



U.S. DEPARTMENT OF
ENERGY



Final Report
E15PG00032
Oil Spill Response and Research
March 22, 2017

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Project Title: **On-Board Flotation System for the Removal of Surface Oil under Arctic Conditions**

Research Topic Area: Innovative Methods to Remove Surface Oil under Arctic Conditions

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This study was funded by the U.S. Department of the Interior, Bureau of Safety and Environmental Enforcement through Interagency Agreement E15PG00032 with the Department of Energy, National Energy Technology Laboratory.

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This final report has been reviewed by the BSEE and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the BSEE, nor does mention of the trade names or commercial products constitute endorsement or recommendation for use.

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Acronyms, Abbreviations, and Symbols

Acronym	Descriptive Name
AECOM	AECOM Corporation
ANS	Alaska North Slope crude
atm	Atmosphere
BSEE	Bureau of Safety and Environmental Enforcement
DI water	Deionized Water
DOE	Department of Energy
DOI	Department of the Interior
2-EH	2-Ethylhexanol
EPA	Environmental Protection Agency
FY	fiscal year
FS-STD	fluorescent response
GC-FID	Gas Chromatography – Flame Ionization Detector
GPC	Gel permeation chromatography
KF titration	Karl Fischer titration
LAMMPS	Large-scale Atomic/Molecular Massively Parallel Simulator
MIBC	Methyl isobutyl carbinol
NETL	National Energy Technology Laboratory
NS crude	North Star crude
ORISE	Oak Ridge Institute of Student Exchange
ppm	part per million
SDSs	Safety Data Sheets
TD-500D	TD-500D Oil Analyzer
TrAPPE	Transferable Potentials for Phase Equilibria

Administrative Summary

The Oil Spill Response and Research on Innovative Methods to Remove Surface Oil under Arctic Conditions project received funding from the Department of the Interior (DOI) /Bureau of Safety and Environmental Enforcement (BSEE).

The United States Department of Energy (DOE) National Energy Technology Laboratory (NETL) conducted research to develop On-Board Flotation System for the Removal of Surface Oil under Arctic Conditions, which was funded by the BSEE under award number E15PG00032. The project investigator was Fan Shi and the recipient institution was National Energy Technology Laboratory (NETL). In response to the finalization delays noted in the previous reporting cycle, a no-cost time extension was approved by DOI/BSEE extending the project cycle to March 31, 2017.

Executive Summary

Oil spill response is a top priority and a demanding task for the oil and gas industry in any environment. However, the ability to prevent and respond to oil spills in arctic regions presents particularly difficult challenges, which is instrumental for obtaining a license to operate. The NETL has performed research to utilize and apply unique flotation skills and equipment, which were previously used for coal preparation, to investigate oil separation under Arctic conditions. Commercial flotation equipment has been used to ensure that all specifications of devices meet industrial standards and are ready for commercialization. The study involved experiments to measure oil separation using a flotation process at simulated Arctic conditions, computational simulations of materials properties, and process optimization. NETL has successfully demonstrated the feasibility of using a flotation process for rapid separation of crude oil under Arctic Ocean conditions. Oil separation efficiency with different surfactants, temperature, and ice conditions has been investigated for both batch Denver cell and continuous column operations. Authentic crude oils and surrogate seawater have been used in all experiments. The largest advantage of using the flotation process is that it provides a high oil/water ratio product, thus significantly reducing the storage requirement for oil spill waste water. Oil separation in the presence of ice has been tested. The hydrophobicity difference between oil contaminated ice (hydrophobic) and cleaned ice (hydrophilic) enables easier separation of oily ice particles from water using the flotation method. Computer simulation has also been performed to optimize materials and equipment. The resulting dataset from the experiment and simulation has been collected and used to develop a prototype of an onsite oil clean-up flotation column system. Overall, the flotation process has successfully demonstrated excellent oil separation from seawater under lab scale simulated Arctic conditions.

1. Introduction

An oil spill is a form of pollution and typically refers to the unintentional release of liquid petroleum products, i.e., crude oil, into the environment. It can happen during oil drilling or transportation and poses a threat to the local environment. Cleanup and recovery of oil spills at sea, typically spread for hundreds of nautical miles in a thin oil slick, are generally much more difficult than those on land, especially in the Arctic, because extreme environmental conditions may prevent human response.

A large thin slick area can be quickly formed by the spread of spilled oil on seawater. Figure 1 shows a general schematic of an oil spill dispersion process on seawater. Different response strategies may be employed depending upon where the spill occurs, where the slick is moving, and the different types of equipment available. In all cases, the earlier the slick is contained and the oil concentrated, the easier the recovery operations will be.

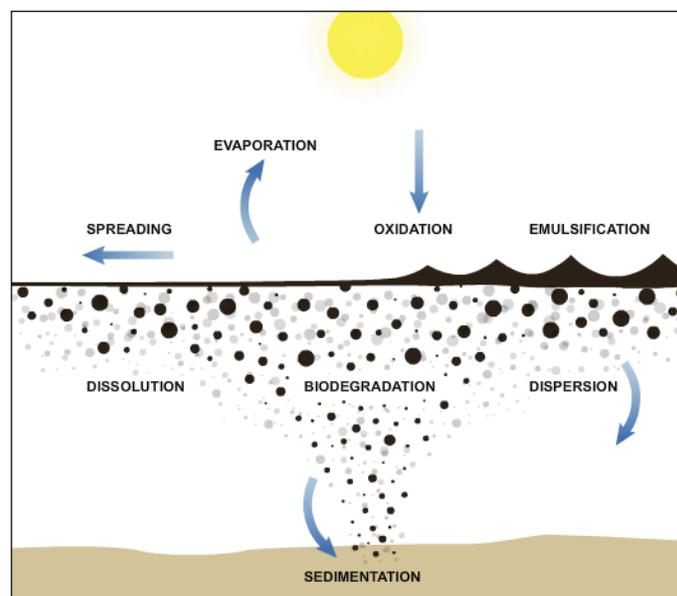


Figure 1. Schematic of oil spill dispersion process on sea (courtesy: Seapro.org).^[1]

A number of advanced technologies are available for controlling oil spills and minimizing impacts on the environment. Mechanical methods, including both containment and recovery, are considered the most environmentally friendly strategies for removing spilled oil from water surfaces. Normally, mechanical containment booms work with skimmers to respond to oil spills. Floating booms are used to temporarily contain spilled oil and help concentrate oil in thicker layers before being properly recovered and disposed of. Skimmers are typically made of rotating brush belts, drums, or ropes, which are towed by vessels through the slick. Several skimmers have been designed specifically for recovering oil in the presence of small ice pieces. These novel skimmers, equipped with advanced systems, including heating elements, ice deflection frames, and other devices, deal with viscous oil/water/ice mixtures.

Arctic operating conditions and spilled oil's physical properties must be considered when selecting the best suited in-situ mechanism for oil removal. Considering the operating environment in Arctic areas, it is important that an oil recovery system can be used in both open

waters and water with ice cover. Traditional oil recovery technologies are challenged by the Arctic operating environment, i.e., cold ice-covered waters. Some modified skimmers^[2] have been used for spilled oil removal in cold environments, including oleophilic skimmer systems for light to medium-viscosity oils; weir skimmers for both light and heavier oil products but with a less efficient and high water-to-oil ratios; vacuum skimmers with less efficient and high water-to-oil ratios; and other mechanical skimmers.

The main challenges presented to mechanical skimmers are: 1) water contents in recovered oil vary with the viscosity of spilled oil and the type of skimmers; 2) skimmer encounter rates are low in the presence of ice; 3) a calm surface is required at all times during skimming operation; and 4) the reliability of skimmers varies at very low temperatures. Another issue is the storage, transfer, and disposal of the recovered oil/ice/water mixture, which is a special challenge in remote Arctic areas with limited infrastructure. After oil, water, and ice mixture is recovered, there is a lack of infrastructure available in the Arctic to allow offload of the recovered product. Therefore, the ability to separate the water from the oil after recovery will allow for more efficient storage. So, the flotation system will be used for that separation process.

The objective of this study is to initiate the development of a prototype flotation system for the treatment of recovered spilled oil/water/ice mixture under Arctic conditions. With a proper design of flotation devices and optimized operating parameters, the prospect of using a froth flotation scheme for separating spilled crude oils from Arctic seawater is excellent.

2. Background

Flotation is an aqueous, efficient, inexpensive, and well-developed commercial technology which has been widely applied in various industrial processes, including mineral processing and wastewater treatments ^[3-7]. After spilled oil is collected by mechanical skimmers, on-board flotation treatments do not depend on the conditions of an oil slick at sea, even with harsh weather conditions and ocean movement. The flotation process has flexible operating ranges and can still be effective for oil separation under different harsh environments and in low temperatures.

The flotation process generates gas bubbles within a solid-liquid or liquid-liquid suspensions and then attaches and removes solid particles or immiscible liquid droplets from the liquid medium with the rising gas bubbles. As a separation process involving collisions between bubbles and particles (or droplets), the principle of flotation is based upon the particle's ability to attach onto an air bubble and then float to the liquid's surface. Hydrophobic fine particles are easily attachable to gas bubbles in a flotation process. Most of the oily wastes—including greases and oils such as light/heavy hydrocarbons, lubricants, fats and fatty oils—are naturally hydrophobic. Hydrophobic particles can be easily captured and carried upward by gas bubbles to the surface of a slurry. Figure 2 shows oil-laden foam from a flotation process.



Figure 2. Oil separation using a Jameson flotation column; courtesy of Jameson Cell. ^[8]

2.1. Principle of Flotation

Oily wastes consist of emulsified and/or non-emulsified (free floating) oils. Floating oil can be removed economically and efficiently by using gravity separation. However, the treatment of emulsified oil is more complex and costly. Although many water treatment technologies are commonly applied in the oil and gas industries, many lack the technical capability to efficiently treat a large quantity of oily seawater at a competitive price. Flotation is a process to separate particles (including oil droplets) from aqueous phase with the help of air bubbles. First applied in mineral processing, fine particle flotation separations began in the early twentieth century. As a

separation process involving collisions between bubbles and particles (or droplets), the principle of flotation is stated in the conception of floatability, which is discussed in the following section.

Floatability

For a given particle or droplet, floatability means its ability to attach onto an air bubble and to be floated to the surface of liquid. It can be measured indirectly in terms of the surface properties of particle, including hydrophobicity and zeta potential.

Particles are said to be hydrophobic when they are easily attachable to gas bubbles, such as oil. Otherwise, they are hydrophilic. So the floatability of a particle is determined by the hydrophobicity of particles. Hydrophobic particles can be easily captured and carried upward by gas bubbles to the surface of slurry, while hydrophilic particles cannot. Contact angle is the most commonly used parameter to indicate the hydrophobicity of particles. The measurement of contact angle in pure water is used to serve as a measurement of hydrophobicity of solid particles. Hydrophobic particles are associated with large contact angles, while hydrophilic particles are coupled with small contact angles. Usually, only particles with contact angles larger than 30 degrees can be separated efficiently by the flotation method ^[9]. Most crude oil and its fractions, including original crude oil, deasphalted oil (original crude oil without asphaltenes), and deacidified oil (original crude oil without naphthenic acids), have contact angles much greater than 30 degrees, i.e., a heavy Brazilian crude of contact angles in a range from 100 to 160 degrees on different solid surface ^[10], indicating their natural hydrophobic behaviors.

Mechanism of Capture of Droplets

Besides the floatability of the particles, there are other factors affecting flotation efficiency. For a given type of particles and a set of operation conditions, the chance of flotation can be expressed in terms of three probabilities, as shown in following equation ^[11]:

$$\text{Chance of flotation} = \text{probability of particle-bubble collision} \times \text{probability of attachment} \times \text{probability of retention of attachment}$$

The probability of collision between particles and bubbles is controlled by hydrodynamic conditions of flotation process. There are three ways bubbles and particles make contact: collision, entrapment and precipitation. Collision is induced by turbulent mixing or caused by direct contact between a rising bubble and a sedimenting particle. Entrapment and precipitation refer to bubbles are entrapped in, or particles precipitate on a growing floc structure, respectively. For oil flotation, collision and entrapment play significant roles.

The probability of attachment is affected by interfacial properties, such as interfacial tensions. The retention of attachment is affected by turbulent condition and adhesive force of attached particles on bubbles. In general, the probability of attachment and the retention of attachment are considered as one factor called *the efficiency of attachment*. Figure 3 shows the aggregation of fine solid particles and air bubbles in a flotation process ^[12].

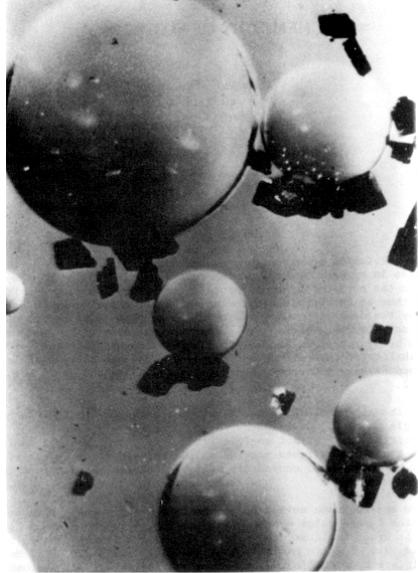


Figure 3. Attachment of fine particles on bubbles. ^[12]

Hydrodynamics in flotation

Hydrodynamic conditions in the flotation process can significantly affect flotation operating behaviors. Two major hydrodynamic parameters in this study are gas bubble size and gas holdup. Bubble generation is an essential step in flotation because rising bubbles provide the lift force for components (in the form of fine particles or droplets) to separate. The effectiveness of flotation mostly depends on the interfacial properties of components in the mixtures to be separated. These interfacial properties can be modified by adding different surface active reagents, called surfactants. In a flotation process, typical surfactants are frother, collector, and modifier. Adding frother can change surface tension and therefore to stabilize bubble formation. The function of modifier is to modify the physical properties of the solution to enhance the separation. Collectors are typically used in mineral process, which won't be discussed in the report. However, there are very few literature references that focus on the investigation of hydrodynamic parameters under arctic conditions.

Gas bubbles generated in a flotation column are relatively small and spherical with a typical bubble Sauter mean diameter as small as 1 mm ^[13], when frother concentration is 15 ppm, and superficial gas velocity is greater than 2.0 cm/s. It is a big advantage, comparing with an average bubble size of 2~4 mm in conventional flotation devices under similar operation conditions. Small bubbles not only give high gas holdup, they also provide more opportunities for collisions between particles and bubbles.

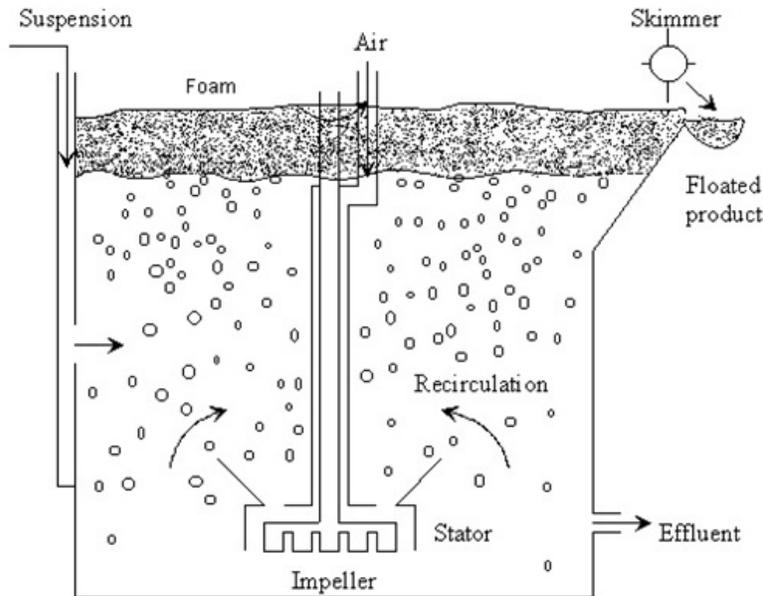
Gas holdup is the ratio of gas phase volume to the total volume of liquid/gas phase in a bubble device. The increase of gas holdup will increase the opportunities of contact and attachment between particles and gas bubbles. Gas holdups increase with the dosage of frother and superficial gas velocity. It is desirable in flotation column to have high gas holdup, because it provides large interfacial area, which favors bubble-particle to form aggregates. Flotation column has a high gas holdup up to 30% ^[14].

The introduction of surfactants to the interface of oil and seawater could modify their interfacial properties significantly. Researchers have tested a large number of surfactants to improve the

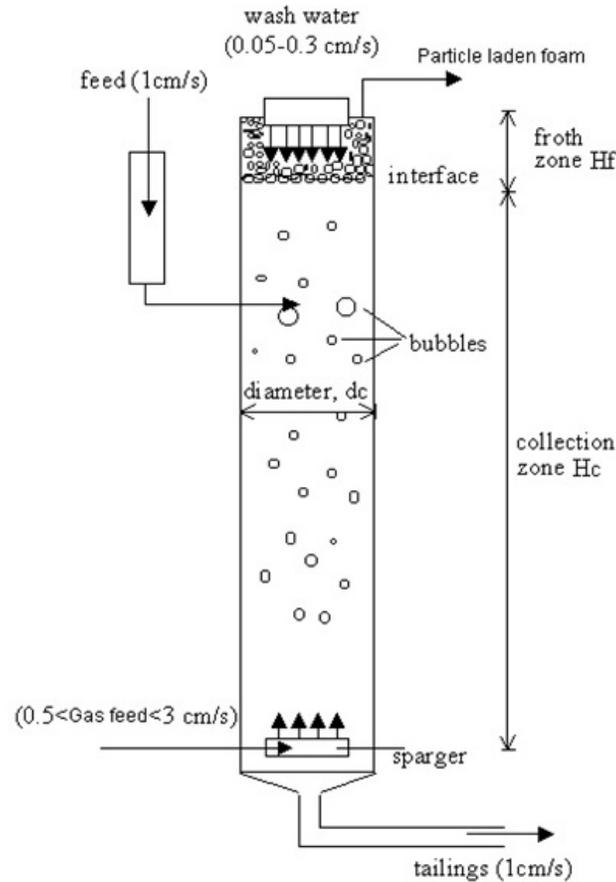
flotation separation efficiency ^[15]. Typical dosages of these chemicals are tens of ppm. Only a trace amount of these surfactants remains in treated water ^[16]. Concentration of frother can be measured by oil analyzer or gas chromatography (GC).

2.2. Flotation Equipment

Although many different types of flotation devices are available, the mechanic agitated flotation cell and the flotation column are the two most widely used configurations. A schematic of a laboratory mechanic agitated flotation cell, the Denver cell, is shown in Figure 4a^[9]. After the slurry is fed into the cell, an impeller is installed. Air is sucked into the cell through a hollow shaft of an agitator, and then the air stream is broken by the agitating impeller so small bubbles are emitted from the end of the impeller blades. Then, rising bubbles, together with attached particles/droplets, form a foam layer on top of the dispersion phase. The foam layer is skimmed off mechanically from the top. Non-floated components and clean water are withdrawn from the bottom of the cell. The rotating impeller of the agitator not only introduces air bubbles into the flotation cell, but also mechanically breaks them into small sizes. In addition, agitation induces turbulent mixing to promote particle bubble collisions. It can be used to duplicate all the operations in large-scale commercial flotation cells, such as the conditioning of the pulp with reagents, aeration of the pulp, and production of high-grade mineral froth, which can be removed at definite time intervals. The results obtained in testing can be compared to actual operating practice. In this study, a Denver cell will be used for proof of concept study, and screen surfactants will be used at simulated Arctic environments.



(a)



(b)

Figure 4. a) Schematic diagram of a conventional flotation cell, and b) a flotation column, respectively.

The flotation column has been recognized as a promising wastewater treatment technique because of its high separation efficiency, small foot print, low capital investment, and ease of operation. As shown in Figure 4b^[4], oil separation in a flotation column involves the selective attachment of oil droplets (hydrophobic) and oil contaminated ice particles (partially hydrophobic) in liquid suspension onto gas bubbles, which provide levitation for oil droplets. The resulting bubble-oil aggregates rise to the surface of the dispersion and form an oil-laden foam layer (froth zone), which is then skimmed off and separated from the dispersion phase. Therefore, oil products from a flotation column feature a high oil-to-water ratio. In this study, a 3" outside diameter (O.D.) flotation column is used to investigate oil removal in the presence of ice particles at cold temperatures to verify oil removal efficiency at simulated Arctic conditions.

Flotation is also a preferable technique for emulsified oily water treatment, typically of oil emulsion less than 1%. Flotation columns-in-series or flotation coupled with an API separator is also a feasible solution for the treatment of water with high content of emulsified oil. Therefore, flotation could be used as both a primary and a secondary stage treatment to reach high oil separation efficiency.

2.3. A Challenge in Oil Recovery under Arctic Conditions

Operating temperature is one of the most important variables affecting crude oil recovery. The environment and temperatures in the Arctic Ocean are very different from other open waters. The typical temperature of the surface of the Arctic Ocean is constant, near the freezing point of seawater, i.e., $-1.8\text{ }^{\circ}\text{C}$ ($28.8\text{ }^{\circ}\text{F}$). During the summer, temperatures may rise to around $10\text{ }^{\circ}\text{C}$.^[17] The varying Arctic environment and physical properties of oils present challenges to oil clean-up. Cold temperatures cause the properties of spilled oil, including viscosity, density and surface tension, to be significantly different than at ambient conditions. At low temperatures, oil viscosity is high and attachment to the air bubbles becomes difficult. As shown in Figure 5, when temperatures increase, oil viscosity is reduced and approaches the viscosity of water. Oil forms a thin film and envelopes air bubbles, providing far more stable attachments. At cold conditions, oil droplets become very viscous and can only colloid and attach to air bubbles. Oil recovery rates may, therefore, greatly decrease at low operating temperatures. Conditions for this series of experiments have been selected that are representative of Arctic conditions at low temperatures.

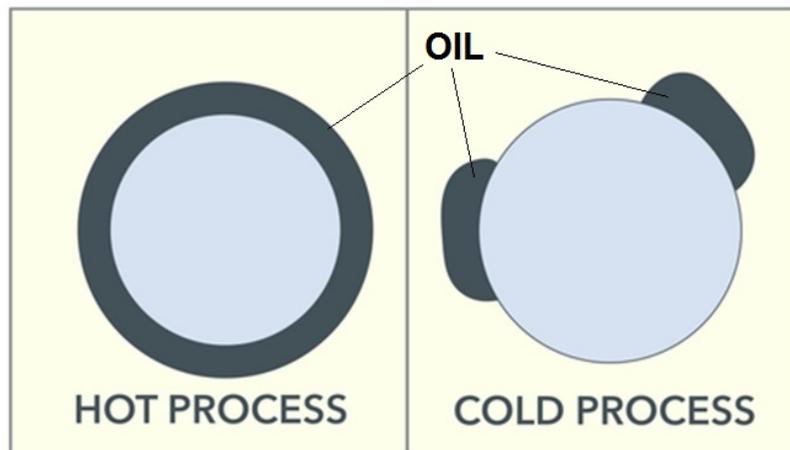


Figure 5. Oil air bubble interaction at different temperatures (courtesy: oilsandsmagazine.com).^[18]

In an Arctic environment, ice contamination caused by an oil spill is also a significant threat to the environment. The flotation method looks promising for the separation of oil contaminated ice from clean ice. Because clean ice particles are naturally hydrophilic, they will not stick to rising air bubbles in a flotation process. Due to the hydrophobic nature of oil droplets, like other hydrophobic particles, oil-contaminated ice particles can be easily picked up by bubbles and separated from clean ice. The uncontaminated ice particles will remain in water rather than in the froth zone, as shown in Figure 4b.

3. Objectives

Generally the objectives of this research were to theoretically and experimentally investigate the feasibility of using flotation technology for spilled oil clean up under arctic conditions. The ultimate goal of the proposed work was to provide an onboard flotation solution for effectively separating recovered oil in arctic conditions. In this study, a batch operated Denver cell was used for materials screening and optimizing operating conditions. After the proof of concept investigation was completed in a Denver cell, NETL used a compact froth flotation column system for the continuous treatment of an oily (ice) seawater mixture. A flotation unit developed for oily water treatment was used as a basis for oil removal operations in a simulated arctic environment. NETL selected several different types of oils, including an ANS, and other crudes provided by BSEE, to simulate spilled oils for the investigation. Oil contaminated ice was also treated using the same flotation system.

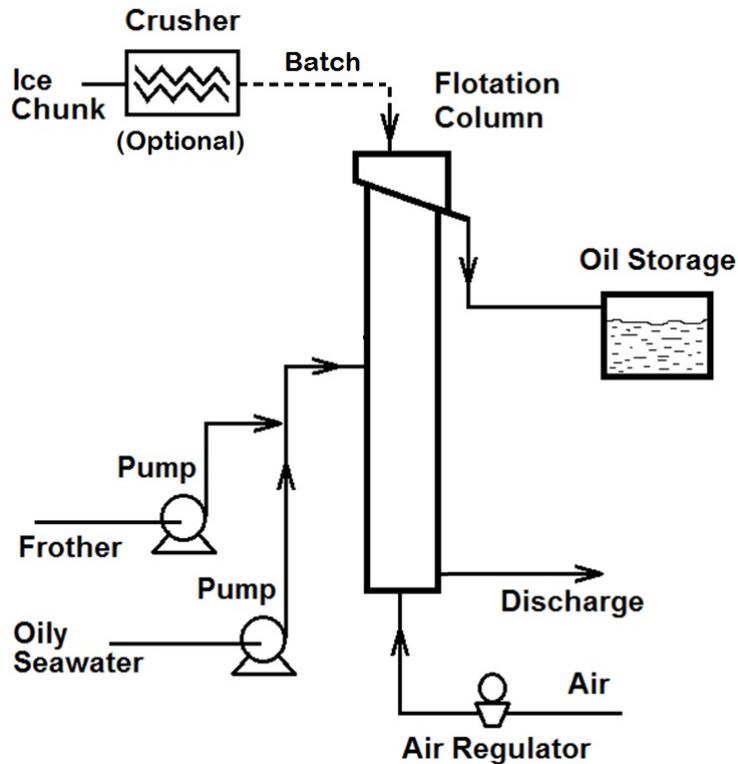


Figure 6. Flotation column system for oily ice water treatment.

The oil/ice clean up flotation column system contains an external ice crusher, a flotation unit, several pumps, a feed tank (not shown) and an oil storage tank, as shown in Figure 6. Simulated seawater was prepared following the standard practice (ASTM D1141-98) procedure. Oily seawater was directly fed into the side of the flotation column. Frother was carefully added to the stream using a syringe pump, as shown in Figure 6, or was pre-mixed with oily water in the feed tank. A single flotation column was the preferable treatment methodology for low oil/particle content water. Thus, the concentration of oil/ice was diluted within an operating range, as discussed in the following sections. Before being fed into the flotation unit, i.e., Denver cell or flotation column, large ice chunks were ground by an external ice crusher to reduce the size of the ice for a better flotation separation operation. For the column setup, ice particles were fed

into the column in a batch mode. Oil-laden foam was collected from the top froth layer and then transferred to an oil storage tank. Light weight oil foam layer in a flotation column was thicker than 4 inches. Waves of oil foam due to ship motion have negligible effect on the oil separation process. A column stabilization mechanism was also under investigation to enable continuous operation of flotation column under different ocean movement.

Computational simulation provided a theoretical understanding of the interactions between materials under different conditions and helped in the selection of proper materials for a better performance. The process simulation also helped to optimize the design of flotation system as well as operating conditions. Both simulations were critical to the study.

The proposed onboard at-sea flotation system is a completely new concept for the separation of spilled oil from seawater in arctic conditions. Due to oil's hydrophobic nature, this flotation system provided high oil separation rates and high oil/water ratio oil products. As an onboard system, this flotation system is also able to deal with oil spills under harsh conditions including low temperatures, sea ice, bad weather, strong ocean movement, etc.

4. Experimental

4.1. Materials Requisition

ASTM D1141-98 sea salt (Fisher Scientific), with composition shown in Table 1, was used for the preparation of simulated seawater. Other chemicals, such as pH buffers, frothers, and demulsifiers, were ordered from major chemical vendors or from manufacturers. Crude oils, including ANS and North Star, were provided by BSEE. Simulated seawater was prepared following the ASTM D11410-98 procedures^[19] and chilled to operating temperatures. Five frothers, including three glycol ethers based Custo527, FloMin F660 and FloMin F672, Methyl isobutyl carbinol (MIBC) and 2-Ethylhexanol (2-EH), were used in the flotation tests. Commercially available demulsifiers, including Dow Corning: DM-2 demulsifier, AkzoNobel: RTC-330 water clarifier, and Dow Chemical: ROMAX 6000 water clarifiers were tested. NETL has received four authentic crude oil samples, including North Star (NS), and ANS crude from the OHMSETT facility in New Jersey. Safety Data Sheets (SDSs) of selected chemicals are shown in the Appendix A.

Table 1. Composition of Sea Salt (ASTM D1141-98)

NaCl	58.490%
MgCl ₂ - 6H ₂ O	26.460%
Na ₂ SO ₄	9.750%
CaCl ₂	2.765%
KCl	1.645%
NaHCO ₃	0.477%
KBr	0.238%
H ₃ BO ₃	0.071%
SrCl ₂ - 6H ₂ O	0.095%
NaF	0.007%

*Density of Seawater equals 1.025 g/mL at 15 °C

4.2. Materials Preparation

Simulated seawater (ASTM D1141 - 98(2013))

A sea salt mix was used in the standard practice (ASTM D1141-98)^[19] to prepare substitute ocean water. It standardizes seawater conditions for many uses, including chemical research testing. To prepare one liter of artificial seawater, 41.953 grams of Sea Salt ASTM D1141-98 was dissolved in enough Deionized (D.I.) or distilled water to yield a total of one liter synthetic/artificial seawater solution. After mixing, pH to 8.2 using 0.1 N solution of sodium hydroxide or hydrochloric acid was adjusted. In the following discussions, “water” refers to “simulated seawater” (ASTM D1141-98), unless otherwise specified.

Emulsified oily water preparation

In an oil spill response, emulsified oil, or weathered oil, is floating on the surface of the water

and has weathered and taken up water typically greater than 50 wt%. Previous research [4, 20-22] reported the preparation of oil emulsion simulant for the wastewater treatment. Typically, daily operation of a 3" column flotation consumes more than 30 liters of oily water solution. To obtain a large volume of stable oily emulsion water solution (emulsified oil in water), a previously designed mixing procedure was employed [4]. A three-step pre-mixing procedure was followed to prepare 50 liters stable 500 ppm (by wt.) emulsified oil solution. Total weight of 25.0 gram of crude oil was needed. First, a half weight of oil was added to 20 liters of fresh seawater, pumped through two parallel static mixers, and recycled back into a feed tank until a stable solution was made. This step took about 60 minutes. Then, another half weight of crude oil was added, and 20 more liters of water was added, up to a total of 40 liters. This oily water was also pumped through static mixers and recycled back into a feed tank for another 60 minutes to obtain a stable oily water solution. Finally, the remaining 10 liters of seawater was added, and the oil/water mixture was diluted to 50 liters. The last mixing step took at least 90 minutes to achieve a stable 50 liters, 500 ppm (by wt.) emulsified oil solution. This prepared oily water solution showed no visible oil film agglomerates, and it remained stable for more than 12 hours.

4.3.Equipment Setup

TD-500D oil sample analyzer

The TD-500D (Turner Designs) is a dual-channel, handheld fluorometer designed for quick measurements of crude oil and refined hydrocarbons in water. The dual-channel design makes the TD-500D applicable to a wide range of hydrocarbon types and concentration ranges [23]. Channel A provides the extreme sensitivity required to monitor very low hydrocarbon concentrations and for detecting weakly fluorescent hydrocarbons such as gas condensates, diesel fuel and refined petroleum products. Channel B was designed for those applications where a wide concentration range was more important than high sensitivity. Channel B is ideal for the analysis of produced water samples where crude oil concentrations can vary from low levels to over one thousand parts-per-million (ppm). The TD-500D is normally used with a solvent-extraction method such as the Oil in Water Analysis Procedure described below.

The TD-500D responds to the fluorescent aromatic compounds in the target hydrocarbon. The instrument must be calibrated by measuring the intensity of fluorescent light that is generated by a known concentration of hydrocarbon. Once calibrated, the instrument converts the fluorescent light intensity from an unknown sample into units of concentration. Every hydrocarbon will have a detection limit and a linearity limit. The detection limit is the lowest concentration that the instrument can detect. The linearity limit varies with each type of hydrocarbon. The linear range is defined by the concentration span from the detection limit to the linearity limit. As concentration increases beyond the linearity limit, the slope of the line begins to reduce. At very high concentrations, the slope may become negative. For most crude oils the linearity limit is well beyond 1000ppm.



Figure 7. A TD-500D oil analyzer.

The TD-500D must operate in the linear range to display accurate results. The linear range can be optimized for any monitoring application by selecting the best combination of measurement channel (A or B) and cuvette size (mini-cell or 8 mm cuvette). First, select a channel and proper cuvette size and calibrate the TD-500D with a standard solution of known concentration then prepare a new standard that is half the concentration of the standard solution by mixing equal volumes of the original calibration standard and pure blank. Place the diluted standard in a sample cell, place the cell in the TD-500D and press <READ>. If the displayed result is greater than half the original standard concentration, the linear range has been exceeded. The linear range is dependent upon the sample cuvette size and the optical channel of the TD-500D; thus, using a smaller cuvette size and/or changing the optical channel can extend the linear range.

GC-SPE method for frother measurement

Due to the high salt content of the aqueous portion of the flotation samples, a GC method with solid phase extraction (SPE) using a 3 ml Discovery SPE column was developed to ensure the ability to quantify amount of the glycol-based frother in the water without interferences from the salt. An Agilent 6890 GC-FID equipped with a 60m Stabil wax-DA capillary GC-column was used for quantitation with a 200 °C injector temperature and 1 µl injection at 10:1 split ratio. The oven was programmed to start at 100 °C and ramp to 200 °C at a rate of 15 °C/min followed by a 3 min 250 °C post run. Thus a single analytical run took approximately 36 min of total run time.

An internal standard was identified for quantification of any frother that remained in the aqueous layer. This standard is a structurally similar compound with similar chemical properties to the frother, yet is chromatographically separable from the frother components. As such, triethyleneglycol monomethylether (TEGME) was spiked into analytical samples at 10 ppm concentrations prior to SPE workup. A retention time (RT) of 12.94 ± 0.002 min was found for the internal standard. The frother, F660 showed a RT of 17.62 ± 0.002 min at a 5 ppm concentration. The method detection limit (MDL) for F660 was found to be 0.08 ppm and the limit of quantitation was found to be approximately 1 ppm.



Figure 8. (a) Agilent 6890N gas chromatography, and (b) a SPE module, respectively.

The general procedure for a single sample work-up and analysis was as follows:

A 1000 ppm stock solution of TEGME was prepared in DI water. An unknown frother sample was spiked with 10 $\mu\text{l}/\text{ml}$ sample with stock TEGME to a final concentration of 10 ppm. The spiked sample was then extracted on the solid phase extraction column, filtered and analyzed by GC-FID.

Column procedure was as follows:

- Pre-wash column with 3 ml methanol then 3 ml of water
- Load sample (2-2.5 ml) and evacuate all liquid with slight N_2 pressure
- Elute with 2 ml methanol
- Filter with 0.2 μm Luerlock syringe filter

GPC method for Demulsifier

Dow's commercial ROMAX demulsifiers are a mixture of polyacrylic acids with molecular weights ranging from 500,000 to 750,000. Gel permeation chromatography (GPC), also known as size exclusion chromatography, (SEC), is well-known technique for determining the molecular weight distribution of polymers. It is, therefore, key to studying biodegradable materials by giving insight into the rate at which a material might degrade, and revealing the presence of degraded polymer chains in a sample. NETL initiated the development of GPC method for the measurement of trace amount of polyacrylic acids in seawater by using a Waters e2695 system, as shown in Figure 9.



Figure 9. Waters GPC e2695 system.

KF titration

Standard Test Method D4928—12 “Water in Crude Oils by Coulometric Karl Fischer Titration”—was followed for water content measurement of water samples from flotation experiments. This test method covered the determination of water in the range of 0.02 to 5.00 mass or volume % in crude oils. This test method is intended for use with standard commercially available coulometric Karl Fischer reagent.



Figure 10. A 860 Thermoprep KF titration unit (Metrohm).

After homogenizing the crude oil sample, a test specimen of that sample was injected into the titration cell of a Karl Fischer apparatus, a 860 Thermoprep unit (Metrohm) as shown in Figure 10, in which iodine for the Karl Fischer reaction is generated coulometrically at the anode. When all the water was titrated, excess iodine was detected by an electrometric endpoint detector and the titration was terminated. Based on the stoichiometry of the reaction, one mole of iodine reacts with one mole of water, so the quantity of water could not be determined.

Bubble size measurement

As noted, bubble sizes directly affect hydrodynamics and, therefore, the efficiency of the flotation process. Bubble size was measured using a “digital photography method.” A high resolution digital camera/camcorder with a wide conversion lens was used to take digital images of moving bubbles. To “freeze” moving bubbles, the shutter speed was set at 1/2,000 second and exposure was set at F4.8. Two 120 V, 300 W Radius tungsten halogen lamps provided a background light source. Individual digital video images of gas bubbles were analyzed using Adobe Photographic Software^[24] and Public Image Analysis Software ImageJ 1.20s^[25], developed by the National Institutes of Health. Bubbles were assumed to be spherical because bubble sizes are very small (less than 2 mm) in the presence of a frother. More than 500 individual bubbles were counted for each measurement to achieve statistical reliability and to provide a representation of bubble size distribution under investigation.

Ice Crusher selection

Drift ice is common in the Arctic Ocean. *Young ice* is not flexible and tends to break under wave action and ranges in thickness from 10 centimeters (3.9 in) to 30 centimeters (12 in). Current technology has difficulty dealing with oil spills when drift ice is present. Oil-contaminated ice presents an environmental threat and can be a partially hydrophobic solid. To treat contaminated ice with a flotation process, the ice size must be reduced. Some crushers are mobile and can crush rocks as large as 60 inches. Typically, crushing stages are followed by milling stages if the materials require further reduction.

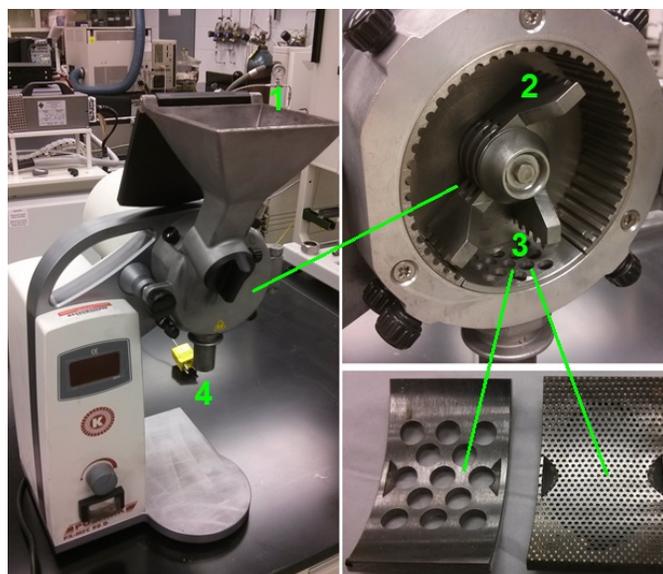


Figure 11. Laboratory hammer/cutting mill for solid sample grinding.

A hammer/cutting mill, PX-MFC 90 D, as shown in Figure 11, is suitable for solid sample grinding in the laboratory. This equipment has been successfully used for grinding solids, such as coal and biomass. The blade grinding attachment (consisting of a rotor with 3 hardened blades, 2 in Figure 11, and stator to accommodate an exchangeable screen, 3 in Figure 11) is used for processing solid materials. The resulting materials are collected from discharge port 4. Sieves can be chosen from 0.2 to 6 mm mesh size for desired particle sizes. Other tools, such as an ice shaver, can also be used to reduce the size of the ice. Different size ice particles will be used to study size effects on oil separation efficiency.

4.4. Separation Systems Setup

Benchtop Denver Cell

A bench-top flotation device, a Denver cell, was used for proof-of-concept and screening surfactants, and to investigate the effects of bubble size, ice content, and other operating conditions, as shown in Figure 12a. This benchtop flotation machine incorporated the same basic principle of operation as the commercial size flotation machine. It is considered as a single stage batch mode flotation cell. The size of each batch ranged from 250 mL to 2,000 mL. A compressed air supply at 40 psig was recommended. Operating temperature was controlled by a recirculating chiller. Details can be found in Table 2.

Flotation column

A commercial pilot scale 3" flotation column laboratory system was purchased from ErieZ, as shown in Figure 12b. To minimize the heat exchange between the flotation column and the environment, a heavy-duty insulation blanket was installed. The 3" flotation column was an ideal tool for evaluating the flotation characteristics of oily cold seawater with or without ice. The laboratory cell included air and water flowmeters to control the bubble generator and froth wash water flow and a variable speed feed slurry mixer to assure proper feed conditioning. Constructed of acrylic and stainless steel, this flotation column was built to handle a wide range of feed materials. ErieZ's 52-gallon propeller agitator tank provided a thorough mixing of slurry,

and was also large enough to meet typical daily flotation column operation requirement. Test sample results and operating parameters were directly scaled up to determine full-sized column flotation cell performance and requirements. Detailed parameters can be found in Table 2. This 3” flotation column system was installed and ISO audited by the NETL/DOE team for quality control purpose.

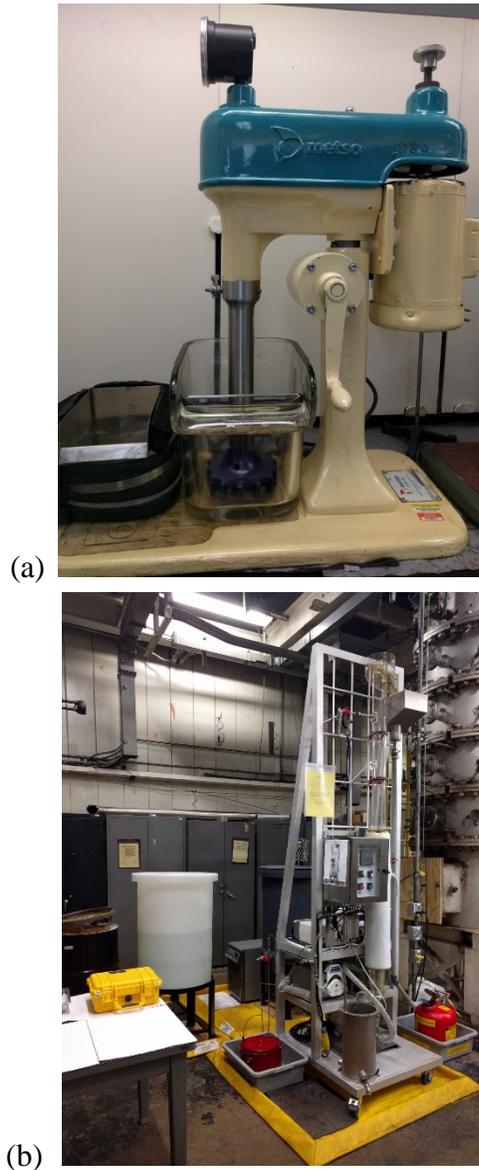


Figure 12. (a) A Metso Denver cell, and (b) an ErieZ 3” flotation column system, respectively.

Table 2. Capabilities of Flotation Facility

Benchtop Denver Cell	
Pressure	Ambient pressure to 40 psig
Temperature	Down to freezing (-2 °C)
Fluids	Simulated seawater (ASTM D1141-98), ice slurry, surfactants, oils, air
Batch size	1-250, 1-500, 1-1,000, 1-2,000 gram tanks, respectively
Mixing systems	Tachometer, ½ hp motor
Sample collection	Scrubber kit
Sample analysis	Oil analyzer, TD-500D
Flotation Cell	
Pressure	Hydrostatic pressure, ~10 psig
Temperature	Down to freezing (-2 °C)
Fluids	Simulated seawater (ASTM D1141-98), ice slurry, surfactants, oils, air
Column size	3” O.D. x 180-inch height
Mixing systems	circulated bubble generating systems, compressed air
Feed system	Peristaltic feed pump, 200 to 1,500 mL/minute
Sample collection	Peristaltic tailings pump
Sample analysis	Oil analyzer, TD-500D

5. Technical Results and Discussions

Flotation experiments were performed in a batch D-12 Denver cell as a proof-of-concept study, and in a continuous 3” flotation column as a scaled up investigation. The outcomes of the Denver cell study provided a basis for a large-scale continuous flotation column operation. Experimental results also served to inform computational simulations for operating optimization, both in material selection as well as the optimization of the flotation process. Experiment results are listed in Appendix B.

5.1. Denver cell test: Proof of Concept

In Denver cell testing, analytical methods were developed. Different materials and operating conditions were investigated and screened, including frothers, oil types, and ice conditions. Details are discussed in the following sections.

5.1.1. Effect of Frothers and usage

NETL developed methods to measure residual frother in water by TD-500D, GC-FID, and GC-SPE, respectively.

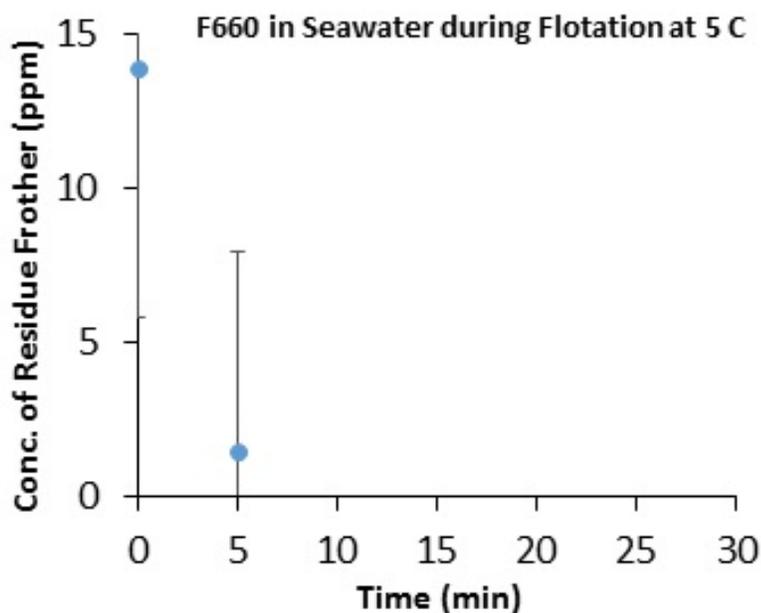
Residual frother concentration measurement using TD-500D

TD-500D detects fluorescent hydrocarbons by measuring fluorescent response (FS-STD). Generally, for hydrocarbons with FS-STD larger than 2% (i.e. 2-100 %), the instrument provides very reliable results of concentration. The values of FS-STD of four frothers are listed in Table 3. Because all four frothers were not strong fluorescent hydrocarbons (FS-STD < 2 %), TD-500D was only used to qualitatively measure F660 and F672, which were used in following flotation experiment.

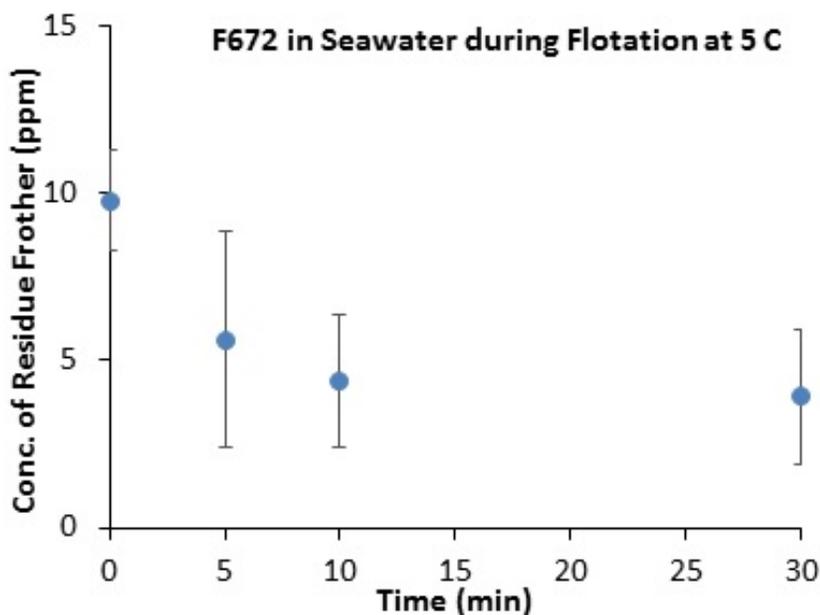
Table 3. FS-STD Values of Four Frothers

Frother	FS-STD (%)
MIBC	0
2-EH	0
F660	0.8
F672	1.5

Concentration of residual frother, i.e., F660 and F672, in seawater was measured during blank tests without crude oil added, as shown in Figures 13a and 13b. Water samples were collected during flotation operation. As indicated in Figures 13 a and b, concentrations decreased for both tests along with the operating time. Because F660 was of a weak FS-STD, it was not detected by TD-500D after 5 minutes of flotation operation. At the steady state, residual concentration was less than 5 ppm for both F660 and F672. Results indicated that the majority of frother didn't remain in water after flotation operations.



(a)



(b)

Figure 13. (a) Residual F660, and (b) residual F672 in seawater during flotation at 5 °C, respectively.

Residual frother concentration measurement using GC-FID

GC-FID provided a good resolution for measuring a trace amount of frother in DI water. An Agilent 6890 GC-FID equipped with a 60m Stabil wax-DA capillary GC-column was employed for quantitation with a 200 °C injector temperature and 1 µl injection at 10:1 split ratio. For the F660 frother, NETL was able to measure from 13 ppm to 120 ppm, as shown in Figure 14a and

14b. However, salt compounds in seawater had strong impact on the FID, which failed quickly after seawater sample injection.

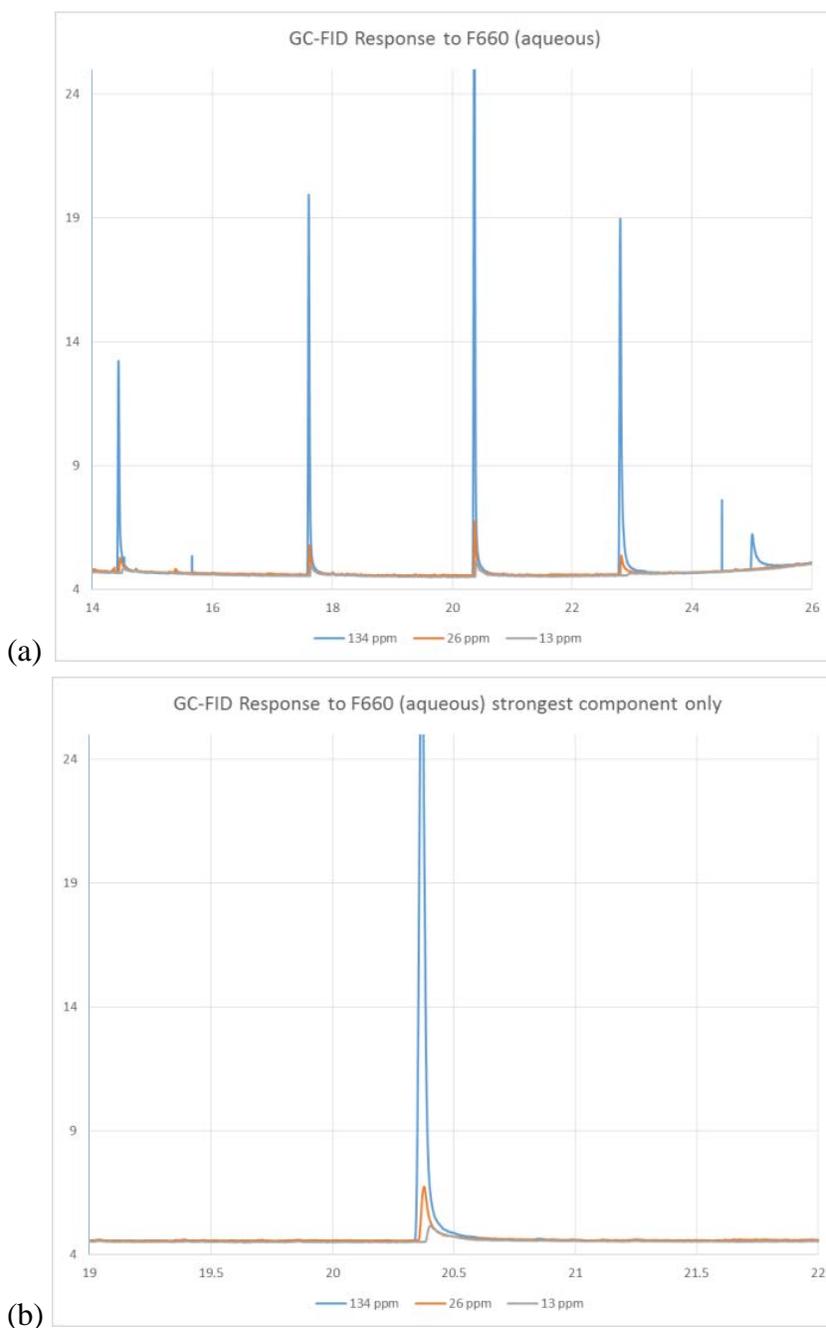


Figure 14. (a) A full spectrum, and (b) representative peaks of F660 frother at different concentrations in water, respectively.

Residual frother concentration measurement using GC equipped with SPE

A GC method with solid phase extraction (SPE) was developed for the measurement of frother in seawater in the presence of trace amount of crude oil. The same Agilent 6890 GC-FID equipped with a 60m Stabil wax-DA capillary GC-column was used. The oven was programmed to start at

100 °C and ramp to 200 °C at a rate of 15 °C/min followed by a 3 min 250 °C post run. Thus a single analytical run took approximately 36 min of total run time.

SPE pretreatment was used to remove harmful salt compounds from seawater samples while leaving target chemicals in the water for quantitative analysis. Triethyleneglycol monomethylether (TEGME), an internal standard, was identified for quantification of any frother that remained in the aqueous layer. TEGME was spiked into analytical samples at 10 ppm concentrations prior to SPE workup. A 1000 ppm stock solution of TEGME was prepared in DI water. An unknown frother sample was spiked with 10 µl/ml sample with stock TEGME to a final concentration of 10 ppm. The spiked sample was then extracted on the solid phase extraction column, filtered and analyzed by GC-FID. Results are shown in Table 4.

Table 4. Recovery of TEGME, an Internal Standard, in Each Experiment

Quality Control				
	sample 1	sample 2	sample 3	sample 4
TEGME expected (ppm)	20	20	20	20
TEGME found (ppm)	15.8	18.5	23.5	20.6
recovery TEGME (%)	79.2	92.5	117.4	103.2
TEGME RSD (%)	16.5			

Results of frother concentrations before (fresh) and during (2 min) the flotation experiment are shown in Table 5. Similar to previous results, frother concentrations decreased along the flotation time.

Table 5. Frother Concentrations Before and During the Flotation Experiment

#	Sample ID	F660 (ppm)	Custo527 (ppm)
1	Fresh sample, ANS, 5C	15.2	
2	Treated sample, 2 min, ANS, 5C	12.9	
3	Fresh sample, ANS, 5C		22.6
4	Treated sample, 2 min, ANS, 5C		16.9

Gas holdup measurement for frothers

Gas holdup is one of the most important parameters characterizing the hydrodynamics of a flotation process. It can be defined as the percentage by volume of gas in a total volume of bubble slurry in a flotation device. In Denver cell test, gas holdup can be measured by following equation:

$$Gas\ Holdup(GH) = \frac{(H_{air\ on} - H_{air\ off})}{H_{air\ on}} \times 100\%$$

Where, H is the height of liquid level under different operating conditions, i.e. air on or air off.

Gas holdup measurement for different frother conditions in DI water and simulated seawater was investigated in Denver cell, as shown in Figures 15 (a-d), and 16 (a-b), respectively. As shown in Fig 15, a small amount of frother, i.e., 30 ppm of F672, significantly reduced air bubble sizes, and therefore increased overall gas holdup.

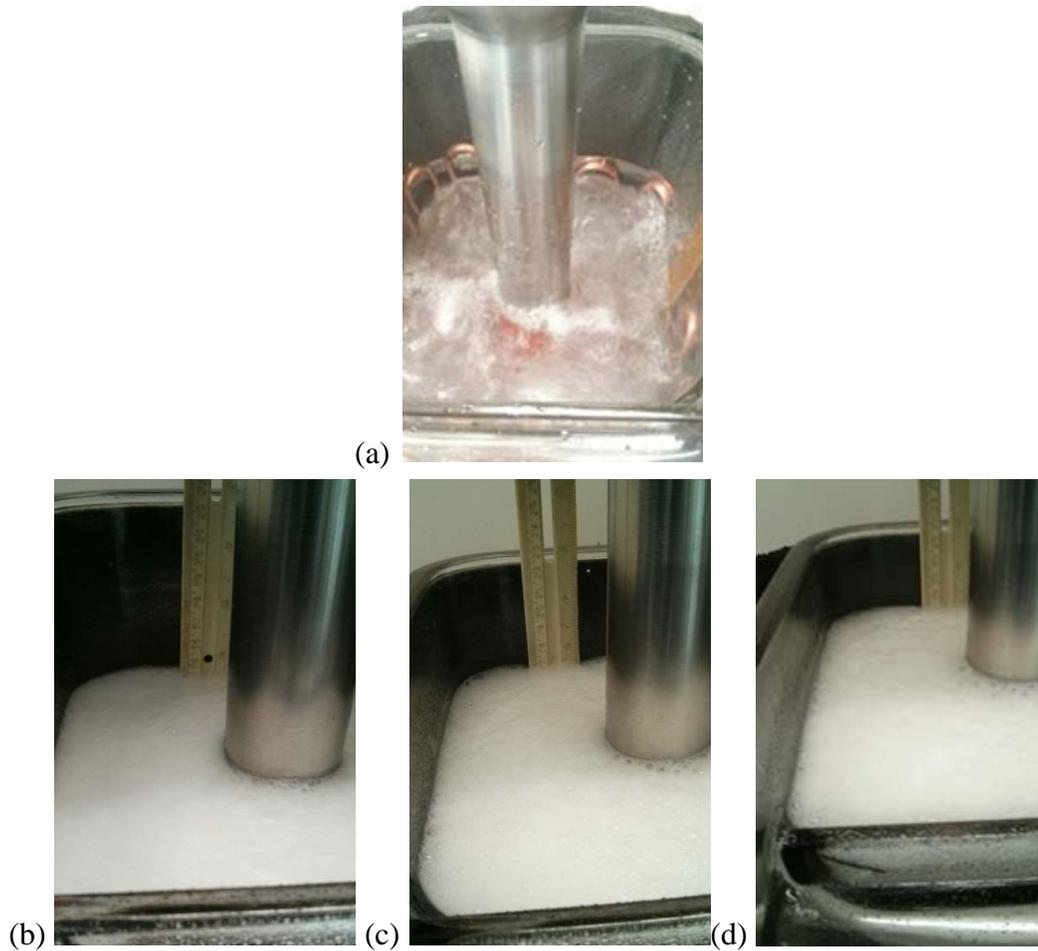
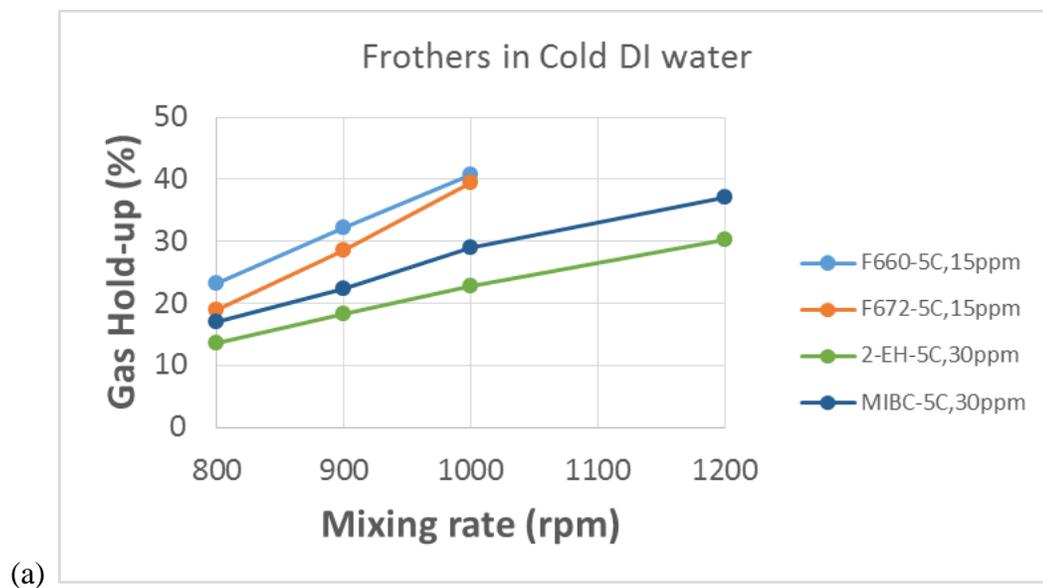


Figure 15. Gas holdup measurement in Denver cell: a) No frother, 1200 rpm, GH < 10 %; and 30 ppm of F672, b) 800 rpm, GH=14.5 %; c) 900 rpm, GH=25.9 %; and d) 1000 rpm, GH = 33 %, respectively.



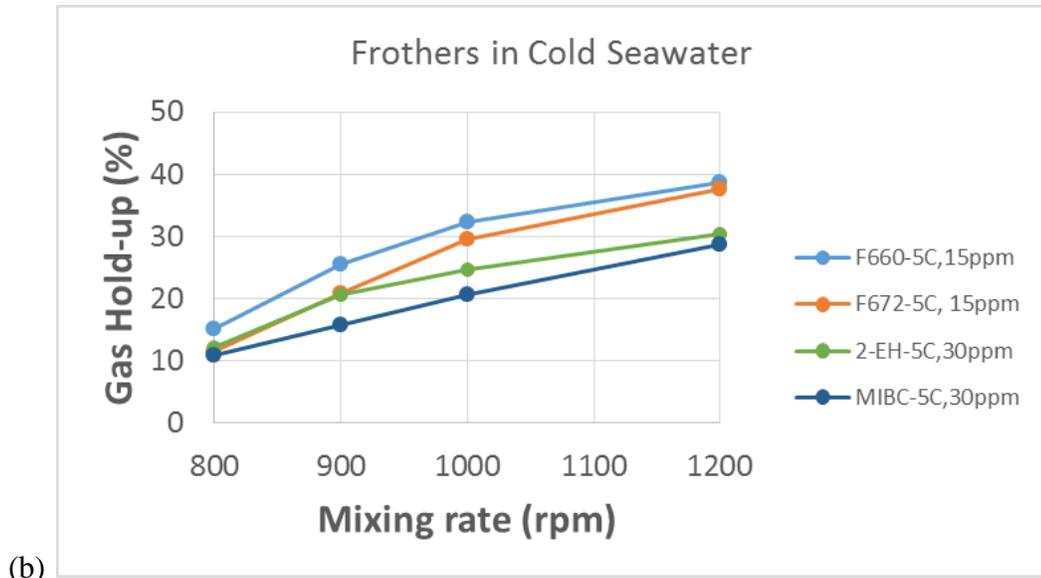


Figure 16. Performance of frothers in (a) DI water, and (b) seawater at 5°C, respectively.

As shown in Figure 16a, all four frothers generated more than 30% of gas holdup in cold DI water at 5°C. Two commercial frothers, i.e., F660 and F672, showed much higher gas holdups than those of 2-EH and MIBC, even at a half of dosage (15 ppm) and a low mixing rate. Among all four frothers, F660 showed the highest gas holdup with the lowest amount of dosage. Figure 16b showed that the gas holdup of F660, F672 and MIBC in cold seawater at 5°C was slightly reduced compared to those resulting from DI water, although still greater than 30%. Of particular note, the gas holdup of 2-EH was not affected by the salinity of water. Among all four frothers, F660 still showed the highest gas holdup in seawater at very low frother dosages.

Gas holdup measurement for frothers at freezing point (-2 °C)

Gas holdup measurements for different frother conditions in seawater at the freezing point were investigated in Denver cell as shown in Figure 17. As shown, gas holdups of all four frothers in freezing seawater at -2°C were similar to those results from 5°C. Overall, among all four frothers, F660 showed the highest gas holdup in seawater at very low frother dosages. F660 also showed a low residual concentration during flotation operation. Therefore, F660 has been selected for further flotation investigations, unless otherwise mentioned.

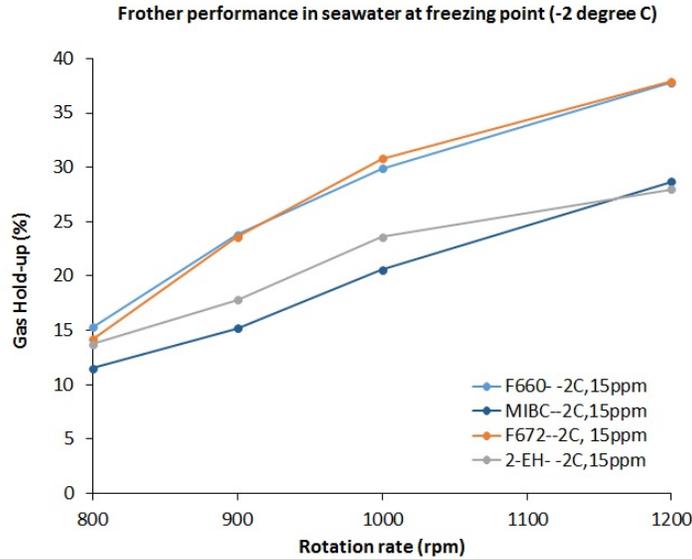


Figure 17. Performance of frothers in freezing seawater at -2°C.

5.1.2. Flotation of crude oil in seawater

In a typical test, seawater with 15 ppm of frother F660 was conditioned for 10 minutes at 5°C under mixing (Reynolds number >10000) in a 1-liter Denver cell. A stable froth layer with 30% gas holdup was formed at the top section of the cell. About 100 ml of crude oil was carefully poured into the surface, and the froth immediately turned into oil-laden froth, as shown in Figure 18a. Because they are naturally hydrophobic, crude oil droplets can easily attach to rising bubbles and float to the surface and be skimmed off from flotation devices. Oil laden froth either overflowed into a sample pan or was skimmed from froth surface. After most of the oil froth was skimmed off, the mixer was turned off and the water sample at the bottom of cell was withdrawn for analysis. The whole experiment was completed within a few minutes. Pictures of a typical oil sample and a water sample for different Denver cell tests are shown in Figures 18 b&c.



(a)

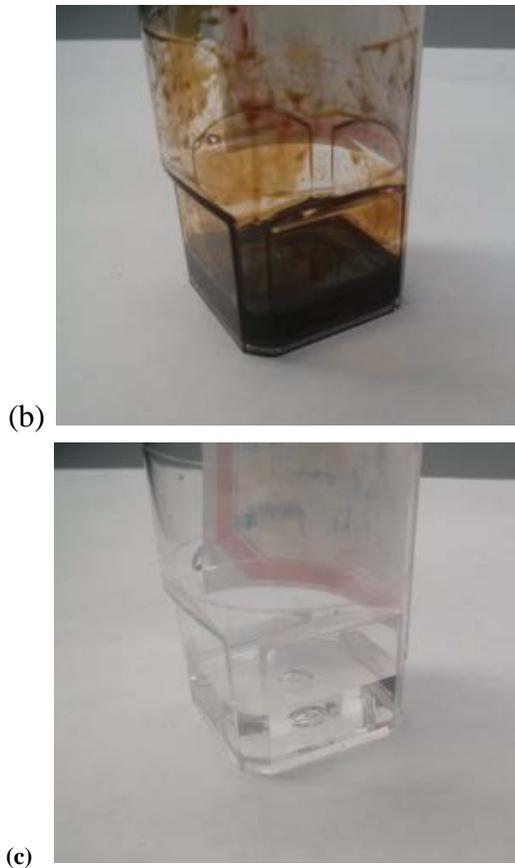


Figure 18a: (a) Oil flotation operating in a Denver cell; typical (b) crude sample, and (c) water sample collected from a Denver cell flotation operation, respectively.

Arctic ANS oil tests

NETL performed flotation tests for arctic crude oils, i.e., both ANS and NS crude oils, using Denver cell at -2°C and 5°C , respectively. Results were summarized in Table 6. Residual oil and water were analyzed by TD-500D and by Karl Fischer titration, respectively. For baseline tests without frother, residual oil in water varied significantly, probably due to high turbulence in cell.

For NS tests, oil content was as low as 13.3 ppm in the water sample which already met the EPA's 15 ppm disposal standards ^[26]. However, water in oil products was more than 10 %. This high-water content in oil was primarily because NS's viscosity was close to that of water, which resulted a thin oil film. At a high turbulence situation, it was difficult to collect oil product from froth layer with a regular skimming blade. It should also be emphasized that these results were collected via a single batch operation.

For ANS tests, a second frother, Custo527, was used to compare to F660. Results were summarized in Table 6. Typical contact time between oil and water was less than 5 minutes, depending on oil collecting speed. As expected, residual oil in all of water samples was found less than 15 ppm. Also as expected, water content in most oil samples was less than 15%. It was found that at a low frother dosage, i.e. 5 ppm, most values of water in oil were no more than those with a high frother concentration. The difference was primarily because of the entrapment of tiny bubbles generated at a relatively high frother concentration.

Overall, oil contents in treated seawater samples from the most of arctic crude oil flotation tests satisfied EPA’s 15 ppm disposal standards.

Table 6. Experiment Results of Arctic Crude Oils at Different Operating Conditions

Crude	Temperature (°C)	Frother	Frother (ppm)	Residual oil in water (ppm)	Residual water in oil (%)
NS	-2	n/a	-	7.3-69	-
NS	5	n/a	-	9.1-77	-
NS	-2	F660	15	<12	20.9
NS	5	F660	15	<14	11.0
ANS	-2	n/a	-	8.7	9.7
ANS	5	n/a	-	30.2	8.0
ANS	5	F660	15	1.0	7.7
ANS	-2	F660	15	2.9	9.5
ANS	5	F660	5	<1	8.2
ANS	-2	F660	5	<2	6.8
ANS	5	Custo527	5	5.9	4.5
ANS	-2	Custo527	5	2.2	7.5
ANS	5	Custo527	15	<2	12.5
ANS	-2	Custo527	15	<2	15.0

Gulf (Non-Arctic) free oil test and baseline test

Flotation experiments of two non- arctic crude oils, i.e., Ewing Bank and Harmony crude oils, were investigated using Denver cell at 30°C. Three frother conditions were applied, including a baseline (no frother), 15 ppm of F660, and Custo527, respectively. Results of experiments were summarized in Table 7. Similar to the results of arctic crude oils tests, less than 15 wt% of water in oil products was found, while only trace amount of residual crude in the treated seawater (< 2 ppm). All residual concentrations of non-arctic crude oil after flotation operations met the EPA’s 15 ppm disposal standards. Therefore, flotation operation has been proven to be capable of removing different crude oils successfully from seawater, even at warm temperatures.

Table 7. Experiment Results of Non-Arctic Crude Oils at Different Operating Conditions

Crude	Temperature (°C)	Frother	Frother (ppm)	Residual oil in water (ppm)	Residual water in oil (%)
Ewing Bank	30	-	0	<1	2.4
Ewing Bank	30	F660	15	<2	13.6
Ewing Bank	30	Custo527	15	<2	14.6
Harmony	30	-	0	<1	4.3
Harmony	30	F660	15	<1	9.0
Harmony	30	Custo527	15	<1	15.7

Emulsified arctic oil test

To investigate the flotation process for the emulsified arctic oil, ANS emulsion solutions were prepared. The preparation of the ANS emulsion was briefly described as follows: 1) add 2 g of ANS oil into 2 liters of freezing seawater under stirring for 2.5 hrs; 2) add another 2 g of ANS oil

while keeping stirring for another 2.5 hrs. At the end of 5 hours, a solution of 300-400 ppm of ANS crude emulsion will be obtained, which was stable for up to 3 hours.

Results of flotation of emulsified ANS crude were shown in Figure 19. The concentrations of emulsified ANS dropped dramatically in the first 10 minutes of flotation, even without frother. After 20 minutes of flotation, less than 20 ppm of oil was found in treated water in the presence of frothers. The oil concentration decreased gradually below 15 ppm after 60 min of flotation. When no frother was used, less than 5 ppm of oil was found after 60 min flotation. This could possibly be attributed to the difference of bubble size generated with and without frothers. In a Denver cell, due to high turbulence, oil attached small bubbles generated by the frother can be easily back-mixed into bulk water and hence the final oil concentration was higher than that of flotation of “big” bubbles without frother. Overall, flotation operation successfully removed emulsified ANS crude oil from seawater under arctic conditions.

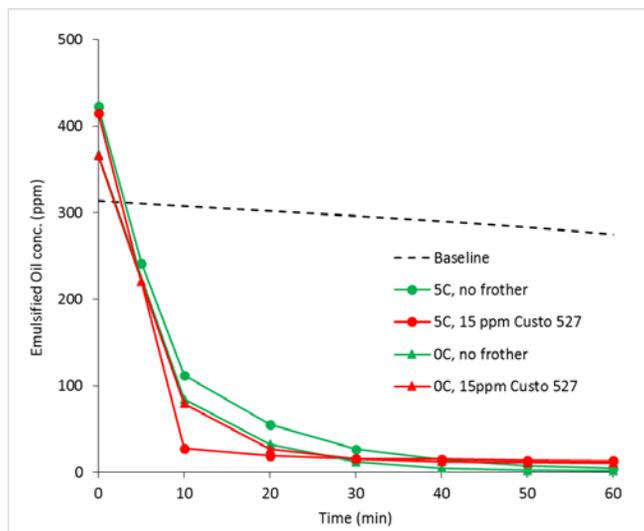


Figure 19. Flotation experiment results of emulsified ANS crude oil.

5.1.3. Oil Contaminated Ice Tests

To find out separation behaviors of arctic oil contaminated ice in a Denver cell, NETL performed separation tests under baseline and frother operating conditions. The average sizes of ice particles were ~5 mm and ~15 mm, respectively. The oil concentration was 10 wt% of ice, while the ice concentration was 10 wt% of water. Oil and ice mixtures were stored in a freezer for overnight to simulate arctic temperature.

Pictures of before and after oil contaminated ice (size ~ 15 mm) flotation test in a Denver cell were shown in Figures 20 *a&b*, respectively. Results of oil in water and oil recovery rates are summarized in Table 8 demonstrating that the flotation process can successfully separate oil contaminated ice particles from clean ice particles. For small ice particles, the oil recovery rate was up to 95% and residual oil concentration was as low as less than 2 ppm. For large ice particles, oil recovery rates were still greater than 90% and residual oil concentration was less than 5 ppm. Therefore, flotation process successfully separated oil and oily ice from clean ice.

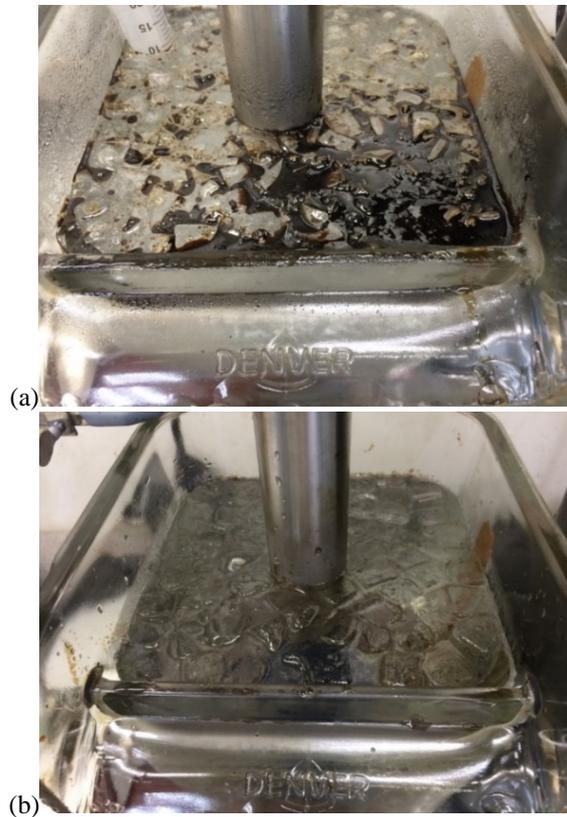


Figure 20. Oil contaminated ice (a) before and (b) after flotation in Denver cell. Ice size is 15 mm.

Table 8. Results of oil contaminated ice flotation test in a Denver Cell

Oil conc. (wt%)	Ice conc. (wt%)	Frother	Frother Conc. (ppm)	Ice size (mm)	Oil recovery (wt%)	Oil in water conc. (ppm)
10	10	n/a	0	3-5	>95	0.8
		Custo527	15		>95	1.5
		n/a	0	5-15	>90	2.4
		Custo527	15		>90	1.1

Overall, the concept of using flotation operations to separate ANS crude oil from seawater near arctic conditions was successfully proved in a Denver cell. The flotation can also separate emulsified oil as well as oil contaminated ice at simulated arctic temperatures. All treated water samples have met the EPA's regulation (<15 ppm) for discharged water from vehicles at sea.

5.2. Flotation Column Test

Results of Denver cell experiments clearly demonstrated the feasibility of using flotation process for spilled crude clean up under arctic as well as gulf conditions. A flotation column was used to further verify the effectiveness of continuous flotation operation for crude oil cleanup under arctic conditions.

5.2.1. Shakedown of flotation column

The complete setup of a flotation column system included: 1) column installation; 2) feed systems installation; 3) sample collection; 4) control panel connection; 5) waste collections; and 6) safety devices installations.

- 1) 3" flotation column, as shown in Figure 21, was commercial available lab scale equipment manufactured by ErieZ company, PA. The original design of column was for the processing of mineral products, i.e., coal products. This unit was to verify the efficiency of flotation column for spilled oil separation from seawater under arctic temperatures.
- 2) Feed systems included air supply, seawater feed, and oil feed. Air was supplied using house air with up to 70 psig of pressure. Seawater feed system included a foam insulated 55-gal feed tank with a 1.5 hp mixer, and a Polystat 1/4 HP Recirculating Chiller (Cole Parmer) to provide cooling to the tank. Oil tank was modified from a 3-gal Justrite safety can. Masterflex peristaltic pumps were used to pump seawater and crude oil.
- 3) The water sample port was at the bottom of flotation column. The flow rate of tailing water was controlled by PID via a Masterflex peristaltic pump. The oil sample overflowed from the top of column and flowed into an oil collection container (a 5-gal Justrite safety can) through a collection funnel and pipe.
- 4) Functions of the control panel included the control of liquid level and the adjustment of bubbles via sparger.
- 5) Waste oil and oily pads were stored in a 55-gal waste oil drum and a waste drum.
- 6) Other safety devices for the project included eye washes, ground connection, oil boom, and oil spill kit, such as oil adsorption pad and oil socks.



Figure 21. A 3" flotation column system for oil separation study.

5.2.2. Hydrodynamic Study

Different from experiments in a Denver cell, in this study more operating parameters were introduced to the continuous flotation column process. To understand the hydrodynamics in a flotation column, liquid properties and operating parameters, including bubble size, gas flow rate, gas holdup, liquid feed rate and oil collection rate, were investigated for process modeling.

As indicated, bubble sizes directly affect hydrodynamics and, therefore, the efficiency of flotation process. Bubble size was measured using a “digital photography method.” A Pentax K series Digital-SLR Camera with a wide conversion lens was used to take digital images of moving bubbles. To “freeze” moving bubbles, shutter speed was set at 1/2000 second and exposure was set at F4.8. Individual digital images of gas bubbles, as shown in Figure 22, were analyzed using Adobe Photographic Software and Image Analysis Software ImageJ 1.20, developed by the National Institutes of Health. According to literature ^[4] and experiment measurement, bubbles with diameter smaller than 2 mm were assumed to be spherical. More than 500 individual bubbles were counted for each measurement to achieve statistical reliability and to provide a representation of bubble size distribution under investigation. Results of bubble sizes and gas holdups of seawater flotation without frother and in the presence of 15 ppm of Custo527 at 5°C were summarized in Table 9. It can be seen that average bubble sizes decreased but gas holdup increased, as expected, in the presence of a frother. In addition, bubble sizes were quite uniform along the column when frother was added. Under no frother case, bubble sizes varied a lot along the column.



Figure 22. A digital image of gas bubbles analyzed using ImageJ 1.20.

Table 9. Hydrodynamic Parameters of Flotation Column Operations

Temperature (°C)	Sparger setting	No frother		15 ppm of Frother	
		6	8	6	8
5	Bubble size (mm)	0.57-0.83	0.35-0.48	0.38	0.38-0.40
	Gas holdup (%)	4.6	5.9	10.9	13.5
-2	Bubble size (mm)	0.56-1.00	0.45-0.60	0.30-0.34	0.35-0.37
	Gas holdup (%)	2.4	3.1	8.3	11.0

5.2.3. ANS Crude Separation in flotation column

Baseline Tests for ANS crude

To investigate the effectiveness of column flotation process for crude oil cleanup, ANS crude oil was tested without frother in seawater at 5 °C as a baseline. Oil feed rate was up to 200 mL/min. Water co-feed rate was up to 100 ml/min. To reach the steady state operation, a typical flotation column test lasted up to 45 mins, unless stated otherwise. Results were summarized in Table 10. Typical oil overflow from the top of column was shown in Figure 23. There was less than 15 ppm of oil in all water samples. Oil samples contained less than 10% of water when there was no co-feeding water, while water content can go as high as 50% in presence of water co-feeding.



Figure 23. Typical oil overflows from the top of flotation column.

Table 10. Summary of ANS Oil Column Flotation Results

Crude Conc. (%)	Flow rate (ml/min)	Temp. (°C)	Frother (ppm)	Demulsifier (ppm)	Residual oil in water (ppm)	Residual water in oil (%)
>90	200	5	0	0	<15	<10
60-70	300	5	0	0	<15	~50
			15	0	<15	<30
			0	10-50*	<10	<15
		-2	0	0	<15	<15
			15	0	<5	<10
			0	10	<5	<10

ANS crude oil in presence of frother test

The effect of frother to crude oil separation was studied: ANS crude oil was tested with a small amount of frother in seawater at 5 °C. Results were listed in Table 10. Typical oil flotation operation in presence of frother was shown in Figure 24. The total feed rate of oil and seawater was up to 300 mL/min. The concentration of frother Custo527 was at 15 ppm. Water sample was of less than 15 ppm of total oil. However, oil froth sample contained up to 30% of water.



Figure 24. ANS crude in presence of Custo527 (15 ppm) overflowed from the top of flotation column.

Bottle test for demulsifiers and water-in-oil emulsion study

NETL observed the formation of water-in-oil emulsion during co-feeding of seawater and oil, which was different from free oil tests, in a Denver cell during flotation column operation. As shown in Figure 25, a drop of water was entrapped inside of a thin oil film along with a droplet of crude oil, surrounded by lots of rising bubbles. Water-in-oil emulsion resulted high water contents in oil froth sample, as high as 50% in case of no surfactant, in oil froth samples, as shown in Table 11.



Figure 25. Water-in-oil emulsion was found in flotation operation.

To break this thin oil film and release water from oil, surfactants, i.e., demulsifier or water clarifier (reverse demulsifier), were recommended and NETL requested these commercial chemicals from manufacturers.

- Dow Corning: DM-2 demulsifier
- AkzoNobel: RTC-330 water clarifier
- Dow Chemical: ROMAX 6000 water clarifier

Procedures of bottle tests were modified from Dow chemical and AkzoNobel's S.O.P. Oily emulsion (~33% of water) with up to 500 ppm of demulsifiers were tested at ambient conditions. Before and after results of adding selected surfactants are shown in Figure 26. It showed that both demulsifier (DM-2) and water clarifiers (Romax6630 and RTC 330) separated water from oily water emulsion effectively in a short period of time. NETL was to develop a GPC method to analyze residual chemical concentrations in water samples from these bottle tests.

As a result, NETL decided that Romax 6000 water clarifier would be used for continuous oil flotation tests in 3" column.

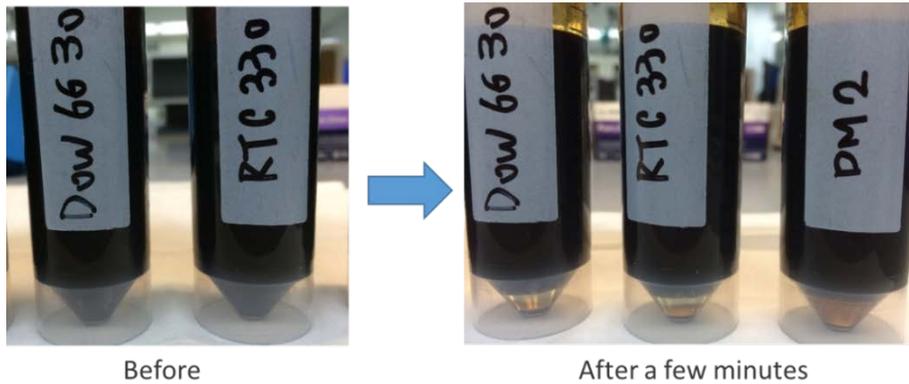


Figure 26. Water-in-oil emulsions (a) before and (b) after 120 seconds of adding 500 ppm of selected surfactants.

ANS crude oil in presence of water clarifier test

To investigate the effectiveness of the water clarifier to the column flotation for crude oil cleanup, ANS crude oil was tested with 10 ppm of Romax 6000 in seawater at 5 °C. Oil and water co-fed into column at rates of 200 mL/min and 100 mL/min, respectively. To obtain the steady state operation data, a typical test lasted for up to 45 mins. Oil flotation operation with Romax 6000 was shown in Figure 27. It showed that after adding 10 ppm of Romax 6000, the shape of oil droplets became spherical while oil/water emulsion bubbles were shapeless without the demulsifier. Results were also summarized in Table 10. Water found in oil froth samples from 10 ppm of Romax 6000 experiments was as low as 10%. By changing oil residence time in column, NETL believed that a better oil froth sample with less water can be obtained. Clean water samples were found containing less than 5 ppm of oil. Water samples were saved for future residual chemical analysis by the GPC method still in developing.



Figure 27. ANS crude in the presence of 10 ppm of Romax 6000 in flotation column at 5 °C.

Emulsified arctic oil test

To investigate continuous flotation process for the emulsified arctic oil, similarly to the previous Denver Cell batch operation, ANS emulsion solutions were prepared. The same preparation procedure was followed to prepare at least 30 liters of a stable solution of ~500 ppm of emulsified ANS oil in water.

Different from Denver cell tests, instead of using frother, Romax 6000 water clarifier was used. For all flotation tests, oil emulsion concentration was about 500 ppm. Water samples were taken after 30 minutes of flotation operation. Results were summarized in Table 11. It showed that more than 80% of oil emulsion was removed from water after flotation treatment. With the addition of a small amount of Romax 6000, oil emulsion was reduced to as low as 15 ppm. It was also found that temperature played a negative effect on the removal of oil emulsion primary due to the low efficiency of Romax 6000 at a temperature below the freezing point. A more detailed investigation was suggested. Overall, flotation column operation was able to successfully remove emulsified ANS crude oil from seawater near Arctic conditions.

Table 11. Experiment Results of Emulsified ANS Crude Oil

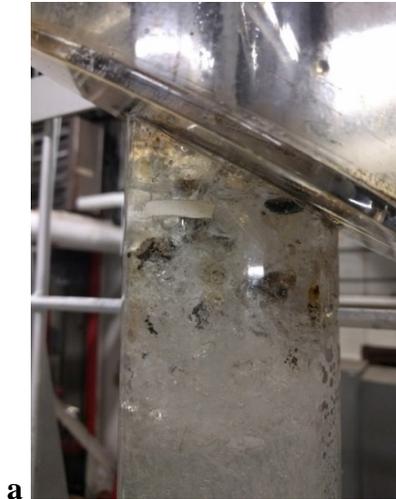
Oil Con. (wt%)	Frother	Frother con. (ppm)	Temp. (°C)	Residual oil in water (ppm)
0.05	None	0	5	<50
			-2	<100
	Romax 6000	10	5	<15
			-2	<30

5.2.4. Oil Contaminated Ice Tests

NETL has performed oil contaminated ice separation tests using a 3” column under baseline and frother operating conditions, similar to the previously discussed Denver cell tests. The average sizes of ice particles were ~5 mm and ~15 mm, respectively. The oil concentration was 10 wt% of ice, while the ice concentration was 10 wt% of water. Oil and ice mixtures were stored in a freezer overnight to simulate arctic temperature.

Results of oil contaminated ice (size ~ 15 mm) tests without the addition of surfactant before and after flotation treatment in a 3” column was shown in Figures 28 *a-c*. It showed that a flotation process successfully separated oil and oil contaminated ice particles from clean ice particles. The same conclusion was drawn for small oil contaminated ice particles in the presence of Romax 6000. Results were summarized in Table 12. During experiments, it was noted that the operating time for small ice particles was shorter than when dealing with large ice particles. However, the difference in time was not significant.

Therefore, again, flotation column process successfully demonstrated the separation of oil and oily ice from clean ice.



a



b



c

Figure 28. Oil contaminated ice (a) before flotation treatment, and (b) side view and (c) top view after flotation treatment in 3" column, respectively. (Baseline condition, ice particle size is 15 mm.)

Table 12. A Summary of oil contaminated ice tests

Oil con. (wt%)	Ice Con. (wt%)	Temp. (°C)	Frother	Frother Con. (ppm)	Ice size (mm)	Residual oil in water (ppm)
10	10	-2	None	0	1-5	<5
					5-15	<5
			Romax 6000	10	1-5	<5
					5-15	<5

5.3 Computational Modeling

Computational simulation is an effective tool for selecting the most efficient flotation solvents and exploring the kinetics/hydrodynamics in the floatation cell. In this project, by employing multi-scale modeling methods, NETL focused on 1) materials property at cold temperature, and 2) kinetics/hydrodynamics in flotation cell to improve the technology and explore the mechanisms behind each process.

The property of crude oil and surfactants and their interactions could change when the temperature drops very low (<0 °C). With molecular dynamics simulations, the temperature effects on the viscosity and surface tension of various chemicals have been calculated and the hydrophobic/hydrophilic interactions of surfactants with oil and salt water have been investigated. Based on the calculated results, a good surfactant can be selected for further testing.

The kinetics/hydrodynamics in a flotation cell has been simulated and analyzed for different oil feed and column geometries, using the computational fluid dynamics module of a COMSOL package, based on experimental data. The optimal working conditions and process will be used for future scale up investigation.

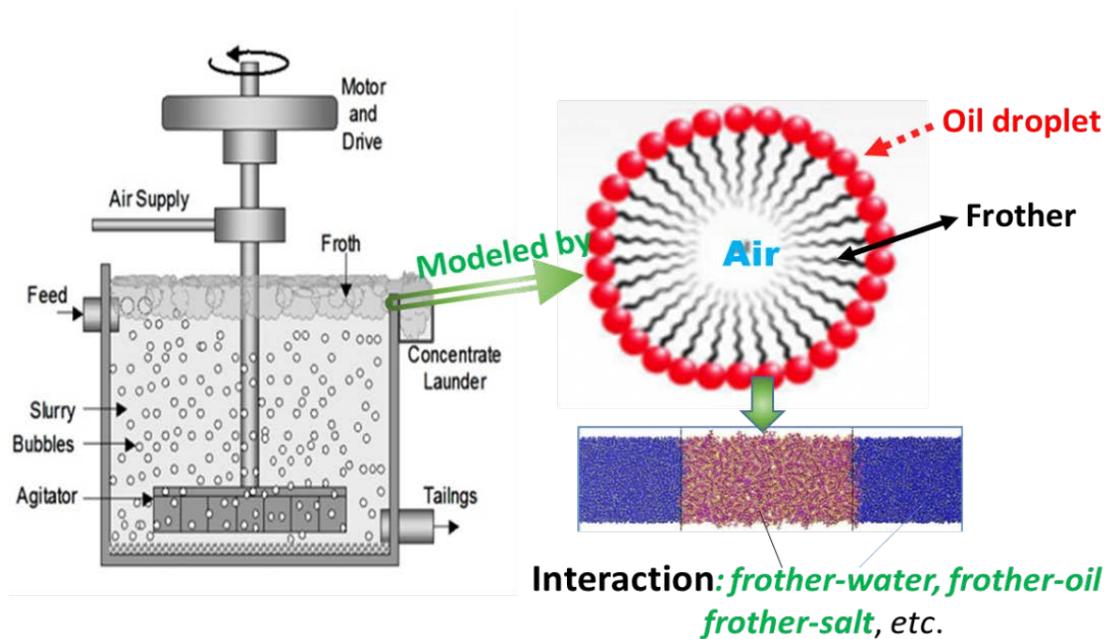


Figure 29. A schematic chart of interactions among air, frother, oil, and seawater.

5.3.1 Nanoscale Interface Modeling

To select the most effective frothers and explore the interactions among crude oil and cold saltwater, NETL applied a molecular dynamics model to simulate those properties which can guide experimental exploration. NETL focused on i) calculating the surface tensions of several frothers, ii) simulating the interactions between frother and crude oil in cold water, iii) exploring the properties of air-frother-H₂O and oil-frother-air. For each type of interface, surface tension calculations are of importance in assessing frother quality because such measurements are indicative of froth and bubble size and stability. As shown in the above schematic Figure 29, from the simulated results, a few frothers with good performance will be identified for experimental validation. The optimal work conditions will be predicted for experimental measurement with high performance of oil removal.

NETL has chosen to model a fluid-fluid interface model. The reasoning being that the effectiveness of oil recovery via froth flotation was dependent on the bubble properties with the water-air interface acting as the site for oil adsorption. The hypothesis was that smaller bubbles result in a larger total surface area that maximizes the available area for oil collision. Low surface tension fluids are known to stabilize small bubbles and inhibit their coalescence. Thus, there was interest in decreasing the surface tension at the water-air interface using frothers and other surfactant-like molecules.

Since this process was based on mineral froth flotation, NETL screened frothers that have been used for mineral froth flotation. Classes of molecules such as aliphatic alcohols, cyclic alcohols, and glycol ethers are well-established frothers in their pure forms and in commercial mixtures. From these options, NETL selected three frothers. Their chemical structures are displayed in Figure 30: methyl isobutyl carbinol (MIBC) is an aliphatic alcohol, alpha terpineol is cyclic alcohol and a component of pine oil, and ethyl glycol butyl ether (EGBE) is an elementary unit of polyglycol ethers. The above frothers were selected because of their low molecular weight and simple chemical structures that can allow us to isolate the effects of each functional group.

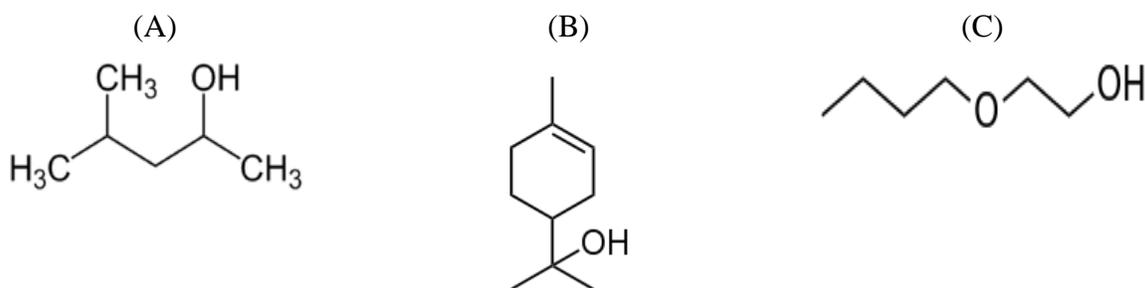


Figure 30. Chemical structures of (A) MIBC, (B) terpineol, and (C) EGBE

Assumptions are made regarding the compositions and chemical structures. After consulting a United States Geological Survey (USGS) report on crude oil samples^[27], NETL has decided to use a linear alkane of 17 carbons (C17) as the initial representative molecule of oil. Once the composition of Gulf and Arctic oil samples are known, future systems will likely contain additional oil impurities of varying chain lengths, cycloalkanes, and aromatics. In addition, the saltwater composition was assumed to have the 3.5 wt% salinity of seawater with NaCl as the

primary salt. The saltwater will thus contain water molecules with the appropriate concentration of Na⁺ and Cl⁻ ions.

The molecular dynamics method is a physics-based process capable of simulating nanoscale systems that can span several nanoseconds. Based on their initial positions, atoms experience potential energy derived from charges, bonds, angles, and other terms. Resulting force, acceleration, velocity, and displacement vectors are then applied to each atom. Time is incremented on the order of femtoseconds, and the atoms' positions are updated. By repeating this algorithm, NETL can monitor thermodynamic properties such as temperature, pressure, and energy over time until the interface reaches equilibrium. At that point, NETL samples the atom trajectories in the calculation of surface tension.

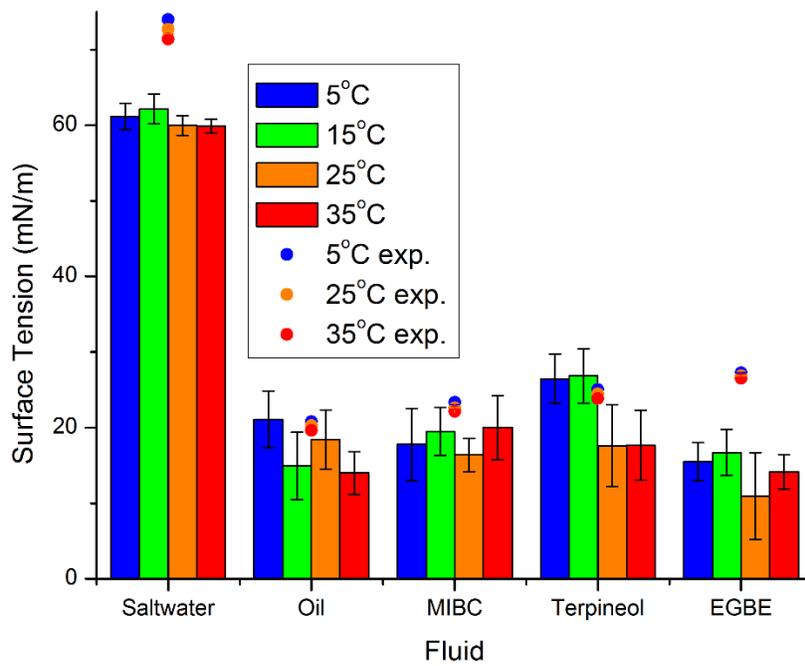
For surface tension calculations, the integral is expressed as following:

$$\int_0^{L_z} \left(\frac{P_{xx} + P_{yy}}{2} - P_{zz} \right) dz$$

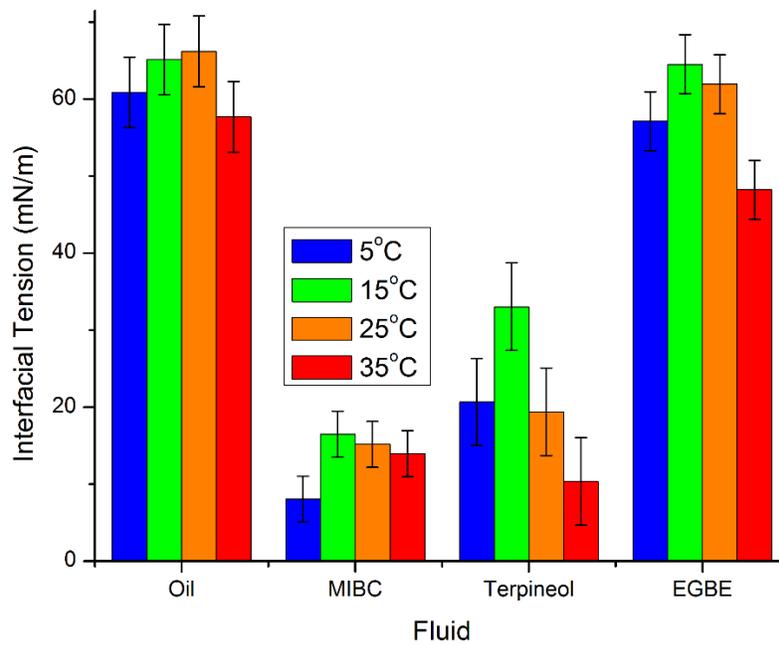
For an interface plane that is to the xy plane, L_z is the length of the simulation box, and the diagonals of the pressure tensor are P_{xx} , P_{yy} , and P_{zz} .

NETL focused on building interface systems to simulate the oil adsorption on bubbles for three types of interfaces that may arise: binary, frother-saltwater-air, and oil-frother-saltwater-air. By modeling binary interfaces of liquid-air, NETL can validate with experiments and assess compatibility of the model. Also, binary liquid-liquid interface simulations aim to observe the miscibility of each fluid to determine how mixing can affect the surface tension calculations of the other interfaces. Measuring this property for frother-saltwater-air interfaces enables NETL to draw connections between frother surface coverage with bulk concentration.

Finally, the oil-frother-saltwater-air interface serves as a possible interface configuration that may occur when oil adsorbs to bubbles or froth. The simulation results will provide insights on the interactions among frother, oil and water, and on the mechanisms of separation, therefore, to guide experimental measurements under optimal conditions and with most effective frothers. Further, by combining with chemical engineering and process modeling, some useful guidelines (such as the system design and scale-up, optimal working conditions, the overall costs, *etc.*) will be obtained and used for improving the performance of oil removal technology.



(a)



(b)

Figure 31. Surface tensions of (a) liquids with air and (b) frothers and oil with saltwater.

5.3.1.1 Binary Interfaces

After simulating the binary interfaces, NETL obtained, calculated, and compiled their surface tensions into Figure 31. The surface tensions of liquids with air agree well with experimental data with a majority of the simulated results slightly underestimating the actual surface tensions. Figure 31a effectively demonstrates that NETL's model is effective in predicting the trends where frother-air interfaces possess lower surface tensions than water saltwater-air interfaces. It is important to note the oil-air surface tension is also as low as the frother-air surface tension, suggesting miscibility that can occur between oil and frother. NETL advances the model further by simulating saltwater-frother interfaces as depicted in Figure 32. Doing so enables NETL to focus on specific binary interfaces that can arise when bulk liquids of oil and frother encounter saltwater. Those surface tension results are shown in Figure 31b. The saltwater-oil interfacial tension is approximately equal to the saltwater-air surface tension.

Unlike the frother-air surface tensions, the saltwater-frother interfacial tensions exhibit distinguishing features between each frother. The interfacial tensions with saltwater appear to follow the trend: EGBE > terpeneol > MIBC. EGBE has a markedly higher interfacial tension with saltwater when compared to the other frothers likely due to the lack of mixing between EGBE and saltwater. Additionally, NETL's model found very little relationship between surface tensions and temperature. The expected pattern of low surface tensions for high temperatures was barely observed in experiments. Some statistical noise from random atomic motion may be affecting the surface tension measurements of nanoscale interfaces.

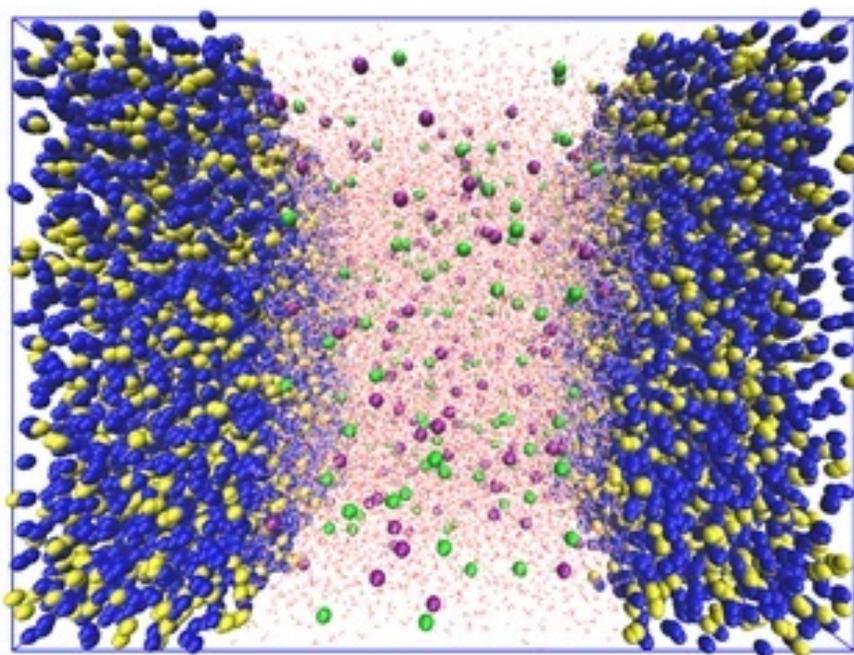


Figure 32. EGBE-saltwater interface: EGBE (blue and yellow), water (red and white), and salt (purple and green)

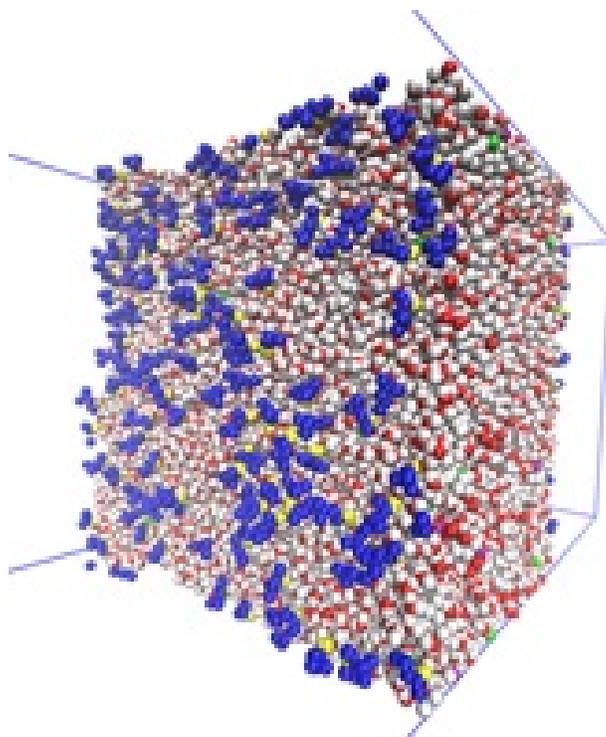


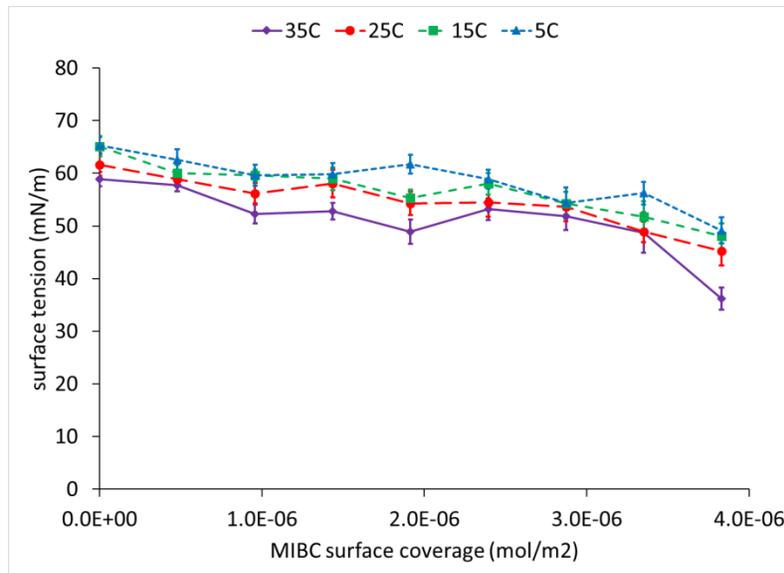
Figure 33. MIBC on saltwater-air interface: MIBC (blue and yellow), water (red and white), and salt (purple and green)

NETL simulated saltwater interfaces with oil and frothers as part of the liquid-liquid interfacial tension calculations. In this configuration, NETL found that EGBE-saltwater had higher surface tensions and smaller interface thicknesses than terpineol-saltwater and MIBC-saltwater. Also, NETL performed frother-oil modeling to better understand the interface stability. Negative surface tensions of frother-oil interfaces as reported in Table 13 should not be feasible and likely indicate mixing. Density profiles confirm significant mixing for all frothers with oil such that practical interfacial tension calculations could not adequately characterize the interface.

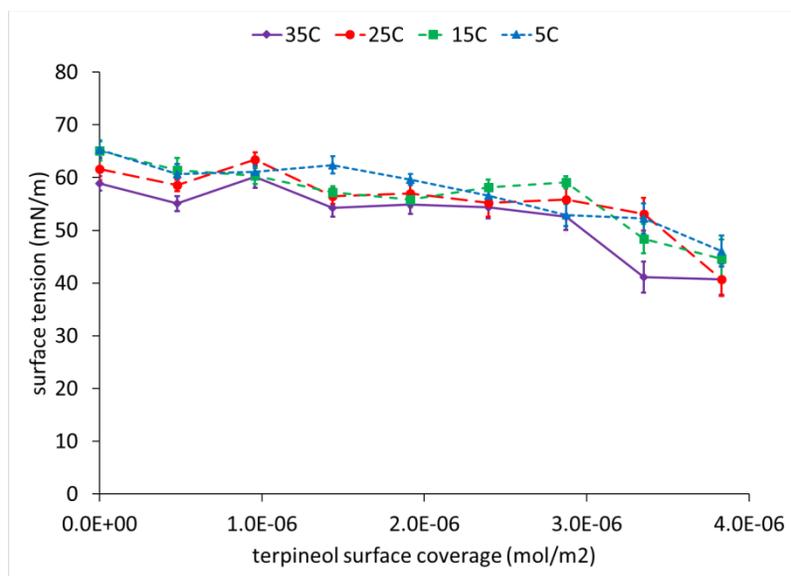
The differences in miscibility of the frothers with oil and the frothers with saltwater revealed the overall hydrophobic nature of the frothers. Of the three frothers tested, only MIBC and terpineol were shown to have comparable liquid-air surface tensions with experiments which supports the applicability of NETL's model for these two frothers. Since MIBC-saltwater interfacial tensions are lower than terpineol-saltwater interfacial tensions, an emulsion of MIBC in saltwater is theoretically more stable than terpineol in saltwater. Further conclusions regarding oil adsorption to air bubbles and froth required modeling saltwater-air interfaces with varying surface concentrations of frother and oil. During froth flotation, the presence of three phases, frother-oil-saltwater, will be common and is the logical next step in modeling to develop NETL's understanding of froth stability and improve oil separation.

Table 13. The calculated interfacial tensions of binary systems (mN/m). Values in parentheses are from experiments.

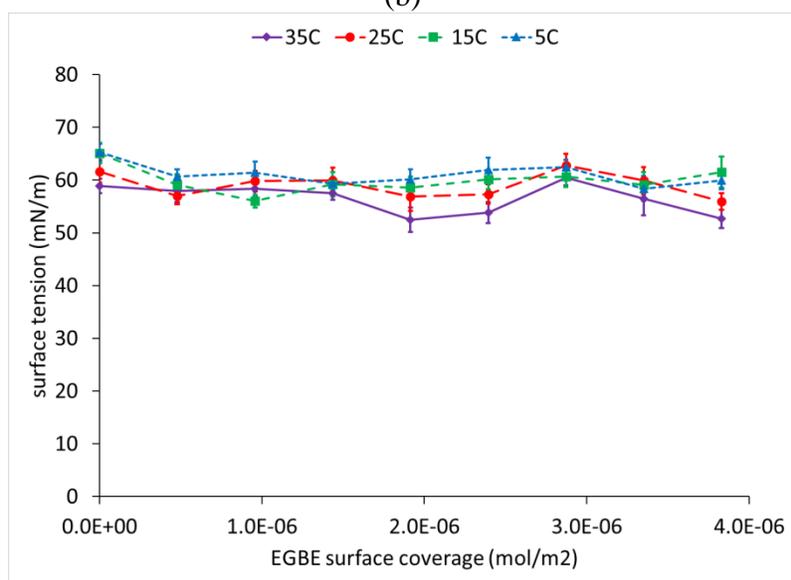
Liquid A	Liquid B	5°C	15°C	25°C	35°C
Saltwater	Air	61.16 (73.99)	62.15 (73.5)	59.96 (72.7)	59.89 (71.39)
Oil	Air	21.1 (20.79)	14.94	18.46 (20.27)	14.01 (19.63)
MIBC	Air	17.77 (23.37)	19.49	16.39 (22.64)	20.03 (22.14)
Terpineol	Air	26.48 (25.07)	26.85	17.63 (24.5)	17.68 (23.85)
EGBE	Air	15.49 (27.28)	16.72	10.95 (26.88)	14.14 (26.53)
Oil	Saltwater	60.87 (52.48)	65.15 (51.86)	66.2 (51.24)	57.69
MIBC	Saltwater	8.05	16.5	15.16	13.96
Terpineol	Saltwater	20.66	33.02	19.39	10.34
EGBE	Saltwater	57.14	64.53	61.94	48.21
MIBC	Oil	-6.16	-9.29	-2.9	4.31
Terpineol	Oil	2.51	-20.05	3.72	-5.34
EGBE	Oil	6.04	12.95	1.93	-2.59



(a)



(b)



(c)

Figure 34. Saltwater-air surface tensions as a function of surface concentration for (a) MIBC, (b) terpineol, and (c) EGBE

5.3.1.2 Frother-Saltwater-Air Interfaces

Figure 33 is a snapshot of the ternary system that evolved from the binary saltwater-air interface. Frother molecules are gradually added to the saltwater surface to determine the relationship between surface tension and the concentration of frother at the surface. Despite the relatively large ratio of water to frother molecules, the frother remains predominantly at the surface without diffusing into the bulk. As shown in Figure 34, the surface tensions mostly decrease, as in the case of MIBC and terpineol, or remains constant, as in the case of EGBE. Over the range of surface concentrations tested, the surface tensions decrease was greatest for MIBC and lowest for EGBE. At 25°C, the theoretical surface tension of terpineol decreased from 61 mN/m to 40

mN/m while the experimental surface tension decreased from 73 mN/m at 0 ppm to 52 mN/m at 500 ppm. Although the absolute value of the surface tensions is offset, the magnitude of the surface tension decrease is similar between the simulation and experiments. A similar emerges for EGBE where an increase in surface coverage and increase bulk concentration yields only small surface tension decrease of less than 5 mN/m. The combined data demonstrates the potential for NETL's model to link surface coverage, a nanoscale property, with bulk concentration, macroscale property.

5.3.1.3 Oil-Frother-Saltwater-Air Interface

To introduce oil into the interface, NETL adds a large amount of C17 molecules, as shown in Figure 35, to the surfaces saltwater-air interfaces with frother molecules present. The result is a volume of saltwater bounded by a layer of frother ($1.915 \times 10^{-6} \text{ mol/m}^2$), bulk oil, and then air. The system is intended to mimic the spreading oil droplet across an air bubble that is stabilized by frother. With the addition of oil, the surface tensions exhibit a noticeable increase in surface tensions as illustrated in Figure 36. This occurred regardless of the type of frother present and is an expected outcome since oil and water naturally do not mix. However, this phenomenon may hinder the stability of bubbles and counteract the lowering of surface tension caused by the frothers. It is worth noting the surface tensions are higher than the values found for bulk saltwater-oil from Figure 31b. This suggests that the dilute amount of frother added between the oil and saltwater is having a significant effect on the overall surface tension calculation. There is also an issue of the amount of oil present at the interface because bubbles may encounter free floating oil or emulsified oil. This difference may play a role in how much oil should be at the interface.

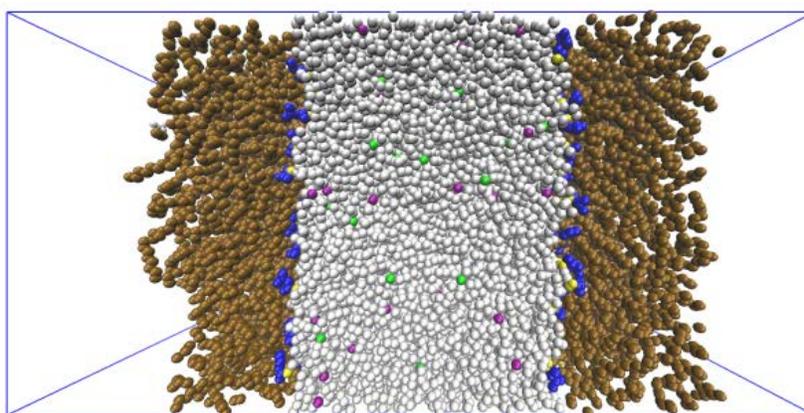
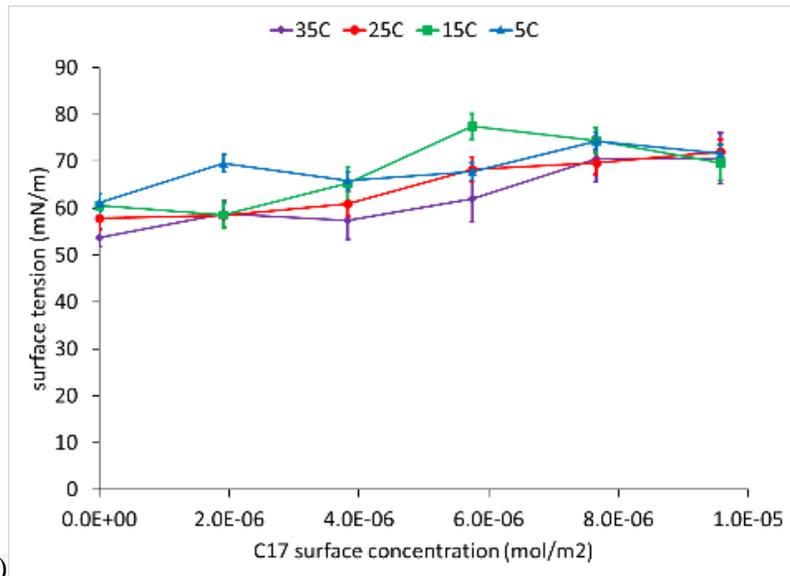


Figure 35. C17 and MIBC on saltwater-air interface: C17 (brown), MIBC (blue and yellow), water (white), and salt (purple and green)

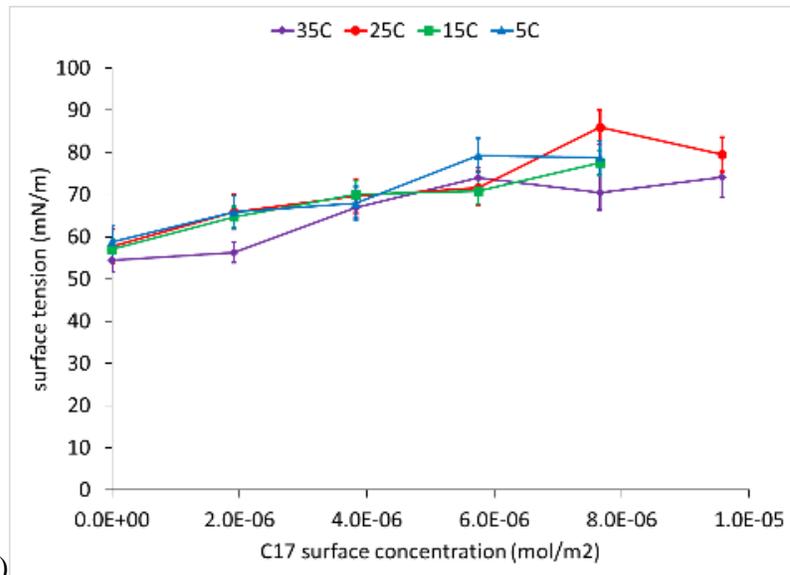
5.3.1.4 Summary of Interface Modeling

By calculating the surface tension of liquid-air interfaces, NETL found MIBC and terpineol appear to be closest in agreement with experimental measurements. Furthermore, the frother-saltwater interfaces are consistently immiscible whereas the frother-oil interface undergoes significant mixing. In addition, a sweep of the surface coverages of the frothers on saltwater-air

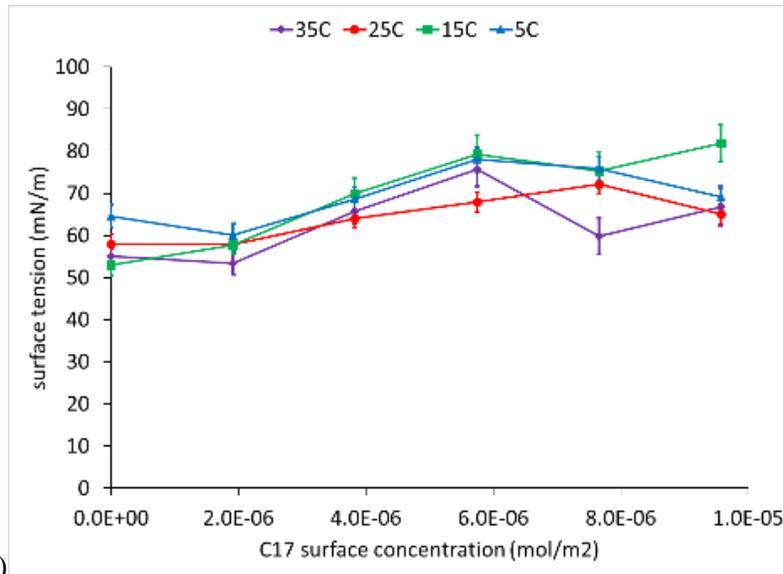
interfaces demonstrate 0 to 3.83×10^{-6} mol/m² is a range of surface coverages that can predict a similar trend for frother bulk concentrations 0 to 500 ppm. The addition of oil to saltwater-air interfaces with dilute frother coverage demonstrate a rise in surface tensions and a possible risk to froth and bubble stability. The results on interface modeling have been presented at the 2016 American Institute of Chemical Engineers (AIChE) meeting and submitted to the Journal of Physical Chemistry B.



(a)



(b)



(c) Figure 36. Surface tensions as a function of C17 surface concentration for (a) MIBC, (b) terpineol, and (c) EGBE frothers at the saltwater-air interface

5.3.2 Froth Flotation Column Hydrodynamics

COMSOL Multiphysics is a finite element analysis solver that is a software package that consists of various modules that can couple different physics phenomena when combined. The finite element method breaks down complex geometries into discrete units and solves the multiple local equations to converge to an overall solution. The solver requires coefficients for the physics equations and boundary values for each point, area, and volume domains of the geometry.

NETL utilized the computational fluid dynamics module from COMSOL 4.3b. The laminar bubbly flow model from this module is suitable for two phases and can track liquid and gas velocities, volume fractions, dynamic viscosities, and densities.

5.3.2.1 Column Setup

NETL used COMSOL simulation software to model the flow of water and air in the flotation column to better understand the hydrodynamics and scale-up possibilities. As depicted in Fig. 37, NETL designed a cylindrical geometry that matches current experimental setup. Beginning with an initial volume of water, air is introduced at bottom while a liquid feed is injected at the upper half of the column. For the purposes of studying only hydrodynamics, the feed consists of only water instead of oily water. A small percentage of the water volume injected is siphoned off at the top of the column lip in order to represent froth overflow. Additionally, tailings outlet at the column bottom removes a majority of the injected water. The model for our base case takes on the dimensions of our experimental column. It is 2 m tall and 7.5 cm in diameter with a feed injection site and gas injection site 1.667 m and 0.250 m from the bottom, respectively. There is also a 0.635 cm diameter tailings outlet located at the bottom of the column. The diameters of the feed inlet and tailings outlet are 0.635 cm, and the diameter of the gas inlet is 2.5 cm. The top of the column is the gas outlet capping a 1 cm rim forming the lip area acting as the overflow. The flow rates for each outlet and inlet are laid out in Figure 37. The operating conditions are assumed to be 25°C and 1 atm with an air density of 28.97 g/mol. The bubble sizes are set to 0.4

mm in diameter which was the average bubble diameter observed in the experimental column. Since the two phases consist of air and water, the surface tension was 72 mN/m. Table 14 summarizes the range of column parameters tested.

Table 14. Preliminary scale up study for flotation column

Parameter	Baseline (Fig. 38a)	Scaled-Up Parameters				
		Gas Flow Rate (Fig. 38b)	Height (Fig. 38c)	Radius (Fig. 38d)	Spargers (Fig. 38e)	Feed Flow Rate
Height (m)	2.0	2.0	1.7	2.0	2.0	2.0
Radius (cm)	3.75	3.75	3.75	11.25	11.25	3.75
Feed Flow Rate (L/min)	0.1	0.1	0.1	0.1	0.1	0.2
Gas Flow Rate (L/min)	1.0	0.2	1.0	1.0	1.0	1.0
Sparger	1	1	1	1	5	1

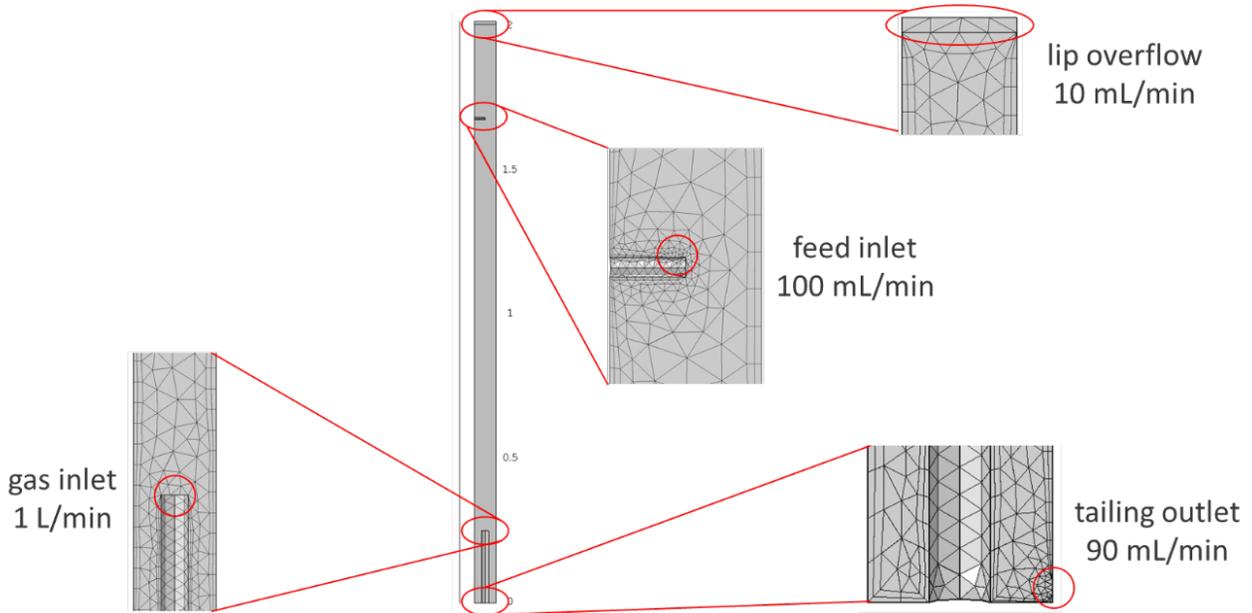


Figure 37. Base column model for COMSOL simulations

5.3.2.2 Simulation Results

Scale-up simulations are critical for studying how geometry changes affect flow and so results mostly track liquid velocity along various axes. As Figure 38 lays out, NETL initially focused on adjusting flow rates, heights, radius and sparger configuration. By lowering the gas flow rate from 1 L/min to 0.2 L/min, our simulations found the shape of the velocity profile across the column diameter to be largely similar as depicted in Figure 39. The width of the distribution of positive upwards liquid velocity remains 0.04 m in both cases with negative downwards velocities in the area outside the center stream. One key difference between the two flow rates are the peak velocities. At 1 L/min gas flow rate, the peak centerline velocity is 0.05 m/s. This velocity decreases to 0.025 m/s when the gas flow rate is reduced to 0.2 L/min. Thus, a reduction

in gas flow rate does not reduce the liquid velocity by the same proportion. This relationship is also present in the downward liquid velocities near the column walls.

To obtain a better picture of how the liquid velocities are affected by changes in column geometry, the distributions along the center of the column and at 1 m were plotted in Figure 40a. After 40 s of simulated operation, the peak velocity is observed to be greater and fluctuates more around the center for larger radii. This reveals an interesting effect where wider radii result in a slightly higher peak velocity for the liquid. On the other hand, thinner columns exhibited larger downward velocities near the walls. The inverse relationship between the center and near-wall velocities regarding the column radii is worth additional simulations that can determine the limit for the peak velocity. Also, shown in Figure 40b, the peak velocity and decay appear to be constant for each height and the feed inlet causes velocity increases regardless of location. However, the results presented are of only small changes in the column height. Modeling a higher range of the column may be able to discern how constant velocity decay will settle to an equilibrium liquid velocity.

Four gas spargers were added to the bottom of the column in a cross pattern. All spargers had inlet openings that were a fifth of the area of the original single sparger. This allowed the inlet gas velocities to be equal to the original inlet velocity. As Figure 38e shows, the velocity distribution across the diameter is broader than with a single sparger, although significant horizontal fluctuations to the peak velocity are still present.

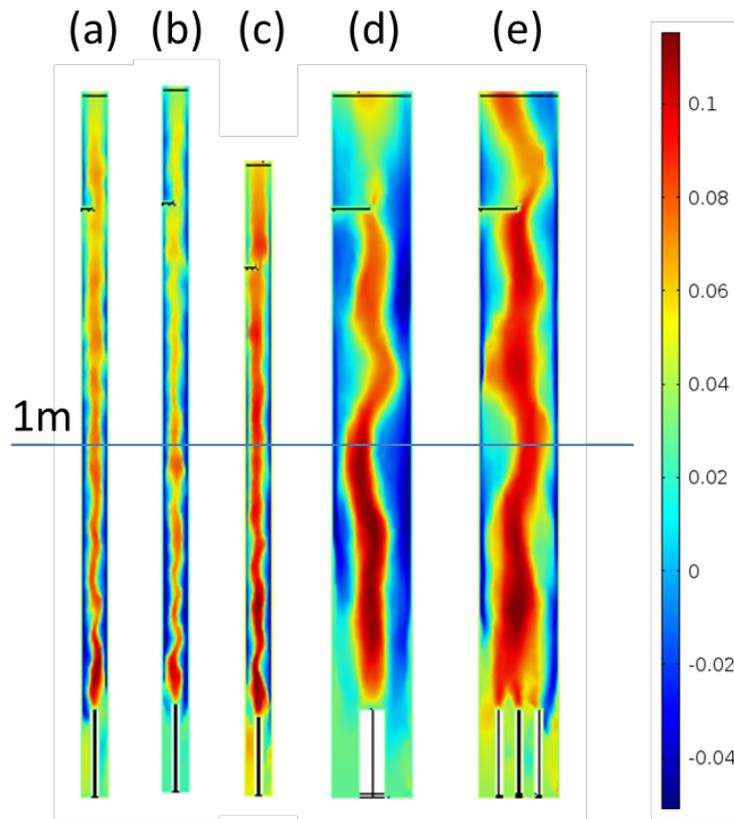
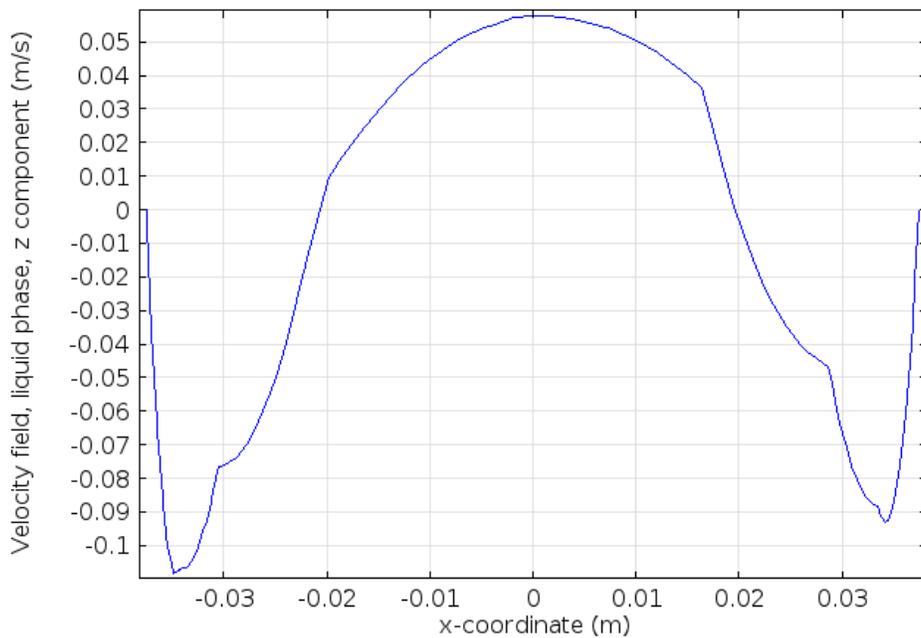
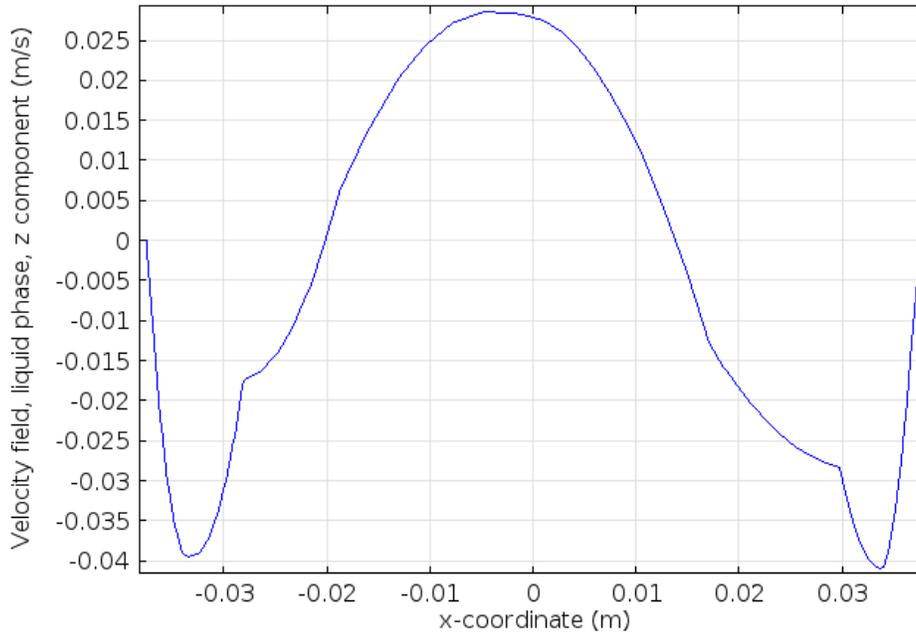


Figure 38. Liquid velocities of scale-up changes to the (a) initial column by (b) reducing flow, (c) reducing height, (d) increasing radius, and (e) multiplying spargers. The figure on the right is the legend in m/s.

The theoretical velocity of the liquid feed is 3.77×10^{-4} m/s. For a diameter of 7.5 cm, density of 1 g/mL, and viscosity of 0.89 cP, the theoretical Reynold's number is 31.791. As the velocity distributions show, the simulated centerline velocities are significantly larger due to the high gas flow rate. They range from about 0.03 m/s to 0.05 m/s which results in Reynold's numbers ranging from 2528.1 to 4213.5. This is important to note because, for a flow in a pipe, a Reynolds number of less than 2000 constitutes laminar flow whereas a Reynolds number greater than 4000 is turbulent flow. Thus, the current settings for the column results in a flow that lies in the laminar-turbulent transition region. For the purpose of froth flotation, it is preferable to maintain a laminar flow to avoid bubble instability and excessive oil mixing. If the downward liquid velocity near the walls are used to calculate the Reynold's number, it would be 5056.2 to 8427 for a velocity range 0.06 m/s to 0.1 m/s which would a sufficiently turbulent flow. If the gas flow rate was reduced to 0.2 L/min, as done in Figure 39b, the maximum liquid velocity would be less than 0.04 m/s and thus a Reynold's number of less than 3370.8.



(a)

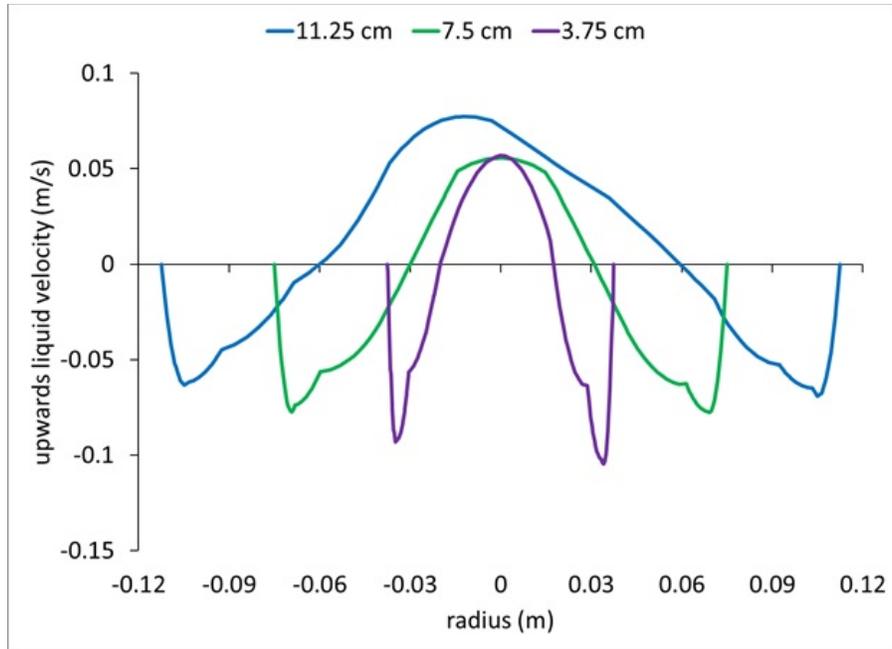


(b)

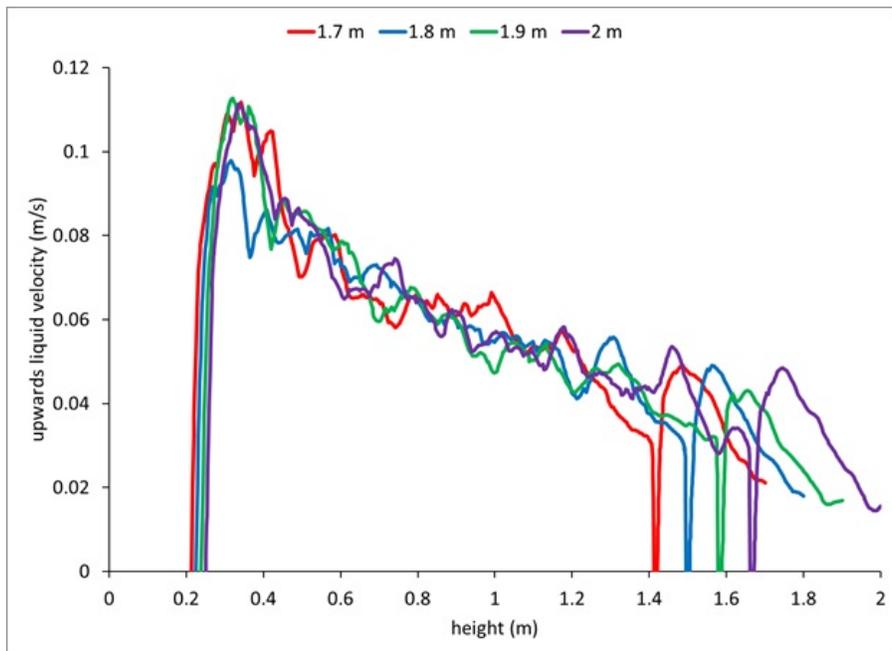
Figure 39. Water velocity profiles at 1 m height for (a) 1 L/min and (b) 0.2 L/min gas flow rate

5.3.2.3 Scale-Up Conclusions

The above findings provide some key pointers for scaling up the column. Oil adsorption to the air bubbles and froth are dependent on the hydrodynamics. An optimal behavior for fluid flow allows for higher residence time of bubbles to collide with oil droplets. As the velocity profiles have shown for all geometries and flow rates, there is an upward flow at the center of the column with a downward flow at the walls. This poses a risk for oil to be drawn towards the tailings outlet where cleaned water should exit. A low gas flow rate has shown that this downward liquid flow can be reduced. When this is combined with the observation that the gas flow is positive for all positions in the column, the fluid action at the walls is thus a countercurrent flow where liquid flows down and air flows up. Maximizing oil interaction with air bubbles near the column wall may be beneficial in that regard. However, it is critical to keep a low gas flow rate because it has the largest effect on liquid velocity which, in turn, contributes to a transition to turbulent flow. It is important to emphasize these simulation observations are based on water and air only but have revealed useful hydrodynamics data on flow patterns. Therefore, simulation tools that can incorporate oil as the third phase will be significantly more appropriate in drawing conclusions for scaling up.



(a)



(b)

Figure 40. Water velocity profiles at (a) 1 m height for 3 column radii and (b) the centerline for 4 column heights

Since these preliminary results are very useful for designing new floatation column and improving the efficiency of separation, further simulations should be focused on pilot-scale level to optimize the column size, to design the flexible column head used onboard, and to explore the effective inlet fluid flow pattern for high separation performance.

5.3.3 Deliverables

- **Publications**

- 1) Leebyn Chong, Yungchieh Lai, McMahan Gray, Yee Soong, Fan Shi, Yuhua Duan, “*Molecular Dynamics Study of Frother Interfaces with Oil, Saltwater, and Air*”, **Journal of Physical Chemistry B**, (2017), DOI: 10.1021/acs.jpcc.6b13040
- 2) Leebyn Chong, Yunchieh Lai, McMahan Gray, Yee Soong, Fan Shi, Yuhua Duan, “*Molecular Dynamics Study of Frothers and Oil at Saltwater-Air Interfaces*”, **Journal of Physical Chemistry B**, (2017) to be submitted

- **Presentations**

- 1) Leebyn Chong, Yungchieh Lai, Fan Shi, McMahan Gray, Yee Soong, Yuhua Duan, “*Molecular Dynamics Study of Surfactant and Oil Interactions at Saltwater-Air Interface*”, **AIChE 2016 Annual Meeting**, Nov. 13-18, 2016, San Francisco, CA
- 2) Leebyn Chong, Yungchieh Lai, Fan Shi, McMahan Gray, Yee Soong, Yuhua Duan, “*Surface Tensions of Frothers and Oil at Saltwater-Air Interfaces: A Computational Study*”, **ACS Fall Meeting**, Aug. 20-24, 2017, Washington DC

6. Summary

The NETL team achieved the primary goals of the proposed work. The strength of the NETL team continues to allow for an efficient development of the work with a strong focus on quality.

In summary, NETL reached these conclusions and developed the following remarks:

- Milestone 1: Experimental planning and preparation was successfully accomplished
 - NETL completed the Safety Analysis Review System (SARS) package for the BSEE project. The project has been ISO approved by DOE/NETL.
 - NETL developed a testing matrix for the oil flotation experiment in a Denver cell and in a 3” flotation column. The experimental plan (Task 1D) has been reviewed and approved by BSEE.
 - Materials preparation (Task 1A) was accomplished. All materials for the project were purchased *via* collaborators and vendors, respectively. NETL has requested ANS, North Star (NS), Ewing Bank (EB) and Harmony (HC) crude oils from BSEE. A 55-gal ANS crude oil drum has been received and stored per the safety regulation of the DOE.
 - Analytical methods for the measurement of trace amount of oil and frother in seawater were successfully developed. Oil analysis, KF titration, and GC-SPE methods have been developed for the analysis of water and oil samples.
 - Bench scale Denver cell and a continuous 3” flotation column were set up for oil tests in cold seawater.
- Milestone 2: NETL has successfully accomplished the proof of concept study using a benchtop Denver cell.
 - Four crude oils, i.e., ANS, NS, EB, and Harmony, were tested using a Denver cell under different operating conditions.
 - A GC method with solid phase extraction (SPE) was used for the measurement of frother in treated seawater with residual crude oil. Residual frother in bulk seawater decreased along the operating time. Residual concentration of F660 was less than 2 ppm after five minutes of flotation testing.
 - Commercial frothers, including 2-EH, MIBC, F660, F672, and Custo527, were tested. Based on the investigation of frother performance, NETL selected F660 and Custo527 for further crude oil clean up tests under Arctic conditions.
 - Flotation process was able to remove different types of crude oils, even emulsified oil, and oily ice, from seawater under varied conditions.
 - Both ANS and NS crude oils were successfully cleaned-up at -2 and 5 °C from seawater by using a Denver cell flotation treatment. Results showed that residual NS crude and ANS crude in bulk seawater were less than 15 ppm and 5 ppm, respectively. It met EPA’s discharge standard. Water in oil is less than 20 wt%.

- Crude oil clean-up of both Ewing Bank and Harmony from seawater were successfully accomplished at 30 °C. Results showed that residual crudes in bulk seawater was less than 5 ppm. Water in oil was less than 15 wt%.
 - The treatment of 400 ppm of ANS crude emulsion was successfully demonstrated in a Denver Cell. At the end of 60 minutes of flotation operation, less than 5 ppm of residual oil was detected in bulk water.
 - Oily ice cleanup in a Denver cell was successfully demonstrated. For small ice particles (1~5 mm), oil recovery rate was up to 95% and residual oil concentration was as low as less than 2 ppm. For large ice particles (5~15 mm), oil recovery rate was still greater than 90% and residual oil concentration was less than 5 ppm.
- Milestone 3: Continuous removal of crude oil was effectively performed using a 3” flotation column.
 - NETL and ErieZ (Industrial partner) have signed the NDA form for an oily water separation project.
 - Continuous clean-up of ANS crude oil from seawater was successfully tested at -2 and 5 °C, respectively. Results showed that residual ANS crude in bulk seawater was less than 10 ppm in presence of 10 ppm of water clarifier (Dow Chemical’s ROMAX 6000 Demulsifier). It met EPA’s discharge standard. Water in oil was as low as 10 wt%.
 - Tests for emulsified ANS (500 ppm) and oily ice were successfully accomplished with outstanding results obtained.
- Milestone 4: Computational simulation was performed.
 - Fluid-fluid interface model was successfully applied at the molecule level simulation to understand effects of function groups of chemicals, such as aliphatic alcohols, cyclic alcohols, and glycol ethers, used for oil separation from salty water at simulated Arctic temperatures.
 - Simulation of the interfacial property among demulsifier, oil and seawater was initiated.
 - Process simulation to better understand the hydrodynamics and scale-up possibilities of column flotation was accomplished by using the COMSOL software.
 - Hydrodynamics were investigated at varied operating conditions.
 - Different column geometries and feed positions were studied.
- Overall, flotation operations have shown excellent ability to successfully remove crude oils from seawater at simulated arctic and gulf temperatures.

7. Recommendations

A technology transfer goal of the Department of Energy's National Energy Technology Laboratory is intended to facilitate the commercialization of energy-related technologies with promising commercial potential that are developed at its facilities. This goal is part of a broader set of initiatives to foster stronger partnerships among government facilities, private sector companies, and other entities involved in bringing energy-related technologies to the marketplace. The collaboration on the current project with the Department of Interior's Bureau of Safety and Environmental Enforcement has greatly assisted in the maturation of this flotation technology that will potentially remediate oil spills in the Arctic, as well as in the Gulf of Mexico, Atlantic Ocean, and Pacific Ocean. Although significant research and development advances have been made over the duration of this project, the current Technical Readiness Level (2-3) could be further increased to attract interest by outside commercial entities. It is suggested that this relatively modest increase in Technical Readiness Level (TRL)^[28] be conducted under a further collaboration with the Bureau of Safety and Environmental Enforcement.

Upon the recent completion of the proof-of-concept and laboratory-scale testing (TRL 2-3), it is necessary to conduct pilot-scale field testing (TRL 4-5) with actual oil streams to determine potential impacts on the flotation process. With the commercialization path in mind, NETL would like to recommend the following topics for further discussion and development of the technology:

- To continue the development of a pilot-scale onboard flotation skid at a feed rate greater than 50 liter/min (TRL 4-5):
 - Retrofit current 3" bench scale flotation column unit and continue separation testing on varied conditions.
 - Investigate weathered crude oils from different regions, including Pacific, Atlantic, Gulf of Mexico, and others. (Note that although the onus of the current work has been with Arctic crude, NETL has performed some ancillary testing with Gulf of Mexico crudes).
 - Modify and retrofit ErieZ's 12" pilot scale flotation column, as shown in Figure 41, for crude oil/seawater separation at a feed rate greater than 50 liter/min.
 - Accomplish field tests for oil separation at a feed rate of 50 Liter/min.
 - Plan a series of continuously outdoor field tests at ErieZ Company, or at the BSEE's Ohmsett Facility, NJ.
 - Conduct an economic analysis for the separation of crude oil from seawater using flotation technology.
 - Optimize the cost/size/weight of flotation skid for commercial applications.
 - Complete the design of 20(24)" full-size demonstration scale flotation column at a feed rate greater than 500 liter/min.
- Oil spill response efficiency can be significantly limited by the onboard storage capacity for recovered spilled oil mixture. The effective storage capacity may be improved by integrating an on-boat flotation separation technology with a current skimmer to reduce

water content in recovered oil/water mixture at sea, enhancing overall oil spill response efficiency.

This project has produced intellectual property that will be of major importance in the future commercialization of the technology. Recently, the project concept was accepted by our internal patent review board and will be submitted to patent examiners for formal approval. The additional maturity of the technology along with the intellectual property will facilitate the commercialization of the technology. As such, the Department of Energy conducts a Technology Commercialization Fund Solicitation every year and a synopsis of this technology in the form of a proposal will eventually be submitted. The solicitation requires a commercial partner and we plan on attracting commercial entities such as Mobil, Exxon, etc. in our proposal. Already a manufacturer of flotation devices (ErieZ Company, PA) has expressed interest in our work and will be instrumental in completion of the design/construction of a prototype unit of our “onboard flotation system.”

The following Figure 41 shows a future 3-year roadmap of scaling up the flotation technology for oil spill response at sea.

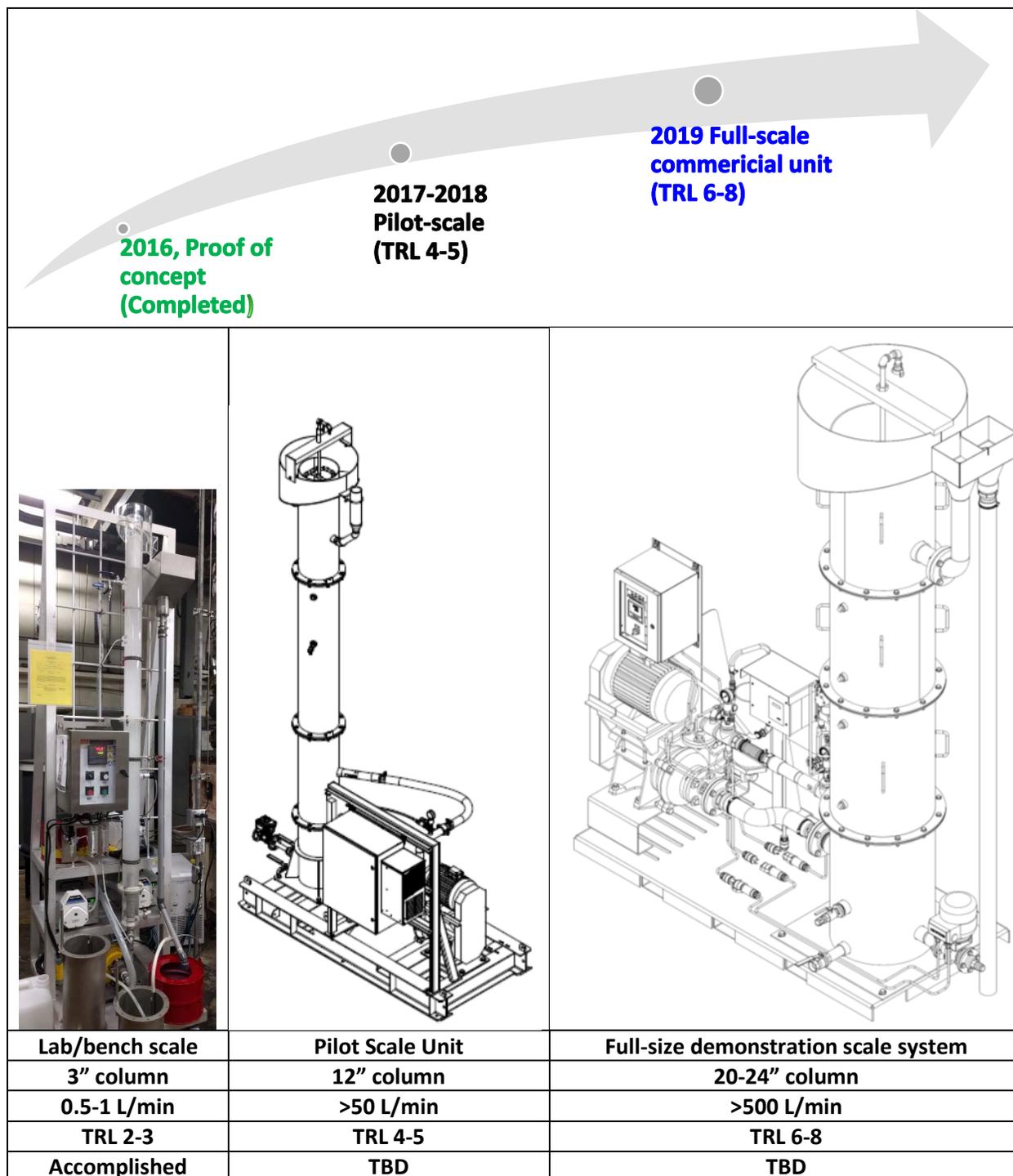


Figure 41. A scale-up process of flotation technology for oil spill response: Research, Development, and Demonstration.

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MATERIAL SAFETY DATA SHEET

Crude Oil, Alaska North Slope

1. PRODUCT AND COMPANY IDENTIFICATION

Product Name: Crude Oil, Alaska North Slope

Synonyms: ANS Crude
Alaska North Slope (ANS) Crude Oil
Crude Oil (ANS Type)
Earth Oil
Petroleum Oil
Rock Oil

Chemical Family: Petroleum Hydrocarbon

Responsible Party: CPA
A Subsidiary of ConocoPhillips
P.O. Box 100360
700 G. Street
Anchorage, AK 99510-0360

For Additional MSDSs 800-762-0942

Technical Information: 907-659-7812

EMERGENCY OVERVIEW

24 Hour Emergency Telephone Numbers:

Spill, Leak, Fire or Accident

California Poison Control System: (800) 356-3129

Call CHEMTREC

North America: (800)424-9300

Others: (703)527-3887 (collect)

Health Hazards/Precautionary Measures: May contain or liberate poisonous hydrogen sulfide gas. Harmful if inhaled. Skin cancer hazard. Causes eye and skin irritation. Overexposure to a component may cause damage to the peripheral nervous system. Aspiration hazard if swallowed. Can enter lungs and cause damage. Use ventilation adequate to keep exposure below recommended limits, if any. Avoid breathing vapor or mist. Avoid contact with eyes, skin and clothing. Do not taste or swallow. Wash thoroughly after handling.

Physical Hazards/Precautionary Measures: Flammable liquid and vapor. Keep away from heat, sparks, flames, static electricity or other sources of ignition.

Appearance: Thicl light yellow to dark black

Physical form: Oily liquid

Odor: Petroleum hydrocarbon

NFPA Hazard Class:

Health: 2 (Moderate)

Flammability: 3 (High)

Reactivity: 0 (Least)

HMIS Hazard Class

Health: 2* (Moderate)

Flammability: 3 (High)

Physical Hazard: 0 (Least)

*Indicates possible chronic health effects.

2. COMPOSITION/INFORMATION ON INGREDIENTS

<u>HAZARDOUS COMPONENTS</u>	<u>% VOLUME</u>	<u>EXPOSURE GUIDELINE</u>		
		<u>Limits</u>	<u>Agency</u>	<u>Type</u>
Crude Oil (Petroleum) CAS# 8002-05-9	100	(See: Oil Mist, If Generated)		
Hydrogen Sulfide CAS# 7783-06-4	<5 ppm dissolved in liquid	10 ppm 15 ppm 20 ppm 100 ppm 50 ppm	ACGIH ACGIH OSHA NIOSH OSHA	TWA STEL CEIL IDLH 10 min. peak; once per 8-hr shift
n-Hexane CAS# 110-54-3	1-2.1	50 ppm 500 ppm 1100 ppm	ACGIH OSHA NIOSH	TWA-SKIN TWA IDLH
Toluene CAS# 108-88-3	1-1.5	50 ppm 200 ppm 300 ppm 500 ppm 500 ppm	ACGIH OSHA OSHA NIOSH OSHA	TWA-SKIN TWA CEIL IDLH 10 min. peak; once per 8-hr shift
Xylenes CAS# 1330-20-7	0.3-1.4	100 ppm 150 ppm 100 ppm 900 ppm	ACGIH ACGIH OSHA NIOSH	TWA STEL TWA IDLH
Benzene CAS# 71-43-2	<0.5	0.5 ppm 2.5 ppm 1 ppm 5 ppm 500 ppm	ACGIH ACGIH OSHA OSHA NIOSH	TWA-SKIN STEL-SKIN TWA STEL IDLH
Ethyl Benzene CAS# 100-41-4	<0.5	100 ppm 125 ppm 100 ppm 800 ppm	ACGIH ACGIH OSHA NIOSH	TWA STEL TWA IDLH

REFERENCEEXPOSURE GUIDELINE

	<u>Limits</u>	<u>Agency</u>	<u>Type</u>
Oil Mist, If Generated	5 mg/m ³	ACGIH	TWA
CAS# None	10 mg/m ³	ACGIH	STEL
	5 mg/m ³	OSHA	TWA
	2500 mg/m ³	NIOSH	IDLH
	5 mg/m ³	NOHSC	TWA

Note: State, local or other agencies or advisory groups may have established more stringent limits. Consult an industrial hygienist or similar professional, or your local agencies, for further information.

1%=10,000 PPM.

Contains benzene. If exposure concentrations exceed the 0.5 ppm action level, OSHA requirements for personal protective equipment, exposure monitoring, and training may apply (29CFR1910.1028). Also see Section 4.

Total sulfur approximately 1.0 to 1.8 wt%
Vanadium approximately 26 to 100 ppm

All components are listed on the TSCA inventory.

3. HAZARDS IDENTIFICATION

Potential Health Effects:

Eye: Contact may cause mild eye irritation including stinging, watering, and redness.

Skin: Contact may cause mild skin irritation including redness, and a burning sensation. Prolonged or repeated contact can worsen irritation by causing drying and cracking of the skin leading to dermatitis (inflammation). No harmful effects from skin absorption are expected.

Inhalation (Breathing): Low to moderate degree of toxicity by inhalation.

May contain or liberate hydrogen sulfide - see Other Comments section below.

Ingestion (Swallowing): Low degree of toxicity by ingestion. ASPIRATION HAZARD - This material can enter lungs during swallowing or vomiting and cause lung inflammation and damage.

Signs and Symptoms: Effects of overexposure may include irritation of the nose and throat, irritation of the digestive tract, nausea, vomiting, diarrhea and transient excitation followed by signs of nervous system depression (e.g., headache, drowsiness, dizziness, loss of coordination, disorientation and fatigue).

Cancer: Skin cancer hazard (see Sections 11 and 15).

Target Organs: Inadequate data available for this material.

Developmental: Potential hazard to the fetus (see Section 11).

Other Comments: This material may contain or liberate hydrogen sulfide, a poisonous gas with the smell of rotten eggs. The smell disappears rapidly because of olfactory fatigue so odor may not be a reliable indicator of exposure. Effects of overexposure include irritation of the eyes, nose, throat and respiratory tract, blurred vision, photophobia (sensitivity to light), and pulmonary edema (fluid accumulation in the lungs). Severe exposures can result in nausea, vomiting, muscle weakness or cramps, headache, disorientation and other signs of nervous system depression, irregular heartbeats, convulsions, respiratory failure, and death.

This material may contain polynuclear aromatic hydrocarbons (PNAs) which have been known to produce a phototoxic reaction when contaminated skin is exposed to sunlight. The effect is similar in appearance to an exaggerated sunburn, and is temporary in duration if exposure is discontinued. Continued exposure to sunlight can result in more serious skin problems including pigmentation (discoloration), skin eruptions (pimples), and possible skin cancers.

Pre-Existing Medical Conditions: Conditions aggravated by exposure may include skin disorders and respiratory (asthma-like) disorders.

4. FIRST AID MEASURES

Eye: If irritation or redness develops, move victim away from exposure and into fresh air. Flush eyes with clean water. If symptoms persist, seek medical attention.

Skin: Wipe material from skin and remove contaminated shoes and clothing. Cleanse affected area(s) thoroughly by washing with mild soap and water and, if necessary, a waterless skin cleanser. If irritation or redness develops and persists, seek medical attention.

Inhalation (Breathing): If respiratory symptoms or other symptoms of exposure develop, move victim away from source of exposure and into fresh air. If symptoms persist, seek immediate medical attention. If victim is not breathing, clear airway and immediately begin artificial respiration. If breathing difficulties develop, oxygen should be administered by qualified personnel. Seek immediate medical attention.

Ingestion (Swallowing): Aspiration hazard: Do not induce vomiting or give anything by mouth because this material can enter the lungs and cause severe lung damage. If victim is drowsy or unconscious and vomiting, place on the left side with the head down. If possible, do not leave victim unattended and observe closely for adequacy of breathing. Seek medical attention.

Note To Physicians: Acute aspirations of large amounts of oil-laden material may produce a serious aspiration pneumonia. Patients who aspirate these oils should be followed for the development of long-term sequelae. Inhalation exposure to oil mists below current workplace exposure limits is unlikely to cause pulmonary abnormalities.

Federal regulations (29 CFR 1910.1028) specify medical surveillance programs for certain exposures to benzene above the action level or PEL (specified in Section (i)(1)(i) of the Standard). In addition, employees exposed in an emergency situation shall, as described in Section (i)(4)(i), provide a urine sample at the end of the shift for measurement of urine phenol.

5. FIRE FIGHTING MEASURES

Flammable Properties: Flash Point: <24°F/<-4°C Estimated
OSHA Flammability Class: Flammable liquid
LEL%: 1.1 (ASTM E-681) / UEL%: 7.3
Autoignition Temperature: 720°F/382 (ASTM 659-78)

Unusual Fire & Explosion Hazards: This material is flammable and can be ignited by heat, sparks, flames, or other sources of ignition (e.g., static electricity, pilot lights, or mechanical/electrical equipment, and electronic devices such as cell phones, computers, calculators, and pagers which have not been certified as intrinsically safe). Vapors may travel considerable distances to a source of ignition where they can ignite, flash back, or explode. May create vapor/air explosion hazard indoors, in confined spaces, outdoors, or in sewers. Vapors are heavier than air and can accumulate in low areas. If container is not properly cooled, it can rupture in the heat of a fire.

Extinguishing Media: Dry chemical, carbon dioxide, or foam is recommended. Water spray is recommended to cool or protect exposed materials or structures. Carbon dioxide can displace oxygen. Use caution when applying carbon dioxide in confined spaces. Water may be ineffective for extinguishment, unless used under favorable conditions by experienced fire fighters.

Fire Fighting Instructions: Long-duration fires involving crude or residual fuel oil stored in tanks may result in a boilover. The contents of the tank may be expelled beyond the containment dikes or ditches. All personnel should be kept back a safe distance when a boilover is anticipated (reference NFPA 11 or API 2021).

For fires beyond the incipient stage, emergency responders in the immediate hazard area should wear bunker gear. When the potential chemical hazard is unknown, in enclosed or confined spaces, or when explicitly required by DOT, a self contained breathing apparatus should be worn. In addition, wear other appropriate protective equipment as conditions warrant (see Section 8).

Isolate immediate hazard area, keep unauthorized personnel out. Stop spill/release if it can be done with minimal risk. Move undamaged containers from immediate hazard area if it can be done with minimal risk.

Water spray may be useful in minimizing or dispersing vapors and to protect personnel. Cool equipment exposed to fire with water, if it can be done with minimal risk. Avoid spreading burning liquid with water used for cooling purposes.

6. ACCIDENTAL RELEASE MEASURES

Flammable. Keep all sources of ignition and hot metal surfaces away from spill/release. The use of

explosion-proof equipment is recommended.

Stay upwind and away from spill/release. Notify persons down wind of the spill/release, isolate immediate hazard area and keep unauthorized personnel out. Stop spill/release if it can be done with minimal risk. Wear appropriate protective equipment including respiratory protection as conditions warrant (see Section 8).

Prevent spilled material from entering sewers, storm drains, other unauthorized drainage systems, and natural waterways. Dike far ahead of spill for later recovery or disposal. Use foam on spills to minimize vapors (see Section 5). Spilled material may be absorbed into an appropriate absorbent material.

Notify fire authorities and appropriate federal, state, and local agencies. Immediate cleanup of any spill is recommended. If spill of any amount is made into or upon navigable waters, the contiguous zone, or adjoining shorelines, notify the National Response Center (phone number 800-424-8802).

7. HANDLING AND STORAGE

Handling: Open container slowly to relieve any pressure. Bond and ground all equipment when transferring from one vessel to another. Can accumulate static charge by flow or agitation. Can be ignited by static discharge. The use of explosion-proof equipment is recommended and may be required (see appropriate fire codes). Refer to NFPA-704 and/or API RP 2003 for specific bonding/grounding requirements.

Do not enter confined spaces such as tanks or pits without following proper entry procedures such as ASTM D-4276 and 29CFR 1910.146. The use of appropriate respiratory protection is advised when concentrations exceed any established exposure limits (see Sections 2 and 8).

Do not wear contaminated clothing or shoes. Keep contaminated clothing away from sources of ignition such as sparks or open flames. Use good personal hygiene practices.

Before working on or in tanks which contain or have contained this material, refer to OSHA regulations, ANSI Z49.1, and other references pertaining to cleaning, repairing, welding, or other contemplated operations.

Storage: Keep container(s) tightly closed. Use and store this material in cool, dry, well-ventilated areas away from heat, direct sunlight, hot metal surfaces, and all sources of ignition. Post area "No Smoking or Open Flame." Store only in approved containers. Keep away from any incompatible material (see Section 10). Protect container(s) against physical damage. Outdoor or detached storage is preferred. Indoor storage should meet OSHA standards and appropriate fire codes.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Engineering controls: If current ventilation practices are not adequate to maintain airborne concentrations below the established exposure limits (see Section 2), additional engineering controls may be required. Where explosive mixtures may be present, electrical systems safe for such locations must be used (see appropriate electrical codes).

Personal Protective Equipment (PPE):

Respiratory: A NIOSH certified air purifying respirator with an organic vapor cartridge may be used under conditions where airborne concentrations are expected to exceed exposure limits (see Section 2).

Protection provided by air purifying respirators is limited (see manufacturer's respirator selection guide). Use a NIOSH approved self-contained breathing apparatus (SCBA) or equivalent operated in a pressure demand or other positive pressure mode if there is potential for an uncontrolled release, exposure levels are not known, or any other circumstances where air purifying respirators may not provide adequate protection.

A respiratory protection program that meets OSHA's 29 CFR 1910.134 and ANSI Z88.2 requirements must be followed whenever workplace conditions warrant a respirator's use.

When benzene values equal or exceed applicable exposure limits the use of respiratory protection should comply with the requirements in OSHA 29 CFR 1910.1028-Benzene.

Skin: The use of gloves impervious to the specific material handled is advised to prevent skin contact, possible irritation, absorption, and skin damage (see glove manufacturer literature for information on permeability).

Use nitrile, PVA, or Viton® gloves to prevent skin contact. Use Viton® gloves for extended use (more than 4 hours) or immersion. Depending on conditions of use, apron and/or arm covers may be necessary.

Eye/Face: Approved eye protection to safeguard against potential eye contact, irritation, or injury is recommended. Depending on conditions of use, a face shield over safety glasses or goggles may be necessary.

Other Protective Equipment: Eye wash and quick-drench shower facilities should be available in the work area. Thoroughly clean shoes and wash contaminated clothing before reuse. It is recommended that impervious clothing be worn when skin contact is possible.

Full body clothing may include CPF2, CPF3, or CPF4 or Saranex® coated Tyvek®, depending on the task.

Suggestions for the use of specific protective materials are based on readily available published data. Users should check with specific manufacturers to confirm the performance of their products.

9. PHYSICAL AND CHEMICAL PROPERTIES

Note: Unless otherwise stated, values are determined at 20°C (68°F) and 760 mm Hg (1 atm).

Appearance: Thick light yellow to dark black

Physical State: Oily liquid

Odor: Petroleum hydrocarbon

pH: Not applicable

Vapor Pressure (mm Hg): 2.6-6.2 lbs. (Reid psi) @100°F

Vapor Density (air=1): >1

Boiling Point/Range: -54 to 1100°F / -18 to 593°C

Freezing/Melting Point: No Data

Solubility in Water: Negligible

Specific Gravity: 0.8939

Percent Volatile: 1-50

Evaporation Rate (nBuAc=1): <1

Bulk Density: 6.9 lbs/gal

Flash Point: <24°F / <-4°C Estimated

Flammable/Explosive Limits (%): LEL: 1.1 (ASTM E-681) / UEL: 7.3

10. STABILITY AND REACTIVITY

Stability: Stable under normal ambient and anticipated storage and handling conditions of temperature and pressure. Flammable liquid and vapor. Vapor can cause flash fire.

Conditions To Avoid: Avoid all possible sources of ignition (see Sections 5 and 7).

Materials to Avoid (Incompatible Materials): Avoid contact with nitric acid and strong oxidizing agents.

Hazardous Decomposition Products: Combustion can yield carbon, nitrogen and sulfur oxides and other organic compounds.

Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Crude Oil (Petroleum) (CAS# 8002-05-9)

Carcinogenicity: Chronic application of crude oil to mouse skin resulted in an increased incidence of skin tumors. IARC concluded in its Crude Oil Monograph that there is limited evidence of carcinogenicity in animals, and that crude oil is not classifiable as to its carcinogenicity in humans (Group 3). It has not been listed as a carcinogen by NTP or OSHA.

Developmental: Dermal exposure to crude oil during pregnancy resulted in limited evidence of developmental toxicity in laboratory animals. Decreased fetal weight and increased resorptions were noted at maternally toxic doses. No significant effects on pup growth or other developmental landmarks were observed postnatally.

n-Hexane (CAS# 110-54-3)

Target Organ(s): Excessive exposure to n-hexane can result in peripheral neuropathies. The initial symptoms are symmetrical sensory numbness and paresthesias of distal portions of the extremities. Motor weakness is typically observed in muscles of the toes and fingers but may also involve muscles of the arms, thighs and forearms. The onset of these symptoms may be delayed for several months to a year after the beginning of exposure. The neurotoxic properties of n-hexane are potentiated by exposure to methyl ethyl ketone and methyl isobutyl ketone.

Prolonged exposure to high concentrations of n-hexane (>1,000 ppm) has resulted in decreased sperm count and degenerative changes in the testes of rats but not those of mice.

Toluene (CAS# 108-88-3)

Target Organ(s): Epidemiology studies suggest that chronic occupational overexposure to toluene may damage color vision. Subchronic and chronic inhalation studies with toluene produced kidney and liver damage, hearing loss and central nervous system (brain) damage in laboratory animals. Intentional misuse by deliberate inhalation of high concentrations of toluene has been shown to cause liver, kidney, and central nervous system damage, including hearing loss and visual disturbances.

Developmental: Exposure to toluene during pregnancy has demonstrated limited evidence of developmental toxicity in laboratory animals. The effects seen include decreased fetal body weight and increased skeletal variations in both inhalation and oral studies.

Xylenes (CAS# 1330-20-7)

Target Organ(s): A six week inhalation study with xylene produced hearing loss in rats.

Developmental: Both mixed xylenes and the individual isomers produced limited evidence of developmental toxicity in laboratory animals. Inhalation and oral administration of xylene resulted in decreased fetal weight, increased incidences of delayed ossification, skeletal variations and resorptions.

Benzene (CAS# 71-43-2)

Carcinogenicity: Benzene is an animal carcinogen and is known to produce acute myelogenous leukemia (a form of cancer) in humans. Benzene has been identified as a human carcinogen by NTP, IARC and OSHA.

Ethyl Benzene (CAS# 100-41-4)

Carcinogenicity: Rats and mice exposed to 0, 75, 250, or 750 ppm ethyl benzene in a two year inhalation study demonstrated limited evidence of kidney, liver, and lung cancer. Ethyl benzene has been listed as a possible human carcinogen by IARC. Ethyl benzene has not been listed as a carcinogen by NTP, or OSHA.

12. ECOLOGICAL INFORMATION

Environmental Fate: No Data Found

Ecotoxicity:

Aquatic toxicity:

90 ppm/24hr/juvenile American shad/TLm/fresh water

91 ppm/24hr/juvenile American shad/Tm/salt water

3 ppm/time period not specified/fresh water fish/toxic/fresh water

200 ppm/24 hr/coral sporites/20-90% 8.1 normal response/salt water

Biochemical Oxygen Demand (BOD): 8%, 5 days

13. DISPOSAL CONSIDERATIONS

Maximize recovery for reuse or recycling.

This material, if discarded as produced, would be a RCRA "characteristic" hazardous waste due to the characteristic(s) of ignitability (D001), benzene (D018). If the spilled or released material impacts soil, water, or other media, characteristic testing of the contaminated materials may be required prior to their disposal. Further, this material, once it becomes a waste, is subject to the land disposal restrictions in 40 CFR 268.40 and may require treatment prior to disposal to meet specific standards. Consult state and local regulations to determine whether they are more stringent than the federal requirements.

Container contents should be completely used and containers should be emptied prior to discard. Container rinsate could be considered a RCRA hazardous waste and must be disposed of with care and in full compliance with federal, state and local regulations. Larger empty containers, such as drums, should be returned to the distributor or to a drum reconditioner. To assure proper disposal of smaller empty containers, consult with state and local regulations and disposal authorities.

14. TRANSPORT INFORMATION

DOT Shipping Description: Petroleum Crude Oil,3,UN1267,II

Non-Bulk Package Marking: Petroleum Crude Oil, UN 1267

Non-Bulk Package Label: Flammable

Bulk Package Placard/Marking: Flammable/1267

Hazardous Substance/RQ None

Packaging References 49 CFR 173.150, 202, 242

Emergency Response Guide: 128

15. REGULATORY INFORMATION

EPA SARA 311/312 (Title III Hazard Categories):

Acute Health: Yes

Chronic Health: Yes

Fire Hazard: Yes

Pressure Hazard: No

Reactive Hazard: No

SARA 313 and 40 CFR 372:

This material contains the following chemicals subject to the reporting requirements of SARA 313 and 40 CFR 372:

Component	CAS Number	Weight %
Hydrogen Sulfide	7783-06-4	<5 ppm dissolved in liquid
n-Hexane	110-54-3	0.8-1.6
Toluene	108-88-3	1-1.5
Xylenes	1330-20-7	0.3-1.4
Benzene	71-43-2	<0.5
Ethyl Benzene	100-41-4	<0.5

California Proposition 65:

Warning: This material contains the following chemicals which are known to the State of California to cause cancer, birth defects or other reproductive harm, and are subject to the requirements of California Proposition 65 (CA Health & Safety Code Section 25249.5):

Component	Effect
Benzene	Cancer, Developmental and Reproductive Toxicant
Various Polycyclic Aromatic Hydrocarbons	Skin Cancer
Toluene	Developmental Toxicant

Carcinogen Identification:

This material has not been identified as a carcinogen by NTP, IARC, or OSHA. See Section 11 for carcinogenicity information of individual components, if any.

EPA (CERCLA) Reportable Quantity:

--None--

Canada - Domestic Substances List: Listed**WHMIS Class:**

B2-Flammable Liquid
 D1A-Materials causing immediate and serious toxic effects - Very Toxic Material
 D2B-Materials causing other toxic effects - Toxic Material

This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all the information required by the CPR.

16. OTHER INFORMATION

Issue Date: 02/28/03

Previous Issue Date: 04/26/01

Revised Sections: New Format

MSDS Number: 791002

Status: Final

Disclaimer of Expressed and Implied Warranties:

The information presented in this Material Safety Data Sheet is based on data believed to be accurate as of the date this Material Safety Data Sheet was prepared. **HOWEVER, NO WARRANTY OF MERCHANTABILITY, FITNESS FOR ANY PARTICULAR PURPOSE, OR ANY OTHER WARRANTY IS EXPRESSED OR IS TO BE IMPLIED REGARDING THE ACCURACY OR COMPLETENESS OF THE INFORMATION PROVIDED ABOVE, THE RESULTS TO BE OBTAINED FROM THE USE OF THIS INFORMATION OR THE PRODUCT, THE SAFETY OF THIS PRODUCT, OR THE HAZARDS RELATED TO ITS USE.** No responsibility is assumed for any damage or injury resulting from abnormal use or from any failure to adhere to recommended practices. The information provided above, and the product, are furnished on the condition that the person receiving them shall make their own determination as to the suitability of the product for their particular purpose and on the condition that they assume the risk of their use. In addition, no authorization is given nor implied to practice any patented invention without a license.

MATERIAL SAFETY DATA

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product name: Flomin F 660 Frother
Chemical Name: Polyethylene glycol monobutyl ether mixture
Synonyms: None
Molecular Formula: Mixture
Molecular Weight: Mixture
Manufacturer: Flomin, Inc.
7500 FM 1405
Baytown, TX 77523
Emergency Phones: 800-424-9300 CHEMTREC (North America)
+1-703-527-3887 CHEMTREC (Worldwide)
912-884-3366 (Flomin Inc., 24-Hour)

2. HAZARDS IDENTIFICATION

Product vapors or mist may be irritating to eyes and respiratory system. Product liquid may cause eye and skin burns. Harmful if swallowed.

POTENTIAL HEALTH EFFECTS: (See Section 11 for toxicological data.)

Eye: Eye irritant. May cause tearing, blurring of vision and corneal damage.

Skin: Skin irritant. Effects of skin contacts may include rash, burns and ulcerations.

Ingestion: Ingestion can cause irritation or burns of the mouth, throat, esophagus, and stomach with nausea and vomiting. Central nervous system effects may include headaches, weakness, and nausea.

Inhalation: Inhalation of vapors is irritating to the respiratory system, may cause throat pain and cough. Central nervous system effects may include headaches, weakness, and nausea.

3. COMPOSITION / INFORMATION ON INGREDIENTS

<u>Component</u>	<u>CAS #</u>	<u>Weight, %</u>	<u>Exposure Limit</u>
Triethylene glycol monobutyl ether	143-22-6	70 - 80	Not established
Tetraethylene glycol monobutyl ether	1559-34-8	15 - 25	Not established
Diethylene glycol monobutyl ether	112-34-5	<5	Not established

4. FIRST AID MEASURES

- Inhalation:** Move to fresh air immediately. Artificial respiration and/or oxygen may be necessary. Seek medical attention if irritation persists.
- Skin contact:** Remove soaked clothing immediately and wash affected skin with soap and water. Get medical treatment for burns and persistent irritation. Launder contaminated clothing before reuse.
- Eye contact:** Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician. Keep eye wide open while rinsing and lift upper and lower lids to ensure complete removal of chemical.
- Ingestion:** Do not induce vomiting. Call a physician immediately.

5. FIRE FIGHTING MEASURES**Flammable properties:**

- **Flash point** >200°F (>93°C), PM CC
- **Flammable limits** 0.8 - 3.8 vol%
- **Autoignition temp.** 397°F (203°C)
- **Decomposition temp.** Not determined

Extinguishing media: Use water spray, carbon dioxide, or dry chemical.

Fire fighting precautions: Avoid contact with liquid or mist. Keep personnel removed and upwind of fire.

Protective equipment: Wear NIOSH approved, positive pressure, self-contained respirator. Wear full protective equipment for body and eyes.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions: Wear full protective equipment for eyes, body and respiratory system. Ventilate spill area. Remove all sources of ignition.

Clean up methods: Dike spill and collect for disposal or reuse. Use adsorbents on residual material. Flush spill area with water.

Environmental: Keep flush material out of waterways. Dispose of cleanup material in an approved manner.

7. HANDLING AND STORAGE

Handling: Wear protective equipment for eyes, body and respiratory system.

Storage: Store in closed containers in a cool area away from sources of heat or ignition. Store this product in steel, stainless steel, polyethylene, or polypropylene containers.

8. EXPOSURE CONTROLS / PERSONAL PROTECTION**Personal protection equipment:**

- **Respiratory:** Wear a properly fitted NIOSH/MSA approved respirator whenever significant exposure to vapor or mist is likely.

- **Hand:** Neoprene, polyvinyl, butyl rubber or nitrile rubber gloves are suitable.

- **Eye:** Wear chemical splash proof goggles or face shield.

- **Skin:** Wear coveralls and/or chemical apron and rubber footwear where physical contact can occur.

Hygiene: Wash hands before breaks and immediately after handling the product. Do not eat or store food and drinks where this product is used.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance:	Colorless liquid
Odor:	Slight odor typical of glycol ethers
Boiling point:	532°F (278°C)
Freezing point:	Not determined
Vapor pressure:	Not determined
Specific gravity:	~0.95 @ 68°F (20°C)
Vapor density:	Not determined (Air =1)
Flash point:	>200°F (>93°C), PM CC
pH:	Not applicable
Water solubility:	Complete
Viscosity:	Not determined

10. STABILITY AND REACTIVITY

Stability: Stable. No hazardous polymerization will occur.

Conditions to avoid: High temperatures and ignition sources. Avoid strong acids, strong bases and oxidizing agents.

Hazardous decomposition

Products: Carbon monoxide, carbon dioxide and hydrocarbons may form under fire conditions.

11. TOXICOLOGICAL INFORMATION**Acute toxicity:**

- **Oral:** Acute oral LD₅₀ (rat) = >5000 mg/kg
- **Dermal:** Acute dermal LD₅₀ (rabbit) = 3480 mg/kg
- **Inhalation:** LC₅₀ (1 hr, rat) = N/A

Irritation:

- **Skin:** Moderate skin irritant.
- **Eyes:** Moderate eye irritant.

Sensitization: May sensitize some individuals toward asthmatic conditions and allergic skin reactions

12. ECOLOGICAL INFORMATION

Aquatic toxicity: LC₅₀ (Leuciscus Idus, 96h) = 2,200 mg/l
EC₅₀ (Daphnia Magna, 48h) = >500 mg/l
EC₅₀ (Scenedesmus Subspictus, 72h) = >500 mg/l

Biodegradation: Product is readily biodegradable.

13. DISPOSAL CONSIDERATIONS

Disposal: Dispose of only in accordance with regulations. Do not contaminate any lakes, streams, ponds, groundwater or soil.

Containers: Empty containers retain product residues (liquid and vapor) and can be dangerous. Do not cut or burn or expose empty containers to heat, flame or sources of ignition. Empty containers should be completely drained and disposed in accordance with regulations.

14. TRANSPORT INFORMATION

Agency **UN#, Proper shipping name, Hazard class, Packing group, Advisory placard**

DOT: This product is not regulated as a hazardous material as defined by the DOT.

IMO: This product is not regulated as a dangerous good as defined by the IMDG Code for marine transport.

ICAO/IATA: This product is not regulated as a dangerous good as defined by the ICAO/IATA for air transport.

Canada: This product is not regulated as a dangerous good as defined by the WHIMS classifications.

15. REGULATORY INFORMATION

Components of this product are listed on the TSCA (US), DSL (Canada), and EINECS (Europe) inventories.

SARA 302: Contains no chemicals subject to 40 CFR 302 reporting.

SARA 311/312: Not hazardous.

SARA 313: Contains no chemicals subject to 40 CFR 313 reporting.

16. OTHER INFORMATION

Hazard Ratings:

	<u>Health</u>	<u>Flammability</u>	<u>Instability</u>	<u>Physical Hazard</u>
- NFPA	1	1	0	---
-HMIS	1	1	---	0

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SAFETY DATA SHEET

1. Identification

Product identifier CustoFroth 527

Other means of identification

Product Code ZF74527

Recommended use Frother.

Recommended restrictions None known.

Manufacturer/Importer/Supplier/Distributor information

Manufacturer

Company name ArrMaz
Address 4800 State Road 60 East
Mulberry, FL 33860
Telephone EHS Contact Phone Number 863.578.1221
(US):
E-mail MSDSinfo@arrmaz.com
Emergency phone number CANUTEC (Canadian (613) 996-6666
Transport.):
CHEMTREC (US (800) 424-9300
Transportation):
ArrMaz QA 24-hr Number (863) 578-1206
(US):

2. Hazard(s) identification

Physical hazards Not classified.

Health hazards Acute toxicity, oral Category 4
Skin corrosion/irritation Category 2
Serious eye damage/eye irritation Category 1

Environmental hazards Hazardous to the aquatic environment, long-term hazard Category 3

OSHA defined hazards Not classified.

Label elements



Signal word Danger

Hazard statement Harmful if swallowed. Causes skin irritation. Causes serious eye damage. Harmful to aquatic life with long lasting effects.

Precautionary statement

Prevention Wash thoroughly after handling. Do not eat, drink or smoke when using this product. Avoid release to the environment. Wear eye protection/face protection. Wear protective gloves.

Response If swallowed: Call a poison center/doctor if you feel unwell. If on skin: Wash with plenty of water. If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Immediately call a poison center/doctor. Rinse mouth. If skin irritation occurs: Get medical advice/attention. Take off contaminated clothing and wash before reuse.

Storage Store away from incompatible materials.

Disposal Dispose of contents/container in accordance with local/regional/national/international regulations.

Hazard(s) not otherwise classified (HNOC) None known.

Supplemental information 50.03% of the mixture consists of component(s) of unknown acute oral toxicity. 61.42% of the mixture consists of component(s) of unknown long-term hazards to the aquatic environment.

3. Composition/information on ingredients

Mixtures

Chemical name	Common name and synonyms	CAS number	%
Blend of Alcohols and Polyglycol Ethers		Proprietary	95 - 100

*Designates that a specific chemical identity and/or percentage of composition has been withheld as a trade secret.

4. First-aid measures

Inhalation	Move to fresh air. Call a physician if symptoms develop or persist.
Skin contact	Remove contaminated clothing. Wash with plenty of soap and water. If skin irritation occurs: Get medical advice/attention. Wash contaminated clothing before reuse.
Eye contact	Immediately flush eyes with plenty of water for at least 15 minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Get medical attention immediately.
Ingestion	Rinse mouth. If vomiting occurs, keep head low so that stomach content doesn't get into the lungs. Get medical advice/attention if you feel unwell.
Most important symptoms/effects, acute and delayed	Severe eye irritation. Symptoms may include stinging, tearing, redness, swelling, and blurred vision. Permanent eye damage including blindness could result. Skin irritation. May cause redness and pain.
Indication of immediate medical attention and special treatment needed	Provide general supportive measures and treat symptomatically. Keep victim warm. Keep victim under observation. Symptoms may be delayed.
General information	Ensure that medical personnel are aware of the material(s) involved, and take precautions to protect themselves. Show this safety data sheet to the doctor in attendance.

5. Fire-fighting measures

Suitable extinguishing media	Water fog. Foam. Dry chemical powder. Carbon dioxide (CO ₂).
Unsuitable extinguishing media	Do not use water jet as an extinguisher, as this will spread the fire.
Specific hazards arising from the chemical	During fire, gases hazardous to health may be formed.
Special protective equipment and precautions for firefighters	Self-contained breathing apparatus and full protective clothing must be worn in case of fire.
Fire-fighting equipment/instructions	Move containers from fire area if you can do so without risk.
Specific methods	Use standard firefighting procedures and consider the hazards of other involved materials.
Unusual fire & explosion hazards	None known.

6. Accidental release measures

Personal precautions, protective equipment and emergency procedures	Keep unnecessary personnel away. Keep people away from and upwind of spill/leak. Wear appropriate protective equipment and clothing during clean-up. Do not touch damaged containers or spilled material unless wearing appropriate protective clothing. Ensure adequate ventilation. Local authorities should be advised if significant spillages cannot be contained. For personal protection, see section 8 of the SDS.
Methods and materials for containment and cleaning up	Prevent product from entering drains. Large Spills: Stop the flow of material, if this is without risk. Dike the spilled material, where this is possible. Cover with plastic sheet to prevent spreading. Absorb in vermiculite, dry sand or earth and place into containers. Following product recovery, flush area with water. Small Spills: Wipe up with absorbent material (e.g. cloth, fleece). Clean surface thoroughly to remove residual contamination.
Environmental precautions	Never return spills to original containers for re-use. For waste disposal, see section 13 of the SDS. Avoid release to the environment. Inform appropriate managerial or supervisory personnel of all environmental releases. Prevent further leakage or spillage if safe to do so. Avoid discharge into drains, water courses or onto the ground.

7. Handling and storage

Precautions for safe handling

Do not get this material in contact with eyes. Do not taste or swallow. Avoid contact with eyes, skin, and clothing. Avoid prolonged exposure. When using, do not eat, drink or smoke. Provide adequate ventilation. Wear appropriate personal protective equipment. Wash hands thoroughly after handling. Avoid release to the environment. Observe good industrial hygiene practices.

Conditions for safe storage, including any incompatibilities

Store in original tightly closed container. Store away from incompatible materials (see Section 10 of the SDS).

8. Exposure controls/personal protection

Occupational exposure limits

No exposure limits noted for ingredient(s).

Biological limit values

No biological exposure limits noted for the ingredient(s).

Appropriate engineering controls

Good general ventilation (typically 10 air changes per hour) should be used. Ventilation rates should be matched to conditions. If applicable, use process enclosures, local exhaust ventilation, or other engineering controls to maintain airborne levels below recommended exposure limits. If exposure limits have not been established, maintain airborne levels to an acceptable level. Eye wash facilities and emergency shower must be available when handling this product.

Individual protection measures, such as personal protective equipment

Eye/face protection

Do not get in eyes. Wear eye/face protection. Chemical goggles and face shield are recommended. Provide an emergency eye wash fountain and quick drench shower in the immediate work area.

Skin protection

Hand protection

Wear appropriate chemical resistant gloves. The choice of an appropriate glove does not only depend on its material but also on other quality features and is different from one producer to the other. Nitrile gloves are recommended.

Other

Wear appropriate chemical resistant clothing.

Respiratory protection

In case of insufficient ventilation, wear suitable respiratory equipment. If engineering controls do not maintain airborne concentrations below recommended exposure limits (where applicable) or to an acceptable level (in countries where exposure limits have not been established), an approved respirator must be worn.

Thermal hazards

Wear appropriate thermal protective clothing, when necessary.

General hygiene considerations

Keep away from food and drink. 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.

9. Physical and chemical properties

Appearance

Amber liquid.

Physical state

Liquid.

Form

Liquid.

Color

Light brown to brown.

Odor

Mild, characteristic.

Odor threshold

Not available.

pH

< 11

Melting point/freezing point

Not available.

Initial boiling point and boiling range

Not available.

Flash point

> 220 °F (> 104 °C)

Evaporation rate

Not available.

Flammability (solid, gas)

Not applicable.

Upper/lower flammability or explosive limits

Flammability limit - lower (%)

Not available.

Flammability limit - upper (%)

Not available.

Explosive limit - lower (%)

Not available.

Explosive limit - upper (%)

Not available.

Vapor pressure

Not available.

Vapor density	Not available.
Relative density	Not available.
Solubility(ies)	Soluble in water.
Partition coefficient (n-octanol/water)	Not available.
Auto-ignition temperature	Not available.
Decomposition temperature	Not available.
Viscosity	< 200 cP
Viscosity temperature	77 °F (25 °C)

Other information

Density	8.07 lb/gal
Density temperature	77 °F (25 °C)
Explosive properties	Not explosive.
Flammability class	Combustible IIIB estimated
Oxidizing properties	Not oxidizing.
Specific gravity	0.97
Specific gravity temperature	77 °F (25 °C)

10. Stability and reactivity

Reactivity	The product is stable and non-reactive under normal conditions of use, storage and transport.
Chemical stability	Material is stable under normal conditions.
Possibility of hazardous reactions	No dangerous reaction known under conditions of normal use.
Conditions to avoid	Contact with incompatible materials.
Incompatible materials	Strong oxidizing agents.
Hazardous decomposition products	Carbon oxides. Sulfur oxides. Sodium oxides. Irritating and/or toxic fumes and gases may be emitted upon the products decomposition.

11. Toxicological information

Information on likely routes of exposure

Inhalation	Prolonged inhalation may be harmful.
Skin contact	Causes skin irritation.
Eye contact	Causes serious eye damage.
Ingestion	Harmful if swallowed.

Symptoms related to the physical, chemical and toxicological characteristics	Severe eye irritation. Symptoms may include stinging, tearing, redness, swelling, and blurred vision. Permanent eye damage including blindness could result. Skin irritation. May cause redness and pain.
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Information on toxicological effects

Acute toxicity	Harmful if swallowed.
Skin corrosion/irritation	Causes skin irritation.
Serious eye damage/eye irritation	Causes serious eye damage.

Respiratory or skin sensitization

Respiratory sensitization	Not classified.
Skin sensitization	This product is not expected to cause skin sensitization.

Germ cell mutagenicity	No data available to indicate product or any components present at greater than 0.1% are mutagenic or genotoxic.
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Carcinogenicity	This product is not considered to be a carcinogen by IARC, ACGIH, NTP, or OSHA.
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IARC Monographs. Overall Evaluation of Carcinogenicity

Not listed.

OSHA Specifically Regulated Substances (29 CFR 1910.1001-1050)

Not regulated.

US. National Toxicology Program (NTP) Report on Carcinogens

Not listed.

Reproductive toxicity	This product is not expected to cause reproductive or developmental effects.
Specific target organ toxicity - single exposure	Not classified.
Specific target organ toxicity - repeated exposure	Not classified.
Aspiration hazard	Not classified. May be harmful if swallowed and enters airways.
Chronic effects	Prolonged inhalation may be harmful.

12. Ecological information

Ecotoxicity Harmful to aquatic life with long lasting effects.

Components	Species	Test Results
Alcohols, C10-16, Ethoxylated, Sulfates, Sodium Salts (CAS 68585-34-2)		
Aquatic		
Crustacea	EC50 Water flea (Ceriodaphnia dubia)	2.33 - 4.81 mg/l, 48 hours

* Estimates for product may be based on additional component data not shown.

Persistence and degradability	No data is available on the degradability of this product.
Bioaccumulative potential	No data available.
Mobility in soil	No data available.
Other adverse effects	No other adverse environmental effects (e.g. ozone depletion, photochemical ozone creation potential, endocrine disruption, global warming potential) are expected from this component.

13. Disposal considerations

Disposal instructions	Collect and reclaim or dispose in sealed containers at licensed waste disposal site. Do not allow this material to drain into sewers/water supplies. Do not contaminate ponds, waterways or ditches with chemical or used container. Dispose of contents/container in accordance with local/regional/national/international regulations.
Local disposal regulations	Dispose in accordance with all applicable regulations.
Hazardous waste code	The waste code should be assigned in discussion between the user, the producer and the waste disposal company.
Waste from residues / unused products	Dispose of in accordance with local regulations. Empty containers or liners may retain some product residues. This material and its container must be disposed of in a safe manner (see: Disposal instructions).
Contaminated packaging	Since emptied containers may retain product residue, follow label warnings even after container is emptied. Empty containers should be taken to an approved waste handling site for recycling or disposal.

14. Transport information

DOT

Not regulated as dangerous goods.

IATA

Not regulated as dangerous goods.

IMDG

Not regulated as dangerous goods.

Transport in bulk according to Annex II of MARPOL 73/78 and the IBC Code Not established.

15. Regulatory information

US federal regulations This product is a "Hazardous Chemical" as defined by the OSHA Hazard Communication Standard, 29 CFR 1910.1200.

TSCA Section 12(b) Export Notification (40 CFR 707, Subpt. D)

Not regulated.

CERCLA Hazardous Substance List (40 CFR 302.4)

Not listed.

Country(s) or region	Inventory name	On inventory (yes/no)*
New Zealand	New Zealand Inventory	Yes
Philippines	Philippine Inventory of Chemicals and Chemical Substances (PICCS)	Yes
United States & Puerto Rico	Toxic Substances Control Act (TSCA) Inventory	Yes

*A "Yes" indicates that all components of this product comply with the inventory requirements administered by the governing country(s)

A "No" indicates that one or more components of the product are not listed or exempt from listing on the inventory administered by the governing country(s).

16. Other information, including date of preparation or last revision

Issue date 11-05-2015

Version # 01

HMIS® ratings
 Health: 3
 Flammability: 1
 Physical hazard: 0
 Personal protection: X

NFPA ratings
 Health: 3
 Flammability: 1
 Instability: 0

Disclaimer ArrMaz cannot anticipate all conditions under which this information and its product, or the products of other manufacturers in combination with its product, may be used. It is the user's responsibility to ensure safe conditions for handling, storage and disposal of the product, and to assume liability for loss, injury, damage or expense due to improper use. The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.



Safety Data Sheet

The Dow Chemical Company

Product Name: ROMAX(TM) Fit for Use Water Clarifier Kit

Issue Date: 09/21/2015

Print Date: 21 Sep 2015

RESEARCH SAMPLE.

The Dow Chemical Company encourages and expects you to read and understand the entire (M)SDS, as there is important information throughout the document. We expect you to follow the precautions identified in this document unless your use conditions would necessitate other appropriate methods or actions.

1. Identification

Product Name

ROMAX™ Fit for Use Water Clarifier Kit

Identified uses

Research sample.

COMPANY IDENTIFICATION

The Dow Chemical Company
2030 Willard H. Dow Center
Midland, MI 48674
United States

Customer Information Number:

800-258-2436

SDSQuestion@dow.com

EMERGENCY TELEPHONE NUMBER

24-Hour Emergency Contact:

989-636-4400

Local Emergency Contact:

989-636-4400

2. Hazards Identification

Classification of the substance or mixture This material is hazardous under the criteria of the Federal OSHA Hazard Communication Standard 29CFR 1910.1200.

Hazard Class:

Acute toxicity (Oral)	Category 4
Specific target organ toxicity - repeated exposure (Oral)	Category 2

Label elements

Hazard Symbol:

®(TM)*Trademark



Signal Word: Warning

Hazards of product:

Harmful if swallowed.

Causes skin irritation.

Causes serious eye irritation.

May cause damage to organs through prolonged or repeated exposure.

Precautionary Statements:

Prevention: Wash skin thoroughly after handling. Do not eat, drink or smoke when using this product. Do not breathe dust or mist.

Response: IF SWALLOWED: Call a POISON CENTER or doctor/ physician if you feel unwell. Rinse mouth. Get medical advice/ attention if you feel unwell.

Disposal: Dispose of contents/container in accordance with applicable regulations.

3. Composition Information

This product is a mixture.

Component	CAS #	Amount
Acrylic polymer(s)	Not Available	>= 0.0 - <= 50.0 %
Ethylene glycol	107-21-1	>= 0.0 - <= 50.0 %
Water	7732-18-5	>= 0.0 - < 99.5 %
Unknown Component(s); based on supplier information provided	Not available	>= 0.0 - <= 12.0 %
Polyethylene glycol	25322-68-3	>= 0.0 - <= 30.0 %
Propylene glycol	57-55-6	>= 0.0 - <= 50.0 %
Ethylene glycol monobutyl ether	111-76-2	>= 0.0 - <= 10.0 %
Diethylene glycol monobutyl ether	112-34-5	>= 0.0 - <= 5.0 %
L-Proline, 5-oxo-, compd. with chitosan	117522-93-7	>= 0.0 - <= 5.0 %
Cationic hydroxyethyl cellulose	68610-92-4	>= 0.0 - <= 5.0 %

4. First-aid measures

Description of first aid measures

General advice: First Aid responders should pay attention to self-protection and use the recommended protective clothing (chemical resistant gloves, splash protection). If potential for exposure exists refer to Section 8 for specific personal protective equipment.

Inhalation: Move person to fresh air; if effects occur, consult a physician.

Skin Contact: Immediately flush skin with water while removing contaminated clothing and shoes. Get medical attention if symptoms occur. Wash clothing before reuse. Contaminated leather items such as shoes should be disposed of properly. Suitable emergency safety shower facility should be immediately available.

Eye Contact: Flush eyes thoroughly with water for several minutes. Remove contact lenses after the initial 1-2 minutes and continue flushing for several additional minutes. If effects occur, consult a physician, preferably an ophthalmologist. Suitable emergency eye wash facility should be available in work area.

Ingestion: Do not induce vomiting. Seek medical attention immediately. If person is fully conscious give 1 cup or 8 ounces (240 ml) of water. If medical advice is delayed and if an adult has swallowed

several ounces of chemical, then give 3-4 ounces (1/3-1/2 Cup) (90-120 ml) of hard liquor such as 80 proof whiskey. For children, give proportionally less liquor at a dose of 0.3 ounce (1 1/2 tsp.) (8 ml) liquor for each 10 pounds of body weight, or 2 ml per kg body weight [e.g., 1.2 ounce (2 1/3 tbsp.) for a 40 pound child or 36 ml for an 18 kg child].

Most important symptoms and effects, both acute and delayed

Aside from the information found under Description of first aid measures (above) and Indication of immediate medical attention and special treatment needed (below), any additional important symptoms and effects are described in Section 11: Toxicology Information.

Indication of immediate medical attention and special treatment needed

If several ounces (60 - 100 ml) of ethylene glycol have been ingested, early administration of ethanol may counter the toxic effects (metabolic acidosis, renal damage). Consider hemodialysis or peritoneal dialysis & thiamine 100 mg plus pyridoxine 50 mg intravenously every 6 hours. If ethanol is used, a therapeutically effective blood concentration in the range of 100 - 150 mg/dl may be achieved by a rapid loading dose followed by a continuous intravenous infusion. Consult standard literature for details of treatment. 4-Methyl pyrazole (Antizol®) is an effective blocker of alcohol dehydrogenase and should be used in the treatment of ethylene glycol (EG), di- or triethylene glycol (DEG, TEG), ethylene glycol butyl ether (EGBE), or methanol intoxication if available. Fomepizole protocol (Brent, J. et al., New England Journal of Medicine, Feb. 8, 2001, 344:6, p. 424-9): loading dose 15 mg/kg intravenously, follow by bolus dose of 10 mg/kg every 12 hours; after 48 hours, increase bolus dose to 15 mg/kg every 12 hours. Continue fomepizole until serum methanol, EG, DEG, TEG or EGBE are undetectable. The signs and symptoms of poisoning include anion gap metabolic acidosis, CNS depression, renal tubular injury, and possible late stage cranial nerve involvement. Respiratory symptoms, including pulmonary edema, may be delayed. Persons receiving significant exposure should be observed 24-48 hours for signs of respiratory distress. In severe poisoning, respiratory support with mechanical ventilation and positive end expiratory pressure may be required. Maintain adequate ventilation and oxygenation of the patient. If lavage is performed, suggest endotracheal and/or esophageal control. Danger from lung aspiration must be weighed against toxicity when considering emptying the stomach. Treatment of exposure should be directed at the control of symptoms and the clinical condition of the patient. No specific antidote.

5. Fire Fighting Measures

Suitable extinguishing media

Water fog or fine spray. Dry chemical fire extinguishers. Carbon dioxide fire extinguishers. Foam.

Special hazards arising from the substance or mixture

Hazardous Combustion Products: Under fire conditions some components of this product may decompose. The smoke may contain unidentified toxic and/or irritating compounds. Combustion products may include and are not limited to: Carbon monoxide. Carbon dioxide.

Unusual Fire and Explosion Hazards: This material will not burn until the water has evaporated. Residue can burn. Container may rupture from gas generation in a fire situation.

Advice for firefighters

Fire Fighting Procedures: Keep people away. Isolate fire and deny unnecessary entry. Stay upwind. Keep out of low areas where gases (fumes) can accumulate. Use water spray to cool fire exposed containers and fire affected zone until fire is out and danger of reignition has passed. Fight fire from protected location or safe distance. Consider the use of unmanned hose holders or monitor nozzles. Immediately withdraw all personnel from the area in case of rising sound from venting safety device or discoloration of the container. Move container from fire area if this is possible without hazard.

Special Protective Equipment for Firefighters: Wear positive-pressure self-contained breathing apparatus (SCBA) and protective fire fighting clothing (includes fire fighting helmet, coat, trousers, boots, and gloves). If protective equipment is not available or not used, fight fire from a protected location or safe distance.

6. Accidental Release Measures

Personal precautions, protective equipment and emergency procedures: Isolate area. Keep unnecessary and unprotected personnel from entering the area. Refer to Section 7, Handling, for additional precautionary measures. Keep personnel out of low areas. Use appropriate safety equipment. For additional information, refer to Section 8, Exposure Controls and Personal Protection.

Environmental precautions: Prevent from entering into soil, ditches, sewers, waterways and/or groundwater. See Section 12, Ecological Information.

Methods and materials for containment and cleaning up: Contain spilled material if possible. Collect in suitable and properly labeled containers. See Section 13, Disposal Considerations, for additional information.

7. Handling and Storage

Handling

General Handling: Do not swallow. Avoid contact with eyes. Wash thoroughly after handling. Store in tightly closed container. See Section 8, EXPOSURE CONTROLS AND PERSONAL PROTECTION.

Storage

Store away from incompatible materials. See STABILITY AND REACTIVITY section.

8. Exposure Controls / Personal Protection

Exposure Limits

Component	List	Type	Value
Ethylene glycol	ACGIH	Ceiling Aerosol.	100 mg/m ³
	Dow IHG	TWA	50 mg/m ³
	Dow IHG	STEL	100 mg/m ³
Polyethylene glycol	AIHA WEEL	TWA Particulate.	10 mg/m ³
Propylene glycol	WEEL	TWA Aerosol.	10 mg/m ³
Ethylene glycol monobutyl ether	ACGIH	TWA	20 ppm BEI
	OSHA Table Z-1	PEL	240 mg/m ³ 50 ppm SKIN
Diethylene glycol monobutyl ether	Dow IHG	TWA	35 ppm
	ACGIH	TWA Inhalable fraction and vapor.	10 ppm

Personal Protection

Eye/Face Protection: Use chemical goggles.

Skin Protection: Use protective clothing chemically resistant to this material. Selection of specific items such as face shield, boots, apron, or full body suit will depend on the task. When handling hot material, protect skin from thermal burns as well as from skin absorption.

Hand protection: Use gloves chemically resistant to this material. Use gloves with insulation for thermal protection, when needed.

Respiratory Protection: Respiratory protection should be worn when there is a potential to exceed the exposure limit requirements or guidelines. If there are no applicable exposure limit requirements or guidelines, wear respiratory protection when adverse effects, such as respiratory irritation or discomfort have been experienced, or where indicated by your risk assessment process. For most conditions no respiratory protection should be needed; however, if discomfort is experienced, use an approved air-purifying respirator. The following should be effective types of air-purifying respirators: Organic vapor cartridge with a particulate pre-filter.

Ingestion: Avoid ingestion of even very small amounts; do not consume or store food or tobacco in the work area; wash hands and face before smoking or eating.

Engineering Controls

Ventilation: Use local exhaust ventilation, or other engineering controls to maintain airborne levels below exposure limit requirements or guidelines. If there are no applicable exposure limit requirements or guidelines, general ventilation should be sufficient for most operations. Local exhaust ventilation may be necessary for some operations.

9. Physical and Chemical Properties

Appearance

Physical State	Liquid.
Color	Colorless to yellow
Odor	No information available.
Odor Threshold	No test data available
pH	4.4 - 6.4
Melting Point	No test data available
Freezing Point	No test data available
Boiling Point (760 mmHg)	No test data available.
Flash Point - Closed Cup	No test data available
Flash Point - Open Cup	No test data available
Flammable Limits In Air	Lower: No test data available Upper: No test data available
Vapor Pressure	No test data available
Vapor Density (air = 1)	<=1.11
Specific Gravity (H2O = 1)	No test data available
Solubility in water (by weight)	No test data available
Partition coefficient, n-octanol/water (log Pow)	No data available for this product. See Section 12 for individual component data.
Autoignition Temperature	No test data available
Decomposition Temperature	No test data available
Dynamic Viscosity	< 4,000 cPs

10. Stability and Reactivity

Reactivity

No dangerous reaction known under conditions of normal use.

Chemical stability

Stable under recommended storage conditions. See Storage, Section 7.

Possibility of hazardous reactions

Polymerization will not occur.

Conditions to Avoid: Product can oxidize at elevated temperatures. Generation of gas during decomposition can cause pressure in closed systems.

Incompatible Materials: Avoid contact with: Oxidizers. Strong acids. Strong bases. Avoid unintended contact with isocyanates. The reaction of polyols and isocyanates generates heat.

Hazardous decomposition products

Decomposition products depend upon temperature, air supply and the presence of other materials. Decomposition products can include and are not limited to: Carbon dioxide. Alcohols. Ethers. Hydrocarbons. Ketones. Polymer fragments.

11. Toxicological Information

Acute Toxicity

Ingestion

Oral toxicity is expected to be moderate in humans due to ethylene glycol even though tests with animals show a lower degree of toxicity. Ingestion of quantities (approximately 65 mL (2 oz.) for diethylene glycol or 100 mL (3 oz.) for ethylene glycol) has caused death in humans. May cause nausea and vomiting. May cause abdominal discomfort or diarrhea. Excessive exposure may cause central nervous system effects, cardiopulmonary effects (metabolic acidosis), and kidney failure. For Ethylene glycol: Lethal Dose, Human, adult 3 Ounces

As product: Single dose oral LD50 has not been determined. Based on information for component(s): Estimated. LD50, rat > 2,000 mg/kg

Aspiration hazard

Based on physical properties, not likely to be an aspiration hazard.

Dermal

Prolonged skin contact is unlikely to result in absorption of harmful amounts. Repeated skin exposure to large quantities may result in absorption of harmful amounts.

As product: The dermal LD50 has not been determined. Based on information for component(s): Estimated. LD50, rabbit > 2,000 mg/kg

Inhalation

At room temperature, exposure to vapor is minimal due to low volatility. With good ventilation, single exposure is not expected to cause adverse effects. If material is heated or areas are poorly ventilated, vapor/mist may accumulate and cause respiratory irritation and symptoms such as headache and nausea.

As product: The LC50 has not been determined.

Eye damage/eye irritation

May cause eye irritation. May cause corneal injury.

Skin corrosion/irritation

Prolonged contact may cause slight skin irritation with local redness. Repeated contact may cause skin irritation with local redness. Repeated contact may cause flaking and softening of skin. May cause more severe response if skin is abraded (scratched or cut).

Sensitization

Skin

Based on information for component(s): Did not cause allergic skin reactions when tested in humans. Did not cause allergic skin reactions when tested in guinea pigs.

Respiratory

No relevant data found.

Repeated Dose Toxicity

Based on information for component(s): Observations in humans include: Nystagmus (involuntary eye movement). Human red blood cells have been shown to be significantly less sensitive to hemolysis than those of rodents and rabbits. In animals, effects have been reported on the following organs: Kidney. Liver. Blood. In rare cases, repeated excessive exposure to propylene glycol may cause central nervous system effects. Recent findings of kidney failure and death in burn patients, as well as some studies using animal burn models, suggest that polyethylene glycol may have been a factor. The use of topical applications containing this material may not be appropriate in severely burned patients or individuals with impaired renal function.

Chronic Toxicity and Carcinogenicity

For the minor component(s): In long-term animal studies with ethylene glycol butyl ether, small but statistically significant increases in tumors were observed in mice but not rats. The effects are not

believed to be relevant to humans. If the material is handled in accordance with proper industrial handling procedures, exposures should not pose a carcinogenic risk to man.

Carcinogenicity Classifications:

Component	List	Classification
Ethylene glycol monobutyl ether	ACGIH	Confirmed animal carcinogen with unknown relevance to humans.; Group A3

Developmental Toxicity

For the major component(s): Based on animal studies, ingestion of very large amounts of ethylene glycol appears to be the major and possibly only route of exposure to produce birth defects. Exposures by inhalation or skin contact, the primary routes of occupational exposure, had minimal effect on the fetus, in animal studies. For the minor component(s): Has been toxic to the fetus in laboratory animals at doses toxic to the mother.

Reproductive Toxicity

For the major component(s): Ingestion of large amounts of ethylene glycol has been shown to interfere with reproduction in animals. For the minor component(s): In laboratory animal studies, effects on reproduction have been seen only at doses that produced significant toxicity to the parent animals.

Genetic Toxicology

In vitro genetic toxicity studies were negative for component(s) tested. Genetic toxicity studies in animals were negative for component(s) tested.

Component Toxicology - Ethylene glycol

Inhalation	LC50, 6 h, Aerosol, rat, male and female > 2.5 mg/l
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Component Toxicology - Polyethylene glycol

Inhalation	Typical for this family of materials. No deaths occurred at this concentration. LC50, 4 h, Aerosol, rat > 2.86 mg/l
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Component Toxicology - Propylene glycol

Inhalation	No deaths occurred at this concentration. LC50, 2 h, Aerosol, rabbit 317.042 mg/l
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12. Ecological Information

Toxicity

Data for Component: Acrylic polymer(s)

No relevant data found.

Data for Component: Ethylene glycol

Material is practically non-toxic to aquatic organisms on an acute basis (LC50/EC50/EL50/LL50 >100 mg/L in the most sensitive species tested).

Fish Acute & Prolonged Toxicity

LC50, Pimephales promelas (fathead minnow), static test, 96 h: 72,860 mg/l

Aquatic Invertebrate Acute Toxicity

EC50, Daphnia magna (Water flea), static test, 48 h, immobilization: > 100 mg/l

Aquatic Plant Toxicity

ErC50, Pseudokirchneriella subcapitata (green algae), Growth rate inhibition, 96 h: 6,500 - 13,000 mg/l

Toxicity to Micro-organisms

EC50, activated sludge test (OECD 209), Respiration inhibition, 30 min: 225 mg/l

Data for Component: Unknown Component(s); based on supplier information provided

No relevant data found.

Data for Component: Polyethylene glycol

For this family of materials: Material is practically non-toxic to aquatic organisms on an acute basis (LC50/EC50/EL50/LL50 >100 mg/L in the most sensitive species tested).

Data for Component: Propylene glycol

Material is practically non-toxic to aquatic organisms on an acute basis (LC50/EC50/EL50/LL50 >100 mg/L in the most sensitive species tested).

Fish Acute & Prolonged Toxicity

LC50, *Oncorhynchus mykiss* (rainbow trout), static test, 96 h: 40,613 mg/l

Aquatic Invertebrate Acute Toxicity

LC50, *Ceriodaphnia Dubia* (water flea), static test, 48 h: 18,340 mg/l

Aquatic Plant Toxicity

ErC50, *Pseudokirchneriella subcapitata* (green algae), Growth rate inhibition, 96 h: 19,000 mg/l

Toxicity to Micro-organisms

EC50, activated sludge test (OECD 209), Respiration inhibition, 3 h: > 1,000 mg/l

Aquatic Invertebrates Chronic Toxicity Value

Ceriodaphnia Dubia (water flea), semi-static test, 7 d, number of offspring, NOEC: 13020 mg/l

Data for Component: Ethylene glycol monobutyl ether

Material is practically non-toxic to aquatic organisms on an acute basis (LC50/EC50/EL50/LL50 >100 mg/L in the most sensitive species tested).

Fish Acute & Prolonged Toxicity

LC50, *Oncorhynchus mykiss* (rainbow trout), static test, 96 h: 1,474 mg/l

Aquatic Invertebrate Acute Toxicity

EC50, *Daphnia magna* (Water flea), static test, 48 h, immobilization: 1,550 mg/l

Aquatic Plant Toxicity

EbC50, *Pseudokirchneriella subcapitata* (green algae), static test, biomass growth inhibition, 72 h: 911 mg/l

Toxicity to Micro-organisms

IC50; Bacteria: > 1,000 mg/l

Fish Chronic Toxicity Value (ChV)

Danio rerio (zebra fish), semi-static test, 21 d, NOEC:> 100 mg/l

Aquatic Invertebrates Chronic Toxicity Value

Daphnia magna (Water flea), semi-static test, 21 d, Other, NOEC: 100 mg/l

Data for Component: Diethylene glycol monobutyl ether

Material is practically non-toxic to aquatic organisms on an acute basis (LC50/EC50/EL50/LL50 >100 mg/L in the most sensitive species tested).

Fish Acute & Prolonged Toxicity

LC50, *Lepomis macrochirus* (Bluegill sunfish), static test, 96 h: 1,300 mg/l

Aquatic Invertebrate Acute Toxicity

EC50, *Daphnia magna* (Water flea), static test, 48 h, immobilization: > 100 mg/l

Aquatic Plant Toxicity

ErC50, alga *Scenedesmus* sp., static test, Growth rate inhibition, 96 h: > 100 mg/l

ErC50, alga *Scenedesmus* sp., static test, biomass growth inhibition, 96 h: > 100 mg/l

Toxicity to Micro-organisms

EC50; Bacteria, static test: 255 mg/l

Data for Component: L-Proline, 5-oxo-, compd. with chitosan

No relevant data found.

Data for Component: Cationic hydroxyethyl cellulose

Material is moderately toxic to aquatic organisms on an acute basis (LC50/EC50 between 1 and 10 mg/L in the most sensitive species tested).

Fish Acute & Prolonged Toxicity

LC50, *Pimephales promelas* (fathead minnow), static test, 96 h: 2.4 - 3.7 mg/l

Aquatic Invertebrate Acute Toxicity

LC50, *Daphnia magna* (Water flea), static test, 48 h: 34 - 48 mg/l

Toxicity to Micro-organisms

EC50, OECD 209 Test; activated sludge, Respiration inhibition, 3 h: > 1,000 mg/l

EC50; Bacteria, 16 h: 2,500 mg/l

Persistence and Degradability

Data for Component: Acrylic polymer(s)

No relevant data found.

Data for Component: Ethylene glycol

Material is readily biodegradable. Passes OECD test(s) for ready biodegradability. Material is ultimately biodegradable (reaches > 70% biodegradation in OECD test(s) for inherent biodegradability).

OECD Biodegradation Tests:

Biodegradation	Exposure Time	Method	10 Day Window
90 - 100 %	10 d	OECD 301A Test	pass
90 %	1 d	OECD 302B Test	Not applicable

Theoretical Oxygen Demand: 1.29 mg/mgData for Component: Unknown Component(s); based on supplier information provided

No relevant data found.

Data for Component: Polyethylene glycol

For this family of materials: Based on stringent OECD test guidelines, this material cannot be considered as readily biodegradable; however, these results do not necessarily mean that the material is not biodegradable under environmental conditions.

OECD Biodegradation Tests: For this family of materials:

Biodegradation	Exposure Time	Method	10 Day Window
48 %	28 d	OECD 301D Test	fail

Biological oxygen demand (BOD): For this family of materials: For this family of materials:

BOD 5	BOD 10	BOD 20	BOD 28
0 - 17 %	3 - 56 %	39 - 77 %	

Theoretical Oxygen Demand: 1.67 - 1.77 mg/mgData for Component: Propylene glycol

Material is readily biodegradable. Passes OECD test(s) for ready biodegradability.

Biodegradation may occur under anaerobic conditions (in the absence of oxygen).

OECD Biodegradation Tests:

Biodegradation	Exposure Time	Method	10 Day Window
81 %	28 d	OECD 301F Test	pass
96 %	64 d	OECD 306 Test	Not applicable

Indirect Photodegradation with OH Radicals

Rate Constant	Atmospheric Half-life	Method
1.28E-11 cm ³ /s	10 h	Estimated.

Biological oxygen demand (BOD):

BOD 5	BOD 10	BOD 20	BOD 28
69.0 %	70.0 %	86.0 %	

Chemical Oxygen Demand: 1.53 mg/mg**Theoretical Oxygen Demand:** 1.68 mg/mgData for Component: Ethylene glycol monobutyl ether

Material is readily biodegradable. Passes OECD test(s) for ready biodegradability. Material is ultimately biodegradable (reaches > 70% biodegradation in OECD test(s) for inherent biodegradability).

OECD Biodegradation Tests:

Biodegradation	Exposure Time	Method	10 Day Window
90.4 %	28 d	OECD 301B Test	pass

Biological oxygen demand (BOD):

BOD 5	BOD 10	BOD 20	BOD 28
5.2 %	57 %	72.2 %	

Chemical Oxygen Demand: 2.21 mg/g**Theoretical Oxygen Demand:** 2.30 mg/mgData for Component: Diethylene glycol monobutyl ether

Material is readily biodegradable. Passes OECD test(s) for ready biodegradability.

OECD Biodegradation Tests:

Biodegradation	Exposure Time	Method	10 Day Window
89 - 93 %	28 d	OECD 301C Test	Not applicable
100 %	28 d	OECD 302B Test	Not applicable
Indirect Photodegradation with OH Radicals			
Rate Constant	Atmospheric Half-life	Method	
3.62E-11 cm ³ /s	11 h	Estimated.	
Biological oxygen demand (BOD):			
BOD 5	BOD 10	BOD 20	BOD 28
27 %	60 %	81 %	

Theoretical Oxygen Demand: 2.17 mg/mg

Data for Component: **L-Proline, 5-oxo-, compd. with chitosan**

No relevant data found.

Data for Component: **Cationic hydroxyethyl cellulose**

Biodegradation under aerobic static laboratory conditions is low (BOD₂₀ or BOD₂₈/ThOD between 2.5 and 10%).

Biological oxygen demand (BOD):

BOD 5	BOD 10	BOD 20	BOD 28
5.000 %	5.000 %	5.000 %	

Chemical Oxygen Demand: 1.27 mg/mg

Bioaccumulative potential

Data for Component: **Acrylic polymer(s)**

Bioaccumulation: No data available.

Data for Component: **Ethylene glycol**

Bioaccumulation: Bioconcentration potential is low (BCF < 100 or Log Pow < 3).

Partition coefficient, n-octanol/water (log Pow): -1.36 Measured

Data for Component: **Unknown Component(s); based on supplier information provided**

Bioaccumulation: No data available.

Data for Component: **Polyethylene glycol**

Bioaccumulation: For this family of materials: No bioconcentration is expected because of the relatively high water solubility.

Data for Component: **Propylene glycol**

Bioaccumulation: Bioconcentration potential is low (BCF < 100 or Log Pow < 3).

Partition coefficient, n-octanol/water (log Pow): -1.07 Measured

Bioconcentration Factor (BCF): 0.09; Estimated.

Data for Component: **Ethylene glycol monobutyl ether**

Bioaccumulation: Bioconcentration potential is low (BCF < 100 or Log Pow < 3).

Partition coefficient, n-octanol/water (log Pow): 0.81 Measured

Data for Component: **Diethylene glycol monobutyl ether**

Bioaccumulation: Bioconcentration potential is low (BCF < 100 or Log Pow < 3).

Partition coefficient, n-octanol/water (log Pow): 1 Measured

Data for Component: **L-Proline, 5-oxo-, compd. with chitosan**

Bioaccumulation: No relevant data found.

Data for Component: **Cationic hydroxyethyl cellulose**

Bioaccumulation: No bioconcentration is expected because of the relatively high molecular weight (MW greater than 1000).

Mobility in soil

Data for Component: **Acrylic polymer(s)**

Mobility in soil: No data available.

Data for Component: **Ethylene glycol**

Mobility in soil: Given its very low Henry's constant, volatilization from natural bodies of water or moist soil is not expected to be an important fate process., Potential for mobility in soil is very high (Koc between 0 and 50).

Partition coefficient, soil organic carbon/water (Koc): 1 Estimated.

Henry's Law Constant (H): 8.05E-09 atm*m³/mole; 25 °C Estimated.

Distribution in Environment: Mackay Level 1 Fugacity Model:

Air	Water.	Biota	Soil	Sediment
0.03 %	100 %	0 %	0 %	0 %

Data for Component: **Unknown Component(s); based on supplier information provided**

Mobility in soil: No data available.

Data for Component: **Polyethylene glycol**

Mobility in soil: No data available.

Data for Component: **Propylene glycol**

Mobility in soil: Given its very low Henry's constant, volatilization from natural bodies of water or moist soil is not expected to be an important fate process., Potential for mobility in soil is very high (Koc between 0 and 50).

Partition coefficient, soil organic carbon/water (Koc): < 1 Estimated.

Henry's Law Constant (H): 1.2E-08 atm*m3/mole Measured

Data for Component: **Ethylene glycol monobutyl ether**

Mobility in soil: Potential for mobility in soil is high (Koc between 50 and 150).

Partition coefficient, soil organic carbon/water (Koc): 67 Estimated.

Henry's Law Constant (H): 1.60E-06 atm*m3/mole Measured

Data for Component: **Diethylene glycol monobutyl ether**

Mobility in soil: Given its very low Henry's constant, volatilization from natural bodies of water or moist soil is not expected to be an important fate process., Potential for mobility in soil is very high (Koc between 0 and 50).

Partition coefficient, soil organic carbon/water (Koc): 2 Estimated.

Henry's Law Constant (H): 1.52E-09 atm*m3/mole; 25 °C Estimated.

Data for Component: **L-Proline, 5-oxo-, compd. with chitosan**

Mobility in soil: No relevant data found.

Data for Component: **Cationic hydroxyethyl cellulose**

Mobility in soil: No data available.

Results of PBT and vPvB assessment

Data for Component: **Acrylic polymer(s)**

This substance has not been assessed for persistence, bioaccumulation and toxicity (PBT).

Data for Component: **Ethylene glycol**

This substance is not considered to be persistent, bioaccumulating and toxic (PBT). This substance is not considered to be very persistent and very bioaccumulating (vPvB).

Data for Component: **Unknown Component(s); based on supplier information provided**

This substance has not been assessed for persistence, bioaccumulation and toxicity (PBT).

Data for Component: **Polyethylene glycol**

This substance is not considered to be very persistent and very bioaccumulating (vPvB).

Data for Component: **Propylene glycol**

This substance is not considered to be persistent, bioaccumulating and toxic (PBT). This substance is not considered to be very persistent and very bioaccumulating (vPvB).

Data for Component: **Ethylene glycol monobutyl ether**

This substance is not considered to be persistent, bioaccumulating and toxic (PBT). This substance is not considered to be very persistent and very bioaccumulating (vPvB).

Data for Component: **Diethylene glycol monobutyl ether**

This substance is not considered to be persistent, bioaccumulating and toxic (PBT). This substance is not considered to be very persistent and very bioaccumulating (vPvB).

Data for Component: **L-Proline, 5-oxo-, compd. with chitosan**

This substance has not been assessed for persistence, bioaccumulation and toxicity (PBT).

Data for Component: **Cationic hydroxyethyl cellulose**

This mixture has not been assessed for persistence, bioaccumulation and toxicity (PBT).

Other adverse effects

Data for Component: **Acrylic polymer(s)**

This substance is not in Annex I of Regulation (EC) No 1005/2009 on substances that deplete the ozone layer.

Data for Component: **Ethylene glycol**

This substance is not in Annex I of Regulation (EC) No 1005/2009 on substances that deplete the ozone layer.

Data for Component: Unknown Component(s); based on supplier information provided

This substance is not in Annex I of Regulation (EC) No 1005/2009 on substances that deplete the ozone layer.

Data for Component: Polyethylene glycol

This substance is not in Annex I of Regulation (EC) No 1005/2009 on substances that deplete the ozone layer.

Data for Component: Propylene glycol

This substance is not in Annex I of Regulation (EC) No 1005/2009 on substances that deplete the ozone layer.

Data for Component: Ethylene glycol monobutyl ether

This substance is not in Annex I of Regulation (EC) No 1005/2009 on substances that deplete the ozone layer.

Data for Component: Diethylene glycol monobutyl ether

This substance is not in Annex I of Regulation (EC) No 1005/2009 on substances that deplete the ozone layer.

Data for Component: L-Proline, 5-oxo-, compd. with chitosan

No relevant data found.

Data for Component: Cationic hydroxyethyl cellulose

This substance is not in Annex I of Regulation (EC) No 1005/2009 on substances that deplete the ozone layer.

13. Disposal Considerations

Disposal methods

NOTICE: Research sample for use by qualified personnel only. Upon completion of tests, dispose of material and container safely and in accord with federal, state/provincial and local laws and regulations. If further information is needed on disposal or use, consult The Dow Chemical Company.

14. Transport Information

DOT Non-Bulk

NOT REGULATED

DOT Bulk

NOT REGULATED

IMDG

NOT REGULATED

ICAO/IATA

NOT REGULATED

This information is not intended to convey all specific regulatory or operational requirements/information relating to this product. Additional transportation system information can be obtained through an authorized sales or customer service representative. It is the responsibility of the transporting organization to follow all applicable laws, regulations and rules relating to the transportation of the material.

15. Regulatory Information

OSHA Hazard Communication Standard

This product is a "Hazardous Chemical" as defined by the OSHA Hazard Communication Standard, 29 CFR 1910.1200.

Superfund Amendments and Reauthorization Act of 1986 Title III (Emergency Planning and Community Right-to-Know Act of 1986) Sections 311 and 312

Immediate (Acute) Health Hazard	Yes
Delayed (Chronic) Health Hazard	Yes
Fire Hazard	No
Reactive Hazard	No
Sudden Release of Pressure Hazard	No

Superfund Amendments and Reauthorization Act of 1986 Title III (Emergency Planning and Community Right-to-Know Act of 1986) Section 313

This product contains the following substances which are subject to the reporting requirements of Section 313 of Title III of the Superfund Amendments and Reauthorization Act of 1986 and which are listed in 40 CFR 372.

Component	CAS #	Amount
Ethylene glycol	107-21-1	>= 0.0 - <= 50.0 %
Ethylene glycol monobutyl ether	111-76-2	>= 0.0 - <= 10.0 %
Diethylene glycol monobutyl ether	112-34-5	>= 0.0 - <= 5.0 %

Pennsylvania (Worker and Community Right-To-Know Act): Pennsylvania Hazardous Substances List and/or Pennsylvania Environmental Hazardous Substance List:

The following product components are cited in the Pennsylvania Hazardous Substance List and/or the Pennsylvania Environmental Substance List, and are present at levels which require reporting.

Component	CAS #	Amount
Ethylene glycol	107-21-1	>= 0.0 - <= 50.0 %
Propylene glycol	57-55-6	>= 0.0 - <= 50.0 %
Ethylene glycol monobutyl ether	111-76-2	>= 0.0 - <= 10.0 %
Diethylene glycol monobutyl ether	112-34-5	>= 0.0 - <= 5.0 %

Pennsylvania (Worker and Community Right-To-Know Act): Pennsylvania Special Hazardous Substances List:

To the best of our knowledge, this product does not contain chemicals at levels which require reporting under this statute.

California Proposition 65 (Safe Drinking Water and Toxic Enforcement Act of 1986)

WARNING: This product contains a chemical(s) known to the State of California to cause cancer.

Component	CAS #	Amount
Acetaldehyde	75-07-0	>= 0.0 - <= 1.62 PPM
1,4-Dioxane	123-91-1	>= 0.0 - <= 1.4 PPM
Ethyl acrylate	140-88-5	>= 0.0 - < 0.0001 %
Formaldehyde	50-00-0	>= 0.0 - <= 0.0099 %
Ethanol	64-17-5	>= 0.0 - <= 0.01 %
Ethylene oxide	75-21-8	>= 0.0 - <= 1.35 PPM

California Proposition 65 (Safe Drinking Water and Toxic Enforcement Act of 1986)

WARNING: This product contains a chemical(s) known to the State of California to cause birth defects or other reproductive harm.

Component	CAS #	Amount
Ethylene oxide	75-21-8	>= 0.0 - <= 1.35 PPM

US. Toxic Substances Control Act

This product contains chemical substance(s) not on the TSCA Inventory. It may be used for research and development purposes only, and only under the supervision of a technically qualified individual. All persons engaged in research and development with this product must be informed of the hazard information in this Material Safety Data Sheet (MSDS).

16. Other Information**Revision**

Identification Number: 1063790 / 1001 / Issue Date 09/21/2015 / Version: .0

Most recent revision(s) are noted by the bold, double bars in left-hand margin throughout this document.

Legend

N/A	Not available
W/W	Weight/Weight
OEL	Occupational Exposure Limit
STEL	Short Term Exposure Limit
TWA	Time Weighted Average
ACGIH	American Conference of Governmental Industrial Hygienists, Inc.
DOW IHG	Dow Industrial Hygiene Guideline
WEEL	Workplace Environmental Exposure Level
HAZ_DES	Hazard Designation
Action Level	A value set by OSHA that is lower than the PEL which will trigger the need for activities such as exposure monitoring and medical surveillance if exceeded.

The Dow Chemical Company urges each customer or recipient of this (M)SDS to study it carefully and consult appropriate expertise, as necessary or appropriate, to become aware of and understand the data contained in this (M)SDS and any hazards associated with the product. The information herein is provided in good faith and believed to be accurate as of the effective date shown above. However, no warranty, express or implied, is given. Regulatory requirements are subject to change and may differ between various locations. It is the buyer's/user's responsibility to ensure that his activities comply with all federal, state, provincial or local laws. The information presented here pertains only to the product as shipped. Since conditions for use of the product are not under the control of the manufacturer, it is the buyer's/user's duty to determine the conditions necessary for the safe use of this product. Due to the proliferation of sources for information such as manufacturer-specific (M)SDSs, we are not and cannot be responsible for (M)SDSs obtained from any source other than ourselves. If you have obtained an (M)SDS from another source or if you are not sure that the (M)SDS you have is current, please contact us for the most current version.

Material Safety Data Sheet

Updated 6/8/15

1. Product and Company Identification

Product Name: "Sea-Salt" ASTM D 1141-98, Formula A, Table X1.1
Product Code: 114120 and 114150
Supplier: Lake Products Company LLC
PO Box 2658
Florissant, MO 63032
314-770-2299
Mark.Youngberg@lakeproductscompany.com
www.lakeproductscompany.com

Synonym: "Sea-Salt" ASTM D 1141-52, Formula A, Table 1, Section 4
Material Uses: Corrosion studies, ocean instrument testing, chemical testing, and biological testing

Validation date: 7/2/13
In case of emergency: 314-494-7443 / 24 hours a day

2. Hazards Identification

Emergency Overview: Caution!
May cause eye and skin irritation
May cause damage to the following organs: skin, eyes, stomach.
Avoid contact with eyes, skin and clothing. Wash thoroughly after handling.

Physical state: Solid. [Granular solid. Crystals.]
OSHA/HCS: This material is considered hazardous by the OSHA Hazard Communication Standard (29 CFR 1910.1200).
Routes of entry: Ingestion

Potential acute health effects:

Inhalation: No known significant effects or critical hazards.
Ingestion: When ingested in large quantities may cause gastrointestinal upset or diarrhea
Skin: May cause skin irritation
Eyes: May cause eye irritation

Potential chronic health effects:

Carcinogenicity: No known significant effects or critical hazards.
Mutagenicity: No known significant effects or critical hazards.
Teratogenicity: No known significant effects or critical hazards.
Developmental: No known significant effects or critical hazards.
Fertility effects: No known significant effects or critical hazards.
Target organs: May cause damage to the following organs: skin, eyes, stomach

Medical conditions aggravated
by overexposure:

Pre-existing disorders involving any target organs mentioned in this MSDS as being at risk may be aggravated by over-exposure to this product.

See toxicological information (section 11)

3. Composition/information on ingredients

<u>Name</u>	<u>CAS Number</u>	<u>% by weight</u>
Sodium Chloride	7647-14-5	58.490
Magnesium Chloride	7791-18-6	26.460
Sodium Sulfate	7757-82-6	9.750
Calcium Chloride	10043-52-4	2.765
Potassium Chloride	7447-40-7	1.645
Sodium Bicarbonate	144-55-8	0.477
Potassium Bromide	7758-02-3	0.238
Boric Acid	10043-35-3	0.071
Strontium Chloride	10025-70-4	0.095
Sodium Fluoride	7681-49-4	0.007

4. First Aid Measures

Eye Contact: Check for and remove any contacted lenses. Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyeballs.

Skin Contact: In case of contact, immediately flush skin with plenty of water

Inhalation: Move exposed person to fresh air.

Ingestion: Give several glasses of water to drink to dilute. If large amounts were swallowed, get medical advice.

5. Fire-Fighting Measures

Flammability of the product:	No Specific fire or explosion hazard.
Extinguishing media:	Use an extinguishing agent suitable for the surrounding fire.
Not suitable:	None known.
Special exposure hazards:	Promptly isolate the scene by removing all persons from the vicinity of the incident if there is a fire. No action shall be taken involving any personal risk or without suitable training.
Hazardous thermal Decomposition products:	Decomposition products may include the following materials: Halogenated compounds; Metal oxide/oxides
Special protective Equipment for fire-fighters:	Fire-fighters should wear appropriate protective equipment and self-contained breathing apparatus (SCBA) with a full face-piece operated in positive pressure mode.

6. Accidental Release Measures

Personal precautions:	Provide adequate ventilation. Wear appropriate respirator when ventilation is inadequate. Put on appropriate personal protective equipment (see section 8)
Environmental precautions:	Avoid dispersal of spilled material and runoff and contact with soil, waterways, drains and sewers. Inform the relevant authorities if the product has caused environmental pollution (sewers, waterways, soil or air)
Methods of cleaning up spill:	Vacuum or sweep up material and place in a designated labeled container.
Handling:	Do not ingest. Avoid contact with eyes, skin and clothing. Keep in the original container or an approved alternative made from a compatible material, kept tightly closed when not in use.
Storage:	Store in accordance with local regulations. Store in original container, protected from direct sunlight. Keep container tightly closed and sealed until ready for use.

7. Handling and Storage

Safe Handling:	Avoid breathing dust Promptly cleanup spills Keep away from eyes
Storage:	Keep containers closed Store away from oxidizing agents Protect from extreme temperatures and humidity during storage

8. Exposure Controls/Personal Protection

Consult local authorities for acceptable exposure limits.

Engineering measures: No special ventilation requirements. Good general ventilation should be sufficient to control worker exposure to airborne contaminants. If this product contains ingredients with exposure limits, use process enclosures, local exhaust ventilation or other engineering controls to keep worker exposure below any recommended or statutory limits.

Hygiene measures: Wash hands, forearms and face thoroughly after handling chemical products, before eating, smoking and using the lavatory and at the end of the working period. Appropriate techniques should be used to remove potentially contaminated clothing. Wash contaminated clothing before reusing. Ensure that eyewash stations and safety showers are close to the workstation location.

Personal protection

Respiratory:	Use a properly fitted, air-purifying or air-fed respirator complying with an approved standard if a risk assessment indicates this is necessary. Respirator selection must be based on known or anticipated exposure levels, the hazards of the product and the safe working limits of the selected respirator.
Hands:	Chemical-resistant, impervious gloves complying with an approved standard should be worn at all times when handling chemical products if a risk assessment indicates this is necessary. Recommended: nitrile rubber gloves
Eyes:	Safety eyewear complying with an approved standard should be used when a risk assessment indicates this is necessary to avoid exposure to

	liquid splashes, mists or dusts. Recommended: safety glasses with side-shields
Skin:	Personal protective equipment for the body should be selected based on the task being performed and the risks involved and should be approved by a specialist before handling this product. Recommended: lab coat
Environmental exposure Controls:	Emissions from ventilation or work process equipment should be checked to ensure they comply with the requirements of environmental protection legislation.

9. Physical and Chemical Properties

Physical State:	Solid [Granular solid Crystals]
Color:	White
Odor:	Odorless
Molecular weight:	Not available
Molecular formula:	Not available
pH:	Not available
Boiling/condensation point:	Not available
Melting/freezing point:	Not available
Relative density:	Not available
Vapor pressure:	Not available
Vapor density:	Not available
Odor threshold:	Not available
Evaporation rate:	Not available
Solubility:	Soluble in the following materials: water

10. Stability and reactivity

Chemical stability:	The product is stable
Possibility of hazardous reactions:	Under normal conditions of storage and use, hazardous reactions will not occur.
Hazardous polymerization:	Under normal conditions of storage and use, hazardous reactions will not occur.
Conditions to avoid:	No specific data.
Hazardous decomposition products:	Under normal conditions of storage and use, hazardous reactions will not occur.

11. Toxicological information

<u>Acute toxicity:</u>	Product/ingredient name: "Sea Salt" ASTM D 1141-52, formula a
Health hazards (Acute):	Irritation of the eyes, skin and respiratory tract. Ingestion of large doses may cause gastro-intestinal distress and vomiting.
<u>Carcinogenicity:</u>	No known significant effects or critical hazards.
<u>Mutagenicity:</u>	No known significant effects or critical hazards.
<u>Teratogenicity:</u>	No known significant effects or critical hazards.

12. Ecological information

<u>Aquatic ecotoxicity:</u>	Product/ingredient name: "Sea Salt" ASTM D 1141-52, formula a Aquatic ecotoxicity is that of seawater when mixed to correct concentration
Environmental effects:	No known significant effects or critical hazards.
Other adverse effects:	No known significant effects or critical hazards.

13. Disposal considerations

The information presented only applies to the material as supplied. The identification based on characteristic(s) or listing may not apply if the material has been used or otherwise contaminated. It is the responsibility of the waste generator to determine the toxicity and physical properties of the material generated to determine the proper waste identification and disposal methods in compliance with applicable regulations. Disposal should be in accordance with applicable regional, national and local laws and regulations.

14. Transport information

DOT Classification – not regulated (Not Hazardous)

15. Regulatory information

United States

HCS Classification:	Irritating material Target organ effects
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U.S. Federal regulations: United States inventory (TSCA 8b): This material is listed or exempted. TSCA (Toxic Substance Control Act): This product is listed on the TSCA Inventory.

Canada

WHMIS (Canada): Not controlled under WHMIS (Canada)

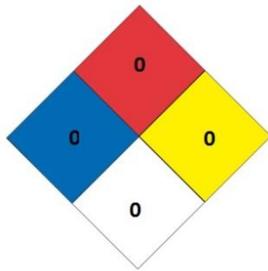
EU regulations

Risk phrases: This product is not classified according to EU legislation.

16. Other information

National Fire Protection:
Association (U.S.A.)

Health – 0 Flammability – 0 Instability – 0 Special - 0



Notice to reader

The statements contained herein are based upon technical data that Lake Products Company believes to be reliable, are offered for information purposes only and as a guide to the appropriate precautionary and emergency handling of the material by a properly trained person having the necessary technical skills. Users should consider these data only as a supplement to other information gathered by them and must make independent determinations of suitability and completeness of information from all sources to assure proper use, storage and disposal of these materials and the safety and health of employees and customers and the protection of the environment.

Appendix B: Experiment Results

(DOI/BSEE E15PG00032)

Characterization of crude oils

Crude type	Viscosity (cP)	Sulfur content (wt%)
ANS	~15	2.0
North Star	~6	1.4
Harmony	~1500	5.7
Ewing Bank	~600	3.0

Denver cell experiments

Residue F660 in seawater during flotation at 5 °C, respectively

Time (min.)		Residue Frother (ppm)			
		0	5	10	30
Run 1	test1	17.7	2.0	BDL	BDL
	test2	13.6	3.4	BDL	BDL
	test3	16.4	4.9	BDL	BDL
Run 2	test1	19.3	1.0	BDL	BDL
	test2	24.4	1.2	BDL	BDL
	test3	21.6	2.1	BDL	BDL
Run 3	test1	11.3	2.7	BDL	BDL
	test2	8.3	2.3	BDL	BDL
	test3	8.7	6.9	BDL	BDL

Residue F672 in seawater during flotation at 5 °C, respectively

Time (min.)		Residue Frother (ppm)			
		0	5	10	30
Run 1	test1	9.9	3.5	5.2	1.9
	test2	9.5	2.2	4.2	1.2
	test3	10.1	0.8	7.6	3.1
Run 2	test1	11.2	4.1	5.7	4.7
	test2	11.0	5.7	6.9	6.4
	test3	11.6	6.4	8.2	5.6
Run 3	test1	8.2	8.2	0.8	2.1
	test2	8.6	9.6	4	6.5
	test3	7.9	10	0	3.7

Gas holdup measurement of frothers in DI water at 5 °C, respectively

Rotation rate (rpm)	Gas holdup (%)			
	15 ppm of F660	15 ppm of F672	30 ppm of 2-EH	30 ppm of MIBC
800	23.2	18.9	13.7	17.1
900	32.2	28.6	18.3	22.4
1000	40.8	39.4	22.9	28.9
1200	n/a	n/a	30.2	37

Gas holdup measurement of 15 ppm of frothers in seawater at 5 °C, respectively

Rotation rate (rpm)	Gas holdup (%)			
	MIBC	F660	F672	2-EH
800	10.9	15.2	11.39	12.1
900	15.8	25.5	20.9	20.7
1000	20.6	32.3	29.6	24.7
1200	28.7	38.6	37.5	30.3

Gas holdup measurement of 15 ppm of frothers in seawater at -2 °C, respectively

Rotation rate (rpm)	Gas holdup (%)			
	MIBC	F660	F672	2-EH
800	11.5	15.3	14.2	13.7
900	15.2	23.8	23.6	17.8
1000	20.6	29.9	30.8	23.6
1200	28.7	37.8	37.9	28

Flotation of arctic oils in seawater at different operating conditions (RPM=800)

Crude	Temperature (°C)	Frother	Frother (ppm)	Residue oil in water (ppm)	Residue water in oil (%)
NS	-2	n/a	-	7.3	-
				58.7	
				68.9	
	5			9.1	-
				66.2	
				77.3	
-2	F660	15	11.2	20.9	
			13.3	11.0	
ANS	5	n/a	-	30.2	8.0
	-2			8.7	9.7
	5	F660	15	1.0	7.7
	-2			2.9	9.5
	5		5	0.3	8.2
	-2			1.2	6.8

	5	Custo527	5	5.9	4.5
	-2			2.2	7.5
	5		15	<2	12.5
	-2			<2	15.0

Flotation of non-arctic crude oils in seawater at 30 °C (RPM=800)

Crude	Frother	Frother (ppm)	Residue oil in water (ppm)	Residue water in oil (%)
Ewing Bank	-	0	0.5	2.4
	F660	15	0.3	13.6
	Custo527	15	0.3	14.6
Harmony	-	0	0.9	4.3
	F660	15	0.2	9.0
	Custo527	15	0.2	15.7

Flotation of emulsified ANS crude in seawater at 5 °C (RPM=800)

Time (min)	Residual oil conc. (ppm)	
	no frother	15 ppm Custo 527
0	422.5	414.8
5	241.2	n/a
10	111.9	27.2
20	55.0	18.7
30	26.4	15.5
40	14.3	14.9
50	7.6	13.5
60	4.3	13

Flotation of emulsified ANS crude in seawater at 0 °C (RPM=800)

Time (min)	Residual oil conc. (ppm)	
	no frother	15 ppm Custo 527
0	367.1	365.6
5	n/a	220.3
10	84.2	79.2
20	32.3	26.5
30	11.7	15.3
40	4.5	12.1
50	2.0	11.0
60	1.6	10.5

Flotation of oil contaminated ice in seawater at -2 °C (RPM=800)

Oil conc. (wt%)	Ice conc. (wt%)	Frother	Frother Conc. (ppm)	Ice size (mm)	Oil recovery (wt%)	Oil in water conc. (ppm)
10	10	n/a	0	3-5	98.1	0.8
		Custo527	15		95.2	1.5
		n/a	0	5-15	91.0	2.4
		Custo527	15		90.1	1.1

GC-SPE analysis

Blank Tests: No oil was added during the test. Experiment was conducted at room temperature. The nominal starting concentration of frother was 15 ppm. The impeller speed was 800 rpm.

Sample #	Sample ID	F660	F527 (ppm)
1	standard 10 ppm F660	13.7	BDL
2	F660 0 min test	16.5	BDL
3	F660 10 min test	16.8	BDL
4	F660 20 min test	15.6	BDL
5	F660 30 min test	17.6	BDL
6	F660 in froth (collected between 0-10min)	12.5	BDL
7	Standard 10 ppm Custo 527	BDL	13.5
8	Custo 527 0 min	BDL	23.8
9	Custo 527 10 min	BDL	13.9
10	Custo 527 20 min	BDL	6.6
11	Custo 527 30 min	BDL	5.4
12	Custo 527 in froth (collected between 0-10min)	BDL	35.3

Free ANS Oil Test: The test was conducted for 30 min at 0 °C. The impeller speed was 800 rpm. The nominal starting concentration of frother was 15 ppm.

Sample #	Sample ID	F660 (ppm)	Custo527 (ppm)
1	Before test, with Custo 527	BDL	22.6
2	After test, with Custo 527	BDL	16.9
3	Before test, with F660	15.2	BDL
4	After test, with F660	12.9	BDL

Free EB and NS Oil Test: The nominal starting concentration of frother was 15 ppm. Tests were conducted at 30 °C for 30 min. The impeller speed was 1200 rpm.

Sample #	Sample ID	F660 (ppm)	Custo527 (ppm)
1	After-EB Oil-Seawater-F660	15.7	BDL
2	Afetr-NS Oil-Seawater-F660	11.1	BDL

Emulsified oil test: The nominal starting concentration of frother was 15 ppm. The test was conducted for 60 mins at 0 °C. The impeller speed was 800 rpm.

Sample #	Sample ID	F660 (ppm)	Custo527 (ppm)
1	Before flotation -ANS oil-Seawater-527	BDL	20.7
2	After 60min flotation-ANS oil-Sewater-527	BDL	14.1

Flotation column experiments

Gas holdup measurement in a 3” flotation column

Circulation pump setting	Gas holdup (%)	
	- 2 °C	5 °C
<i>no frother</i>		
speed 6	2.41	4.08
speed 8	3.09	5.22
<i>15ppm Custo527</i>		
speed 6	8.25	10.90
speed 8	10.98	13.50
<i>10ppm Romax6000</i>		
speed 6	3.23	n/a
speed 8	4.88	n/a

Bubble size measurement without frother at -2 °C in a 3” flotation column

	sparger speed 6		sparger speed 8	
	bottom	top	bottom	top
average size (mm)	0.56	1.00	0.45	0.60
standard deviation	0.38	0.73	0.37	0.54
bubble #	bubble size (mm)	bubble size (mm)	bubble size (mm)	bubble size (mm)
1	0.61	2.09	2.59	2.64
2	0.29	0.68	0.24	1.81
3	0.65	0.53	0.24	0.80
4	0.32	1.07	0.32	0.54
5	1.54	0.61	0.30	0.51
6	0.35	0.80	0.26	0.49
7	0.48	0.73	0.22	0.76
8	0.84	0.82	0.24	0.55
9	0.35	0.65	0.26	0.97
10	0.45	0.75	0.40	0.66
11	0.38	0.62	0.34	0.49
12	0.38	0.61	0.31	1.17
13	0.56	0.43	0.38	1.37
14	0.70	0.75	0.97	1.02

15	0.74	0.64	0.72	0.57
16	0.31	3.34	1.80	0.44
17	0.18	0.58	1.99	0.60
18	0.30	1.33	0.30	0.35
19	0.24	0.48	0.43	0.26
20	0.28	0.26	0.37	0.45
21	0.22	1.26	0.28	0.37
22	0.32	0.48	0.22	0.24
23	0.34	0.66	0.24	0.37
24	0.36	0.48	0.32	0.26
25	0.30	0.55	0.34	0.31
26	0.27	0.52	0.39	0.36
27	0.34	0.52	0.66	0.28
28	0.27	0.36	0.32	0.40
29	0.27	0.51	0.26	0.21
30	0.29	0.55	0.33	0.27
31	0.78	0.47	0.28	0.23
32	0.36	0.71	0.22	0.38
33	0.80	0.50	0.37	0.27
34	0.34	0.48	0.13	0.22
35	0.60	0.66	0.87	0.32
36	0.35	0.64	0.24	0.27
37	0.61	0.55	0.43	0.32
38	0.40	0.51	0.24	0.23
39	0.30	0.46	0.35	0.27
40	0.29	0.41	0.38	1.10
41	0.23	0.57	0.32	0.22
42	0.32	0.56	0.32	0.26
43	0.78	0.70	0.24	0.24
44	0.27	3.68	0.15	0.24
45	0.41	4.07	1.78	0.18
46	0.47	1.25	0.49	1.04
47	0.27	2.08	0.17	0.30
48	0.42	0.62	0.30	0.22
49	0.41	0.73	0.37	0.21
50	0.62	0.99	0.37	0.17
51	0.43	0.75	0.35	0.31
52	0.41	1.55	0.28	0.15
53	0.30	2.55	1.06	0.21
54	0.26	2.86	1.32	0.31
55	0.26	0.47	0.92	0.27
56	0.35	0.51	0.37	0.52
57	0.40	0.41	0.34	0.24

58	0.28	0.51	0.30	0.38
59	0.41	0.44	0.27	0.21
60	0.34	0.46	0.34	0.26
61	0.49	0.76	0.35	0.23
62	0.32	0.48	0.45	0.24
63	0.48	0.44	0.28	0.27
64	0.32	0.48	1.29	0.29
65	0.41	0.57	0.29	0.24
66	1.12	0.94	0.22	0.14
67	0.87	1.58	0.45	0.21
68	0.47	0.36	0.30	0.19
69	0.41	0.45	0.44	0.20
70	0.32	0.41	0.28	0.19
71	0.53	3.66	0.32	0.30
72	0.37	0.69	0.33	0.30
73	0.28	1.82	0.43	0.42
74	0.27	0.63	1.33	0.31
75	1.50	0.74	1.05	0.64
76	0.96	2.05	0.35	0.42
77	0.82	2.12	1.25	0.32
78	0.30	1.78	2.53	0.23
79	0.37	1.07	2.19	0.28
80	0.39	0.87	0.74	0.27
81	0.42	0.64	1.83	0.47
82	0.47	0.79	1.59	0.33
83	0.29	0.57	0.34	0.35
84	0.83	0.75	0.34	0.49
85	0.40	0.75	1.48	0.40
86	1.83	0.99	0.33	0.41
87	1.14	1.26	0.30	0.36
88	0.56	0.83	0.37	0.72
89	0.39	0.78	0.27	0.40
90	0.87	0.86	0.28	0.45
91	0.50	0.73	0.39	2.49
92	0.54	0.68	0.44	2.89
93	0.64	0.97	0.38	1.07
94	0.49	0.62	0.51	0.66
95	0.63	0.93	0.37	0.37
96	0.30	0.53	0.43	0.54
97	0.46	0.79	0.50	0.43
98	0.35	0.72	0.26	0.81
99	0.36	0.62	0.34	0.54
100	0.41	1.04	0.41	0.67

101	0.37	0.64	0.32	0.56
102	0.34	0.64	0.40	0.46
103	0.37	0.56	0.40	0.38
104	0.18	0.57	0.35	1.24
105	0.41	0.79	0.39	0.74
106	0.60	0.56	0.32	1.20
107	0.55	0.91	0.34	0.38
108	0.34	0.58	0.43	0.33
109	0.30	2.21	0.56	0.37
110	0.29	2.71	0.39	0.28
111	0.38	3.21	0.27	0.33
112	1.83	2.02	0.39	0.33
113	0.45	1.94	0.17	0.33
114	0.31	0.79	0.34	0.58
115	0.34	0.82	0.32	0.59
116	0.67	1.00	0.32	0.40
117	0.29	0.89	0.28	0.33
118	0.52	0.75	0.30	0.40
119	0.35	0.82	0.32	0.48
120	0.35	0.58	0.35	0.41
121	0.52	0.64	0.38	0.62
122	0.35	1.02	0.25	0.76
123	0.40	1.59	0.35	0.54
124	0.41	0.75	0.31	0.54
125	0.40	0.73	0.57	0.43
126	0.31	0.51	0.22	0.63
127	0.48	0.68	0.25	0.31
128	0.31	0.79	0.26	0.52
129	0.69	0.72	0.39	0.73
130	0.60	0.84	0.37	0.45
131	0.60	1.51	0.34	0.44
132	0.31	0.72	0.38	0.57
133	0.56	1.59	0.31	0.64
134	0.37	1.75	0.29	0.51
135	0.39	0.97	0.22	0.83
136	1.62	0.56	0.32	0.51
137	0.39	1.04	0.45	2.66
138	1.68	2.36	0.30	3.36
139	0.53	2.04	0.34	2.35
140	0.45	2.01	0.25	3.56
141	0.78	3.27	0.20	1.22
142	1.69	2.46	0.22	0.62
143	1.34	0.69	0.37	0.63

144	0.52	0.96	0.24	0.45
145	0.50	1.75	0.40	0.38
146	0.43	0.61	0.28	0.73
147	0.43	0.70	0.27	0.56
148	0.42	0.72	0.28	0.72
149	0.37	0.66	0.41	0.70
150	1.25	0.84	0.23	0.45
151	1.39	0.42	0.63	0.34
152	1.94	0.56	0.45	0.57
153	0.36	0.81	0.30	0.45
154	0.30	3.39	0.63	2.29
155	0.33	1.64	0.30	0.47
156	0.52	0.78	0.36	0.49
157	0.75	0.79	0.48	0.69
158	0.56	1.28	0.32	0.57
159	0.32	0.87	0.48	0.63
160	0.56	0.55	0.28	2.04
161	0.72	0.95	0.30	0.63
162	0.46	0.66	0.34	0.43
163	0.38	1.61	0.39	0.42
164	0.27	1.05	0.30	0.42
165	0.33	0.69	0.34	0.31
166	0.25	1.32	0.47	0.48
167	0.96	2.31	0.40	0.51
168	0.93	0.97	0.47	0.56
169	2.08	0.50	0.34	0.49
170	0.35	0.90	0.57	0.62
171	1.43	0.64	0.44	0.60
172	0.38	0.42	1.84	0.60
173	1.33	0.59	0.35	0.67
174	1.74	0.78	1.22	0.53
175	1.03	0.72	0.24	1.05
176	0.42	0.55	0.48	1.22
177	0.34		0.43	1.52
178	0.46		0.19	0.95
179	0.22		0.24	0.48
180	0.25		0.35	0.42
181	0.38		0.33	0.30
182	0.58		0.33	0.48
183	1.84		0.30	0.88
184	0.36		0.40	0.75
185	0.28		0.37	0.83
186	0.47		0.30	0.84

187	0.50		0.24	0.71
188	1.52		0.30	0.55
189	1.40		0.24	0.67
190	1.10		0.39	0.46
191	0.44		0.47	0.80
192	0.42		0.34	0.36
193	0.36		0.39	0.48
194	0.74		0.33	0.41
195	0.37		0.34	0.93
196	0.32		0.44	0.57
197	0.90		0.38	0.34
198	0.67		0.41	0.29
199	0.52		0.38	0.33
200	0.59		0.33	0.45
201			0.19	0.96
202			0.41	1.21
203			0.29	
204			0.39	
205			0.40	
206			0.45	
207			0.32	
208			0.45	
209			0.45	
210			0.51	
211			0.54	
212			0.45	
213			0.45	
214			0.47	
215			0.34	
216			0.29	
217			0.34	
218			0.34	
219			0.53	
220			0.28	
221			0.30	
222			0.24	
223			0.29	
224			0.24	
225			0.29	
226			0.32	
227			0.33	
228			0.37	
229			0.34	

230			0.34	
231			0.28	
232			0.38	
233			0.38	
234			0.40	
235			0.41	
236			0.37	
237			0.41	
238			0.72	
239			0.42	

Bubble size measurement with 15ppm Custo527 at -2 °C in a 3” flotation column

	sparger speed 6		sparger speed 8	
	bottom	top	bottom	top
average size (mm)	0.34	0.30	0.38	0.35
standard deviation	0.10	0.11	0.12	0.10
bubble #	bubble size (mm)	bubble size (mm)	bubble size (mm)	bubble size (mm)
1	0.66	0.54	0.61	0.62
2	0.34	0.41	0.39	0.57
3	0.31	0.34	0.40	0.27
4	0.27	0.38	0.33	0.30
5	0.45	0.29	0.38	0.33
6	0.21	0.28	0.39	0.44
7	0.28	0.29	0.29	0.40
8	0.30	0.24	0.38	0.29
9	0.27	0.32	0.35	0.33
10	0.21	0.53	0.49	0.36
11	0.39	0.27	0.44	0.46
12	0.24	0.38	0.29	0.40
13	0.36	0.34	0.40	0.26
14	0.35	0.30	0.42	0.33
15	0.27	0.20	0.27	0.81
16	0.27	0.27	0.33	0.23
17	0.32	0.38	0.29	0.32
18	0.21	0.34	0.43	0.43
19	0.23	0.25	0.34	0.37
20	0.41	0.34	0.27	0.34
21	0.36	0.24	0.35	0.29
22	0.24	0.28	0.36	0.28
23	0.21	0.37	0.38	0.40
24	0.29	0.58	0.37	0.48
25	0.23	0.51	0.68	0.26

26	0.23	0.21	0.42	0.31
27	0.21	0.31	0.27	0.34
28	0.56	0.26	0.26	0.52
29	0.19	0.22	0.34	0.46
30	0.23	0.28	0.39	0.50
31	0.24	0.34	0.22	0.25
32	0.27	0.21	0.26	0.50
33	0.21	0.20	0.26	0.26
34	0.23	0.24	0.49	0.37
35	0.26	0.25	0.32	0.34
36	0.34	0.23	0.25	0.49
37	0.21	0.23	0.20	0.46
38	0.21	0.21	0.38	0.33
39	0.18	0.20	0.26	0.33
40	0.27	0.16	0.25	0.47
41	0.24	0.17	0.34	0.33
42	0.50	0.32	0.48	0.26
43	0.21	0.17	0.56	0.31
44	0.30	0.28	0.25	0.40
45	0.30	0.35	0.27	0.59
46	0.38	0.35	0.32	0.33
47	0.52	0.37	1.18	0.40
48	0.33	0.25	0.29	0.56
49	0.33	0.30	0.30	0.49
50	0.36	0.20	0.38	0.43
51	0.34	0.21	0.38	0.48
52	0.21	0.32	0.24	0.35
53	0.28	0.20	0.43	0.25
54	0.60	0.30	0.37	0.39
55	0.23	0.32	0.50	0.42
56	0.27	0.21	0.43	0.37
57	0.24	0.33	0.55	0.49
58	0.28	0.36	0.37	0.46
59	0.30	0.32	0.39	0.43
60	0.32	0.42	0.21	0.46
61	0.30	0.20	0.42	0.33
62	0.34	0.38	0.21	0.34
63	0.30	0.28	0.38	0.60
64	0.35	0.37	0.37	0.29
65	0.24	0.33	0.26	0.37
66	0.50	0.66	0.26	0.47
67	0.39	0.21	0.32	0.30
68	0.27	0.26	0.30	0.41

69	0.24	0.19	0.29	0.23
70	0.34	0.33	0.35	0.23
71	0.36	0.35	0.26	0.29
72	0.34	0.47	0.31	0.31
73	0.34	0.43	0.44	0.28
74	0.33	0.36	0.30	0.27
75	0.55	0.26	0.29	0.31
76	0.45	0.28	0.30	0.25
77	0.31	0.43	0.29	0.23
78	0.39	0.18	0.32	0.29
79	0.56	0.28	0.37	0.33
80	0.33	0.13	0.32	0.37
81	0.51	0.17	0.25	0.35
82	0.51	0.31	0.41	0.27
83	0.30	0.21	0.26	0.28
84	0.30	0.41	0.30	0.44
85	0.44	0.21	0.22	0.33
86	0.38	0.25	0.33	0.37
87	0.32	0.23	0.20	0.32
88	0.40	0.48	0.37	0.32
89	0.28	0.23	0.45	0.44
90	0.21	0.27	0.26	0.23
91	0.28	0.41	0.29	0.19
92	0.39	0.14	0.18	0.48
93	0.42	0.18	0.35	0.35
94	0.68	0.35	0.38	0.36
95	0.29	0.18	0.48	0.31
96	0.29	0.23	0.21	0.25
97	0.29	0.30	0.21	0.35
98	0.43	0.18	0.40	0.30
99	0.34	0.22	0.34	0.27
100	0.34	0.46	0.26	0.28
101	0.29	0.34	0.39	0.35
102	0.30	0.26	0.26	0.36
103	0.36	0.85	0.40	0.33
104	0.43	0.37	0.22	0.37
105	0.40	0.29	0.18	0.38
106	0.34	0.22	0.37	0.33
107	0.46	0.21	0.37	0.29
108	0.33	0.28	0.30	0.42
109	0.33	0.18	0.26	0.41
110	0.32	0.21	0.37	0.23
111	0.29	0.25	0.41	0.59

112	0.36	0.23	0.27	0.36
113	0.21	0.26	0.37	0.52
114	0.32	0.31	0.41	0.23
115	0.23	0.29	0.41	0.44
116	0.26	0.22	0.50	0.43
117	0.26	0.35	0.78	0.33
118	0.83	0.29	0.61	0.48
119	0.30	0.27	0.53	0.44
120	0.28	0.17	0.39	0.44
121	0.28	0.19	0.29	0.53
122	0.27	0.18	0.35	0.52
123	0.24	0.21	0.29	0.33
124	0.34	0.28	0.40	0.27
125	0.33	0.31	0.59	0.32
126	0.27	0.21	0.38	0.31
127	0.31	0.17	0.56	0.25
128	0.56	0.23	0.41	0.29
129	0.26	0.24	0.26	0.32
130	0.30	0.25	0.24	0.31
131	0.46	0.35	0.35	0.27
132	0.42	0.28	0.26	0.29
133	0.27	0.24	0.35	0.20
134	0.59	0.22	0.35	0.40
135	0.27	0.39	0.38	0.30
136	0.24	0.21	0.50	0.35
137	0.40	0.20	0.53	0.47
138	0.36	0.24	0.41	0.28
139	0.42	0.28	0.56	0.25
140	0.55	0.39	0.43	0.25
141	0.31	0.38	0.35	0.25
142	0.40	0.34	0.40	0.22
143	0.30	0.31	0.55	0.32
144	0.25	0.29	0.59	0.31
145	0.36	0.28	0.38	0.36
146	0.28	0.27	0.58	0.40
147	0.46	0.33	0.48	0.32
148	0.41	0.29	0.34	0.39
149	0.45	0.21	0.38	0.30
150	0.45	0.39	0.38	0.25
151	0.32	0.36	0.44	0.28
152	0.40	0.39	0.56	0.37
153	0.34	0.15	0.35	0.45
154	0.40	0.29	0.35	0.30

155	0.32	0.22	0.49	0.25
156	0.33	0.95	0.47	0.26
157	0.29	0.26	0.42	0.38
158	0.36	0.23	0.29	0.57
159	0.30	0.21	0.40	0.25
160	0.28	0.25	0.34	0.33
161	0.39	0.23	0.41	0.44
162	0.38	0.31	0.56	0.35
163	0.35	0.20	0.45	0.30
164	0.48	0.38	0.48	0.26
165	0.24	0.31	0.26	0.26
166	0.24	0.35	0.30	0.29
167	0.21	0.29	0.39	0.28
168	0.42	0.41	0.33	0.22
169	0.28	0.29	0.29	0.19
170	0.33	0.20	0.37	0.29
171	0.39	0.25	0.29	0.29
172	0.30	0.35	0.47	0.32
173	0.30	0.35	0.33	0.32
174	0.33	0.41	0.42	0.34
175	0.31	0.29	0.29	0.36
176	0.30	0.30	0.41	0.26
177	0.62	0.40	0.40	0.24
178	0.22	0.39	0.65	0.42
179	0.19	0.31	0.33	0.26
180	0.56	0.24	0.38	0.34
181	0.29	0.33	0.43	0.37
182	0.36	0.41	0.47	0.21
183	0.34	0.36	0.40	0.30
184	0.59	0.39	0.56	0.33
185	0.53	0.27	0.51	0.37
186	0.34	0.33	0.46	0.22
187	0.29	0.28	0.52	0.29
188	0.40	0.39	0.25	0.31
189	0.44	0.33	0.34	0.26
190	0.43	0.21	0.25	0.28
191	0.41	0.28	0.38	0.41
192	0.26	0.17	0.32	0.57
193	0.30	0.20	0.47	0.44
194	0.21	0.50	0.43	0.26
195	0.25	0.26	0.26	0.29
196	0.28	0.37	0.26	0.53
197	0.27	0.28	0.68	0.32

198	0.39	0.21	0.26	0.35
199	0.30	0.46	0.48	0.47
200	0.36	0.35	0.35	0.17
201	0.26	0.52	0.41	0.25
202	0.44	0.34	0.86	0.20
203	0.43		0.25	0.21
204	0.34		0.26	0.30
205	0.34		0.35	0.32
206	0.43			

Bubble size measurement with 10ppm of Romax 6000 at -2 °C in a 3” flotation column

	sparger speed 6		sparger speed 8	
	bottom	top	bottom	top
average size (mm)	0.49	0.49	0.39	0.42
standard deviation	0.19	0.20	0.16	0.22
bubble #	bubble size (mm)	bubble size (mm)	bubble size (mm)	bubble size (mm)
1	1.50	1.23	0.57	0.38
2	0.41	0.66	0.37	0.41
3	0.50	0.33	0.38	0.48
4	0.46	0.55	0.28	0.58
5	0.52	0.39	0.48	0.47
6	0.61	0.50	0.53	0.40
7	0.75	0.93	0.20	0.30
8	0.65	0.83	0.52	0.76
9	0.43	0.38	1.00	0.33
10	0.54	0.46	0.86	0.36
11	0.28	0.25	0.57	0.30
12	0.45	0.27	0.28	0.46
13	0.39	0.87	0.31	0.46
14	0.51	0.40	0.33	0.41
15	0.33	0.88	0.32	0.31
16	0.67	0.43	0.43	0.85
17	0.41	0.46	0.45	0.65
18	0.32	0.74	0.40	0.28
19	0.45	0.54	0.34	0.54
20	0.68	0.35	0.45	0.43
21	0.55	0.37	0.32	0.30
22	0.52	0.41	0.46	0.28
23	0.41	0.29	0.29	0.54
24	0.97	0.35	0.55	0.34

25	0.91	0.46	0.32	0.41
26	0.37	0.50	0.26	0.54
27	0.52	0.75	0.34	0.25
28	0.61	0.61	0.60	0.35
29	0.41	0.83	0.32	1.17
30	0.29	0.33	0.29	0.50
31	0.82	0.19	0.26	0.38
32	0.41	0.29	0.34	0.35
33	0.37	0.22	0.52	0.45
34	0.45	0.96	0.28	0.43
35	0.40	0.46	0.28	0.32
36	0.62	0.48	0.41	0.38
37	0.54	0.22	0.36	0.92
38	0.38	0.33	0.69	0.39
39	0.51	0.26	0.57	0.54
40	0.35	0.73	0.45	0.45
41	0.30	0.39	0.37	0.31
42	0.39	0.50	0.48	0.41
43	0.63	0.37	0.20	0.52
44	0.56	0.49	0.33	0.58
45	0.46	0.30	0.74	0.39
46	0.32	0.29	0.57	0.31
47	0.46	0.71	0.29	0.25
48	0.35	0.87	0.24	0.30
49	0.26	0.64	0.68	0.35
50	0.23	0.46	0.63	0.31
51	0.44	0.41	0.27	0.50
52	0.34	0.29	0.37	0.52
53	0.32	0.33	0.50	0.54
54	0.41	0.61	0.78	0.65
55	0.32	0.94	0.51	0.35
56	0.59	0.50	0.36	0.32
57	1.26	0.84	0.14	0.20
58	0.30	0.39	0.34	0.27
59	0.26	0.30	0.32	0.58
60	0.42	0.25	0.47	0.22
61	0.32	0.37	0.34	0.28
62	0.47	0.41	0.29	0.31
63	0.37	0.47	0.32	0.39
64	0.76	0.61	0.36	0.61
65	0.37	0.71	0.24	0.49
66	0.30	0.33	0.32	0.64
67	0.45	0.31	0.26	0.29

68	0.42	0.29	0.27	0.33
69	0.85	0.37	0.87	0.29
70	0.41	0.29	0.29	0.41
71	0.47	0.88	0.28	0.29
72	0.51	0.42	0.31	0.41
73	0.26	0.52	0.51	0.98
74	0.42	0.41	0.48	0.51
75	0.69	0.46	0.18	0.35
76	0.43	0.37	0.34	1.00
77	0.54	0.38	0.49	0.98
78	0.60	0.37	0.36	0.77
79	0.29	0.34	0.34	0.40
80	0.17	0.25	0.37	0.42
81	0.69	0.35	0.28	0.66
82	0.39	0.52	0.48	0.54
83	0.41	0.32	0.34	0.24
84	0.59	0.35	0.40	0.35
85	0.35	0.25	0.48	0.52
86	0.46	0.48	0.33	0.27
87	0.46	0.50	0.31	0.42
88	0.46	0.18	0.23	0.35
89	0.37	1.08	0.29	0.27
90	0.42	0.32	0.32	0.86
91	0.56	0.52	0.28	0.46
92	0.41	0.98	0.18	0.57
93	0.62	0.67	0.31	0.41
94	0.60	0.57	0.31	0.75
95	0.88	0.68	0.40	0.75
96	0.54	0.27	1.05	0.62
97	0.41	0.91	0.26	0.28
98	0.56	0.37	0.37	0.23
99	0.36	0.52	0.28	0.27
100	0.60	0.24	0.37	0.16
101	0.63	0.43	0.20	0.35
102	0.37	0.37	0.90	0.19
103	0.30	0.15	0.45	0.31
104	0.38	0.58	0.40	0.31
105	0.50	0.35	0.31	0.31
106	0.42	0.37	0.18	0.27
107	0.33	0.49	0.32	0.28
108	0.43	0.34	0.31	0.38
109	0.33	0.66	0.20	0.33
110	0.33	0.70	0.31	0.22

111	0.33	0.44	0.29	0.30
112	0.50	0.39	0.26	0.26
113	0.87	0.29	0.61	0.38
114	0.26	0.37	0.36	0.39
115	0.33	0.35	0.24	0.30
116	0.46	0.12	0.24	0.23
117	0.53	0.30	0.25	0.27
118	0.28	0.22	0.47	0.99
119	0.32	1.02	0.34	0.46
120	0.66	0.82	0.45	1.07
121	0.39	0.66	0.42	0.27
122	0.62	0.41	0.68	0.19
123	0.43	0.31	0.27	0.55
124	0.46	0.26	0.31	1.36
125	0.30	0.62	0.24	0.23
126	0.50	0.46	0.29	0.29
127	0.39	0.52	0.29	0.27
128	0.45	0.41	0.50	0.27
129	0.42	0.60	0.38	0.23
130	0.32	0.38	0.60	0.23
131	0.43	0.38	0.33	0.15
132	0.92	0.65	0.26	0.21
133	0.47	0.42	0.36	0.16
134	0.43	0.53	0.23	0.25
135	0.32	0.26	0.26	0.21
136	0.39	0.33	0.46	0.17
137	0.41	0.43	0.34	0.46
138	0.39	0.90	0.36	0.44
139	0.43	0.42	0.29	0.61
140	0.43	0.37	0.29	0.41
141	1.09	0.35	0.68	0.26
142	0.92	0.49	0.47	0.44
143	0.63	0.39	0.20	0.41
144	0.31	0.95	0.34	0.16
145	0.50	0.39	0.40	0.31
146	0.33	0.34	0.38	0.27
147	0.34	0.35	0.11	0.25
148	0.33	0.38	0.23	0.84
149	0.50	0.50	0.34	0.21
150	0.50	0.22	0.25	0.50
151	0.51	0.73	0.34	0.64
152	0.33	0.61	0.28	0.31
153	0.28	0.78	0.29	0.57

154	0.48	0.39	0.31	0.38
155	0.36	0.48	0.57	0.43
156	0.85	0.56	0.29	0.28
157	0.36	0.27	0.43	0.32
158	0.50	0.42	0.61	0.27
159	0.47	0.38	0.28	0.25
160	0.58	0.47	0.15	0.14
161	0.49	0.79	0.24	0.59
162	0.56	0.59	0.42	0.22
163	0.46	0.58	0.20	0.26
164	0.43	0.41	0.28	0.40
165	0.39	0.26	0.57	0.28
166	0.19	0.58	0.34	0.36
167	0.46	0.46	0.36	0.55
168	0.52	0.41	0.40	0.35
169	0.54	0.94	0.51	0.36
170	0.44	0.50	0.50	0.73
171	0.37	0.97	0.31	0.26
172	0.36	0.69	0.51	0.45
173	0.37	0.30	0.34	0.28
174	0.42	0.40	0.46	0.25
175	0.54	0.49	0.48	0.22
176	0.76	0.41	0.52	0.26
177	0.58	0.52	0.52	0.60
178	0.33	0.34	0.51	0.41
179	0.44	0.44	0.34	0.53
180	0.24	0.37	0.58	0.22
181	0.29	0.55	0.52	0.57
182	0.35	0.53	0.27	0.44
183	0.45	0.58	0.31	0.27
184	0.58	0.52	1.13	0.35
185	0.35	0.46	0.32	0.33
186	0.64	0.61	0.25	0.36
187	0.43	0.78	0.31	0.36
188	0.67	0.66	0.41	0.36
189	0.54	0.37	0.38	0.25
190	0.59	0.56	0.55	0.33
191	0.44	0.61	0.56	0.23
192	0.45	0.54	0.36	0.30
193	0.65	0.67	0.27	1.26
194	0.47	0.29	0.27	0.47
195	0.60	0.61	0.39	0.35
196	0.42	0.37	0.39	0.28

197	1.17	0.81	0.75	0.39
198	1.01	0.88	0.49	0.70
199	0.83	0.53	0.50	0.38
200	0.75	0.53	0.39	0.57
201	0.47	0.35	0.25	0.54
202	0.63	0.69	0.22	0.35
203	0.87	0.46	0.23	0.32
204	0.54	0.32	0.31	0.41
205	0.58	0.57	0.12	1.50
206	0.44	0.67	0.28	0.50
207	0.56		0.72	0.45
208	0.58		0.26	0.28
209	0.47		0.57	0.31
210				0.31
211				0.38
212				0.54
213				0.54
214				0.73

Hydrodynamic parameters in a 3" flotation column

		No frother		15 ppm of Frother	
Temperature (°C)	Sparger setting	6	8	6	8
5	Bubble size (mm)	0.57-0.83	0.35-0.48	0.38	0.38-0.40
	Gas holdup (%)	4.6	5.9	10.9	13.5
-2	Bubble size (mm)	0.56-1.00	0.45-0.60	0.30-0.34	0.35-0.37
	Gas holdup (%)	2.4	3.1	8.3	11.0

3" Column flotation of ANS crude oil at 5 °C

Crude Conc. (%)	Flow rate (ml/min)	Custo527 (ppm)	Romax6000 (ppm)	Time (min.)	Residual oil in water (ppm)	Residual water in oil (%)
>90	200	0	0	5	5.6	9.5
				10	10.3	7.8
				20	12.3	8.9
				30	8.1	9.1
				60	3.2	7.8
60-70	300	0	0	5	1.3	45.6
				10	11.5	41.0
				20	14.3	44.0
				30	11.1	47.9
				60	9.1	32.8

		15	0	5	2.2	24.6
				10	11.1	25.6
				20	0.8	17.8
				30	5.6	22.1
				60	2.1	23.9
		0	10-50	5	3.6	11.4
				10	7.9	10.8
				20	6.5	6.4
				30	6.9	12.2
				60	4.1	7.9

3" Column flotation of ANS crude oil at -2 °C

Crude Conc. (%)	Flow rate (ml/min)	Custo527 (ppm)	Romax6000 (ppm)	Time (min.)	Residual oil in water (ppm)	Residual water in oil (%)
60-70	300	0	0	5	1.3	12.6
				10	8.4	6.3
				20	15.9	5.4
				30	12.1	5.6
				60	11.1	5.7
		15	0	5	1.6	12.6
				10	1.8	9.6
				20	2.2	6.1
				30	3.0	5.3
				60	4.3	7.6
		0	10	5	2.2	-
				10	4.2	5.7
				20	2.1	5.3
				30	2.2	6.3
				60	1.8	5.5

3" Column flotation of emulsified ANS crude oil

Oil Con. (wt%)	Frother	Frother con. (ppm)	Temp. (°C)	Time (min.)	Residual oil in water (ppm)
0.05	None	0	5	5	22.3
				10	41.0
				20	26.9
				30	18.7
				60	22.5
			-2	5	70.2
				10	50.8
				20	66.1

	Romax 6000	10	5	30	17.8
				60	30.2
				5	10.5
				10	14.4
				20	6.6
				30	8.9
			-2	60	11.7
				5	23.6
				10	28.1
				20	8.8
				30	17.9
				60	10.7

3" Column flotation of oil contaminated ice in seawater at -2 °C

Oil con. (wt%)	Ice Con. (wt%)	Frother	Frother Con. (ppm)	Ice size (mm)	Time (min.)	Residual oil in water (ppm)	
5	10	None	0	1-5	5	2.5	
					10	2.0	
					20	4.3	
					30	1.7	
					60	1.9	
				5-15	5	4.0	
					10	0.9	
					20	1.4	
					30	1.8	
					60	1.5	
		Romax 6000	10	1-5		5	2.0
						10	3.9
						20	1.4
						30	1.6
						60	1.5
				5-15		5	1.6
						10	1.5
						20	1.4
						30	2.9
						60	1.0

Bottle tests of 50 ppm of demulsifiers for breaking seawater-in-oil (water/ANS=1/2) emulsion at room temperature (no stir)

Demulsifier	Vol % of water separated			
	<i>2 min.</i>	<i>5 min.</i>	<i>15 min.</i>	<i>30 min.</i>
DM-2	1.0	4.2	5.5	7.8
RTC-330	1.0	3.8	4.6	6.5
Romax 6000	1.0	6.6	9.5	9.7
Romax 6001	5.3	5.5	6.6	10.8
Romax 6005	2.5	4.8	9.0	11.5
Romax 6020	3.1	4.8	7.8	10.8
Romax 6023	2.9	3.5	6.3	7.5
Romax 6030	2.0	4.2	5.2	10.1
Romax 6035	1.1	2.5	4.5	8.4
Romax 6080	0	4.5	4.8	4.8
Romax 6082	1.4	2.0	4.8	5.0
Romax 6100	1.2	4.5	4.8	11.5
Romax 6620	0	2.3	2.7	3.2
Romax 6630	0	1.8	2.0	2.2
Romax 6640	2.2	1.7	1.5	2.2
Romax 6650	0	1.3	1.3	1.3
Romax 6660	0	0	2.7	5.0
Romax 6668	2.0	1.8	2.3	6.0