

SOLID-LIQUID SEPARATION OF WASTE PETROLEUM DRILLING MUD IN OFFSHORE NEWFOUNDLAND AND LABRADOR

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Solid-Liquid Separation of Waste Petroleum Drilling Mud in Offshore Newfoundland and Labrador

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Executive Summary

Waste drilling mud is the second largest waste volume produced in the oil and gas exploration industry after wastewater and cannot be disposed of or landfilled without proper treatment to meet regulation requirements. Several contaminants are present in the waste drilling mud and cuttings, including petroleum hydrocarbons, heavy metals, BTEX (benzene, toluene, ethyl benzene, and xylenes), polycyclic aromatic hydrocarbons (PAHs), and other hazardous materials typically originating from the base drilling fluids. Strict environmental regulations are in place regarding the disposal of the waste drilling mud and cuttings to minimize their effect on the environment. Therefore, the waste drilling mud and cuttings must be properly treated before being released into the environment. Different technologies have been proposed for waste drilling mud remediation; however, most of them are unable to meet the strict environmental regulation limits.

In this report, physical treatment technologies (centrifuge and surfactant-enhanced washing) were employed to first, separate the liquid and solid phases of the waste drilling mud and then, to remove the petroleum hydrocarbons. The main aim of this study is to investigate the ability of surfactant-enhanced washing to treat the waste drilling mud and remove the hazardous hydrocarbons to meet the strict environmental regulations. The specific objectives of the study are to: (1) characterize the waste drilling mud using particle size distribution, X-ray diffraction (XRD), induced coupled plasma optical emission spectrometer (ICP-OES), and gas chromatography GC analyses; (2) screen and select the best surfactants for drilling mud remediation using interfacial tension and sorption analyses; and (3) experimentally determine the impacts of significant factors on the efficiency of surfactant-enhanced washing.

Part I. Literature Review

Drilling fluids are used to prevent the blowouts, balance and control formation pressure, minimize formation damage and corrosion, lubricate, cool, and remove the drill cuttings from the well by transporting them through the drill string, and up the annulus to the surface (Shaikh, 2010). The drilling mud is separated from the drill cuttings in a shale shaker. The drilling mud is a solid-liquid slurry with very high viscosity, high oil content, heavy metals, and other ingredients such as bentonite, barite, and other polymers (Khodja et al., 2010; Wada et al., 2006). Figure 1 illustrates the drilling fluid injection and recovery system.

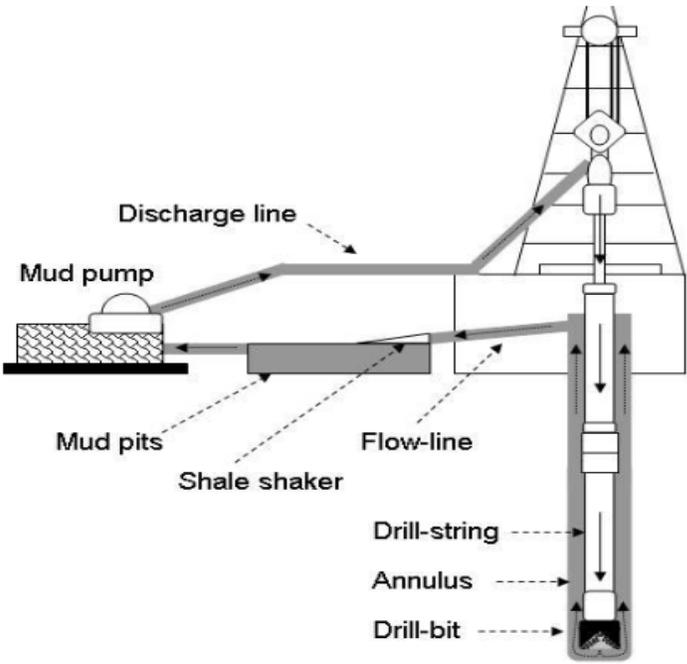


Fig. 1 – Drilling fluid injection and recovery system (Growcock and Harvey, 2005)

The composition of the waste drilling mud is complex and dependent on the type of the drilling fluid in use. The geological formation of the drilled rock can also change the composition of the waste drilling mud. For instance, deeper wells increase the complexity of the produced drilling mud (Fink, 2011; Pettersen, 2007). The drilling fluid types are being discussed as they affect the metal concentration and contamination of the waste drilling mud. There are two primary types of drilling fluids: non-aqueous drilling fluids (NADFs) and water based fluids (WBFs) (Hossain and Al-Majed, 2015). A typical composition of non-aqueous drilling fluids is illustrated in Figure 2(a). Figure 2(b) illustrates a typical composition of water-based drilling fluids (Force, 2009).

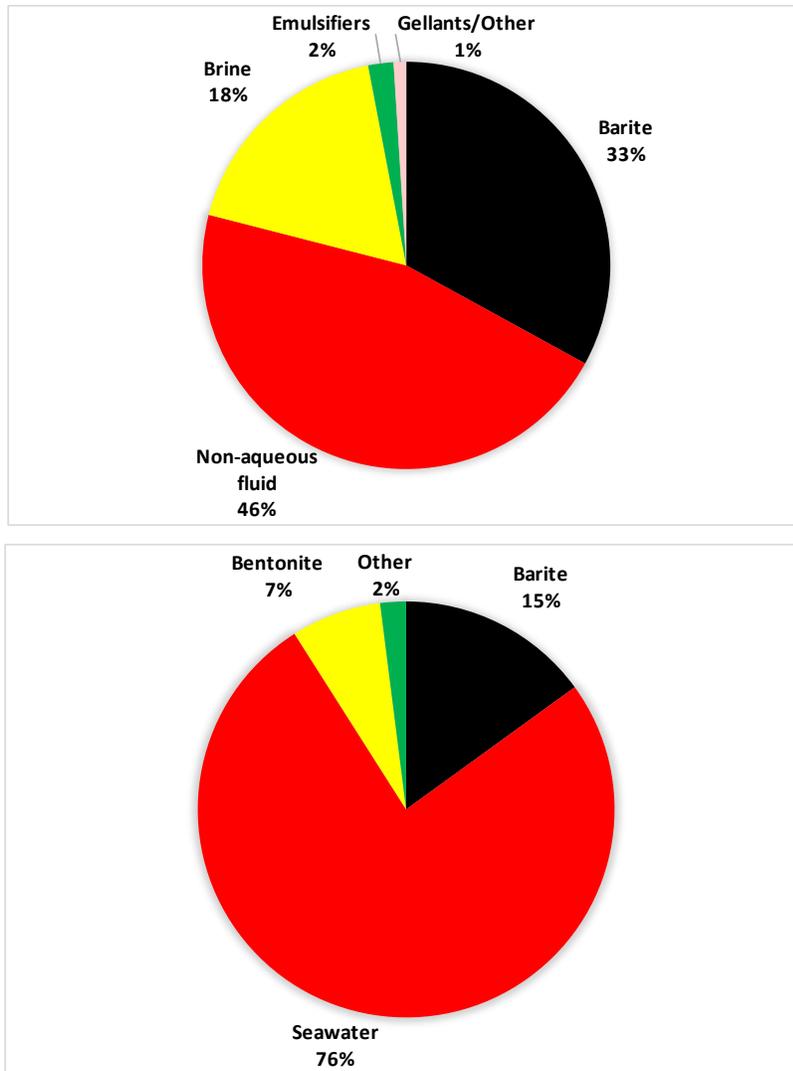


Fig. 2 – Typical composition of the (top) non-aqueous and (bottom) water-based drilling fluids (Force, 2009)

- **Physical treatment technologies**
 - **Centrifugal separation**

Waste drilling mud is a solid-liquid mixture. Centrifugal separation has been widely accepted as a fast and efficient method for solid-liquid separation (Bobo and Hoch, 1954); however, the technology is less effective at removal of contaminants bound to the surface of the fine solid phase of the waste drilling mud. In addition, the centrifuge must be operated carefully as there is a risk

of further dispersing fines into the liquid phase. As such, centrifuges could only be considered as a pre-treatment process to separate liquid from the solid phase.

- **Supercritical fluid extraction**

Supercritical fluid extraction (SFE) has been tested for the removal of various forms of hydrocarbons from the waste drilling mud. In supercritical fluid extraction, a solvent is heated and compressed to above the critical temperature and critical pressure (Saldana et al., 2005). Supercritical fluids have liquid-like densities, gas-like viscosities, and zero surface tension as well as pressure-dependent solvating power (McHugh and Krukonis, 2013). The high diffusivity of the supercritical fluid extraction process can improve the mass transfer and generates a more rapid rate for hydrocarbon and oil removal and recovery from porous materials such as waste drilling mud (Lopez-Gomez, 2004). This method has several advantages over some common remediation technologies, including lower solvent usage, shorter extraction time and most notable, easy separation of pollutants from the solvent by a slight change of temperature and/or pressure (Saldana et al., 2005). In contrast, there are some disadvantages that should be resolved to increase the application of this method for the waste drilling mud remediation. Depending on the type of solvent, there may be safety issues regarding the pressure and temperatures at critical conditions. It also requires high capital investment (Lopez-Gomez, 2004).

Carbon dioxide (CO₂) is the most widely used supercritical fluid due to its non-flammability and being chemically inert (Thanyamanta, 2003). Carbon dioxide is considered a non-toxic solvent with low adverse environmental impacts that possesses relatively low critical temperature and pressure ($T_c = 31^\circ\text{C}$ and $P_c = 74$ bar) (Saldana et al., 2005; Lopez-Gomez, 2004). It is also available in its high purity at a relatively low cost and it can be easily removed from the solid phase after

the extraction process. Modifiers, normally solvents, such as methanol, toluene, and acetone, are added either directly to the solid phase before the supercritical extraction process, or added to the supercritical fluid using a separate modifier pump.

Various studies have been conducted to investigate the possibility of using the supercritical fluid extraction process for the drilling waste remediation. Research on drilling mud treatment using supercritical fluid extraction are summarized in Table 1

Table 1 - Supercritical fluid extraction processes tested for waste drilling mud and cuttings

Supercritical Fluid	Pressure (bar)	Temperature (°C)	Efficiency (%)	Reference
HFC 134a and Propane	44.8	121	98	(Eldridge, 1996)
CO ₂	100	35	95	(Saintpere and morillon-Jeanmaire, 2000)
CO ₂	124	50	96	(Odusanya and Guigard, 2002)
Propane and Butane	34.5	23	96	(Seaton and Hall, 2005)
CO ₂	145	40	98	(Street and Guigard, 2006; Street et al., 2007)
CO ₂	200	79.5	49.1	Goodarznia and Esmailzadeh, 2006

- **Surfactant-enhanced washing**

Surfactants are surface active compounds containing a hydrophilic head and a hydrophobic tail (Urum and Pekdemir, 2004; Urum et al., 2005) and are categorized, based on the net charge of the hydrophilic head group, to anionic, cationic, non-ionic, and zwitterionic surfactants (Park and

Bielefeldt, 2003). Surfactants possess specific physical properties such as solubility, surface tension, and critical micelle concentration (CMC) that can significantly affect surfactant-enhanced washing processes. Figure 3 illustrates how these physical properties typically change with increasing surfactant concentration. Critical micelle concentration is the most important property of the surfactant and from an economic perspective, lower CMC values are desirable.

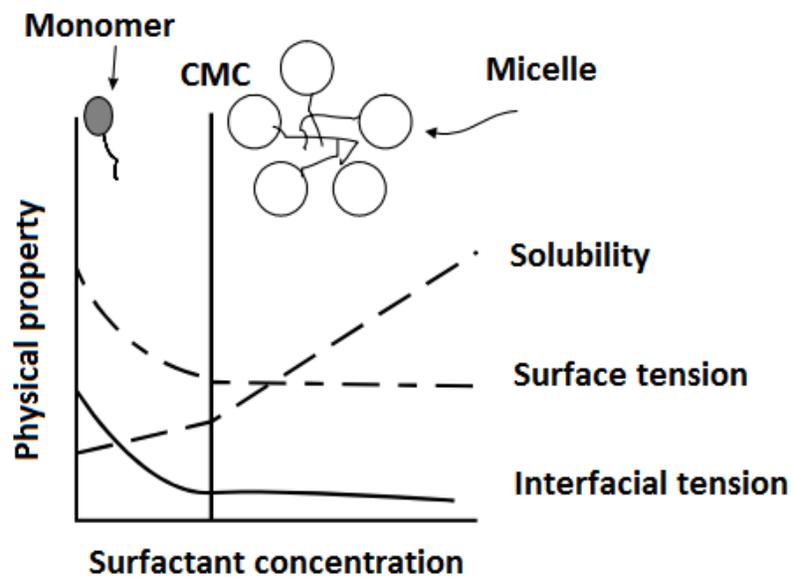


Fig. 3 – Surfactant properties with surfactant concentration (Mulligan et al., 2001)

Generally, the removal mechanisms involved in surfactant-enhanced washing processes are categorized into three main streams: roll-up, snap-off or emulsification, and solubilisation mechanisms (Childs et al., 2005).

Roll-up involves the adsorption of the surfactant at the oil-water interface, thus increasing the contact angle between the oil and the solid phase (θ) and decreasing the interfacial tension between oil and water ($\gamma_{o/w}$). In this mechanism, oil can be removed from the surface with minimal mechanical agitation (energy). Figure 4 illustrates the roll-up mechanism. By adding surfactant, the contact angle between the oil and the solid surface increases and thus, it would be easier to remove the entire oil droplet from the solid surface by mechanical agitation.

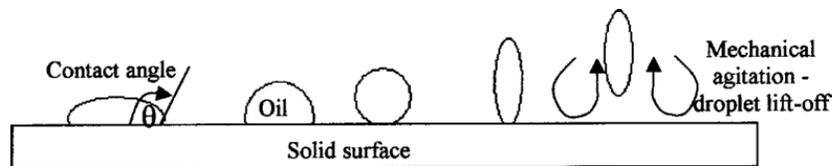


Fig. 4 – Roll-up mechanism for oil removal (Childs et al., 2005)

Snap-off occurs when the contact angle is not high enough for the entire droplet to detach from the substrate, but a portion breaks off the deposited oil film, as shown in Figure 5. Interfacial tension of the oil portion and the water decreases in the presence of the surfactant and thus, a portion of the oil can be easily removed from the bulk using mechanical force. The snap-off mechanism is related to the interfacial tension, by the work of cohesion (W_c), $W_c = 2 \gamma_{o/w}$ (Childs et al., 2005). Similar to roll-up, the snap-off oil removal is facilitated by lower oil/water interfacial tension because less mechanical energy is required to overcome the work of cohesion of the oil.

Roll-up and snap-off mechanisms are desirable for oil removal as (i) the oil is liberated from the surface as a free phase top layer that can be skimmed from the bath, and (ii) they require low surfactant concentrations (less environmental risks and more economical feasibility). This

mechanism happens in concentrations less than or slightly over the surfactant critical micelle concentration.

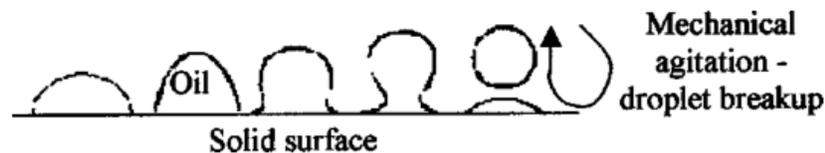


Fig. 5 – Snap-off mechanism for oil recovery from solid surface, also known as emulsification

(Childs et al., 2005)

Solubilisation mechanism is based on the partition of the oil molecules inside the hydrophobic core of micelles. This mechanism is only relevant at high surface concentrations when large numbers of micelles are present (Childs et al., 2005).

Surfactant-enhanced washing process has been considered an easy and cost-effective method (Chu, 2003; Han et al., 2009; Iturbe et al., 2004; Torres et al., 2005; Torres et al., 2006; Zacarias-Salinas et al., 2013) which has widely been employed for drilling mud treatment. In this project, the most suitable surfactant was selected based on two analyses: interfacial tension analysis and soil sorption analysis. Furthermore, the surfactant-enhanced washing experiments (with the selected surfactant) were conducted after centrifuging the waste drilling mud to investigate the efficiency of the process and the effects of significant parameters.

In the next parts of the report, characterization of the waste drilling mud, surfactant selection based on the two analyses, and the results of the surfactant-enhanced washing of the waste drilling mud are reported in detail.

Part II. Waste Drilling Mud Characterization

The properties of the waste drilling mud can significantly affect the efficiency of the surfactant-enhanced washing process. These factors include, but are not limited to, pH, composition, particle size distribution, and types of contaminants on the solid surface (Mulligan et al., 2001). The characterization of the waste drilling mud is listed in Table 2. Solid particle size analysis was conducted for 10 samples to achieve consistent data and the average amounts are presented. All other analyses, such as pH and moisture content tests were conducted four times and the average values are presented in Table 2. The solid surface charge is more negative at higher pH values (Bohn et al., 2015; Paria and Khilar, 2004; Farn, 2008). The pH was measured to be approximately nine, hence the surface charge is expected to be negative. This finding shows that cationic surfactants have high potentials for adsorption to the waste drilling mud sample while anionic surfactants are less likely to face loss.

Table 2 – Waste drilling mud sample characterization

Characteristic		Value
Moisture (%)		13.49 ± 0.77
Solid percent (mass/mass %)		97
pH		8.96 ± 0.26
Electrical conductivity (µS)		839 ± 9
Density g/cm ³		1.97 ± 0.20
Solid particle size	Average mean size (µm)	25.62 ± 5.47
	Average median size (µm)	16.92 ± 2.22
BET surface area (m ² /g)		1.92 ± 0.26

C10-C16 hydrocarbons (mg/kg)	65,298 ± 470
C16-C34 hydrocarbons (mg/kg)	34,320 ± 613
C34-C50 hydrocarbons (mg/kg)	1,800 ± 300
Total petroleum hydrocarbons (mg/kg)	101,418 ± 478

Figure 6 illustrates the particle size distribution of the waste drilling mud solid particles before any processing or treatment. The y-axis is the % by volume of the corresponding particle size shown on the x-axis. This distribution shows the pattern of fine particles for the waste drilling mud sample.

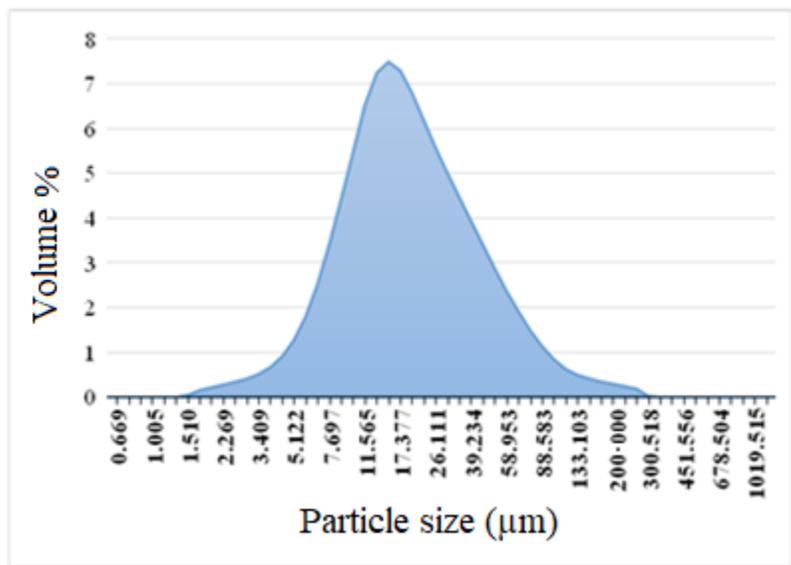


Fig. 6 – Particle size distribution of the waste drilling mud before any treatment or processing

Table 3 lists the average results and the limits of the Environmental Protection Agency and the Government of Newfoundland and Labrador for hazardous metal concentrations. It can be implied

from Table 3 that all the metal concentrations are below the limit required by landfilling regulations. Therefore, the waste drilling mud could be landfilled if the petroleum hydrocarbon concentration is reduced to meet the maximum hydrocarbon concentration permitted by regulatory agencies.

Table 3 – ICP-OES analysis results and applicable regulations on hazardous metal concentrations

Metal	Pb	Cd	Cr	As	Ba
Metal concentration (mg/l)	3.78 ± 0.41	<0.01	0.43 ± 0.10	0.24 ± 0.03	29.74 ± 11.47
EPA limit (mg/l)	5.00	1.00	5.00	5.00	100.00
Newfoundland Limit (mg/l)	5.00	0.50	5.00	2.5	100.00

X-ray diffraction analysis was completed to determine the composition of the waste drilling mud. The results are illustrated in Figure 7. Two different samples of the waste drilling mud generated almost identical results implying that the waste drilling mud was homogenous. Compounds were identified by matching the peak positions and intensities to the database software. The results showed that calcite (CaCO_3), barite (BaSO_4), and quartz (SiO_2) are the three most abundant minerals in the waste drilling mud with the normalized percentages of 77.1% ($\pm 5\%$), 12.7% ($\pm 0.8\%$), and 10.2% ($\pm 0.7\%$) respectively. $\text{CaMg}(\text{CO}_3)_2$, also known as dolomite, may also be present in the sample; however, its quantity was measured to be below the detection limits. The abundance of calcite in the sample typically generates an alkaline pH (Chesworth et al., 2008).

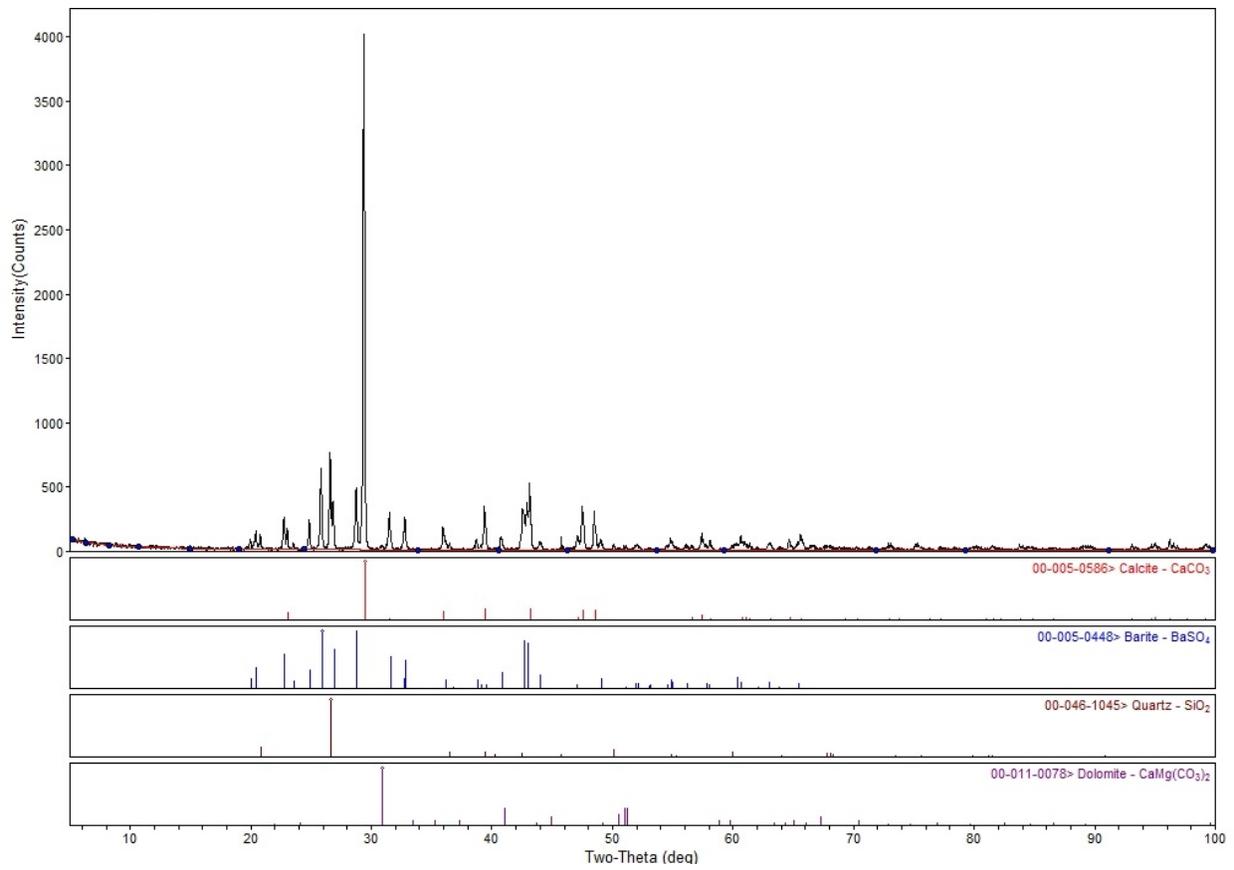


Fig. 7 – X-ray diffraction results for the waste drilling mud

Part III. Surfactant Selection Strategy

Interfacial tension analysis was employed as the screening method for selection of the most suitable surfactant for the surfactant-enhanced washing of the waste drilling mud. Three surfactants were selected from the literature due to their potential for waste remediation (low interfacial tension) and their low sorption to solid particles. They are listed in Table 4 and their structures are illustrated in Figures 8-10.

Table 4 – Three surfactants used in this study; Triton 100, Alfoterra 145-8S 90, and Saponin

Surfactant	Provider	Type	State
Triton 100	Fisher Scientific	Non-ionic	Liquid
Alfoterra 145-8S 90	Sasol Inc.	Anionic	Liquid
Saponin	Acros Organics	Non-ionic biosurfactant	Solid

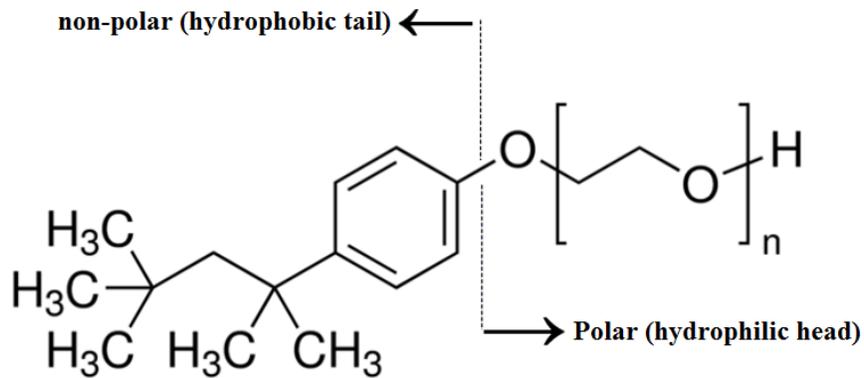


Fig. 8 – Chemical structure of Triton 100

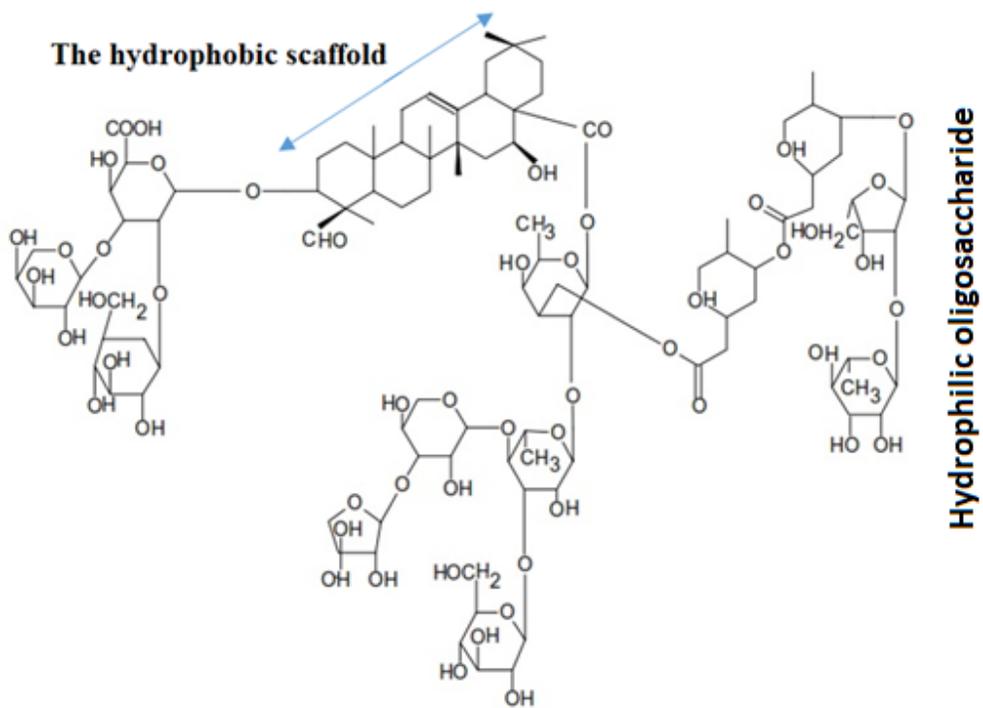


Fig. 9 – Chemical structure of Saponin

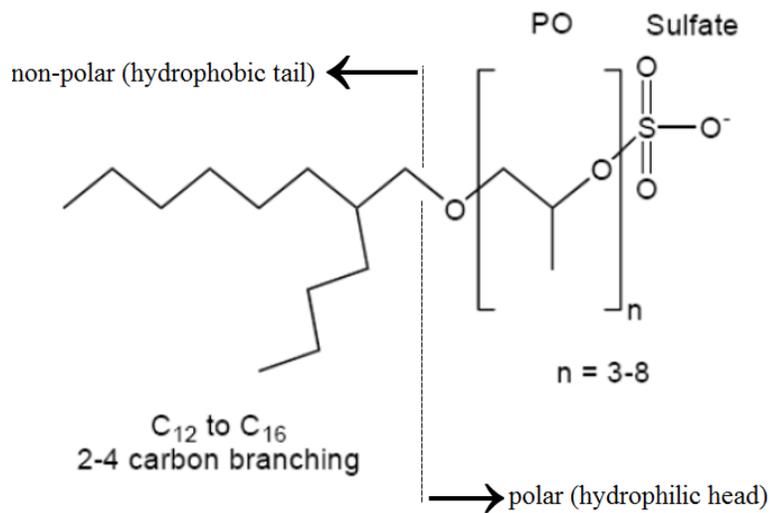


Fig. 10 – Chemical structure of Alforterra surfactants

The Pendant Bubble method was used to measure the interfacial tension between the surfactant solutions and oil. Diluted surfactant solutions with deionized water were prepared at wt.% concentrations of 0.1%, 0.25%, 1.25%, 2.5%, and 5%. Deionized water was used to dilute the surfactant solutions and the interfacial tension diagrams were plotted against each other. Interfacial tension analysis is an excellent screening test as lower interfacial tensions correspond to more complete remediation.

In this study, interfacial tension analysis with the base oil of synthetic drilling fluid was also employed to investigate the loss of surfactant due to sorption into the waste drilling mud solid particles. In our experiments, the interfacial tension between the three surfactants (Alfoterra 145-8S 90, Saponin, and Triton 100) and the base oil of synthetic drilling fluid was measured in different surfactant concentrations for the fresh surfactant solutions and the mud-surfactant solutions supernatants. The two measurements were plotted at the same scale and the amount of surfactant sorption was determined.

The results of the interfacial tension analysis (between surfactant aqueous solutions and the base oil of synthetic drilling fluid) are plotted in Figure 11. The interfacial tension is concentration-dependant and typically decreases with increasing surfactant concentration. The slope of the changing interfacial tension value is higher at the lower concentrations and it moves toward a constant slope at higher concentrations and the value of interfacial tension remains constant afterwards. Lower interfacial tension indicates the oil and surfactant solution are closer to miscibility. When the interfacial tension between the aqueous and hydrophobic compounds is reduced, the forces holding the hydrophobic compounds to the solid particles are also equally reduced (Urum et al., 2005). Therefore, Triton 100 is the most effective surfactant for reducing the interfacial tension between the oil and the surfactant solution while Saponin is the least effective

surfactant. The lowest interfacial tension achieved by Triton 100 at the concentration of 5% wt.% was 0.285 (± 0.005) mN/m. The difference between the three surfactants' interfacial tension was at its highest at the surfactant concentration of 0.25% (wt./wt.%). In practice, employing solutions with lower surfactant concentrations is desirable for economic reasons and as the Triton 100 has the capability of reducing the interfacial tension at lower concentrations, it can be considered the most suitable surfactant.

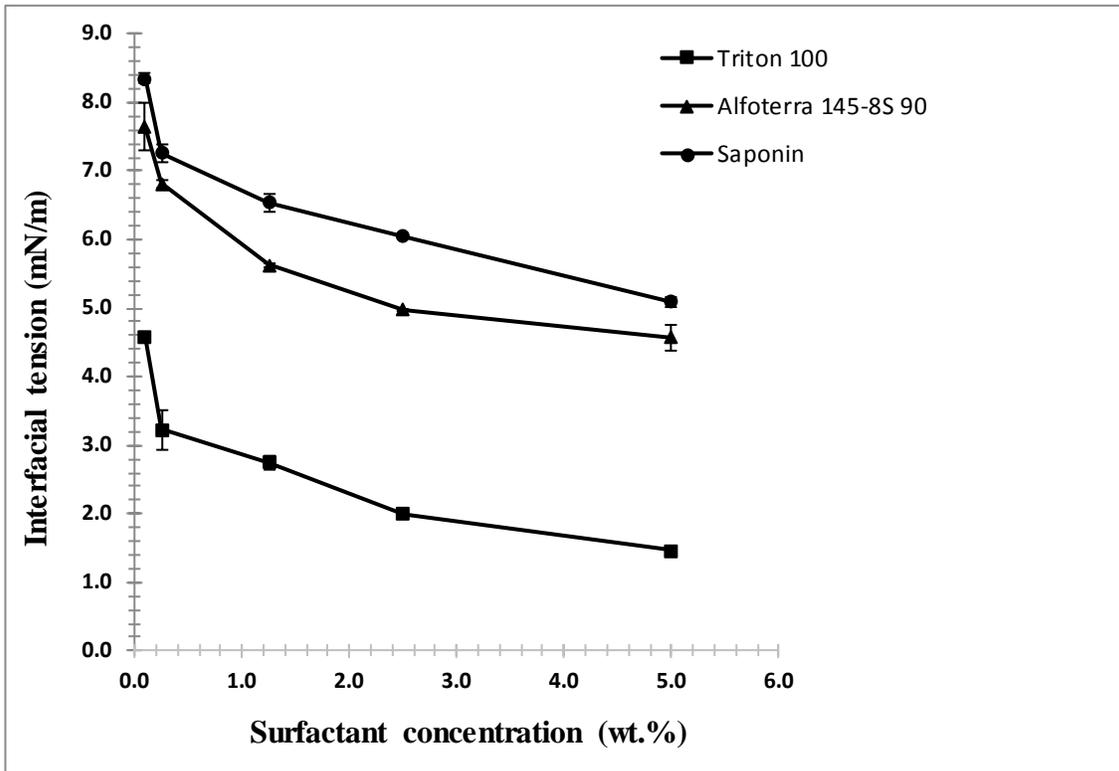


Fig. 11 – Interfacial tension of diluted surfactants and synthetic drilling fluid at 22°C

The results of the sorption analysis are also listed in Table 5.

Table 5 – Surfactant loss due to sorption to drilling mud for different surfactants

Surfactant	Inflection point concentration (mN/m)		Surfactant loss (%)
	Pure surfactant	Supernatants	
Triton 100	1.316	1.504	12.50%
Saponin	1.182	1.654	28.54%
Alforterra 145-8S 90	1.317	1.363	3.37%

It can be observed that Triton 100 had an intermediate sorption to the mud solid particles while Saponin loss is higher than both Triton 100 and Alforterra 145-8S 90. Considering the intermediate sorption and low interfacial tension (with the base oil of synthetic drilling fluid) of our non-ionic surfactant, Triton 100, it was selected as the most suitable surfactant for the surfactant-enhanced washing of our waste drilling mud sample.

Part IV: Laboratory surfactant-enhanced washing experiments

After selection of the most suitable surfactant, i.e. Triton 100, for the surfactant-enhanced washing of the waste drilling mud, based on interfacial and sorption analyses, the effect of three significant factors on the surfactant-enhanced washing efficiencies were investigated. The three significant factors were determined to be (i) contact time, (ii) surfactant concentration, and (iii) temperature.

The low and high limits of these factors are listed in Table 6.

Since these three parameters have no or little co-interactions, the effects of the three parameters were experimentally investigated by changing each factor at a time at three levels while keeping other parameters constant.

Table 6 – Parameters influencing petroleum hydrocarbon removal efficiencies for the surfactant-enhanced washing process

Factor	Unit	Range	
		Low level	High Level
Contact time	min	30	120
Surfactant concentration	wt.%	0.05	0.09
Temperature	°C	20	40

After the surfactant washing of the waste drilling mud, the solid particles were separated from the surfactant solution by centrifuging at 1000 rpm for 20 minutes. The aqueous solution was discarded and the remaining solid was analysed for its petroleum hydrocarbon concentrations using the Tier 1 method. The efficiency of the surfactant-enhanced washing process was

determined based on the total petroleum hydrocarbon concentration on the remaining solid after washing.

- **Effect of contact time**

Effects of contact time on the removal of petroleum hydrocarbons from the waste drilling mud is illustrated in Figures 12-13. It can be inferred that the maximum petroleum hydrocarbon removal can be achieved in only 30 minutes, after which the concentrations of some petroleum hydrocarbons in the waste drilling mud slightly increase and thus, the petroleum hydrocarbon removal efficiency decreases. This could be correlated with the surfactant's sorption to the porous structure of the solid particles of the waste drilling mud after a certain amount of time; and therefore, the petroleum hydrocarbon removal decreases as there would be fewer surfactant micelles to remove the oil and petroleum hydrocarbons. This phenomenon can also increase the cost of the process as the surfactant loss may increase after the 30-minute contact time. Thirty minutes should be considered the optimum contact time within the studied range as the maximum efficiency of petroleum hydrocarbon removal up to 70% could be achieved at this stage and the petroleum hydrocarbon removal decreases with increasing the contact time afterwards.

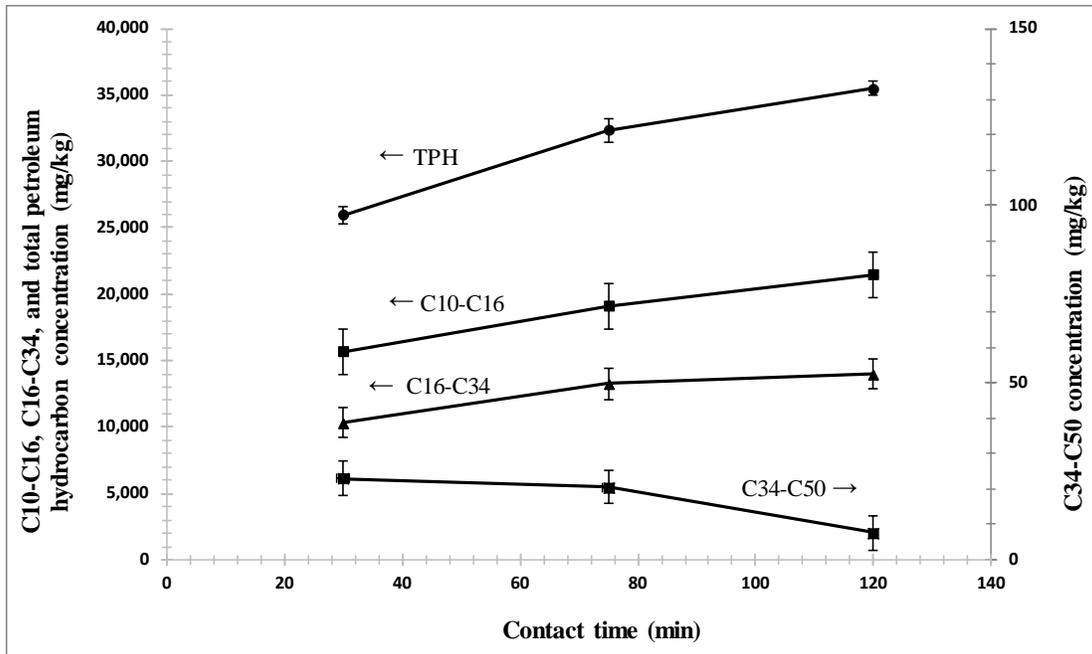


Fig. 4.12 – Fig. 12 – Effect of contact time on different hydrocarbon fraction concentrations in the waste drilling mud (temperature of 30°C and Triton 100 concentration of 0.07wt.%)

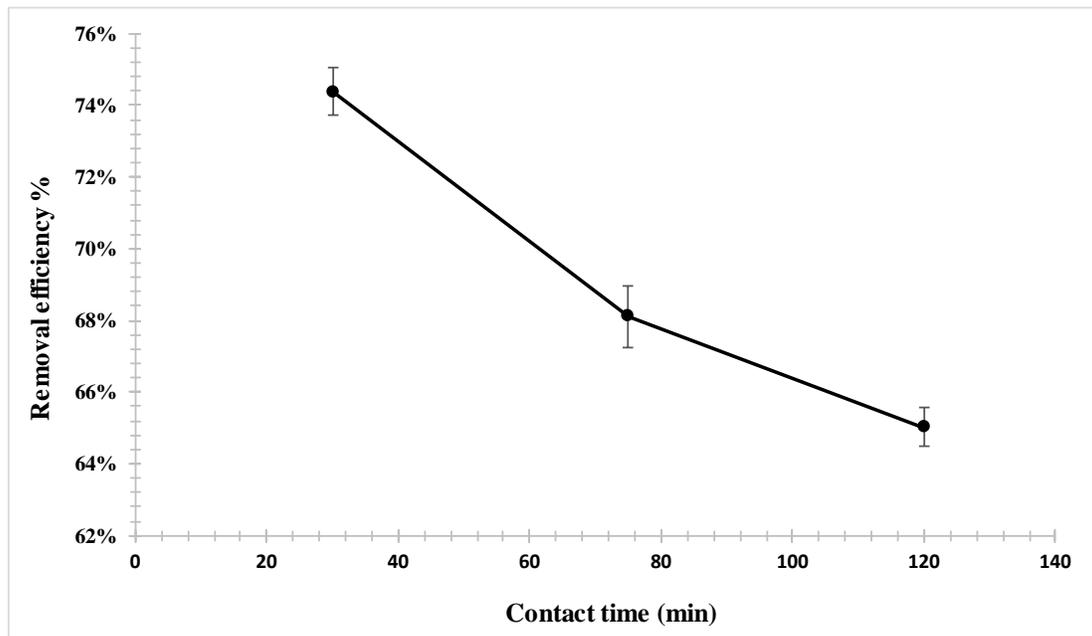


Fig. 13 – Effect of contact time on the petroleum hydrocarbon removal efficiency of the surfactant-enhanced washing process (temperature of 30°C and Triton 100 concentration of 0.07wt.%)

- **Effect of surfactant concentration**

Effects of Triton 100 concentration on petroleum hydrocarbon removal are shown in Figures 14-15. Increasing the surfactant concentration improves the hydrocarbon removal efficiencies as the presence of surfactant micelles increases with increasing the surfactant concentration and thus, more oil and petroleum hydrocarbons could be removed and recovered from the waste drilling mud. However, the effect of the surfactant concentration on the removal efficiency is slightly lower in higher concentrations which could be attributed to the fact that the higher concentration provides more surfactant micelles, thus increasing the possibly of surfactant sorption to the waste drilling mud solid particles.

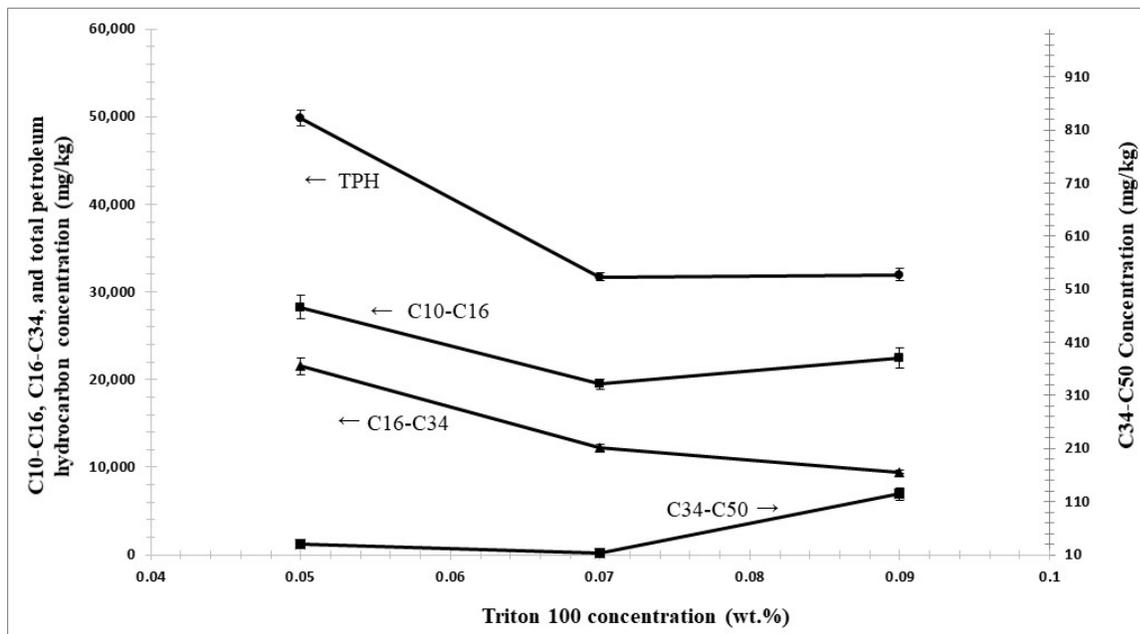


Fig. 14 – Effect of Triton 100 concentrations on the removal of various hydrocarbon fractions in the waste drilling mud (temperature of 30°C and contact time of 75 minutes)

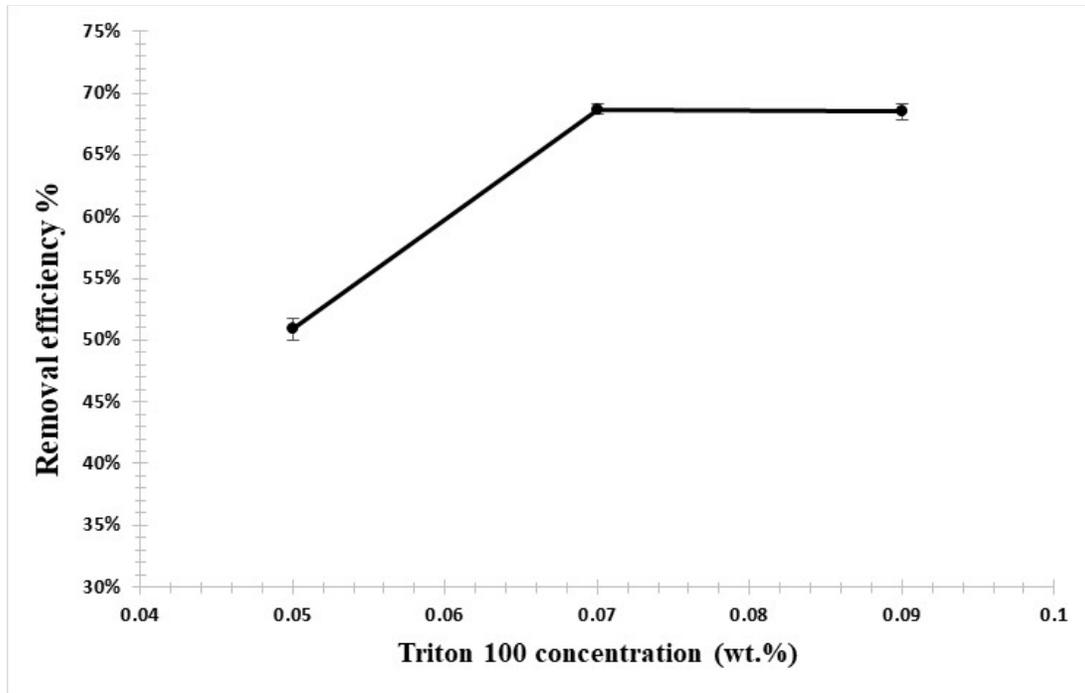


Fig. 15 – Effect of Triton 100 concentrations on the petroleum hydrocarbon removal efficiency of the surfactant-enhanced washing process (temperature of 30°C and contact time of 75 minutes)

- **Effect of temperature**

Effects of temperature on the petroleum hydrocarbon concentrations and the removal efficiencies are illustrated in Figures 16-17. There is slight improvement on the petroleum hydrocarbon removal efficiencies by increasing the temperature of the surfactant-enhanced washing process. The temperature increase typically enhances the mobility of the oil molecules due to the reduced viscosity; however, low impact of temperature increase on the petroleum hydrocarbon removal efficiency can be attributed to the jelly-like nature and higher viscosity of the Triton 100 (Zubair et al., 2013).

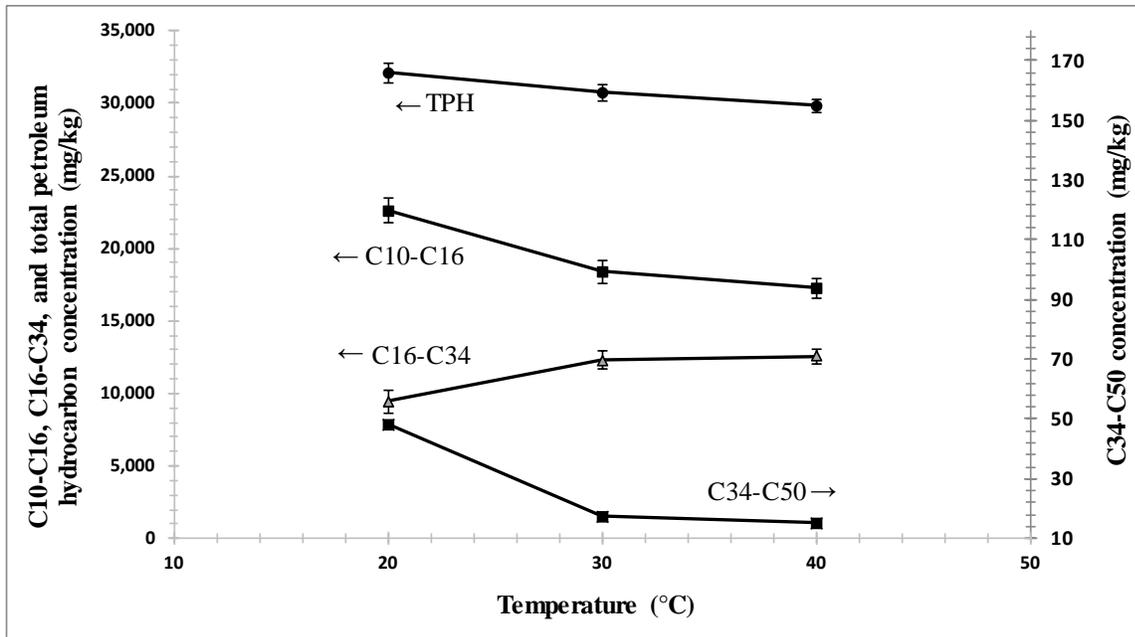


Fig. 16 – Effect of temperature on different hydrocarbon fraction concentrations in the waste drilling mud (contact time of 75 minutes and concentration of 0.07 wt.%)

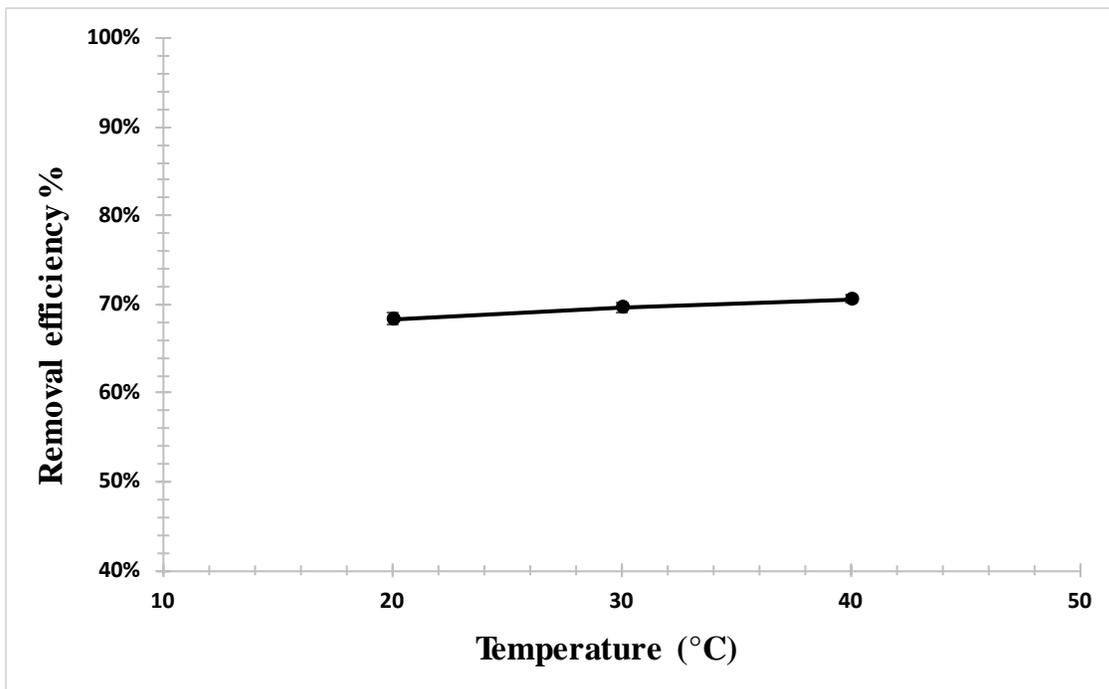


Fig. 17 – Effect of temperature on the removal efficiency of petroleum hydrocarbons in the surfactant-enhanced washing process (contact time of 75 minutes and concentration of 0.07 wt.%)

Summary and conclusions

Three surfactants were experimentally tested for their capability for waste drilling mud surfactant-enhanced remediation. Waste drilling mud was analyzed and surfactants were compared based on their ability to reduce the interfacial tension. Since the surfactant loss is a significant factor to consider for cost-effectiveness of the washing process, the surfactant sorption to the waste drilling mud solid particles was also measured. After the selection of Triton 100 as the most suitable surfactant for the surfactant-enhanced washing process, it was experimentally tested for the waste drilling mud remediation and the effects of contact time, surfactant concentration, and temperature on petroleum hydrocarbon removal efficiencies were determined. The following are the results obtained from this chapter:

- Triton 100 was selected as the most successful surfactant in reducing the interfacial tension between surfactant aqueous solutions and the base oil of synthetic drilling fluid.
- Interfacial tension between base synthetic drilling fluid and the surfactant solutions were the lowest in case of Triton 100 and the highest in case of Saponin.
- Loss of the surfactants due to sorption to solid particles was minimal for Alfoterra 145-8S 90 and maximum for Saponin. The least sorption of the Alfoterra surfactant was attributed to the high pH and the negative charge of the solid surface of the waste drilling mud.
- Thirty minutes were considered as the optimum time to achieve the highest total petroleum hydrocarbon removal efficiency using Triton 100 in the surfactant-enhanced washing process.
- Increasing temperature slightly improved the efficiency of the surfactant-enhanced washing of the waste drilling mud by Triton 100; however, the impact of temperature was

not considered significant. Therefore, a temperature of 20 °C was considered the optimum temperature for our surfactant-enhanced washing process.

- Increasing Triton 100 concentration could result in higher petroleum hydrocarbon removal efficiencies due to the increase in the available micelles in the solution.
- The results suggested that Triton 100 is a good candidate for the remediation of waste drilling mud; however, further treatment may be required to reach the limits of environmental regulations in place for disposal and landfilling of the waste drilling mud.

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