REPORT

SINTEF Materials and Chemistry

Address: NO-7465 Trondheim, NORWAY
Location: Brattørkaia 17B, 4. etg.
Telephone: +47 4000 3730
Fax: +47 930 70730

Enterprise No.: NO 948 007 029 MVA

Centre de Documentation, de Recherche et d'Expérimentations sur les
Centre of Documentation, Research and Experimentation on Ac
Rue Alain Colas - BP 20413 - F 29604 BREST CEDEX
National : Tél. 02 98 33 10 10 - Fax 02 98 44 91 38
International : Tel. +33 2 98 33 10 10 - Fax +33 2 98 44 91 38
E-mail : contact@le-cedre.fr - Internet : http://www.le-cedre.fr

TITLE

Effects of Time on the Effectiveness of Dispersants

Final version

AUTHOR(S)

Janne Lise Myrhaug Resby, Per Johan Brandvik, Per S. Daling
Julien Guyomarch (*Cedre*) and Ingvar Eide (Statoil)

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ABSTRACT

The objective of this study was to determine whether dispersants will remain with treated oil slicks over time and retain effectiveness. This would make it possible to apply dispersants to an oil slick under low energy conditions and delay the dispersion until the energy level increases. One possible scenario could be treating an oil spill captured in ice for later dispersion when the ice melts and sufficient energy for effective dispersion occurs.

The main conclusion from the effectiveness testing of dispersants on the four oils used in this study is that dispersants may be effective for significant time periods, but this is dependant on the oil rheological properties and on the environmental temperature. Naphthenic and asphalthenic oils remained dispersible even after two weeks of prolonged contact time at all temperatures tested (0, 15, and 25ºC).

The dispersant effectiveness did not correlate well with the surfactant content in the oil. That is, high dispersant effectiveness was achieved even when over 75% of the surfactants had leached from the oil in some tests. In other tests, poor dispersant effectiveness was observed even when most of the surfactant remained in the oil. Thus, the evidence from these tests indicates that physical properties of the oil, primarily the formation of wax precipitates, are more important factors controlling dispersant effectiveness.

The dispersant effectiveness after two weeks with a paraffinic oil depended strongly on the test conditions. The potential for retaining good effectiveness is high at temperatures above the pour point of the oil and low at temperatures below the pour point. For the waxy oil tested, the dispersion potential over time is low because it had a high pour point.

No clear conclusion could be drawn from the limited series of leaching experiments with oil in ice. The low dispersant effectiveness can not be explained by leaching of surfactants and was probably caused by the long exposure to low air temperature (-20°C) causing structural changes to the oil (semi-solidification).

KEYWORDS	ENGLISH	NORWEGIAN
GROUP 1	Dispersant	Dispergeringsmiddel
GROUP 2	Dispersant effectiveness	Effekt av dispergeringsmiddel
SELECTED BY AUTHOR	Surfactant leaching	Lekkasje av surfaktant
	ice	is
	oil	olje

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1 Introduction

Application of chemical dispersants to marine oil spills is an important response option that can produce net environmental benefits in certain situations. As shown by the simplified dispersant mechanism in Figure 1.1, the formation and dispersion of oil droplets into the water column requires turbulence. Responders may decide not to apply dispersants if sea-states are too low to promote immediate dispersion.

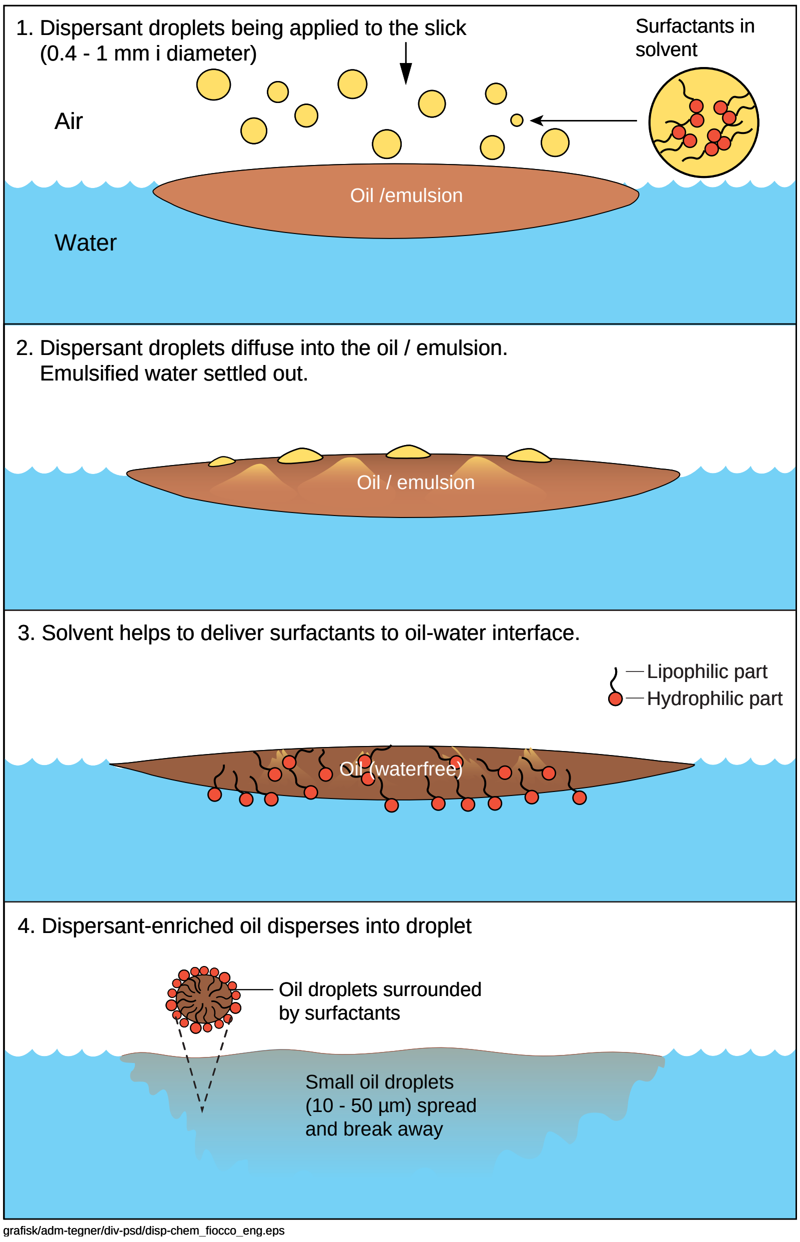

Figure 1.1 Dispersant-enriched oil disperses into droplets with wave action.

Penetration of surfactants into an oil film, however, is a time dependent step. It is most effective when dispersants are applied before the oil becomes too viscous due to weathering processes like evaporation and emulsification. Depending on the characteristics of the spilled oil and the ambient conditions, the time window for effective dispersant use can be small. The dispersant window may also close if dispersant application is postponed because existing sea states are too low.

Calm or low-energy seas often occur but are usually of short duration. In arctic regions, ice floes act to dampen the ambient wave energy needed for oil dispersion, but natural movement of ice to the edge of ice zones or strong storms can produce mixing conditions needed for dispersion even in high concentrations of ice.

A limited literature review focusing on earlier studies performed at *Cedre* and SINTEF was performed as a part of this project. The literature review found earlier work indicating that dispersant effectiveness can be retained during prolonged contact time after dispersant application. The current project extends the earlier work by performing a more detailed study and using a sophisticated quantitative method to determine surfactant content in the oil phase over time.

The review was initiated after the experimental plan for this project was established and no information was found during the review that caused major changes in the project. The literature reviews (SINTEF and *Cedre*) are given in Appendix A and B.

2 Objective

The objective of this study was to determine whether dispersants will remain with treated oil slicks over time and retain effectiveness. If dispersants remain effective for significant periods of time, oil spill responders can consider dispersants in scenarios including calm seas.

3 Scope of work

To achieve the study objective, the project was divided into three scientific tasks:

Task 1: Literature review: Document existing literature relevant to the study objective to avoid repeating earlier efforts (note that this was not a comprehensive literature search and focused mainly on SINTEF/*Cedre* studies). The literature review is attached as Appendix A.

Task 2: Fate of dispersant over time (without freezing): Determine if dispersants remain effective over time when applied to napthenic, asphalthenic, parrafinic, or waxy crude oils in open-water conditions at 0ºC, 15ºC, and 25ºC. The task includes dispersant-effectiveness testing using the IFP method and parallel analysis of dispersant leaching.

Task 3: Fate of dispersant over time (with freezing): Evaluate spill scenarios in ice-prone regions by determining if dispersants remain effective over time when frozen into an ice layer and on top of an ice layer and subsequently thawed. Only oils that were effectively dispersed at 0ºC in Task 2 will be used. The task includes dispersant-effectiveness testing using the IFP method and parallel analysis of dispersant leaching.

4 Experimental design

SINTEF and *Cedre* jointly performed the testing completed for this report. SINTEF performed the tests on the paraffinic and naphthenic oil types and *Cedre* on the asphalthenic and waxy oil types for the open-water study described in Task 2. SINTEF completed the testing under freezing conditions described in Task 3.

4.1 Quality assurance - intercalibration between SINTEF-*Cedre*

Since the dispersant effectiveness testing (IFP) was performed at two different laboratories, an intercalibration was performed before the full laboratory work was started. The calibration between the laboratories was performed on three oil types. One standard oil and two project oils were used for this intercalibration. The results are given in Table 4.1 and Table 4.2.

Table 4.1 Mean and Standard deviation from intercalibration between *Cedre* and SINTEF (IFP%) using the SINTEF standard calibration procedure. Three replicates were performed.

	Cedre	**SINTEF**
Oil[1]	SINTEF reference	SINTEF reference
Dispersant	Dasic NS	Dasic NS
Height of the beater (mm)	33 mm	33 mm
Temperature (°C)	13 °C	13 °C
Pre. treatment 50°C[2]	Yes	Yes
Effectiveness (%)[3]	**77,0**	**76,8**
Standard deviation[4]	**1,1**	**2,1**

1) Reference oil for the SINTEF IFP calibration (Sture Blend 200ºC, ID: 1999-0514)
2) Preheating to 50°C to exclude differences in thermal history of the oil
3) Expected IFP% for the reference oil is: 75.6%
3) Expected standard deviation for this reference oil is: 5.1

Expected mean and standard deviation above are based on 38 replicate IFP experiments performed by five different laboratory technicians at SINTEF.

Table 4.2 Results from intercalibration between *Cedre* and SINTEF (IFP%) using two oils and the IFP procedure used in the project (1 minute contact time). Two replicates were performed of each dispersant/oil combination.

	Balder 200ºC		**Ringhorne 150ºC**		**Pooled**
	Corexit 9500	**Finasol OSR52**	**Corexit 9500**	**Finasol OSR52**	**Standard Dev**
Cedre	95,6	87,0	62,8	42,0	4,8
SINTEF	93,6	95,2	66,3	37,8	4,6
Difference	2,0	-8,2	-3,5	4,2	0,2

The calibration test with the standard oil (Table 4.1) showed an excellent agreement in the IFP results delivered of the two laboratories. The mean values and difference in dispersant effectiveness was only a small fraction of the expected standard deviation. The experimental uncertainties (standard deviation) were also smaller than expected for the calibration test.

Such IFP calibrations are performed and documented with regular intervals at both laboratories as a part of their internal QA systems.

Also the results from IFP testing with the two project oils (Table 4.2) did not show any differences between the two laboratories which were larger that two StDev (9.2) which were the requirement in the project description.

The project description describes a procedure with duplicate IFP measurements instead of the standard triplicate replicates. However, if the two replicates showed a difference more than two StDev for the method, a third replicate was performed, and all three replicates used to calculate the IFP effectiveness. This extra IFP replicate was performed in 22% of the IFP experiments performed in the project.

4.2 Selection of test oils

Four crude oils representing four broad oil categories were used for this project. The oils were selected from a set of 70 oils that SINTEF has performed standardized weathering studies on, 40-50 of these oils had also been tested for dispersability.

Based on the fresh and weathered oil properties, the oils can roughly be placed into four different oil characteristic groups shown in Figure 4.1

- Naphthenic oil: Biodegraded, rich in saturated cyclic components and branched alkanes.
- Asphalthenic oil: Rich in polar resins, asphaltenes and aromatic components.
- Paraffinic oil: Rich in paraffins and saturated components.
- Waxy oil: High pour point, rich in higher molecular-weight saturated components.

One representative oil from each group was selected for further testing. The following four oils were selected:

1. Troll B (naphthenic oil), SINTEF id 2000-0052. Sampled in 2000.
2. Balder (asphalthenic oil)¸ SINTEF id 1999-0527. Sampled in 1999.
3. New Oseberg Blend (paraffinic oil), SINTEF id 2005-0837. Sampled in 2005.
4. Ringhorne (waxy oil), SINTEF id 2000-0652. Sampled in 1997.

Each crude oil underwent simulated weathering to represent 12-24 hours of weathering at sea. Simulated at-sea weathering was completed using a modified ASTM distillation (Stiver and Mackay, 1984). The distillation proceeded until the vapour temperature reached 200ºC leaving a 200ºC weathered residue, see Table 4.3 for procedures. The 200ºC residue represent 12-24 hours of weathering at sea, assuming 10 m/s wind, air/water temperatures of both 15ºC and a terminal film thickness of 1 mm. This can be shown with the SINTEF OWM and is verified by field trials.

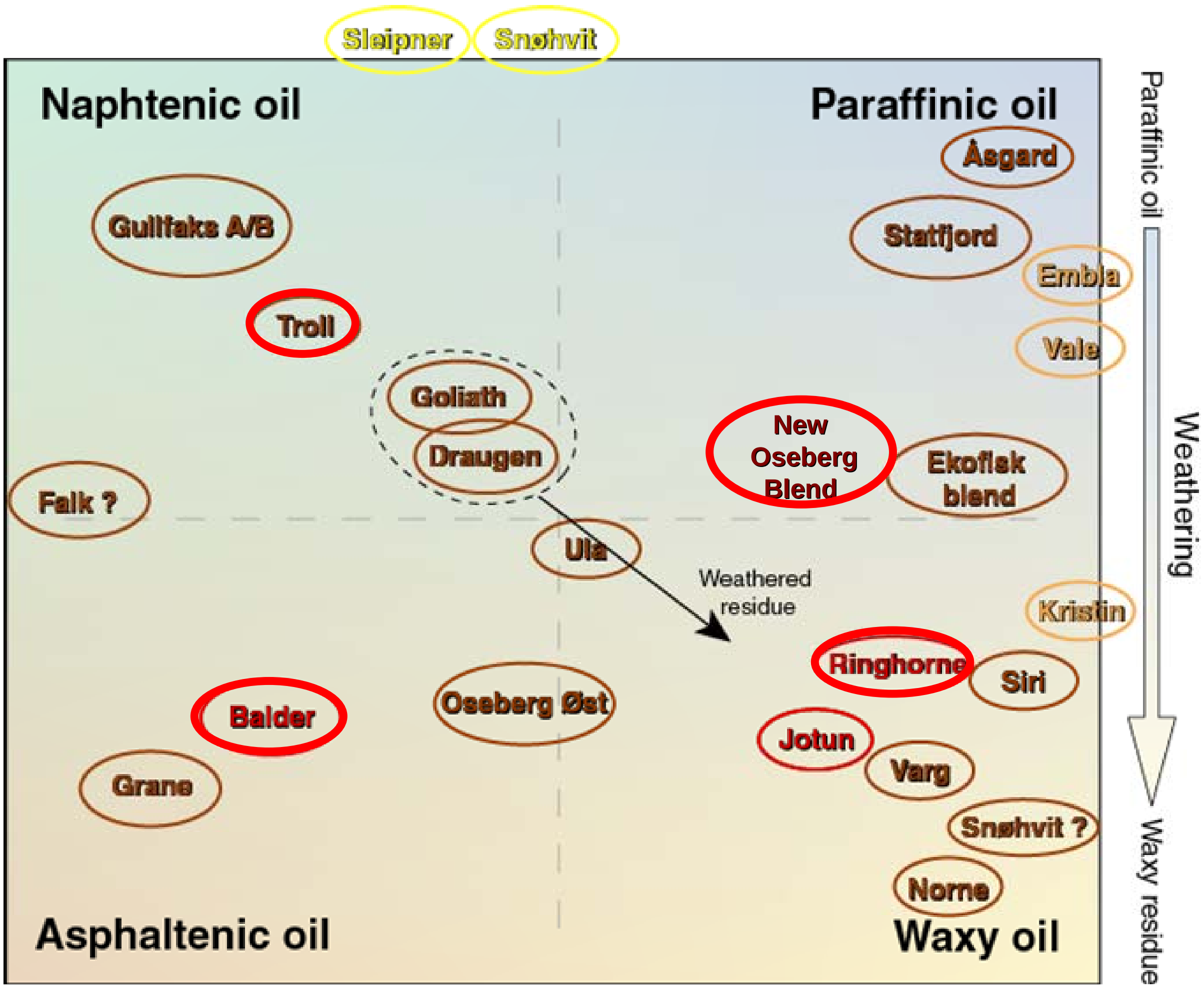

Figure 4.1 Example of categorization of some Norwegian crude oils and showing schematically where the crude oils tested in this project can be placed in this map.

4.3 Physico-chemical analyses

The fresh and weathered crude oils were characterised using the analytical methods listed in Table 4.3.

Table 4.3 Overview physical-chemical analyses.

Analysis	Method
Density	ASTM-method D4052-81.
Viscosity (dynamic)	Physica MCR 300 (McDonaugh et. al., 1995)
Pour Point	ASTM-method D97-66, IP-method 15/67.
Wax content	Insoluble in 2-butanon/dichlormethane at -10°C (Bridié *et al*. 1980).
"Hard" asphaltenes	IP-method 143/84 (precipitation in “hot” heptane).
Interfacial Tension	ASTM-method 971-82.

Crude oil viscosity can vary over two orders of magnitude for the temperature range of interest in this study. Viscosity is also a parameter that significantly influences oil spill response. For these reasons, the viscosity of the weathered crude oils was measured in 0.5°C increments over the temperature range of 30° to 0°C at a shear rate of $10 s^{-1}$ (unless otherwise specified).

4.4 Dispersants tested

The commercially available dispersants used in this project were Corexit 9500, Dasic NS, Superdispersant 25 and Finasol OSR 52. Because manufacturers keep the exact formulation of the dispersants proprietary, a Model dispersant was prepared and used in the testing to quantify the leaching of surfactants. The Model dispersant provided the precise concentrations of the dispersant components required to quantify leaching rates. The formulation of the Model dispersant is given in Table 4.4

Table 4.4 Composition of the Model dispersant

Wgt.%	Name[1]	HLB[2]
6,5	Span-80	4.3
12,9	Tween-80	15
19,1	Tween-85	11
27,8	Aerosol-OT-75 (AOT)	ionic
18,6	Dipropylene-glycol-n-butyl-ether	
15,2	Exxsol-D80	

1) Trade names

2) HLB: Hydrophilic-Lipophilic-Balance

4.4.1 Dispersant effectiveness test method

In this project, the IFP Dispersant effectiveness test was selected. The IFP apparatus is schematically shown in Figure 4.2. The IFP test (Institute Francais du Pétrole test, Bocard et *al*. 1984) is the official method used for approval of dispersants in France and in Norway. Correlations between results of this test and field experiments have been documented in Desmarquest et. al (1985). Compared to many other test methods, the IFP is a low energy test and represents a more realistic approach to field conditions due to continuous dilution, making this method especially suitable for this project.

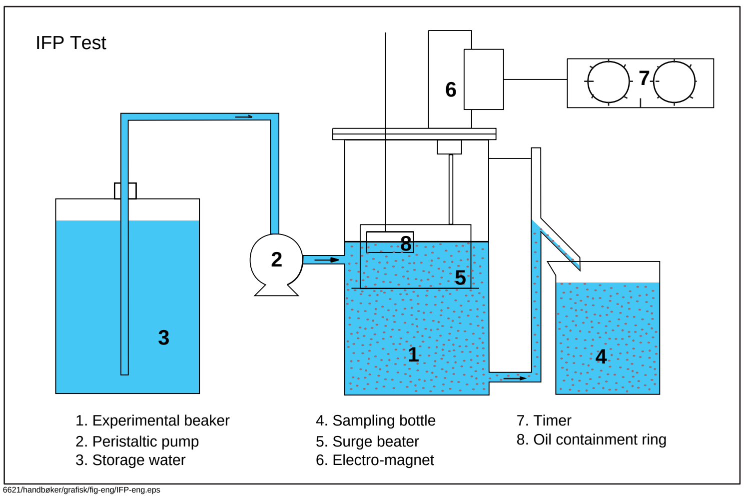

Figure 4.2 Schematic description of IFP method.

4.4.2 Dispersant screening tests

The first experimental work conducted for this study was a screening test to identify the two commercial dispersants being most effective on the four weathered crude oils. These two dispersants were afterwards comprehensively evaluated in Task 2 to maintain a manageable project scope. The screening tests were completed using the standard IFP dispersant effectiveness test conducted at 15°C.

4.4.3 Dispersant tests with extended contact time without freezing, Task 2

The Task 2 research to evaluate dispersion over time in open-water conditions included dispersant effectiveness tests using the IFP test and parallel leaching tests using the IFP apparatus. Two sets of parallel effectiveness and leaching tests were performed at each contact time.

The IFP test was modified by:

1. Extending the contact times between dispersant and oil up to 2 weeks and
2. For tests with the model dispersant only, premixing of oil and dispersant before application of oil in containment ring.

As mentioned, the Model dispersant was included in the testing to accurately characterize the leaching of dispersant components from the crude oils over time. To ensure that the initial concentrations of the dispersant components within the oil were known, the Model dispersant was always premixed with the oil prior to application within the IFP apparatus. Premixing of the Model dispersant was performed for the leaching samples and the parallel dispersant effectiveness tests.

All tests with the commercial dispersants used the standard drop-wise addition of dispersant. Drop-wise addition of dispersant was used with the commercial dispersants to simulate the initial interaction of dispersant with the oil film.

The dispersant effectiveness tests were completed in the standard way while the leaching tests ended by taking the whole oil sample out of the IFP apparatus using a steel ladle. Some water was drained out of the ladle before oil and water were transferred to a specially made funnel with a Teflon valve. The remaining water and oil mixture was allowed to settle in the funnel for 10 minutes before draining the water. Then the oil sample was mixed with a glass rod, transferred to glass vials, and frozen prior to analysis. To determine leaching rates, the concentration of the dispersant components was measured using combined mass spectrometry (MS) and multivariate calibration (see chapter 4.5).

The contact times were 1 min (standard), 6hr, 12hr, 24hr, 72hr, 168hr and 336hr depending on effectiveness and temperature. Test temperatures were 0, 15 and 25°C. The Model dispersant, Corexit 9500, and Finasol OSR 52 were all tested at 15°C. Only the Model dispersant was tested at both 0 and 25°C.

All IFP tests were performed with the standard continuous dilution of 2.5 L/hour of fresh (3.3% seawater) during the entire test period. Effectiveness and leaching tests were performed in separate IFP systems to get a representative oil sample for further quantification of surfactants in oil phase by multivariate analysis on MS spectra.

The dosage of dispersants was 4 wt. % for all tests performed at SINTEF and 5 wt% for all tests performed at *Cedre*. This slight difference in the dosage used at the two institutes should have been avoided, however, we do not expect this difference to influence on the main conclusions and findings in this project.

4.4.4 Dispersant tests with extended contact time with freezing, Task 3

The Task 3 research to evaluate dispersion over time in ice conditions also included dispersant effectiveness tests using the IFP test and parallel leaching tests using the IFP apparatus. Two spill scenarios were evaluated: oil spilled on top of ice and oil spilled on top of water that subsequently froze. With these freezing experiments only single experiments were performed for surfactant leaching.

To freeze the oil-ice mixtures, beakers used for the IFP test were mounted within an insulated, temperature-controlled container that kept the walls and bottom of the beaker at between 0 and 2ºC. This container was placed into a freezer maintained at -20ºC, see Figure 4.3. This setup allowed an ice layer to form on the surface of the water in a manner representing real conditions to simulate first-year ice properties (density, salinity, porosity, and brine channels).

Because wax precipitation was expected to limit dispersion of waxy and paraffinic oils at 0°C, only the asphalthenic and napthenic oils were tested using the Model dispersant in the freezing experiments.

As mentioned earlier, two different series of experiments were performed. The first series included placing premixed oil and dispersant in the IFP containment ring on top of existing ice with a thickness of approximately 10-13 cm. After contact times of 48 hours, 2 weeks or 1.5 months, the ice was thawed (at 20ºC) until the oil-dispersant-ice could be moved to a new IFP beaker maintained at 0°C. The ice was then thawed slowly at 0°C with continuous dilution with seawater (0°C) at a rate of 2.5 L/hour. After thawing the beater ring was placed in the beaker and the IFP tests were performed.

In the second series, the experiments were performed as described earlier but the oil was placed on top of seawater and then frozen into ice with an ice thickness of approximately 10-13 cm. The experiments were then completed as described above.

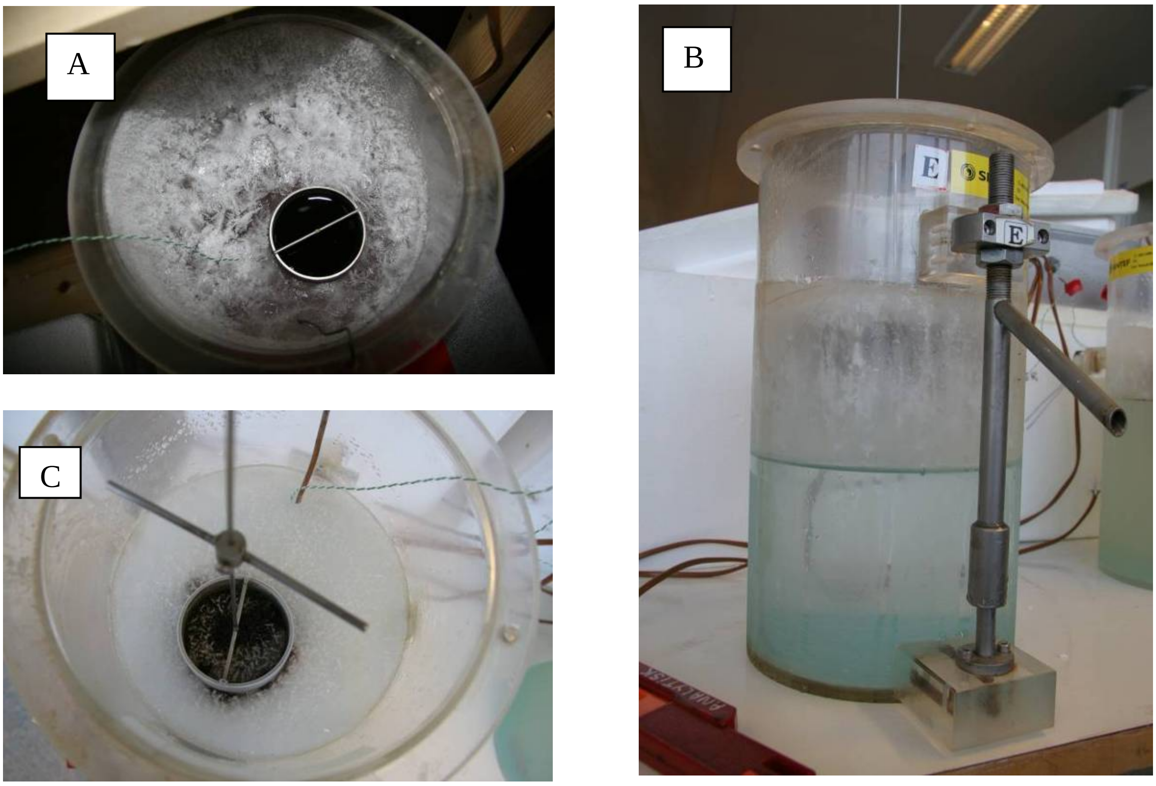

Figure 4.3 Pictures from oil on ice experiment with a premix of Model dispersant and Troll B 200°C+. A: just after application of oil on ice. B and C: after 1.5 month in ice at air temperature -20°C.

4.5 Quantification of surfactant leaching

The initial approach was to quantify the surfactants which had leached in to the waterphase using solid phase extraction and mass spectrometry. However, the complexity was high and the sensitivity was not satisfying. This led to the approach of quantifying the remaining surfactants in the oil using the procedure described in this chapter.

Surfactant content in the oil was compared to dispersant effectiveness by performing leaching and IFP tests in parallel systems using the same conditions. The entire oil sample in the leaching tests was collected for analysis to eliminate uncertainty in surfactant partitioning within the oil.

The total dispersant and individual surfactants remaining in the oil phase were quantified using mass spectrometry (MS) and multivariate calibration. Experimental conditions regarding the MS analysis and the multivariate calibration are briefly described below and in more detail in Appendix D. The preparation of the samples was performed at SINTEF, the MS analysis and data pre-treatment (selecting of masses and calculation of mean spectra) were done at Statoil's research centre in Trondheim and the final multivariate calibration was performed by SINTEF.

4.5.1 Mass Spectrometry (MS)

In preparation for the MS analysis, the frozen leaching samples were thawed, mixed well, and dissolved in dichloromethane (DCM) to give a concentration of 2 mg oil-dispersant/mg of DCM. The samples were analysed using positive electrospray mass spectrometry using a single quadupole LC-MS instrument with direct injection (no chromatographic separation of the surfactants) and without fragmenting the molecules. Each sample was analyzed 5 or 6 times

4.5.2 Determination of total dispersant leaching

To determine the total dispersant concentration in the leaching samples, calibration was performed using a calibration set for each combination of oil (Troll B, Balder, Ringhorne, New Oseberg Blend) and experimental test conditions where the total dispersant concentration was varied from 0 to 6%. A multivariate model for each oil was developed based on MS data for the calibration sets. The model consisted of two principal components explaining 85-95% of the X variance (masses) and 92-99% of the Y variance (amount of dispersant) to describe the relationship between a total surfactant pattern versus the oil pattern in the MS spectra and the known concentration of dispersant in the calibration samples. Correlation between the known concentration and the measured concentration determined by cross validation was in the range of 0.95-0.99 for all oil-experiment type combinations.

These predictions were scaled to represent the totals concentration of surfactants (excluding the solvent package) in the oil phase and they are presented in chapter 5.

4.5.3 Determination of leaching of individual surfactants

To determine the concentration of individual surfactant components in the leaching samples, calibration was performed using a calibration set where the concentration of the four individual surfactants in the Model dispersant was varied using the d-optimal design shown in Table D.0.1. A separate calibration set using the d-optimal design was prepared for all four oils. In addition, the calibration set for each oil included three replicate samples of the dispersant-free oil as a zero surfactant reference.

The calibration sets spanned the expected variation in concentration of the four surfactants in the leaching samples with a minimum of samples (25 + 3). A multivariate model for each oil was developed based on MS data for the calibration sets. The models consisted of four to five principal components explaining 55-75% of the X variance (masses) and 75-95% of the Y variance (amount of the individual surfactants). Correlation between the known concentration and measured concentrations in the calibration sets determined by cross validation was in the range of 0.65-0.98 for all oil-experiment type combinations.

These predictions were scaled to represent the concentration of individual surfactants in the oil phase (excluding the solvent package) and are presented in chapter 5. The total surfactant content was also predicted by summarising all the individual surfactants. These summary figures were in good agreement with the total surfactant predictions in chapter 4.5.2 (± 15%).

4.6 Documentation of spontaneously formed micron sized oil droplets

In the request for proposal prior to this project, observations of a cloudy layer interpreted as oil dispersed without energy input was described. This mechanism has earlier been discussed by Canevari (in McCarty et. al: ASTM Special Technical Publ. 659, 1978); At that time, the so-called "self-mix dispersants" (concentrates), when applied to the oil on water, were thought to have a "driving force" to disperse oil droplets from the oil phase to the water phase. Similar visual observations, of cloudy layers in the water phase, have also been made at SINTEF in connection with dispersant performance testing of very low-viscosity oils (e.g. fresh crude oils). In this project, special attention was given attempting to observe such a process.

5 Results and discussion

In the figures in this report, the dispersant effectiveness is denoted as "IFP%", to identify the test method used to determine the dispersant effectiveness.

5.1 Quality assurance

The results from the intercalibration between *Cedre* and SINTEF were well within the specifications in the project description. There is no indication that the two-laboratory approach has created additional uncertainty to the generated data.

5.2 Oil characteristics

Measured physical and chemical characteristics of the four crude oils evaluated in this study are listed in Table 5.1. The viscosity of each oil as a function of temperature is shown in Figure 5.1.

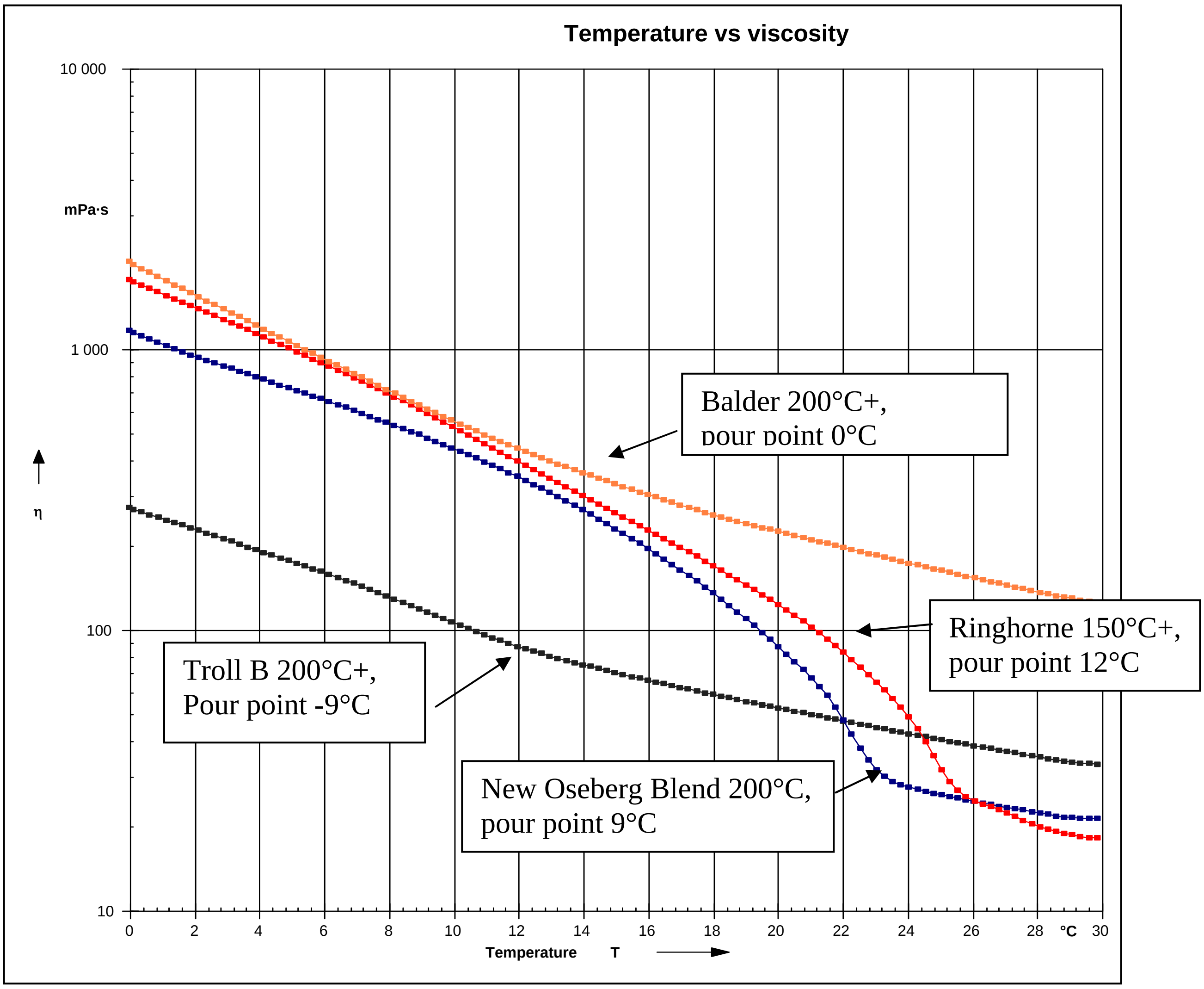

Figure 5.1 Viscosity as function of temperature for the four tested oils. Viscosity is measured at a shear rate of 10^{-1}.

Figure 5.1 shows the significant increase in viscosity that occurs for each oil as the temperature is reduced from 30°C to 0°C. This increase is almost two orders of magnitude for the paraffinic and waxy oils (New Oseberg Blend and Ringhorne).

At roughly 23°C for New Oseberg and 26°C for Ringhorne the slope of the viscosity curves changes indicating the onset of wax precipitation that forms a lattice structure in the oil. At lower temperatures, more wax precipitates and strengthens the lattice. This causes the viscosity to follow a steeper slope with respect to temperature, compared to Balder and Troll. The Balder and Troll oils with low wax contents and pour points have a more loglinear viscosity temperature dependence, see Figure 5.1.

Table 5.1 Physical and chemical properties of the oils used in the study

Oil type	Residue	Evap (vol.%)	Density (g/mL)	Pour Point (°C) [1]	Viscosity (cP) 13°C	Asphaltenes (Wt. %) [2]	Wax (wt. %) [3]
Troll B (Naphthenic)	Fresh	0	0.891	-18	36	0.1	1.9
	200°C+	17	0.908	-9	55	0.1	1.6
Balder (Asphalthenic)	Fresh	0	0.914	-6	220	1.0	0.5
	200°C+	11	0.929	0	990	0.9	0.5
New Oseberg blend (Paraffinic)	Fresh	0	0.859	-12	6	0.5	2.1
	200°C+	30	0.884	9	260	0.6	2.9
Ringhorne (waxy)	Fresh	0	0.830	6	66	0.2	4.8
	150°C+	20	0.860	12	1270	0.2	5.9
	200°C+	32	0.875	24	3510	0.3	6.8

1) Pour point determined using ASTM D97-77
2) Asphaltenes determined using IP 143/90
3) Waxes determined by extracting with 2-butanone/DCM at –10ºC (Bridie *et al*. 1980)

5.3 Screening of dispersants

The results from the screening tests are shown in Figure 5.2. Initially 200°C+ residues were selected for all oil types. However, all dispersants had low effectiveness on the 200°C+ residue of the waxy Ringhorne oil even with only 1 minute of contact due to the high pour point (24°C). Wax precipitation likely reduced dispersant penetration into the oil and increased the oil cohesiveness. A 150°C+ Ringhorne residue was substituted to allow some dispersion in further testing.

The model dispersant, with a known surfactant composition was also included since it was used for the leaching studies and detailed surfactant analysis. The experiments with the Model dispersant were performed with the dispersant premixed into the oil before placing in the IFP device. This was done to ensure initial surfactant concentration in the oils was known for the leaching experiments.

Superdispersant 25 had in general a lower effectiveness than the other three dispersants, especially with the asphaltenic, paraffinic, and waxy crude. The other three dispersants (Corexit, Dasic, and Finasol) were relatively similar in effectiveness, except Finasol had slightly lower effectiveness than Corexit 9500 and Dasic on the waxy Ringhorne.

The steering committee decided to use Corexit 9500 and Finasol OSR 52 for the testing at extended contact times.

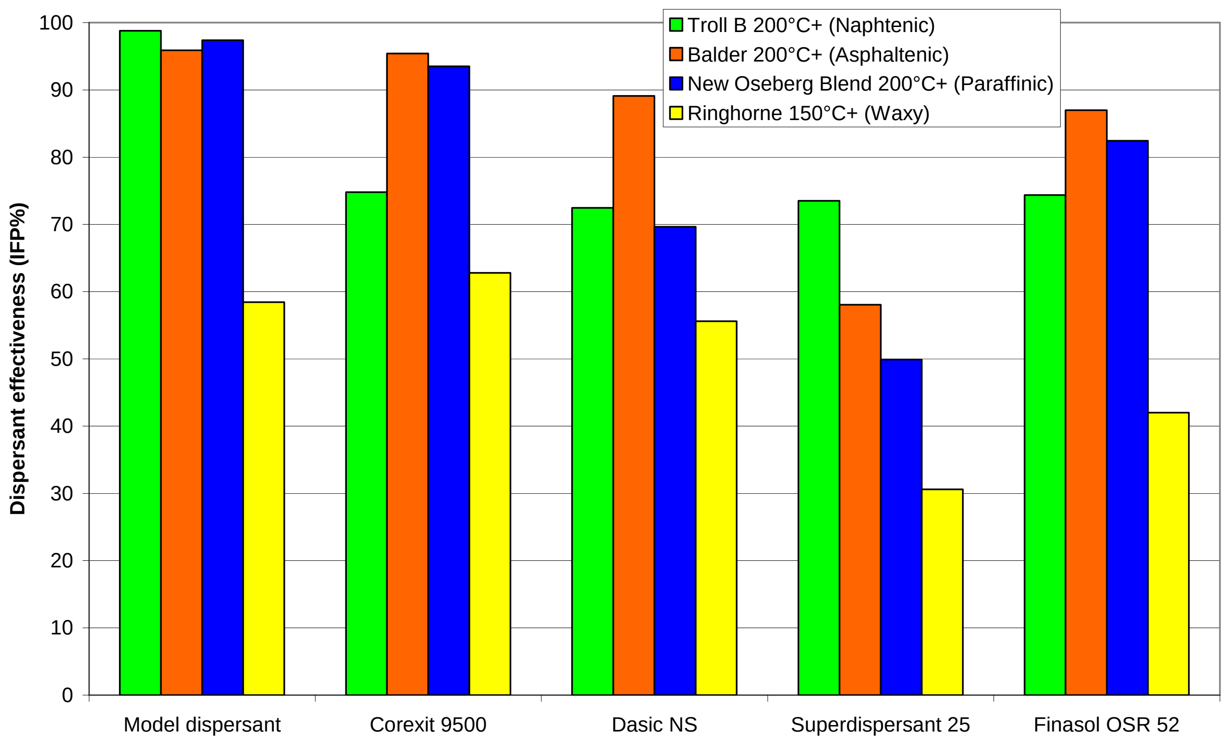

Figure 5.2 Screening tests of dispersant effectiveness at 15°C using the IFP test standard 1 minute contact time.

5.4 Dispersant Effectiveness versus leaching of surfactants

This chapter presents the combined results from both dispersant effectiveness testing and surfactant leaching experiments. Dispersant effectiveness was measured with three different dispersants (Model, Corexit, and Finasol) and all four oil types after prolonged contact time between oil and dispersant at seawater temperature 25, 15, and 0°C. The experimental matrix in Table 5.2 shows the dispersant effectiveness results and experiments that included surfactant leaching are marked in red.

All combinations of dispersants and oil types were tested at 15°C. The waxy Ringhorne oil wasn't tested at 0°C because it wasn't expected to disperse. The naphthenic Troll oil wasn't tested at 25°C because it was expected to readily disperse.

Table 5.2: *Dispersant effectiveness as a function of dispersant type, oil type, temperature and leaching time. Red indicates replicate IFP experiments for surfactant analysis.*

Oil type	**Troll B 200°C+**			**Balder 200°C+**			**Oseberg 200°C+**			**Ringhorne 150°C+**		
	Naphthenic			**Asphalthenic**			**Paraffinic**			**Waxy**		
Temp Hours	***MD***	***9500***	***OSR52***	***MD***[1]	***9500***[2]	***OSR52***[3]	***MD***	***9500***	***OSR52***	***MD***	***9500***	***OSR52***
25°C 0.017				95			100			90		
1				94			100			97		
6												
24				91						76		
72												
168				90			84			37		
336				59			83			24		
15°C 0.017	99	75	74	96	95	87	97	94	82	58	63	42
1		79										
6										56	55	38
24	98	91	74	97	95	88	92	95	87	24	40	21
72										13	18	7
168	99	100	77	83	84	58	75	63	86		7	
336	92	97	52	76	67	44	53	32	32			
0°C 0.017	98			100			38					
1	97			98			38					
6												
24												
72												
168	92			77			27					
336	86			88			15					

1) *MD: Model dispersant* 2) *9500: Corexit 9500* 3) *OSR52: Finasol OSR-52*

5.4.1 Dispersant effectiveness (all dispersants and oil types at 15°C)

This section provides the dispersant effectiveness test results for the different combinations of oils and dispersants at 15ºC. All combinations of dispersants and oil types were tested at 15°C.

Naphthenic oil (Troll B 200°C+)

The effectiveness of the Model dispersant and Corexit 9500 was very good and above 90% after two weeks. Finasol OSR52 had a lower initial effectiveness (above 70) which was reduced to 50 after two weeks of leaching (see *Figure* 5.3 for details).

The Model dispersant had initially (the first 24 hours) a higher effectiveness than Corexit 9500, likely due to the premixing of the Model dispersant into the oil. The increasing effectiveness of Corexit 9500 with increasing contact time could be explained by the surfactants arrangement in the oil. It may take time after application of dispersant for the surfactants to completely penetrate and uniformly mix into the oil. In the premixed Model dispersant tests, the surfactants were homogenously mixed from the start.

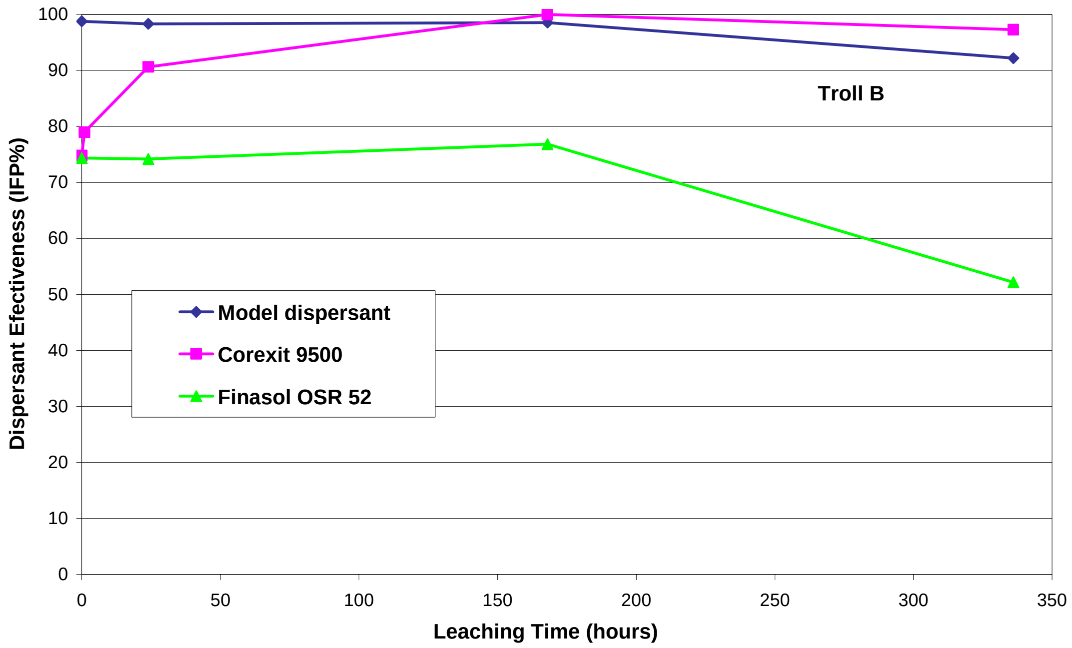

Figure 5.3 Effectiveness of the three dispersants at 15C° on the naphthenic Troll 200°C+l. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks).

Asphalthenic oil (Balder 200°C+)
The effectiveness of all three dispersants was initially very high with Balder (above 85%). However, during the first week the effectiveness slowly declined for all three dispersants. After two weeks the effectiveness for the Model dispersants and Corexit 9500 was still high (76% and 67%), while Finasol OSR52 dropped to 44% (see Figure 5.4 for details).

Comparing results, the asphaltenic oil had a larger decrease in effectiveness at 2 weeks contact than the napthenic oil.

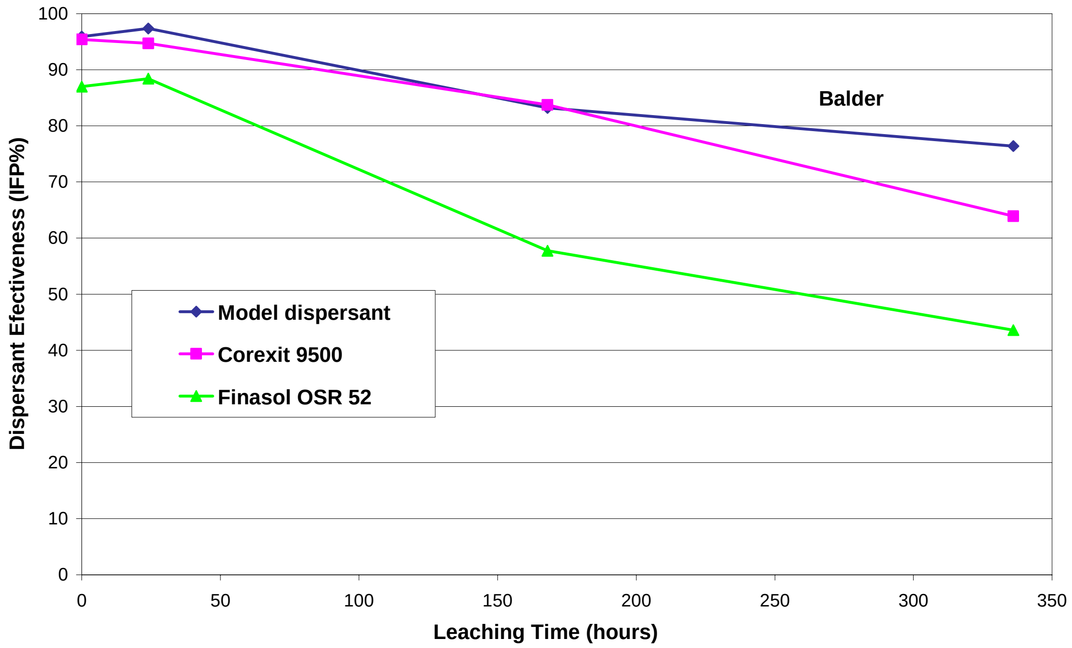

Figure 5.4 *Effectiveness of the dispersants at 15C° on the asphalthenic Balder 200°C+. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks).*

Paraffinic oil (Oseberg 200°C+)
As with the previous Troll and Balder crudes, the effectiveness of all three dispersants are initially very high (above 85 IFP%), but with Oseberg they decline at a slightly higher rate and after two weeks only the Model dispersant is above 50%, while the Corexit 9500 and Finasol OSR52 gave only 32% (see *Figure* 5.5 for details).

The larger decrease in effectiveness for this oil compared to the two previous oils is probably due to precipitation of waxes. The pour point of the 200°C+ residue is 9°C, but Figure 5.1 shows that wax starts to precipitate already at 23°C. Thus, wax precipitation is likely to occur during the test period at 15°C (see also Chapter 5.2 Oil characteristics).

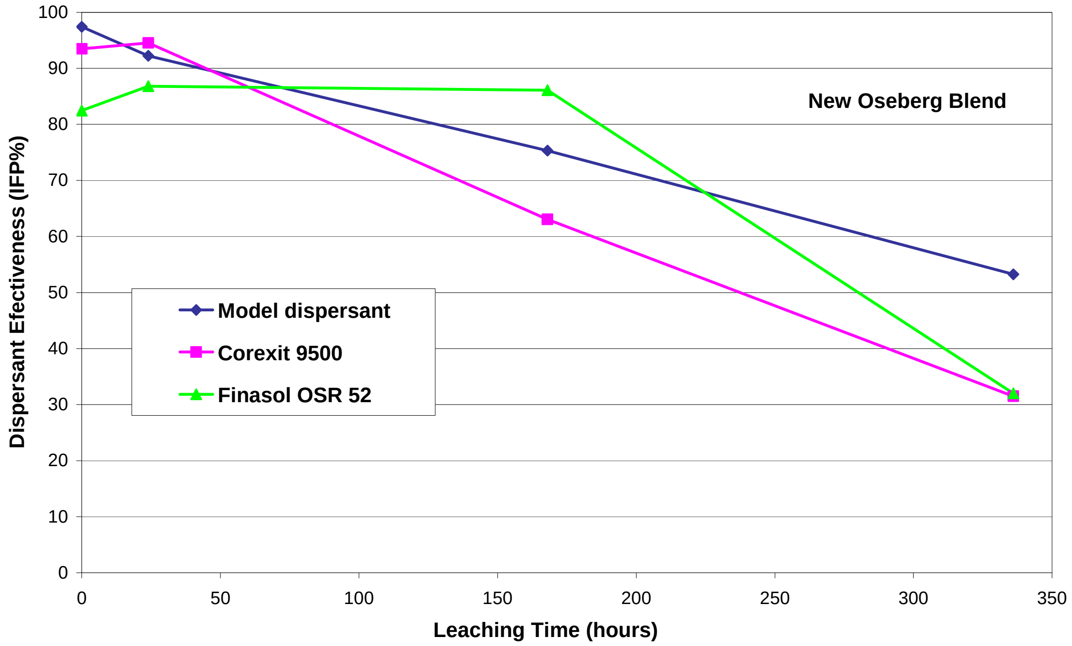

Figure 5.5 Effectiveness of the three dispersants at 15C° on the paraffinic new Oseberg blend 200°C+. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks).

Waxy oil (Ringhorne 150°C+)
The effectiveness of all three dispersants was initially lower for the waxy Ringhorne crude (40-60 IFP%, see *Figure* 5.6 for details). All dispersants showed a rapid decrease in effectiveness with increasing contact time. After only 3 days the effectiveness had decreased below 20%.

The decrease in effectiveness for this oil was likely due to wax precipitation over time and solidification of the oil, since the pour point of the oil (12°C) was close to the seawater temperature. During the test period there may also be a slight evaporation from the 150ºC-residue used with Ringhorne. This may cause formation of a wax lattice and lead to a semi-solid (cohesive) oil phase, which will reduce the potential of using dispersants.

Figure 5.7 shows the average 15ºC effectiveness for all three dispersants and all four oils at 2 weeks. The data show that the low wax content oils (Troll B and Balder) maintained relatively good dispersion throughout the 2 week test period. The New Oseberg Blend with a significant wax content, had somewhat lower dispersion at 2 weeks. The 5.9% wax content and the resulting wax precipitation in the Ringhorne oil appeared to rapidly limit dispersion and completely eliminate it by 2 weeks.

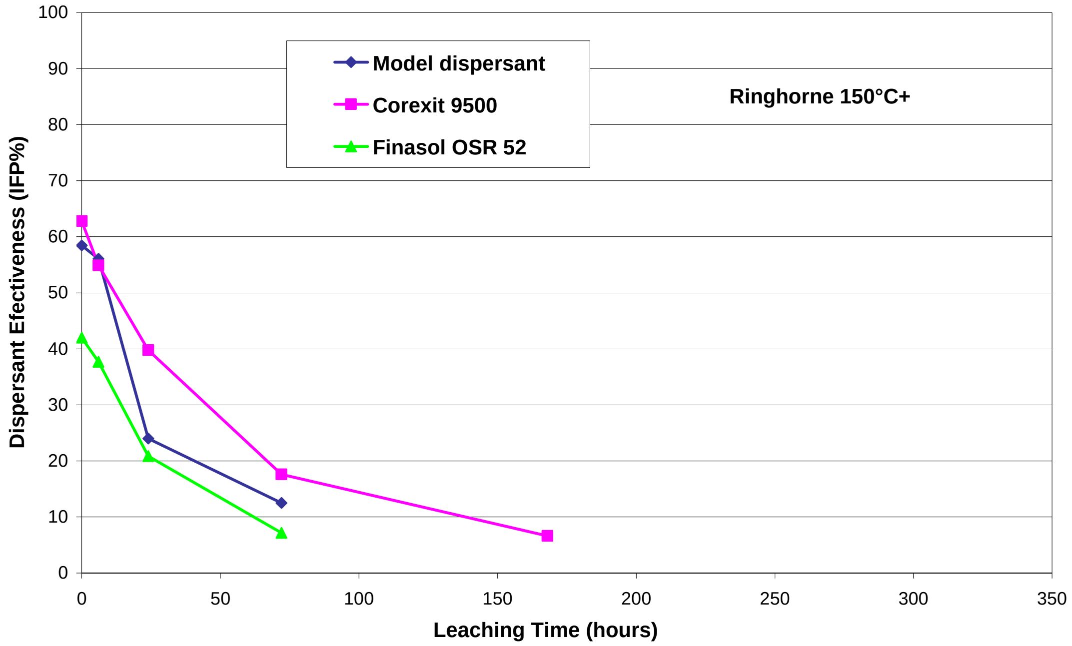

Figure 5.6 Effectiveness of the three dispersants at 15C° on the waxy Ringhorne 150°C+. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks).

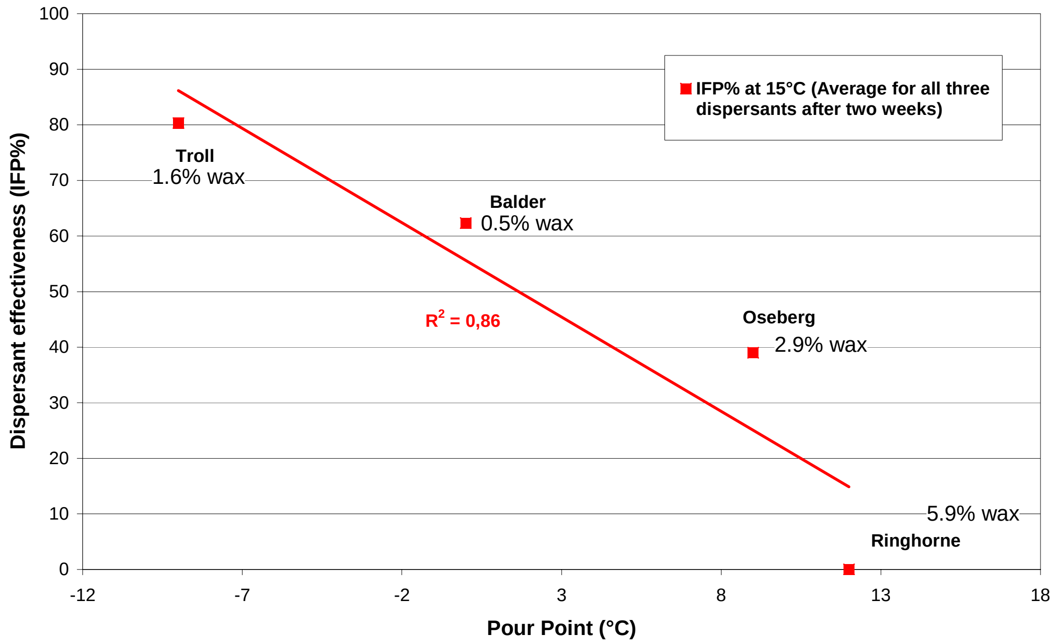

Figure 5.7 *Averaged dispersant effectiveness (%) at 2 weeks and 15°C for all three dispersants (Model dispersant, Corexit 9500 and Finasol OSR52).*

However, it is expected that the surfactant leaching rate is also controlled by the pour point to some extent, since the molecular diffusion should slow as a waxy skin forms at the oil-water interface. The combined variation in effectiveness and surfactant leaching is discussed in the next section.

5.4.2 Dispersant effectiveness versus surfactant leaching at 0, 15, and 25°C

Dispersant effectiveness (%) as a function of both concentration of individual surfactants (AOT, Tween-80, Tween-85, and Span-80) and total dispersant is given from Figure 5.8 to Figure 5.17. The concentrations in the figures of both the single surfactants and the total dispersant are concentrations in the oil.

The interpretation of leaching trends in these and the figures to follow is based on the interpolated exponential trend lines. These exponential trend lines are shown as linear lines in the figures due to logarithmic scaling of the dispersant effectiveness y-axis. It is reasonable to believe that the surfactant leaching follows an exponential decay rather than a linear trend as most concentration equilibrium driven processes. In most cases the fit between the experimental data and the trend lines is good, correlation (R^2) better than 0.6. However, in some experiments this correlation is lower. The lower correlations are likely the result of a loss in experimental accuracy when measuring the low concentration of surfactants that remained at 2 weeks for many tests.

The leaching of the individual surfactant in the Model dispersant (AOT, Span-80, Tween-85 and Tween-80), the leaching of the total dispersant and the dispersant effectiveness (%) for the napthenic Troll oil at 15ºC are presented in *Figure* 5.8.

The total dispersant concentration in the oil was reduced from 2.6 to approximately 1.4% at 2 weeks due to surfactant leaching after prolonged contact with water. However, all four surfactants are present in the oil at 2 weeks in relative proportions not very different from the test start. This significant loss of surfactant content at 2 weeks had little influence on the dispersant effectiveness for this napthenic oil (dispersant effectiveness still over 90%).

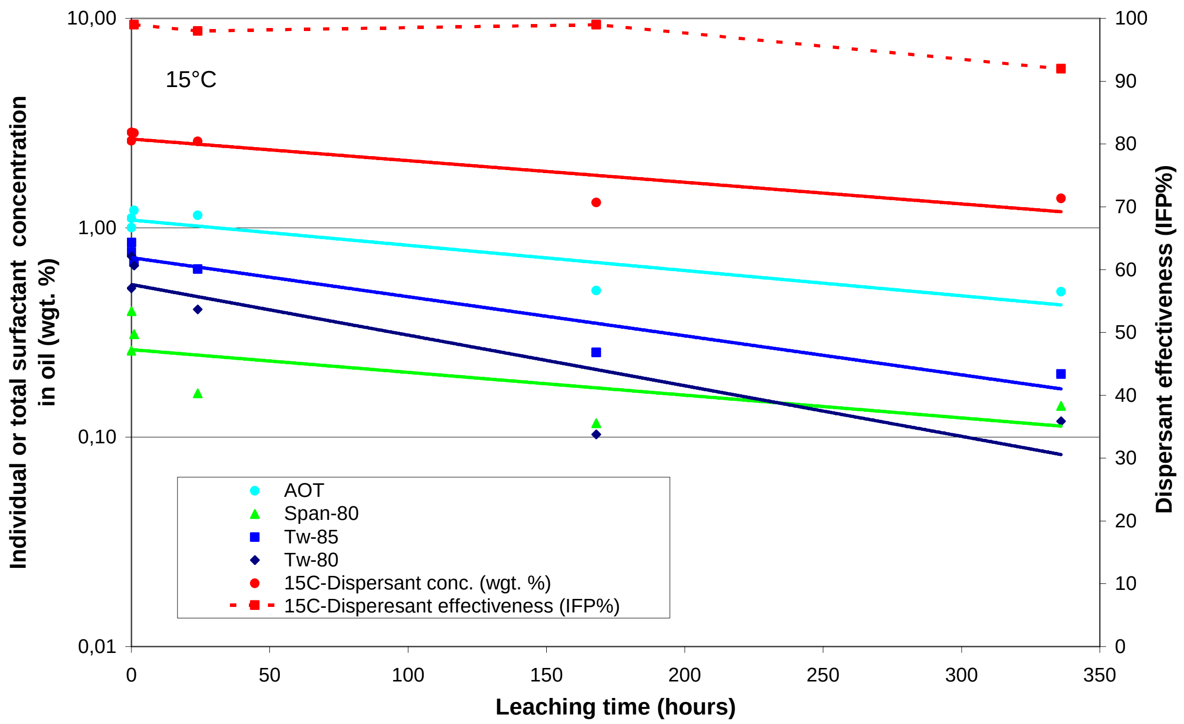

Figure 5.8 Effectiveness and surfactant leaching for the model dispersant at 15ºC with the 200°C+ Troll B crude from 1 minute to 2 weeks.

At 0ºC (Figure 5.9), the total dispersant loss from the Troll oil was very similar to the 15°C tests and reduced to 1.4% at 2 weeks. The behaviour of the individual surfactants was somewhat different with a larger reduction of AOT at 2 weeks while the other surfactants had less leaching. This resulted in a different relative composition of surfactants in the oil at the test end. Similar to the 15°C tests, the napthenic oil retained a high dispersability after 2 weeks (86%) even with a significant surfactant loss.

As mentioned, the napthenic Troll B oil was not tested at 25°C because it was expected to disperse at least as well as it did at 15°C.

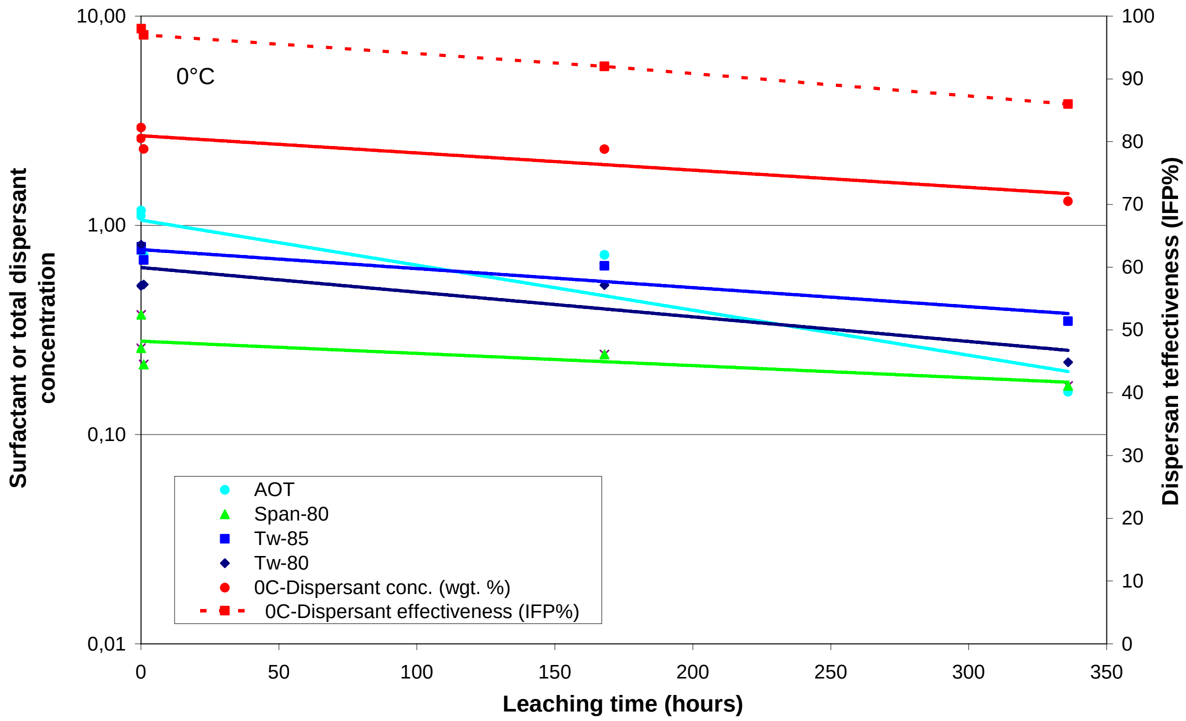

Figure 5.9 Effectiveness and surfactant leaching for the model dispersant at 0ºC with the 200°C+ Troll B crude from 1 minute to 2 weeks.

The leaching of surfactants and the dispersant effectiveness (%) for the asphaltenic and low wax content Balder oil are presented in *Figure* 5.10 to *Figure* 5.12 at 25, 15, and 0°C, respectively.

The reduction of the total dispersant concentration from the initial 3.3% in the oil is very dependant on the temperature. The dispersant content after two weeks is approximately 0.1% at 25°C, 1.5% at 15°C and 2.8% at 0°C. The same trend is seen with the ionic surfactant AOT, which has corresponding contents of 0.01, 0.39, and 0.95%. The initial content of AOT premixed in the oil is 1.4%. The loss of the other polar surfactants Tween-80 and 85 (high HLB) shows similar trends versus temperature.

For the Balder oil, dispersant effectiveness actually increased from 69 to 88% as the test temperature was reduced from 25°C to 0°C. Thus, for the asphalthenic and low-wax content oil, the reduction in effectiveness is correlated with the surfactant leaching and not with the reduction in temperature.

The concentrations of the unpolar Span-80 (low HLB) are increasing in the figures for 15 and 0ºC (see *Figure* 5.11 and *Figure* 5.12). This is probably an artefact of the multivariate calibration used in this study. In the d-optimal calibration set (see Table D.0.1) no samples have low concentrations of both tween-80 and tween-85. This is the situation in some of these analysed samples, due to the very high leaching from the Balder oil. Some of these surfactant predictions (very high Span and very low Tweens) are based on extrapolations outside the calibration set and should be used with caution.

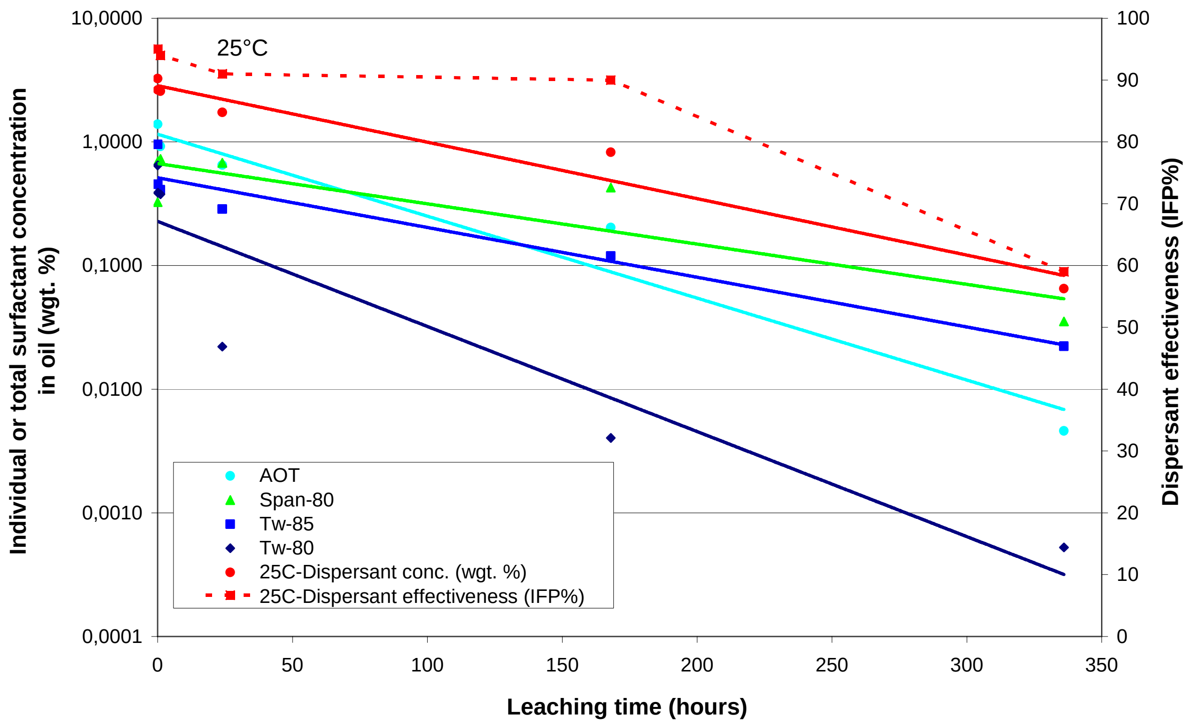

Figure 5.10 Effectiveness and surfactant leaching for the model dispersant at 25ºC with the 200°C+Balder crude from 1 minute to 2 weeks.

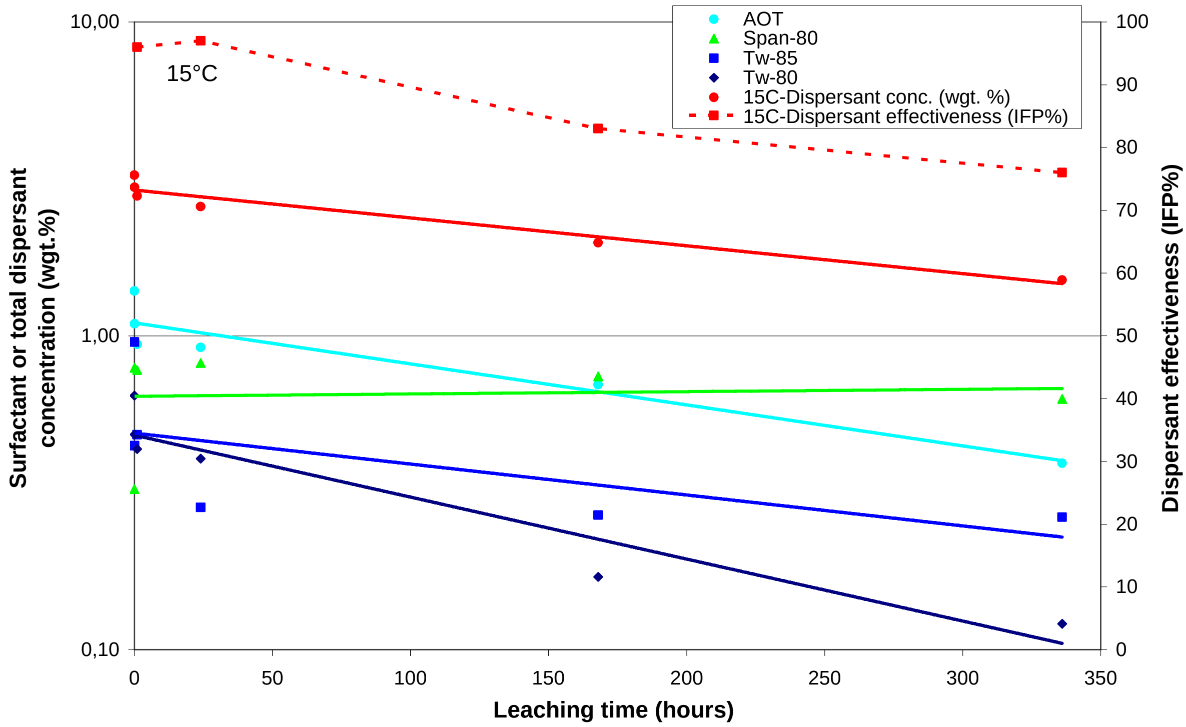

Figure 5.11 Effectiveness and surfactant leaching for the model dispersant at 15ºC with the Balder crude 200°C+ from 1 minute to 2 weeks.

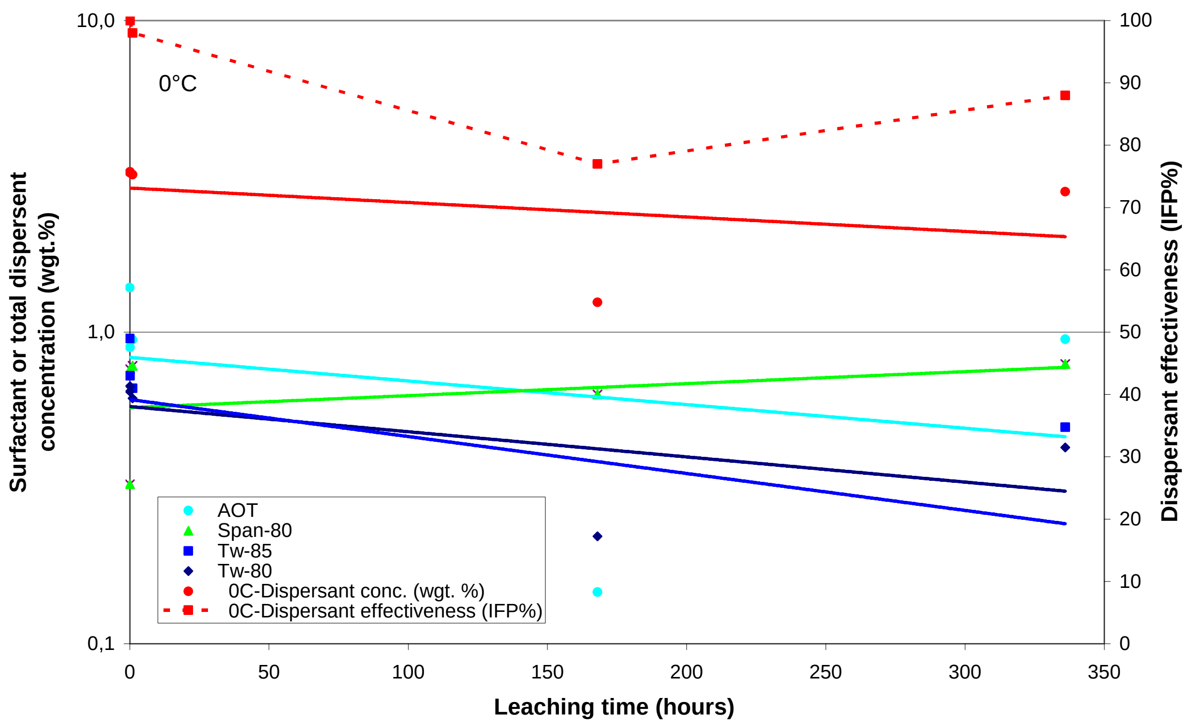

Figure 5.12 Effectiveness and surfactant leaching for the model dispersant at 0ºC with the Balder crude 200°C+ from 1 minute to 2 weeks.

The leaching data and the dispersant effectiveness data for the paraffinic Oseberg are presented in *Figure* 5.13 to *Figure* 5.15.

The reduction of the total dispersant concentration from the initial 2.6% in the oil is very dependant on the temperature. The dispersant contents after two weeks are approximate 1.1% at 25°C and 2.1 at 15°C, and 2.2% at 0°C. The same trend is seen with individual surfactants versus temperature, but the leaching rates for the four surfactants are similar causing only small changes in the dispersant formulation.

Based on the leaching data (1-2% dispersant remained at 2 weeks and unchanged formulation) a high dispersant effectiveness would be expected, but we observe a clear decreasing trend with temperature and time (83% at 25°C, 53% at 15°C, and 15% at 0°C at 2 weeks) probably due to wax precipitation for this oil that contained 2.9% wax. The pour point of the 200°C+ Oseberg sample was 9°C, but Figure 5.1 shows that wax start to precipitation begins at 23°C. This probably results in significant wax precipitation during the test period at both 15°C and 0°C.

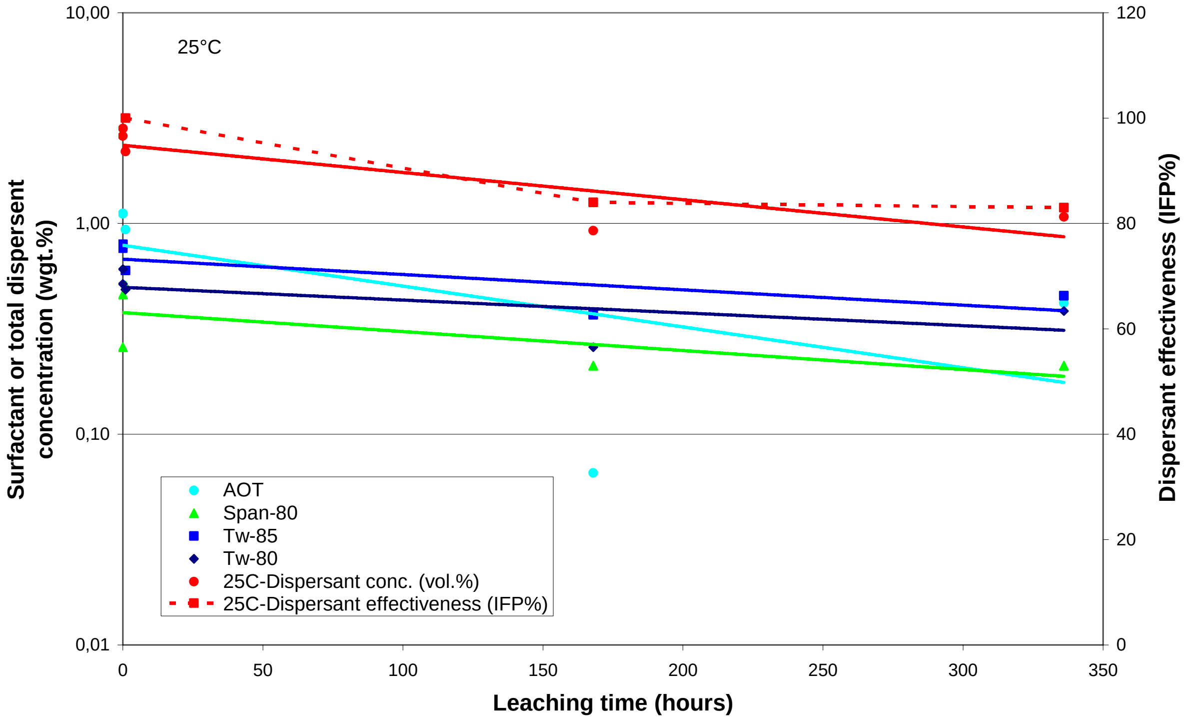

Figure 5.13 *Effectiveness and surfactant leaching for the model dispersant at 25ºC with the Oseberg crude 200°C+. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks).*

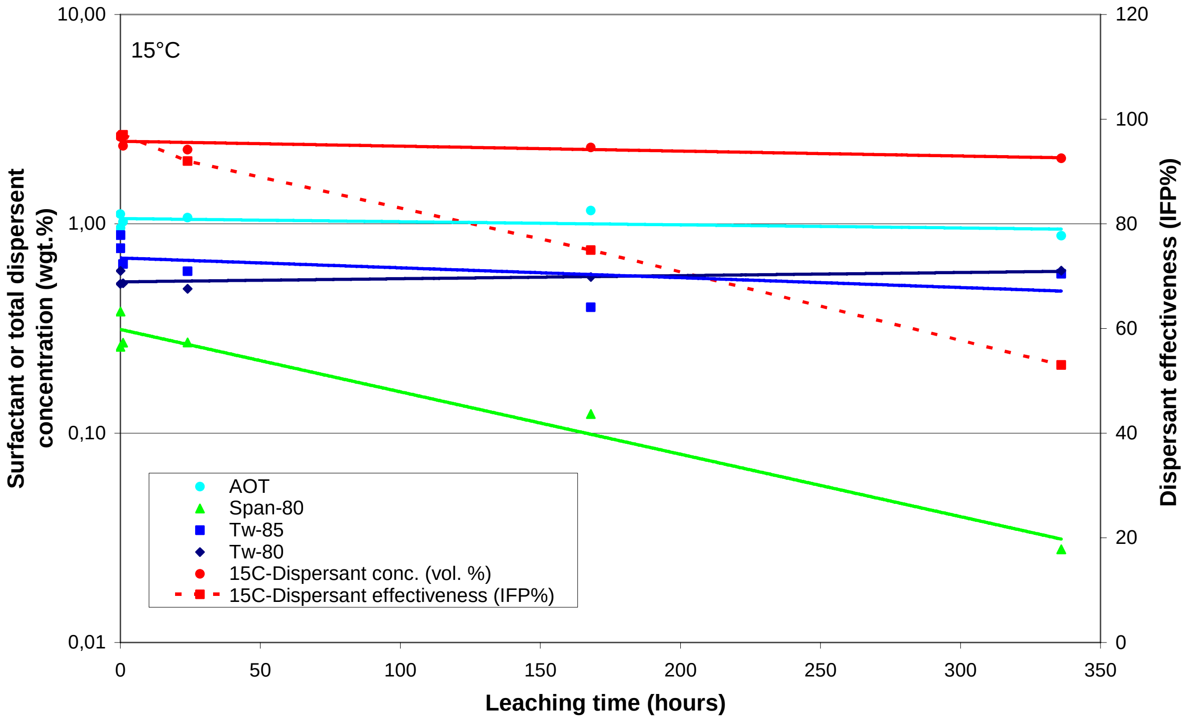

Figure 5.14 *Effectiveness and surfactant leaching for the model dispersant at 15ºC with the Oseberg crude 200°C+. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks)*

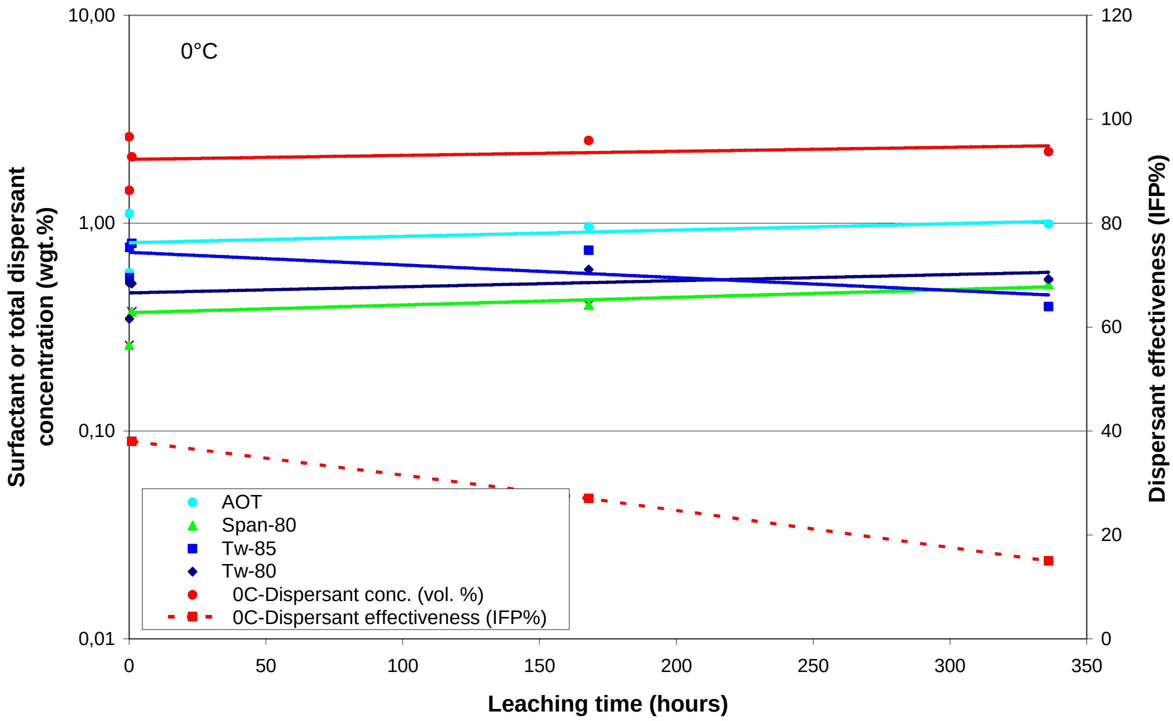

Figure 5.15 Effectiveness and surfactant leaching for the model dispersant at 0ºC with the Oseberg crude 200°C+. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks)

The leaching data and the dispersant effectiveness data for the waxy Ringhorne oil are presented in Figure 5.16 and Figure 5.17.

The reduction of the total dispersant concentration from the initial 3.3% in the waxy Ringhorne oil is dependant on temperature. The dispersant content after two weeks is only 0.4% at 25, but 1.7% at 15°C. The same trend is seen with the individual surfactants versus temperature, but the leaching rates for the four surfactants are similar causing only small changes in the dispersant formulation.

Based on the dispersant leaching data (1.7% residual dispersant and relatively unchanged formulation) a high dispersant effectiveness would be expected, but we observe a clear decreasing trend with temperature and time due to high pour point and wax precipitation in this oil. This causes formation of a wax lattice and leads to a semi-solid (cohesive) oil phase, which reduces the potential of using dispersants.

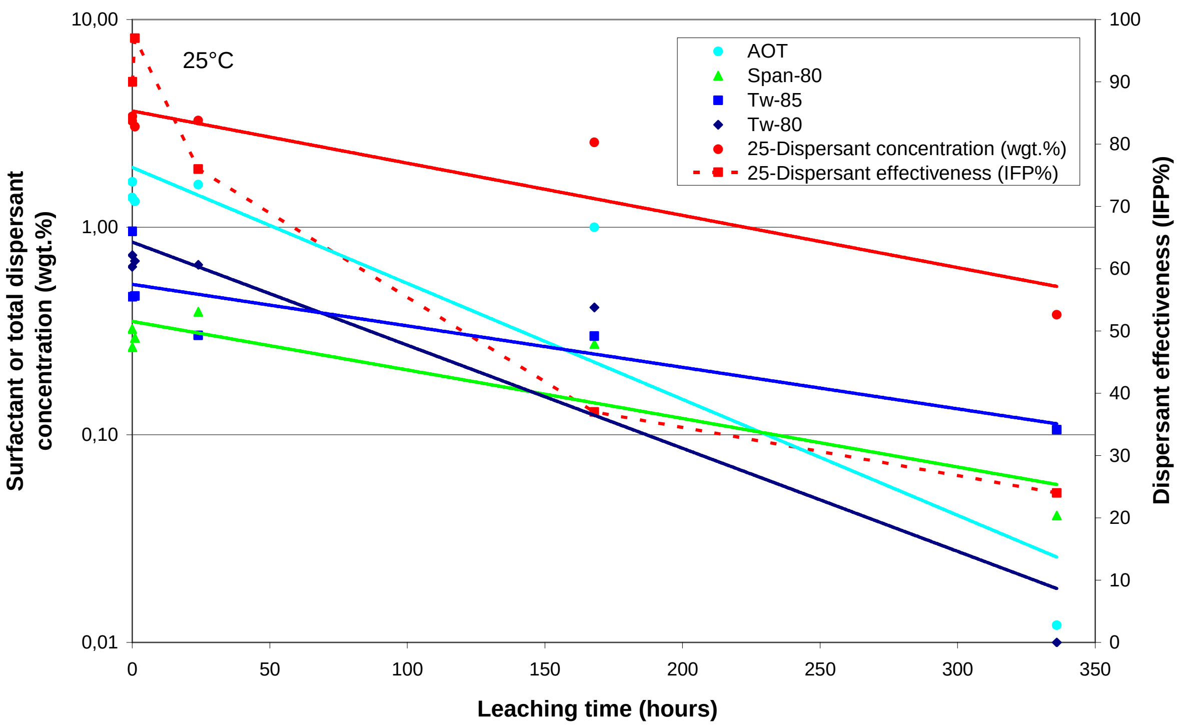

Figure 5.16 Effectiveness and surfactant leaching for the model dispersant at 25ºC with the Ringhorne crude 150°C+. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks)

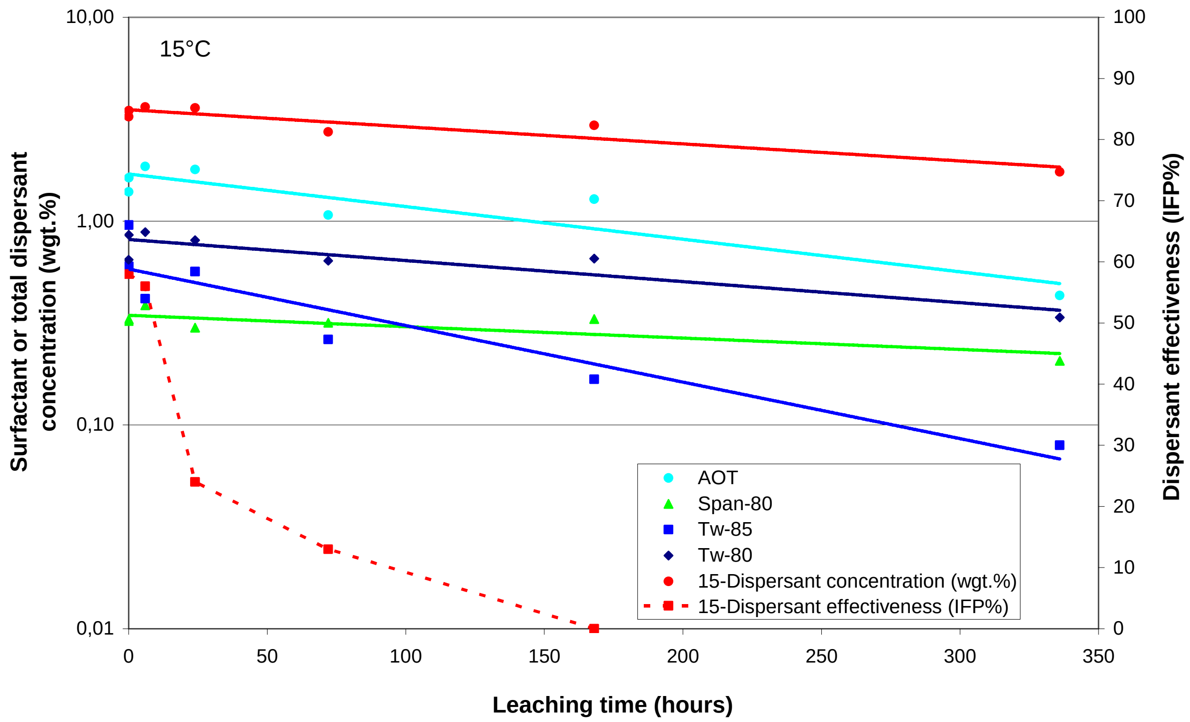

Figure 5.17 *Effectiveness and surfactant leaching for the model dispersant at 15°C with the Ringhorne crude 150°C+. Contact time: 1 minute, 1 hour, 24 hours, 168h (1 week) and 336h (2 weeks)*

5.5 Dispersant Effectiveness versus surfactant leaching after oil being captured in ice

An objective of this project was to evaluate spill scenarios in ice-prone regions by determining if dispersants remain effective over time when frozen into an ice layer and when placed on top of an ice layer and the ice subsequently thawed at 0°C. Experiments were performed only with the napthenic and asphalthenic oils (Troll and Balder), due to their low wax content and low pour points.

In the first series oil was placed in the IFP containment ring on top of an ice layer in the IFP beaker. Contact/freezing time was 48 hours, 2 weeks and 1.5 months at an air temperature of -20°C. These experiments were performed on the naphthenic oil (Troll B, 200°C+) and the asphalthenic oil (Balder 200°C+). The second series included placing oil in the containment ring on top of seawater, lowering the air temperature to -20°C and slowly freezing the top 15 cm of water to ice. Contact/freezing time was 48 hours, 2 weeks and 1.5 months at an air temperature of -20°C. These experiments were performed on the naphthenic oil (Troll B, 200°C+) and the asphalthenic oil (Balder 200°C+).

According to the project specification a limited number of experiments were used for these ice experiments. Three different leaching times were used and only single replicates performed to quantify surfactant leaching. See the experimental section for further details. The deviation in dispersant effectiveness was also larger for these ice experiments (StDev > 10), compared to the ordinary open water experiments (StDev<5).

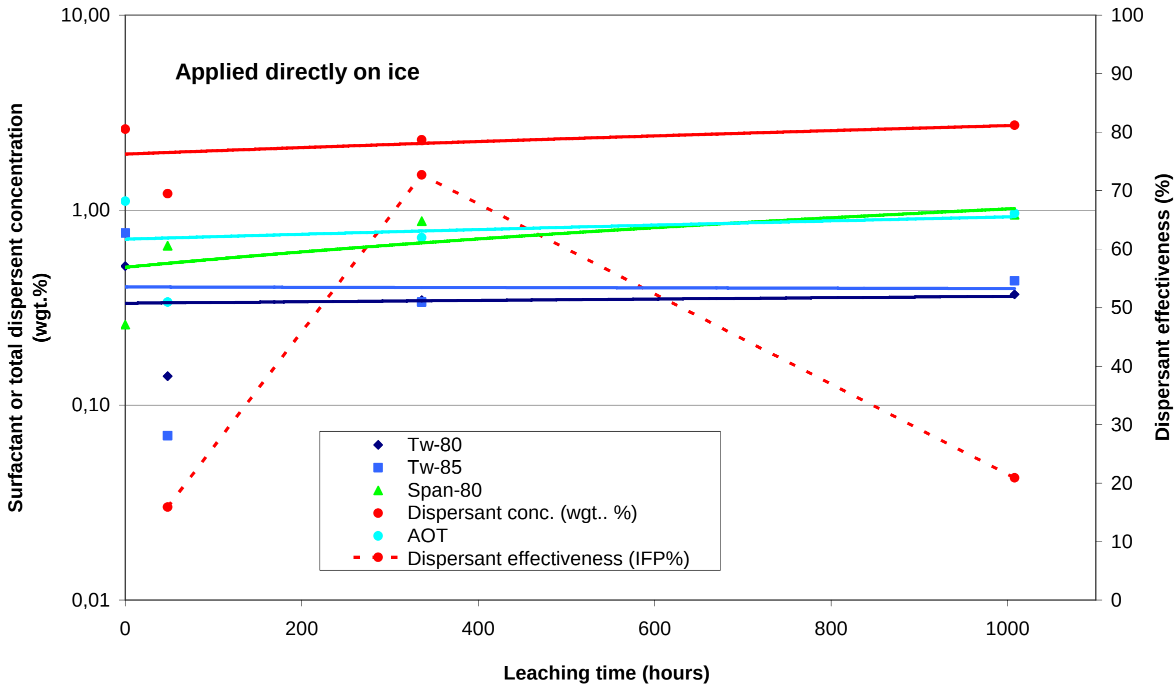

Figure 5.18 *Effectiveness and surfactant leaching for the model dispersant with the Balder crude 200°C+ applied directly on ice. Contact time: 48 hours, 336h (2 weeks) and 1008 hours (6 weeks).*

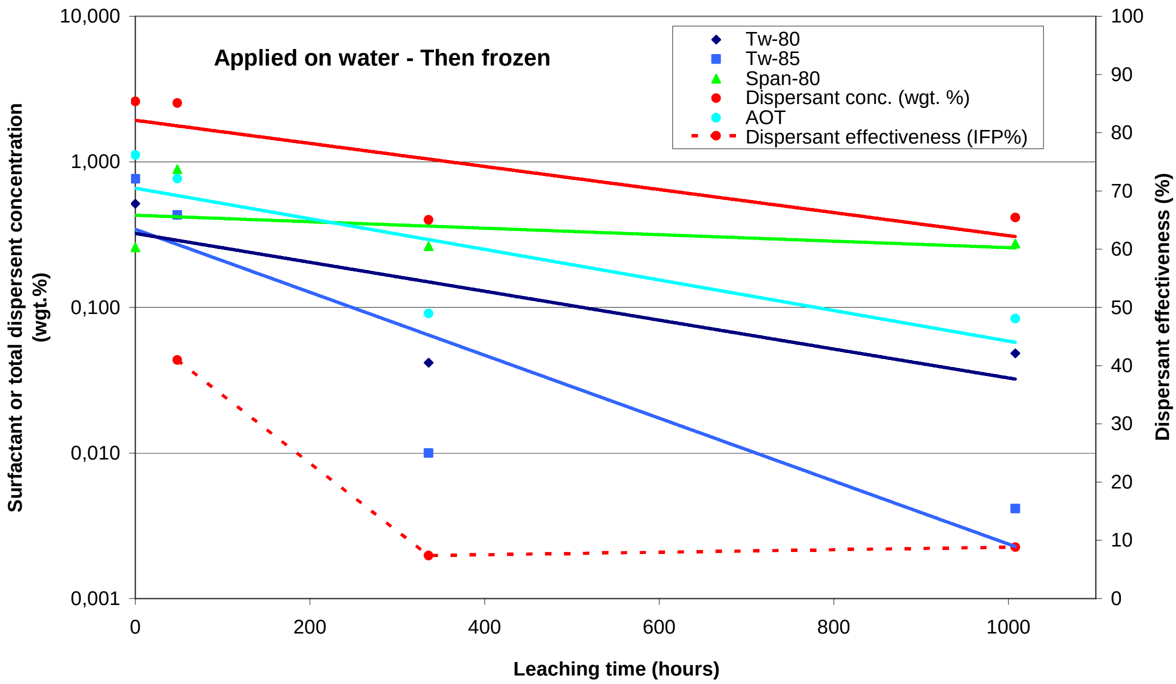

Figure 5.19 *Effectiveness and surfactant leaching for the model dispersant with the Balder crude 200°C+ first applied on water, then frozen. Contact time: 48 hours, 336h (2 weeks) and 1008 hours (6 weeks).*

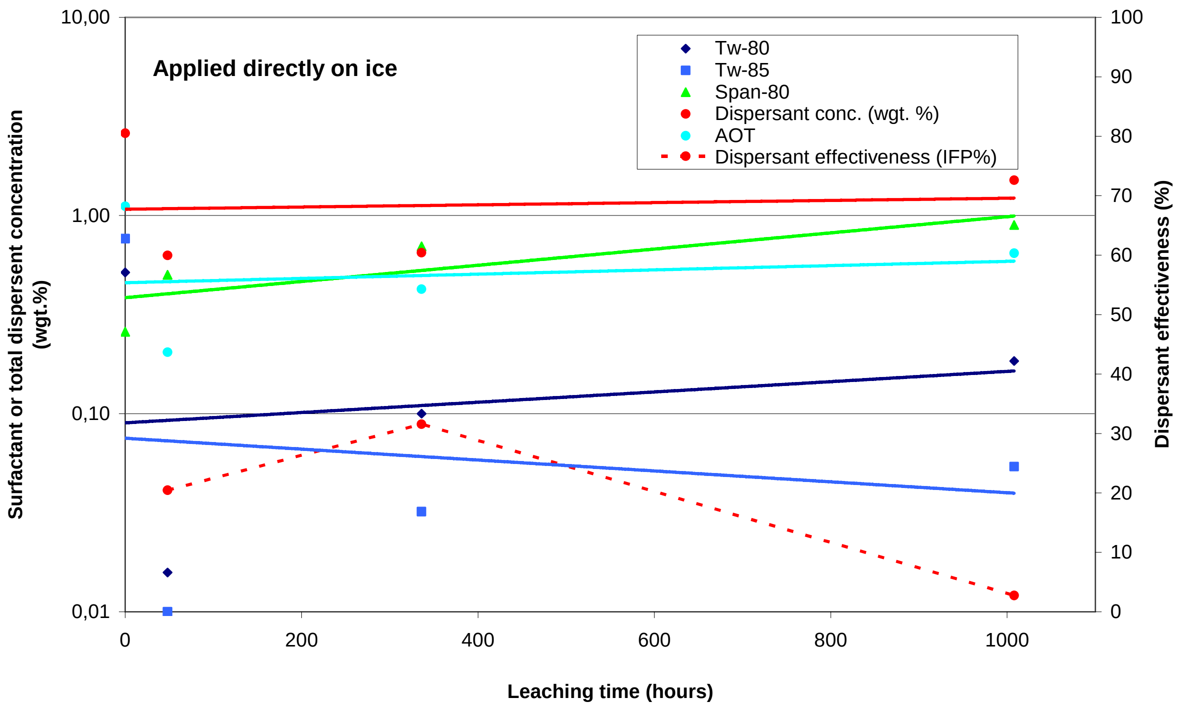

Figure 5.20 Effectiveness and surfactant leaching for the model dispersant with the Troll crude 200°C+ applied directly on ice. Contact time: 48 hours, 336h (2 weeks) and 1008 hours (6 weeks).

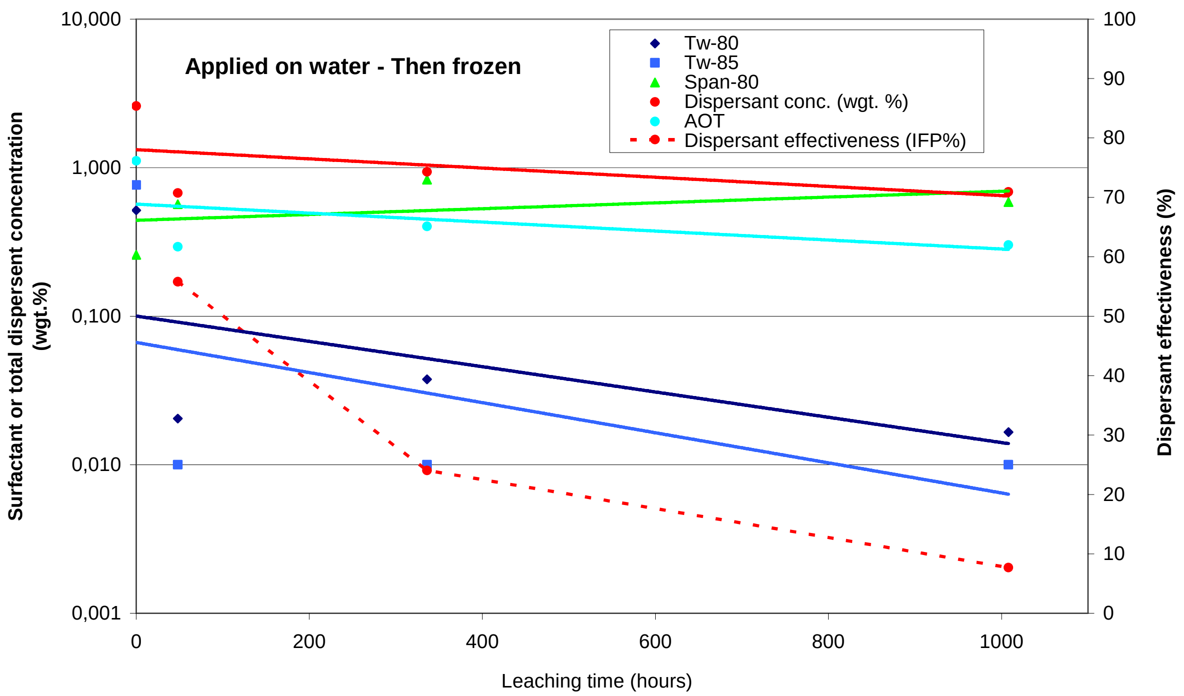

Figure 5.21 Effectiveness and surfactant leaching for the model dispersant with the Troll B crude 200°C+ first applied on water, then frozen. Contact time: 48 hours, 336h (2 weeks) and 1008 hours (6 weeks).

These limited experiments with leaching of surfactants from oil trapped in ice show a clear reduction in dispersant effectiveness. This reduction to below 20% is very large compared to testing with the same oils in open water (*Figure* 5.8 and *Figure* 5.9). The reason for this could be the extended leaching time, but more probable the exposure of the oil to the low air temperature (-20°). This extended period at low temperature has probably altered the structure of, even these low wax content, oils to give a very low dispersant effectiveness at the end of the experiment.

5.6 Documentation of possible spontaneously formed micron sized oil droplets

No spontaneous formation of micron sized oil droplets were observed in the experiments, but a "milky cloud" was observed after a contact time between oil and water of 24-48 hours in some of the experiments. Figure 5.22 and Figure 5.23 show the "milky cloud" below two different oil types. The size of the "milky cloud" was different from oil type to oil type.

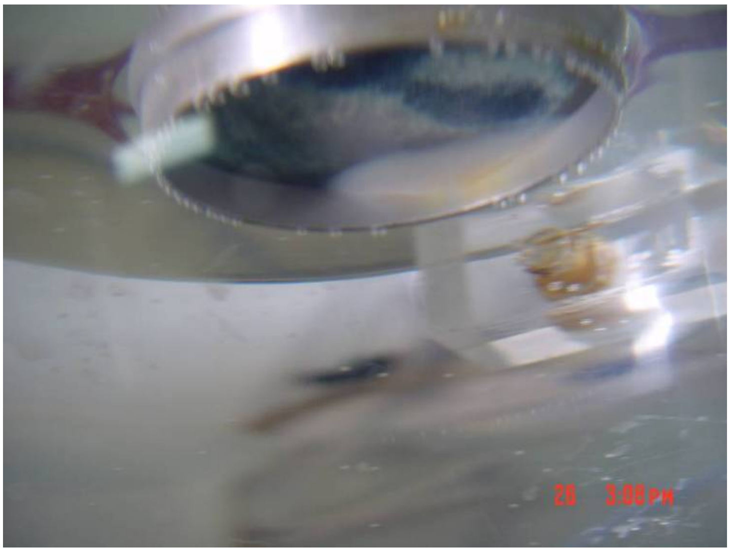

Figure 5.22 Observation of "milky cloud" under a premix of Model dispersant and Troll 200°C after 48 hours oil/water contact time at 15°C.

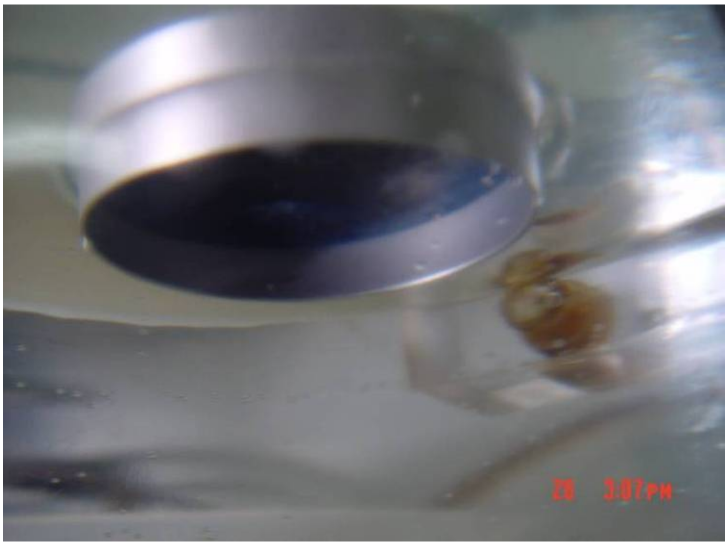

Figure 5.23 Observation of "milky cloud" under a premix of Model dispersant and New Oseberg Blend 200°C after 48 hours oil/water contact time at 15°C.

Several samples were taken from these "milky clouds" and analyzed by fluorescence microscopy and gas chromatography. This was done to detect and quantify microbial or fungal activity or oil droplets. No increased numbers of micro organisms or oil patterns were detected in these samples.

The most likely source for these “milky clouds” is the surfactants leaching out of the oil phase and creating micelles. Micelles are clusters of surfactants and are formed when the surfactant concentration increases above their solubility in water. The appearance of the “milky clouds” after 24-48 hours also strengthens this hypothesis, since the surfactant leaching is at its highest rate during this time interval (see *Figure* 5.8 as an example).

6 Conclusions and recommendations

The overall objective of this study was to determine whether dispersants will remain with treated oil slicks over time and retain effectiveness. This would make it possible to apply dispersants to an oil slick under low energy conditions and delay the dispersion until the energy level increases. One possible scenario could be treating an oil spill captured in ice for later dispersion when the ice melts and sufficient energy for effective dispersion occurs.

6.1 Conclusions

The main conclusion from the effectiveness testing of dispersants on the four oils used in this study is that dispersants may retain good effectiveness for significant time periods but this is dependant on the oil rheological properties and on the environmental temperature.

The dispersant effectiveness did not correlate well with the remaining surfactant content in the oil. That is, high dispersant effectiveness was achieved even when over 75% of the surfactants had leached from the oil in some tests. In other tests, poor dispersant effectiveness was observed even when most of the surfactant remained in the oil. Thus, the evidence from these tests indicates that physical properties of the oil, primarily the formation of wax precipitates at low temperatures, are important factors controlling dispersant effectiveness.

Naphthenic and asphalthenic oils remained dispersible even after two weeks of prolonged contact time at all temperatures (0-25ºC) in the ice-free tests.

The dispersant effectiveness after two weeks with a paraffinic oil depended strongly on the spill conditions. The potential for retaining good effectiveness is high at sea temperatures higher than the pour point of the oil and low at sea temperatures lower than the pour point.

For waxy oils the potential for retaining good effectiveness of dispersant by time is low, because the pour point is high and normally lower or close to sea temperature. This will cause formation of a wax lattice and lead to a semi-solid (cohesive) oil phase, which will reduce the potential of using dispersants.

No clear conclusion could be drawn from the limited series of leaching experiments with oil in ice. The low dispersant effectiveness can not be explained by leaching of surfactants and are probably caused by the long exposure to low air temperature causing structural changes to the oil (semi-solidification).

6.2 Recommendations

The results from this study show that dispersant effectiveness is still high after two weeks of prolonged contact time, even when the formulation of the dispersant is changed due to different leaching rates of the individual surfactants and the amount of residual dispersant is low (<1%). The following activities are proposed to investigate this further:

1. Produce dispersants formulated to reflect the changing formulation measured by the leaching studies and test these formulas on the study oils. This would verify the relationship between surfactant leaching, changes in dispersant composition and possible changes in dispersant effectiveness.
2. Since the leaching of surfactants seems to "level out" at a residual concentration of <1% in the oil, experiments with a lower initial concentration should be performed. This could reduce the leaching, since the initial concentration gradient is lower.

The non-conclusive result from the leaching in ice experiments is probably caused by the experimental setup with prolonged exposure to the low air temperature. New experiments should be performed with a more realistic air temperature exposure.

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Appendix A Literature Review - SINTEF References

Relates to Laboratory Study of the Effects of time on the Effectiveness of Dispersants. Task 1 in PERF 2004-05 JIP Agreement, UR-01704

SINTEF

SINTEF MEMO

SINTEF Materials and Chemistry

Address:	NO-7465 Trondheim, NORWAY
Location:	Brattørkaia 17B, 4. etg.
Telephone:	+47 4000 3730
Fax:	+47 930 70730

Enterprise No.: NO 948 007 029 MVA

TITLE

Literature Rewiev of SINTEF References that Relates to Laboratory Study of the Effects of time on the Effectiveness of Dispersants.

Task 1 in PERF 2004-05 JIP Agreement, UR-01704

AUTHOR(S)

Janne Lise Myrhaug Resby, Per Johan Brandvik

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ExxonMobil Upstream Research Company

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	2005-10-25	Tore Aunaas	

ABSTRACT

A complete list of references from SINTEF databases is provided in this document. The references are marked for the relevans for SINTEFs overall understanding of oil weathering, physico-chemical properties and dispersibility. References used in the planning and design of the PERF project are marked in the reference list. Original abstracts or front pages of reports of references used in the planning and design of the PERF project is scanned and attached. The abstracts or front pages in Norwegian are translated to English.

References form *Cedre* are reported in a separate report.

The literature review of earlier work at SINTEF and *CEDRE* give indications that dispersant effectiveness can be retained during prolonged contact time with sea water after dispersant application.

There is identified a need for more sophisticated analysis of surfactant content in the oil phase or in the water phase to quantify surfactant leaching.

However, no information has been found during this review that introduces a need for major changes in the established plan or design of the “Laboratory study of the effects of time on the effectiveness of dispersants (PERF project)”.

KEYWORDS	ENGLISH	NORWEGIAN
GROUP 1	Dispersants	Dispergeringsmiddel
GROUP 2	Leaching	Utlekking
SELECTED BY AUTHOR	Litterature review	Litteratur gjennomgang
	Oil	Olje
	Surfactants	Surfaktanter

8 Introduction

In relation to Task 1 in PERF 2004-05 JIP Agreement, UR-01704: Laboratory study of the effects of time on the effectiveness of dispersants (PERF project), a literature review should be prepared in accordance to Task 1. The objective of literature search was to include a complete list of references from SINTEF and Cedre. The abstracts from references that relate to this project were to be translated to English, if necessary, and a brief analysis of relevance was to be prepared.

9 Complete list of SINTEF references

A complete list of references from SINTEF databases is provided in this document. The references are marked for the relevance for SINTEFs overall understanding of oil weathering, physico-chemical properties and dispersibility. References used in the planning and design of the PERF project are marked in the reference list. Original abstracts or front pages of reports of references used in the planning and design of the PERF project is scanned and attached. The abstracts or front pages in Norwegian are translated to English.

Table 3 Complete list of SINTEF references. The references are marked for the relevance for SINTEFs overall understanding of oil weathering, physico-chemical properties and dispersibility. References used in the planning and design of the PERF project are marked as references that relate to PERF project.

		Importance for SINTEFs overall understanding of oils:			
Ref #	References:	Weathering processes	Physico-chemical properties	Dispers-ibility	References that relate to PERF project
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721	AAMO, O. M., REED, M., RYE, H., DALING, P. S. 1995: Oil spill combat simulations for Esso Slagentangen ESCOST Report no. 20 Final report. IKU Report 22.2049.00/01/95 31p. Restricted.				
722	AARESKJOLD, K. 1993: Spredning av olje og kondensat i sjøvann. IKU Report 22.2080.00/01/93 32p. Restricted.				

10 References used in the planning and design of the laboratory study of the effect of time on the effectiveness of dispersants.

10.1 Ref # 4

Oil Spill R&D in Norwegian Arctic Waters with special Focus on Large-scale Oil Weathering Experiments

P. J. Brandvik[1], I. Singsaas[2] and P. S. Daling[2]

[1] University centre at Svalbard (UNIS), Pb 156, N-9171 Longyearbyen, Norway
[2] SINTEF Materials and Chemistry, Marine Environmental Technology, N-7465 Trondheim, Norway

Abstract

The blow-out on Ekofisk in 1977 showed that the Norwegian preparedness for handling offshore oil spills was limited. The release lasted for seven days and totally 13 000 tons of crude oil were released. Sampling and monitoring of this first major Norwegian oil spill showed that the evaporative loss and natural dispersion were surprisingly high for this light North Sea crude. These findings initiated several substantial national R&D programs focusing on modeling of oil drift, mechanical recovery off-shore, environmental consequences and weathering processes in marine oil spills.

In the decade from 1985 exploration in the Barents Sea, and even on Svalbard, initiated several programs to develop new or adapt existing oil spill technology to Arctic conditions. With Arctic conditions we here mean low temperatures, possible presence of ice, darkness in the winter season and often long distances and lack of infrastructure. Development of skimmers, operationalisation of in-situ burning and the use of dispersants and studies of bioremediation were important R&D activities. Due to lack of major oil discoveries, the oil companies lost interest for the Norwegian Arctic areas in the late 1990ties and the funding for Arctic related R&D dried up. At present the interest in Norway for oil spill countermeasures in northern areas is again increasing, partly due to reopening of the Barents Sea for exploratory drilling and partly due to the increasing tanker traffic outside the Norwegian coast from Russia to Europe and USA.

To study the difference between an oil spill in temperate open water and in broken ice conditions important oil properties as evaporative loss, water content, emulsion viscosity and oil density are compared for two large-scale experimental oil releases (30 and 26 m^3 of crude oil).

State-of-the-art trajectory and oil weathering models can be used to predict both oil drift and weathering processes of oil spills in cold waters (without ice) with a accuracy sufficient for most operational purposes. This is possible after several decades with full-scale field experiments in Norway combined with the effort of several R&D programs. The present situation regarding knowledge and modeling capability concerning Arctic oil spills (broken ice) is however far from this. Large-scale field experiments in broken ice are very limited and there is a lack of knowledge regarding oil weathering and the dependence of environmental conditions in a broken ice scenario.

Since both oil transport and exploration are increasing in Arctic waters increased understanding of oil weathering processes under these conditions is needed. This is important both for environmental risk assessment studies, for oil spill contingency planning and to increase the operational capability for handling oil spills in Arctic areas.

10.2 Ref # 28

IKU SINTEF GROUP	REPORT
IKU Petroleumsforskning a.s IKU Petroleum Research N-7034 Trondheim, Norway Phone: +47 7 59 11 00 Fax: +47 7 59 11 02 (aut.) Telex: 55 434 iku n	TITLE **Testing of dispersants under arctic conditions - a laboratory study.** **DIWO Report no. 18** AUTHOR(S) P.J. Brandvik, M. Moldestad, O.Ø. Knudsen and P.S. Daling.
CLASSIFICATION Unrestricted REPORT NO. 22.2030.00/18/93	CLIENT(S) Fina Exploration Norway u.a.s. Att.: Olaf Gram

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93.158	22-NOV-93	Ole Øystein Knudsen	Ole Ø Knudsen
NO. OF PAGES	NO. OF APPENDICES	SCIENTIFIC CONTROL	SIGN.
50	-	Ivar Singsaas	Ivar Singsaas

SUMMARY

This report presents a laboratory screening of the effectiveness of 14 commercial dispersants at low temperatur and both high and low salinity (0.5 and 3.5%). An extended testing of the five most promising dispersants from the screening test with several oiltypes is also given. In addition, dispersant effectiveness as a function of salinity is tested with the most effective dispersant reported at high and low salinity.

The results from this study show that many dispersant which previously have shown a high effectiveness at high salinity (3.5%), may give a very low effectiveness under low salinity conditions (0.5%). This is of significant operational importance in arctic oil spill combat operations since the salinity of the surface water may vary e.g. in a ice melting situation.

Recently developed products especially designed for low salinity use are very effective at low salinities, but suffers from a rather poor effectiveness at higher salinities.

This study of dispersant effectiveness under arctic conditions shows the need for further development of dispersants with high effectiveness both at low temperature (0°C) and over a wider range of salinities (e.g. from 0.5% to 3.5%).

KEYWORDS ENGLISH	KEYWORDS NORWEGIAN
Oil Spill	Oljesøl
Oil weathering	Forvitring av olje
Dispersant	Dispergeringsmiddel
Arctic conditions	Arktiske betingelser

10.3 Ref # 56

SINTEF REPORT

SINTEF

SINTEF Applied Chemistry

Address: N-7034 Trondheim, NORWAY
Location: S.P. Andersens vei 15 b
Telephone: +47 73 59 28 73
Fax: +47 73 59 70 51

Enterprise No.: NO 948 007 029 MVA

TITLE

Procedures for analysis of oil spill chemicals

DIWO Report no. 28.

AUTHOR(S)

Ole Øystein Knudsen, Jorun Nerbø Hokstad and Per Johan Brandvik

CLIENT(S)

Fina Exploration Norway Inc.

REPORT NO.	CLASSIFICATION	CLIENT'S REF.	
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CLASS. FRONT PAGE	**ISBN**	**PROJECT NO.**	**NO. OF PAGES/APPENDICES**
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Diwo-rep-28.doc		Per Johan Brandvik PJB	Ivar Singsaas
FILE CODE	**DATE**	**APPROVED BY (NAME, POSITION, SIGN.)**	
	05.03.97	Tore Aunaas, Research Manager	

ABSTRACT

The objective with this activity in the DIWO-2 project has been to find analytical methods for quantifying and identifying surfactants in commercial oil spill chemicals and in water samples. The methods were used for analysing commercially available oil spill chemicals and to study surfactant leaching.

Different chromatographic techniques (HPLC, GC, Iatroscan TLC) have been tested, but finding a chromatographic method which could quantify the complex mixture of surfactants used in dispersants turned out to be very difficult. The main reasons for this are that dispersants consists of surfactants with very different chromatographic properties (ionic, lipophilic and hydrophilic components) and that they have weak UV/VIS absorbance which make them difficult to detect. Each type of surfactants e.g. a specific ethoxylated sorbitan was also present in the dispersant as many different modifications (ethoxylation degree, number of and types of fatty acids, homologues, isomers etc.).

The strategy was therefore changed, and different methods found in the literature for analysing each **group** of surfactants in the oil spill chemicals were established.

Anionic surfactants in water samples and in neat oil spill chemicals was quantified by forming a complex with methylene blue which could be determined colorimetrically. Poly ethoxylated surfactants were quantified after derivatisation of the poly ethoxy chain to 1,2-dibromo ethane which was quantified by GC. Quantitative and qualitative analysis of sorbitan esters were done by hydrolysing the ester, methylation of the fatty acids and identification/quantification of the fatty acid by GC analysis. Surfactants in water samples were concentrated by solid phase extraction on C_{18} Maxi-Clean cartridges.

The composition of 9 commercial dispersants were analysed.

KEYWORDS	ENGLISH	NORWEGIAN
GROUP 1	Chemistry	Kjemi
GROUP 2	Contingency	Beredskap
SELECTED BY AUTHOR	Analysis, Oil Spill Chemicals	Analyse, Oljevernkjemikalier
	Surfactants, Solvent	Surfaktanter, Løsningsmiddel

10.4 Ref # 76

Report in Norwegian, See next page for a translation of the title and the summary.

IKU
SINTEF GRUPPEN

Institutt for kontinentalsokkelundersøkelser og petroleumsteknologi A/S
Continental Shelf and Petroleum Technology Research Institute
S.P. Andersen veg 15 b * N-7034 Trondheim, Norway
Tel.: + 47 7 591100 * Telex: 55 434 iku n * Telefax: + 47 7 591102 (aut.)

Front page is now clasified as Open
Oct-2007: PJB

RAPPORT

REG.NR.:	TILGJENGELIGHET:
91.148	Begrenset

RAPPORT TITTEL:

TESTING AV DISPERGERINGSMIDLERS EFFEKTIVITET UNDER ARKTISKE BETINGELSER

RAPPORT NR.: 22.2008.00/01/91

FORFATTER(E):

Per S. Daling, Ivar Singsaas, Jorunn Nerbø Hokstad

DATO:	ANT. SIDER:	ANT. BILAG:	PROSJEKTLEDER:	SIGN.:
13 des.-91	76	-	Per S. Daling	Per S. Daling
OPPDRAGSGIVER: NOFO v/ Widar Skogly			GODKJENT AV FAGLIG ANSVARLIG: Liv Schou	SIGN.: Liv Schou

SAMMENDRAG:

Gjennom en systematisk uttesting av ulike dispergeringsmidlers effektivitet i laboratoriet ved IKU har vi kommet frem til hvilke dispergeringsmidler som har størst effektivitet på relevante norske råoljer med forskjellig grad av forvitring/emulsjons-dannelse og ved ulike arktiske betingelser (lave temperaturer, lav salinitet, tilstedeværelse av is etc.).

Videre er effekten av utvasking av dispergeringsmiddel fra en oljefilm og ned i underliggende vannmasser undersøkt.

Potensialet for bruk av dispergeringsmidler på olje på is og i smeltevannsdammer ble studert i et mindre feltforsøk på Svalbard.

Prosjektet danner et viktig grunnlag for utvelgelse av aktuelle produkter til videre studier i meso-skala is-renneforsøk og til fremtidige stor-skala feltforsøk i arktiske farvann.

STIKKORD:	KEY WORDS:
Arktisk oljevern	Arctic oilspill combat
Dispergeringsmidler	Dispersants
Effektivitet	Effectiveness

Skipnes

Title: Testing of dispersant's efficiency in Arctic conditions

Summary: IKU has, through systematic laboratory testing of dispersant efficiency, determined which dispersants have the highest/best effect on relevant Norwegian crude oils with different degrees of weathering / emulsion formation and at different Arctic conditions (low temperatures, low salinity, presence of ice etc.).

In addition, the effect of leaching of dispersant from an oil film and down into underlying water masses has also been examined/tested.

The potential for use of dispersants on oil on ice and in melting pools was studied in minor field trials on Svalbard.

The project is an important basis for the choice of current products for further studies in meso-scale flume testing, and for future large-scale field trials in Arctic waters.

10.5 Ref # 201

IKU SINTEF GROUP **IKU Petroleumsforskning a.s** **IKU Petroleum Research** N-7034 Trondheim, Norway Telephone: +47 73 59 11 00 Fax: +47 73 59 11 02 (aut.) Telex: 55 434 iku n Enterprise no.: NO 936 882 331 MVA	**IKU REPORT** TITLE **Oil-surfactant interaction and mechanism studies - Part 1: Leaching of surfactants from oil to water. Chemical composition of dispersed oil.** **ESCOST report no. 21. DRAFT VERSION.** AUTHOR(S) Jorunn Nerbø Hokstad, Børre Knudsen and Per S. Daling. Front page is now clasified as Open Oct-2007: PJB

CLASSIFICATION Restricted		CLIENT(S) Esso Norge a.s Att.: Geir Indrebø	
REPORT NO. 22.2043.00/21/95			
REG. NO. 95.xxx	DATE 9 February, 1996	PROJECT MANAGER Jorunn Nerbø Hokstad	SIGN. Jorunn Nerbø Hokstad
NO. OF PAGES 63	NO. OF APPENDICES	APPROVED Ivar Singsaas	SIGN. Ivar Singsaas

SUMMARY

The overall objective of Task C3 "Oil-surfactant interactions and mechanism studies" has been to achieve a more fundamental understanding of the natural processes taking place when oil is spilt at sea and also of the mechanisms involved when an oil slick is treated with oil spill chemicals.

The part of the work reported here, Activity 2 "Leaching Studies" and Activity 3 "Selectivity studies", has been concentrated on the chemical aspects of oil-surfactant interactions and the dispersion process.

Leaching of surfactants from an oil film to sea water
The leaching of three different surfactants from oil to sea water was studied in a near static, closed system. The three surfactants represent three different classes often used in oil spill dispersants: I) Sulpho succinates, II) poly-ethoxylated sorbitan esters and III) non-ethoxylated sorbitan esters. Standard methods found in the literature have been applied for the analysis of I and II. A method which can be used to analyse surfactants of type III in model systems has been developed.

The results indicated that non-ionic sorbitan ester surfactants (Spans) have a very low tendency to leach from oil to water. Both the anionic surfactant dioctyl sulphosuccinate, AOT, and the non-ionic, polyethoxylated sorbitane ester surfactant Tween 80 showed a high tendency to leach from oil to water when they were the only surfactant present in the oil. This was, however, drastically reduced when they were in mixtures containing all the three surfactants Span, Tween and AOT. Such a mixture of different surfactant types is most often the case of real dispersants.

Chemical composition of dispersed oil
The objective of Activity 3 was to investigate whether the dispersion process is selective with respect to certain ranges of n-alkanes in the oil corresponding to the alkyl chain of the oleophilic part of the surfactants.

Evaluation of the results indicated that there were a depletion of the n-alkanes from nC-16 and above in the dispersion compared to the original oil. This is probably due to wax chrystals / particles being more difficultly dispersed than the bulk oil. It could not be found any support for the theory of selective dispersion of n-alkanes.

KEYWORDS ENGLISH		KEYWORDS NORWEGIAN
Leaching	Span	
Surfactants	Tween	
Oil Spill Chemicals	Sulpho succinate	
Analysis		
Dispersed oil		

10.6 Ref # 270

REPORT

IKU
SINTEF GROUP

IKU Petroleumsforskning a.s
IKU Petroleum Research

N-7034 Trondheim, Norway
Phone: +47 7 59 11 00
Fax: +47 7 59 11 02 (aut.)
Telex: 55 434 iku n

TITLE

Screening testing of surfactants for use in oil spill chemicals.

DIWO Report no. 20
DRAFT REPORT

AUTHOR(S)

O.Ø. Knudsen, P.J. Brandvik, M.Ø. Moldestad, K. Aareskjold

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REG. NO. 93.082	DATE 25-MAY-93	PROJECT MANAGER Ole Øystein Knudsen	SIGN. Olef Knudsen
NO. OF PAGES 24	NO. OF APPENDICES -	SCIENTIFIC CONTROL Ivar Singsaas	SIGN.

SUMMARY

This report presents a screening testing of surfactants for future use in oil spill chemicals, performed in activity 2, task 1 in the DIWO-2 project. The objective of this work has been to test a large number of surfactants for their ability to lower the interfacial tension between oil and water, and their performance as wetting agents. The spinning drop test was used for measuring of interfacial tension and draves test was used for measuring of wetting properties.

Draves test: 63 surfactants from different surfactants classes were tested in distilled and brackish water (0 and 1.75% salinity). 10 surfactants gave wetting times less than 20 seconds at both salinities, which is comparable to AOT, a surfactant commonly used in oil spill chemicals. Two of these are recommended to be tested as emulsion breakers and in dispersants.

Spinning Drop: Several problems with the apparatus made the test time consuming and expensive, and therefor not as many surfactants as initially planned could be tested. The lowest interfacial tension was achieved with AOT and Tween 85, which are commonly used in dispersants. For surfactants tested in dispersants earlier, there were consistency between the results in this test and their performance in dispersants. Therefor it is recommended to work with the apparatus problems so that the test can be used in future projects.

KEYWORDS ENGLISH	KEYWORDS NORWEGIAN
Oil spill chemicals	Oljevernkjemikalier
Screening	Screening
Surfactants	Surfactanter

10.7 Ref # 276

<table>
<tr><td colspan="2" rowspan="3">SINTEF

SINTEF Applied Chemistry

Address: N-7034 Trondheim, NORWAY
Location: S.P. Andersens vei 15 b
Telephone: +47 73 59 28 73
Fax: +47 73 59 70 51

Enterprise No.: NO 948 007 029 MVA</td><td colspan="2">SINTEF REPORT

TITLE

Leaching of surfactants used in oil spill chemicals from the oil to the water phase

DIWO Report no. 26</td></tr>
<tr><td colspan="2">AUTHOR(S)

Ole Øystein Knudsen, Jorun Nerbø Hokstad and Per Johan Brandvik</td></tr>
<tr><td colspan="2">CLIENT(S)

Fina Exploration Norway Inc.</td></tr>
<tr><td>REPORT NO.

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Gisle Nødtvedt</td></tr>
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Per Johan Brandvik</td><td>CHECKED BY (NAME, SIGN.)

Ivar Singsaas</td></tr>
<tr><td>FILE CODE</td><td>DATE

17.10.05</td><td colspan="2">APPROVED BY (NAME, POSITION, SIGN.)

Tore Aunaas, Research Manager</td></tr>
</table>

ABSTRACT

The work presented in this report is a part of the DIWO-2 project (Task 1, activity 4, leaching of surfactants). The purpose of this activity has been to study the leaching of potential surfactants for the dispersant development in task 2. Leaching in this context is loss of surfactants in oil spill chemicals from the oil to the water phase. Leaching has been studied by quantifying the amount of surfactants in the water as a function of time.

The anionic surfactants tested showed an extensive leaching into the water. The leaching was faster in low salinity water and was slightly reduced by using Ca^{2+} and Mg^{2+} counter ions instead of Na^{+}. The nonionic surfactant tested in this study did not seem to leach into the water phase to a significant extent. However, significant leaching of nonionic surfactants have been found in later work at SINTEF (Hokstad et al., 1996).

Ca^{2+} dodecyl benzenesulphonate had the least salinity dependant leaching rates of the anionic surfactants. It is therefore expected that the use of this surfactant will give less salinity dependant dispersants than the other anionic surfactants tested. This indicate that calsium salts of ionic surfactants should be further used for dispersant optimisation.

Due to the leaching of anionic surfactants, they are not recommended to be used as emulsion breakers or emulsion inhibitors directly on the sea, unless they are used in a mixture with other surfactants that will stay in the oil and prevent re-emulsification of the oil.

KEYWORDS	ENGLISH	NORWEGIAN
GROUP 1		
GROUP 2		
SELECTED BY AUTHOR	Leaching, Surfactants	Lekkasje, Surfaktanter
	Oil Spill Chemicals	Oljevernkjemikalier

10.8 Ref # 278

SINTEF	SINTEF REPORT
SINTEF Applied Chemistry Address: N-7034 Trondheim, NORWAY Location: S.P. Andersens vei 15 b Telephone: +47 73 59 28 73 Fax: +47 73 59 70 51 Enterprise No.: NO 948 007 029 MVA	TITLE **Optimisation of oil spill dispersant for Arctic conditions - low temperature and varying salinity.** **DIWO Report no. 29.** AUTHOR(S) Per Johan Brandvik and Ole Øystein Knudsen CLIENT(S) Fina Exploration Norway Inc.

REPORT NO.	CLASSIFICATION	CLIENT'S REF.	
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FILE CODE	DATE	APPROVED BY (NAME. POSITION, SIGN.)	
	17.10.05	Tore Aunaas, Research Manager	

ABSTRACT

Earlier work in the DIWO programme have shown that the commercially dispersants available today have a effectiveness which is very dependant of water salinity. Specialised dispersants exists for both high (e.g. 3.5%) and low salinity (0.5%), but no single dispersant gives an acceptable effectiveness over a broad salinity range e.g. 0.5-3.5%.

The objective with this task of the DIWO-2 project has been to develop a dispersant for arctic conditions. Arctic conditions have been defined as 0°C water temperature and a salinity varying between 0.5 and 3.5%.

In an earlier study in the DIWO programme studying surfactant leaching from dispersants, it was concluded that the use of calcium salts of the anionic surfactants, instead of sodium salts, probably would reduce the salinity dependence of dispersants. Many different mixtures with calcium salts of dioctyl sulphosuccinate and dodecyl benzene sulphonate with various non-ionic surfactants were therefore tested for use in dispersants as a part of this study.

The effectiveness of the final optimised product (SINTEF-17) was similar to the effectiveness of a state-of-the art dispersant as Corexit 9500 at medium to high salinity (2 to 3.5% salinity) and better at low salinity (between 2 and 0.5%).

KEYWORDS	ENGLISH	NORWEGIAN
GROUP 1		
GROUP 2		
SELECTED BY AUTHOR	Arctic	Arktis, Dispergeringsmidler
	Oil Spill	Oljevern

10.9 Ref # 279

SINTEF REPORT

SINTEF Applied Chemistry

Address: N-7034 Trondheim, NORWAY
Location: S.P. Andersens vei 15 b
Telephone: +47 73 59 28 73
Fax: +47 73 59 70 51

Enterprise No.: NO 948 007 029 MVA

TITLE

Procedures for analysis of oil spill chemicals

DIWO Report no. 28.

AUTHOR(S)

Ole Øystein Knudsen, Jorun Nerbø Hokstad and Per Johan Brandvik

CLIENT(S)

Fina Exploration Norway Inc.

REPORT NO.	CLASSIFICATION	CLIENT'S REF.	
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FILE CODE	DATE	APPROVED BY (NAME. POSITION, SIGN.)	
	05.03.97	Tore Aunaas, Research Manager	

ABSTRACT

The objective with this activity in the DIWO-2 project has been to find analytical methods for quantifying and identifying surfactants in commercial oil spill chemicals and in water samples. The methods were used for analysing commercially available oil spill chemicals and to study surfactant leaching.

Different chromatographic techniques (HPLC, GC, Iatroscan TLC) have been tested, but finding a chromatographic method which could quantify the complex mixture of surfactants used in dispersants turned out to be very difficult. The main reasons for this are that dispersants consists of surfactants with very different chromatographic properties (ionic, lipophilic and hydrophilic components) and that they have weak UV/VIS absorbance which make them difficult to detect. Each type of surfactants e.g. a specific ethoxylated sorbitan was also present in the dispersant as many different modifications (ethoxylation degree, number of and types of fatty acids, homologues, isomers etc.).

The strategy was therefore changed, and different methods found in the literature for analysing each **group** of surfactants in the oil spill chemicals were established.

Anionic surfactants in water samples and in neat oil spill chemicals was quantified by forming a complex with methylene blue which could be determined colorimetrically. Poly ethoxylated surfactants were quantified after derivatisation of the poly ethoxy chain to 1,2-dibromo ethane which was quantified by GC. Quantitative and qualitative analysis of sorbitan esters were done by hydrolysing the ester, methylation of the fatty acids and identification/quantification of the fatty acid by GC analysis. Surfactants in water samples were concentrated by solid phase extraction on C_{18} Maxi-Clean cartridges.

The composition of 9 commercial dispersants were analysed.

KEYWORDS	ENGLISH	NORWEGIAN
GROUP 1	Chemistry	Kjemi
GROUP 2	Contingency	Beredskap
SELECTED BY AUTHOR	Analysis, Oil Spill Chemicals	Analyse, Oljevernkjemikalier
	Surfactants, Solvent	Surfaktanter, Løsningsmiddel

10.10 Ref # 346

IKU SINTEF GROUP

IKU Petroleumsforskning a.s
IKU Petroleum Research

N-7034 Trondheim, Norway
Telephone: +47 73 59 11 00
Fax: +47 73 59 11 02 (aut.)
Telex: 55 434 iku n

Enterprise no.:
NO 936 882 331 MVA

IKU REPORT

TITLE

Optimisation of dispersant formulations.

ESCOST report no. 23.

AUTHOR(S)

Alun Lewis

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NO. OF PAGES 30	NO. OF APPENDICES	APPROVED Ivar Singsaas	SIGN. Ivar Singsaas

SUMMARY

The results obtained in Tasks C4 and C6 of the ESCOST project indicated that there was further scope for the development of oil spill dispersants with improved performance. Corexit 9554 had been developed, but failed to conform to the maximum low temperature viscosity requirement of the UK specification. The first part of this project was to evaluate alternative solvents and surfactant blend concentrations for effectiveness and physical properties.

The work confirmed that Corexit 9500 has significant performance advantages over previous commercial dispersants, including Corexit 9527. The powerful emulsion breaking effect of Corexit 9500 was demonstrated in several meso-scale flume tests and confirmed by observations at the 1994 NOFO dispersant sea trial. Whilst Corexit 9500 is, in general an excellent dispersant, it is a poor performer in low salinity water and will not disperse some oils effectively in the very low salinity conditions that will prevail in estuarine areas or in the upper layers of the Arctic oceans during ice-melt.

Exxon research and Engineering supplied a series of experimental formulations which were comprehensively tested. Formulations were identified that are highly effective against many oils types and w/o emulsions in both high salinity and low salinity conditions. These have not been further tested or marketed by Exxon for commercial reasons.

KEYWORDS ENGLISH	KEYWORDS NORWEGIAN

11 Conclusions

The literature review of earlier work at SINTEF and CEDRE give indications that dispersant effectiveness can be retained during prolonged contact time with e\water after dispersant application.

There is identified a need for more sophisticated analysis of surfactant content in the oil phase or in the water phase to quantify surfactant leaching.

However, no information has been found during this review that introduces the need for major changes in the established plan or design of the “Laboratory study of the effects of time on the effectiveness of dispersants (PERF project)”.

Appendix B Literature Review - *CEDRE* References

PERF PROJECT

LITTERATURE REVIEW: REFERENCES FROM *CEDRE*

CENTRE DE DOCUMENTATION

DE RECHERCHE ET D'EXPERIMENTATIONS

SUR LES POLLUTIONS ACCIDENTELLES DES EAUX

PERF PROJECT

LITTERATURE REVIEW: REFERENCES FROM *CEDRE*

P.05.XX JG October 2005

LITTERATURE REVIEW

1. BIBLIOGRAPHICAL REFERENCES

[1] **Guyomarch J., E. Mamaca, M. Champs and F-X. Merlin, 2002**. "Oil Weathering and Dispersibility Studies: Laboratory, Flume, Mesocosms and Field Experiments", in *Proceedings of the 3rd IMO R&D Forum*.

[2] **Guyomarch J., E. Morin, A. Goutard and F-X. Merlin, 2001**. "Experimental Oil Weathering Studies in Hydraulic Canal and Open Pool to Predict Oils Behaviour in Case of Casual Spillage", in *Proceedings of the 2001 International Oil Spill Conference*, American Petroleum Institute, Washington, D.C.

[3] **Guyomarch J. and F-X. Merlin, 2000.** "Methodology for Assessing Oil Weathering in a dedicated Hydraulic Canal: Evolution of the Physical-Chemical Properties and Dispersibility of various Crudes", in *Proceedings of the 23rd Arctic and Marine Oilspill Program (AMOP) Technical Seminar*, 2000, Environment Canada, Ottawa, Ontario.

[4] **Guyomarch J., O. Kerfourn and F-X. Merlin, 1999.** "Dispersants and Demulsifier: Studies in the Laboratory, Harbor and Polludrome", in *Proceedings of the 1999 International Oil Spill Conference,* American Petroleum Institute, Washington, D.C., pp. 195-202.

[5] **Guyomarch J., F-X. and S. Colin, 1999.** "Study of the feasability of chemical dispersion of viscous oils and water in oil emulsions", in *Proceedings of the 22nd Arctic and Marine Oillspill Program (AMOP) Technical Seminar,* June 2-4, 1999, Calgary, Alberta, Canada.

[6] **Bocard B., G. Castaing, J. Ducreux, C. Gatellier, J. Croquette and F-X. Merlin, 1987.** "PROTECMAR: The French Experience from a Seven-Year Dispersant Offshore Trials Program", in *Proceedings of the 1987 International Oil Spill Conference,* American Petroleum Institute, Washington, D.C., pp. 225-229.

1. *CEDRE*'S REPORTS

[7] **Francois Xavier Merlin, 2005.** "Expérimentation DEPOL 04 – Etude de la Dispersion et du Comportement d'Hydrocarbures en mer et Intercalibration des Moyens de Télédétection – Synthèse Préliminaire", *Cedre*'s report n° R.05.05 C/3214, 12 p, Février 2005.

Translated Title: *"DEPOL 04 Experiment – Study of the Hydrocarbons Dispersion and Behavior at Sea & Intercalibration of Remote Sensing Means – Preliminary Synthesis"*

[8] **Julien Guyomarch, 2002.** "Etude du Comportement des Produits Pétroliers Bruts et Raffinés Déversés en Milieu Marin – Synthèse des Essais menés sur la Période 1999-2003", *Cedre*'s report n° R.04.16 C/3013, 12 p, Mars 2004.

Translated Title: *" Study of the Crude Oils and Refined Products Behaviour at Sea – Synthesis of Experiments Conducted During the Period 1999-2003"*

[1] **Guyomarch J., E. Mamaca, M. Champs and F-X. Merlin, 2002**. “Oil Weathering and Dispersibility Studies: Laboratory, Flume, Mesocosms and Field Experiments”, in *Proceedings of the 3rd IMO R&D Forum*.

ABSTRACT

In order to provide responders with more reliable predictions, *Cedre* has performed various weathering studies, at different scales, to assess the oils evolution in real conditions. In this view, a specific methodology was developed in an hydraulic canal (flume test, the Polludrome), which allows to simulate open sea conditions realistically. Data obtained were completed by additional weathering studies, in floating mesocosms set in a harbour water body, and by field trials or real cases. All these elements enabled to calibrate the flume test experiments and thus, providing a realistic view of the potential evolutions at sea.

In addition to these studies, investigations have been undertaken to review the possibilities of chemically dispersing high viscosity oils and water-in-oil emulsions with modern dispersants. This was conducted in two steps, in the laboratory using standard dispersibility tests, the WSL (Warren Spring Laboratory) method and the IFP (Institut Français du Pétrole) dilution method, which are both used to assess the efficacy of dispersants, and at a larger scale, in the Polludrome, in floating mesocosms and during field trials. The laboratory methods produced high efficiency results for high viscosity oils, peculiarly the WSL method, but, for emulsified oils, the efficiency was much lower than in the flume, which simulates somewhat more realistic sea conditions.

Moreover, laboratory tests, especially the IFP test, seems not to be suitable for testing oils with viscosities over 15,000 cSt and unrealistic results have been observed. Laboratory tests, which were originally developed for dispersant approval purposes, are not very reliable methods for studying the dispersion of high viscosity and density oils.

Finally, flume tests (Polludrome) and the field trial showed that the oil viscosity limit for dispersion, which had been defined in the eighties in France and set at 2000 cSt, can be upgraded to reflect the improvements made in the formulations of the modern dispersants.

Relevance for the study

This paper consist in a review of the tests performed at Cedre over a period of 5 years. Particularly, dispersibility tests conducted according to various laboratory protocols were compared to experimental results obtained from pilot scale experiments. This study shows the interest of the IFP protocol as it is quite close to more realistic conditions such as the ones recreated in the flume test, but also when comparing with field trials. Moreover, it shows the limitations of the WSL test when dealing with emulsions as the dispersibility is strongly reduced as far as the oil incorporates significant amount of water, the threshold value being close to 20%.

[2] **Guyomarch J., E. Morin, A. Goutard and F-X. Merlin, 2001**. “Experimental Oil Weathering Studies in Hydraulic Canal and Open Pool to Predict Oils Behaviour in Case of Casual Spillage”, in *Proceedings of the 2001 International Oil Spill Conference*, American Petroleum Institute, Washington, D.C.

ABSTRACT

The objective of this study was to get experimental data on the behavior of crude oils from different oil fields. The various weathering processes were simulated realistically in *Cedre*’s hydraulic canal, in which different marine water conditions can be re-created: wind, waves and UV light. All the experiments were carried out with the same agitation level and at two temperatures, 10 and 20°C. Six different oils were tested and the different parameters measured or assessed were : density, viscosity, water content and kinetics of emulsification, chemical composition and kinetics of evaporation, flash point, emulsion stability, oil adhesion and chemical dispersibility. The evolutions proved to vary considerably according to the nature of the oil, the temperature and the photo-oxidation process.

The weathering of one crude was also assessed outside in a large pool to provide a calibration of the evaporation and emuslification kinetics in realistic conditions compared to the flume test. The canal speeds up these processes by a factor between 4 and 6.

<u>Relevance for the study</u>

This paper describes the evolution of physical-chemical parameters of different crude oils for two pilot scale experiments. It shows that the weathering varies considerably according to the oil nature, but also compares flume test kinetics to open sea conditions. This calibrations allows to predict oil behavior for various environmental conditions.

[3] **Guyomarch J. and F-X. Merlin, 2000.** "Methodology for Assessing Oil Weathering in a dedicated Hydraulic Canal: Evolution of the Physical-Chemical Properties and Dispersibility of various Crudes", in *Proceedings of the 23rd Arctic and Marine Oilspill Program (AMOP) Technical Seminar*, 2000, Environment Canada, Ottawa, Ontario.

ABSTRACT

When spilled at sea, crude oil is subjected to weathering processes such as evaporation, emulsification, dispersion, and photo-oxidation. These processes occur under natural conditions due to sea surface agitation by wind, waves, and currents and exposure of the oil to solar light. The chemical composition and physical properties of the oil are constantly changing according to its weathering stage. Understanding these changes is a key element in evaluating the potential impacts, optimizing the response options, and implementing the emergency response plan to an oil spill.

The objective of this study was to obtain experimental data on the behaviour of crude oils from different oil fields. The various weathering processes were realistically simulated in Cedre's hydraulic canal, in which different marine water conditions can be recreated: wind, waves, and UV light. All the experiments were carried out with a similar agitation level and at two temperatures. The parameters measured or assessed were: density, viscosity, water content and kinetics of emulsification, chemical composition and kinetics of evaporation, flash point, emulsion stability, oil adhesion, and chemical dispersibility.

Relevance for the study

During the flume test experiments, several parameters were monitored for all the collected samples. In particular, the oil dispersibility was assessed according to the WSL test. The standard procedure was slightly modified by introducing 3 mixing times (1, 5 and 15 minutes) instead of the regular 2 minutes duration. Results of these experiments proved to be very low in comparison with previous tests performed on non-emulsified oils for similar viscosity [4]. Moreover, the effect of a prolonged time of energy proved to be low: the mixing energy being initially high, this protocol is not very adapted when considering different levels of energy as planned in the study.

[4] **Guyomarch J., O. Kerfourn and F-X. Merlin, 1999.** “Dispersants and Demulsifier: Studies in the Laboratory, Harbor and Polludrome”, in *Proceedings of the 1999 International Oil Spill Conference*, American Petroleum Institute, Washington, D.C., pp. 195-202.

ABSTRACT

When spilled at sea, many oils are known to form emulsions. These emulsions are often of high water content and viscosity, poorly dispersible, hard to recover and to pump, and are likely to remain as a persistent pollutant that may come ashore. To avoid these difficulties, demulsifiers have been used, either to inhibit emulsion formation or to break emulsions that have already been created. *Cedre* has studied the efficiency of several demulsifiers on the rate of emulsion formation and on the dispersibility of emulsified oils of different types. This study was conducted in three stages:

- Firstly; a study of the rate and extent of emulsification was conducted in the laboratory.

- Secondly, the effect of demulsifiers was studied in floating mesocosms placed in a harbor. The demulsifiers did not succeed in totally preventing emulsion formation, but they inhibited the degree of emulsification of the oils for some time.

- Thirdly, the dispersibility of weathered oils was studied in laboratory using the IFP and WSL test methods and then in the Polludrome where the effects of different treatment strategies combining demulsifiers and dispersants applications were assessed.

<u>Relevance for the study</u>

This paper describes several investigations carried out by using WSL tests. It shows that this test method can be adapted for making differences between products only when considering high viscosities, from 10 000 to 20 000 cSt. In the field of interest of our study, this method do not seem to be adapted as oils are less viscous. Moreover, the WSL method is a high energy test, which is not realistic in the light of the spill scenario of the study: the dispersant is applied in lack of energy and the dispersion is achieved later, generally in presence of a low level of agitation.

Moreover, some tests were performed in the flume test in order to assess the possibility of using dispersants for viscous emulsions (close to 17 000 cSt). The dispersion was successfully achieved by adopting a double treatment strategies. However, it must be noticed that the dispersant proved to be as efficient as the demulsifier for the first application. This result can be applied to our study: the application of dispersant in low energy conditions is liable to prevent a rapid emulsification of the oil thereafter (considering that a low concentration of the product is not sufficient to disperse the oil but can prevent the formation of emulsions).

[5] **Guyomarch J., F-X. and S. Colin, 1999.** "Study of the feasability of chemical dispersion of viscous oils and water in oil emulsions", in *Proceedings of the 22nd Arctic and Marine Oillspill Program (AMOP) Technical Seminar,* June 2-4, 1999, Calgary, Alberta, Canada.

ABSTRACT

Investigations have been undertaken to review the possibilities of chemically dispersing high viscosity oils and water-in-oil emulsions with recently developed modern dispersants. This study was conducted in two steps, in the laboratory using standard dispersibility tests, the WSL (Warren Spring Laboratory) method and the IFP (Institut Français du Pétrole) dilution method, which are both used to assess the efficacy of dispersants, and at a larger scale, in the Polludrome. The laboratory methods produced high efficiency results for high viscosity oils; the WSL method produced of up to 50% efficiency for oils with viscosities of up to 10,000 to 20,000 cSt, depending on the dispersant used. However, for emulsified oils, the efficiency was much lower, <15% for similar viscosities. In the Polludrome, which simulates somewhat more realistic sea conditions, the dispersion efficiency measured with viscous oils was lower, which suggested the laboratory tests over-estimate dispersion. In tests in the Polludrome, it was necessary to adopt special treatment strategies such as double dispersant applications to get significant dispersion of emulsions. Such strategies are very difficult to reproduce realistically in the laboratory tests. Moreover, laboratory tests, especially the IFP test, seem not to be suitable for testing oils with viscosities over 15,000 cSt and unrealistic results have been observed. Laboratory tests, which were originally developed for dispersant approval purposes, are not very reliable methods for studying the dispersion of high viscosity oils. This can be overcome by using larger testing facilities, such as the Polludrome, in which the environmental conditions can be more realistically simulated. Tests with the Polludrome showed that the oil viscosity limits for dispersion, which had been defined in the eighties, can be upgraded to reflect the improvements made in the formulations of the modern dispersants. For dispersing emulsions, multiple application strategies can be carried out, possibly using demulsifiers and dispersants. However, care should be taken to choose the products as the study proved that, under some conditions, not all products are compatible.

Relevance for the study

This paper describes for several test protocols the evolution of dispersant effectiveness versus viscosity. In addition to the standard procedures using either the WSL or the IFP test, some experiments were conducted in the flume test. These pilot scale proved to be closer to the reality than the laboratory tests with a drop of the dispersant efficiency in a range of viscosity close to field experiments. Results of IFP tests were slightly lower than the flume but the drop of efficiency was obtained for similar viscosities. These observations justify the use of the IFP protocol for our study as the limitations in terms of viscosity mentioned in [4] are not reached.

[6] **Bocard B., G. Castaing, J. Ducreux, C. Gatellier, J. Croquette and F-X. Merlin, 1987.** “PROTECMAR: The French Experience from a Seven-Year Dispersant Offshore Trials Program”, in *Proceedings of the 1987 International Oil Spill Conference,* American Petroleum Institute, Washington, D.C., pp. 225-229.

ABSTRACT

Six campaigns of dispersant offshore trials were conducted from 1979 to 1985 off the French Mediterranean and Brittany coasts. Altogether, 30 slicks were treated with several dispersants applied from ships by different spraying systems, from helicopter e quipped with an underslung bucket, and from a Canadair CL 215 aircraft.

Despite the difficulty of getting a mass balance of dispersed oil on the basis of oil concentration measurements and remote sensing techniques, the trials resulted in identifying the different effects of dispersants (short term dispersion of oil, delayed dissemination) and the limiting parameters (minimum energy of sea surface, high dispersant/oil ratio needed, negative herding effect).

Different techniques were tested in order to optimize the application of dispersant in different situations: use of a variable flow-rate system to spray neat concentrates from ships, methods of operating ships and aircraft to reach a selective distribution of dispersant and get good coverage of slicks.

<u>Relevance for the study</u>

This paper describes several field trials conducted between 1979 and 1985 in order to assess the operational efficiency of dispersant treatments. In particular, the last field trial was characterized by low energy conditions and observations made immediately after the treatment showed a significant dispersion for the oil treated by the ship due to the agitation generated by repeated runs. However, observations made the day after revealed that much less oil was remaining as regards the slick treated by the helicopter, thus showing the possibility of a delayed effect.

[7] **Francois Xavier Merlin, 2005.** “Expérimentation DEPOL 04 – Etude de la Dispersion et du Comportement d’Hydrocarbures en mer et Intercalibration des Moyens de Télédétection – Synthèse Préliminaire”, *Cedre*’s report n° R.05.05 C/3214, 12 p, Février 2005.

Translated Title: *“DEPOL 04 Experiment – Study of the Hydrocarbons Dispersion and Behavior at Sea & Intercalibration of Remote Sensing Means – Preliminary Synthesis”*

ABSTRACT

A field experiment, named DEPOL 03 because initially planned in 2003 but postponed in 2004, aimed at combining, in the framework of the Nebajex European project, several studies in relationship with various topics such as oil weathering at sea, assessment of different strategies of dispersant treatments and conditions of application (from plane and from ships) and estimation of limitations of use in terms of viscosity. Finally, all these themes were completed by an intercalibration exercise associating various remote sensing techniques from various European countries.

The DEPOL 04 field trial was conducted in 2004 in order to achieve the objectives initially assigned to the DEPOL 03 experiment. Three slick of around 10 m3 were formed. The first day, a paraffinic oil was poured onto the water surface, regularly sampled for characterisation purposes, and thereafter successfully treated with dispersant by using the POD system.

The second day, an asphaltenic mixture was used but the lack of mixing energy due to a very calm weather did not allowed assessment of treatment strategies as no emulsions were formed. However, a comparison of treatments from boats and plane was conducted and the oil proved to be totally dispersed two days after (but not the following day).

<u>Relevance for the study</u>

The environmental conditions that characterized the second day of the experiment correspond to levels of energy similar to those concerned by the study. In lack of waves, the oil was dispersed with the propellers of the ships but proved to coalesced to form thin slicks. However, two days after, when the environmental conditions turned to more favorable ones, the oil disappeared, thus suggesting the possibility of a delayed effect of dispersants.

Concerning the behaviour and fate of paraffinic oils, the questions raised previously [7] were partially answered: the natural dispersion of the oil proved to be significant but not as marked as in the flume. Finally, the effect of chemical dispersants proved to be slightly less efficient as expected in terms of viscosity of the products, but not as inefficient as suggested by IFP tests.

[8] **Julien Guyomarch, 2002.** “Etude du Comportement des Produits Pétroliers Bruts et Raffinés Déversés en Milieu Marin – Synthèse des Essais menés sur la Période 1999-2003”, *Cedre*’s report n° R.04.16 C/3013, 12 p, Mars 2004.

Translated Title: *“ Study of the Crude Oils and Refined Products Behaviour at Sea – Synthesis of Experiments Conducted During the Period 1999-2003”*

ABSTRACT

The various experiments conducted at *Cedre* during the 1999-2003 period have demonstrated an acceleration of weathering process in the flume, especially when dealing with viscous products. For light crudes, the kinetics study have shown divergences according to the parameter considered.

This study presents a synthesis of the experiments conducted either in the flume or in the open pools. It appears that, for light paraffinic crudes, opposite behaviours were observed: emulsification in the open pools and natural dispersion in the flume, the two pilot scale device being respectively characterised by low and high mixing energies. Moreover, samples collected in the flume for dispersibility test (IFP protocol) proved to be poorly dispersible in spite of low viscosity. On the contrary, the asphaltenic products did not show a significant natural dispersion in the flume and were sensitive to chemical dispersants with limits of efficiencies in agreement with the literature. These points should be cleared at the occasion of the DEPOL 04 experiment planned in year 2004.

On the other hand, some relationships were established between the chemical composition and the evolution in the flume for different oils. Moreover, the flume test data could be extrapolated to different environmental conditions. This work, mainly performed on asphaltenic crudes, has to be completed on some aspects, especially concerning differences of maximum viscosities, the flume seeming to underestimate this parameter.

<u>Relevance for the study</u>

This report proposes a synthesis of the work performed at *Cedre* and raises questions concerning the differences observed between the different pilot scale device. Moreover, it shows that paraffinic light crudes are liable to be naturally dispersed or emulsified according to the level of energy applied. Finally, it shows that laboratory protocols such as the IFP test can underestimate the efficiency of dispersants on these products.

Appendix C Dispersant effectiveness and surfactant leaching data

This appendix contains the tabulated data used for the figures in the report.

Table C.0.1 Dispersant Screening data: IFP efficiency of model dispersant, Corexit 9500, Dasic NS, Superdispersant 25 and Finasol OSR 52 on the four study oils at 15°C.

Oil type:	Residue	Modell dispersant	Corexit 9500	Dasic NS	Super-dispersant 25	Finasol OSR 52
Troll B (naohthenic)	200°C+	99	75	72	74	74
Balder (apshalthenic)	200°C+	96	95	89	58	87
Oseberg (paraffinic)	200°C+	97	94	67	50	82
Ringhorne (waxy)	150°C+	58	63	56	31	42
Ringhorne (waxy)	200°C+	-	3	5	2	3

-*: not performed*

Table C.0.2 T3 Freezing experiments: IFP efficiency of model dispersant applied on top of oil and on water(then frozen) for Troll and Balder 200°C+

	Troll B 200°C		Balder 200°C	
Hours	Applied directly on ice	Applied on water then frozen	Applied directly on ice	Applied on water then frozen
48	21	56	16	41
336	34	24	73	7
1056	31	8	21	9

Table C.0.3 Individual surfactant and total dispersant concentration predicted by multivariate statistics from the MS data for the Troll 200°C+. Dispersant effectiveness (IFP%) is also included.

Troll 15C						
Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	4	
0,016	16,7	19,4	9,1	22,8	4,4	99
1	15,1	15,7	7,1	27,8	4,4	
24	10,2	15,9	4,1	28,8	4,0	98
168	5,1	12,5	5,7	24,7	2,0	99
336	5,6	9,4	6,6	23,3	2,1	92
Troll 0C						
Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	4	
0,016	17,9	17,5	8,3	26,1	4,5	98
1	14,6	19,2	6,1	20,2	3,6	97
168,0	14,6	18,0	6,8	20,3	3,6	92
336,0	11,1	17,4	8,5	13,1	2,0	86

Table C.0.4 Individual surfactant and total dispersant concentration predicted by multivariate statistics from the MS data for the Ringhorne 150°C+. Dispersant effectiveness (IFP%) is also included.

Ringhorne 25C						
Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	5	
0,016	14,0	8,8	5,0	31,5	5,3	90
1	14,6	9,9	6,3	28,4	4,7	97
24	13,1	6,0	7,8	31,9	5,0	76
168	10,4	7,6	7,0	25,4	3,9	37
336	0,0	18,1	7,0	2,1	0,6	24
Ringhorne 15C						
Time(h)	**Tw-80**	**Tw-85**	**Span-80**	**AOT**	**Disp. conc. (wgt. %)**	**Disp. effectiveness (IFP%)**
0	12,9	19,1	6,5	27,8	5	
0,016	16,0	11,2	6,1	30,4	5,4	58
6	9,3	7,5	5,0	17,1	2,8	56
24	14,6	10,2	5,4	32,4	5,5	24
72	15,1	6,2	7,5	25,4	4,2	13
168	14,5	3,7	7,3	28,3	4,5	-
336	12,5	3,0	7,7	16,1	2,7	-

Table C.0.5 Individual surfactant and total dispersant concentration predicted by multivariate statistics from the MS data for the Oseberg 200°C+. Dispersant effectiveness (IFP%) is also included.

Oseberg 25C						
Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	4	
0,016	14,0	18,4	10,6	25,7	4,3	100
1	11,2	13,8	11,7	21,6	3,4	100
168	6,0	8,5	4,9	10,5	1,4	84
336	8,9	10,5	4,9	9,7	1,7	83

Oseberg 15C						
Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	4	
0,016	13,7	20,3	8,8	22,0	4,1	97
1	12,0	14,8	6,2	23,6	3,6	
24	11,3	13,7	6,3	24,7	3,5	92
168	12,8	9,2	2,8	26,7	3,6	75
336	13,8	13,3	0,6	20,2	3,2	53

Oseberg 0C						
Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	4	
0,016	8,0	12,6	12,5	13,3	2,2	38
1	11,8	18,4	8,7	18,3	3,2	38
168	13,8	17,1	9,3	22,1	3,8	27
336	12,4	9,1	11,7	22,8	3,4	15

Table C.0.6 Individual surfactant and total dispersant concentration predicted by multivariate statistics from the MS data for the Balder 200°C+. Dispersant effectiveness (IFP%) is also included.

Balder 25C Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	5	
0,016	9,6	11,2	17,2	23,4	4,0	95
1	9,5	10,3	18,4	23,2	4,0	94
24	0,8	10,7	25,4	24,4	2,7	91
168	0,3	9,4	33,7	16,0	1,3	90
336	0,5	22,3	35,3	4,6	0,1	59

Balder 15C Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	5	
0,016	10,6	9,8	17,3	23,9	4,6	96
1	10,2	11,3	18,1	21,9	4,3	
24	10,2	7,2	20,6	23,2	3,9	97
168	5,6	8,8	24,4	23,0	2,0	83
336	5,2	11,4	27,2	17,0	0,8	76

Balder 0C Time(h)	Tw-80	Tw-85	Span-80	AOT	Disp. conc. (wgt. %)	Disp. effectiveness (IFP%)
0	12,9	19,1	6,5	27,8	5	
0,016	13,3	14,4	15,1	17,8	5,0	100
1	12,4	13,4	15,8	19,2	4,9	98
168	11,5	4,8	32,9	7,6	1,9	77
336	9,8	11,4	18,2	21,8	4,3	88

Appendix D Quantification of dispersant and individual surfactants

Residual total dispersant and individual surfactants in the oil phase were quantified using mass spectrometri (MS) and multivariate calibration. The preparation of the samples was performed at SINTEF, the MS analysis and data pre-treatment (selecting of masses and calculation of mean spectra) were done at Statoils research centre in Trondheim. The data were sent to SINTEF as Excel files and the final multivariate calibration and quantitative analysis were performed by SINTEF in close cooperation with Statoil.

Mass Spectrometri (MS)
Samples of oil (with or without surfactants) were dissolved in dichloromethane (DCM) in a concentration of 2 mg/mL. The samples were analyzed by positive electrospray mass spectrometry (ESI-MS) using a single quadrupole LC-MS instrument with direct injection (no chromatographic separation of oils or surfactants) and without fragmentation of the molecules. Each sample was analyzed 5 or 6 times. The instrument was operated in the mass number (m/z) range from 65 to 1400. The methodology and its application on surfactants are described in more detail elsewhere (Eide et al. 2006).

With direct injection, each analysis takes one min. One spectrum was obtained from each individual analysis. The acquisition of spectra was performed by a post-run macro to ensure identical data collection between different injections. Table construction was performed by a specially designed macro in Microsoft Access. Further pre-treatment was performed in Microsoft Excel. The final Excel worksheets with non-normalized mean spectral values were submitted to SINTEF for multivariate calibration and quantification.

Multivariate calibration
Both the total dispersant leaching and the leaching between individual surfactants were quantified by this approach using commercial software for multivariate data analysis, Unscrambler© ver. 9.2. The MS dataset consisting of averaged spectra (5 or 6 replicates) were normalised (divided on average) to remove variation caused by e.g. sample concentrations, injected volumes, detector sensitivity etc. Both the analysis of dispersant leaching (relative to oil) and individual surfactant leaching (relative to oil) were more efficient using normalised data, avoiding variation from the quantitative/absolute data. The MS dataset were also weighted (1/SD) to give all the variables (masses) equal (unit) variance (Var=1). The advantage with this pre-treatment is that all masses will be given equal importance in the multivariate analysis independent of their numerical scale (large or small). The focus is on their relevant systematic variation (information) which is extracted by the multivariate calibration.

The dispersant/surfactant dataset are mixture data and already normalised (gives a total of surfactant concentration of 60%). Scaling was not used on these data since the dispersant/surfactant variables are within the same numerical range (0-30) and have similar variances, since the calibration set follows a statistical design (d-optimal). The d-optimal design is listed in Table D.0.1. More details can be found in Eide et al. 2006 (quantification of surfactans) and Brandvik and Daling, 1998 (PLS2 and designed experiments).

Determination of dispersant leaching
The calibration was performed using a calibration set where the dispersant concentration was varied from 0 to 6%. A multivariate model was established based on MS data for each combination of oil type (Troll, Balder, Ringhorne, Oseberg) and type of experiment (open water and ice experiments). The multivariate models consisted of two principal components, explaining 85-95% of the X variance (masses) and 92-99% of the Y variance (amount of dispersant). All of these models had a very high ability to describe the relationship between the changes in the total surfactant pattern (dispersant) in the MS-spectra and the known concentration of dispersant in the oil. Correlation between predicted and measured values in the calibration set was for all models in the range of 0.95-0.99 (determined by cross validation). See example in Figure D.1.

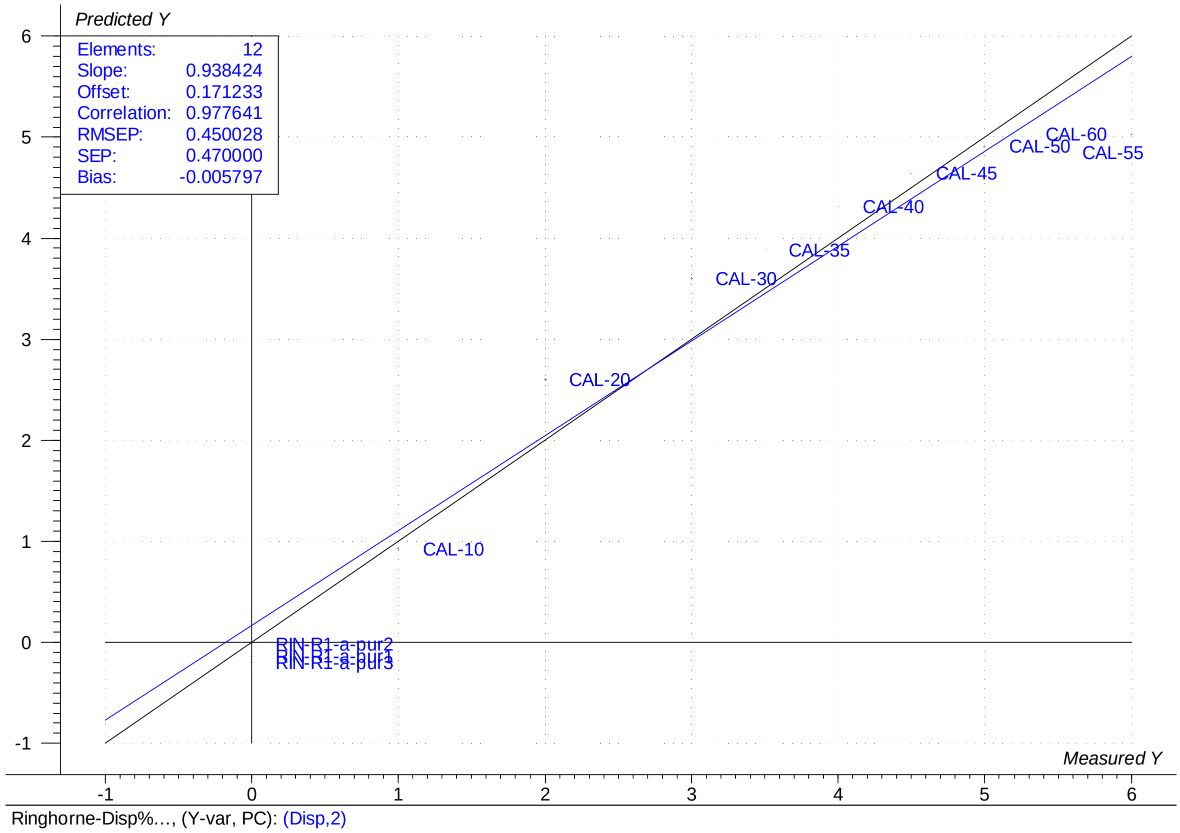

Figure D.1 Example of predicted Y versus measured Y (dispersant content) from the multivariate calibration of total dispersant leaching from the Ringhorne oil phase based on 12 samples in the calibration set. Correlations are 0.98.

Determination of individual surfactant leaching
This calibration was performed using a calibration set where the concentration of the four individual surfactants in the Model dispersant was varied following a d-optimal design, see *Table D.0.1*. A calibration set following the d-optimal design was prepared for all oil types in the original premix concentration of Model dispersant in oil (4 wt.%) and analysed together with the real samples. Three replicates of the relevant oil were included in each calibration set, as a zero reference.

Table D.0.1 D-optimal with 25 different dispersants prepared by varying the four individual surfactants in the Model dispersant.

Dispersant number in calibration set	Tween 80 (wt. %)	Tween 85 (wt. %)	Span 80 (wt. %)	AOT (wt. %)
1	17.6	26.4	0.0	21.1
2	12.8	15.5	6.8	30.0
3	12.9	18.4	10.2	23.4
4	10.5	14.3	10.1	30.1
5	17.7	26.6	6.8	13.8
6	17.8	26.7	10.2	10.1
7	3.5	26.6	10.2	24.7
8	17.7	12.6	5.0	30.0
9	17.5	17.9	0.0	29.9
10	3.4	21.2	10.2	30.1
11	13.0	26.7	10.2	14.9
12	3.3	26.5	5.1	30.1
13	8.8	26.4	0.0	30.0
14	9.9	23.5	1.7	30.0
15	10.2	26.5	5.1	23.2
16	17.7	19.6	5.0	22.8
17	7.3	23.1	7.9	26.6
18	8.1	26.6	10.3	19.8
19	17.6	22.2	0.0	25.5
20	13.2	26.4	0.0	25.6
21	14.4	20.1	2.8	27.9
22	14.5	24.4	5.5	20.7
23	17.6	7.6	10.1	30.0
24	17.7	20.2	10.3	16.8
25	17.7	13.8	9.9	23.5

This approach spans out the expected variation of all the four surfactants in the samples from the leaching experiments with a minimum number of samples (25+3). A multivariate model was established based on MS data for each combination of oil type (Troll, Balder, Ringhorne, Oseberg) and type of experiment (open water and ice experiments). The multivariate models consisted of four to five principal components, explaining 55-75% of the X variance (masses) and 75-95% of the Y variance (amount of the individual surfactants). The calibration was performed with all the four surfactants simultanously (PLS2). Calibration with single surfactants was also tested (PLS1), but did not in general give increased predictive capability. However, for a few of the Tween-85 and AOT predictions, single Y-variable (PLS1) was used since it gave slightly better predictions.

All of these models had a high ability to describe the relationship between the changes in the surfactant pattern (due to leaching of individual surfactants) in the MS-spectra and the known concentration of surfactants in the calibration set (25 + 3 surfactant mixtures). Correlation between predicted and measured values in the calibration set was for all models in the range of 0.65-0.98% (determined by cross validation). See example for Tween -80 in Figure D.2.

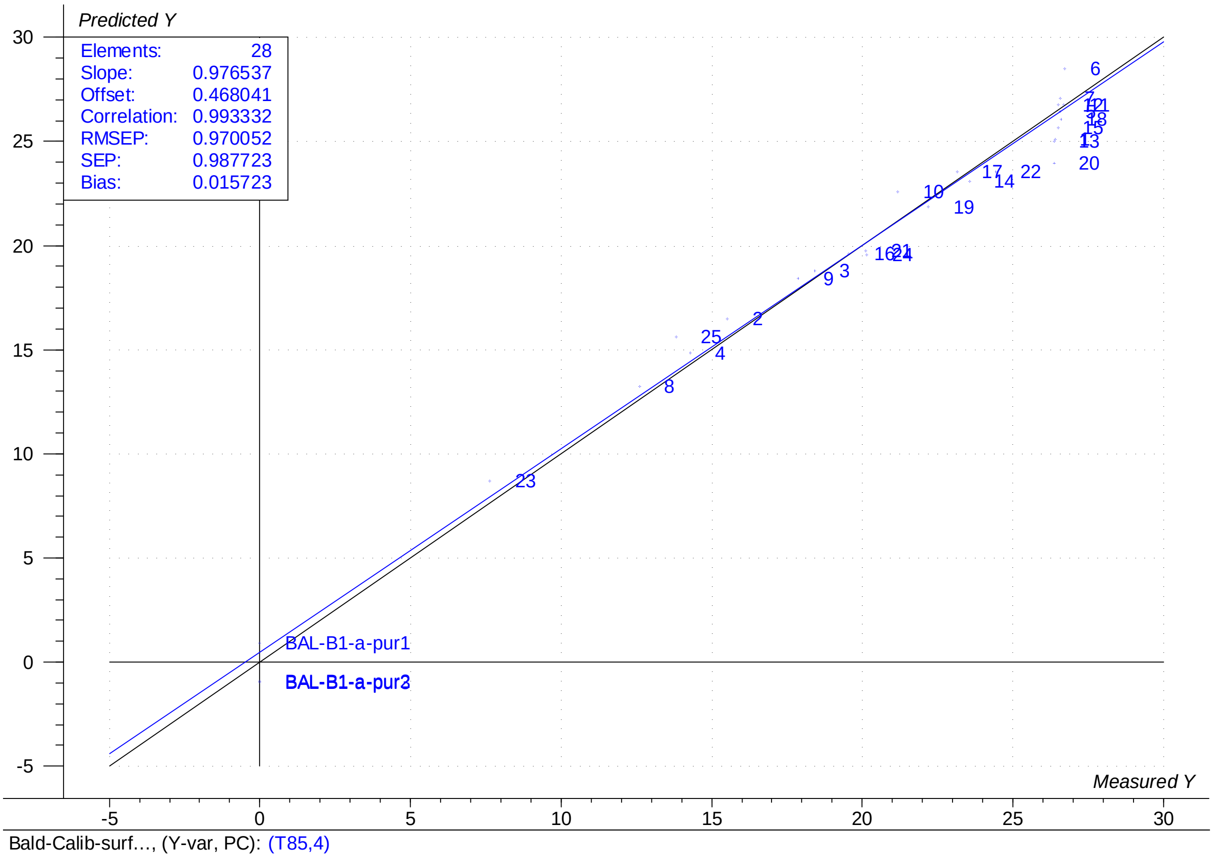

Figure D.2 Example of predicted versus measured Y (Tween-85) from the multivariate calibration of the individual surfactant leaching from the oil phase (Balder) based on the 25+3 samples in the calibration set. Correlation between predicted and measured is 0.99 using cross validation.