heat exchanger (9); fan (10).

Fluidized-bed drying has been used to process a broad range of materials such as powders, crystals and granules (Frosterud et al., 2011) and mostly used in steam drying processes. Steam fluidized-bed dryers require larger particles than flash dryers, typical size of feed materials dried with fluidized-bed dryers range from 0.5 to 10 mm (see Table 5.1). The retention time is longer (8 min for instance) if it is only 5 to 60 s in steam flash dryers (van't Land, 2012). According to van't Land (2012), flash dryer and fluidized-bed dryer are the only convective dryers extensively used for superheated steam drying until now. Spray dryer has been used in only one known case (van't Land, 2012).

Continuous fluid-bed steam dryer has been widely used to dry material of biological nature (van't Land, 2012). Braunschweigische Maschinenbauanstalt AG Germany (BMA) for example sells dryers which have water evaporation capacities of 25 to 56 t/h when operated at steam pressure of 25 bar absolute and 17 to 40 t/h at 15 bar absolute.

The fan powers required for these dryers are in the range of 0.6 to 1.35 MW (van't Land, 2012). Table 5.6 illustrates a design and performance data of a Niro A/S fluid-bed dryer. It is a superheated steam fluid-bed dryer commercialized by Niro A/S Denmark (SUWIC Sheffield University, 2010). The working pressure of this dryer ranges from 200 to 300 kPag. The used steam is recirculated back into the dryer chamber. The average residence time is 300 s. This dryer is used for treating by-product materials such as sludge in the agricultural industry (SUWIC Sheffield University, 2010).

Table 5.6. Design example of a SHS fluid-bed dryer (SUWIC Sheffield University, 2010)

Evaporation (t/h)	27.2
Capacity (t/h)	10.8
Moisture content of the feed (%)	70.8
Moisture of product or discharge (%)	10
Thermal requirements	
Without heat recovery (kWh/ton-evap*)	958
With latent heat recovery (kWh/ton-evap)	111
Blower power (kWh/ton-evap)	67

* ton-evap: tonnes of evaporated water

Based on Table 5.6, for drill cuttings of assumed 15 % water content, an energy (as heating source and blower power) of 26.7 kWh/t of cuttings is required for complete drying. The estimation is based on assuming that the latent heat is recovered.

Chapter 6: Laboratory Testing for Study of Chemical Dewatering Method for Drill Cuttings Pre-treatment

A new dewatering method based on chemical destabilization and mechanical separation of contaminated drill cuttings is evaluated in this chapter. The mud coating the drill cuttings is first destabilized using chemicals called MudSplit before separating away the water (and oil) from the cuttings by centrifugation. MudSpilt chemicals were used to destabilize and break water-in-oil emulsion and split OBM waste into three phases: oil, water and solids (Smith, 2014). The experiments carried out in this thesis will address the effect of MudSplit chemicals on OBM drill cuttings. Different solvents will also be used to find out if the solvent extraction would be more efficient with destabilized mud. The first phase of the laboratory testing was dedicated to verify the effect of the chemicals on water and oil separation and to estimate the conditions for efficient phase separation. The second phase consisted of evaluating if MudSplit chemicals will enhance the oil and water separation when used in combination with solvent extraction.

Solvent extraction is a common technique used to remove harmful compounds from contaminated materials (Reis, 1996). It has been shown efficient in removing hydrophobic organic contaminants such as petroleum hydrocarbons from soils (EPA, 2001 as cited in Li et al., 2012). Solvent extraction is accepted as an alternative method for remediation of organic contaminants (Li et al., 2012). However, McGill and Rowell (1980) stipulate that water-in-oil emulsions reduce the extraction efficiency of solvent. Therefore, the use of emulsion destabilizer chemicals such as MudSplit can be a way to improve the process. Combining MudSplit with solvents will also further verify the mud destabilization and may open up for more effective and environmentally friendly solvents

For the second phase, the choice of solvents is a crucial step before performing the tests. A survey carried out by the Victorian Environmental Protection Authority (VicEPA) showed that the following solvents and mixture of solvents are preferred to extract petroleum hydrocarbon from soil: dichloromethane (DCM), DCM/methanol (1:1), DCM/acetone (1:1), hexane/acetone (3:1), pentane/acetone (1:1) and alcohols such as methanol and isopropanol.

The (1:1) or (3:1) mean 1 ml or 3 ml of solvent A is mixed with 1 ml of solvent B. Figure 6.1 indicates the results from this survey of 23 laboratories investigated.

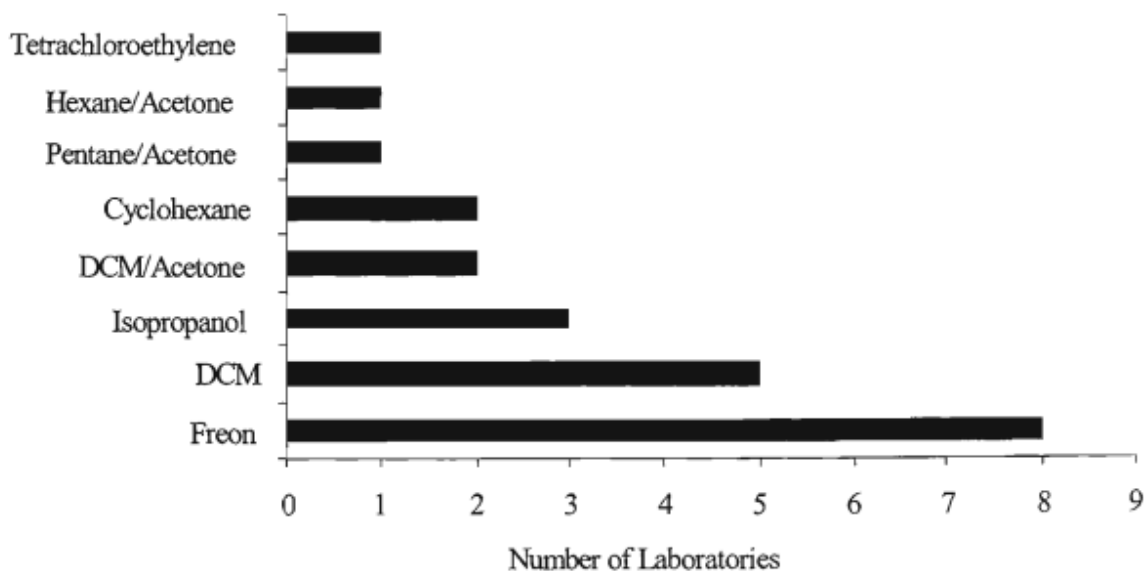


Figure 6.1: Solvents used for TPH extractions (VicEPA as cited in Buddhadasa, 2002).

The choice of the solvents used in this thesis was also based on this survey as well as other factors such as extraction efficiency, low cost, availability of the solvents, low toxicity and regulatory requirements. Based on these criteria, hexane/acetone (3:1) and DCM were chosen. Hexane is less expensive and safe solvent. It is conventionally used to extract petroleum hydrocarbons from soils. Acetone is relatively less expensive and presents low toxicity. DCM is also cheap and has high extraction efficiency towards semi-volatile TPH according to Weisman (1998) as cited in Buddhadasa (2002). However, DCM is more toxic than the two solvents previously cited. In addition, petroleum ether was also used. This solvent has high extraction efficiency, not expensive and easy to evaporate from the cuttings samples. According to Sui et al. (2014), petroleum ether is capable to remove 76 to 94 % of TPH.

Materials and Methods

This research was conducted by extracting water and oil from OBM contaminated drill cuttings using MudSplit chemicals and solvents. Water and oil separation rate are then considered to be the important indicators of the extraction method efficiency. Methods such as retort analysis, soxhlet and soxtec extractions were used to determine the water and oil content of the treated drill cuttings.

6.1. Chemicals and Reagents

The chemicals and reagents used during this study consist of MudSplit and solvents. MudSplit chemicals are manufactured and supplied by Norwegian-Group AS. For the experiments, MudSplit 02 and MudSplit 04, as presented in Figure 6.2, were used. No information are available concerning the composition of these chemicals. Tests performed by the supplier and verified during this study showed that they are hydrophilic and thus dissolve in water. Mixing these chemicals with water resulted in a homogenous mixture. However, they are not soluble in nonpolar solvents such as hexane.



Figure 6.2: MudSplit 02 (left) and MudSplit 04 (right) chemicals

The solvents used were also supplied by Norwegian-Group AS. These include analytical grade *Sigma-aldrich* petroleum ether, n-hexane and DCM, and *VWR chemicals* technical acetone. Table 6.1 indicates a summary of the properties of these solvents which were taken from the safety data sheet of each solvent. These latter and together with the current prices of the products are available at the manufacturers' websites (Sigma-aldrich and VWR chemicals). The three parameters designated with asterisk in Table 6.1 were taken from Riddick et al. (1986) and are described as follows:

a. Solubility parameter δ

This parameter describes the relationship between the physical properties of the solvent and its effectiveness in dissolving specific solutes. It is used in the selection of the appropriate solvent for dissolving a particular substance (Riddick et al., 1986). According to the same authors, one substance dissolves easily in another if their solubility parameters are similar.

b. Dipole moment μ

This parameter expresses the polarity or the electrical dissymmetry of a molecule. The compounds with higher value of dipole moment are polar. They are nonpolar if the value of the dipole moment is low. According to Riddick et al. (1986), polar compounds are generally less volatile than nonpolar compounds. Polar substances are soluble in other polar substances and nonpolar in other nonpolar (like dissolves like rule).

c. Evaporation rate ER

The evaporation rate is a parameter used to quantify how fast a specified amount of solvent can evaporate from a surface. Butyl acetate is commonly used as a reference solvent and designated as BuOAc = 1. The higher the value of the evaporation rate, the faster the solvent can evaporate.

Table 6.1: Properties of Petroleum ether, Hexane, DCM and Acetone

	Petroleum ether	<i>n</i>-Hexane	DCM	Acetone
Product reference	77399-7L	32293-2.5L	32222N-2.5L	20063.365
Brand	Sigma-Aldrich	Sigma-Aldrich	Sigma-Aldrich	VWR Chemicals
CAS No	101316-46-5	110-54-3	75-09-2	67-64-1
Boiling point (°C)	30-40	69	39.8-40	56.2
Form	Liquid	Liquid	Liquid	Liquid
Solubility parameter δ at 25° C (J^{1/2}/cm^{3/2})*	No data available (NA)	14.87	20.21	20.5
Dipole moment μ (D)*	NA	0.085 (25 ° C)	1.14 (25 ° C)	2.69 (20 ° C)
Polarity	Non-polar	Non-polar	Polar	Polar
ER (BuOAC)*	NA, extremely volatile	8.9	14.4	5.59
Hazards Identification	Extremely flammable liquid and vapor	Highly flammable liquid and vapor	Nonflammable	Highly flammable liquid and vapor
Toxicological information	Suspected human reproductive toxicant	Slightly hazardous for human health	Possibly carcinogenic to humans	No evidence of carcinogenic properties
Ecological Information	Low bioaccumulation potential ⁷ but toxic to aquatic life	Low bioaccumulation potential ⁷ but toxic to aquatic life	Does not bioaccumulate but not readily biodegradable	Does not bioaccumulate and readily biodegradable
Current Price (NOK)	726.75 (7 L)	1 708.50 (2.5 L)	603.50 (2.5 L)	335,00 (5 L)

6.2. Samples Preparation

The laboratory work was performed at the University of Stavanger. The samples to be treated are constituted of OBM cuttings from an oil field in the North Sea and supplied by the International Research Institute of Stavanger AS (IRIS AS) via ThermTech AS. The cuttings composition are fine clay particles. During the whole period of study, the cuttings were stored in plastic barrel at 4°C in a cooling chamber to prevent water and light hydrocarbon evaporation.

A batch of cuttings from the barrel was collected in a plastic container. The supernatant liquid was poured back into the barrel while the solids fraction was processed in two different ways. First, enough cuttings were collected to perform the whole experiments and then well-mixed within the plastic container. These samples are called “raw cuttings” in this report.

Second, the solids fraction was transferred into four 250 ml wide neck plastic bottles and centrifuged at 2000 rpm for 2 minutes using a *Hettich ROTOFIX 46* centrifuge with a maximum possible speed of 4000 rpm (Figure 6.3). This equipment was used to centrifuge all the samples prepared during the experiments. This procedure was used to provide dryer and easy to homogenize cuttings samples with stable oil and water concentration. After centrifugation, the cuttings were split into three phases consisting of liquid as the top layer, followed by small and light particles in the midst and larger solids at the bottom of the sample containers. The two top layers were decanted and the rest (larger cuttings) was collected into a plastic container. The same process was repeated until enough solid cuttings were collected. The samples prepared this way are called “centrifuged cuttings” (Figure 6.4).



Figure 6.3: Hettich ROTOFIX 46 lab benchtop centrifuge (Hettich Instruments LP)

The two plastic containers (with raw and centrifuged cuttings each) were afterwards lidded and stored at 4 °C in the cooling chamber. The raw cuttings were well mixed prior to each sampling. Figure 6.4 presents two samples taken from the centrifuged (left) and raw cuttings (right). The average oil and water content of these cuttings were determined by retort analysis which is described and discussed in details in Section 6.4. Determination of Water and Oil Content in Drill Cuttings.



Figure 6.4: Centrifuged cuttings (left) and raw cuttings (right)

6.3. Extraction Process

The tests were performed in two phases: 1) the effect of MudSplit chemicals on drill cuttings in terms of water and oil separation was evaluated. The most efficient MudSplit was identified as well as the parameters (centrifugation force and dosage) yielding the best water and oil separation; 2) the pre-treatment method identified above was combined with solvent extraction to determine if the water and oil separation would be enhanced. These tests were performed using the following process.

Approximately 100 g of cuttings (raw or centrifuged) were collected into 250 ml wide neck plastic bottle. A known volume of water, MudSplit chemical and/or solvent was added into the sample. The sample was agitated and then allowed to rest for two minutes after addition of each chemicals (MudSplit and solvent). This was done to allow the chemicals to react. The oil and water were afterwards separated from the cuttings by means of centrifugation and decantation. The centrifugal speed (rpm) was alternated with duration time of 1 min and 30 sec. After centrifugation, the liquid or sludge fraction was decanted in a graduated glass cylinder for reading before being transferred into 125 mL narrow-mouth plastic flask and stored in the cooling chamber. Samples from the remaining cuttings were taken for oil and water content analysis. Three samples were prepared and analyzed for each parameter studied, unless for some cases in the solvent extraction.

6.3.1. Phase 1: Oil and Water Extraction using MudSplit Chemicals

Some parameters were analysed and compared to determine the optimum conditions for the MudSplit chemicals. Figure 6.5 illustrates the extraction process used.

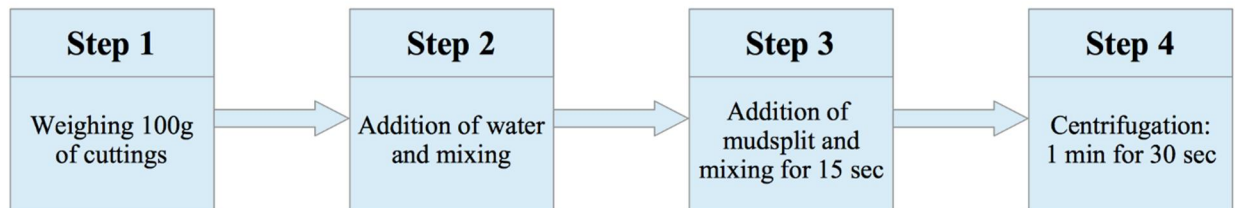


Figure 6.5: Extraction process for studying MudSplit efficiency

a. Treatment with MudSplit vs no MudSplit

To evaluate the effect of the MudSplit chemicals on the contaminated cuttings, 12 samples were prepared: six from the raw cuttings and six from the centrifuged cuttings. Three of the raw cuttings samples were treated using steps 1 to 4 in Figure 6.5, with 40 ml water, 5 ml MudSplit 02 (M2), centrifuged at 1500 rpm and denoted RM2. The three other raw cuttings samples were treated using the same amount of water and procedure except step 3 (i.e. no addition of M2) and designated as Rw.

The six centrifuged cuttings samples were treated the same way as the raw cuttings above. The samples were denoted CM2 and Cw for samples treated with and without M2 respectively.

b. MudSplit 02 vs MudSplit 04

In order to determine which of the two MudSplit chemicals is most efficient for water and oil separation, six samples (three from raw cuttings and three from centrifuged cuttings) were treated with MudSplit 04 (M4) using the same procedure used for M2 above. The raw cuttings samples were called RM4 and the centrifuged cuttings samples CM4.

c. Alternating the g-forces

The centrifuge speed was varied in order to identify the most efficient centrifugation parameter to be used for later experiments. To carry out the experiments, 12 raw cuttings samples were prepared using steps 1 to 4 in Figure 6.5, with 40 ml water and 2 ml M2 added. Three samples each were treated at 1500 rpm, 2000 rpm, 3000 rpm and 4000 rpm.

Since the centrifugation speed is not a good measure of the force applied on the samples during the centrifugation process (Høiland et al., 1986), the rpm data have been converted into relative centrifugal force RCF. Equation (6.1) was used for the conversion (Hettich Instruments LP):

$$RCF = P \left(\frac{RPM}{1000} \right)^2 \cdot r \cdot 1.118 \quad (6.1)$$

Where RCF is the relative centrifugal force, RPM is the rotational speed (rpm), r is the centrifugal radius in mm, which is the 173 mm for the centrifuge used during this thesis.

d. Alternating the Dosage of MudSplit

To find the optimal M2 dosage, three samples each were treated with 1 ml, 3 ml and 10 ml of M2, following steps 1 to 4 in Figure 6.5 and using 40 ml water. 2 ml and 5 ml were already used in previous experiments and therefore not tested. The samples were centrifuged at 1500 rpm.

e. Alternating the Amount of Water

The centrifugation speed and M2 dosage determined to be most efficient in steps c. and d. were used to treat a set of three raw cuttings samples with 40 ml water added. This amount of water was also reduced to determine if it will affect the separation efficiency. Six samples were prepared for this purpose, three treated with 30 ml water and three with 20 ml.

f. Pre-mixing M2 with Water before Addition

Since all previous experiments were conducted by adding water prior to M2 addition, another test was carried out with three raw cuttings samples in which the M2 was diluted or pre-mixed with water before the mixture was added into the samples. The rpm, amount of water and dosage of M2 determined to be most efficient in the previous experiments were used for this test.

6.3.2. Phase 2: Combining MudSplit with Solvent Extraction

To evaluate if the water and oil extraction can be enhanced by MudSplit, this latter was added in addition to solvent, and compared with solvent extraction alone. Hexane/Acetone (3:1), DCM and Petroleum ether were used. The efficiency was compared. The extraction process is as presented in Figure 6.6.

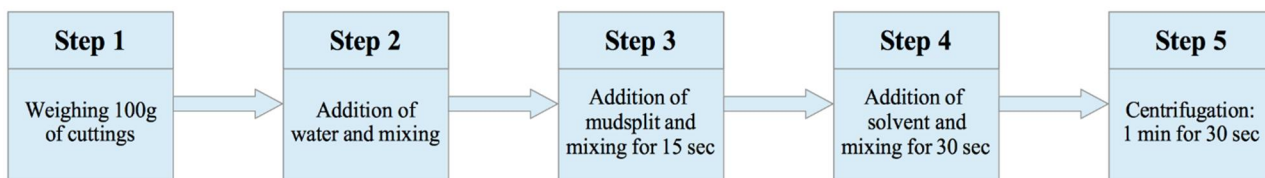


Figure 6.6: Extraction process for studying the efficiency of MudSplit combined with solvent extraction.

To perform this test, 24 samples were prepared. Two different centrifugation rpm setups were evaluated: 1500 rpm and 3000 rpm. The solvent used, the amount of water, M2 and solvent, as well as the centrifugation rpm used, are presented in Table 6.2.

Table 6.2: Parameters analyzed

Solvents used	Total number of samples	No. of samples	Water (ml)	M2 (ml)	Solvent (ml)	Centrifugation speed (rpm)
Hexane/ Acetone (3:1)	12	4	30	3	10	1500
		4	30	3	10	3000
		4	30	0	10	3000
DCM	6	2	30	3	10	1500
		2	30	3	10	3000
		2	30	0	10	3000
Petroleum ether	6	2	30	3	10	1500
		2	30	3	10	3000
		2	30	0	10	3000

6.4. Determination of Water and Oil Content in Drill Cuttings

To evaluate the efficiency of the cuttings treatment methods used, oil and water on cuttings were considered to be the important parameters and were determined. The cuttings remaining after the extraction were used for the analysis. Both water and oil content of untreated samples and samples treated without solvent were determined by retort analysis. For solvent extracted samples, only water content was determined by retort analysis. It was not possible to determine the oil content with this method. Therefore, soxhlet extraction was used. However, the challenges encountered during the analysis with this method led to the use of soxtec. The methods used and the associated challenges are discussed in this section.

a. Retort Analysis

Retort analysis is a method used to extract oil and water from solids using distillation. The oil and water are allowed to vaporize by heating a known mass of sample in an *OFITE* 50-mL electronic retort kit (230 V) as presented in Figure 6.7.



Figure 6.7: Running a retort analysis with OFITE Electronic Retort Kit

The temperature of the retort was set to 480°C and the distillation process was run for at least 45 min. The oil and water vapors were condensed and then collected to determine the water content and the oil retention on cuttings (ROC).

This method is well described in the API RP 13B-2 and recommended by several guidelines in the oil and gas industry to determine the oil content of cuttings after solids control processes or prior to cuttings discharge (NOROG, 2015, API). Equation 6.2 was used to calculate the water content and Equations 6.3 and 6.4 to calculate the ROC. This latter is expressed in % of oil by wet and dry mass of cuttings (ROCwet and ROCdry respectively) and the water content is expressed in % of water by wet mass.

$$\text{Water content (\%)} = \frac{m_{\text{water}}}{m_{\text{wet cuttings}}} \times 100 \quad (6.2)$$

$$\text{ROCwet (\%)} = \frac{m_{\text{oil}}}{m_{\text{wet cuttings}}} \times 100 \quad (6.3)$$

$$\text{ROCdry (\%)} = \frac{m_{\text{oil}}}{m_{\text{dry cuttings}}} \times 100 \quad (6.4)$$

Here m_{water} is the mass of water recovered, m_{oil} the mass of oil recovered, $m_{\text{wet cuttings}}$ the mass of initial wet cuttings, and $m_{\text{dry cuttings}}$ the mass of dry retorted cuttings.

To calculate the water and oil removal, Equations 6.5 and 6.6 were used:

$$\text{Water separation (\%)} = \frac{\text{Water content}_{R_x} - \text{Water content}_s}{\text{Water content}_{R_x}} \times 100 \quad (6.5)$$

$$\text{Oil separation (\%)} = \frac{\text{ROC dry}_{R_x} - \text{ROC dry}_s}{\text{ROC dry}_{R_x}} \times 100 \quad (6.6)$$

Water content $_{R_x}$ is the water content of untreated raw cuttings and Water content $_s$ is the water content of the sample to be evaluated. ROCdry $_{R_x}$ is the ROCdry of untreated raw cuttings and ROCdry $_s$ is the ROCdry of the sample to be evaluated.

It is important to note that if the sum of the mass of liquid (water and oil) recovered (m_w and m_o) and the dry cuttings (m_d) was not within 5 % of the mass of initial wet cuttings used (Equation 6.7), the test was invalid and repeated.

$$0.95 < \frac{m_d + m_w + m_o}{m_{wet}} < 1.05 \quad (6.7)$$

The water and oil content of all the samples (51 samples) prepared in phase 1 of this study (M2 extraction), and six untreated samples (three raw cuttings and three centrifuged cuttings) were analyzed using this method. It was, however, challenging to determine the oil content when solvent treated cuttings were to be analyzed. The condensate obtained consisted of water at the bottom layer of the cylinder and a mixture of oil and solvent at the top. The results can not be read straight away. The water has to be separated through a separatory funnel and the solvent has to be evaporated off.

To evaporate the solvent, two methods were used:

- The oil and solvent mixture was transferred into a round-bottom flask suitable for a rotary evaporator and the solvent was evaporated using this device. The rotary evaporator was run at 40°C and 120 rpm.
- The flask was maintained by a holder and a stand above a hot water (65°C) heated with a heating plate. A magnetic stirrer was placed within the flask to ensure good mixing of the heat. The samples were frequently weighed with a *Shimadzu Aux 120* analytical balance until stable value was obtained.

Due to the complexity of this procedure, only the water content of the solvent extracted samples was analyzed by retort analysis. Their oil content was determined using Soxtec.

For the samples from phase 2, 15 samples were analyzed for water content: nine from the samples extracted with hexane/acetone (3:1) (three for each condition analyzed), and six from the samples extracted with DCM and petroleum ether (one from each condition evaluated).

b. Soxhlet Extraction

Soxhlet extraction is a method used to extract nonvolatile and semivolatile organic compounds from solids, such as cuttings, using solvent (EPA, 1996). A known mass of sample contained in an extraction thimble is placed within a soxhlet extractor. This latter is connected to a condenser at the top part and a flask containing solvent at the bottom. During this laboratory experiment, approximately 30 g of samples were analyzed each time. Hexane was used as extracting solvent. It dissolves and washes off the oil from the cuttings and then recirculated into the flask (Figure 6.8). After appropriate extraction time, the experiment was stopped and the oil and solvent mixture were transferred into another flask suitable for the rotary evaporator. Or, as discussed earlier, the solvent can be evaporated using a heating plate. Detailed description of the method can be found in EPA SW-846 series method 3540c (EPA, 1996).

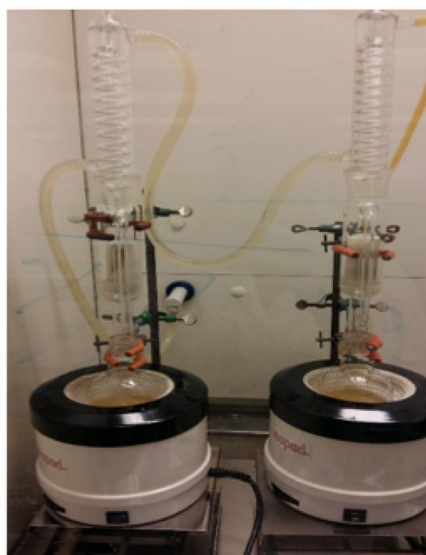


Figure 6.8: Oil extraction with Soxhlet

This method has the limitation on running period. If the centrifuged untreated samples were for instance extracted for six hours, the oil content obtained was lower than expected. Raw cuttings extracted for 18 hours gave results more or less identical to those obtained with retort analysis. Six hours extraction time is considered insufficient. This method is not convenient to determine the water content of the samples.

c. Soxtec Extraction

This method uses the same principle as Soxhlet extraction. However, Soxtec is run through three stage extraction (boiling, rinsing and evaporation), which makes it able to achieve the same oil recovery as in Soxhlet extraction in a shorter time. Furthermore, with this equipment six samples can be analyzed at the same time. A Tecator Soxtec System HT 1043 extraction unit and 1046 service unit were used (Figure 6.9). The equipment was tested by a previous user and validated to give similar results as with retort analysis using centrifuged cuttings and light petroleum ether. This solvent was chosen due to its low boiling point and high evaporation rate, which facilitate solvent separation from the oil extract after the analysis.



Figure 6.9: Tecator Soxtec System HT 1043 extraction unit (right) and 1046 service unit (left)

Two tests were run. The first comprised of six samples prepared with hexane/acetone (3:1) (two samples from each condition analyzed). The second test consisted of drill cuttings prepared with DCM and petroleum ether, one sample from each condition analyzed. The samples were first transferred into porcelain dishes and dried at room temperature (under fume hood) for 3 hours. Then they were stored in a transparent plastic box with lid at 4°C in the cooling chamber prior to the Soxtec analysis. As recommended in the EPA SW-846 series method 3541 (EPA, 1994), the boiling time and rinsing time were set for 60 min each and 50 ml petroleum ether was used for the extraction. The boiling temperature was set to 106°C. After rinsing, the heat was turned off and the solvent in the extract was evaporated off by placing the sample cups under a fume hood. The samples were weighed with an OHAUS AX523 analytical balance every 2 to 5 minutes until stable values were obtained.

Chapter 7: Results and Discussion

The results obtained from the two phases of laboratory experiments are presented in this chapter. The discussion follows the results in each sub-chapter.

7.1. Samples Characterization

The oil and water content of untreated raw and centrifuged cuttings determined by retort analysis are given in Table 7.1 and Table 7.2 respectively.

Table 7.1: Characterization of the raw cuttings samples

Parameters	ROC wet	ROC dry	Water content
Samples	(%)	(%)	(%)
Rx1	10.1	15.34	22.07
Rx2	9.77	15.14	22.09
Rx3	9.6	14.58	22.29
Mean (μ)	9.82	15.02	22.15
σ	0.21	0.32	0.1
U (95%)	± 0.29	± 0.44	± 0.14

Rx: Untreated raw cuttings samples

Table 7.2: Characterization of the centrifuged cuttings samples

Parameters	ROC wet	ROC dry	Water content
Samples	(%)	(%)	(%)
Cx1*	8.30	11.13	15.87
Cx2*	8.17	10.97	15.92
Cx3	7.93	10.64	15.76
μ	8.13	10.91	15.85
σ	0.15	0.20	0.06
U (95%)	± 0.21	± 0.28	± 0.09

Cx: Untreated centrifuged cuttings samples, *: not performed by the author of this thesis

As presented in Table 7.1, the raw cuttings have an average ROC wet of 9.82 ± 0.29 %, ROC dry of 15.02 ± 0.44 % and water content of 22.15 ± 0.14 %. These parameters decreased to 8.03 ± 0.21 %, 10.91 ± 0.28 % and 15.85 ± 0.09 % respectively (Table 7.2) after the cuttings were centrifuged at 2000 rpm for 2 min and the two top layers which consisted of a mixture of clay, water and oil (mud) were decanted.

7.2. Water and Oil Separation

The water and oil separation, which determine the efficiency of the treatments evaluated, are presented in this section. Conversion of rpm into RCF using Equation 6.1 is given in Table 7.3.

Table 7.3: Conversion of RPM to RCF

RPM	1500	2000	3000	4000
RCF	435	774	1741	3095

7.2.1. Water and Oil Separation by MudSplit Chemicals Alone

The values in the figures are based on the average of three samples analyzed with the retort. All the abbreviations used are explained in Table 7.4

Table 7.4: Abbreviations used in this section

Abbreviations	Explanations
Rx	Untreated raw cuttings
Rw	Raw cuttings samples treated without MudSplit, only water and centrifugation
RM2/ RM4	Raw cuttings samples treated with water, M2 or M4 and centrifuged
Cx	Untreated centrifuged cuttings
Cw	Centrifuged cuttings samples treated without MudSplit, only water and centrifugation
CM2/ CM4	Centrifuged cuttings samples treated with water, M2 or M4 and centrifuged

7.2.1.1. Treatment with Mudsplit vs without Mudsplit

The liquid fractions of samples RM2, Rw, CM2 and Cw (from left to right) decanted from the flasks after centrifugation are presented in Figure 7.1. These samples were centrifuged at 435 g and RM2 and CM2 were treated with 5 ml M2 each. The liquid fractions of raw cuttings samples were solids-free but present a thin layer of oil on the surface. The liquid fractions of centrifuged samples contained some solid particles. The liquid fractions of CM2 and RM2 samples are also colored due to the M2 chemicals. A greater amount of water was removed from RM2 compared to Rw whereas the amount of water removed from CM2 and Cw are approximately similar.

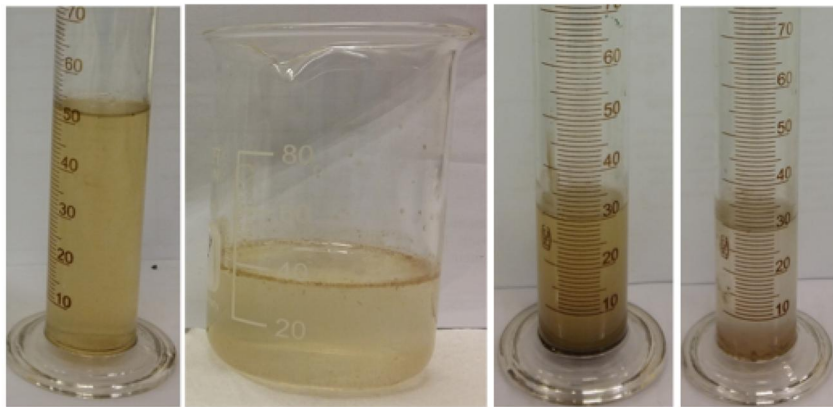


Figure 7.1: Liquid fractions of RM2, Rw, CM2 and Cw (from the left to the right)

Figures 7.2 and 7.4 illustrate a comparison between the oil and water content of these samples (RM2, Rw, CM2 and Cw) as well as for untreated cuttings (Rx and Cx). The ROC and water content of samples treated with water only (Rw and Cw) are presented to give an indication of the effect of M2 on cuttings.

As illustrated in Figure 7.2, the water content of the samples treated with M2 (RM2) is below those of the samples untreated or treated without M2 (Rx and Rw respectively). As indicated by the arrows, the water content decreased from an average of 22.15 % to 18.65 % after treatment with M2. There was an increase from 22.15 % to 25.59 % if M2 was not used.

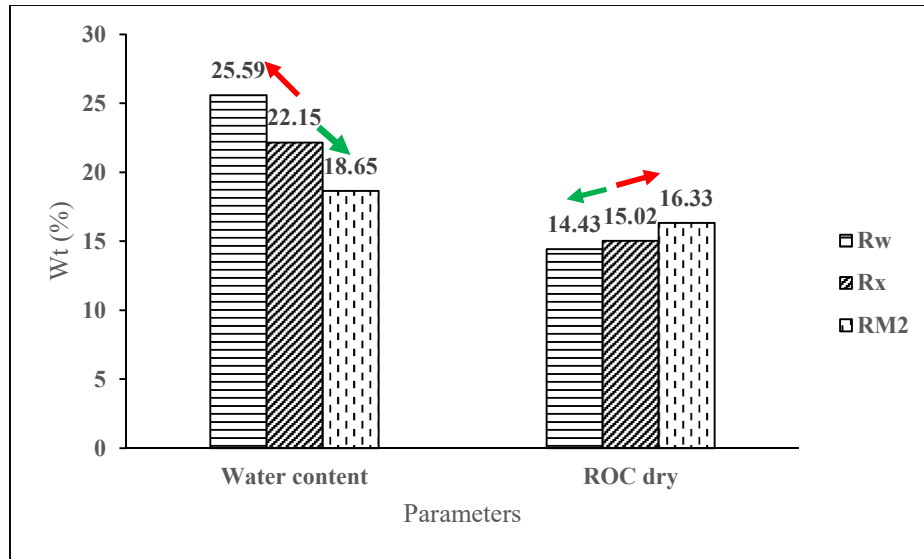


Figure 7.2: Oil and water content in untreated and treated raw cuttings

These results show that M2 chemical has an effect with respect to water separation. The high water content in Rw indicates that some of the water added into the samples during the treatment could not be removed by centrifugation alone. This can be due to the ability of clay to adsorb layers of water between their unit cells (Hensen and Smit, 2002). According to Wårnheim and Sjöblom (1985), the swelling clays or smectites which are present in significant amount in drill cuttings (see Table 2.3 in Chapter 2) are negatively charged at their face surfaces. Their edges are positively charged at alkaline, neutral and weakly acid pH. The face surfaces of these clay particles can attract and adsorb cations or polar molecules if they are not coated by oil wetting agents such as amines or Calcium alkyl aryl sulfonates (CaLAS) used in mud formulations (Wårnheim and Sjöblom, 1985). The water adsorbed in clay cuttings layers are difficult to separate. Høiland et al. (1985) demonstrated that a centrifugal force below 7000 g is unable of removing water from OBM cuttings. The RCF 435 g used to centrifuge the samples in this experiment is far below this value 7000 g. Therefore, no adsorbed water is expected to be removed.

However, for samples treated with M2, not only the additional water but also some of the water initially present in the cuttings were removed. This indicates that M2 may be able to destabilize the water-in-oil emulsions and facilitate water separation by centrifugation.

It is also worth noting that while agitating the samples after addition of M2, a viscosity drop was noticeable. The samples treated with M2 were less viscous compared to those without. As discussed in Chapter 2 (section 2.1.1.2.a. Composition of OBM), water-in-oil emulsions provide viscosity to the mud system and the destabilization of this emulsion will result in a viscosity drop. M2 is then capable of displacing the surfactants (emulsifiers and oil wetting agents) maintaining the original water-in-oil emulsion and inverting it to oil-in-water emulsion. The relocated oil wetting agents may be attracted to the face surfaces of the clay particles preventing a significant swelling process to occur.

Some of the oil kept in solution within the newly formed oil-in-water emulsion should be easy to remove by centrifugation. The thin layer of oil present at the surface of the liquid fraction (see Figure 7.1) also indicates that a little amount of oil was separated from the samples. However the results in Figure 7.2 illustrate that there was no oil separated from samples treated with M2 (RM2), their oil content increased by 1.31 % instead. A decrease of 0.59 % was obtained with samples treated without M2 (Rw). The oil separated in Rw and the oil present in RM2 liquid fraction are due to the centrifugal force applied (RCF 435 g). According to Høiland et al. (1986) and as discussed in Chapter 2 (section 2.1.4. Drilling Mud and Drill Cuttings Bonding System), the adhesively and capillary bound oil can be separated by centrifugal force. The 0.59 % oil separated here is then part of the adhesively/capillary bound oil.

For M2 treated samples, the oil-in-water emulsion formed after destabilization of the emulsifiers may have reacted with the displaced oil wetting agents and become strongly bound into the clay cuttings making them difficult to separate by centrifugation. Or they may have deposited into the cuttings during the resting time. Wårnheim and Sjöblom (1985) argued that a relatively short contact time should be provided to the washing system made of surfactants to prevent re-deposition of the emulsified oil.

The possible explanation for the increase in oil content of the samples treated with M2 observed after retort analysis despite the presence of oil in the liquid fraction can be based on their structure. Surfactants-like molecules have phase inversion (change in hydrophilicity or hydrophobicity) depending on some physical parameters.

A switchable deoiling agent (SDA) based on amine, formic acid, and formaldehyde solution, for example can switch from being hydrophobic to hydrophilic when a CO₂ gas was passed through a container with the surfactant solution and water. The same surfactant system could be reverse to be hydrophobic by addition of air, Ar or N₂ gas (Liu et al., 2014). (Velásquez et al., 2009). (2009) also stipulate that an increase in temperature decreases the hydrophilicity of nonionic surfactants and some anionic surfactants such as the alkyl polypropylene oxide sulfate type.

The high temperature up to 480 °C used during the retort process may have changed some of the M2 remaining in the cuttings into hydrophobic molecules. This results in the M2 able to mix with the oil fraction collected in the cylinder after condensation. This may have led to an erroneous reading of the results (Figure 7.3).

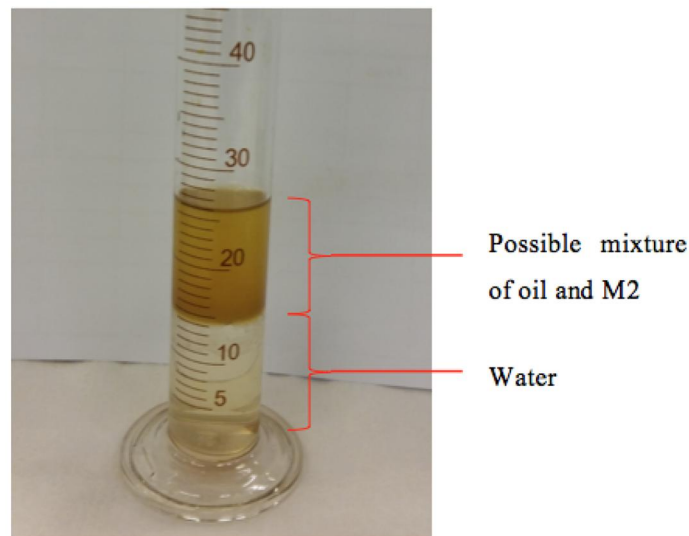


Figure 7.3: Liquid condensate from retort analysis of a sample treated with 5 ml M2 and centrifuged at 435 g.

For centrifuged samples, the water content of CM2 is approximately the same as for Cw with a value of 24.17% compared to 24.31% for Cw. These water contents are both high compared to the initial water content of the centrifuged cuttings which is 15.85 % (Figure 7.4).

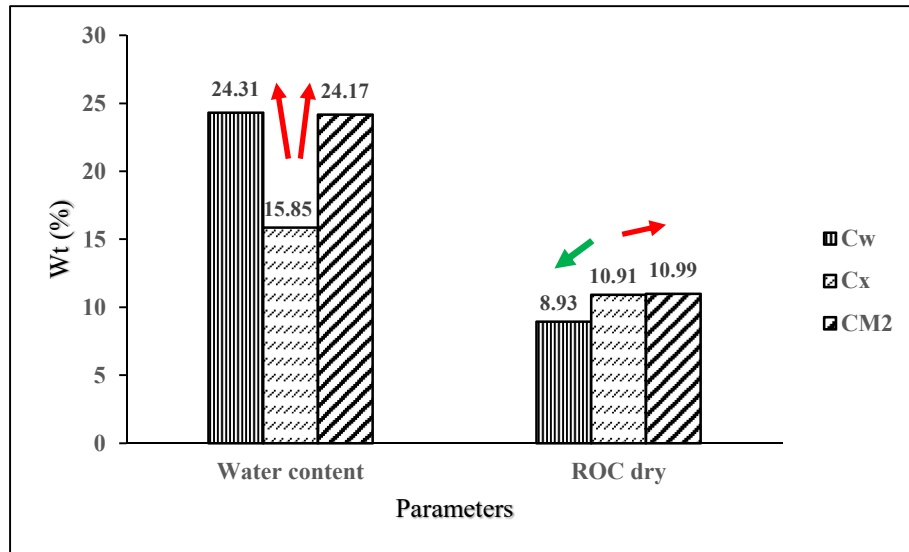


Figure 7.4: Oil and water content in untreated and treated centrifuged cuttings

The results indicate that M2 chemical is not efficient in treating centrifuged cuttings. The mud that M2 should destabilize has been decanted during the samples preparation process (centrifugation at 2000 rpm for 2 min and two top layers decanted). Therefore, there is no reaction between the samples and M2 chemical. The results are the same as for samples treated without M2, the water are adsorbed into the swelling clays.

For oil content, similar results as with raw cuttings are observed. The oil content of the samples increased after treatment with M2 (CM2) and decreased without M2 (Cw). The reason is as discussed previously. A phase inversion of M2 may have occurred during the distillation process with retort.

The liquid fractions of samples RM4 (left) and CM4 (right) are presented in Figure 7.5. RM4 liquid fractions were solids-free, orangish (due to the presence of M4) and contain a very thin layer of oil at the surface. CM4 was dark due to the presence of solid particles from the cuttings. Greater amount of water was removed from RM4 compared to CM4.

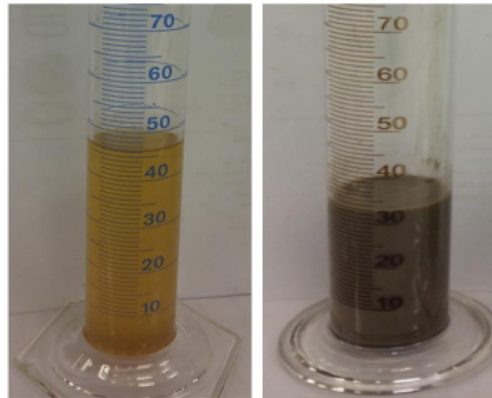


Figure 7.5: Liquid fractions of RM4 (left) and CM4 (right)

Figure 7.6 illustrates a comparison between the water and oil content of raw cuttings samples treated with M2 and M4. The water content of the samples RM4 with a value of 22.20 % is the same as the initial water content (22.15 %) and is high compared to that of RM2 (18.65 %).

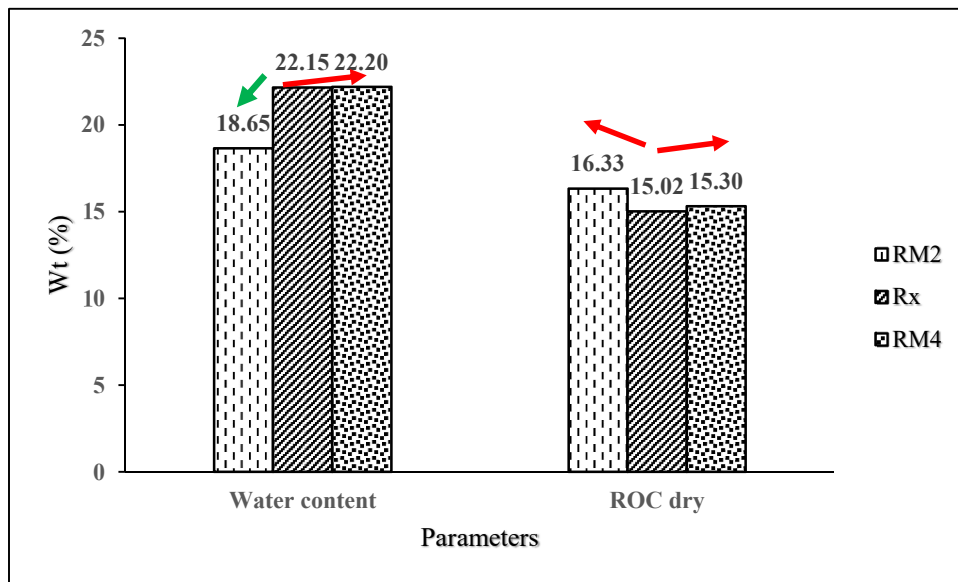


Figure 7.6: Comparison between M2 and M4 efficiencies in treating raw cuttings

It can be seen that M4 is not efficient in treating raw cuttings. It is not capable of displacing the emulsifiers but it can prevent clays to swell with water.

The water content of the cuttings (RM4) did not increase significantly. This may be due to adsorption of the M4 into the face surfaces of the clay particles.

The oil content of RM4 samples are similar to the initial oil content of the cuttings (15.30 % and 15.02 % respectively). It indicates that there was no oil separation in RM4 samples. The thin layer of oil presents in the liquid fraction in Figure 7.5 is due to centrifugation and very insignificant.

For centrifuged cuttings samples, the same results as with M2 were obtained with M4. The water content of CM4 samples increased from 15.85 % (initial value) to 24.17 % (Figure 7.7). This means M4 is also ineffective in treating centrifuged cuttings. It does not have any effect in the samples. Furthermore the face surfaces of the centrifuged cuttings may not be compatible for the M4 chemical to adhere into and resulting in swelling of cuttings with water.

The decrease in oil content in CM4 samples is due to light cuttings present in their liquid fractions (Figure 7.5). These light cuttings are probably coated with oil.

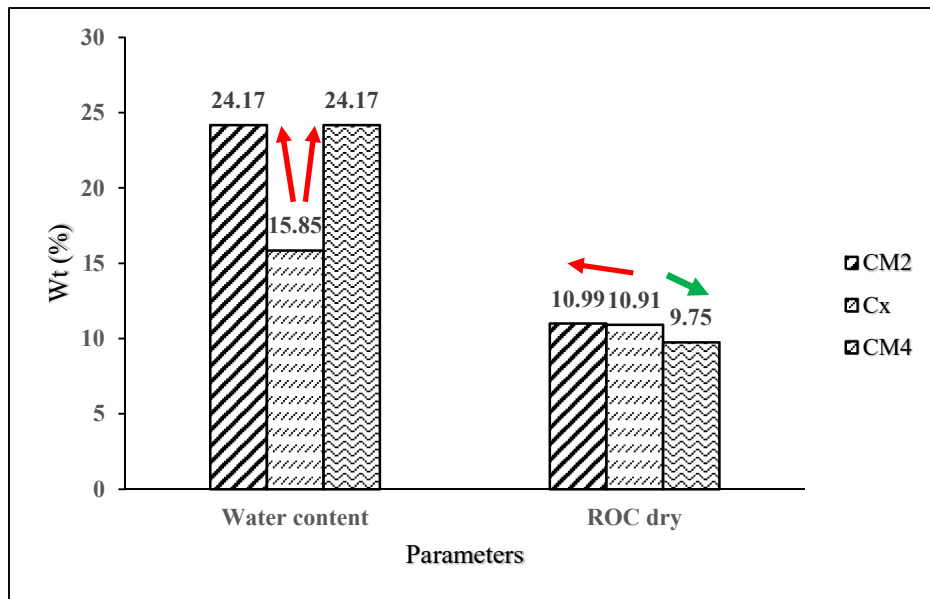


Figure 7.7. Comparison between M2 and M4 efficiencies in treating centrifuged cuttings

In terms of water separation, Figure 7.8 demonstrates the effectiveness of each chemical in treating raw and centrifuged cuttings. The data were calculated using Equation 6.5. Only raw samples treated with M2 present positive result with a removal of 15.80 %. The negative results mean there was increase in water content in the samples as presented and discussed earlier.

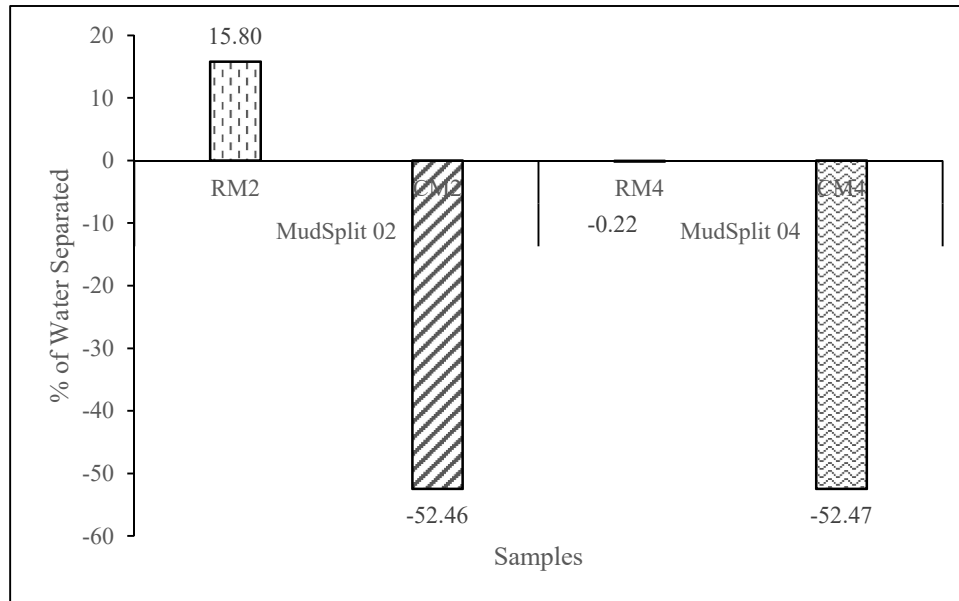


Figure 7.8: Water removal

7.2.1.2. Alternated Centrifugal Forces

M2 is the only chemical demonstrated to be efficient in treating raw cuttings (see Figure 7.8). Raw cuttings and M2 are tested for alternating centrifugal force.

Figure 7.9 illustrates liquid fractions removed from the raw cuttings samples treated with 2 mL M2 as a function of the centrifugal force. The liquid fractions obtained from samples centrifuged at 435 g and 774 g were clear and without any trace of oil nor solids (visual observation). The samples centrifuged at 1741 g were less clear and contained a floating oil layer (Figure 7.9 right). For samples treated at 3095 g, clay was observed in the decanted liquid. Figure 7.9 left shows the sludge fraction decanted from the samples treated at 3095 g after centrifugation. Each flask in Figure 7.9 right contains liquid removed from two samples treated.



Figure 7.9: Liquid/sludge fraction removed from RM2 samples centrifuged at 3095 g (left) and liquid fraction versus relative centrifugal force (right).

A separation of the treated samples with a centrifugal force between 435 g and 1741 g is considered to be suitable for the cuttings since. High centrifugal force (3095 g) resulted in removal of oily light cuttings which will be more difficult to treat. The highest amount of water separated was obtained with samples centrifuged at 1741 g.

The corresponding amount of water and oil removed from these samples in percentage is presented in Figure 7.10. These values were calculated from the water and oil on cuttings determined by retort analysis.

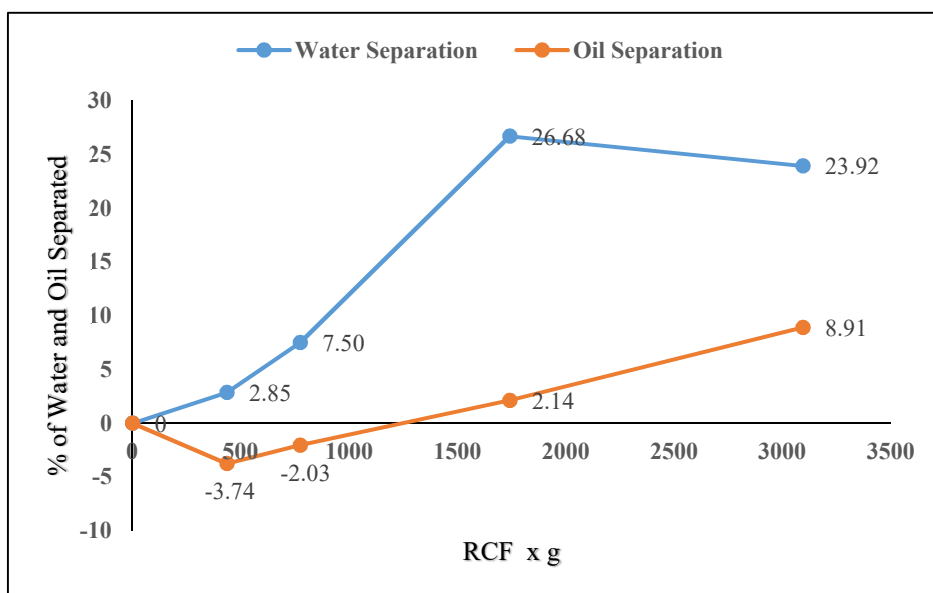


Figure 7.10: Percentage of water and oil separated versus relative centrifugal force.

As illustrated in Figure 7.10, the amount of water separated increases with increasing centrifugal force until a maximum removal of 26.68 % was reached at 1741 g. From this point, the curve levels off to 23.92% despite the high centrifugal force applied.

There was no oil separated from samples centrifuged at 435 g and 774 g. At 1741 g, 2.14% of oil were separated and 8.91% at 3095 g. The oil separated in samples centrifuged at 1741 g was due to the centrifugal force applied. The high oil separation obtained with samples centrifuged at 3095 g is most likely due to the oily light cuttings decanted along with the liquid fraction as shown in Figure 7.9 left. High centrifugal force is considered to be detrimental for the separation process using M2 chemical.

7.2.1.3. Alternated Dosage of MudSplit 02

The liquid fractions of samples treated with 1 mL, 2 mL, 3 mL, 5 mL and 10 mL M2 (from left to right) and centrifuged at 435 g are presented in Figure 7.11. All the liquid fractions were clear unless for samples treated with 1 mL M2 which contained solids causing the dark color. The highest water separation was observed with samples treated with 3 mL. The higher the amount of M2 added, the browner the color of the liquid fraction.



Figure 7.11: Liquid fractions decanted from RM2 samples centrifuged at 435 g versus M2 dosage.

The percentage of water and oil separated, calculated from data obtained from retort analysis of the residual cuttings, are plotted in Figure 7.12.

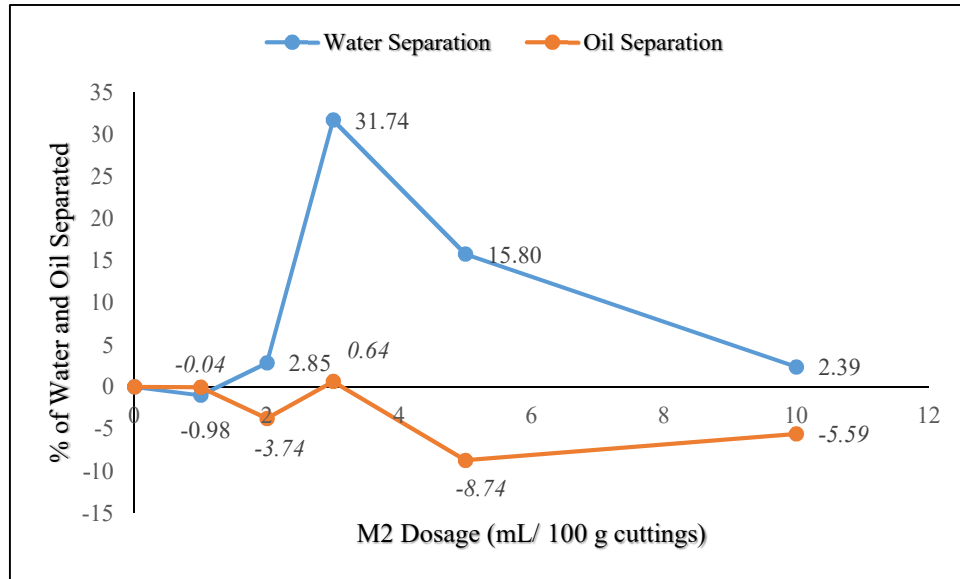


Figure 7.12: Percentage of water and oil separated from RM2 samples versus M2 dosage.

Figure 7.12 indicates that the water separation increases rapidly with increased concentration of M2 until it reaches a maximum value of 31.74 % at 3 mL. From that point, it decreased with increasing dosage of M2. Based on these results, 3 ml M2 per 100 g cuttings is considered to be the optimal dosage of M2 yielding high water separation.

For all the cases evaluated, there was no significant oil separation obtained. Almost all the samples indicated an increased oil content after the treatment. The variation of the oil content may depend on the amount of M2 left in the samples after each treatment.

7.2.1.4. Alternated Amount of Water

The centrifugal force of 1741 g in combination with 3 mL M2 was considered the optimal conditions. These parameters were used further while the water content was alternated.

Figure 7.13, from the left to the right, presents the liquid fractions of samples treated with 40 ml, 30 ml and 20 ml water. In each sample, a thin layer of oil was observed at the surface of a mixture of water and M2.

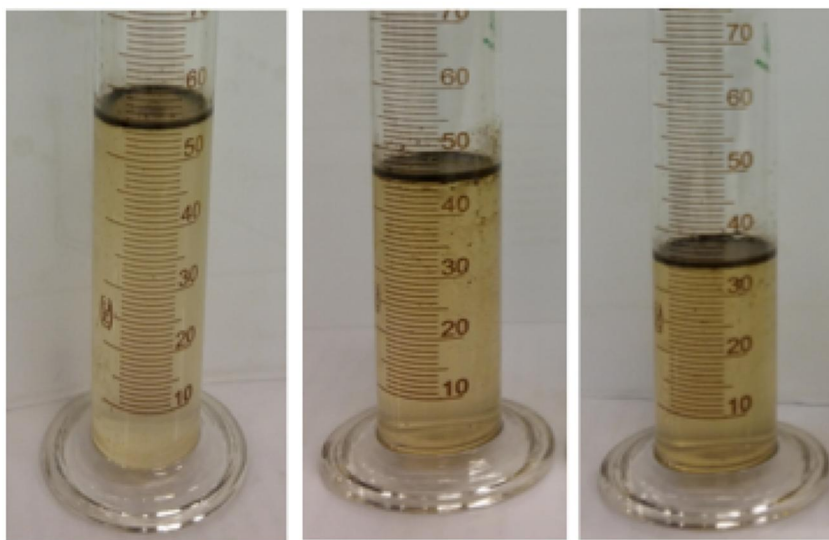


Figure 7.13: Liquid fractions decanted from RM2 samples centrifuged at 1741 g and treated with 40 ml, 30 ml and 20 ml water (from left to right).

The corresponding water and oil separation obtained with these samples are presented in Figure 7.14. Water separation of 40.18 % was found for samples treated with 40 ml water. When the amount of water was reduced to 30 ml, the water separation was the same (40.17 %). When 20 ml water was used, the separation efficiency of M2 decreased with a value of 34.29 %. Therefore, 30 ml of water will be used for later experiments because it resulted in similar water separation as with 40 ml. The amount of waste to be handled will also be decreased.

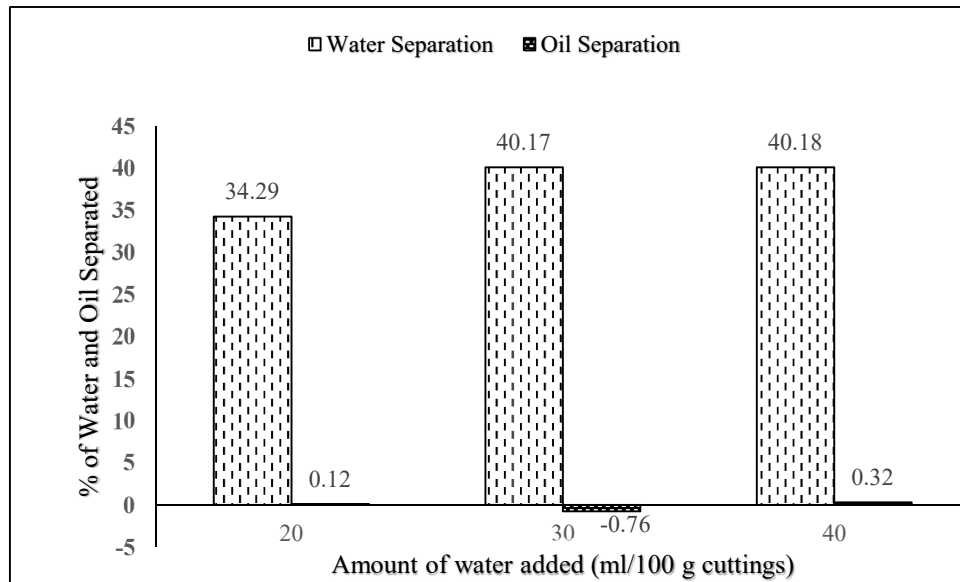


Figure 7.14: Percentage of water and oil separated from RM2 samples versus amount of water added.

Even though 1741 g resulted in an oil removal of about 2 % in samples treated with 2 ml M2. Here, with 3 ml M2 used, the oil removal was very insignificant. An increase in oil content was observed for samples treated using 30 ml water. This can be explained by the fact that the oil content of the samples may vary depending on the amount of M2 left in the samples after each treatment.

7.2.1.5. MudSplit 02 Pre-diluted with Water before Addition

The liquid fractions obtained were the same for the two cases: M2 pre-diluted or as in normal process. There was presence of thin layer of oil floating on the mixture of water and M2 (see Figure 7.13).

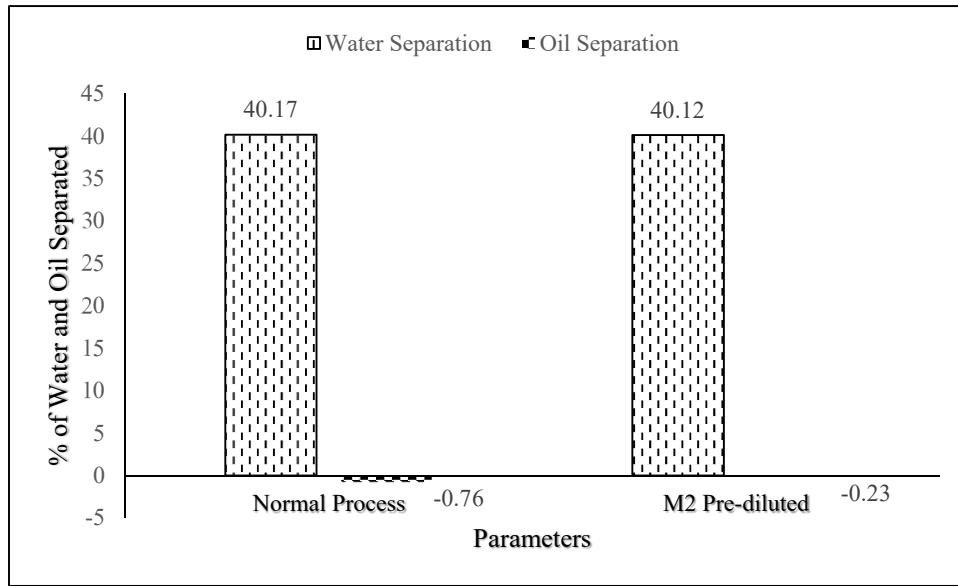


Figure 7.15: Percentage of water and oil separated from RM2 samples versus type of water addition process.

As indicated by Figure 7.15, mixing M2 with water prior to adding them into the samples did not enhance the water separation. The result obtained 40.12% was similar to that obtained with water addition followed by M2 addition (40.17%). In both cases, there was no oil separation.

7.2.2. Water and Oil Separation by MudSplit Combined with Solvent

The M2 successfully dewater drill cuttings. This may be an indication of mud destabilization which is the main application of M2. If water-in-oil emulsions are destabilized, the effect of solvent extraction may increase. This section is dedicated on evaluating the efficiency of the extraction process when M2 is used in combination with solvents. The abbreviations used are explained in Table 7.5.



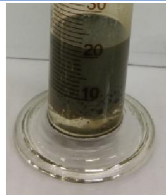

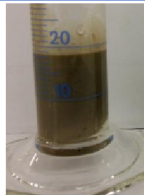
Table 7.5: Abbreviations used in this section



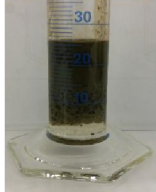
Abbreviations	Explanations
Hex/Acet	Samples treated with a mixture of hexane and acetone (3:1)
DCM	Samples treated with DCM
PE	Samples treated with petroleum ether
M2/435	Samples treated with M2 and centrifuged at 435 g RCF
M2/1741	Samples treated with M2 and centrifuged at 1741 g RCF
1741	Samples treated without M2 (solvent alone) and centrifuged at 1741 g RCF

The liquid fractions decanted after treatment with solvents as well as the observed layers are presented in Table 7.6. During the extraction process, the viscosity drop noticed after addition of M2 increased further when solvents were added whereas the samples treated with solvents alone were more viscous.

As illustrated in Table 7.6, the samples centrifuged at 435 g split into three layers: a mixture of clay and solvents at top layer, water in the midst and light cuttings at the bottom. For samples treated with petroleum ether, the light cuttings formed flocs (aggregation of particles). When stirred, the flocs floated to the surface. Samples centrifuged at 1741 g split into two layers, a mixture of solvent and clays floating on top of water. Almost no water was recovered from the samples treated with solvents alone, without M2. These results confirm again that M2 is effective for water separation.

Table 7.6: Layers distribution of the decanted liquid fractions

Solvents	Hexane/Acetone (3:1)			DCM		
Samples	M2/435	M2/1741	1741	M2/435	M2/1741	1741
Top layer	Solvent with clay	Solvent with clay	Solvent with clay	Solvent with clay	Solvent with clay	Solvent with clay
Mid layer	Water	-	-	Water	-	-
Bottom layer	Light cuttings	Water	Water	Light cuttings	Water	Water
Illustrations						

Solvents	Petroleum ether		
	M2/435	M2/1741	1741
Top layer	Solvent with clay	Solvent with clay	Solvent with clay
Mid layer	Water	-	-
Bottom layer	Light cuttings	Water	Water
Illustrations			

7.2.2.1. Water Separation

The water separation obtained from these samples are presented in Figure 7.16. The data for samples treated with hexane/acetone (3:1) is based on the average of three samples whereas the others (DCM and PE) are from one sample each.

For samples centrifuged at 435 g, the efficiency of M2 was reduced from 31.74 % to 27.53 %, and 27.08 % when a mixture of hexane/acetone (3:1) and petroleum ether were added respectively. For samples centrifuged at 1741 g, it decreased from 40.17 % to 20.93 % and 10.75 %. However, when M2 was used in combination with DCM, an increase in water separation was observed. The water separation are 51.35 % and 46.32 % for samples centrifuged at 435 g and 1741 g respectively. Høiland et al. (1986) also found that DCM is capable of dissolving other organic material in the cuttings in addition to oil and facilitating the discharge of water. For all the cases evaluated, solvent extraction alone is not favorable in separating water from cuttings, the added water was adsorbed into the clay cuttings resulting in increase of the water content.

Figure 7.16 illustrates also that centrifugation at 435 g is more efficient than at 1741 when the M2 is combined with solvent extraction. Among the three solvents tested, DCM is the most effective, followed by hexane/acetone (3:1) and petroleum ether.

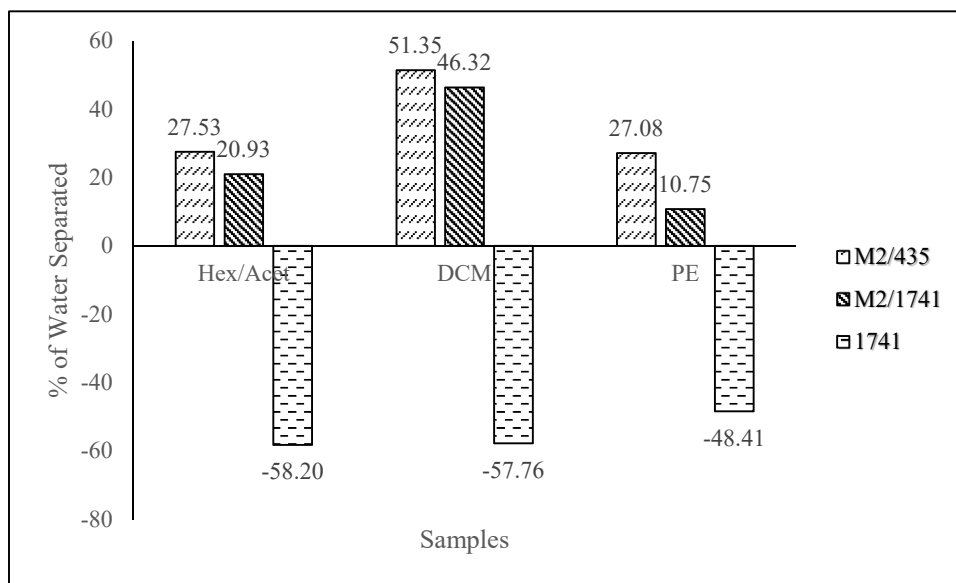


Figure 7.16: Water separation versus solvent or mixture of solvents used

7.2.2.2. Oil Separation

Figure 7.17 indicates that all the cases evaluated resulted in oil separation. The data for samples treated with hexane/acetone (3:1) is based on the average of two samples whereas the others (DCM and PE) are from one sample each.

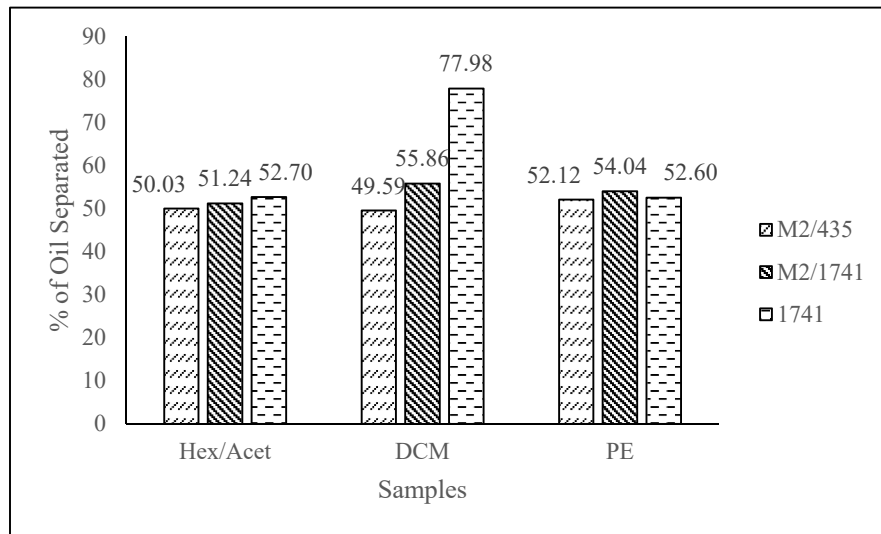


Figure 7.17: Oil separation versus solvent or mixture of solvents used

For hexane/acetone (3:1) and petroleum ether, the results from the three conditions evaluated (M2/435, M2/1741 and 1741) were approximately the same with an extraction efficiency of about 50 %. Addition of M2 seems to be only advantageous in terms of water separation. It does not affect the oil separation. Li et al. (2012) found that hexane/acetone (3:1) is able to remove 60 to 90 % of oil from contaminated soils (depending on the type of the petroleum hydrocarbons). However the solvent soil ratio of 6:1 used by these authors is significantly high compared to that used in this research. A solvent cuttings ratio of 1:10 was used in addition to the use of water (solvent diluted). A solvent soil ratio of 8:1 was used by Sui et al. (2014) to extract TPH from soils using petroleum ether and yielded in an extraction efficiency of 76 to 94 %. This solvent soil ratio is also very high compared to the one used in this thesis (1:10).

For DCM, there is a significant difference between the three cases evaluated. DCM is more efficient when used alone than combined with M2. It resulted in oil separation of 77.98% compared to 49.59% and 55.86% for samples treated with DCM in combination with M2 (centrifuged at 435 g and 1741 g respectively). This indicates that addition of M2 has negative effect when used with DCM.

The results also indicate that the base oil used for the formulation of OBM (in this case) has a good affinity to DCM rather than the other two solvents (hexane/acetone 3:1 and petroleum ether). According to Hansen (2007) and Riddick et al. (1986) solvent extraction relies on “like dissolves like” rule. The polarity of the base oil may be similar to that of DCM.

Chapter 8: Conclusion and Recommendation

The laboratory testing was carried out with the aim of demonstrating the possibility of using MudSplit chemicals in combination with decanter centrifuge as a pre-treatment method for drill cuttings. The main objective was to separate water from the cuttings and oil separation was considered to be additional advantage if achieved with the same process. The experiments served also to verify the effect of MudSplit, alone and in combination with solvent. Splitting mud emulsion may allow the solvent to work more efficiently which could in principle open up for more environmentally friendly solvents.

Of the two chemicals tested (M2 and M4), M2 was demonstrated to be effective on separating water from raw cuttings with a separation of 15.80 %. It was not efficient in treating centrifuged cuttings. This indicate that when more mud is present in the samples such as in raw cuttings, M2 is more efficient to separate water. If the top (mud) fraction from the barrel was also treated along with the solids from the bottom fraction, better results could be achieved. Although, there was no oil separation observed, the results give an indication of possible capability of M2 to destabilize OBM in cuttings.

A centrifugal force of 1741 g and a M2 dosage of 3 ml/ 100 g cuttings were found to be the parameters yielding optimal water separation of 40.17 %. Decreasing the water used during the extraction process into 30 ml did not affect the performance of M2 but 20 ml resulted in lower water separation of 34.29 %.

Diluting M2 with water or adding them directly into the samples did not have significant effect on M2 performance. A dosage ranging from 0.075 to 0.1 mL M2/mL water was found to be the optimum for treating 100 g cuttings. This dosage can be considered as very low.

When M2 was combined with solvent extraction, both water and oil separation were observed. Only oil could be extracted by solvents used alone. However the water separation decreased when hexane/acetone (3:1) and petroleum ether were used in combination with M2. It increased by 11.18 % and 6.15 % when DCM was used in addition to M2 and the samples were centrifuged at 435 g and 1741 g respectively.

The oil separation obtained was similar for samples treated with hexane/acetone (3:1) and petroleum ether with or without M2. But a substantial difference was observed for samples treated with DCM. Treatment with DCM without M2 was most effective in terms of oil removal with a value of 77.98 % compared to 49.59 % and 55.86 % for the samples treated with M2 and centrifuged at 435 g and 1741 g respectively.

Based on the main objective of this thesis, all of these results show the feasibility of using M2 followed by centrifugal separation as a pre-treatment method for OBM drill cuttings.

Recommendations

Due to time limitation, several parameters could not be evaluated to determine the optimum conditions for the method evaluated in this thesis. Therefore the following are recommended for further studies:

- The agitation is one parameter that should be studied further. During the laboratory testing, the agitation method used was manually and the mixing force was not constant. This parameter is of high importance because it affects the efficiency of M2. Wärnheim and Sjöblom (1985) stipulated that a rapid agitation is required while mixing a surfactant with the samples. But violent agitation increases the disintegration of the cuttings leading to the formation of small particles which may clog the capillaries containing trapped oil. A violent agitation also decreases the size of the water-in-oil emulsions droplet and making them more stable and difficult to destabilize (Wärnheim and Sjöblom 1985). An equipment which provides controllable agitation is then necessary.
- The contact time is also another parameter which may be of interest. It can affect the efficiency of M2 and should be studied further.
- The mechanism of water separation by the M2 should be investigated to provide better understanding of the effect of this chemical on OBM cuttings.
- The liquid fraction and residual cuttings should be analyzed for oil content with a method which does not involve heat. This would indicate whether M2 provides oil separation or not. Retort analysis seems to not be suitable for determining the oil content of samples treated with MudSplit chemicals.
- The possibility of recycling or reusing recovered liquid phase containing M2 into the separation process can be investigated.
- The water and oil extraction based on combining M2 with solvents may serve as potential for a stand alone full treatment unit localized onshore. Further studies should be carried out for this purpose.

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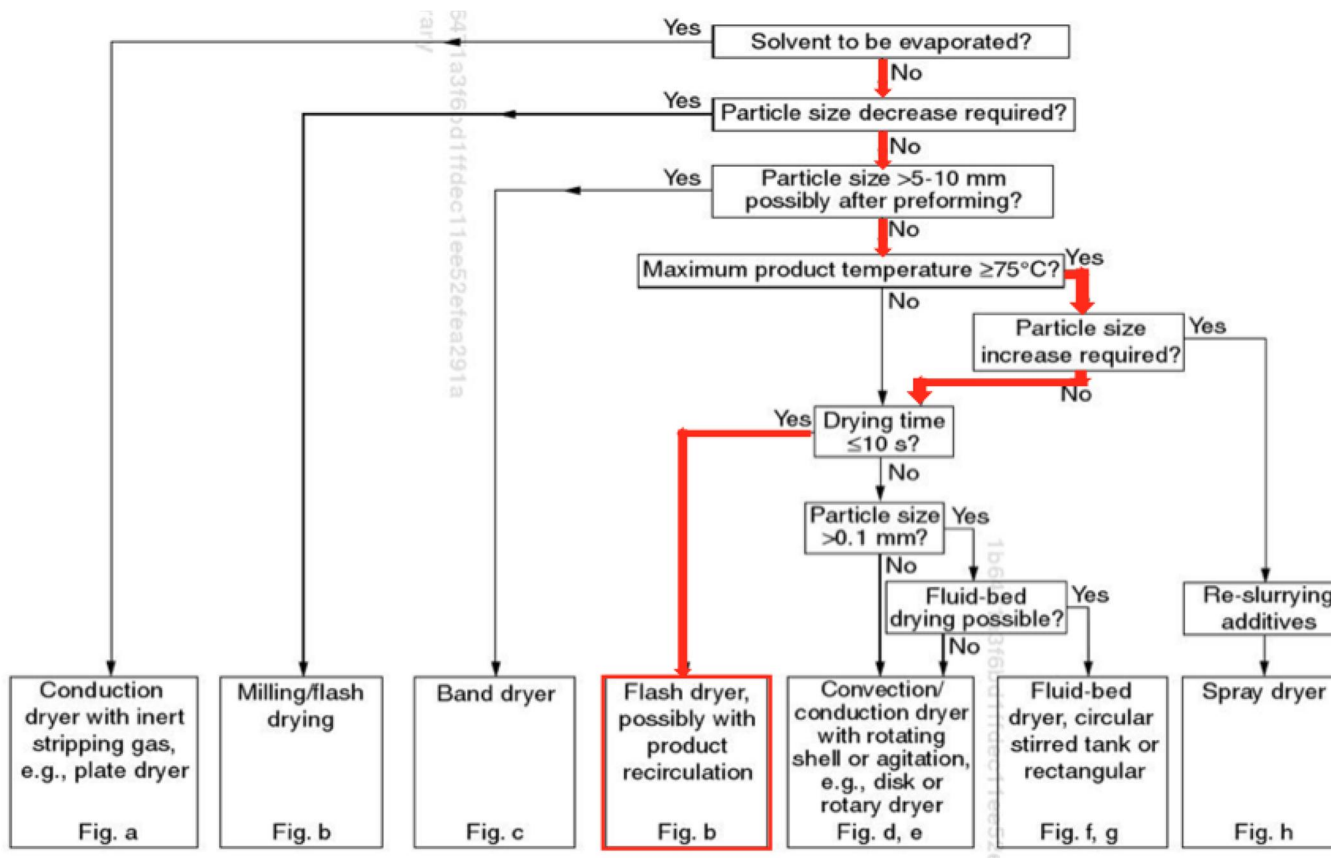
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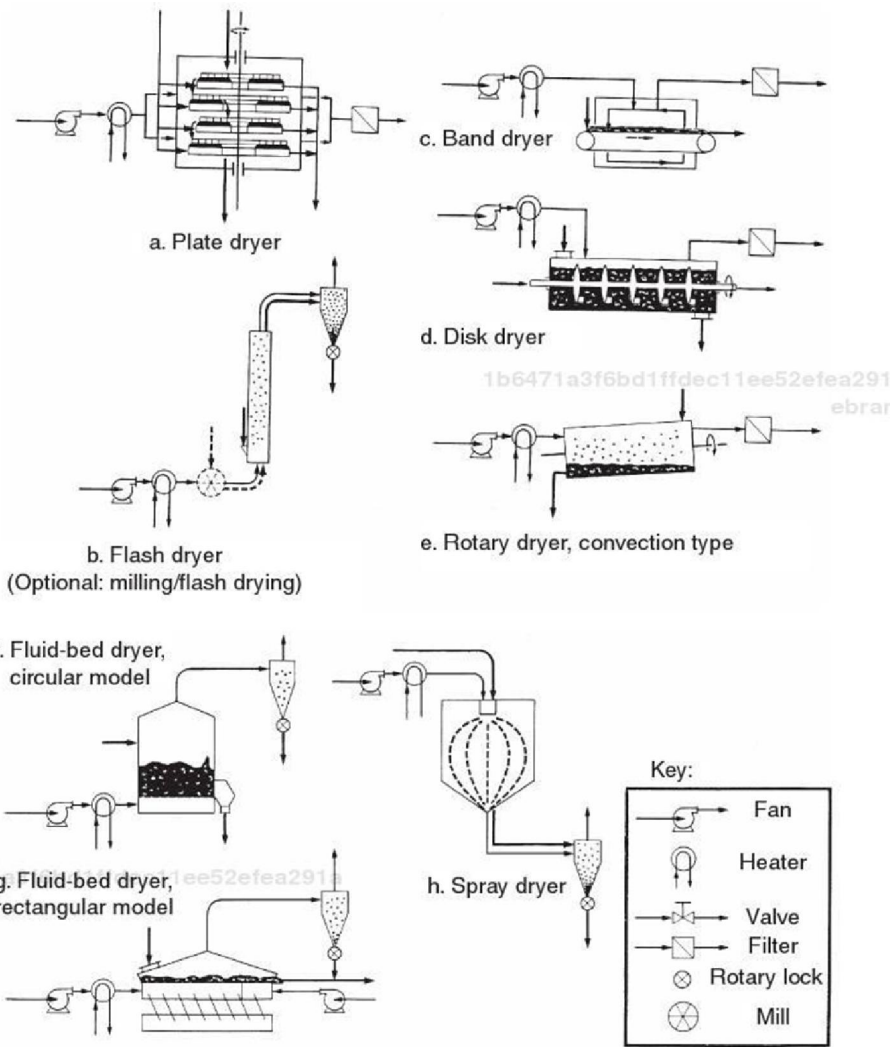
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Appendix

A1: Decision tree for choosing a suitable dryer and the corresponding figure of each type





B1: Retort analysis of untreated raw and centrifuged cuttings

Samples	Rx			Cx		
	Wet cuttings (g)	87,50	86,00	86,10	95,50	96,40
Water (ml)	19,50	19,00	19,00	15,20	15,30	15,10
Oil (g)	8,40	8,40	8,70	7,80	8,00	7,60
Dry cuttings (g)	57,60	55,50	56,70	71,10	71,90	71,40
Water content (%)	22,29	22,09	22,07	15,92	15,87	15,76
ROC wet (%)	9,60	9,77	10,10	8,17	8,30	7,93
ROC dry (%)	14,58	15,14	15,34	10,97	11,13	10,64

B2: Retort analysis of raw cuttings samples treated with water only and with M2

Samples	Rw			RM2 5ml		
Wet cuttings (g)	80,30	81,90	82,10	80,40	80,40	84,60
Water (ml)	21,00	20,50	21,00	14,50	14,80	16,50
Oil (g)	6,50	7,70	7,80	9,00	8,80	9,60
Dry cuttings (g)	48,80	51,70	51,70	55,50	55,20	57,00
Water content (%)	26,15	25,03	25,58	18,03	18,41	19,50
ROC wet (%)	8,09	9,40	9,50	11,19	10,95	11,35
ROC dry (%)	13,32	14,89	15,09	16,22	15,94	16,84

B3: Retort analysis of centrifuged cuttings treated with water only and with M2

Samples	Cw 1500			CM2 5ml		
Wet cuttings (g)	82,90	77,10	86,90	80,70	87,40	88,10
Water (ml)	20,00	19,00	21,00	18,50	21,50	22,00
Oil (g)	4,90	4,90	5,30	6,00	6,50	6,30
Dry cuttings (g)	57,30	52,30	59,70	54,80	58,10	58,10
Water content (%)	24,13	24,64	24,17	22,92	24,60	24,97
ROC wet (%)	5,91	6,36	6,10	7,43	7,44	7,15
ROC dry (%)	8,55	9,37	8,88	10,95	11,19	10,84

B4: Results from retort analysis of raw and centrifuged cuttings treated with M4

Samples	RM4 5ml			CM4 5ml		
Wet cuttings (g)	86,20	85,80	84,80	88,70	88,90	88,90
Water (ml)	19,00	19,00	19,00	22,00	20,00	22,40
Oil (g)	8,60	8,90	8,50	5,90	5,90	5,60
Dry cuttings (g)	57,30	56,60	56,00	59,30	59,70	59,50
Water content (%)	22,04	22,14	22,41	24,80	22,50	25,20
ROC wet (%)	9,98	10,37	10,02	6,65	6,64	6,30
ROC dry (%)	15,01	15,72	15,18	9,95	9,88	9,41

B5: Retort analysis of raw cuttings samples treated with alternated centrifugal force

Samples	RM2 774 g			RM2 1741 g			RM2 3095 g		
Wet cuttings (g)	83,10	84,70	84,50	83,60	79,40	83,00	81,40	83,10	83,60
Water (ml)	16,20	16,50	19,00	15,00	12,00	13,00	14,00	14,00	13,80
Oil (g)	8,00	9,10	8,90	8,90	7,90	8,70	7,40	8,70	8,20
Dry cuttings (g)	57,60	57,00	55,20	58,00	56,80	58,60	58,70	58,70	60,20
Water content (%)	19,49	19,48	22,49	17,94	15,11	15,66	17,20	16,85	16,51
ROC wet (%)	9,63	10,74	10,53	10,65	9,95	10,48	9,09	10,47	9,81
ROC dry (%)	13,89	15,96	16,12	15,34	13,91	14,85	12,61	14,82	13,62

B6: Retort analysis of raw cuttings samples treated with alternated M2 dosage

Samples	RM2 1ml			RM2 2ml			RM2 3ml			RM2 10 ml		
Wet cuttings (g)	83,30	81,20	83,70	83,40	84,70	82,90	80,40	78,70	84,10	83,90	85,10	85,40
Water (ml)	19,00	18,50	18,00	19,50	17,50	17,00	12,80	11,00	13,00	18,00	18,50	18,50
Oil (g)	8,30	7,80	8,40	8,50	8,80	8,50	9,20	7,90	8,90	8,50	9,00	9,10
Dry cuttings (g)	54,60	53,20	55,20	53,70	56,00	55,90	58,20	56,30	59,60	55,60	56,10	56,00
Water content (%)	22,81	22,78	21,51	23,38	20,66	20,51	15,92	13,98	15,46	21,45	21,74	21,66
ROC wet (%)	9,96	9,61	10,04	10,19	10,39	10,25	11,44	10,04	10,58	10,13	10,58	10,66
ROC dry (%)	15,20	14,66	15,22	15,83	15,71	15,21	15,81	14,03	14,93	15,29	16,04	16,25

B7: Retort analysis of raw cuttings samples treated with alternated amount of water

Samples	RM2 40ml H ₂ O			RM2 30ml H ₂ O			RM2 20ml H ₂ O			Water and M2 pre-mixed		
Wet cuttings (g)	76,20	76,40	79,10	76,20	79,40	77,60	78,80	79,20	80,40	72,80	80,10	80,70
Water (ml)	10,10	10,10	10,50	10,00	10,40	10,50	11,20	11,50	12,00	9,50	10,50	11,00
Oil (g)	8,40	8,50	8,70	8,70	8,80	8,50	8,80	8,80	8,50	8,30	8,60	9,10
Dry cuttings (g)	56,30	56,00	58,70	56,40	58,30	57,10	57,70	57,60	58,70	54,10	59,50	59,20
Water content (%)	13,25	13,22	13,27	13,12	13,10	13,53	14,21	14,52	14,93	13,05	13,11	13,63
ROC wet (%)	11,02	11,13	11,00	11,42	11,08	10,95	11,17	11,11	10,57	11,40	10,74	11,28
ROC dry (%)	14,92	15,18	14,82	15,43	15,09	14,89	15,25	15,28	14,48	15,34	14,45	15,37

B8: Retort analysis of samples treated with Hex/Acet (3:1)

Samples	RM2+Hex/Acet 1741 g			RM2+Hex/Acet 435 g			Hex/Acet 1741		
	Wet cuttings (g)	86,80	84,50	85,60	76,00	75,90	88,60	77,20	77,40
Water (ml)	15,50	14,50	15,00	13,00	12,00	13,50	26,00	27,00	28,00
Oil and solvent g	7,80	8,40	8,40	9,30	8,10	8,80	4,50	4,70	4,40
Dry cuttings (g)	59,40	58,80	60,10	52,20	54,10	64,60	45,10	44,00	42,60
Water content (%)	17,86	17,16	17,52	17,11	15,81	15,24	33,68	34,88	36,55

B9: Retort analysis of samples treated with DCM and PE

Samples	RM2+DCM 1741	RM2+DCM 435	DCM 1741	RM2+PE 1741	RM2+PE 435	PE 1741
Wet cuttings (g)	79,90	92,80	78,70	86,00	77,40	79,10
Water (ml)	9,50	10,00	27,50	17,00	12,50	26,00
Oil and solvent (g)	8,80	9,80	5,40	7,20	7,50	4,10
Dry cuttings (g)	59,60	69,90	43,10	59,00	55,00	46,70
Water content (%)	11,89	10,78	34,94	19,77	16,15	32,87

C1: Soxtec analysis of samples treated with Hex/Acet (3:1)

Samples	M2+Hex-Acet 1741 g	M2-Hex-Acet 1741 g	M2-Hex-Acet 435 g	M2-Hex-Acet 435 g	Hex-Acet 1741 g	Hex-Acet 1741 g
Cuttings (g)	5.13	5.15	5.31	5.06	5.13	5.62
Oil (g)	0.35	0.40	0.38	0.40	0.38	0.38
ROC (%)	6.78	7.87	7.12	7.89	7.43	6.78

C2: Soxtec analysis of samples treated with DCM and PE

Samples	M2-DCM 1741 g	M2-DCM 435 g	DCM 1741 g	M2-PE 1741 g	M2-PE 435 g	PE 1741 g
Cuttings (g)	5.14	4.29	5.35	5.13	5.16	5.16
Oil (g)	0.34	0.32	0.18	0.35	0.37	0.37
ROC (%)	6.63	7.57	3.31	6.90	7.19	7.12