

PERFORMANCE EVALUATION OF SOLVENT ASSISTED-STEAM INJECTION
PROCESSES WITH ASPHALTENE INSOLUBLE SOLVENTS

A Thesis

by

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ABSTRACT

Adding solvents to steam flooding processes during bitumen production can reduce the water and natural gas usage but may create a new unique set of problems. Most solvents are toxic and are prohibited to be used on sites. Most importantly, since the solvent-bitumen-steam interactions are not well understood, solvent-steam coinjection into bitumen reservoirs may lead to produce lower quality oil. Because of the high asphaltene content of bitumen reservoirs and the presence of clays in the reservoir, the quality of the produced oil is determined mainly by the interaction of solvents with these elements. These interactions can be detrimental on the performance of the steam processes by enhancing the asphaltene precipitation which can plug the reservoir pores and/or by forming emulsions in produced oil which may require additional surface facilities to separate water-in-oil emulsions at the surface due to asphaltene-water interactions. This study will investigate the solvent-asphaltene-clay-steam interactions that occur during solvent-steam injection into high asphaltene content bitumen reservoirs.

Two solvents were used: n-hexane (E1 and E4) and a commercial solvent (CS) (E2 and E5). Five experiments were run: two miscible floodings (E1 and E2), one steam flooding (E3) and two SA-SF (E4 and E5). The performances of the solvents were evaluated according to the oil production and the produced oil quality, the asphaltene clay content, viscosity and emulsions of this oil.

During miscible flooding, E1 (n-hexane flooding) performed only slightly better than E2 (commercial solvent flooding). The quality of produced oil, however, was vastly

different due to the asphaltene and clay content. The commercial solvent is composed of heavy dearomatized hydrocarbons ranging from C_{11} to C_{16} , and therefore is more prone to carrying asphaltenes. During E3, E4, and E5, in which steam was used, the differences were made even clearer. Although E5 had a better recovery than steam alone (E3), the oil quality was very poor, containing high amounts of asphaltene and clay. However, for E4 where steam and n- C_6 were injected more clay was observed than E5 in which steam and commercial solvent (CS) were injected. E5 also produced less oil than E4; the heavier weight of the CS keeps it at a liquid state even at steam temperatures, limiting its coverage of the rock. The high asphaltene content of the produced oil also caused severe emulsions problems, as the asphaltene and clays present deposited in the water oil interface and stabilized the water droplets.

From these results, the use of heavy hydrocarbon solvents is not recommended for SA-SF processes into high asphaltene content reservoirs, as their interactions with the bitumen, reservoir and steam are detrimental to not only the recovery factor, but also the quality of the oil.

DEDICATION

I dedicate this thesis to my family and friends, for all their love and support throughout the years.

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NOMENCLATURE

CEC	Cation Exchange Capacity
CS	Commercial Solvent
DAO	Deasphalted Oil
DSC	Differential Scanning Calorimetry
E1	n-Hexane flooding experiment
E2	Commercial Solvent flooding experiment
E3	Steam flooding experiment
E4	n-Hexane assisted-steam flooding experiment
E5	Commercial Solvent assisted-steam flooding experiment
EDS	Energy dispersive x-ray spectroscopy
EOR	Enhanced Oil Recovery
FTIR	Fourier Transform Infrared (Spectroscopy)
OOIP	Original Oil In Place
MSDS	Material Safety Data Sheet
SAGD	Steam Assisted Gravity Drainage
ES-SAGD	Expanding Solvent Steam Assisted Gravity Drainage
SA-SF	Solvent Assisted-Steam Flooding
SEM	Scanning Electron Microscope
SF	Steam Flooding
TGA	Thermogravimetric Analysis

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INTRODUCTION

Petroleum is a critical component for the sustainability of the modern world. Its versatility allows its use as a fuel for vehicles, as the source of countless chemicals and as an electricity source (EIA 2015). The world's consumption of oil has steadily increased for decades (EIA 2014), fueled by the conventional reservoirs available all over the world. However, forecasts of the future of oil production predict a plateau of the conventional reserves during the next couple of decades, while the demand continues to rise (ExxonMobil 2015, IEA 2011). This gap between production and consumption will therefore be filled by other sources, including unconventional reservoirs.

There is no unified definition of what is an unconventional reservoir. One common generalization is that an unconventional reservoir is any reservoir that cannot be produced with an unstimulated well. Some form of stimulation process is required to improve either the permeability of the rock or the viscosity of the oil, to allow for production (Zou et al. 2013).

Of these unconventional sources oil sands are of great interest. Bitumen reservoirs at Alberta account for over 90% of Canada's oil reserves with 1.7 trillion bbl of OOIP (Alberta Energy Regulator 2015), reaching a total of 172 billion barrels of oil reserves, and making Canada the country with the third largest reserves in the world (EIA 2015). These reservoirs, however, contain bitumen with viscosities that can reach values of millions of centipoise under reservoir conditions, and with little reservoir energy available, limiting the recovery and methods (Murji and Farouq Ali 1994).

One possible means of bitumen production is surface mining, where the oil sands are physically excavated and sent out for treatment. However, only 20% of the oil sands in Canada (equivalent to 3% of the oil sand area) are at shallow enough depths to allow for surface mining, with the other 80% requiring in-situ solutions (Government of Canada 2015, Alberta government 2014).

The viscosity is highly sensitive to temperature and drops rapidly as the temperature increases (Prats 1982). This fact makes thermal Enhanced Oil Recovery (EOR) methods ideal for the recovery of the oil sands as they focus on increasing the temperature of the bitumen (Farouq Ali 1975). Steam injection methods have been the most used and successful processes for bitumen extraction, with attempts to produce the oil sands using steam going as far back as 1930 (Bellows and Bohme 1963).

As steam injection projects started to develop, research focused on improving the efficiency and overall oil production. One of the solutions given was to inject the steam, let it soak in the reservoir, and then produce the oil from the same well. This method is known as cyclic steam stimulation, or “huff and puff” (Payne et. al 1965). This method has been in used for many decades with its flow regimes being studied by (Vittoratos 1991). Variations on its well configuration were analyzed by Chang et al. (2009).

One of the proposed improvements for the process was adding some form of solvent to the steam stream to help dilute and move the oil. Many different solvents were tested throughout the years, with varying degrees of success. Synthetic crude, naphtha, and “Mobil solvent” were tested (Farouq Ali and Abad 1976). It found that there was an ideal minimum steam injection rate, and that small solvent slugs were more effective at

mobilizing the oil. The synthetic crude was the most suited for injection since it does not appear to cause asphaltene precipitation and is readily available. Redford (1982) explored the coinjection of CO₂, ethane, and naphtha, showing that each solvent improves the efficiency of oil production in different ways. CO₂ and ethane produced fluids from the cooler sections of the reservoir through a solution gas-drive, while naphtha mainly reduced the viscosity of the bitumen, allowing for a better use of the energy from the gas drive.

Farouq Ali and Snyder (1973) studied how a pre-slug of solvent (naphtha) would influence the performance of the steam on a 2D tar sand pack. They concluded that the naphtha was efficient in creating openings to allow the steam to flow through homogeneous sand packs; however, the naphtha was quickly vaporized and produced if there were high permeability channels. Farouq Ali et al. (1979) investigated the impact of injection production strategy in solvent assisted-steam flooding processes, finding that the recovery could be improved by also injecting solvent into the production well or even solely into the production well as a plug of optimum size between 10-20% of pore volume. Leung (1983) used a numerical simulation model to determine that CO₂ co-injection could increase production in cyclic steam stimulation wells.

Another prominent method of bitumen recovery using steam is Steam Assisted Gravity Drainage, or SAGD (Butler 1979), in which two parallel horizontal wells are drilled and separated vertically by approximately five meters. Steam is continuously injected through the top well. As the steam enters the formation it expands out and upwards through the reservoir forming a steam chamber. As the steam chamber expands

it heats the bitumen at its margins. This heated and mobilized bitumen then drains down the side of the steam chamber along with condensed water towards the production well. A small pool of bitumen and condensed water forms around the production well, preventing any steam from being produced immediately after injection. The SAGD process has been successfully implemented on the field, demonstrated by the Cold Lake reservoir, developed by ESSO (Butler 1994).

To improve the performance of the SAGD process, Nasr and Isaacs (2001) proposed the coinjection of solvents with the steam. Different solvents were studied along the years. Nasr et al. (2003) evaluated with eight experiments the performance of methane up to octane, concluding that the solvent should condense at the same temperature as the steam when it comes into contact with the bitumen. Orr (2010) reported on an ES-SAGD pilot test completed using Jet B, a mixture of heavy petroleum fractions ($C_7 - C_{12}$). This mixture, despite its higher molecular weight, still vaporized under the operating pressures. The addition of the solvent did not improve the production, and the pilot test was stopped after two months. Govind et al (2008) performed a sensitivity study on the main control factors of the ES-SAGD process, concluding that dynamic dilation factor played a critical role and matching earlier time data.

Ardali et al (2011) used experiments to compare SAGD with ES-SAGD using n-hexane, showing ES-SAGD improved the oil recovery and energy efficiency of the process. Li's and Mamora's (2011a and 2011b) concluded after simulation and experimental work that lighter hydrocarbon solvents such as n-heptane, improve the ES-

SAGD performance; however, they may create a gas phase between the steam and the bitumen that hinders heat transfer. The study suggests the co-injection of a mixture of heavy and lighter hydrocarbon solvents to mitigate this problem.

Mukhametshina (2013) ran SAGD experiments using n-hexane, toluene, and cyclohexane, varying the injection procedure. It was found that injecting solvents with steam improved recovery, and that co-injecting toluene with n-hexane gave the best results, both cyclically or continuously. Asphaltene precipitation was reported in experiments where n-hexane was injected, but it did not affect the oil production rate. Cyclohexane resulted in delayed asphaltene precipitation, which occurred in the production lines.

Learning the interaction of the solvent with oil is essential when selecting the right solvents. Most of the solvents used in solvent-steam injection are asphaltene insoluble. Asphaltenes are known as the portion of the oil that is n-alkane insoluble while at the same time being soluble in aromatic solvents (Speight 2006). Therefore, the asphaltene-solvent interaction will have a greater impact on oil recovery performance. The precipitation of asphaltenes may have some undesired consequences, such as blocking the flow in the pores (Minssieux 1997, Ali and Islam 1998). However, because asphaltenes are the heaviest part of the crude oil, asphaltene precipitation will also result in higher produced oil quality with less metal and impurity contents. Asphaltenes are also known as a polar component of crude oils due to impurity content. High polarity of asphaltenes will also enhance the water-asphaltene interaction (Prakoso 2015, Kar et al.

2016). An increase in asphaltene content in the oil also leads to a higher viscosity (Das and Butler 1994, Ghanavati et al. 2013).

Not only asphaltene-water but also asphaltene-clay interactions will affect oil displacement (Kar et al. 2015), and these two interactions will be enhanced or lessened with the addition of solvents (Kar and Hascakir 2015). As the fines migrate they might block pores, reducing permeability (Gruesbeck et al. 1982, Kar et al. 2015). The viscosity of the oil increases exponentially with the clay content (Coelho and Hascakir 2015).

In this thesis, effectiveness of a commercial solvent that has been reported as biodegradable (see MSDS of solvent in Appendix), was tested for bitumen extraction through solvent-steam flooding. Results were compared with n-hexane which has been reported previously as a successful solvent for solvent-stream processes (Mukhametshina 2013). To better understand the pore scale displacement mechanism, miscible flooding experiments with each solvent and steam flooding performances are also examined.

EXPERIMENTAL PROCEDURE

Characterization of Bitumen

The bitumen studied was from the Peace River region in Alberta, Canada. It has an API gravity of 8.8° and a viscosity of 54,152 cP at room temperature (Mukhametshina and Hascakir 2014). It has high asphaltene content, with 34% of its weight being derived from n-pentane insoluble asphaltenes (ASTM D2007-11). Figure 1 shows the viscosity behavior of the original oil as a function of its temperature. Also, on the same graph a correlation to define temperature-viscosity relation is presented with power law relation.

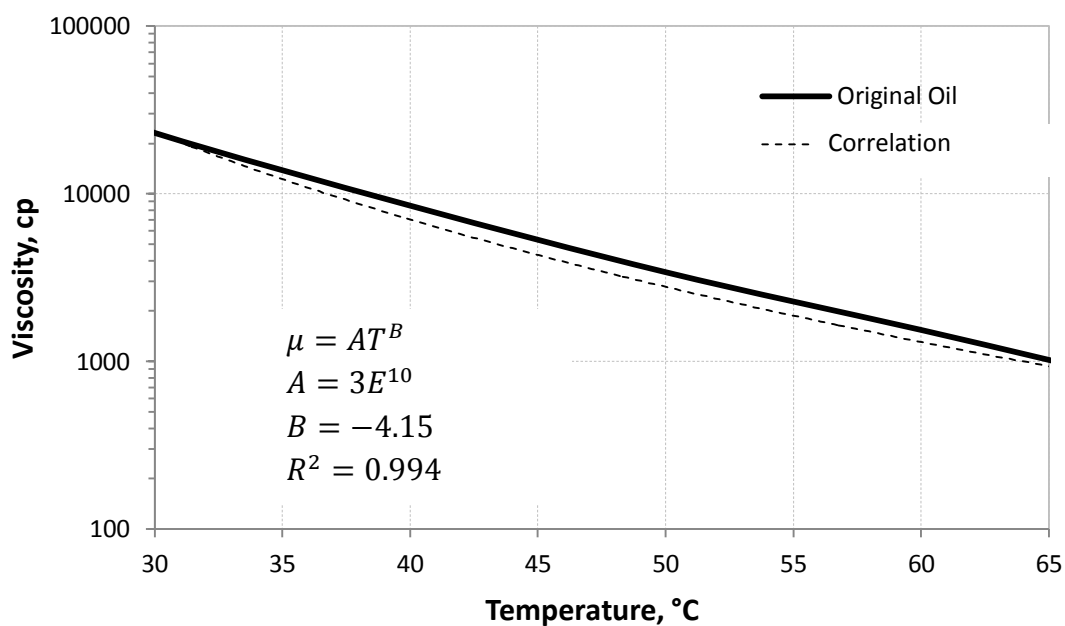


Figure 1 – Viscosity-Temperature Relation for Original Bitumen (Y Axis is in Log. Scale)

Characterization of Solvents Used in This Study

During miscible or solvent assisted-steam flooding experiments, either a commercial solvent or n-hexane was used. The n-hexane used during the experiments is over 99% pure n-hexane. Its flash point is -22.8°C while its density is 666kg/m^3 (Open Chemistry Database 2015). The commercial solvent is composed of dearomatized hydrocarbons, with a carbon number distribution that ranges from C_{11} up to C_{16} . It has a flash point of 96°C and its density at 15.6°C is 799 kg/m^3 (see MSDS of solvent in Appendix). Thermogravimetric analysis and Differential Scanning Calorimetric (TGA/DSC) experiment was carried out on commercial solvent to observe weight loss (TGA) and heat flow (DSC) behavior of the CS at a $10^{\circ}\text{C}/\text{min}$ heating rate until it reached 220°C . The TGA/DSC results of CS are given in the Appendix (Figure A-4). For n-hexane, a TGA and DTA (Differential Thermal Analysis) experiment was carried out, Results are also given in the Appendix (Figure A-6).

Fourier Transform Infrared spectroscopy was also used for solvent characterization. Results are given in Appendix (Figure A-10). Wavenumbers between $2850 - 3000\text{ cm}^{-1}$ in FTIR spectra are the indications of CH stretch while 1470 cm^{-1} and 2850 cm^{-1} are CH_2 and CH_3 (Bellamy 1975). These absorbance peaks provide information on the saturated nature of the two solvents. Since the two solvents are saturated hydrocarbons, their interaction with the high asphaltene content bitumen studied will result in asphaltene precipitation (Hammami et al. 2000, Mitchell and Speight 1973, Sahimi et al. 1997). However, the carbon number of commercial solvent (C_{11} up to C_{16}) is higher than n-hexane (C_6). Therefore, the experiments conducted with

commercial solvent should result in less asphaltene precipitation than the experiment conducted with n-hexane (Mitchell and Speight 1973, Sahimi et al. 1997). However, the asphaltene content of produced oil after interaction with commercial solvent should be higher than after interaction with n-hexane. This study investigates how the difference in volatility, molecular weight and solubility in asphaltenes of the solvents will affect the performance of the recovery processes.

Sample Preparation and Experimental Setup

The sand/clay mixture used was designed to simulate the Peace River region's reservoirs. The reservoir rock composition is reported as 85%wt sand 20-40 mesh and 15%wt clay (Bayliss and Levinson 1976). This mixture generates a void space equivalent of 32% porosity. It was then saturated with 14%v distilled water and 86%v bitumen to replicate real reservoir composition (Hamm and Ong 1995).

The clay used is described as containing 90%wt kaolinite and 10%wt illite (Kar et al. 2015). The oil-sand mixture was then packed in the cylindrical core holder given in the Appendix (Figure A-2), which was held vertical during the experiments. Steam, solvent, and steam with solvent were injected from the top of the cell, and the produced oil, water, gas, and solvents were collected from the bottom of the cell.

The experiments were conducted using the experimental setup described in Figure 2. All experiments were conducted under the same conditions: a backpressure regulator, located at the outlet of the core holder kept the pressure of the oil-sand pack at a constant 75 psi, with the pressure being provided by a nitrogen gas cylinder.

A total of five experiments were conducted; two miscible floodings, one steam flooding, and two solvent assisted-steam floodings. The framework of each experiment is summarized in Table 1. From this point forward the experiments will be named as E1 [n-hexane flooding], E2 [Commercial Solvent flooding], E3 [Steam flooding], E4 [n-hexane + Steam], and E5 [CS + Steam].

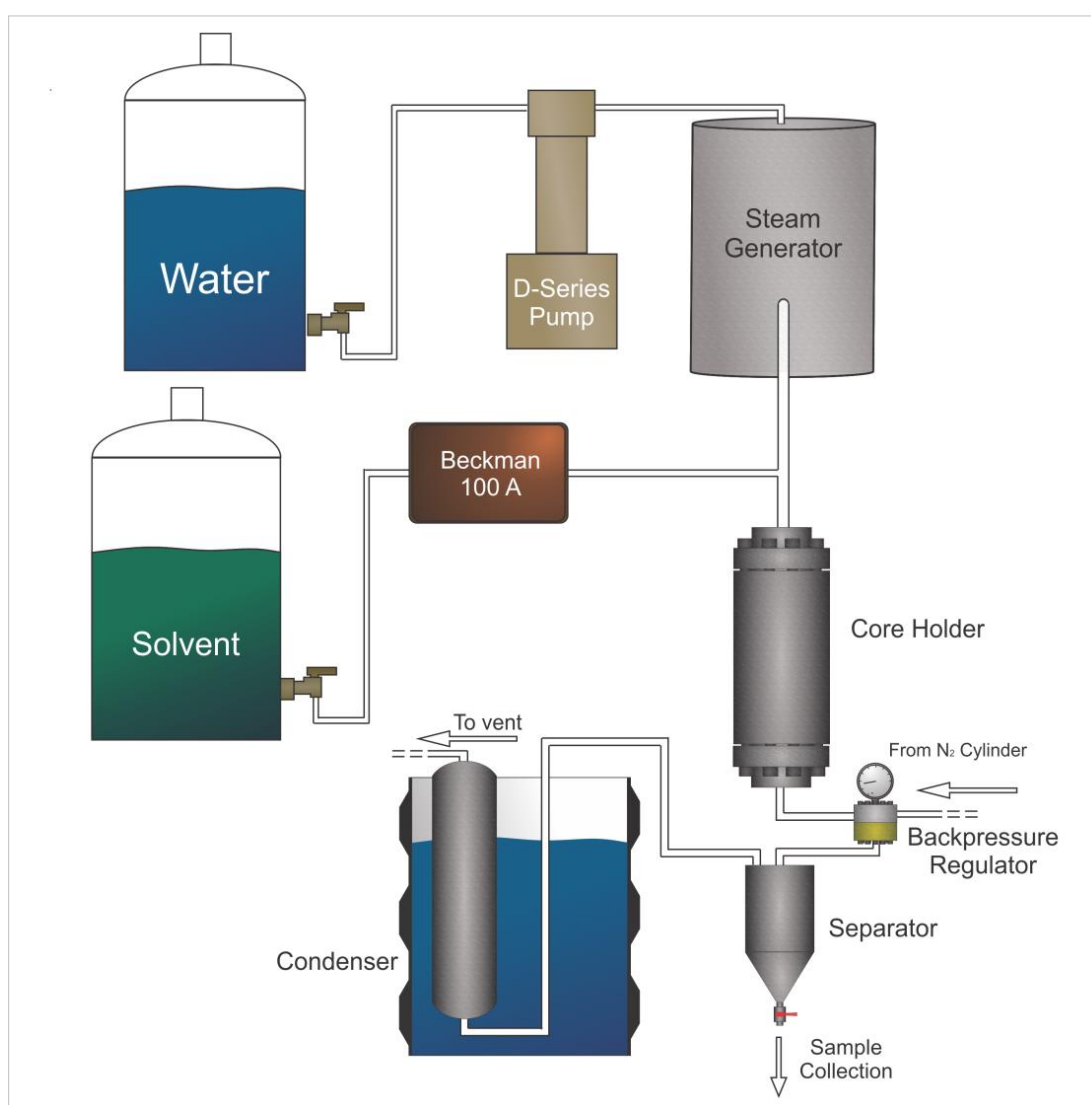


Figure 2 - Experimental Setup

Table 1 - Experimental Conditions

EOR Method	Experiment	Solvent Type	Steam Injection Rate, ml/min CWE*	Solvent Injection Rate, ml/min
Miscible	E1	n-Hexane	0	2
Miscible	E2	Commercial Solvent	0	2
Steam	E3	-	18	0
SA-SF**	E4	n-Hexane	18	2
SA-SF**	E5	Commercial Solvent	18	2

*CWE Cold Water Equivalent
** SA-SF Solvent Assisted-Steam Flooding

Water was injected into the steam generator using two water pumps, a D-Series water Pump and a LC5000 water pump (see Appendix Figure A-1) capable of holding 1,000 ml and 500 ml of water, respectively. The pumps operated alternately to continuously feed the steam generator with water during the refilling of each pump. The pressure was kept at 1,000 psi by a backpressure valve, to ensure constant flowrate and correct operation of the pumps.

The water was heated to ~170°C in the steam generator to achieve 100% steam quality at 75 psi and then directed into the core holder cell. The Beckman 100A pump was used to inject solvents, during the miscible flooding experiments. For the solvent assisted-steam flooding (SA-SF) experiments the solvents were coinjected with the steam. One-way valves were located in the flowlines to prevent any backflow of the fluids.

Experiments were conducted vertically, to achieve a piston-like displacement. Hence, gravitational influence that could result in steam override was minimized (Lookeren 1983). Produced fluids were collected in a two-stage separator system. In the

first stage, the liquids were accumulated and the gases were redirected to the second separator which was immersed into water bath to maintain condensation of steam-solvent gases or light hydrocarbons. Any non-condensable gases were vented. Experiments were run to achieve at least three full hours of production.

Post Experimental Analysis of Produced Oil and Spent Rock Samples

Produced oil samples were analyzed to determine the viscosity and composition. Spent rock samples were studied to find the residual oil content and water/air contact angle.

The viscosities of the produced oil samples were measured using a Brookfield DV-III Ultra Rheometer at temperatures ranging from 30°C up to 65°C. The ASTM-D2007-11 method was used to determine the produced oil composition. The produced oil was mixed with n-pentane to precipitate the n-pentane insoluble asphaltenes present in the bitumen. This mixture was then filtered through a filter paper that collects the precipitated asphaltenes, along with clays present in the oil, letting only the deasphalted oil (DAO) through. The asphaltenes and clays were then mixed with toluene, an aromatic solvent that dissolves the asphaltenes, and again filtered through a filter paper to remove the asphaltenes, leaving only the clays. The produced oil was therefore separated into three main components: deasphalted oil, asphaltenes and clays.

The produced oil water content was measured to examine the emulsion formation during the steam injection experiments. A TGA/DSC analysis was used to determine the water content of the produced oil samples. The samples were heated at a constant

heating rate (10°C/min) under air injection while the weight loss (TGA) and heat flow (DSC) were monitored.

Residual oil content of the spent rocks were calculated by measuring the weight of the rock samples, washing them with n-pentane to remove the DAO, weighing the samples again, washing it with toluene to remove the asphaltenes, and then measuring the final weight of the sample. The residual oil content was therefore separated between DAO and asphaltenes.

The wettability of the spent rock samples was determined using the Kruss DSA30S Drop Shape analyzer. The rock samples were flattened to form an even flat surface and a droplet of 50 μL of water was dropped on it using a high precision syringe. Video recording was then used by the software to calculate the contact angle between the water and the rock surface. The measurements were taken in an air matrix, therefore were air/water contact angles, using the correlation developed by Grate et al. (2012) the values were then converted into oil/water contact angles.

RESULTS AND DISCUSSION*

Solvent-Bitumen Interactions

First, the solvent-bitumen interactions were evaluated through viscosity measurements at varying temperatures. Mixtures of bitumen and solvent at a 9:1 bitumen-solvent weight ratio were prepared, and their viscosities were measured (Figure 3).

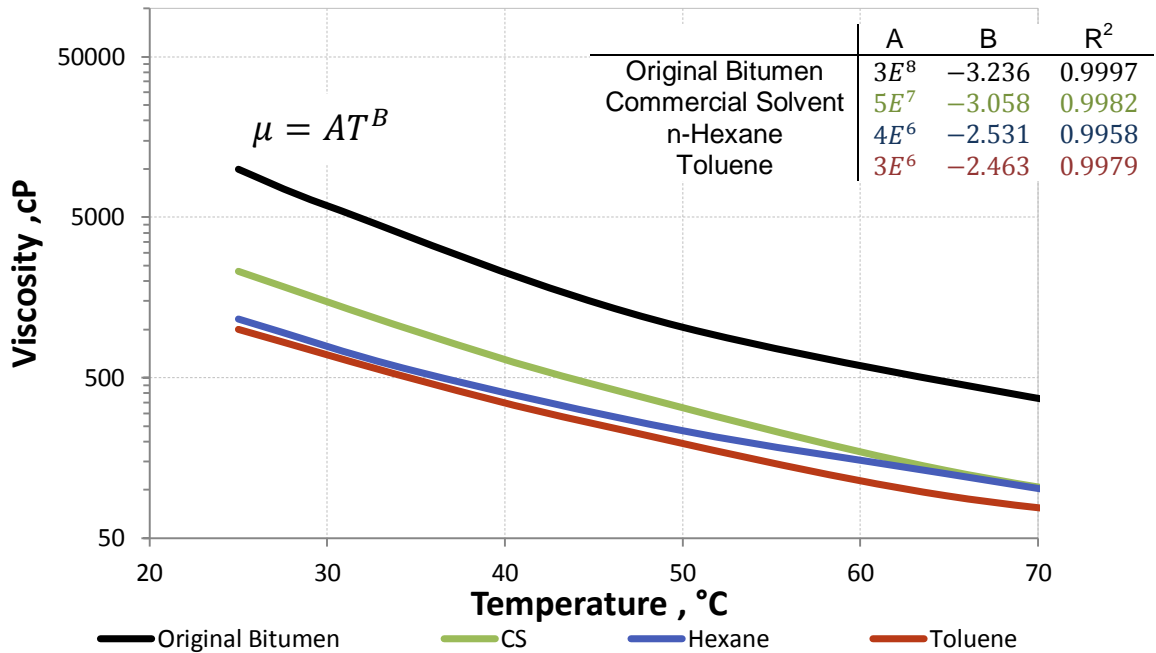


Figure 3 – The Viscosity-Temperature Relations for 90 %wt Bitumen + 10 %wt Solvent Mixtures

Toluene had the highest viscosity reduction as a result of its aromatic composition, which dissolves asphaltenes (Mitchell and Speight 1973). The commercial solvent reduced the viscosity of the bitumen considerably, however, not as much the n-

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hexane or the toluene. This was the first indication that this new solvent might not perform as well as the n-hexane during flooding experiments, while the original bitumen viscosity temperature relation provides information on how viscosity reduction will be accomplished during steam flooding.

Cumulative Oil Production

During all experiments, every 20 minutes, produced oil samples were collected from the separator. The collected liquids were labeled and left stagnant to separate the injected solvents and steam through evaporation. The weight of each bottle was measured in time to observe the evaporation of the remaining solvents or any other gases in the produced oil samples. Figure 4 shows the final cumulative oil production of the miscible flooding experiments (E1 and E2), while Figure 5 shows the results from steam and solvent assisted-steam flooding experiments. All results represent only the oil components excluding water-in-oil emulsions and clays. However, initially emulsions and clays were detected in produced oil samples that will be discussed in the following sections.

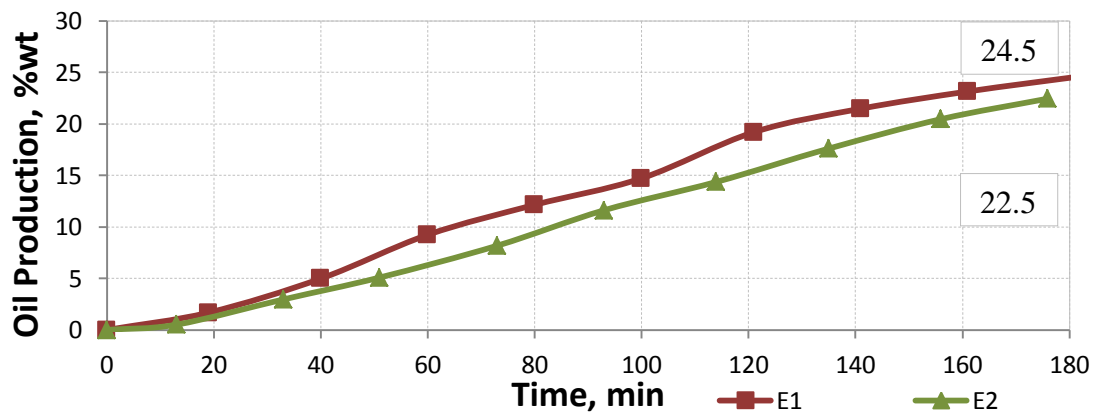


Figure 4 - Cumulative Oil Production from Miscible Flooding Experiments (E1 – n-C₆ Flooding E2 – CS Flooding)

The difference in performance between the two solvents during the miscible flooding experiments was only about 9%, meaning that the CS was almost as efficient as the n-hexane in improving the oil recovery. Their recovery rate was also very similar, with E2 lagging just slightly behind.

The n-hexane was the first to mobilize the oil. In E3, in which steam flooding alone was implemented, less oil recovery was observed than in E1 and E2, in which miscible flooding was implemented. However, solvent-steam flooding methods (E4 and E5) enhanced the oil production significantly; solvents help mobilize the bitumen more rapidly, as they increase its mobility even in lower temperatures.

Solvent addition to the steam stream is definitely a viable way of increasing oil production. In E4, after 60 minutes, the oil production reached almost maximum level at approximately 25%wt recovery. Hence, n-C₆ use along with the steam obviously reduced the steam generation necessity and increased the oil production rate.

All steam experiments have a discernable hump during the production, where the oil rate increases rapidly, and then returns to lower rates, which is an indication of oil bank arrival. During miscible flooding experiments three main zones formed inside the cell that will determine the displacement of the fluids. The first zone was composed solely of the solvent as it was being injected through the top, the second zone is where the solvent makes contact with the bitumen present in the rock. Since the solvents were alkanes they create a miscible zone where they dissolve with the DAO of the oil.

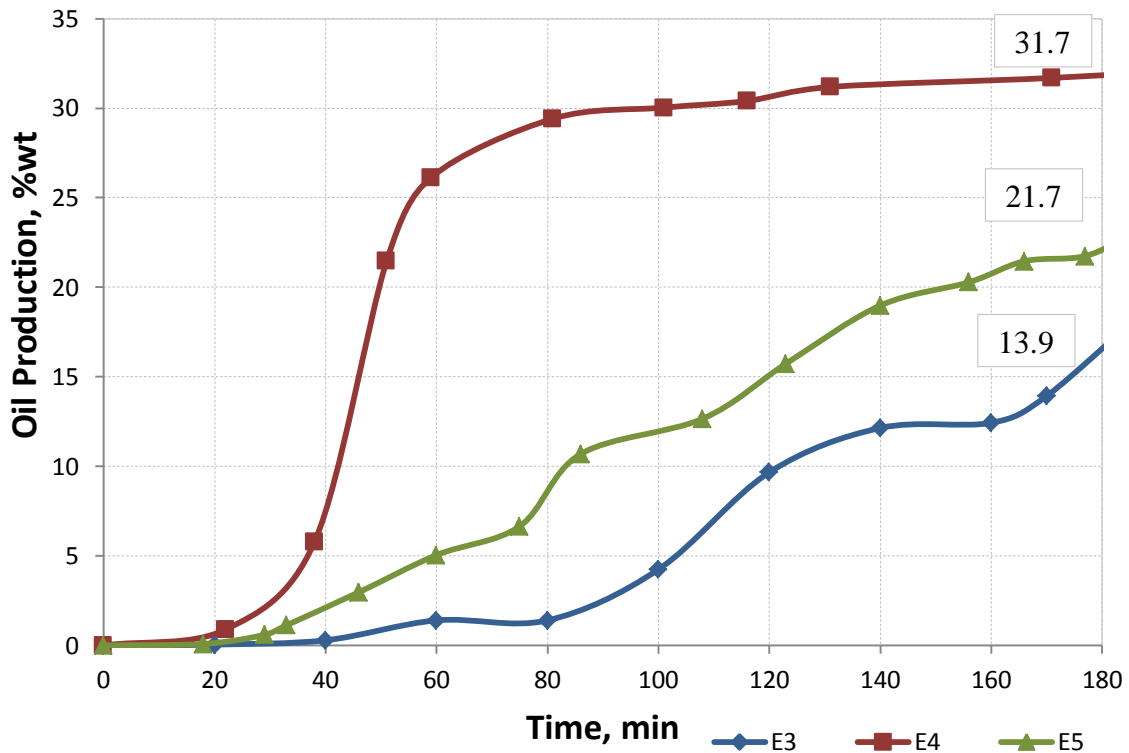


Figure 5 - Cumulative Oil Production from Steam and Solvent Assisted-Steam Flooding Experiments (E3 – Steam Flooding E4 - n-C₆ Assisted Steam Flooding E5 – CS Assisted Steam Flooding)

The asphaltenes are not miscible in the alkanes, and therefore tend to precipitate when in contact with the solvents. The third and final zone is the oil bank, mobilized oil that is being pushed by the mixture bank in the direction of the outlet.

During the steam flooding four zones were expected to form. The first is the steam zone, where the steam was in vapor phase sweeping the rock. The second is the water zone, steam that condensed due to heat transfer to the bitumen. The third is the hot zone that contained the bitumen that was heated by the contact with the steam and therefore mobilized. The final zone is the cold zone, where the original bitumen, not

contacted by the steam, is located. Since the bitumen was still cold its viscosity was very high, and it was not mobile.

For the steam-solvent flooding experiments the zones were similar to the steam flooding experiments. There was a steam and solvent zone, where the injected fluids swept the reservoir. The steam condensed when in contact with the bitumen to again form the water zone. The contact of the steam and solvent with the bitumen formed a miscible hot zone, in which the solvent was mixed with the bitumen while it was heated. And the final zone which contained the cold original bitumen, not contacted by either the solvent or steam.

The oil displacement and consequently oil production are affected by the interaction of in-place fluids with injected fluids. Changes in wettability and interfacial tension have great impact on recovery performances. Thus, the following section will discuss the produced oil quality in terms of asphaltene and clay contents.

Produced Oil Analysis

The produced oil compositions were analyzed using various methods. The samples from each experiment were first left inside an oven at 90°C, to remove the solvents and water through evaporation. Sample weights were measured constantly to observe the evaporation rate. While n-hexane has a 69°C boiling point, CS has 210°C; therefore, n-C₆ was separated quicker than CS.

With the produced oil samples in hand, two main components were measured, the asphaltenes and the clays. Asphaltenes were separated using the ASTM-D2007-11 method, using n-pentane as the precipitating agent. The asphaltenes collected are therefore categorized as n-pentane insoluble. The clays were separated by a toluene bath of the produced oil. This oil was then passed through a filter paper with a pore size that would allow the washed oil to pass, but at the same time would retain any clays present (Unal et al. 2015). The part of the oil that was neither clay nor asphaltene was classified as deasphalted oil (DAO), which contains the lighter elements of the oil. The same procedure was applied to the original oil; the impact on the oil quality of each experiment could therefore be investigated. Using these methods, the composition of the produced oil samples was determined. Figure 6 summarizes the composition of all produced oil samples originating from different experiments showing great variation between them.

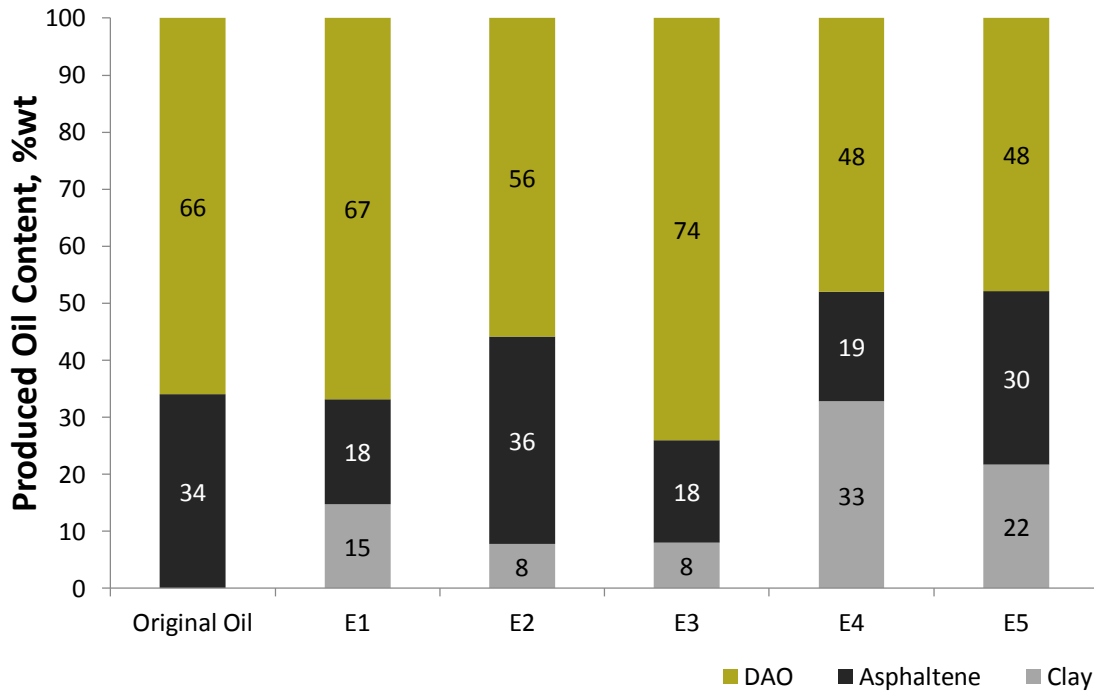


Figure 6 – Produced Oil Components (Clay, Asphaltenes and DAO) – Water and Solvent Removed – Reprinted from Stape et al. (2016)

Clay Content

Throughout all samples it is clearly visible that clay migrates into the produced oil. The clays show more affinity with the solvent of lower molecular weight. During the miscible flooding the oil produced with n-hexane had 15% of its weight coming from the clays, almost twice as much as the CS injection. The same overall trend is seen during the solvent assisted-steam flooding results, where the steam and solvent injection resulted in a clay content of 33%, while the steam and CS injection produced only 22% of clay. The steam injection experiment had a minimal clay production, with only 8% of the oil weight being clays. These results present a scenario where the solvents have a distinct affinity with the clays, increasing their movement and therefore their content in

the final oil. The type of solvent used has a direct impact on this effect, with the affinity for clays decreasing as the molecular weight increases. Steam did not move a lot of clays by itself, but when coupled with solvents their migration increased.

Scanning electron microscope (SEM) images were taken of the original clay used in the experiments, as well as the samples separated from experiments E1 and E2, (Figure 7).

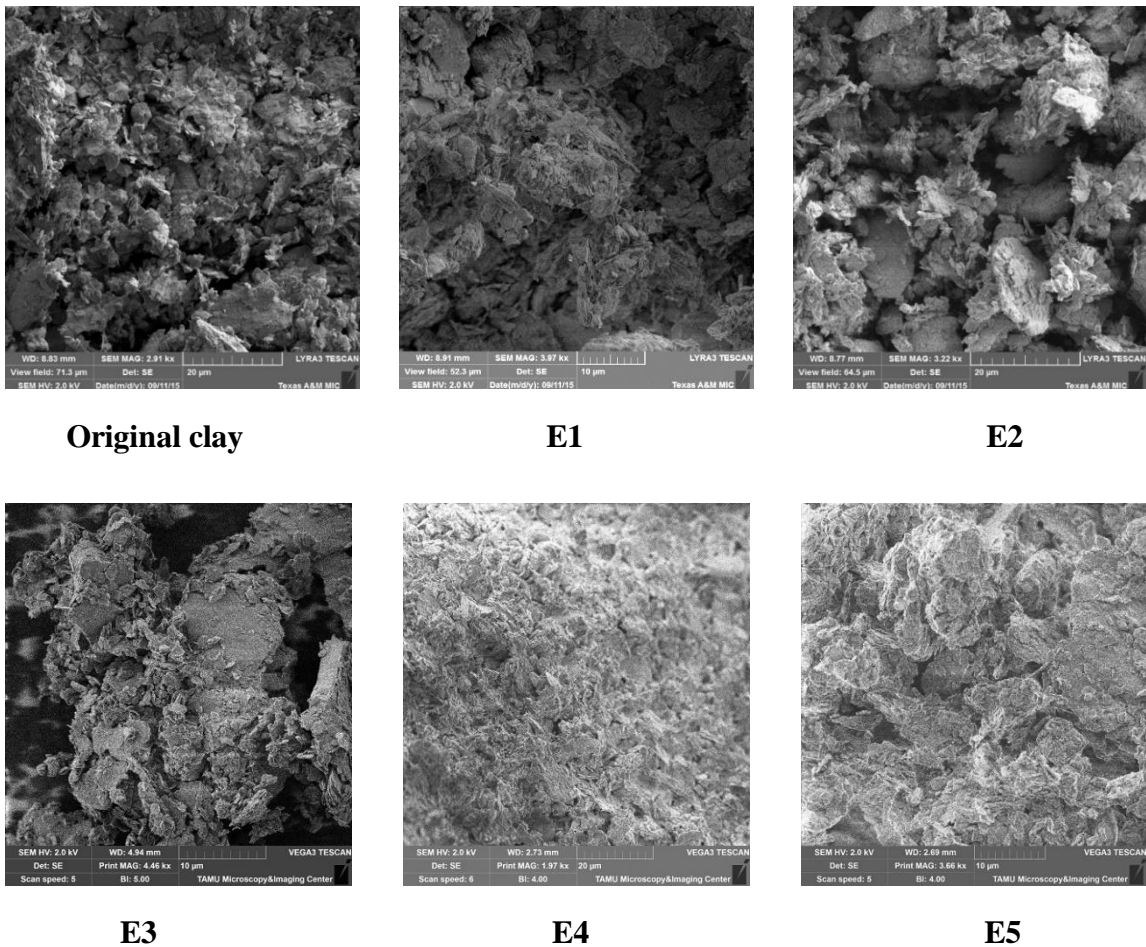


Figure 7 - SEM Images of Original and Produced Clays from E1, E2, E3, E4 and E5

Energy dispersive x-ray spectroscopy (EDS) was performed on these samples to determine their composition. The results are shown in Table 2. Note that the initial clay was composed of 90% kaolinite and 10% illite. Kaolinite does not have potassium in its composition, therefore any potassium traces that are found in the clay comes from the illite. These traces can be used to see if there is any clay transformation between experiments after the interaction with bitumen and steam through cation exchange. Note that kaolinite has an XX cation exchange capacity (CEC) and illite has a YY CEC. Hence illite is expected to have a transformation. From the EDS results (Table 2), while no cation exchange occurred in clays during the experiments, it is possible to have some physical changes in clays morphology due to their pore lining, filling cementing feature (Figure 7).

Table 2 - EDS Results for Clay Composition, %wt

Component	Original Clay	E1	E2	E3	E4	E5
O	57.8	56.0	57.1	69.4	74.4	66.6
Al	18.6	19.4	19.0	15.5	12.6	16.1
Si	21.8	22.9	22.1	14.3	10.9	16.0
K	1.7	1.7	1.8	0.9	0.5	1.2
S					1.2	
Na					0.8	

Asphaltene Content

E1 produced oil with an asphaltene content of 18%, compared to 36% during E2. Both SA-SF had a higher asphaltene production than the SF alone, with E4 having 19% and E5 with 30%, compared to E3 with 18%. The asphaltene production also

accompanied a trend: the higher the molecular weight of the solvent the more asphaltene was carried with it. This follows similar information found in the literature (Mitchell and Speight 1973, Sahimi et al. 1997), where it was found that the higher the carbon number of the solvent that came into contact with the oil, the lower the amount of precipitated asphaltene would be. This means that when heavier hydrocarbons such as the CS (C₁₁-C₁₆) are injected into the reservoir, less asphaltenes should precipitate and more should be carried to the produced oil (Mitchell and Speight 1973, Sahimi et al. 1997), and that is exactly what was observed. The analysis also showed that this continues to be true even when steam is involved, although the discrepancy was not as large between experiments.

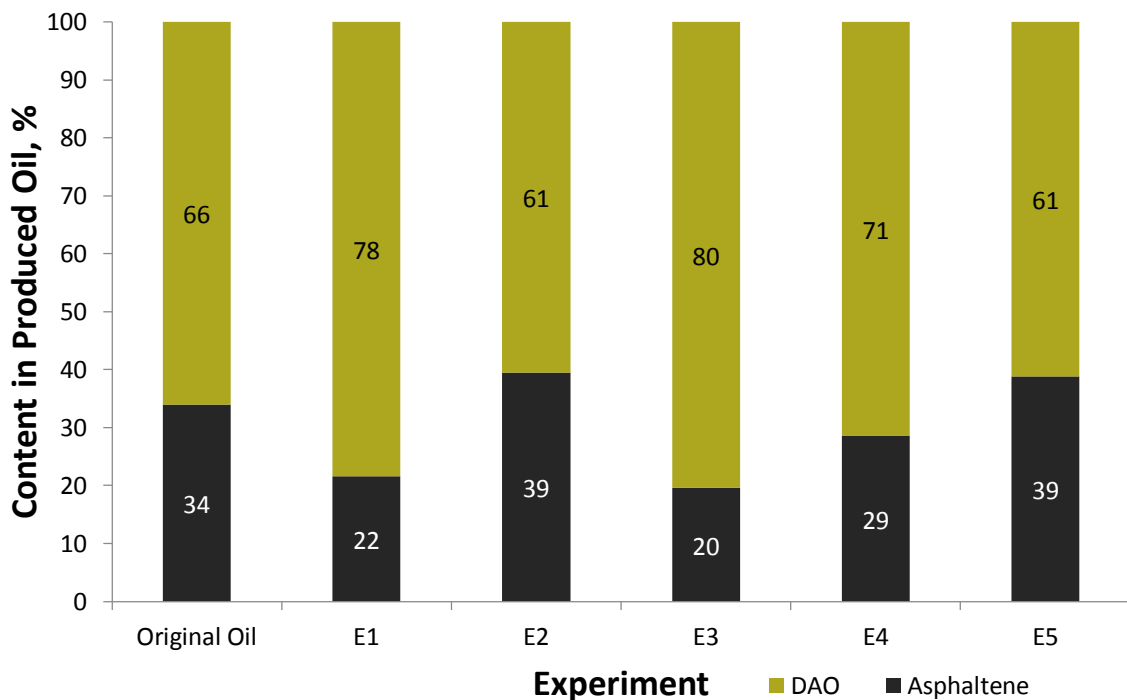


Figure 8 - Composition of the Produced Oil – Normalized by Excluding the Contribution of Clays, Solvent and Water – Reprinted from Stape et al. (2016)

Figure 8 is constructed by excluding the contribution of clays, water, and solvent contents of produced oil samples; hence they are normalized values. CS produced higher asphaltene content than even the original bitumen. Visual inspection of asphaltenes from the five experiments is given in Figure 9. The reason behind the lower quality in produced oil samples from E5 is further analyzed when dealing with emulsions, which will be discussed in the following section.

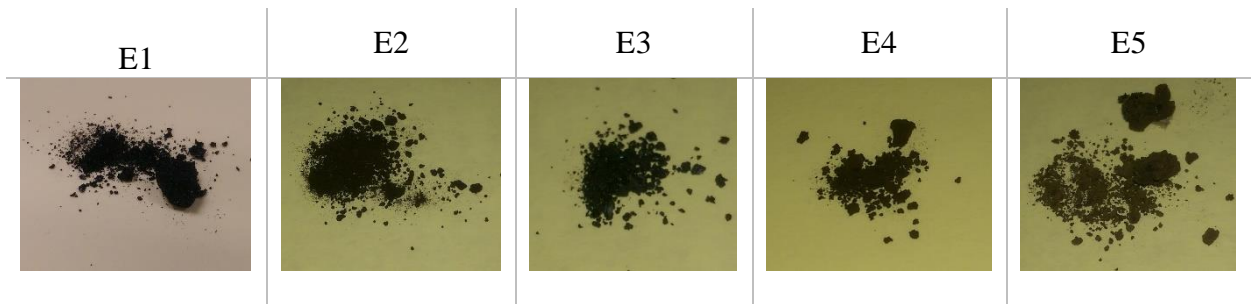


Figure 9 - Produced Asphaltenes from Core Flooding Experiments

Water-in-Oil Emulsions Content of Produced Oils

As any process where water is involved, the solvent assisted-steam flooding methods will be susceptible to emulsions formation (Fingas 2014). The high energy of the steam coupled with the high asphaltene content of the produced oil, makes this scenario especially vulnerable (Kar et al. 2014, Mukhametshina et al. 2015), which will increase the cost of water-oil separation. Experiments E1 and E2 did not involve steam and consequently are not relative for this discussion.

During all experiments involving steam the produced fluids at the time of sampling were heavily mixed. They were left to settle while inside an oven at 90°C. All samples formed a layer of oil at the top of the bottles with a much larger layer of water at

the bottom (Figure A-3). For experiments E3 and E4, the water was quick to evaporate in which steam and n-C₆-steam were injected. However, for E5, the water-in-oil emulsions were much more stable, remaining for much longer and containing much more water. Microscope images at 400x magnification were taken to assess the presence of water. Figure 10 shows the optical microscope images of the produced oil from the steam flooding experiments before oven treatment, confirming the presence of water-in-oil emulsions in the produced oil.

A TGA/DSC analysis was used to determine the water content of the produced oil samples. Results are given in the Appendix (Figure A-7). Accordingly it was determined that the oil from E5 contained as much as 80 %wt water, and it remained that way for a very long period of over a month in the oven at 90°C.

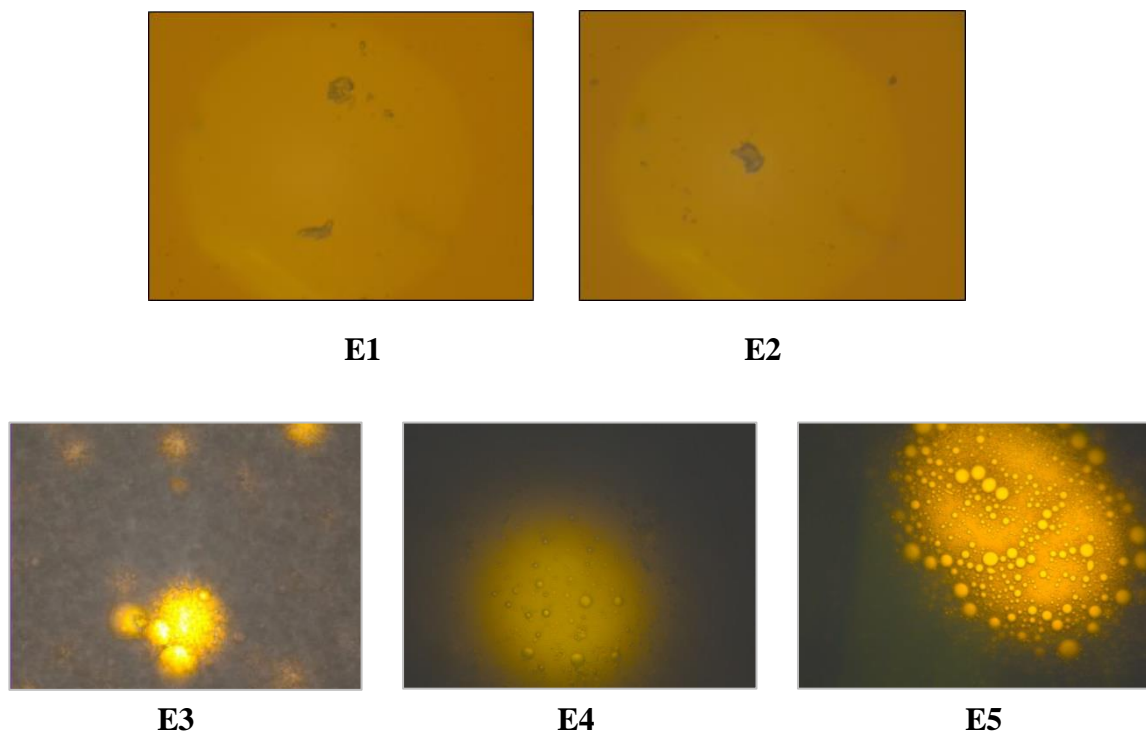


Figure 10 - Microscope Images of Produced Oil at 400x Magnification

The emulsion stability highly depends on the asphaltene content of produced oil (Sjoblom et al. 2007, Tchoukov et al. 2014). As previously seen the CS, due to its high molecular weight, produced more asphaltenes than in the other experiments. This higher asphaltene presence, coupled with the fines (clay) present (which is less than E4 but not insignificant), helps stabilize the water droplets as they settle in the water oil interface, and prevent coalescence (Czarnecki et al. 2012, Levine and Sanford 1985). Due to emulsions problems, coinjection of CS with steam was not recommended. The water is not only produced in emulsion form for E5, but also higher produced water volume was observed in E5 (Figure 11). In other words, injected water/steam could not displace the oil effectively. The n-hexane forms a gas phase layer between the steam and the reservoir, which reduces the heat transfer from steam to oil, explaining its lower water recovery in the samples, as less steam was condensed (Li and Mamora 2011b).

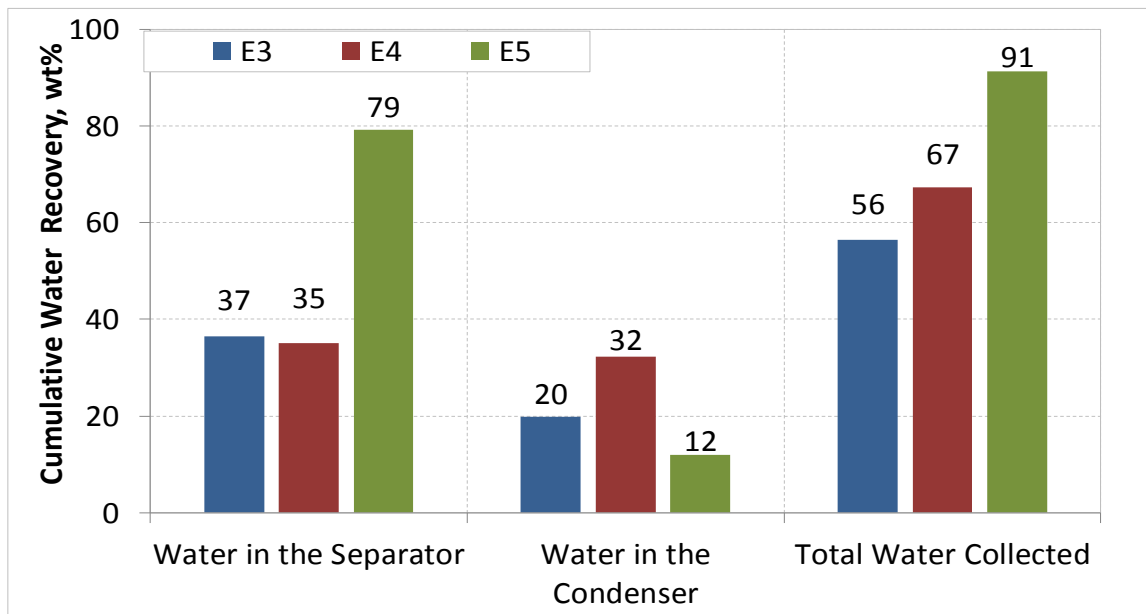


Figure 11 - Water Recovery from Experiments with Steam (E3) and Solvent-Steam (E4, E5) – Reprinted from Stape et al. (2016)

Viscosity of Produced Oil Samples

Viscosity plays a major role in the production process, as it determines the mobility of the oil. The viscosity of the oil produced from each experiment was measured as a function of temperature using a Brookfield DV-III Ultra Rheometer (Figure 12). The viscosity measurements were conducted on produced oil samples with clay, solvent, and water content. At first glance, apart from E1, all other produced oil samples had higher viscosities than the original bitumen.

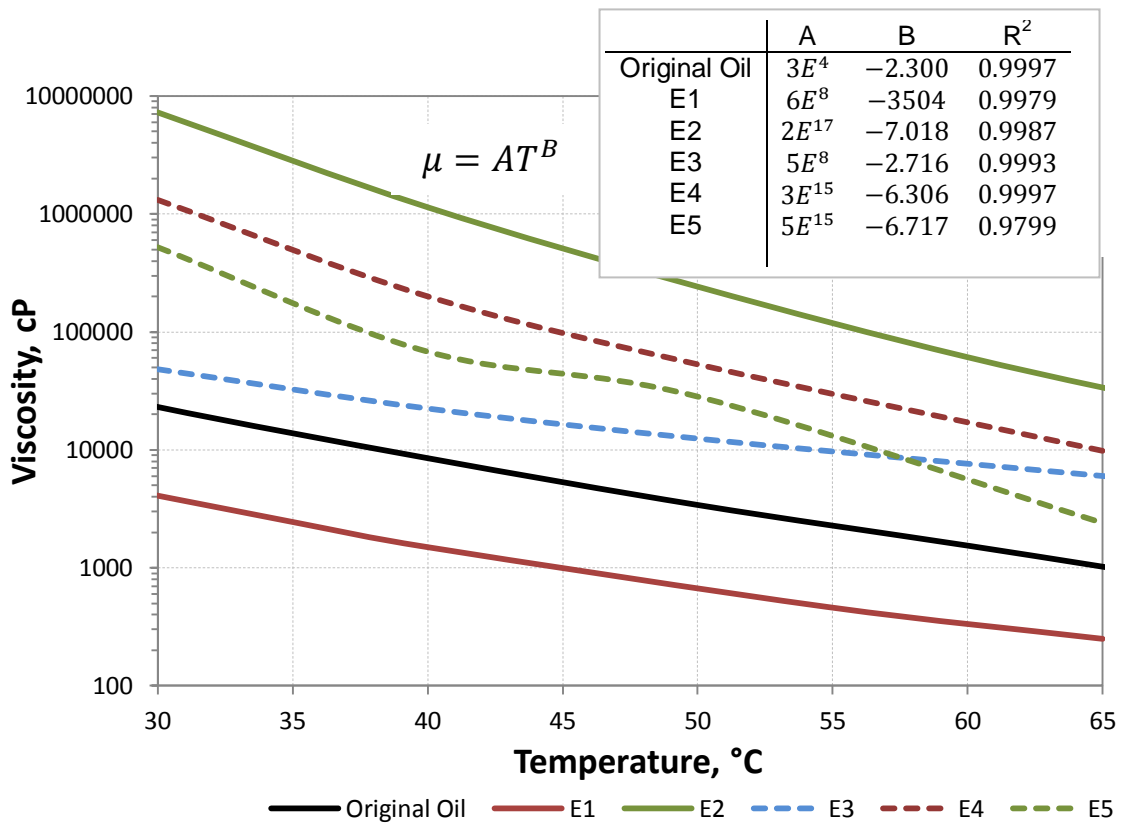


Figure 12 – Viscosity as a Function of Temperature for Produced Oil Samples from All Experiments

While the clay and water content of produced oil causes an increase in viscosity (Coelho and Hascakir 2015, Farah et al. 2005), the solvent content reduces their viscosities (Figure 3). Note that asphaltenes are polar; hence the higher asphaltene content in produced oil increased the water content of produced oil and this in turn also increased the viscosity.

In E1 and E2, in which steam is not involved, the clay content and asphaltene content of the produced oil were responsible for the high viscosity, as E2 produced a high asphaltene content (Figure 6). Conversely, for the steam experiments there was an interesting scenario. The viscosity of E3 was the lowest one among the steam injection experiments (E3, E4 and E5), yet it was still higher than the original bitumen. This discrepancy is caused by the water-in-oil emulsions present in the oil, which increases the viscosity of said oils (Farah et al. 2005). E4 had a higher viscosity than E3, explained again by its higher clay and asphaltene content. E5 had an intermediate viscosity, which was due to the irremovable solvent content. Note that the CS has a very high boiling point of up to 210°C, meaning that it evaporates very slowly. The intermediate viscosity seen for E5 derived from CS still being present in the oil that did not have enough time to evaporate. Had all the solvent been removed, the viscosity would have been the highest for E5.

Postmortem Analyses

In this section, the spent rock samples were analyzed to determine sweep efficiencies obtained by the end of each experiment. Figure 13 shows the visual inspection of post-mortem samples. The top of each picture indicates injection point and the bottom is the production point. Hence flow is from top to bottom.

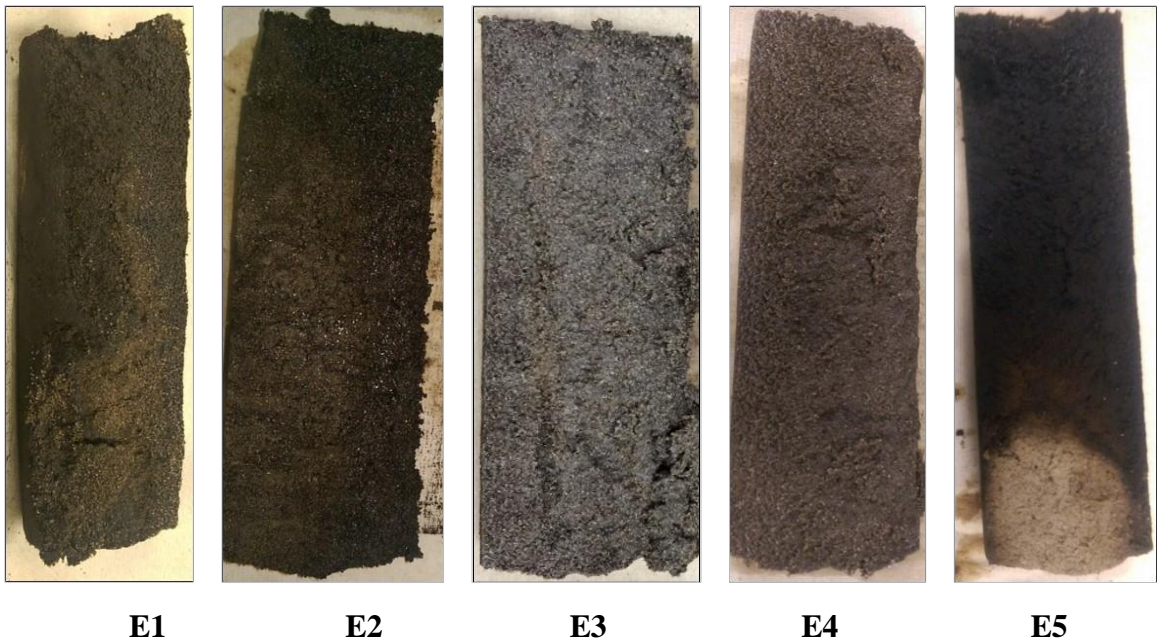


Figure 13 – Postmortem Pictures from Each Experiment (Top of Each Picture is Injection Point and Bottom is Production)

Residual Oil Content

The post-mortem sample was washed with a known quantity of n-pentane to remove the DAO. After weighing, the sample was then washed with toluene to remove any leftover asphaltenes. Table 3 describes the residual oil composition of the post-mortem near the inlet and outlet of each sample. The solvent interaction with the asphaltenes changed the composition of the residual oil. During the miscible flooding

experiments the lower molecular weight solvent (n-hexane) carried less asphaltenes out of the sample, leaving behind and possibly precipitating. E2 had lower asphaltene content on the spent rock, corroborating the produced oil results that showed that the CS was more efficient at dissolving the asphaltenes; the same trend can be seen on E4 and E5. Steam injection shows the highest amount of asphaltene still in the rock, again following the produced oil composition.

Table 3- Residual Oil Composition

Experiment	Position	DAO Content, %	Asphaltene Content, %	Residual Oil, %
E1	Inlet	56	44	59
	Outlet	73	27	20
E2	Inlet	74	26	48
	Outlet	80	20	34
E3	Inlet	89	11	85
	Outlet	89	11	91
E4	Inlet	86	14	49
	Outlet	86	14	44
E5	Inlet	66	34	72
	Outlet	90	10	53

Contact Angle

Contact angle measurements on the postmortem samples were accomplished using the Kruss DSA30S Drop Shape Analyzer. Figure 14 shows images collected during measurements. The Kruss DSA30S Drop Shape Analyzer produces water-in-air contact angle measurements. Due to the high viscosity of the bitumen water-oil contact angle measurements were not possible. However, wettability definition is given on

water-oil contact angles (Anderson 1986). Accordingly contact angles between 0°-75° are considered water wet, 75°-105° are considered intermediate wet and 105°-180° are considered to be oil wet. To determine wettability of the postmortem samples a correlation was used (Grate et al. 2012), which converts water-air contact angles into water-oil contact angles. The measure water-air and calculated water-oil contact angles are given in Table 4.

Table 4 - Contact Angle Values for the Samples from Inlet and Outlet of the Postmortems Samples

Experiment	Position	Contact Angle Air/Water	Contact Angle Oil/Water
E1	Inlet	101.1	142.4
	Outlet	124.1	174.8
E2	Inlet	91.3	128.6
	Outlet	107.2	150.9
E3	Inlet	111.5	157.0
	Outlet	117.5	165.5
E4	Inlet	94.1	132.6
	Outlet	100.1	141.0
E5	Inlet	101.7	143.3
	Outlet	125.1	176.3

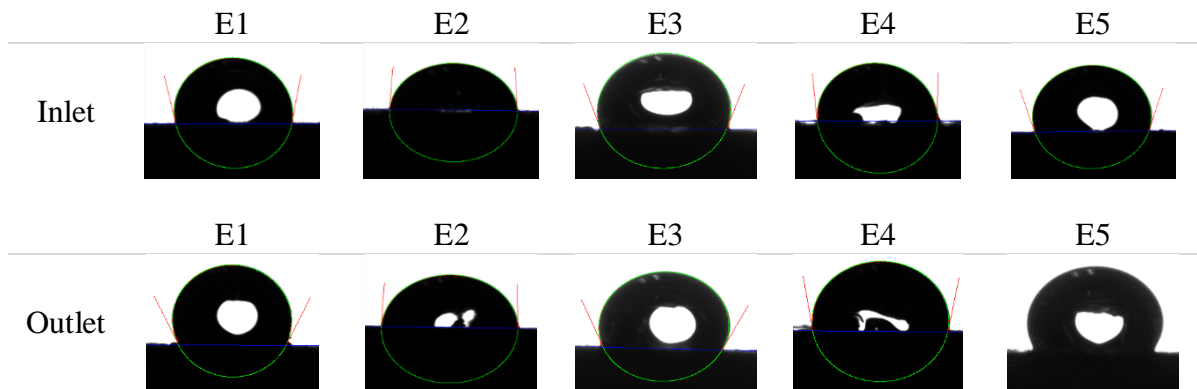


Figure 14 - Contact Angle Images Collected During Contact Angle Measurements, Top Images are from the Inlet and Bottom from the Outlet of the Postmortem Sample

Between E1 and E2 we see that the contact angle was highest for the experiment using the n-hexane. As the asphaltenes precipitate they adsorb onto the rock, increasing its wettability to oil. The asphaltenes precipitated by CS will also be more polar, leading to higher affinity with water and therefore a smaller contact angle (Speight 2006).

On the steam experiments however, this trend is reversed. E4 had a lower contact angle while E5 had the highest, and E3 had a measurement in between. This low contact angle for E4 plays a part in the high recovery rate of this experiment, as wettability changes have great impact during production of high density oils (Prats 1982). The combination of CS with steam appears to be very detrimental to the wettability of the rock. E5 had the highest contact angle, which most certainly leads to its lower performance. The interaction of the steam and solvent with the bitumen-clay system generated a very good environment for oil retention on the surface of the rock. Any asphaltene precipitated in E5 had a very high polarity, and would therefore interact with

the steam, increasing the asphaltene production even more. The accumulation of all these contributing factor caused the eventual lower recovery rate of experiment E5.

Repeatability

To evaluate the repeatability of the results presented a sixth experiment was conducted, with the same experimental conditions as E4. The results of these experiments were then compared to determine their validity.

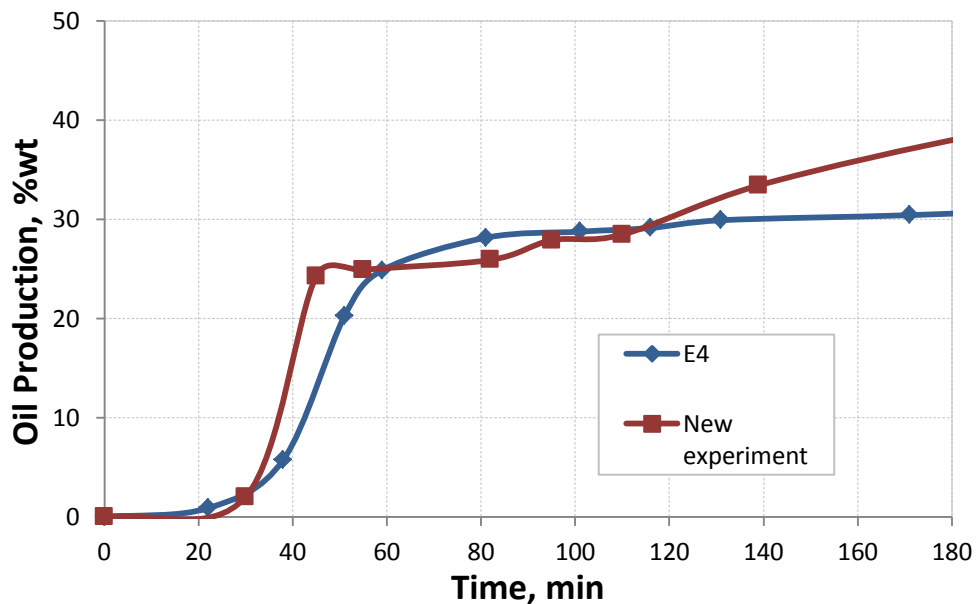


Figure 15 - Cumulative Oil Production from Experiment E4 and the New Experiment Designed to Replicate E4 (E4 and new experiment – n-C₆ + Steam Flooding)

The new experiment had an oil production curve very similar to E4 (Figure 15), in both total oil produced as well as production rate. The experimental results diverged slightly at the end of the run. During the middle of the second experiment the injection flow had to be stopped for approximately 20 minutes due to a leakage, which could be

the cause of this slight difference. The similarities between the two experiments shows that the experimental results achieved during each of the five experiments could be replicated consistently throughout other runs.

A TGA/DTA analyzes was performed on the produced oil of the second experiment, to determine the water content of the oil (see Appendix Figure A-9). It shows that the water content was about 39%, similar to the produced oil from E3 (34%), and very different than the one from E5 (80%).

CONCLUSIONS

Five 1D flooding experiments were run, two miscible floodings, one steam flooding and two solvent assisted-steam flooding. A new high carbon number solvent (CS) was tested for the first time, and its performance compared to n-hexane. Performance evaluation was made by comparing produced oil and residual oil quality.

The new CS showed lower interaction with clays during the miscible or solvent assisted-steam flooding processes. However, CS assisted-steam flooding carried more water in produced oil. When the asphaltene contents of the produced oils were compared, our study suggested that the emulsion formation is favored more by the asphaltene content of oil rather than clay content. CS always produced more asphaltenes than n-hexane, both during miscible flooding and solvent assisted-steam flooding, hence the produced oil quality was observed poorer for CS options.

Based on these observations high molecular weight hydrocarbon solvents such as CS are not recommended for use during SA-SF processes, as they will not perform as well as lighter solvents, and the oil produced will have a higher viscosity, more stable emulsions and higher asphaltene content, resulting in a lower overall quality of the oil.

If the toxicity, handling, and cost of solvents are considered then CS will be a better option. But in terms of oil processing and oil production then n-hexane will produce higher quality oil with less water and asphaltene content. Hence, we recommend fundamental research on solvent asphaltene interaction prior to application of solvents during any solvent based process.

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APPENDIX



Figure A-1 - D-Series Water Pump (on the left) and LC 5000 Water pump (on the right)

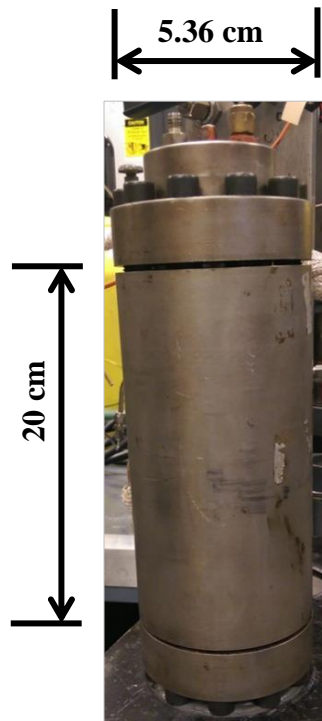


Figure A-2 – Picture of the Core Holder



Figure A-3 – Picture showing the two phase separation of produced fluids in bottles

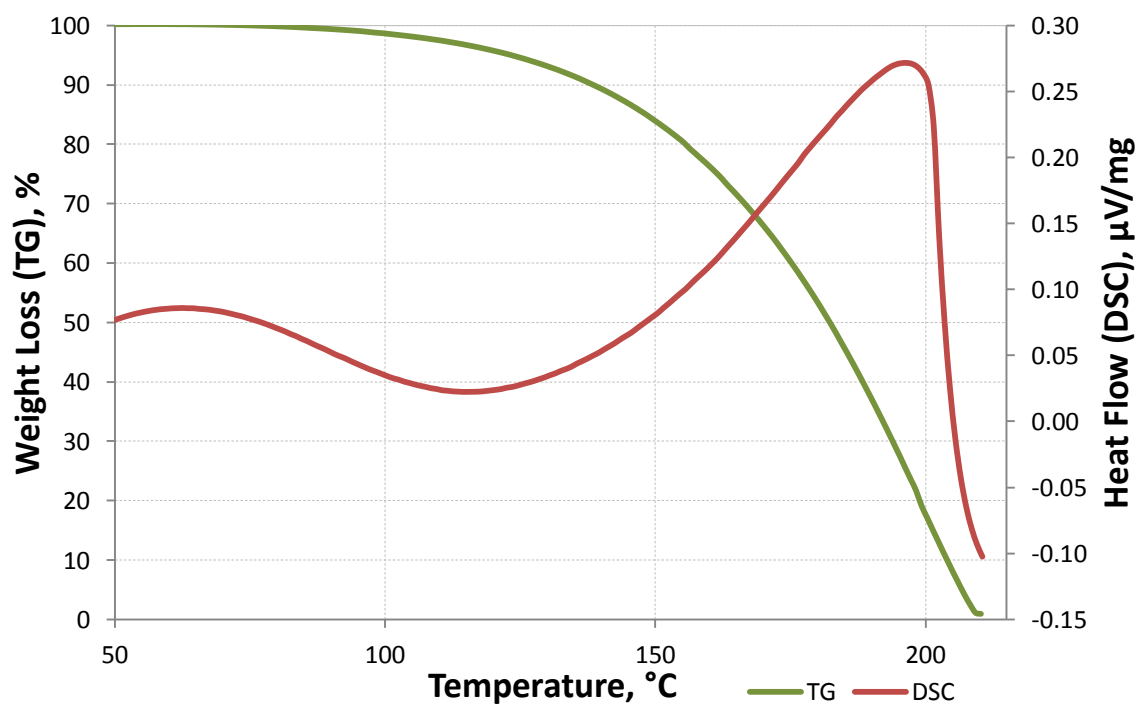


Figure A-4 – Reference TGA/DSC of Commercial Solvent

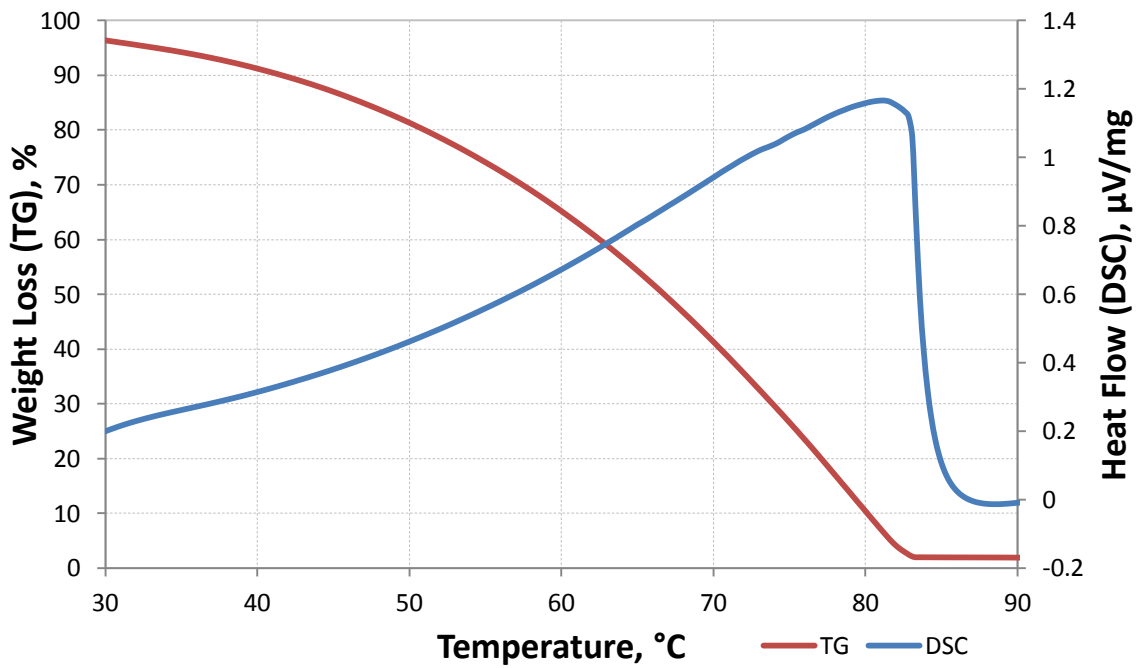


Figure A-5 – Reference TGA/DSC of Distilled Water

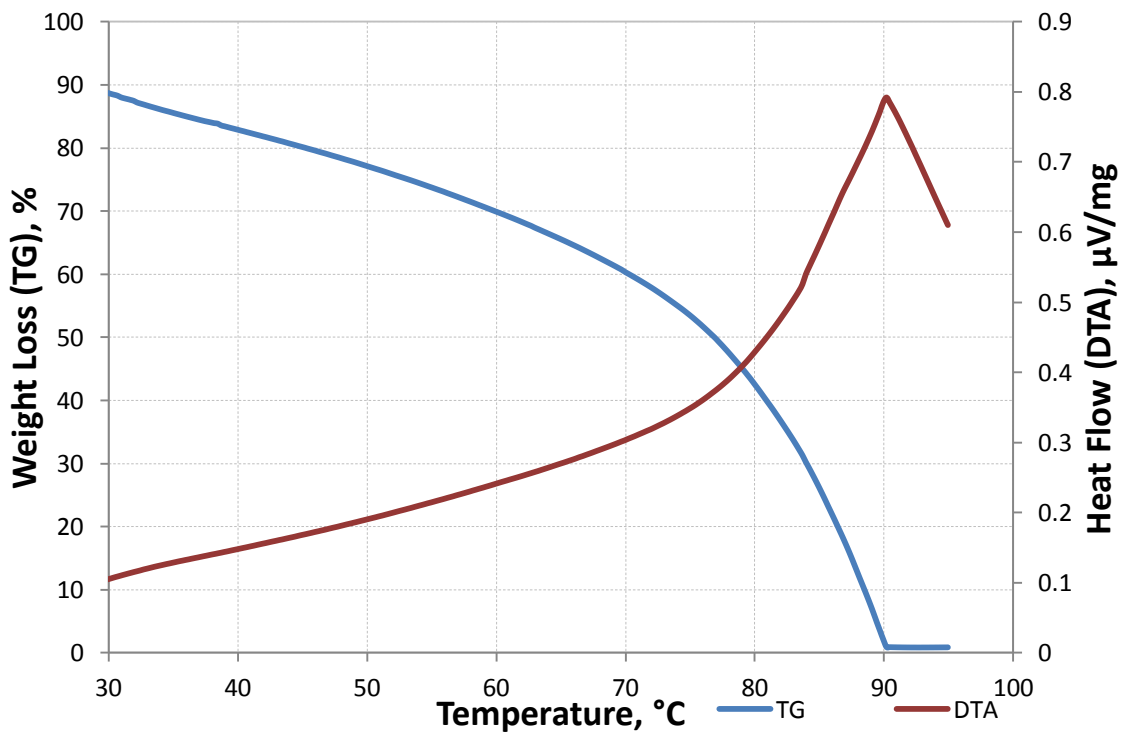


Figure A-6 – Reference TGA/DTA of n-Hexane

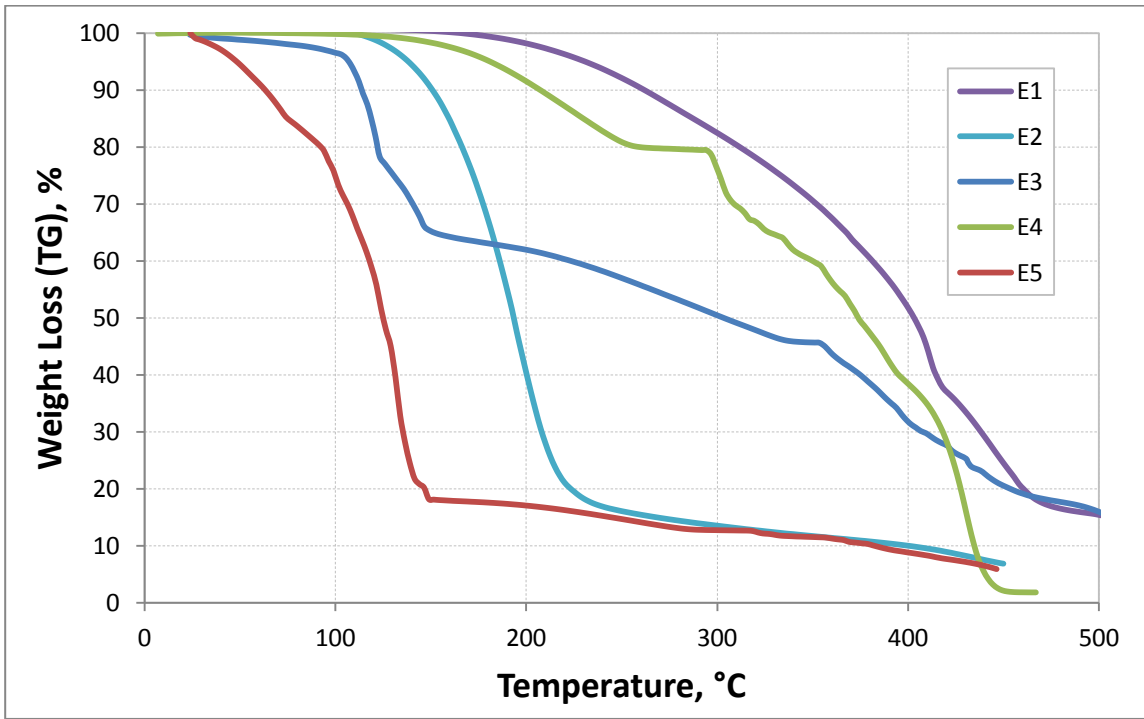


Figure A-7 - TGA of Produced Oil from E1, E2, E3, E4, and E5

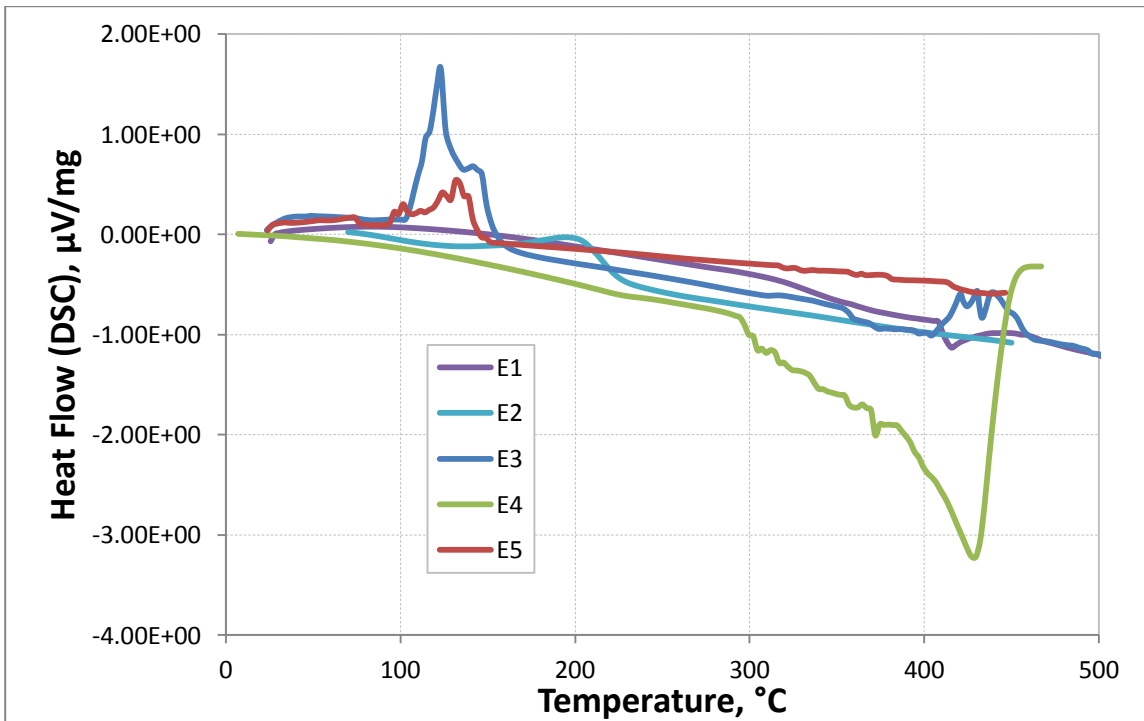


Figure A-8 – Heat Flow Behavior of Produced Oil from experiments E1, E2, E3, E4, and E5

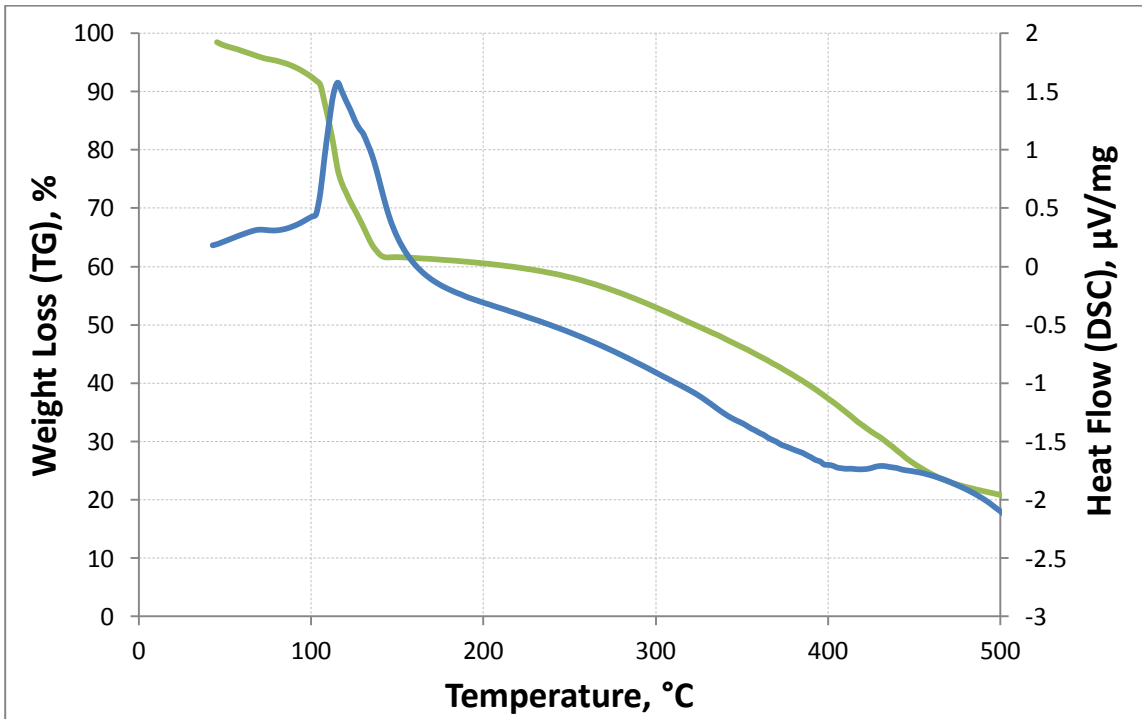


Figure A-9 – Weight Loss and Heat Flow behavior of produced oil from the new experiment designed to test repeatability

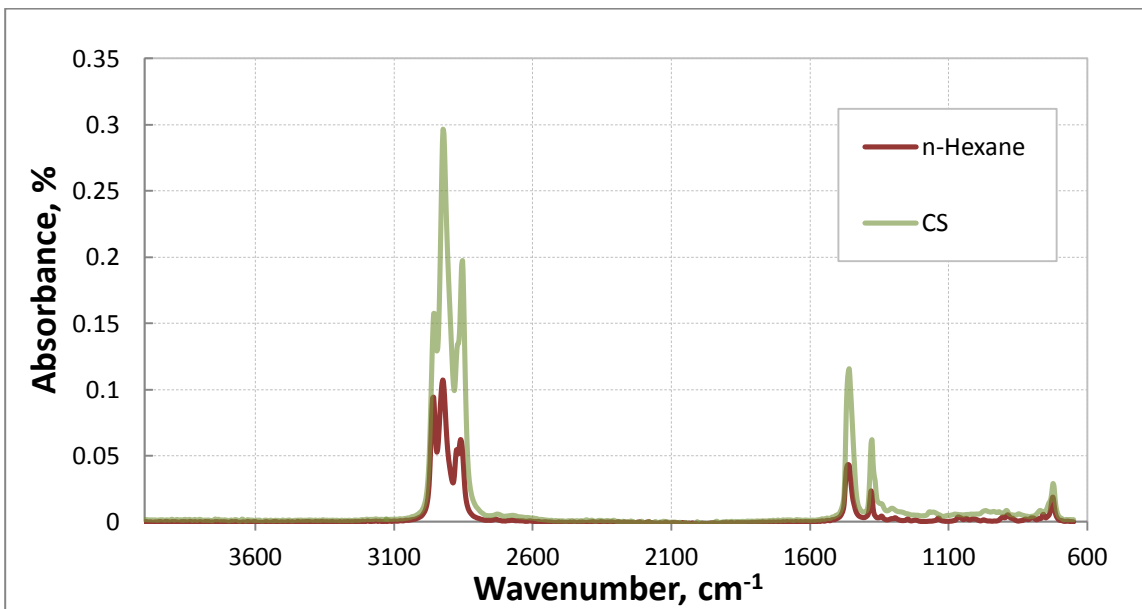


Figure A-10 - Reference Fourier Transform Infrared Spectroscopy results for n-hexane and CS

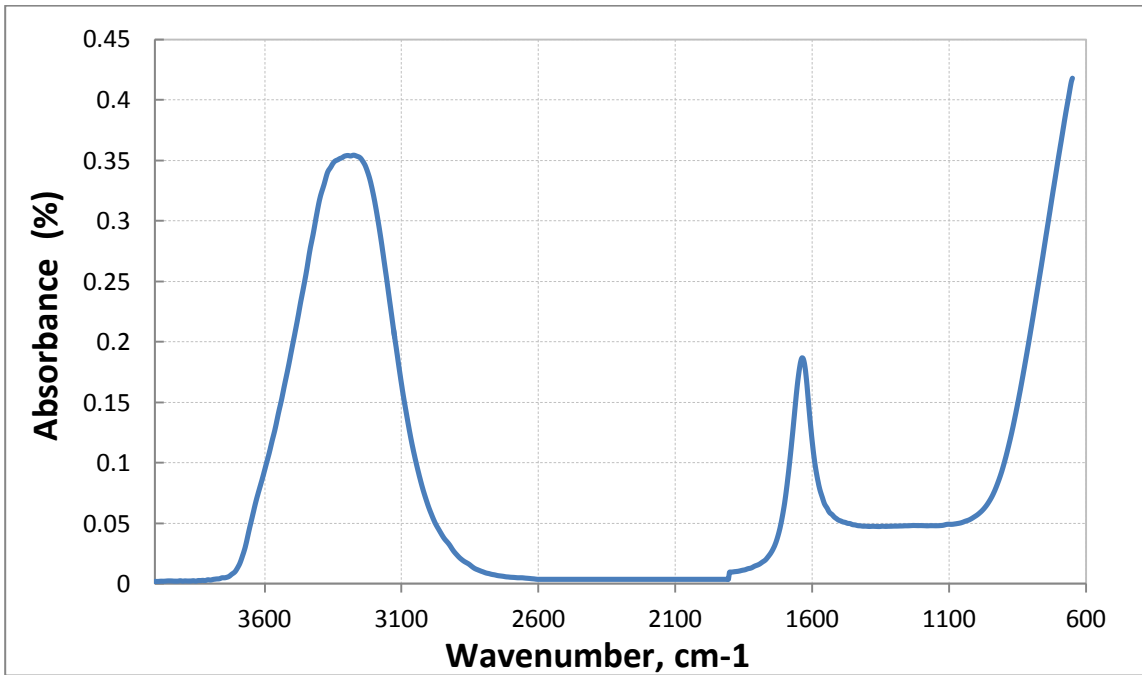


Figure A-11 - Reference Fourier Transform Infrared Spectroscopy results for distilled water

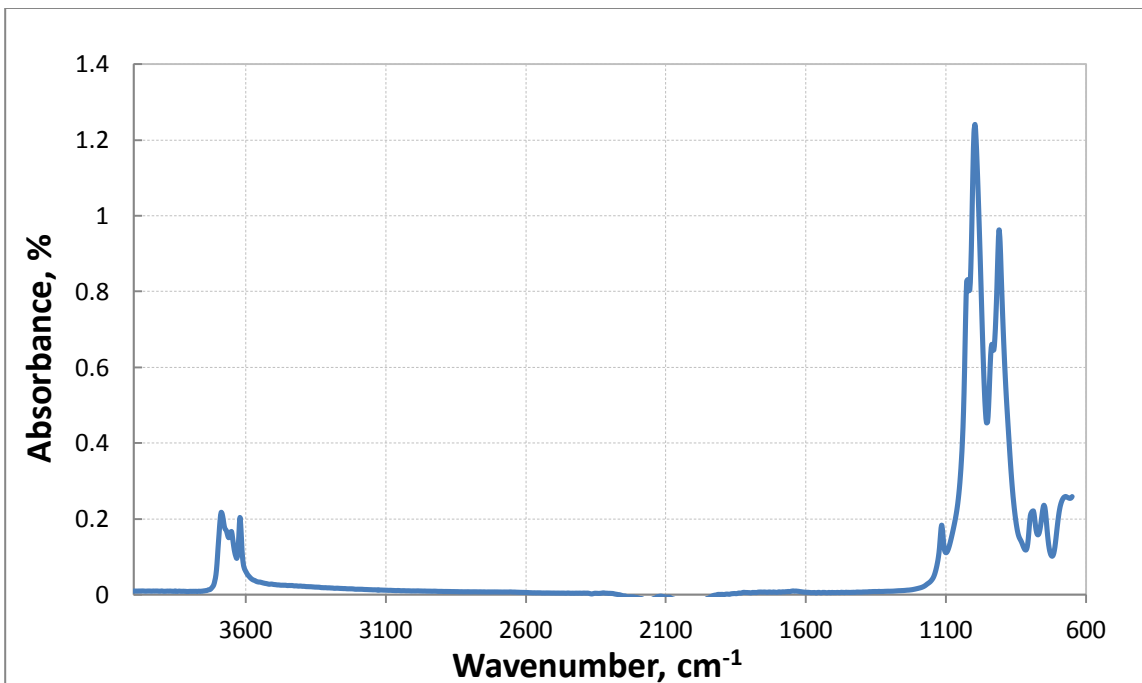


Figure A-12 - Reference Fourier Transform Infrared Spectroscopy results for clay

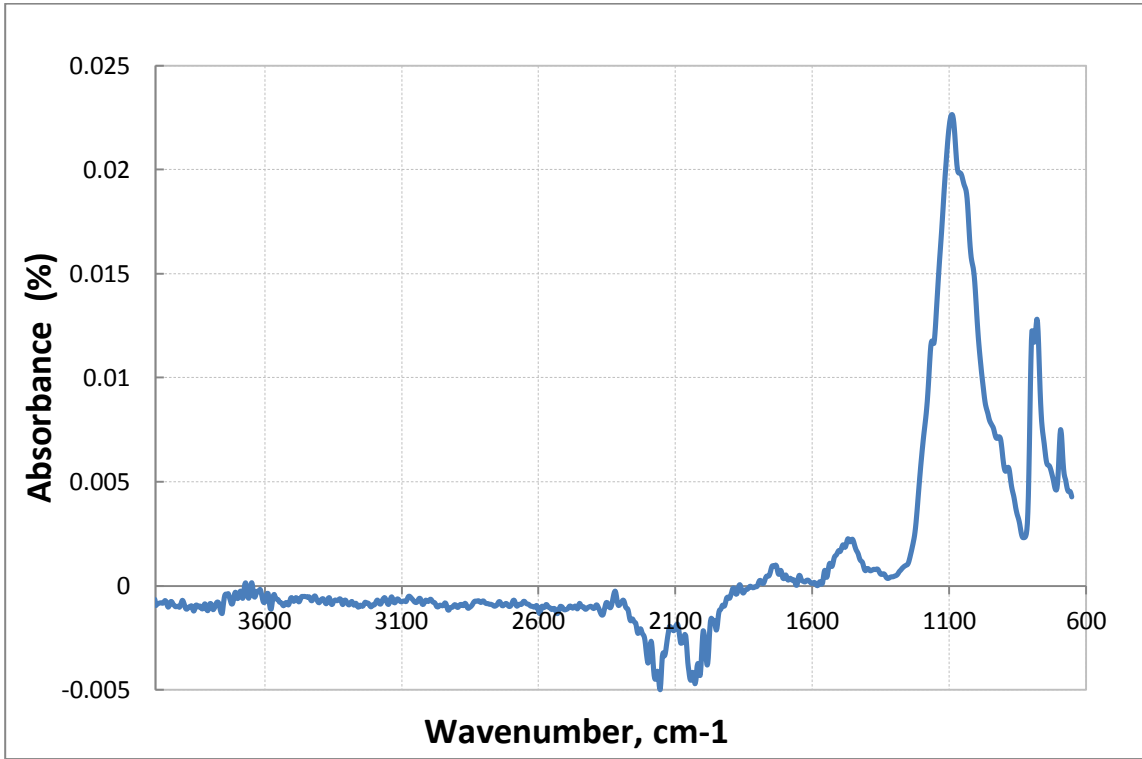


Figure A-13 - Reference Fourier Transform Infrared Spectroscopy results for Ottawa sand

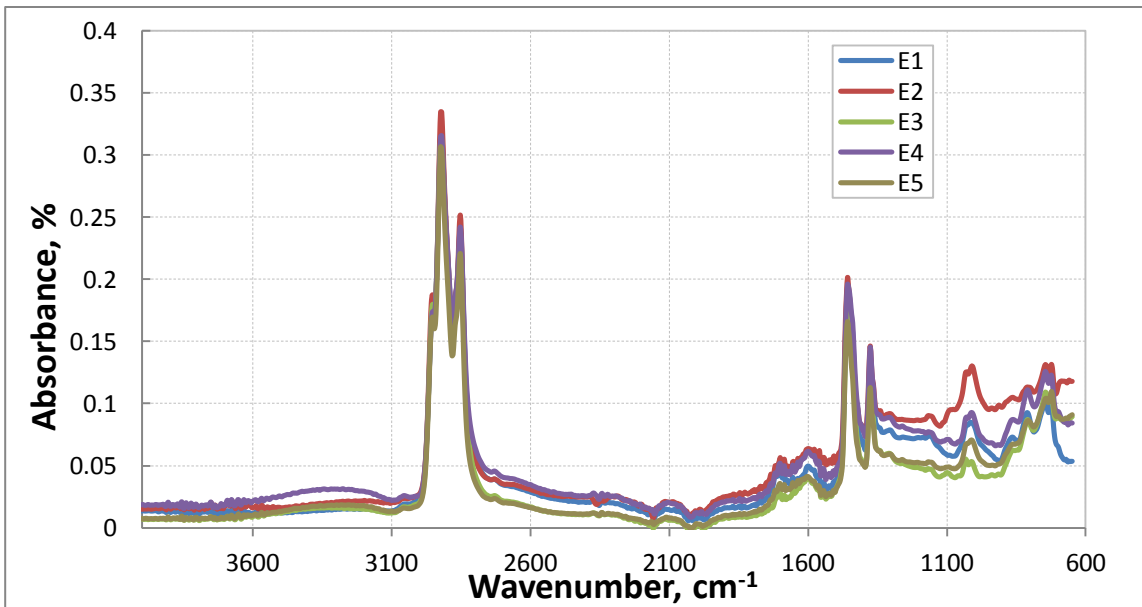


Figure A-14 – Fourier Transform Infrared Spectroscopy results of produced oil from E1 (blue curve), E2 (red curve), E3 (green curve), E4 (purple curve), E5 (brown curve).

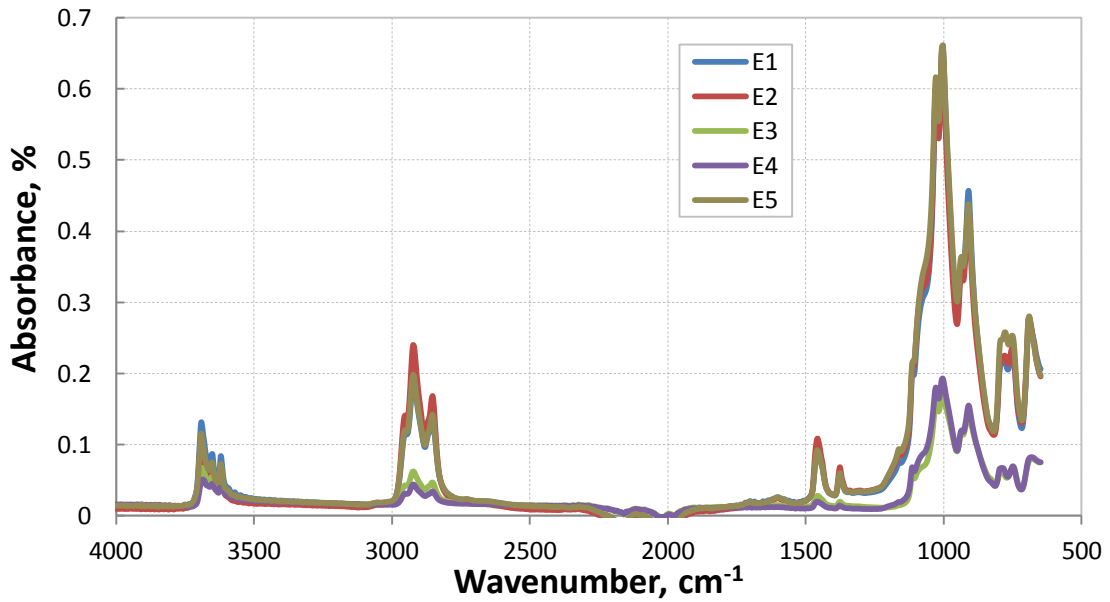


Figure A-15 – Fourier Transform Infrared Spectroscopy results of inlet side of spent rock samples from E1 (blue curve), E2 (red curve), E3 (green curve), E4 (purple curve), E5 (brown curve).

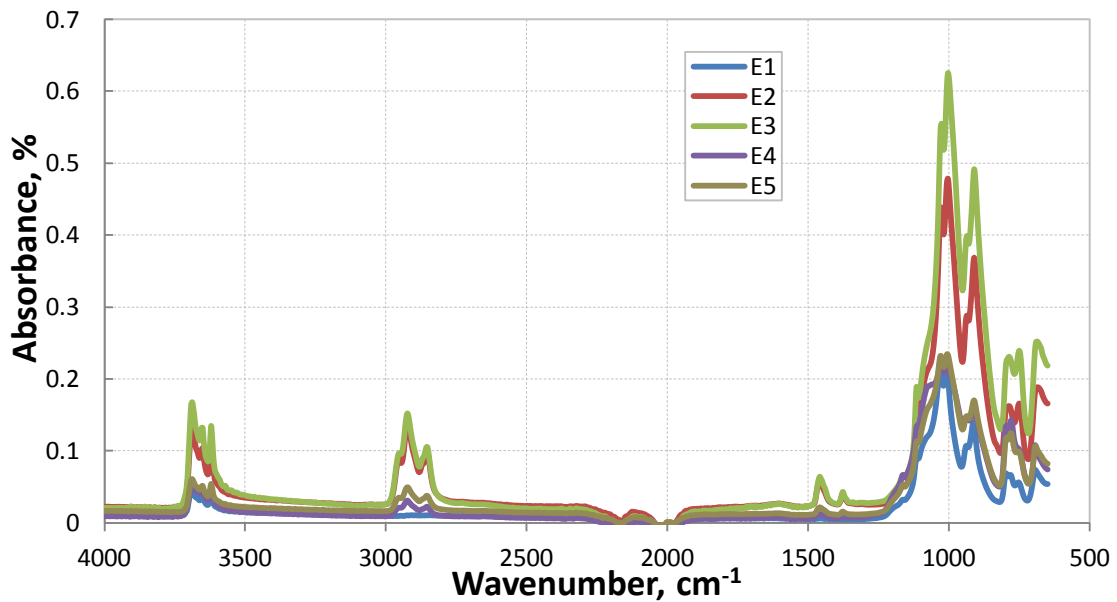


Figure A-16 – Fourier Transform Infrared Spectroscopy results of outlet side of spent rock samples from E1 (blue curve), E2 (red curve), E3 (green curve), E4 (purple curve), E5 (brown curve).

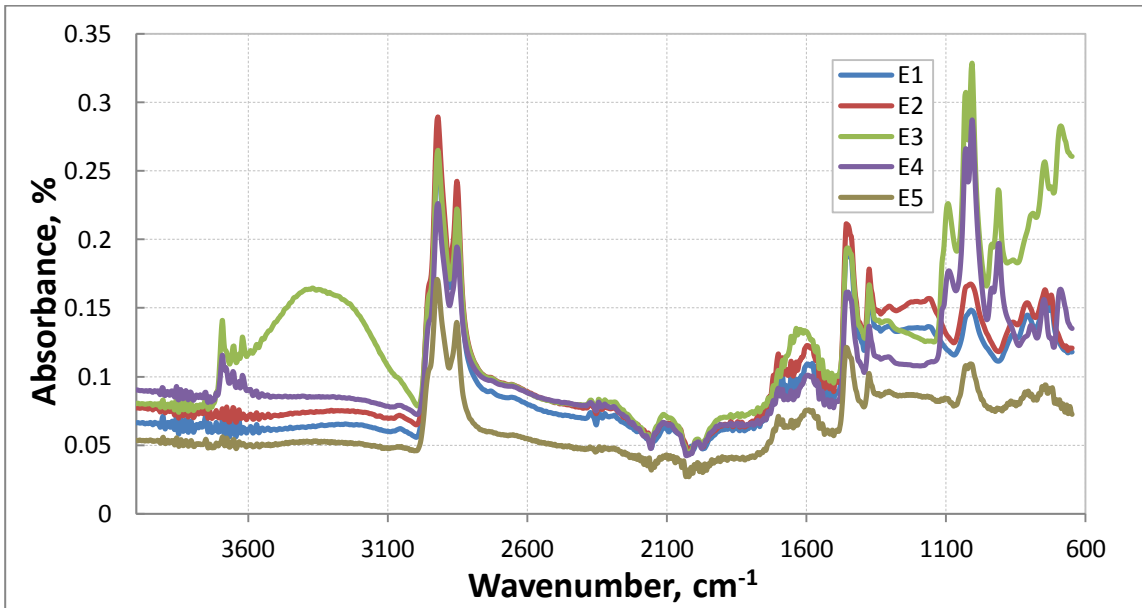


Figure A-17 - Fourier Transform Infrared Spectroscopy results of asphaltenes from produced oil from E1 (blue curve), E2 (red curve), E3 (green curve), E4 (purple curve), E5 (brown curve).

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SAFETY DATA SHEET

SECTION 1 PRODUCT AND COMPANY IDENTIFICATION

PRODUCT

Product Name: ESCAID™ 115
Product Description: Dearomatized Hydrocarbons

Intended Use: Drilling muds, oil-based

COMPANY IDENTIFICATION

Supplier: EXXONMOBIL CHEMICAL COMPANY
P.O. BOX 3272
HOUSTON, TX. 77253-3272 USA

24 Hour Health Emergency	(800) 728-2015
Transportation Emergency Phone	(800) 424-9300 or (703) 527-3887 CHEMTREC
Product Technical Information	(832) 624-8500
Supplier General Contact	(832) 624-8500

SECTION 2 HAZARDS IDENTIFICATION

This material is hazardous according to regulatory guidelines (see (M)SDS Section 15).

CLASSIFICATION:

Aspiration toxicant: Category 1.

LABEL:

Pictogram:



Signal Word: Danger

Hazard Statements:

H304: May be fatal if swallowed and enters airways.

Precautionary Statements:

P301 + P310: IF SWALLOWED: Immediately call a POISON CENTER or doctor/physician. P331: Do NOT induce vomiting. P405: Store locked up. P501: Dispose of contents and container in accordance with local regulations.

Other hazard information:

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HAZARD NOT OTHERWISE CLASSIFIED (HNOC): None as defined under 29 CFR 1900.1200.

PHYSICAL / CHEMICAL HAZARDS

Material can accumulate static charges which may cause an ignition.

HEALTH HAZARDS

May be irritating to the eyes, nose, throat, and lungs. Repeated exposure may cause skin dryness or cracking.

ENVIRONMENTAL HAZARDS

No significant hazards.

NFPA Hazard ID: Health: 1 Flammability: 1 Reactivity: 0
HMIS Hazard ID: Health: 1* Flammability: 1 Reactivity: 0

NOTE: This material should not be used for any other purpose than the intended use in Section 1 without expert advice. Health studies have shown that chemical exposure may cause potential human health risks which may vary from person to person.

SECTION 3 COMPOSITION / INFORMATION ON INGREDIENTS

This material is defined as a substance.

Hazardous Substance(s) or Complex Substance(s) required for disclosure

Name	CAS#	Concentration*	GHS Hazard Codes
DISTILLATES (PETROLEUM), HYDROTREATED LIGHT	64742-47-8	100 %	H304

* All concentrations are percent by weight unless material is a gas. Gas concentrations are in percent by volume.

As per paragraph (i) of 29 CFR 1910.1200, formulation is considered a trade secret and specific chemical identity and exact percentage (concentration) of composition may have been withheld. Specific chemical identity and exact percentage composition will be provided to health professionals, employees, or designated representatives in accordance with applicable provisions of paragraph (i).

SECTION 4 FIRST AID MEASURES

INHALATION

Remove from further exposure. For those providing assistance, avoid exposure to yourself or others. Use adequate respiratory protection. If respiratory irritation, dizziness, nausea, or unconsciousness occurs, seek immediate medical assistance. If breathing has stopped, assist ventilation with a mechanical device or use mouth-to-mouth resuscitation.

SKIN CONTACT

Wash contact areas with soap and water. Remove contaminated clothing. Launder contaminated clothing before reuse.

EYE CONTACT

Flush thoroughly with water. If irritation occurs, get medical assistance.

INGESTION

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Seek immediate medical attention. Do not induce vomiting.

NOTE TO PHYSICIAN

If ingested, material may be aspirated into the lungs and cause chemical pneumonitis. Treat appropriately.

SECTION 5	FIRE FIGHTING MEASURES
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EXTINGUISHING MEDIA

Appropriate Extinguishing Media: Use water fog, foam, dry chemical or carbon dioxide (CO₂) to extinguish flames.

Inappropriate Extinguishing Media: Straight Streams of Water

FIRE FIGHTING

Fire Fighting Instructions: Evacuate area. Prevent runoff from fire control or dilution from entering streams, sewers, or drinking water supply. Firefighters should use standard protective equipment and in enclosed spaces, self-contained breathing apparatus (SCBA). Use water spray to cool fire exposed surfaces and to protect personnel.

Hazardous Combustion Products: Oxides of carbon, Smoke, Fume, Incomplete combustion products

FLAMMABILITY PROPERTIES

Flash Point [Method]: 96°C (205°F) [ASTM D-93]

Flammable Limits (Approximate volume % in air): LEL: 0.6 UEL: 4.9

Autoignition Temperature: 215°C (419°F)

SECTION 6	ACCIDENTAL RELEASE MEASURES
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NOTIFICATION PROCEDURES

In the event of a spill or accidental release, notify relevant authorities in accordance with all applicable regulations. US regulations require reporting releases of this material to the environment which exceed the applicable reportable quantity or oil spills which could reach any waterway including intermittent dry creeks. The National Response Center can be reached at (800)424-8802.

PROTECTIVE MEASURES

Avoid contact with spilled material. See Section 5 for fire fighting information. See the Hazard Identification Section for Significant Hazards. See Section 4 for First Aid Advice. See Section 8 for advice on the minimum requirements for personal protective equipment. Additional protective measures may be necessary, depending on the specific circumstances and/or the expert judgment of the emergency responders.

For emergency responders: Respiratory protection: half-face or full-face respirator with filter(s) for organic vapor and, when applicable, H₂S, or Self Contained Breathing Apparatus (SCBA) can be used depending on the size of spill and potential level of exposure. If the exposure cannot be completely characterized or an oxygen deficient atmosphere is possible or anticipated, SCBA is recommended. Work gloves that are resistant to aromatic hydrocarbons are recommended. Note: gloves made of polyvinyl acetate (PVA) are not water-resistant and are not suitable for emergency use. Chemical goggles are recommended if splashes or contact with eyes is possible. Small spills: normal antistatic work clothes are usually adequate. Large spills: full body suit of chemical resistant, antistatic material is recommended.

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SPILL MANAGEMENT

Land Spill: Stop leak if you can do it without risk. Absorb or cover with dry earth, sand or other non-combustible material and transfer to containers. Recover by pumping or with suitable absorbent.

Water Spill: Stop leak if you can do it without risk. Warn other shipping. Remove from the surface by skimming or with suitable absorbents. Seek the advice of a specialist before using dispersants.

Water spill and land spill recommendations are based on the most likely spill scenario for this material; however, geographic conditions, wind, temperature, (and in the case of a water spill) wave and current direction and speed may greatly influence the appropriate action to be taken. For this reason, local experts should be consulted. Note: Local regulations may prescribe or limit action to be taken.

ENVIRONMENTAL PRECAUTIONS

Large Spills: Dike far ahead of liquid spill for later recovery and disposal. Prevent entry into waterways, sewers, basements or confined areas.

SECTION 7 HANDLING AND STORAGE

HANDLING

Avoid contact with skin. Prevent small spills and leakage to avoid slip hazard. Material can accumulate static charges which may cause an electrical spark (ignition source). When the material is handled in bulk, an electrical spark could ignite any flammable vapors from liquids or residues that may be present (e.g., during switch-loading operations). Use proper bonding and/or ground procedures. However, bonding and grounds may not eliminate the hazard from static accumulation. Consult local applicable standards for guidance. Additional references include American Petroleum Institute 2003 (Protection Against Ignitions Arising out of Static, Lightning and Stray Currents) or National Fire Protection Agency 77 (Recommended Practice on Static Electricity) or CENELEC CLC/TR 50404 (Electrostatics - Code of practice for the avoidance of hazards due to static electricity).

Loading/Unloading Temperature: [Ambient]

Transport Temperature: [Ambient]

Transport Pressure: [Ambient]

Static Accumulator: This material is a static accumulator. A liquid is typically considered a nonconductive, static accumulator if its conductivity is below 100 pS/m (100x10E-12 Siemens per meter) and is considered a semiconductive, static accumulator if its conductivity is below 10,000 pS/m. Whether a liquid is nonconductive or semiconductive, the precautions are the same. A number of factors, for example liquid temperature, presence of contaminants, anti-static additives and filtration can greatly influence the conductivity of a liquid.

STORAGE

The container choice, for example storage vessel, may effect static accumulation and dissipation. Do not store in open or unlabelled containers. Keep container closed. Handle containers with care. Open slowly in order to control possible pressure release. Store in a cool, well-ventilated area.

Storage Temperature: [Ambient]

Storage Pressure: [Ambient]

Suitable Containers/Packing: Tank Cars; Barges; Drums

Suitable Materials and Coatings (Chemical Compatibility): Stainless Steel; Teflon; Polyethylene; Carbon Steel; Polypropylene

Unsuitable Materials and Coatings: Polystyrene; Butyl Rubber; Natural Rubber; Ethylene-propylene-diene

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monomer (EPDM)

SECTION 8 EXPOSURE CONTROLS / PERSONAL PROTECTION

EXPOSURE LIMIT VALUES

Exposure limits/standards (Note: Exposure limits are not additive)

Substance Name	Form	Limit / Standard			NOTE	Source
DISTILLATES (PETROLEUM), HYDROTREATED LIGHT	Vapor.	RCP - TWA	1200 mg/m3	166 ppm	Total Hydrocarbon s	ExxonMobil

NOTE: Limits/standards shown for guidance only. Follow applicable regulations.

No biological limits allocated.

ENGINEERING CONTROLS

The level of protection and types of controls necessary will vary depending upon potential exposure conditions. Control measures to consider:

Adequate ventilation should be provided so that exposure limits are not exceeded.

PERSONAL PROTECTION

Personal protective equipment selections vary based on potential exposure conditions such as applications, handling practices, concentration and ventilation. Information on the selection of protective equipment for use with this material, as provided below, is based upon intended, normal usage.

Respiratory Protection: If engineering controls do not maintain airborne contaminant concentrations at a level which is adequate to protect worker health, an approved respirator may be appropriate. Respirator selection, use, and maintenance must be in accordance with regulatory requirements, if applicable. Types of respirators to be considered for this material include:

Half-face filter respirator

For high airborne concentrations, use an approved supplied-air respirator, operated in positive pressure mode. Supplied air respirators with an escape bottle may be appropriate when oxygen levels are inadequate, gas/vapor warning properties are poor, or if air purifying filter capacity/rating may be exceeded.

Hand Protection: Any specific glove information provided is based on published literature and glove manufacturer data. Glove suitability and breakthrough time will differ depending on the specific use conditions. Contact the glove manufacturer for specific advice on glove selection and breakthrough times for your use conditions. Inspect and replace worn or damaged gloves. The types of gloves to be considered for this material include:

If prolonged or repeated contact is likely, chemical resistant gloves are recommended. If contact with forearms is likely, wear gauntlet style gloves.

Eye Protection: If contact is likely, safety glasses with side shields are recommended.

Skin and Body Protection: Any specific clothing information provided is based on published literature or

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manufacturer data. The types of clothing to be considered for this material include:
If prolonged or repeated contact is likely, chemical, and oil resistant clothing is recommended.

Specific Hygiene Measures: Always observe good personal hygiene measures, such as washing after handling the material and before eating, drinking, and/or smoking. Routinely wash work clothing and protective equipment to remove contaminants. Discard contaminated clothing and footwear that cannot be cleaned. Practice good housekeeping.

ENVIRONMENTAL CONTROLS

Comply with applicable environmental regulations limiting discharge to air, water and soil. Protect the environment by applying appropriate control measures to prevent or limit emissions.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Note: Physical and chemical properties are provided for safety, health and environmental considerations only and may not fully represent product specifications. Contact the Supplier for additional information.

GENERAL INFORMATION

Physical State: Liquid
Form: Clear
Color: Colorless
Odor: Mild Petroleum/Solvent
Odor Threshold: N/D

IMPORTANT HEALTH, SAFETY, AND ENVIRONMENTAL INFORMATION

Relative Density (at 15.6 °C): 0.8
Density (at 15.6 °C): 799 kg/m³ (6.67 lbs/gal, 0.8 kg/dm³)
Flammability (Solid, Gas): N/A
Flash Point [Method]: 96°C (205°F) [ASTM D-93]
Flammable Limits (Approximate volume % in air): LEL: 0.6 UEL: 4.9
Autoignition Temperature: 215°C (419°F)
Boiling Point / Range: 227°C (441°F) - 253°C (487°F)
Decomposition Temperature: N/D
Vapor Density (Air = 1): 6.5 at 101 kPa
Vapor Pressure: 0.004 kPa (0.03 mm Hg) at 20 °C
Evaporation Rate (n-butyl acetate = 1): 0.5
pH: N/A
Log Pow (n-Octanol/Water Partition Coefficient): N/D
Solubility in Water: Negligible
Viscosity: 2.05 cSt (2.05 mm²/sec) at 40 °C | 2.67 cSt (2.67 mm²/sec) at 25°C
Oxidizing Properties: See Hazards Identification Section.

OTHER INFORMATION

Freezing Point: N/A
Melting Point: N/D
Pour Point: -32°C (-26°F)
Molecular Weight: 187
Coefficient of Thermal Expansion: 0.00072 V/DEGC

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SECTION 10	STABILITY AND REACTIVITY
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REACTIVITY: See sub-sections below.

STABILITY: Material is stable under normal conditions.

CONDITIONS TO AVOID: Open flames and high energy ignition sources.

MATERIALS TO AVOID: Strong oxidizers

HAZARDOUS DECOMPOSITION PRODUCTS: Material does not decompose at ambient temperatures.

POSSIBILITY OF HAZARDOUS REACTIONS: Hazardous polymerization will not occur.

SECTION 11	TOXICOLOGICAL INFORMATION
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INFORMATION ON TOXICOLOGICAL EFFECTS

Hazard Class	Conclusion / Remarks
Inhalation	
Acute Toxicity: (Rat) 8 hour(s) LC50 > 5000 mg/m3 (Vapor)	Minimally Toxic. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 403
Irritation: No end point data for material.	Negligible hazard at ambient/normal handling temperatures.
Ingestion	
Acute Toxicity (Rat): LD50 > 5000 mg/kg	Minimally Toxic. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 401
Skin	
Acute Toxicity (Rabbit): LD50 > 5000 mg/kg	Minimally Toxic. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 402
Skin Corrosion/Irritation: Data available.	May dry the skin leading to discomfort and dermatitis. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 404
Eye	
Serious Eye Damage/Irritation: Data available.	May cause mild, short-lasting discomfort to eyes. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 405
Sensitization	
Respiratory Sensitization: No end point data for material.	Not expected to be a respiratory sensitizer.
Skin Sensitization: Data available.	Not expected to be a skin sensitizer. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 406
Aspiration: Data available.	May be fatal if swallowed and enters airways. Based on physico-chemical properties of the material.
Germ Cell Mutagenicity: Data available.	Not expected to be a germ cell mutagen. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 471 473 474 476 478 479
Carcinogenicity: Data available.	Not expected to cause cancer. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 453
Reproductive Toxicity: Data available.	Not expected to be a reproductive toxicant. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 414 421 422
Lactation: No end point data for material.	Not expected to cause harm to breast-fed children.

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Specific Target Organ Toxicity (STOT)	
Single Exposure: No end point data for material.	Not expected to cause organ damage from a single exposure.
Repeated Exposure: Data available.	Not expected to cause organ damage from prolonged or repeated exposure. Based on test data for structurally similar materials. Test(s) equivalent or similar to OECD Guideline 408 413 422

OTHER INFORMATION

For the product itself:

Vapor/aerosol concentrations above recommended exposure levels are irritating to the eyes and respiratory tract, may cause headaches, dizziness, anesthesia, drowsiness, unconsciousness and other central nervous system effects including death.

Prolonged and/or repeated skin contact with low viscosity materials may defat the skin resulting in possible irritation and dermatitis.

Small amounts of liquid aspirated into the lungs during ingestion or from vomiting may cause chemical pneumonitis or pulmonary edema.

The following ingredients are cited on the lists below: None.

--REGULATORY LISTS SEARCHED--

1 = NTP CARC

3 = IARC 1

5 = IARC 2B

2 = NTP SUS

4 = IARC 2A

6 = OSHA CARC

SECTION 12 ECOLOGICAL INFORMATION

The information given is based on data available for the material, the components of the material, and similar materials.

ECOTOXICITY

Material -- Not expected to be harmful to aquatic organisms.

Material -- Not expected to demonstrate chronic toxicity to aquatic organisms.

MOBILITY

Material -- Low solubility and floats and is expected to migrate from water to the land. Expected to partition to sediment and wastewater solids.

PERSISTENCE AND DEGRADABILITY

Biodegradation:

Material -- Expected to be readily biodegradable.

Hydrolysis:

Material -- Transformation due to hydrolysis not expected to be significant.

Photolysis:

Material -- Transformation due to photolysis not expected to be significant.

Atmospheric Oxidation:

Material -- Expected to degrade rapidly in air



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OTHER ECOLOGICAL INFORMATION
VOC (EPA Method 24): 6.668 lbs/gal

SECTION 13 DISPOSAL CONSIDERATIONS

Disposal recommendations based on material as supplied. Disposal must be in accordance with current applicable laws and regulations, and material characteristics at time of disposal.

DISPOSAL RECOMMENDATIONS

Product is suitable for burning in an enclosed controlled burner for fuel value or disposal by supervised incineration at very high temperatures to prevent formation of undesirable combustion products.

REGULATORY DISPOSAL INFORMATION

RCRA Information: The unused product, in our opinion, is not specifically listed by the EPA as a hazardous waste (40 CFR, Part 261D), nor is it formulated to contain materials which are listed as hazardous wastes. It does not exhibit the hazardous characteristics of ignitability, corrosivity or reactivity and is not formulated with contaminants as determined by the Toxicity Characteristic Leaching Procedure (TCLP). However, used product may be regulated.

Empty Container Warning Empty Container Warning (where applicable): Empty containers may contain residue and can be dangerous. Do not attempt to refill or clean containers without proper instructions. Empty drums should be completely drained and safely stored until appropriately reconditioned or disposed. Empty containers should be taken for recycling, recovery, or disposal through suitably qualified or licensed contractor and in accordance with governmental regulations. **DO NOT PRESSURISE, CUT, WELD, BRAZE, SOLDER, DRILL, GRIND, OR EXPOSE SUCH CONTAINERS TO HEAT, FLAME, SPARKS, STATIC ELECTRICITY, OR OTHER SOURCES OF IGNITION. THEY MAY EXPLODE AND CAUSE INJURY OR DEATH.**

SECTION 14 TRANSPORT INFORMATION

LAND (DOT): Not Regulated for Land Transport

LAND (TDG): Not Regulated for Land Transport

SEA (IMDG): Not Regulated for Sea Transport according to IMDG-Code

Marine Pollutant: No

AIR (IATA): Not Regulated for Air Transport

SECTION 15 REGULATORY INFORMATION



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OSHA HAZARD COMMUNICATION STANDARD: This material is considered hazardous in accordance with OSHA HazCom 2012, 29 CFR 1910.1200.

Listed or exempt from listing/notification on the following chemical inventories: AICS, DSL, ENCS, IECSC, KECI, PICCS, TSCA

EPCRA SECTION 302: This material contains no extremely hazardous substances.

CERCLA: This material is not subject to any special reporting under the requirements of the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA). Contact local authorities to determine if other reporting requirements apply.

CWA / OPA: This product is classified as an oil under Section 311 of the Clean Water Act (40 CFR 110) and the Oil Pollution Act of 1990. Discharge or spills which produce a visible sheen on either surface water, or in waterways/sewers which lead to surface water, must be reported to the National Response Center at 800-424-8802.

SARA (311/312) REPORTABLE HAZARD CATEGORIES: Immediate Health. Delayed Health.

SARA (313) TOXIC RELEASE INVENTORY: This material contains no chemicals subject to the supplier notification requirements of the SARA 313 Toxic Release Program.

The following ingredients are cited on the lists below:

Chemical Name	CAS Number	List Citations
DISTILLATES (PETROLEUM), HYDROTREATED LIGHT	64742-47-8	17, 18

--REGULATORY LISTS SEARCHED--

- | | | | |
|---------------|------------------|-------------------|-------------|
| 1 = ACGIH ALL | 6 = TSCA 5a2 | 11 = CA P65 REPRO | 16 = MN RTK |
| 2 = ACGIH A1 | 7 = TSCA 5e | 12 = CA RTK | 17 = NJ RTK |
| 3 = ACGIH A2 | 8 = TSCA 6 | 13 = IL RTK | 18 = PA RTK |
| 4 = OSHA Z | 9 = TSCA 12b | 14 = LA RTK | 19 = RI RTK |
| 5 = TSCA 4 | 10 = CA P65 CARC | 15 = MI 293 | |

Code key: CARC=Carcinogen; REPRO=Reproductive

SECTION 16	OTHER INFORMATION
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This warning is given to comply with California Health and Safety Code 25249.6 and does not constitute an admission or a waiver of rights. This product contains a chemical known to the State of California to cause cancer, birth defects, or other reproductive harm.

N/D = Not determined, N/A = Not applicable

KEY TO THE H-CODES CONTAINED IN SECTION 3 OF THIS DOCUMENT (for information only):

H304: May be fatal if swallowed and enters airways; Aspiration, Cat 1



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Revision Date: 12 Dec 2014
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THIS SAFETY DATA SHEET CONTAINS THE FOLLOWING REVISIONS:

Updates made in accordance with implementation of GHS requirements.

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Internal Use Only

MHC: 1A, 0, 0, 0, 1, 0

DGN: 4409045HUS (1006349)

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