Meso-scale Flume Testing of Dispersant Effectiveness in Frazil Ice

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EXECUTIVE SUMMARY

This report summarizes the results of meso-scale re-circulating flume tank testing of the weathering rate and chemical dispersibility of two oils spilled in frazil ice conditions. The tests were commissioned by the Bureau of Safety and Environmental Enforcement (BSEE) of the US Department of the Interior. Alaska North Slope crude oil was tested in Ottawa by SL Ross Environmental Research and Troll Blend crude oil was tested in Trondheim by SINTEF. The weathering rates and chemical dispersibility of the two oils when spilled in frazil ice conditions are reported.

ANS crude oil weathered for 96 hours in frazil ice, with evaporative losses estimated at 25% to 30% by mass, was difficult to disperse, with efficiencies measured between 1.6% and 6.2% in high energy waves, and between 1.6% and 12.5% in prop wash.

Fresh ANS in frazil ice dispersed between 9% and 49% in high energy waves, and from 25% to 59% in prop wash. Evaporated ANS (17.4% by mass) dispersed between 0% and 19% in high energy waves, and from 8 to 20% in prop wash.

Fresh Troll Blend in frazil ice dispersed between 40% and 83% in high energy waves, and from 67% to 86% in prop wash. Evaporated Troll Blend (22.0 % by mass) dispersed between 45% and 53% in high energy waves, and between 47% and 61% in prop wash.

The frazil ice thickness did not have a significant impact on the evaporative process in these tests, compared to an ice-free scenario, because the majority of the oil quickly migrated to the surface of the ice layer under the low energy conditions that the test tanks were subjected to during the four-day weathering periods. Evaporative losses in frazil ice have been shown to be similar to open water losses as modeled using SINTEF's oil weathering model (OWM).

In contrast, changes in oil viscosity and emulsification (i.e., water uptake) measured in the weathering experiments compared more favourably with simulations using high broken-ice coverage. This is likely due to the similar wave dampening effects between frazil and high concentrations of broken ice.

Ice layer thickness influenced the effectiveness of dispersants on the oils as it generally dampened the energy available to disperse the oil. Weathered oil was not as readily dispersed as fresh oil and resulted in larger oil drop sizes, as would be expected. Oil layer thickness over the range tested did not affect the dispersion process appreciably.



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1. INTRODUCTION

In the event of an offshore oil spill rapid decisions must be made with regard to the best course of action to mitigate potential effects of the spillage. Chemical dispersants are one of the tools to be considered. The initial question to be answered when deciding if dispersant application is an appropriate action is "Will the dispersant be effective in removing a significant percentage of the oil from the surface?" If not, then the dispersant operation would not be considered. If chemical dispersants are deemed to be potentially effective other issues such as a) the logistical feasibility of getting the dispersant to the spill site; b) applying it at the appropriate dosage; and c) the net environmental benefit of using the dispersant use.

Oil exploration activities in the United States are extending into the Chukchi and Beaufort Sea OCS regions where ice conditions of various types exist during the year. Oil spill response planners in these areas need to know whether oil spilled in these regions during periods when ice is present in various forms can be chemically dispersed.

1.1 BACKGROUND

Early research on dispersant effectiveness in ice-infested waters included studies by Mackay et al. 1980; Cox and Shultz 1981; Byford et al. 1983; Brown et al. 1985; and Brown and Goodman 1996. These studies ranged from experiments in small containers, to medium sized-tank experiments, to experiments at a large wave basin in Calgary, Alberta, Canada. More recent work includes tests by SL Ross for ExxonMobil in their wave tank (SL Ross 2005, 2006a) and at the Ohmsett facility (SL Ross 2002, 2006b), and for BP at the Aker ice basin in Finland (SL Ross 2006c). SINTEF has completed dispersant studies with ice in their re-circulating flume (Brandvik et al., 2010), and performed field tests of dispersant application on oil released in the Marginal Ice Zone (MIZ) in the Barents Sea (e.g. Singsaas et al., 1994; Daling et al., 2010).

The overall results of the studies completed to date suggest that ice has a dual effect on the energy available for oil dispersion. One effect is to dampen the wave motion and the other effect is the pumping action imparted to the oil between colliding ice pieces. At low energy levels the damping effect may be more important, while at higher energy levels the pumping action may override the damping effect and generate enough mixing energy to disperse chemically treated oil. In any case, the conclusion from the studies is that the level of dispersant effectiveness in an ice situation will strongly depend on the particular ice regime under consideration. The major factors are the level of surface mixing energy that is available to first of all break up the oil into small droplets and then the energy available to mix and hold the dispersed oil drops in the underlying body of water.

In January 2012, the International Oil and Gas industry launched the Arctic Oil Spill Response Technology Joint Industry Program (JIP) to enhance Arctic oil spill capabilities under the auspices of the International Association of Oil and Gas Producers (OGP). One of the research topics that this program is studying is the use of chemical dispersants in ice-infested waters¹. As part of this research, SINTEF

¹ Details concerning this project can be found at http://www.arcticresponsetechnology.org/



and SL Ross have completed an extensive matrix of dispersant effectiveness tests in their identical meso-scale re-circulating flume tanks, with various oils in broken ice conditions (i.e., chunks of ice at least several centimeters in size and larger).

While the work being conducted under the OGP project addresses some of the concerns identified by BSEE regarding dispersant use under realistic conditions (Faksness et al., 2017), there was an opportunity to extend the investigations to other ice condition scenarios that have not previously been studied. One area that is not being investigated in the OGP study is dispersant use in slush or frazil ice.

Frazil ice is the first stage in the formation of sea ice and resembles soupy slush. It forms in the ocean when air temperatures are at or below -6°C, and the surface water loses heat to the cooler air above. The frazil ice starts as small crystals, which then rapidly grow in size and number. Turbulence in the water due to waves or current initially prevents the formation of a sheet of ice; however, if the air temperature remains cold for a long enough period, the frazil ice crystals will fuse together into a solid sheet (pack ice).

1.2 OBJECTIVE

The objective of the project was to advance the understanding of the effectiveness of chemical dispersants when applied to oil spilled in ice-infested waters, specifically in slush or frazil ice conditions. The goals of the research were to:

- 1. Conduct weathering studies at meso-scale to establish the rate of change of oil properties when spilled in frazil ice;
- 2. Conduct dispersant effectiveness tests at meso-scale to determine the potential for the use of chemical dispersants on both fresh and weathered oil in the presence of frazil ice.

The project was designed to provide the best incremental improvement in the collective understanding of dispersant use in ice.



2. MATERIALS AND METHODS

2.1 TEST OILS AND DISPERSANT

The tests conducted by SL Ross used Alaska North Slope (ANS) crude oil. The tests conducted by SINTEF used Troll Blend crude oil. The physical properties of the fresh test oils are presented in Table 2-1 below.

Crude Oil	Density (g/mL)	Viscosity (cP, 100 s-1)	Pour Point (°C)
Alaska North Slope	0.855 (15.5°C)	22 (2°C)	-30°C
Troll Blend	0.874 (20°C)	40 (0°C)	-18°C

All tests were conducted using Corexit 9500 dispersant applied at a 1:20 dispersant-to-oil ratio (by volume).

2.2 TEST TANKS

The oil weathering and dispersant effectiveness tests in frazil ice were conducted in meso-scale recirculating flumes at SL Ross and SINTEF. The flumes are identical in size and construction, with an outer circumference of 16.6 m, a width of 0.5 m, and a height of 1.5 m. A schematic drawing of the flumes is presented in Figure 2-1. The flumes were filled with salt water (nominally 35 ppt) to a depth of 1 m, for a total water volume of 4,800 L. The temperature of the water during the tests averaged -0.5°C for both the SINTEF and SL Ross tests. The temperature of the air above the water in the tank averaged 9.5°C during the SL Ross tests, and -0.6°C during the SINTEF tests.



Figure 2-1: Sketch of SINTEF and SL Ross meso-scale recirculating flumes.

The flumes are equipped with a wave maker and a fan, as shown, to produce wave action and wind during the oil weathering portion of the tests. Two wave maker settings were used to produce two



different wave energy levels. The wave maker settings are provided in Table 2-2. Low energy waves were used during the weathering portion of the experiments, while high energy waves were used after dispersant was applied to the test oil slicks.

Table 2-2: Wave paddle settings for low and high energy waves

	Application	Wave Paddle Frequency	Wave Paddle Amplitude		
High Energy Wave	Dispersant Testing	зо rpm	16 cm		
Low Energy Wave	Oil Weathering	24 rpm	12 CM		

The recirculating flumes are not equipped with wave absorbers, and the interference patterns that build up can be complex. The presence of ice significantly dampened the waves. Low energy waves were roughly equivalent to gentle swells on the ocean – enough to move the oil and ice, but not enough to cause emulsification. The high energy waves were roughly equivalent to a harbor chop, and would be able to disperse a treated surface slick of dispersible oil in ice-free conditions.

A 1 m² confinement area was set up with removable barriers to contain the oil in one section of the flumes during long-term weathering tests and prior to dispersant application.

A propeller was installed at point A in Figure 2-1 (MinnKota Endura 30 electric trolling motor with twinblade propeller) to provide additional agitation during the prop wash portion of the dispersant effectiveness tests (see Section 2.5). The propeller was inserted into the tank by the operator and held approximately 20 cm below the surface of the water.

A LISST particle size analyzer (Sequoia Scientific model 100x) was used to characterize the droplet sizes and measure oil concentration in the dispersed oil plume. The LISST was positioned 50 cm below the water surface upstream of the containment area at point B in Figure 2-1. The LISST measures particles between 2.5 and 500 µm in diameter in real time using laser diffraction.



Figure 2-2: LISST 100X Type C, mounted in flume



A sampling tube to collect water samples from the tank was located 50 cm below the water surface, at point B in the diagram.

2.3 FRAZIL ICE PRODUCTION

Due to differences in facilities and equipment between SL Ross and SINTEF, two different methods were used to prepare frazil ice for the weathering and dispersant effectiveness tests, as discussed below. The typical appearance of the frazil ice that was produced is shown in Figure 2-3.



Figure 2-3: Frazil ice produced at SINTEF

2.3.1 SL Ross Frazil Ice Production Procedure

Frazil ice for the tests conducted by SL Ross was prepared in a separate tank: the SL Ross wind/wave tank, which measures 11 m long, 1.2 m wide, and 1.2 m high. The tank was filled with water to a depth of approximately 85 cm, at a salinity of 5 ppt. Full ocean salinity water was not used to prepare the frazil ice, since this would have required a lower temperature to form ice, and most of the salt is not retained in the ice after it crystallizes so this does not significantly affect the characteristics of the ice produced.

The tank chiller was set to lower the temperature of the water to o°C, and the wind chiller was set to bring the air temperature over the water to -10°C. A propeller was set in the tank to agitate the water and help produce the super-cooled conditions necessary for the formation of frazil ice. The frazil ice that formed was collected immediately prior to use in the weathering and dispersant effectiveness tests that were conducted in the recirculating flume.

2.3.2 SINTEF Frazil Ice Production Procedure

Frazil ice for the tests conducted by SINTEF was prepared using 35 ppt salinity water in an 800-L tank located inside a refrigerated chamber (see Figure 2-4). The tank was equipped with a mechanical stirrer that was operated while the water cooled and frazil ice was formed. The ice was transferred to the recirculating flume immediately prior to use.





Figure 2-4: Frazil ice tank at SINTEF

2.4 96-HR OIL WEATHERING

To our knowledge no research on the weathering rate of crude oil mixed with frazil ice has previously been conducted (e.g., Lewis and Daling, 2007). It was not clear from the available literature how the presence of small ice particles would affect the rate of weathering and the resulting changes in oil properties. This lack of knowledge is important from both a spill modeling perspective, and a response planning perspective. To address this, oil weathering experiments were conducted in the recirculating flumes.

Frazil ice conditions are unlikely to persist for extended periods in nature, as it is a transitional phenomenon. Consequently, it is unlikely that the opportunity for dispersant use in frazil ice conditions would exist for more than a few days. This puts a limit on the duration required for weathering experiments to adequately simulate exposure to a frazil ice environment. It was estimated that four days (i.e., 96 hours) would be a conservative upper bound on such a scenario.

Weathering tests with Troll Blend and Alaska North Slope crude oils were conducted in the 1-m² containment zones in one of the straight sections of the recirculating flumes. Frazil ice was used in contact with the oil in the containment zone, while machine-made ice from a commercial ice flaker, which was similar in size and shape to the frazil ice, was used in the remainder of the flume. The machine-made ice was not in contact with the oil, and acted only as a thermal buffer to reduce the overall rate of ice melting in the flume. A photo of the oil and ice in the tank is provided in Figure 2-5.



Figure 2-5: ANS crude oil and ice in the SL Ross flume tank (left) and Troll Blend crude oil in the SINTEF flume tank (right).

The tests were done using a low energy wave environment (approximately equivalent to gentle swells in an ice field situation), one water temperature (-2°C), one wind speed (1.2 m/s), and one frazil ice thickness (10 cm).

A number of bench scale tests were conducted prior to the weathering tests to evaluate the behaviour of oil mixed into a moving layer of frazil ice. These tests demonstrated that the majority of the oil migrates to the surface of the ice-water layer regardless of the ice thickness. For this reason, it was decided to use only one ice thickness in the weathering tests.

Three oil loadings of 1, 2 and 5 L of oil in the containment zone were investigated. The middle oil loading was duplicated to assess repeatability of the weathering method (four tests in total). Vapours were drawn away from the air space above the oil using a low velocity vent connected to the laboratory exhaust systems. The test matrix is summarized in Table 2-3.

Test ID	Oil Loading	Frazil Ice Thickness
1L	1 L	10 cm
2 L A	2 L	10 cm
2 L B	2 L	10 CM
5 L	5 L	10 CM

Table 2-3: Test matrix for oil weathering experiments in frazil ice with ANS and Troll Blend crude oils.

The weathering tests continued for a total of 96 hours. During this period, frazil and machine-made ice in the tank was replenished as necessary to maintain the 10 cm thickness. Oil samples were collected during the weathering test and analyzed for density, viscosity, water content, and gas chromatography (GC) analysis at the following times: 3, 6, 18, 24, 48, 72 and 96 hours.



Water content was measured by placing the oil sample of in a 40-mL clear glass vial and heating it in a water bath at 70°C. The amount of free water that separated out was measured by height and compared to the height of oil and the initial height of water to calculate water content in the oil.

The test oils had previously been characterized for spill modeling purposes, and the relationship between evaporative loss and increasing density was known. The density of the samples was used to estimate the evaporative loss of the test oils at each sampling time. The density vs. evaporative loss relationships for the crude oils is provided in Appendix B.

2.4.1 Oil Spill Modeling

The results of the weathering tests and the changes in evaporative loss and viscosity were compared to modeling simulations completed using the SINTEF oil weathering model (OWM), in order to quantify the effects of the frazil ice on the weathering process.

The SINTEF OWM predicts oil properties for the bulk phase of a surface oil slick as a function of weathering time at sea. These predictions are based on the algorithms in the model, the properties of the actual oil (data from a weathering study) for selected environmental conditions (sea state, sea temperature, and ice concentration). The model was developed during several research programs in the late eighties and early nighties (Daling et al., 1989; Aamo et al., 1993) and has been verified through several large-scale experimental field trials (Daling and Strøm, 1999). The model has been used in several previous oil weathering projects (e.g., Daling et al., 1990; Ø. Johansen, 1991; Daling et al., 2014).

The SINTEF OWM includes the ability to model oil weathering in partial ice cover scenarios, based on data from previous laboratory and field studies. Weathering properties of four oil types spanning a large variation on oil properties (paraffinic, asphaltenic, waxy and naphthenic) was studied in the same weathering flume as used in this study with varying ice coverage from open water to 90% (Brandvik et al., 2010). Increasing ice coverage was simulated by filling the flume with ice blocks and reducing the wave action to simulate wave dampening expected to find in a denser ice field.

In addition to the laboratory flume experiments at SINTEF's SeaLab facility described above (10 L of oil for 5 days), larger experiments were performed in an ice basin cut out in the sea ice outside SINTEF's field station at Svea, Svalbard. In these experiments the oil (200 L) was weathered for 48 hours in different ice conditions. A field experiment was also performed under dynamic ice conditions in the Barents Sea, southeast of Svalbard, to verify the main findings from the basin studies (7,000 L for six days). The generated laboratory, basin and field data were used to calibrate and implement oil weathering as a function of dynamic ice coverage in the SINTEF OWM (Brandvik et al., 2010).

2.5 DISPERSANT EFFECTIVENESS

Three different assessments of dispersant effectiveness were conducted:

- Field effectiveness tests;
- Meso-scale dispersant effectiveness tests on the 96-hour weathered ANS remaining at the end of the four on-tank weathering tests; and,
- Meso-scale dispersant effectiveness tests on fresh and intermediate weathered Troll Blend and ANS crude oils.

2.5.1 Field Effectiveness Tests

Field effectiveness tests (FET) were completed on a subset of samples of oil from the weathering tests, which were collected at the same time as those for testing the density and viscosity, to assess how the weathering was affecting the dispersibility. The FET is a simple qualitative method to evaluate the effectiveness of a dispersant on a given oil. The method compares the behaviour of a sample of oil dosed with dispersant against a blank sample of oil alone when they are agitated gently. The test procedure is as follows:

- Two 100-ml glass cylinders are filled with 80 mL of seawater, to which is added 1.5 mL of the test oil.
- Sixty micro-liters of dispersant is added to one of the cylinders (1:25 dispersant to oil ratio).
- No dispersant is added to the second cylinder (it is used as a reference to assess natural dispersion).
- After one minute of contact time between oil and dispersant, both cylinders are gently inverted and returned upright thirty times over one minute (i.e., 30 rpm).

After the agitation, the cylinders are observed and the resulting dispersion is characterized visually. The following general criteria for dispersant effectiveness are used:

- Good Dispersion: formation of small oil droplets (brown dispersion) that will only very slowly rise to the surface at a standstill.
- Reduced Effectiveness: formation of dark/black large oil droplets that quickly rise to the surface.
- Poor Effectiveness: little or no difference from the untreated oil (reference oil). Fast rising of large oil droplets to the surface.

2.5.2 Meso-Scale Dispersant Effectiveness Tests 96-hr Weathered ANS

Meso-scale dispersant effectiveness tests were conducted in the flumes following the four 96-hour weathering tests with ANS crude oil. The protocol for the tests was as follows:

- 1. Following the final sampling of the weathered ANS (at 96 hours), Corexit 9500 dispersant was applied at a nominal rate of 60 mL of per L of oil with a commercial sprayer (Wagner 450 with a 0.5-mm nozzle) to the cordoned-off section of the tank;
- 2. the temporary barriers were removed and the wave paddle was operated at a high setting for 30 minutes; and,
- 3. propeller energy from an electric trolling motor was added to the tank for an additional 30 minutes while high waves continued to operate.

The high energy setting represents the high end of the wave energy that might typically be encountered in an ice field or at an ice field edge. The propeller effect is meant to simulate the addition of energy from the propeller of a vessel, if it is used to add mixing to the treated oil slick.

The LISST particle size analyzer was operated during the dispersant effectiveness tests to measure the oil concentration and droplet size distribution the resulting dispersions over the course of the experiments. As well, oil concentration in water was measured from water grab samples (nominal 1 L) collected from the tank using the sampling tube at point B at the following times:



- immediately prior to dispersant application (background sample);
- after the 30 minutes of high waves; and,
- between 10 and 15 minutes of prop energy and high waves.

The water samples were extracted with dichloromethane and the extracts analysed with a spectrophotometer to quantify the amount of dispersed oil in the water column, and to calculate the dispersant effectiveness. Dispersant effectiveness was calculated as the percentage of the initial oil mass that was dispersed into the water column.

The objective of these tests was to assess the dispersibility of ANS and Troll Blend crude oils at the upper bound of weathering in frazil ice.

2.5.3 Meso-Scale Dispersant Effectiveness Tests, Fresh and Weathered ANS and Troll Blend Crude Oils

Meso-scale dispersant effectiveness tests were conducted on fresh and intermediate weathered Troll Blend and ANS crude oil samples. The test protocol was based on the dispersant effectiveness testing in broken ice conditions completed for the Arctic Oil Spill Response Technology Join Industry Programme (Faksness et al., 2017), but using frazil ice instead of larger ice pieces.

Based on the limited effectiveness of the dispersant tests on the 96-hour weathered ANS crude oil slicks, it was decided to use less weathered samples for this phase of dispersant effectiveness tests in the flume tank. The intermediate weathered tests were conducted with the oil weathered to the same degree as was reached after approximately 18 to 24 hours of weathering in the tank, being nominally 26% for Troll Blend crude oil, and 18% for ANS crude oil (by mass).

The protocol for the tests was as follows:

- 1. a 1-m² section in the straight portion of the tank was isolated with removable barriers extending several cm into the water column;
- 2. the cordoned-off section of the tank was filled with frazil ice to the desired depth, and was distributed by hand as evenly as possible;
- 3. machine-made flaked ice was placed in the remainder of the tank to the same depth;
- 4. the specified volume of oil was added to the isolated section of the tank and gently mixed into the ice to produce an even distribution of oil;
- 5. the wave paddle was operated at a low setting for one hour, to allow the oil to naturally distribute within the ice (it was observed to quickly rise through the frazil ice to the surface);
- 6. Corexit 9500 dispersant was applied at a nominal rate of 60 g of per L of oil with a commercial sprayer (Wagner 450 with a 0.5 mm nozzle) to the cordoned-off section of the tank;
- 7. the temporary barriers were removed and the wave paddle was operated at a high setting for 30 minutes; and,
- 8. propeller energy from an electric trolling motor was added to the tank for an additional 30 minutes while high waves continued to operate.

The machine-made ice was not in direct contact with the oil, except at the upstream and downstream edges of the cordoned-off section of the tank. The machine-made ice served as a place-holder and thermal buffer.



The LISST particle size analyzer was operated during the dispersant effectiveness tests to measure the oil concentration over time and the oil droplet size distributions in the resulting dispersion. As well, water grab samples (nominal 1 L) were collected from the tank at the following times:

- immediately prior to dispersant application (background sample);
- after the 30 minutes of high waves; and,
- between 10 and 15 minutes of prop energy and high waves.

The water samples were extracted with dichloromethane to quantify the amount of dispersed oil in the water column, and to calculate the dispersant effectiveness. Dispersant effectiveness was calculated as the percentage of the initial oil mass that was dispersed into the water column.



3. **RESULTS AND DISCUSSION**

3.1 OIL WEATHERING TESTS

SL Ross conducted four weathering tests with Alaska North Slope (ANS) crude oil, and SINTEF conducted four similar tests with Troll Blend (Troll) crude oil. Each test involved weathering a sample of crude oil in a meso-scale flume with frazil ice over a period of four days. The tests were conducted using three oil-to-ice ratios, a low wave energy (equivalent to swells in an ice-field scenario), with air temperature, water temperature, wind speed and ice thickness held constant. Samples of the oil were collected at regular intervals over the four days and analysed to determine density, viscosity, emulsion formation and dispersibility.

3.1.1 Evaporative Loss

The density of the oil samples was used to calculate the evaporative loss of the test slicks, based on the determined relationship between density and weathering for each crude oil. Figure 3-1 and Figure 3-2 show the evaporative loss over time for the weathering experiments with Troll and ANS crude oils, respectively.



Figure 3-1: Evaporative loss over time for Troll Blend crude oil





Figure 3-2: Evaporative loss over time for ANS crude oil

In general, the amount of oil weathering varied inversely with oil loading (i.e., oil film thickness), as expected. GC traces of the oil samples over time showed the expected reductions in the lighter components of the oil over time. GC traces are provided in Appendix A.

There were some anomalies with the measured density and viscosity data, such as for the 72-hour 1-L and 2-LA Troll blend and ANS tests. It was difficult to obtain a representative sample of the oil from the tank without including water or ice, and some excess water may have affected the analyses. However, the general trend of the measurements of density and viscosity over the course of the experiments was reasonable.

3.1.2 Emulsification

The water content of the recovered oil samples was measured by treating the oil with emulsion breaker and applying heat, and measuring the amount of water that was released (if any). Figure 3-3 and Figure 3-4 show the water content over time for Troll and ANS crude oils, respectively.



Figure 3-3: Water content over time for Troll Blend crude oil





Figure 3-4: Water content over time for ANS crude oil

Emulsion formation and water content was found to vary inversely with oil loading (i.e., lower film thickness and higher weathering), as expected. Both oils had a maximum water content of approximately 50% occur mid-way through the test, and then declined slightly in subsequent samples.

There was some variation in the water content measured between the two repeat tests for both crude oils, which may be a reflection of the difficulty in collecting representative samples from a thin oil film in ice. Some water and ice was invariably collected with the sample that was not part of the emulsion water content and it was difficult to distinguish between the 2 water sources. We attempted to account for this by measuring the oil and water layer approximately 1 hour after sampling to identify the amount of free water sampled. We then measured the water and oil layers after the sample had been treated with an emulsion breaker and placed in a hot water bath for at least 24 hours. The initial free water layer is subtracted form the final total water layer to get the final emulsion water content; however, with oils that form an unstable emulsion, such as ANS crude oil, it is very difficult to get accurate water content measurements under these test conditions.

3.1.3 Viscosity

Viscosity of the oil samples was measured and reported at a shear rate of 10 s⁻¹ and a temperature of 2°C. Figure 3-5 and Figure 3-6, below, show the viscosity over time for Troll Blend and ANS crude oils, respectively.





Figure 3-5: Viscosity over time for Troll Blend crude oil





As expected, viscosity of the crude oils increased over time, although there were fluctuations outside of the general trend for some of the samples likely due to the uneven makeup of the surface oils and the difficultly in getting samples that reflect the average condition of the oil at each time period.

3.1.4 Summary of Oil Weathering Results

The measured properties of the recovered oil samples of Troll Blend and ANS crude oils are provided in **Error! Reference source not found.** and **Error! Reference source not found.**, respectively.

As was expected, the tests with lower oil loading, and therefore thinner oil films, showed higher weathering and emulsification than the tests with higher oil loading. There was generally good agreement between the repeat tests with 2 L of oil, for both crude oils. We note that the viscosities of the weathered samples were measured at a shear rate of 100 s⁻¹, whereas the oil property data was reported at 10 s⁻¹ (see Section 2).



Time		Densit	ty (g/mL)			Water co	ntent (%)		Viscosity 2°C, 10 s ⁻¹ (cP)			
(hrs)	۱L	2 L A	2 L B	5 L	۱L	2 L A	2 L B	5 L	1L	2 L A	2 L B	5 L
0	0.855	0.855	0.855	0.855	0	0	0	0	34	34	34	34
3	0.877	0.866	0.880	0.875	20	0.1	2.5	1.9	995	158	378	335
6	0.885	0.874	0.881	0.879	21	5.7	3.7	1.5	1842	442	304	441
18	0.892	0.888	0.890	0.883	5.6	12	11	1.8	1005	265	668	403
24	0.894	0.888	0.891	0.884	20	5.5	8.2	0.1	1225	1148	789	463
48	0.898	0.894	0.896	0.890	53	26	22	3.2	3581	2264	1251	654
72	0.901	0.895	0.899	0.891	47	20	26	5.6	1881	3400	1397	743
96	0.900	0.897	0.900	0.893	30	14	24	11	2356	2774	1922	931

Table 3-1: Measured properties of Troll Blend crude oil samples.

Table 3-2: Measured properties of ANS crude oil samples

Time		Densit	y (g/mL)			Water C	ontent (%)	Viscosity 2°C, 10 s ⁻¹ (cP)			
(nrs)	۱L	2 L A	2 L B	5 L	۱L	2 L A	2 L B	5 L	۱L	2 L A	2 L B	5 L
0	0.874	0.874	0.874	0.874	0	0	0	0	40	40	40	40
3	0.900	0.898	0.897	o.886	24.1	1.9	0	4.4	275	196	137	78
6	0.906	0.902	0.904	0.893	25.5	1.8	0	3.8	398	341	301	155
18	0.917	0.912	0.910	0.903	41.7	0	17.1	2.8	3070	1051	515	337
48	0.924	0.920	0.916	0.907	50.9	4.7	29.4	0	7580	2970	3212	739
72	0.930	0.936	0.921	0.914	36.2	34.7	34.7	8.3	6231	12180	7064	1069
96	0.925	0.929	0.929	0.917	42.9	21.9	32.1	8.5	6509	5798	5636	1231

3.1.5 Field Effectiveness Test Results

The SINTEF Field Effectiveness Test (FET) was used on selected samples to assess the dispersibility of the weathered crude oils over time. The results of the FET are shown in Table 4, below. The FET test showed that all of the test samples were considered dispersible, with the exception of the 1L test with Troll Crude Oil, which was showing reduced dispersibility at the end of the test.

	Troll Crude Oil				ANS Crude Oil				
Time (hrs)	۱L	2 L A	2 L B	5 L	۱L	2 L B	5 L		
0	NA	NA	NA	NA	Good	Good	Good		
3	NA	NA	NA	NA	Good	Good	Good		
6	NA	NA	NA	NA	Good	Good	Good		
18	NA	Good	NA	NA	Good	Good	Good		
24	NA	Good	Good	NA	NA	NA	NA		
48	NA	Good	Good	NA	Good	Good	Good		
72	NA	Good	Good	NA	Good	Good	Good		
96	Good/reduced	Good	Good	Good	Good	Reduced	Good		

NA is not analysed



3.1.6 Preliminary Dispersant Effectiveness Results for 96-hour Weathered ANS

Corexit 9500 dispersant was applied to the slicks of ANS crude oil at the end of each weathering test, at a ratio of 1:20 by weight of initial oil. The slicks were then subjected to 30 minutes of high energy waves, followed by 30 minutes of high energy waves plus prop wash. Grab samples of tank water were collected at the end of the waves and the end of the waves and prop, and extracted with dichloromethane to determine the concentration of dispersed oil in the water column. Dispersant effectiveness for the ANS tests is provided in Table 3-4, below.

	Waves	Prop
1 L	1.6%	1.6%
2 L A	2.4%	3.4%
2 L B	6.2%	12.5%
5 L	2.0%	3.9%

Table 3-4: Dispersant effectiveness results for 96-hour weathered ANS

The dispersant effectiveness results were low for all tests. The FET results indicated that the oils were generally considered to be dispersible at the end of the weathering period, so we conclude that the presence of frazil ice significantly reduces the energy level of the experimental environment, and reduces the effectiveness of the dispersant.

3.1.7 Oil Weathering Model

In this section the weathering of the two oils Troll Blend (Troll) and Alaska North Slope (ANS) in frazil ice is compared to the weathering in dynamic ice with varying ice coverage (0-90%). This is done by comparing the weathered oil property data from the frazil ice experiments (average for all thicknesses) with predictions from the OWM (both oils) and with experimental basin and field data (where available for the Troll oil).

The oil weathering simulations are based on a batch release of 20 tons of oil, released over 15 minutes. The water temperature is set to -1.8°C and the wind speed to 8 m/sec. This gives a rapid spreading and the stable, initial film thickness is established very quickly (i.e., within a few minutes). The terminal film thickness without any ice is 1 mm. The film thickness for the broken/dynamic ice scenarios is a function of the ice-coverage, as follows: 50% - 1.21 mm, 60% - 1.5 mm, 70% - 2.0 mm, 80% - 2.8 mm and 90% 4.0 mm. These thicknesses are different from the arithmetic numbers and are based on calibration from experimental data.

The frazil ice weathering data (averaged values) from the 96-hour tests are compared with predictions from the SINTEF OWM for dynamic broken ice conditions, with ice coverage varying from 0 to 90% (Figure 3-7 to Figure 3.11).





Figure 3-7: Evaporative loss of ANS in 96-hr flume tests and model predictions with varying ice coverage





The reduced evaporative loss as a function of increased ice coverage is caused by increased oil film thickness when the surface area is occupied by an increasing amount of ice. This is not seen for the frazil ice experiments, when comparing them with the predictions for high ice coverage (see Figure 3-7 and Figure 3-8). The high evaporative loss for the frazil ice experiments (similar to open water), are expected since most of the oil is observed floating on top of the frazil ice. This is different compared to the dynamic broken ice conditions where the oil is mainly floating on the water between the ice sheets.

The evaporative loss for the Troll crude used in the experiments is significantly higher than predicted. This is likely caused by the differences in content of light components between the Troll oil used as basis for the SINTEF OWM simulations (from 2000) and the version used for the experimental work (from 2015). It is common for crude oils to change over time, as the oil in the formation changes, or the blends of the constituent crude oils change.





Figure 3-9: Water content of ANS in 96-hr flume experiments and model predictions with varying ice coverage



Figure 3-10: Water content of Troll Blend in 96-hr flume experiments and model predictions with varying ice coverage

For emulsification (water uptake and emulsion viscosity), a better fit is observed between the frazil ice experiments and simulations of high ice coverage with dynamic broken ice. This agreement is likely due to the similar wave dampening effects caused by both a frazil ice layer and high concentrations of dynamic broken ice, which would tend to reduce emulsification and lower slick viscosity.





Figure 3-11: Viscosity of ANS from 96-hr flume experiments and model predictions with varying ice coverage



Figure 3-12: Viscosity of Troll Blend from 96-hr flume experiments and model predictions with varying ice coverage

3.2 Meso-Scale Dispersant Effectiveness Tests

Based on the limited effectiveness of the dispersant tests on the 96-hour weathered ANS crude oil slicks, it was decided to use less weathered samples for next phase of dispersant effectiveness tests in the flume tank. Both crude oils were tested at near fresh conditions, being allowed to weather in the tank for one hour in ice and waves prior to applying dispersant. A second set of tests was done with the oil weathered to the same degree as was reached after approximately 18 to 24 hours of weathering in the tank, being nominally 26% for Troll Blend crude oil, and 18% for ANS crude oil (by mass).

3.2.1 Dispersant Effectiveness

The results of the dispersant effectiveness tests with Troll Blend crude oil are presented in Table 3-5, below. The effectiveness was calculated from the tank water grab samples collected at the end of the high energy wave period, and in the middle of the propeller wash period (prop wash).



lce Thickness	Oil Weathering	Oil Volume	High Energy	Prop Wash
5 cm Frazil	Fresh	1L	83%	86%
		2 L	74%	74%
	22.0 % (wt.)	1 L	53%	61%
		2 L	50%	47%
10 cm Frazil	Fresh	1 L	81%	79%
		2 L	40%	67%
	22.0 % (wt.)	1 L	45%	47%
		2 L	45%	51%

Table 3-5: Dispersant effectiveness with Troll Blend crude oil

The dispersant effectiveness results are shown on Figure 3-13.



Figure 3-13: Dispersant effectiveness with Troll Blend crude oil

As expected, the results show that the dispersant efficiency of fresh oil is higher than weathered oil. The dispersant effectiveness results in the test with 2 L of fresh oil in 10 cm of frazil ice are lower for the high energy waves than what would be expected based on the other results; however, when prop wash energy was added, the dispersant effectiveness improved.

The results of the dispersant effectiveness tests with ANS Crude Oil in Frazil Ice are presented in Table 3-6, below. The effectiveness was calculated from the tank water grab samples collected at the end of the high energy wave period and in the middle of the propeller period.



lce	Oil	Oil	High Energy	Prop Wash	
Thickness	Weathering	Volume			
5 cm Frazil	Fresh	۱L	49%	59%	
		2 L	27%	32%	
	17.4 % (wt.)	1 L	19%	20%	
		2 L	19%	20%	
10 cm Frazil	Fresh	1 L	9%	23%	
		2 L	24%	34%	
	17.4 % (wt.)	1 L	٥%	6%	
		2 L	5%	6%	

Table 3-6: Dispersant effectiveness with ANS crude oil

The dispersant effectiveness results are shown on Figure 3-14, below.



Figure 3-14: Dispersant effectiveness with ANS crude oil

Higher dispersant effectiveness results were achieved with the fresh ANS, compared to the 17.4% weathered ANS. This is expected, due to the higher viscosity of the weathered oil, which would resist dispersing; however, both oils are considered to be completely dispersible under ice-free conditions based on the FET conducted. In a tank without ice as is simulated in the FET test, we would expect to see dispersant effectiveness approaching 100%, even for the 17.4% weathered ANS.

The propeller energy when added after 30 minutes of high energy waves generally increased dispersant effectiveness by a small margin of between 5 to 15%. The addition of propeller energy tended to have a reduced effect on the weathered oil than on the fresh oil, particularly on the tests in 10 cm of ice.

The overall results show a clear trend of decreasing dispersant effectiveness with ice thickness for both Troll Blend and ANS crude oils. This is illustrated in Figure 3-15 and Figure 3-16, below.







Figure 3-15: Dispersant effectiveness with ice thickness for Troll Blend crude oil



Figure 3-16: Dispersant effectiveness with ice thickness for ANS crude oil

3.2.2 Oil Droplet Size Information

The following descriptive statistics were calculated from the LISST particle size data for each test:

- the average dispersed oil concentration (ppm);
- the 50^{th} percentile oil droplet size (d50, μ m).

In addition, the percentage by volume of dispersed oil present in droplets smaller than 75 μ m (%V < 75 μ m) was calculated for the tests with ANS crude oil. 75 μ m is considered to be the droplet diameter cutoff below which oil droplets are considered to be permanently dispersed. These statistics were calculated over two periods of approximately 8 minutes, at the end of the high energy wave and propeller periods, respectively. The data for the tests is presented in Table 3-7 and Table 3-8, below. Note that no LISST data was available for the test with 2 L of weathered Troll Blend crude oil in 5 cm of frazil ice due to a malfunction with the instrument.

				High Energy		Prop Wash		
Ice Thickness	Oil Weathering	Oil Volume	C _{grab} (ppm)	C _{mean} (ppm)	d5o (μm)	C _{grab} (ppm)	C _{mean} (ppm)	d5o (μm)
5 cm Frazil	Fresh	1L	133	79	40	137	93	49
		2 L	239) 190 27		237	187	26
	17.4 % (wt.)	1L	80	43 285		90	16	295
		2 L	188	No data		218		
10 cm Frazil	Fresh	1L	120	142	101	118	342	178
		2 L	126	.6 122		215	175	97
	17.4 % (wt.)	1L	90	139		85	103	147
		2 L	161 212		147	165	475	180

Table 3-7: Droplet statistics for tests with Troll Blend crude oil

For the Troll Blend crude oil, similar oil concentrations and 50th percentile droplet sizes were measured in the high energy and prop wash periods for the tests in 5 cm of frazil ice. For the tests in 10 cm of frazil ice, significant differences in these results were noted. There was good agreement between oil-inwater concentrations measured by the LISST and by grab samples for the high energy period. The agreement was not as good for the prop wash period, in particular for the 1 L fresh and 2 L weathered tests in 10 cm of frazil ice.

It is possible that the LISST could have measured small ice particles (i.e., less than 500 µm in diameter) that became entrained in the water column, in addition to the oil, particularly during the more turbulent prop wash periods. This could explain the significant increase in mean concentration measured during the 10 cm frazil tests with 1L of fresh and 2 L of weathered Troll Blend crude oil between the high energy wave and prop wash periods.

Table 3-8: Droplet statistics for tests with ANS crude oil.

			High Energy			Prop Wash				
Ice Thickness	Oil Weathering	Oil Volume	C _{grab} (ppm)	C _{mean} (ppm)	d5o (μm)	%V < 75 μm	C _{grab} (ppm)	C _{mean} (ppm)	d5o (μm)	%V < 75 μm
5 cm Frazil	Fresh	1L	83	55	43	73	104	79	63	61
		2 L	93	117	49	70	129	139	65	59
	17.4 % (wt.)	1 L	32	34	72	57	38	53	109	44
		2 L	52	38	79	54	70	60	115	43
10 cm Frazil	Fresh	1 L	15	20	104	41	31	56	181	20
		2 L	82	90	49	71	154	131	87	49
	17.4 % (wt.)	1L	0	2	40	55	15	42	211	16
		2 L	19	28	99	44	34	69	178	25

For the tests with ANS crude oil, the average concentration of dispersed oil was higher during the propeller period, compared to the initial period with high energy waves alone, as expected; however, the droplet size distribution indicated that the oil droplets were significantly larger during the propeller



energy period, with a higher percentage of the dispersed oil being in droplets greater than 75 μm, which may resurface over time. There was good agreement between the concentrations of dispersed oil measured by the LISST and measured by the water grab samples.

Plots of oil concentration (ppm) and volume median oil droplet diameter (d_{50} in μ m) averaged over 30-second intervals, as measured by the LISST particle size analyzer for the tests with Troll Blend crude oil are presented in







Figure 3-23, below.





Figure 3-17: 1 L Fresh Troll Blend in 5 cm of Frazil Ice



Figure 3-18: 2 L Fresh Troll Blend in 5 cm of Frazil Ice

Similar droplet sizes were measured between the tests with 1 and 2 L of fresh oil in 5 cm of frazil ice. The concentration of oil in the water was higher for the test with 2 L of oil, as expected. No significant differences were noted between the high energy and prop wash periods.





Figure 3-19: 1 L Weathered Troll Blend in 5 cm of Frazil Ice

The effect of adding prop wash energy produced a more noticeable effect on the dispersed oil concentration for the test with weathered Troll Blend in 5 cm of frazil ice, and for the tests with fresh and weathered Troll Blend in 10 cm of frazil ice.



Figure 3-20: 1 L Fresh Troll Blend in 10 cm of Frazil Ice





Figure 3-21: 2 L Fresh Troll Blend in 10 cm of Frazil Ice



Figure 3-22: 1 L Weathered Troll Blend in 10 cm of Frazil Ice





Figure 3-23: 2 L Weathered Troll Blend in 10 cm of Frazil Ice

Plots of oil concentration (ppm) and volume median oil droplet diameter (d_{50} in μ m) as measured by the LISST particle size analyzer for the tests with ANS crude oil are presented in Figure 3-24 through Figure 3-31, below.



Figure 3-24: 1 L Fresh ANS in 5 cm of Frazil Ice





Figure 3-25: 2 L Fresh ANS in 5 cm of Frazil Ice

The observed lag in the oil concentration between the start of the high energy waves and the detection of dispersed oil is due to the time required for the oil to travel around the flume with the wave- and wind-induced current, and reach the area where the LISST is suspended. Oil concentrations were observed to be slightly higher for the test with 2 L of ANS, compared to the test with 1 L; 50th percentile droplet sizes were similar with both oil volumes.



Figure 3-26: 1 L 17.4% Weathered ANS in 5 cm of Frazil Ice





Figure 3-27: 2 L 17.4% Weathered ANS in 5 cm of Frazil Ice

Compared to the tests with fresh oil in 5 cm of Frazil Ice, the oil concentrations measured in the water column were significantly lower, and the dispersed oil droplets were significantly larger in size for the tests with 17.4% weathered ANS. Concentrations and droplet size distributions were similar for both oil volumes with the weathered ANS.



Figure 3-28: 1 L Fresh ANS in 10 cm of Frazil Ice





Figure 3-29: 2 L of Fresh ANS in 10 cm of Frazil Ice

The oil concentration profile was significantly different for the test with 2 L of Fresh ANS in 10 cm of Frazil Ice, compared to the test with 1 L. The oil concentrations were significantly higher with the 2 L test, although still lower than were measured during the tests with 5 cm of Frazil Ice (see Figure 3-24). The 50th percentile oil droplet diameters were significantly smaller in the tests with 2 L of oil.



Figure 3-30: 1 L 17.4% Weathered ANS in 10 cm of Frazil Ice





Figure 3-31: 2 L 17.4% Weathered ANS in 10 cm of Frazil Ice

The tests with 17.4% weathered ANS in 10 cm of Frazil Ice showed relatively low concentrations of dispersed oil in the water column, and the oil droplet sizes were relatively large. There were slightly higher concentrations of oil in the water for the test with 2 L of weathered oil, compared to the test with 1 L.



4. CONCLUSIONS AND RECOMMENDATIONS

4.1 CONCLUSIONS

Oil weathering rates and chemical dispersibility of two crude oils have been investigated in meso-scale re-circulating flumes at the SL Ross Environmental Research and SINTEF facilities. It has been shown that frazil ice thickness will not have a significant impact on the evaporative process of oil spilled in frazil ice conditions because the majority of the oil quickly migrates to the surface of the ice layer under even low energy conditions.

The evaporative losses, oil viscosity and water contents measured during the frazil ice weathering experiments conducted in this study have been compared to predicted evaporative losses using SINTEF's oil weathering model (OWM) over a range of open water and broken ice conditions for which the model has been previously validated. The modeled results for the Troll Blend are not as finely tuned as for the ANS crude because the oil data set used in the Troll oil modeling was from analyses completed on a different sample of Troll Blend than used in the weathering study. The measured results for evaporative losses in frazil ice for the ANS and Troll Blend crude oils best match the predicted values for open water conditions. This makes sense since the oil moves through the frazil ice to the surface where it is exposed to weathering conditions similar to open water.

The measured oil viscosities and water contents more closely match the modeled values when a high (90%) broken ice cover is assumed. This would indicate that the energy level present in a frazil ice cover available for the creation of water-in-oil emulsions is similar to that present in a high concentration broken ice cover, likely due to similar wave dampening by the two conditions. The higher evaporative losses in the frazil ice case versus high broken ice cover will result in higher parent oil viscosities but these increases will be small compared to potential viscosity increases due to water uptake and water-in-oil emulsification that can occur in more energetic conditions.

ANS crude oil weathered for 96 hours in frazil ice, with evaporative losses estimated at 25 to 30% by mass, was difficult to disperse, with efficiencies measured between 1.6 and 6.2% in high energy waves, and between 1.6 and 12.5% in prop wash.

Fresh ANS in frazil ice dispersed between 9 and 49% in high energy waves, and from 25 to 59% in prop wash. 17.4% by mass evaporated ANS dispersed between 0 and 19% in high energy waves, and from 8 to 20% in prop wash.

Fresh Troll Blend in frazil ice dispersed between 40 and 83% in high energy waves, and from 67 to 86% in prop wash. 22.0 % by mass evaporated Troll Blend dispersed between 45 and 53% in high energy waves, and between 47 to 61% in prop wash.

Frazil ice layer thickness influenced the dispersibility of the oil as it generally dampened the energy available to disperse the oil. The dispersant effectiveness dropped by about 15% on ANS crude oil and 10% on Troll Blend when the frazil ice thickness increased from 5 cm to 10 cm.

Weathered oil was not as readily dispersed as fresh oil and resulted in larger oil drop sizes, as would be expected. A reduction in dispersant effectiveness, over the comparable fresh oil results, of between 20 and 30% was measured in the weathered Troll Blend tests. The weathered ANS crude was 15 to 40%



less dispersible than the fresh crude oil. During the high wave energy period the volume median oil drop diameters (d50) in the fresh oil dispersions were generally below 50 microns whereas the weathered oil d50s were generally greater than 70 microns.

4.2 **RECOMMENDATIONS**

To improve the modeling work for the Troll Blend oil a full oil property analysis could be conducted on the specific Troll blend used in the study. This would provide more accurate parameters for the OWM and should result in a better match between the modeled and measured data.

Additional duplicate weathering tests in the flume using a range of oil thicknesses could be conducted to provide more precise oil weathering data for comparison to the OWM and to provide data to further refine the OWM for frazil ice conditions. Additional oil types specific to US Arctic waters could also be tested to determine their weathering and dispersibility characteristics.

A larger test scale research program could be conducted at the Ohmsett facility to validate the mesoscale dispersant effectiveness in ice work conducted by both OGP (in broken ice) and BSEE (slush ice).



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APPENDIX A: GC TRACES OF WEATHERED OILS



Troll Blend Crude Oil 96-hour Weathering





ANS 96-hour Weathering





APPENDIX B: DENSITY VS. EVAPORATION CURVES FOR ANS AND TROLL BLEND OILS

For Troll Blend the data of density vs evaporative loss is from artificial weathering of the oil in the laboratory as described in Daling et al. (2014). It is a simple one-stage distillation to vapor temperatures of 150, 175, 200, and 250 °C resulting in oil residues with increasing evaporative loss. The relationship between evaporative loss and density for Troll Blend is shown below.



The relation between density and evaporation for the Alaska North Slope crude oil that was used in the tests was based on analysis of samples artificially weathered in a wind tunnel. The relationship between evaporative loss and density for ANS is shown below.

