SINTEF

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Report

Verification study of weathering properties of Draugen crude oil

Re-check of oil properties related to oil spill response

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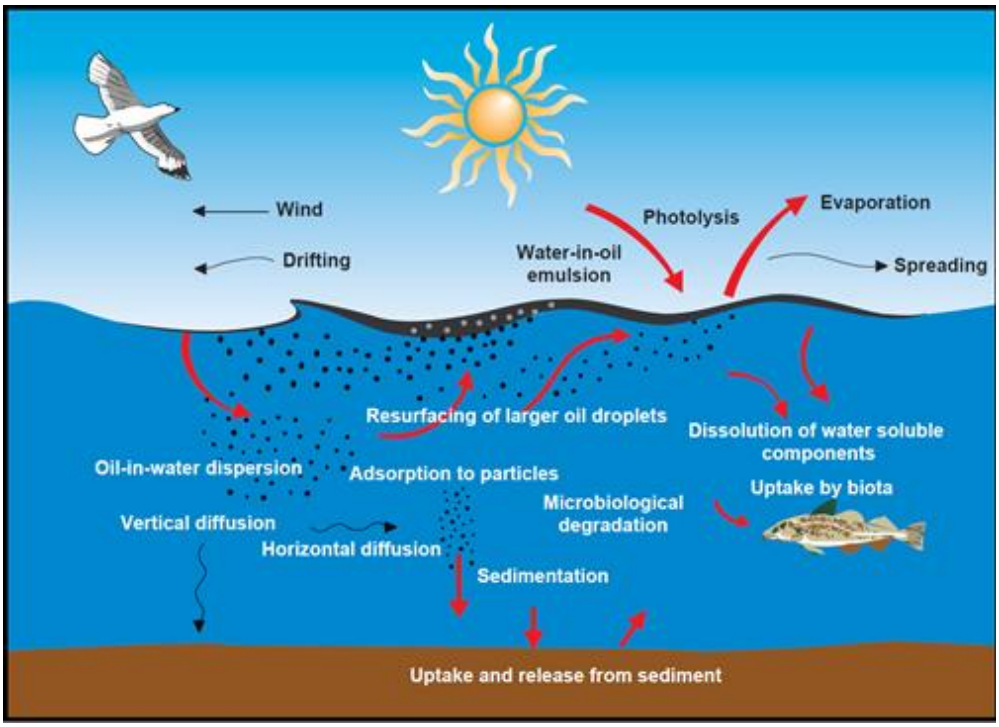

SINTEF Ocean AS
Risk and New Recourses
2018-06-08

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ABSTRACT

SINTEF has conducted a verification study (re-check) of Draugen crude oil collected in 2018 to determine if weathering data collected from Draugen crude oil collected in 2008 is still valid. Weathering properties of Draugen 2018 were obtained in the laboratory following standard methods and compared to the laboratory data of Draugen 2008. The only significant difference between Draugen 2018 and Draugen 2008 were the viscosities of evaporated 250°C+ residue, which decreased from 7190 and 1490 mPa.s in 2008 to 1658 and 645 mPa.s in 2018, for water-free and emulsified residues, respectively. The decrease in the water-free viscosity for Draugen 2018 could be explained by the variation of the topped 250°C+ residue between the oil samples from 2018 and 2008). All other Draugen 2018 analytical data were not significantly different from Draugen 2008.

The weathering data from this 2018 verification study was combined with the existing weathering data of Draugen 2008 as input to SINTEF's Oil Weathering Model (OWM) to run predictions. The time window for dispersant use increased with Draugen 2018 compared to Draugen 2008 based on the lower emulsion viscosities of Draugen 2018. At the same time, the slightly higher pour point in Draugen 2018 may cause the oil to solidify faster on the sea surface compared to Draugen 2008, which may also reduce the effectiveness of dispersant use. Solidification (high pour point) is likely to be the limiting factor for use of dispersant in an oil spill operation, particularly in winter conditions. Use of dispersants will likely be most efficient within 1-2 days after a spill on the sea surface, and may be even shorter at 5 °C. Moreover, the use of weir skimmers should be efficient on the Draugen 2018 emulsions after several days of weathering for viscosities < 15-20 000 mPa.s.

Based on a total evaluation from this study, the existing weathering data of Draugen 2008 can still justify being valid related to oil spill contingency analysis and environmental risk assessment.

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1 Introduction

A/S Shell Norge has requested SINTEF Ocean (dept. Risk and New Recourses) to conduct a verification study (re-check) of the weathering properties of Draugen 2018 and compare the exiting weathering properties with a weathering study of Draugen from 2008 (Leirvik, 2008). In addition, SINTEF also conducted a weathering study on Draugen back in 1994, as described in Almås et al. (1994).

This data report gives a recommendation if the exiting 2008 weathering data are still valid, or if an updated weathering study should be conducted on Draugen oil from 2018. Weathering data of Draugen are utilized in spill contingency planning and environmental risk assessment and are input into modelling tools such as SINTEF's Oil Weathering Model (OWM) and OSCAR oil spill trajectory model.

An image and description of the Draugen platform at the Draugen oil field in the Norwegian Sea (Haltenbanken) is given in Figure 1-1.

The Draugen oil field is operated by Shell in Norway. Other stakeholders are Petoro and VNG. The field lies in block 6407/9 in the Haltenbanken area (Norwegian Sea), which is situated about 150km from Kristiansund, Norway. The field lies in production licence PL093. The water depth is 250m.

Figure 1-1 Draugen oil platform ***https://www.shell.no/about-us/projects-and-sites/draugen.html***

2 Laboratory analysis and results

The analytical methods are described in Appendix A. Draugen 2008 and Draugen 2018 studies were both conducted at 13°C. The results from Draugen 2018 (this verification study) are compared to the Draugen 2008 study (Leirvik et al., 2008). See Table 2-1 for administrative details pertaining to Draugen 2008.

Table 2-1 Report details and scientific reference of the Draugen 2008 study

Oil	SINTEF Id	Report Number	Reference
Draugen 2008	2007-0426	SINTEF A5637	Leirvik, 2008

The verification study of Draugen 2018 included the following analysis parameters:

- Topping / distillation of the fresh oil into residues (200 and 250 °C+)
- Gas chromatographic analysis of hydrocarbon distribution
- Density and viscosity of fresh oil and evaporated water-free residues
- Pour point of fresh oil and residues
- Content by weight % of wax and asphaltenes
- Emulsification kinetics
- Emulsion maximum water uptake
- Emulsion viscosity
- Emulsion stability and addition of emulsion breaker

2.1 Pre-handling and homogenization

On March 8, 2018, SINTEF Ocean received five barrels (total of 60 litres) of Draugen oil (see Figure 2-1). Oil samples were obtained from each barrel and were registered in SINTEF's laboratory information management system (LIMS) and given SINTEF Id: 2018-2014. Upon arrival, oil samples were measured for water content and density. The oil samples had water contents lower than 2 vol. %, and additional laboratory HSE precautions for distillation (topping) were therefore not required. Samples from one of the barrels were temperated (50 °C) and homogenized prior to the physicochemical analysis according to SINTEF`s internal procedures for pre-handling of crude oils.

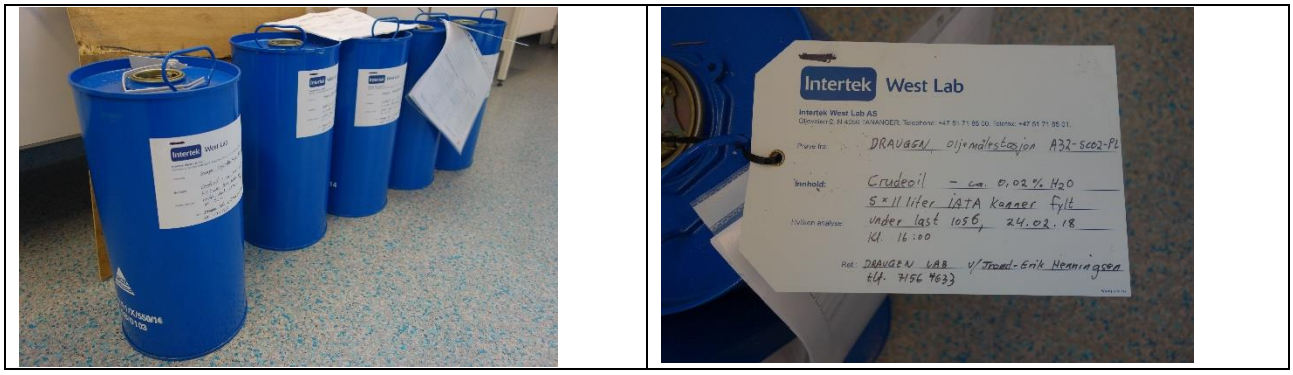


Figure 2-1 Barrels of Draugen crude oil (SINTEF Id: 2018-2014)

2.2 Chemical composition and physical properties

The chemical compositions of the hydrocarbon profiles of *n*-alkanes (nC_5-nC_{36}) for Draugen 2018 and Draugen 2008 are shown in Figure 2-2. The wax and asphaltene contents are given in Table 2-3, and the water uptake times for the evaporated residues of the crude oils are given Table 2-4.

Gas chromatographic flame ionization detector (GC-FID) characterization

The hydrocarbon profiles of Draugen 2018 and Draugen 2008 were analysed by use of Gas Chromatography (GC) coupled with Flame Ionization Detector (FID). Figure 2-2 illustrates the GC-FID output (i.e. gas chromatogram) of the fresh Draugen crude oils (2008 and 2018). Figure 2-3 shows the gas chromatograms of the corresponding evaporated residues topped at two different temperatures (200 and 250°C+). The loss of low molecular weight compounds (shown towards the left of the chromatogram) mimics the natural weathering and provides support for the artificial evaporation of the crude oil by use of distillation (topping) in the laboratory. Draugen 2008 was re-analysed on GC-FID using the same instrumental conditions as for Draugen 2018 for comparison. The inspection of the gas chromatogram of the 200°C+ residue (Figure 2-3) indicates that the sample from 2008 was slightly more topped (evaporated) compared with the 2018 sample.

The gas chromatograms show the *n*-alkanes as systematic narrow peaks. The first peaks in the chromatogram represent components with the lowest boiling points. Some of the more complex components, such as resins and naphthenes, are shown as a broad and poorly defined bump below the sharp peaks and are often described as the "Unresolved Complex Mixture" (UCM). Heavier compounds such as asphaltenes (> nC_{40}) are not possible to analyze with this technique. The chromatograms are normalized to *n*C25, an *n*-alkane that is not influenced by weathering.

The gas chromatograms of fresh Draugen 2018 and 2008 show very similar hydrocarbon distributions (Figure 2-2). These hydrocarbon distributions are typical for biodegraded (naphtenic) crude oils and include a minor content of the lightest *n*-alkanes. Both samples also show systematic paraffinic peaks for the higher molecular weight *n*-alkanes that reflect the wax content (> nC_{20}). Moreover, the gas chromatograms of the 200°C+ residue for Draugen 2018 and 2008 are also very similar.

GC-FID is also a tool in oil spill identification, where common screening parameters are the nC_{17}/pristane, nC_{18}/phytane, and pristane/phytane ratios. These ratios relate the more biodegradable *n*-alkanes to the more recalcitrant isoprenoids (pristane and phytane). Thus, the ratios of nC_{17}/pristane and nC_{18}/phytane are reduced as biodegradation proceeds. These ratios for Draugen 2018 and 2008 are shown in Table 2-2, and the ratios are comparable between the two oil samples.

Table 2-2: Typical ratios used for identifying oil types and biodegradation

Oil	nC_{17}/Pristane*	nC_{18}/Phytane*	Pristane/Phytane
Draugen 2018	1.07	1.85	2.09
Draugen 2008	1.04	1.81	2.06

**Ratios > 2 typical for high paraffinic oils, ratios < 1 typical for very biodegraded /naphthenic oil.*

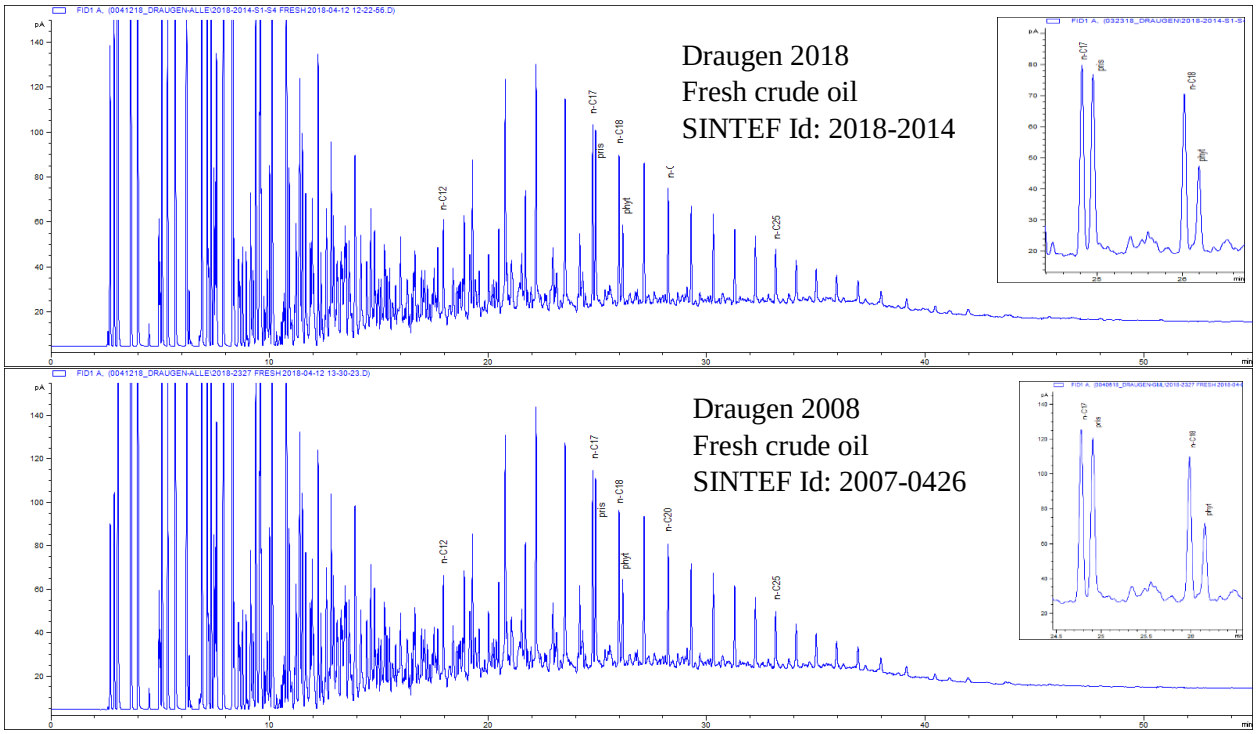
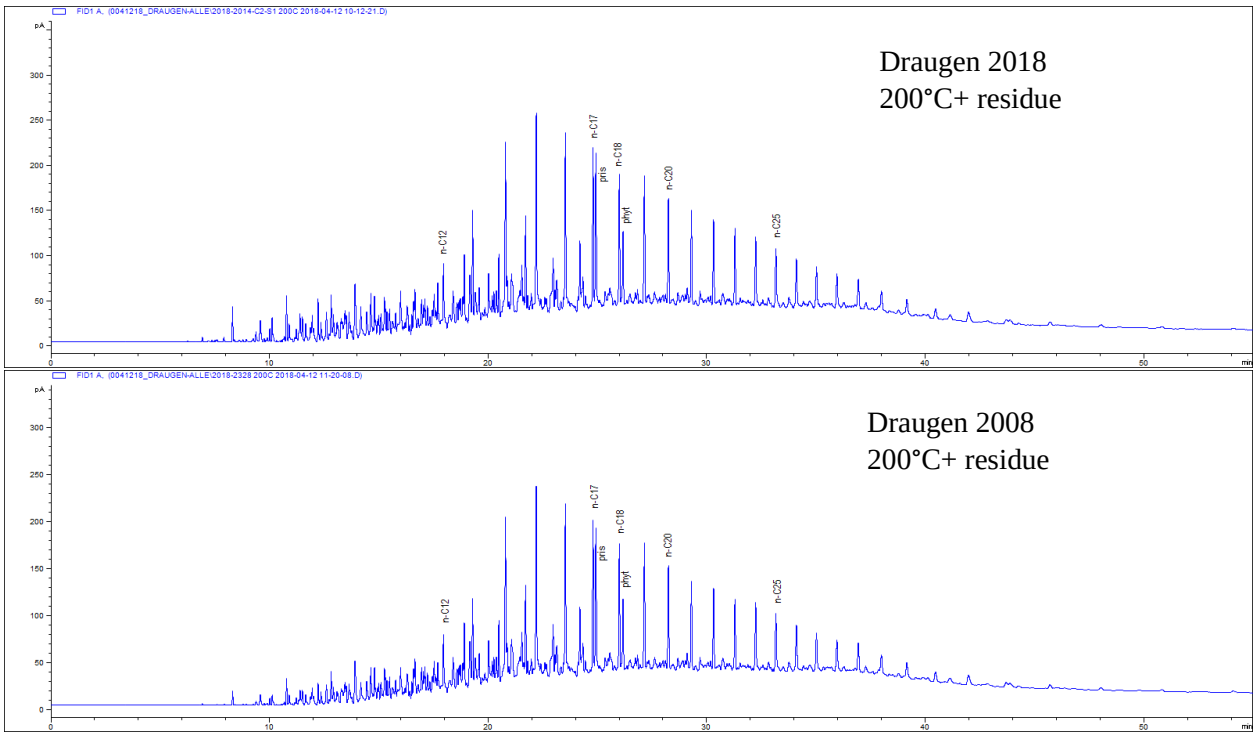


Figure 2-2 Gas chromatogram of n-alkanes (nC5-nC36) of fresh Draugen crude oil from 2018 and 2008. Draugen 2008 was re-analysed on GC-FID using the similar instrumental conditions as for Draugen 2018 for comparison



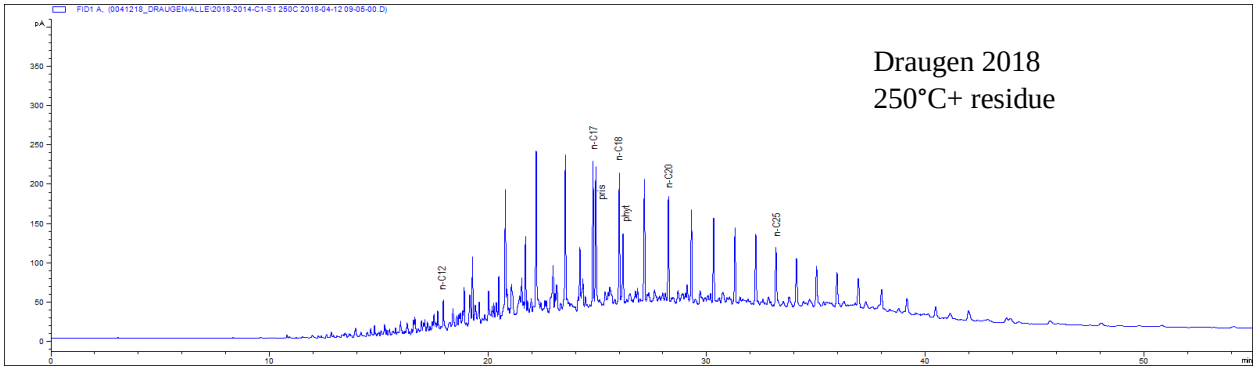


Figure 2-3 GC-FID chromatograms of evaporated residues of Draugen 2018 and Draugen 2008. The samples are analysed with similar instrumental conditions in 2018 for comparison

Physicochemical properties for the fresh and weathered residues

Analysis of relevant physicochemical parameters were performed for the fresh, 200°C+ and 250°C+ residue of Draugen 2018 and compared with the similar parameters from the previously weathering study of Draugen 2008 (see Table 2-3). Overall, the physiochemical properties for Draugen 2018 did not change significantly compared with Draugen 2008. The evaporation loss and density are in the same range for Draugen 2018 and Draugen 2008. The 250 °C+ residue of Draugen 2018 has approx. 4-5 vol% lower evaporated loss compared with Draugen 2008 but is not assumed as a significant difference. However, the viscosity of the 250°C+ residue of Draugen 2018 is lower than Draugen 2008 and could be explained by this small variation from the topping /distillation of the oil. Moreover, the higher wax content of the 250°C+ residue for Draugen 2018 reflects the higher pour point of the same residue.

Table 2-3 Physicochemical properties of Draugen 2018 and 2008

Parameters	**Draugen 2018**			**Draugen 2008**		
	Fresh	**200°C+**	**250°C+**	**Fresh**	**200°C+**	**250°C+**
Evaporated (vol.%)	0	39	48	0	41	53
Residue (wt.%)	100	66	57	100	64	52
Wax content (wt.%)	3.0	4.6	5.3	2.4	3.7	4.6
"Hard" asphaltene (wt.%)*	0.09	0.14	0.16	0.13	0.21	0.26
Density (g/mL)	0.822	0.887	0.897	0.823	0.890	0.904
Viscosity (13°C, 10 s^{-1})	6	188	645	6	284	1490
Pour point (°C)	-24	12	21	-24	12	18

**Precipitated in n-heptane*

2.3 Emulsifying properties

Water uptake (kinetics)

The rate of water uptake (kinetics) was studied by use of the rotating cylinders (Hokstad et al., 1993). The water content in the water-in-oil (w/o) emulsions as a function of time is tabulated in Table 2-4. The $t_{1/2}$ - value is defined as the time (hours) it takes to incorporate half of the maximum water uptake (vol. %) in 24 hours (rotating time). Draugen 2008 emulsified water slightly slower than Draugen 2018.

Table 2-4 Water uptake times for the evaporated residues of Draugen 2018 and Draugen 2008 at 13°C

Residue	Draugen 2018 t ½-value (hours)	Draugen 2008 t ½-value (hours)
200°C+	0.31	0.40
250°C+	0.12	0.16

Maximum water content, stability and efficiency of emulsion breaker

In mechanical recovery operations, separating oil from water enables optimal use of available storage (i.e. facilities/tankers) and the stability and efficiency of this separation can be enhanced by applying emulsion breakers. Stability testing of the emulsion and the efficiency of the emulsion breaker (Alcopol O 60%) are shown in Table 2-5 and Table 2-6.

Draugen 2018 and Draugen 2008 formed stable water-in-oil (w/o) emulsions for the 200 and 250°C+ residues with very similar stability ratios as shown in Table 2-5 and Table 2-6. The emulsion samples also showed similarities in the maximum water content. The effectiveness of the emulsion breaker on emulsion samples was not significantly different from 2018 and 2008, as the emulsion breaker partly broke both (2018 & 2008) emulsions with concentrations of 500 and 2000 ppm (wt.%).

Table 2-5 Stability of emulsion and efficiency of emulsion breaker at 13°C for Draugen 2018

Residue	Emulsion breaker	Draugen 2018 Water-in-oil emulsion (vol. %) at 13 °C		Stability ratio***
		Reference no settling*	24 hours settling**	
200°C+	none	89	87	0.81
250°C+	none	82	82	0.99
200°C+	Alc. O 60 % 500 ppm	89	35	0.07
250°C+	Alc. O 60 % 500 ppm	82	44	0.18
200°C+	Alc. O 60 % 2000 ppm	89	19	0.03
250°C+	Alc. O 60 % 2000 ppm	82	17	0.04

ppm is parts per million;
**: w/o emulsion after 24 hours rotation;*
***: w/o emulsion after 24 hours rotation and 24 hours settling*
****: Stability ratio =1 means stable emulsion; stability ratio=0 means totally broken emulsion. The stability ratio is based on the volumetric water to oil ratio in the emulsion before and after settling.*

Table 2-6 Stability of emulsion and efficiency of emulsion breaker at 13ºC for Draugen 2008

Residue	Emulsion breaker	Draugen 2008 Water-in-oil emulsion (vol. %) at 13 °C		Stability ratio**
		Reference (no settling) **	24 hours settling***	
200ºC+	none	90	90	0.88
250ºC+	none	78	78	0.98
200ºC+	Alc. O 60 % 500 ppm	90	33	-
250ºC+	Alc. O 60 % 500 ppm	78	33	-
200ºC+	Alc. O 60 % 2000 ppm	90	9	-
250ºC+	Alc. O 60 % 2000 ppm	78	12	-

-: No data available; ppm is parts per million
**: w/o emulsion after 24 hours rotation;*
***: w/o emulsion after 24 hours rotation and 24 hours settling*
****: Stability ratio =1 means stable emulsion; stability ratio=0 means totally broken emulsion. The stability ratio is based on the volumetric water to oil ratio in the emulsion before and after settling.*

Viscosities of water-free and emulsified residues

Table 2-7 shows the viscosities of water-free residues and the residues emulsified with maximum water content for Draugen 2018 and Draugen 2008. The viscosity for the 250°C+ residue with the maximum water content (79-81%) was significantly lower for Draugen 2018 compared to 2008 (Table 2-7). This is likely due to the difference in the viscosity of water-free residue (Table 2-3) as the maximum water content was very similar between Draugen 2018 and 2008.

Table 2-7: Viscosity of Draugen 2018 and 2008 for water-free and emulsified residues at 13ºC

Residue	Water content (vol.%) 13ºC	Draugen 2018 Viscosity 13°C $10s^{-1}$ (mPa.s)	Draugen 2008 Viscosity 13°C $10s^{-1}$ (mPa.s)
		10 s^{-1}	10 s^{-1}
Fresh	0	6	6
200°C+	0	188	284
200°C+	87	599	-
250°C+	0	645	1490
250°C+*	81/79	1658	7190

-: no data in 2008 due to supersaturated and unstable emulsion
**: Maximum water uptake for Draugen 2018 / Draugen 2008*

3 Oil weathering predictions of Draugen 2018 vs. Draugen 2008

Laboratory data generated from the verification study of Draugen 2018 were combined with some of the existing weathering data of Draugen 2008. The existing viscosities of the 50 and 75 vol. % emulsions from Draugen 2008 were not utilized since those data were not a part of the current verification study. The updated dataset was used as input for the SINTEF Oil Weathering Model (OWM) to compare relevant weathering properties from the existing 2008 predictions to the updated 2018 predictions. OWM predictions of water uptake, emulsion viscosity, pour point and mass balances are given in Appendix B.

4 Summary & recommendation

Evaluation of weathering data

The hydrocarbon profile of Draugen 2018 shown as GC-FID chromatograms is very similar to Draugen 2008. The chromatograms show that Draugen 2018 and Draugen 2008 are naphtenic crude oils that are highly biodegraded up to < nC_{15}-nC_{18}. Above nC_{18}-nC_{20} the gas chromatograms show a low degree of biodegradation of the *n*-alkanes, which also reflects the wax content of both oils.

The density of Draugen 2018 (0.822 g/mL) was similar to Draugen 2008 (0.823 g/mL). The evaporative loss was about 5 vol.% lower for the 250°C+ residue of Draugen 2018 but is not assumed as a significant difference. The oil samples achieved comparable volumes of water uptakes and water uptake rates, and both oils (2018 and 2008) formed stable w/o emulsions that were partly broken when adding emulsion breaker. Overall, the obtained laboratory weathering data for Draugen 2018 were not found to be significantly different from Draugen 2008, except for the water-free and emulsified viscosities on 250°C+ residue that decreased significantly from 2008 to 2018.

SINTEF OWM predictions and oil spill response

The laboratory data obtained from this verification study (re-check) were used in combination with the existing weathering data of Draugen 2008 as input to the SINTEF OWM. Weathering predictions were performed for water uptake, emulsion viscosities, pour points and mass balances. The predominant deviations in the OWM predictions between the two oils were the lower predicted emulsion viscosities when using weathering data from Draugen 2018, and the higher pour point predictions for 2018 compared with 2008.

The lower emulsion predictions show an increase in the time window for use of dispersants with Draugen 2018 compared to Draugen 2008 for when the oils are expected to have a reduced dispersibility (> 4000 mPa.s). On the other hand, the somewhat higher pour point predictions for Draugen 2018 indicate a shorter time window for use of dispersant if the oil /residue solidifies on the sea surface. If such solidified (low emulsified /water-free) lumps are observed on the sea surface, a lower dispersant effectiveness is likely. Solidification typically arises with pour points 5-15 °C above the sea temperature.

In addition, storage of weathered emulsions, with high pour point of the oil residue, may require heating when transferring recovered oil from the storage tank from the recovery vessel. This phenomenon was experienced during the incident at the Draugen oil field in 2003. Solidification at the sea surface is therefore expected to be more challenging operation at winter conditions of the Draugen crude oil due to the high pour point of the oil /residue. The use of dispersants will therefore likely be most efficient within 1-2 days after a spill on the sea surface for this crude oil, and the time window might be even shorter in winter conditions at higher wind speeds.

Moreover, the use of weir skimmers should be efficient on the Draugen 2018 emulsions after several days of weathering for viscosities due to viscosities < 15-20 000 mPa.s (Leirvik et al., 2001).

Evaluation and recommendation

Based on a total evaluation, using the verified 2018 laboratory data and the combined OWM predictions, the existing weathering data of Draugen 2008 can still justify being valid related to oil spill response. However, due to the lower viscosities of Draugen 2018, the existing weathering data of Draugen 2008 can be considered as a conservative alternative to Draugen 2018.

5 References

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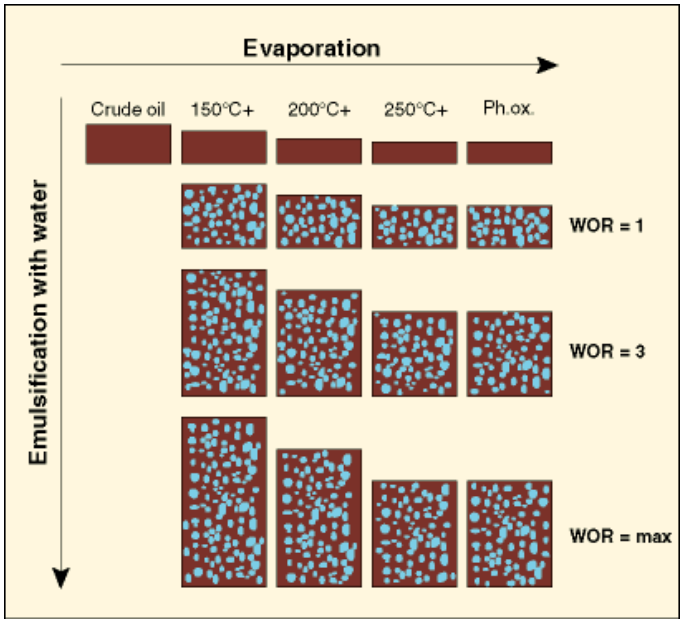
A Experimental setup

The verification study (re-check) was performed at 13 °C.

Small-scale laboratory testing

To isolate and map the various weathering processes at sea, crude oil was exposed to a systematic, stepwise procedure developed at SINTEF (Daling et al., 1990). The general procedure is illustrated in Figure A-1.



WOR: Water to Oil Ratio

WOR=1: 50 vol.% water

WOR=3: 75 vol.% water

WOR=max: the maximum water content

Figure A-1: Small-scale laboratory weathering flow chart of oil

Evaporation

The density of the oil was monitored during the degassing. This was performed before evaporation by standard procedure. The evaporation procedure used is described in Stiver and Mackay (1984). Evaporation of the lighter compounds from the fresh oil was carried out as a simple one-step distillation to vapour temperatures of 150 °C, 200 °C and 250 °C, which resulted in oil residues with an evaporation loss corresponding to approximately 0.5-1-hour, 0.5-1 day and 0.5-1 week of weathering on the sea surface. These residues are referred to as 150°C+, 200°C+ and 250°C+, respectively.

Physical and chemical analysis

The viscosity, density, pour point of the fresh and water-free evaporated residues were analysed. In addition, wax and asphaltene content was measured for the 250°C+ residue. Viscosity for w/o emulsions was also determined. The analytical methods are given in Table A-1 and Table A-2.

Table A-1: Analytical methods used to determine the physical properties

Physical property	Analytical method	Instrument
Viscosity	McDonagh et al, 1995	Physica MCR 300
Density	ASTM method D4052-81	Anton Paar, DMA 4500
Pour point	ASTM method D97	-

Table A-2: Analytical methods used to determine chemical properties

Chemical property	Analytical method
Wax content	Bridiè et al, 1980
"Hard" asphaltene	IP 143/90

Chemical characterization by GC/FID

The distribution of hydrocarbons (*n*C5-*n*C36) was analysed using a Gas Chromatograph coupled with a Flame Ionisation Detector (GC/FID). The Gas Chromatograph was an Agilent 6890N with a 30m DB1 column.

Emulsification properties

The w/o emulsifications were performed by the rotating cylinders method developed by Mackay and Zagorski (1982), which is described in detail by Hokstad et al. (1993). The method includes the measuring of the following parameters:

- Relative water uptake (kinetics)
- Maximum water uptake (volume)
- Stability of the emulsion
- Effectiveness of emulsion breaker (Alcopol 60%)

The principle of the rotating cylinders method is illustrated in Figure A-2. Oil (30 mL) and seawater (300 mL) are mixed and rotated with a rotation speed of 30 rpm in separating funnels (0.5 L). The emulsification kinetics is mapped by measuring the water content at fixed rotation times. The maximum water content is determined after 24 hours of rotation.

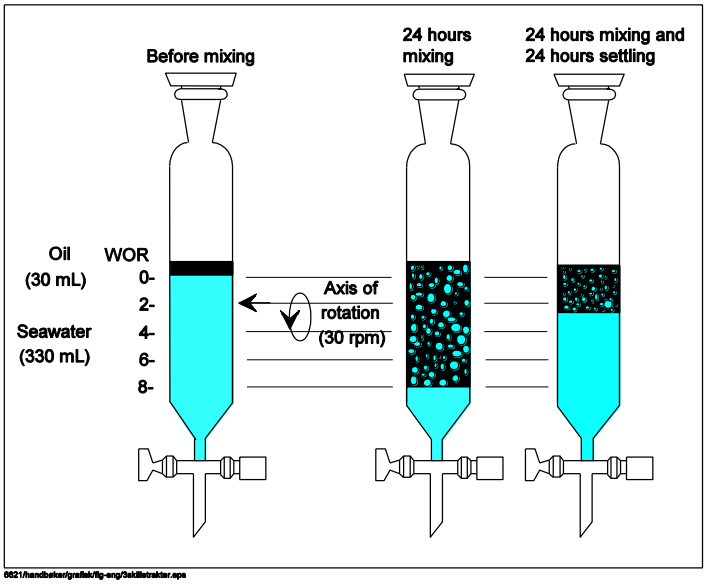


Figure A-2: Principle of the rotating cylinder method

B Predictions with SINTEF Oil Weathering Model (OWM)

The laboratory data used as input to the SINTEF OWM for Draugen 2018 is given in Table B- and Table B-2. The oil weathering predictions were based on a combination of the previously data from 2008 (150 °C+ residue) and the recent data for 2018 (200 and 250 °C+ residue). In addition, the True Boling Point (TBP) curve from Draugen 2008 was used in these predictions, as no updated TBP for 2018 was available. The TBP and the exiting dispersibility limits are found in Leirvik, 2008.

Table B-1: Crude Assay (CA) data for Draugen 2018 at 13 °C

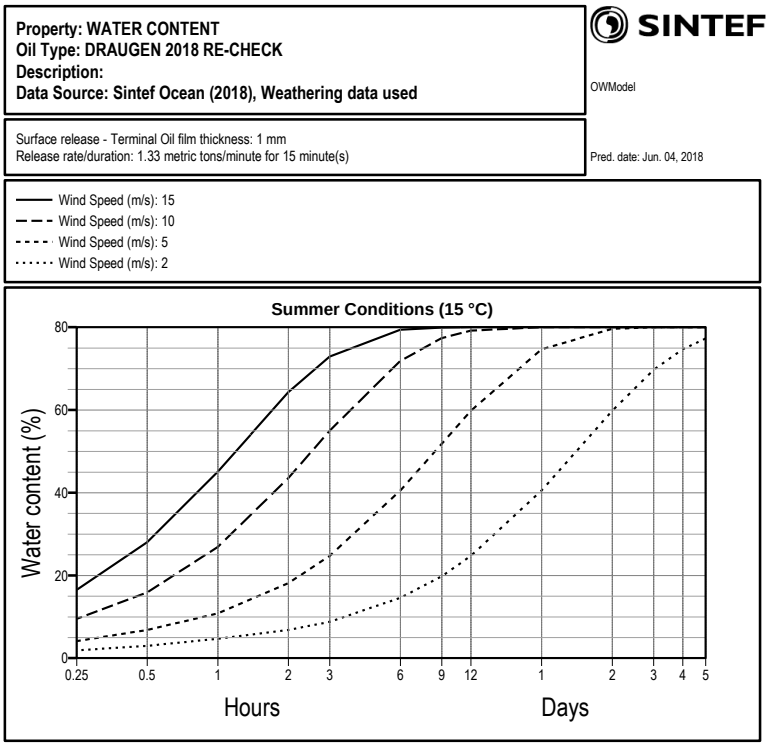
Properties of fresh oil	**Value**
Density (g/mL)	0.822
Pour point (°C)	-24
Reference temperature (°C)	13
Viscosity at ref. temp. (mPa.s = cP) *	6
Asphaltenes (wt. %)	0.09
Wax Content (wt. %)	3.0

** Measured at shear rate 10 s^{-1}*

Table B-2: Laboratory weathering data for Draugen 2018 at 13 °C

Properties	**Fresh**	**150°C+ *****	**200°C+**	**250°C+**
Boiling Point Temp. (°C)	-	201	249	297
Vol. Topped (%)	0	30	39	48
Weight Residue (wt. %)	100	74	66	57
Density (g/mL)	0.822	0.875	0.887	0.897
Pour point (°C)	-24	3	12	21
*Viscosity of water-free residue (mPa.s =cP)	5	34	92	255
**Viscosity of 50% emulsion (mPa.s = cP)	-	424	1100	1689
**Viscosity of 75% emulsion (mPa.s = cP)	-	1510	677	2121
**Viscosity of max water (mPa.s = cP)	-	-	599	1658
Max. water cont. (vol. %)	-	78	89	82
(T1/2) Halftime for water uptake (hrs)	-	0.50	0.30	0.12
Stability ratio	-	1	0.81	0.99

** Measured at shear rate 100 s^{-1} ** Measured at shear rate 10 s^{-1} *** Data from Draugen 2008*
- No data



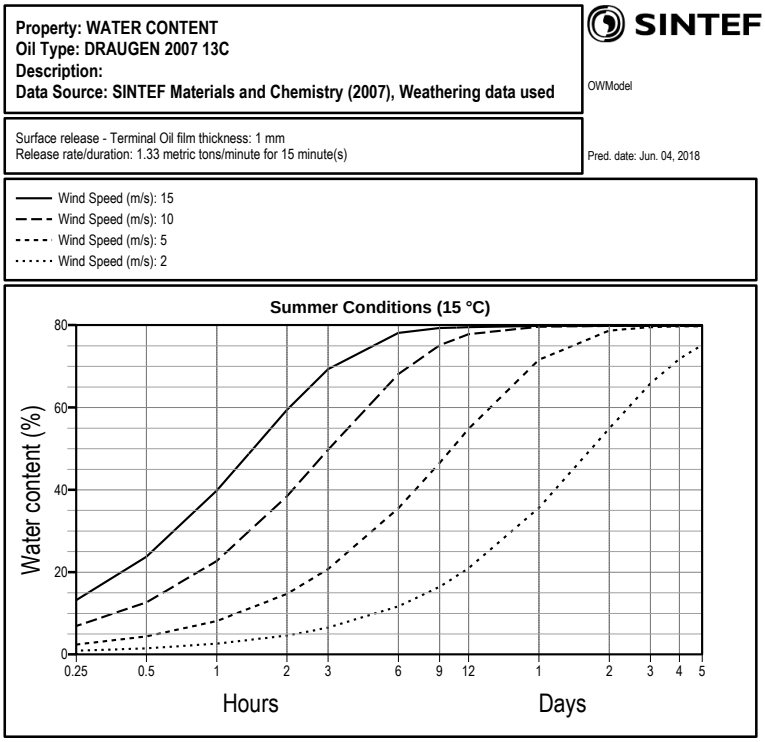
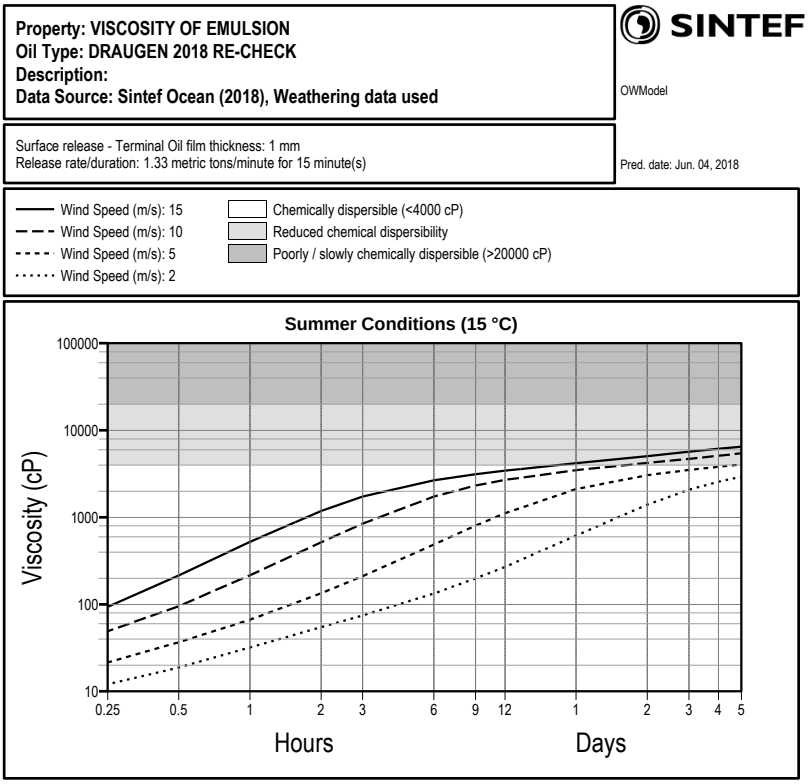


Figure B-1: Water uptake of Draugen 2018 (above) and Draugen 2008 (below)



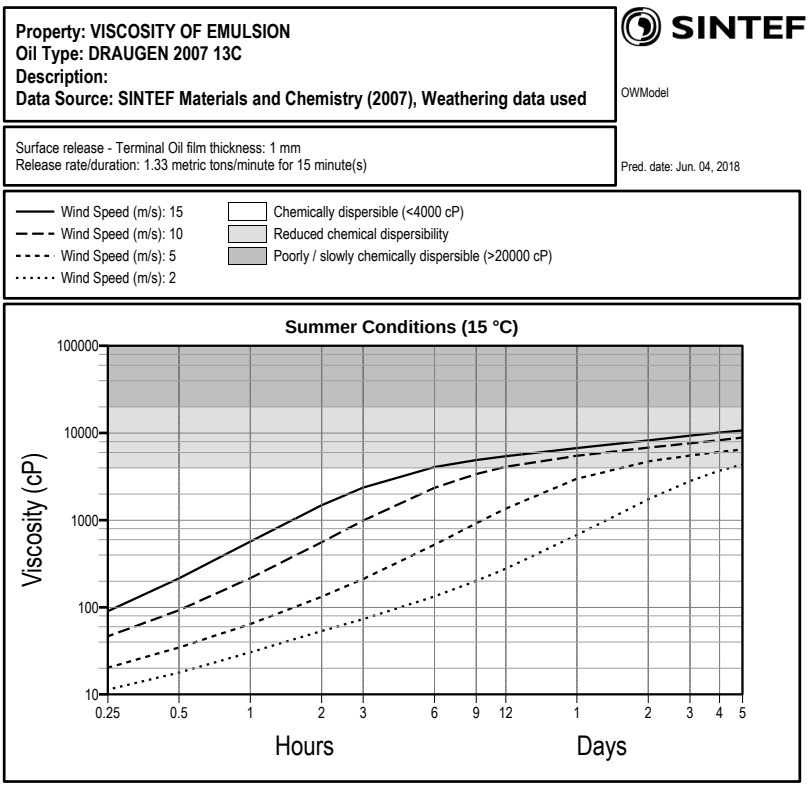
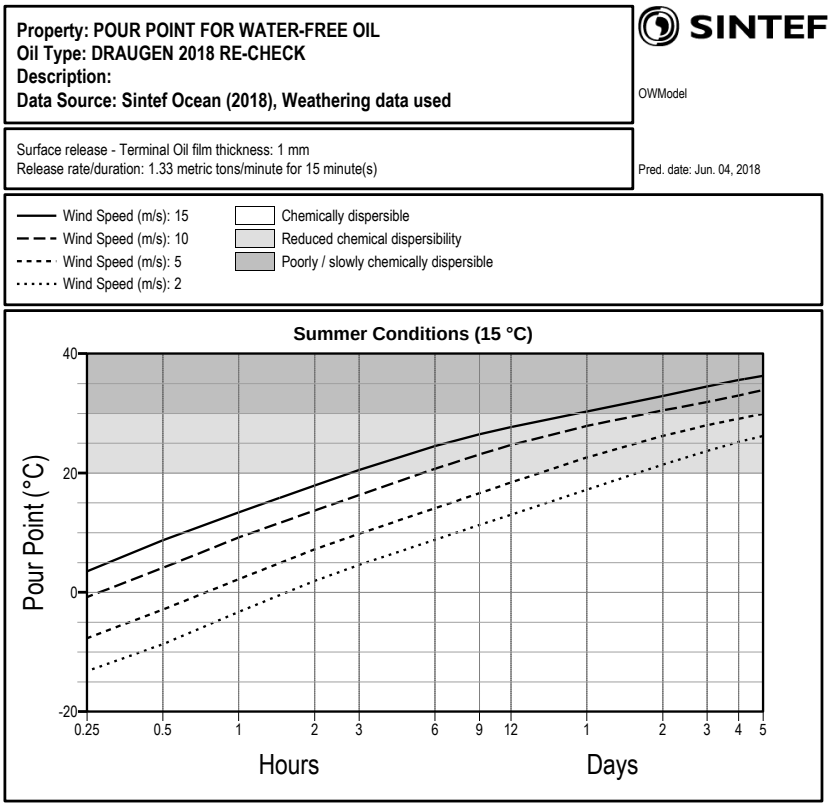


Figure B-2: Emulsion viscosity of Draugen 2018 (above) and Draugen 2008 (below)



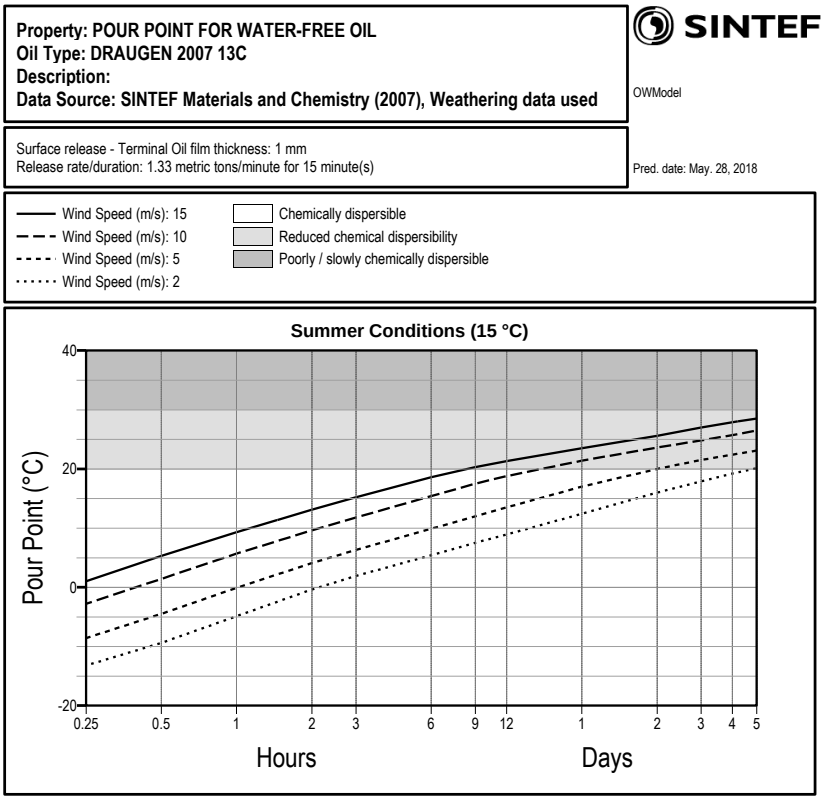


Figure B-3: Pour point of Draugen 2018 (above) and Draugen 2018 (below)

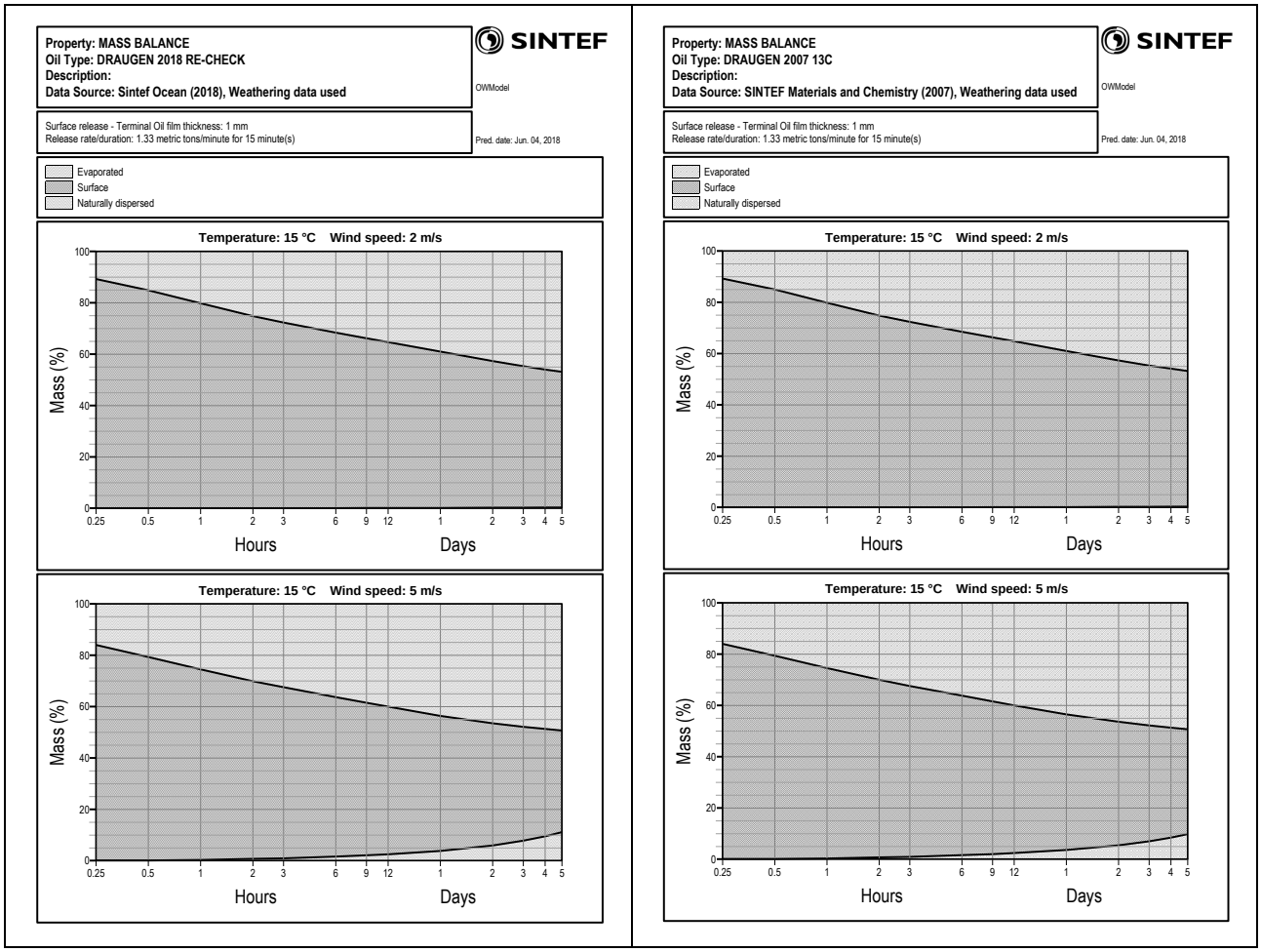


Figure B-4: Mass balance of Draugen 2018 (left) and Draugen 2008 (right) at 2 and 5 m/s and 15 °C

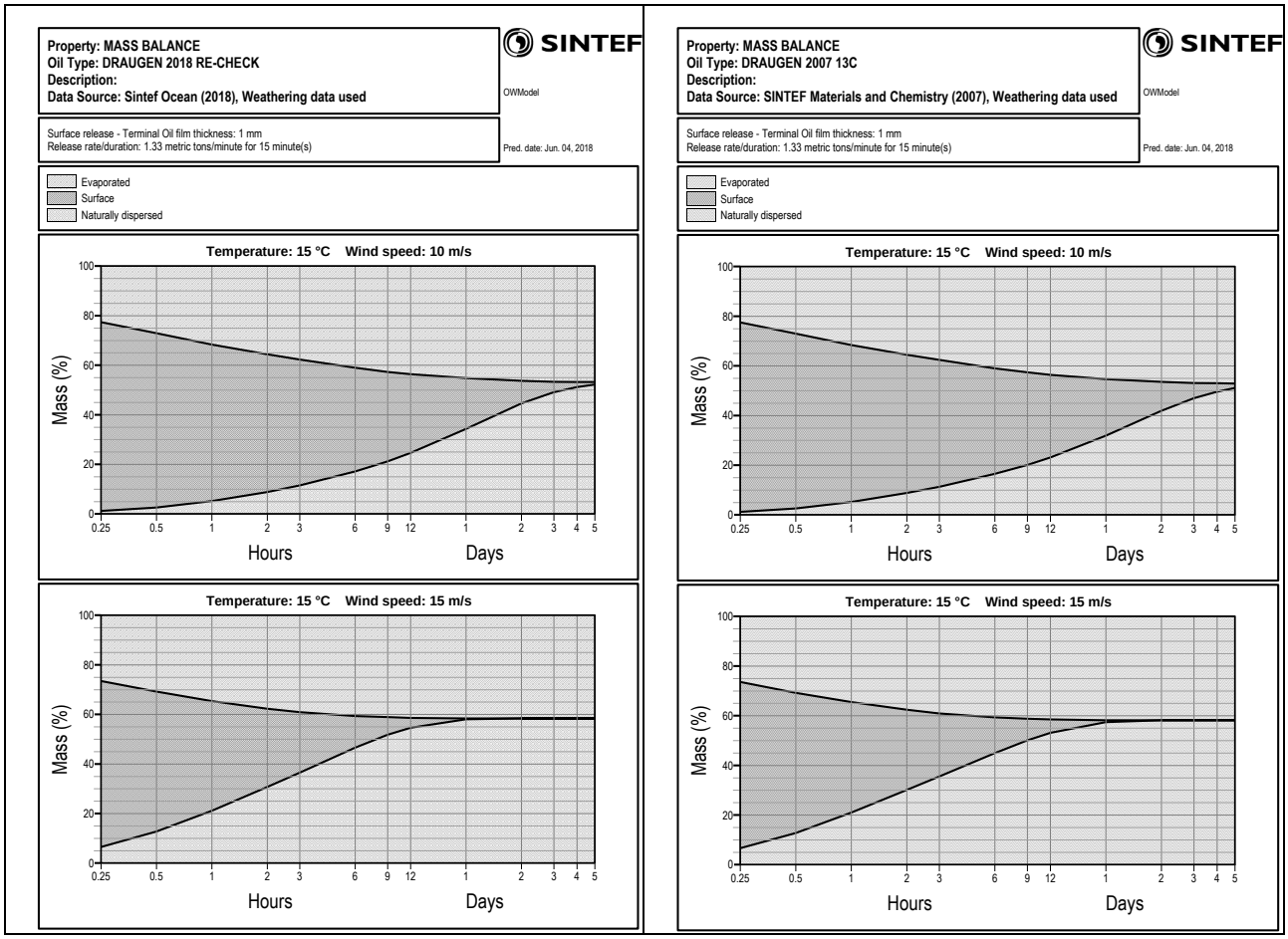


Figure B-5: Mass balance of Draugen 2018 (left) and Draugen 2008 (right) at 10 and 15 m/s and 15 °C

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