Report

Physico-chemical characterization and weathering properties of IM-5 Wakashio

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Report

Physico-chemical characterization and weathering properties of IM-5 Wakashio

Very Low Sulfur Fuel Oil (VLSFO)

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ABSTRACT

This project, to studying the physico-chemical characteristics and weathering properties of IM-5 Wakashio (VLSFO, S ≤0.50 % m/m), has been funded by Norwegian Coastal Administration (NCA). This is a supplement study to the on-going IMAROS/EU project led by the NCA. The main objective has been to conduct a weathering study of the IM-5 Wakashio residual fuel oil including standardized bench-scale and meso-scale flume testing at 2 and 15 °C, reflecting typically seawater temperatures in the arctic/cold climate regions, and summer North Sea conditions. The laboratory data were used as input to the SINTEF Oil Weathering Model (OWM) for weathering predictions of the IM-5 Wakashio fuel oil at 2 and 15 °C.

Findings from this study indicate e.g. that IM-5 Wakashio has lower viscosity compared to other previous tested low sulfur marine residual fuel oils (VLSFO/ULSFO) and bunker fuel oils (IFO-180/380), but has similarities with e.g. IFO-30. IM-5 Wakashio has higher viscosity compared with marine distillates (marine gas oils or diesel oils). Overall, IM-5 Wakashio is not considered as an "extreme" oil and is in the lower range with respect to physico-chemical properties compared with previous tested low sulfur marine residual fuel oils.

Mechanical recovery is expected to be the main strategy for oil spill response on IM-5 Wakashio. Use of dispersants can be an option during the first hours on IM-5 Wakashio, but after several days of weathering the dispersion effectiveness is expected to be low.

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Tables of contents

SINTEF

1 Executive summary

This project to studying the physico-chemical characteristics and weathering properties of IM-5 Wakashio very low sulfur fuel oil (VLSFO, S≤0.50 % m/m) has been funded by Norwegian Coastal Administration (NCA) as a supplementary study as a part of the on-going IMAROS/EU project. IM-5 Wakashio is one of the 13 test oils in the IMAROS /EU project. SINTEF received in this connection about 40 litres of IM-Wakashio through Cedre (France). The oil samples came from the salvage operation after the vessel MV "Wakashio" grounded on the coral reefs in Mauritius 25th July 2020.

The main objective has been to conduct a laboratory oil weathering study of the IM-5 Wakashio residual fuel oil including a standardized bench-scale testing and meso-scale flume basin testing at 2 and 15 °C, reflecting typically seawater temperatures in the arctic or cold climate regions, and summer North Sea conditions. The laboratory data conducted from this study were used as input to the SINTEF Oil Weathering Model (OWM) for weathering predictions of the IM-5 Wakashio fuel oil at 2 and 15 °C.

IM-5 Wakashio showed a low evaporative loss of 12 vol.% (250°C+ residue, reflecting typically 0.5-1 week at sea), and is in the same range of other previous tested residual low sulfur fuel oils at SINTEF that included both VLSFOs and ULSFOs; ultra-low sulfur fuel oils, S ≤0.10 % m/m). Neither the density (0.908 g/mL), pour point (+9 °C), nor the asphaltenes (0.52 wt.%) and wax (5.4 wt.%) are outliers, and the analytical parameters showed also to be in the lower range compared with previous tested residual fuel oils at SINTEF. Also, the viscosity of IM-5 Wakashio (temperature-sweep 50-0 °C) showed a lower level in the range of 2-15 °C compared with other VLSFOs and ULSFOs, and examples of IFO-180/380. Moreover, IM-5 Wakashio has a viscosity of ~33 mPa.s at 50 °C and exhibits a similar viscosity behaviour as intermediate bunker fuel oil (IFO-30) and is in the lower range of the previous tested residual low sulfur fuel oils (except from one of the ULSFOs). The viscosity of IM-5 Wakashio is higher than marine distillates (DMA diesel). Overall, based on the physico-chemical properties, IM-5 Wakashio is not considered as an "extreme" oil type compared with previous tested low sulfur marine residual fuel oils.

IM-5 Wakashio emulsified both at 2 and 15 °C (55 and 65 vol.%, respective) from the bench-scale testing. However, previous experiences have shown that viscous residual fuels may underestimate the water uptake in rotating cylinders (bench-scale) and flume basin testing may therefore be an important supplement (larger scale).

Meso-scale flume weathering were included on IM-5 Wakashio at 2 and 15 °C. The results from the flume basin testing showed consistent trends with increasing viscosity and water uptake during the weathering. The weathering time in the flume was 7 days (168 hours) to harmonize with Cedre's test procedures. The water uptake increased to 66 vol. % at 2 °C, and 80 vol. % at 15 °C, and the emulsion viscosities reached 39 960 mPa.s and 22 083 mPa.s, respective during one-week of weathering in the flume basin. The oil showed low evaporative loss (7-13 vol.%) in the flume and negligible naturally dispersed/entrained oil lumps in the water phase during the one-week weathering. The flume basin experiments were terminated with application of dispersant (Dasic Slickgone NS). The application of dispersant was able to break up the slick into smaller patches or lumps, but required a high amount of dispersant (i.e. dispersant-to-emulsion ratio; DER 1:14 and DER 1:33 at 2 and 15 °C, respective) and 3-4 replications. Moreover, the application of dispersant after one-week of weathering showed low dispersion effectiveness. However, IM-5 Wakashio showed to have a potential to be good dispersible during the first hours (4-6 h) of weathering at 2 and 15 °C, using a simplified FET (field effectiveness test).

The SINTEF Oil Weathering Model (OWM) related oil properties to a chosen set of conditions (oil/emulsion thickness, wind speeds and seawater temperature), and predicts the change of oil properties on the sea surface by time. In this report, the presented predictions span a period from 15 min. to 7 days based on a standard release rate (80 m^3/h). The mass balances show the IM-5 Wakashio is very persistent on the sea surface in non-breaking waves conditions (2-5 m/s wind speeds) at 2 and 15 °C. At higher wind speeds (10-15 m/s wind), the oil is less persistent due to entrained oil into the water column by increasing wave energy. It is however expected that naturally entrained oil would behave as larger lumps and will easily resurface in calmer weather conditions.

As a part of this project, specific analytical data from the bench-scale testing of IM-5 Wakashio were compared with similar parameters conducted at Cedre, as a part of an interlaboratory comparison of results. The physico-chemical parameters of density, viscosity, wax and asphaltenes were in the same range and hence comparable results between the laboratories.

Preliminary evaluation of oil spill response in cold climate region or arctic conditions of IM-5 Wakashio:
Mechanical recovery should be considered as the main strategy for an oil spill of IM-5 Wakashio in cold climate regions/arctic conditions. The emulsion viscosity may reach to about 40.000 mPa.s during one-week of weathering at sea, which is a medium range viscous-emulsions (10-50 000 mPa.s). In addition, the pour point of IM-5 Wakashio is not that high to expect a server degree of solidification at sea that will influence negatively on the mechanical response strategy when using high visc. skimmers (e.g. brush, belt and drum skimmers). The efficacy of traditional weir skimmers will typically be reduced for viscosities > 15 000 mPa.s.

Use of dispersants can be an option during the first hours on IM-5 Wakashio, but after several days of weathering the dispersion effectiveness is expected to be low. The meso-sale flume testing showed that after one week weathering at sea the use of dispersant on IM-5 Wakashio was found to break up the slick into smaller patches, but not as a good dispersion with formation of small oil droplets. This required also breaking wave conditions and successive applications of dispersants with high dosages. Overall, use of dispersants is not recommended as a main strategy for heavily weathered IM-Wakashio due to the short time-window for effective dispersant use.

In-situ burning (ISB) is often considered as a possible primary response operation in arctic and ice-covered areas. The ice can be used to confine the oil and increase the film thickness to increase the ignitability. However, ISB is dependent on other factors such as wind speeds and a water contents (degree of emulsification, e.g. < 30 vol. % water). This means that ISB would likely be an option during the in the first 3-5 hours of IM-5 Wakashio in low wind speeds (<5 m/s), however, the oil may require prolonged time to be heated by burning gelled gasoline /diesel mixture as shown for other VLSFOs and ULSFOs tested at SINTEF.

2 Abbreviations, Acronyms and Symbols

Abbreviations	Definitions
ASTM	American Society for Testing and Materials
BDN	Bunker Delivery Note
b.p.	boiling point
BTEX	Benzene, Toluene, Ethylbenzene and Xylene
COA	Certificate of analysis
cP	centipois (= mPa.s)
DER	Dispersant-to-emulsion ratio
DMA	Marine distillate gas oil (according to ISO 8217:2017)
DOR	Dispersant-to-oil ratio
GC-FID	Gas chromatography – Flame Ionization Detector
HDME 50	Heavy Distillate Marine ECA 50
FET	Field Effectiveness Test
IFO /HFO	Intermediate Fuel Oil / Heavy Fuel Oil
IMO	International Maritime Organization
IMAROS	Project title: "Improving response capacities and understanding the environmental impacts of new generation low sulphur MARine fuel Oil Spills"
ISB	In-situ burning
LIMS	Laboratory Information Management System
LSFO	Low Sulfur Fuel Oil
m/m	mass by mass
mPa.s	millipascal second. Unit for dynamic viscosity
m/s	meter per second
Newtonian fluid	Fluid whose viscosity does not change with rate of flow
NCA	Norwegian Coastal Administration
Non-Newtonian fluid	A non-Newtonian fluid is a fluid that does not follow Newton's law of viscosity, i.e. constant viscosity independent of stress
OWM	Oil Weathering Model
PAH	Polyaromatic hydrocarbon
ppm	parts per million
S	Sulfur
s^{-1}	reciprocal second
SI	International System of Units
SIMDIS	Gas Chromatographic Simulated Distillation
SVOC	Semi-volatile organic compound
TBP	True Boiling Point
ULSFO	Ultra-low sulfur fuel oil: ≤ 0.10 S wt. % (SECA, 2015)
UCM	Unresolved Complex Mixture
VLSFO	Very Low Sulfur Fuel oil: ≤0.50 wt. % (outside SECA from 2020)
VOC	Volatile organic compounds
vol.%	volume percent
WRG	Wide range gas oil
WOR	Water-to-oil ratio
wt. %	weight percent

3 Introduction

3.1 Background

The MV "Wakashio" vessel grounded on the coral reefs in Mauritius 25th July 2020. The vessel contained about 3,900 tons onboard of very low sulfur fuel oil (VLSFO, S≤0.50 % m/m) from two different bunkering of residual fuels, in addition to about 200 tons diesel and some lubricants. A big salvage operation took place during the weeks to emptying the tanks. However, approximately 1000 tons of marine fuel oil were leaked out into the sea. As a part of the on-going IMAROS/EU project led by the Norwegian Coastal Administration (NCA), SINTEF received about 40 litres of the marine residual fuel oil from Cedre (France), hereinafter called IM-5 Wakashio (Table 3-1), for analysis of physico-chemical characteristics and weathering properties. The batch of IM-5 Wakashio came from the salvage operation from one of the bunker fuel tanks onboard the vessel and indicates coming from the most recent bunkering in Singapore 14th July according to BDN (Bunker Delivery Note).

Table 3-1 Overview of reference of IM-5 Wakashio.

IMAROS ref	Cedre ref	SINTEF ref	Product	Country
IM-5	HC-20-167	2020-9915	Wakashio VLSFO	France

3.2 Objective

The main objective has been to conduct a laboratory oil weathering study of the IM-5 Wakashio residual fuel oil including a standardized bench-scale testing and meso-scale flume testing at 2 and 15 °C, reflecting typically seawater temperatures in arctic or cold climate regions, and summer North Sea conditions. The laboratory data conducted from this study were customized for input to the SINTEF Oil Weathering Model (OWM) for weathering predictions of the IM-5 Wakashio fuel oil. In addition, specific analytical data from the bench-scale testing of IM-5 Wakashio were also compared with similar parameters conducted at Cedre, as a part of an interlaboratory calibration for comparison of results.

The project included the following main tasks:

- Physico-chemical analysis of the fresh and 250°C+ residue.
- Emulsion testing at 2 and 15 °C on the fresh oil and 250°C+ residue.
- Meso-scale oil weathering flume testing at 2 and 15 °C, over 7 days weathering time.
- OWM predictions of oil weathering properties of IM-5 Wakashio at 2 and 15 °C.
- Comparison of results of selected parameters between SINTEF and Cedre.
- Evaluation of results and reporting.

4 Bench-scale laboratory testing results

The bench-scale laboratory study of the IM-5 Wakashio was conducted at 2 and 15 °C. The methodology used is described in more details in Appendix A. The generated weathering data from this study were also compared with similar data from previous studies at SINTEF on low sulfur residual fuel oils as shown in Table 4-1, and other low sulfur marine fuel oils when applicable.

Table 4-1 Low sulfur residual fuel oils from previous studies at SINTEF compared with IM-5 Wakashio.

Oil names*	Report ref	SINTEF ref	References	Comments
VLSFO Chevron 2019	VLSFO-1	2019-3599	Sørheim et al.2020	Project supported by DFO/MPRI, ITOPF and NCA
VLSFO Shell 2019	VLSFO-2	2019-7685	Sørheim et al. 2020	Project supported by DFO/MPRI, ITOPF and NCA
ULSFO Shell 2016	ULSFO-1	2016-0233	Hellstrøm, 2017 Hellstrøm et al. 2017	Project supported by NCA
ULSFO Shell 2019	ULSFO-2	2019-11170	Sørheim et al. 2020	Project supported by DFO/MPRI, ITOPF and NCA

**The oil names are denoted as VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2 respectively throughout this report.*

4.1 Registration and pre-handling

Oil samples (8 x 5 litres) cans of the IM-5 Wakashio arrived at SINTEF laboratory on the 4th of December 2020. The cans were registered in LIMS (Laboratory information management system) and given the unique SINTEF identification number: 2020-9915. Free-water from the oil samples (cans) were drained off as a part of pre-handling of the fuel oil, and the water volume varied from zero to 1 litres in the different cans. The free-water was found to be seawater measured by use of a refractometer. Water content in the oil phase was measured by use of Karl-Fisher titration, as a part of the HSE risk assessment prior to distillation (evaporation) of the fresh oil (chapter 4.2). Oil samples (cans) with water content <2 vol.% were thoroughly homogenized before distillation and further analysis. Figure 4-1 shows example of one of the cans received at the laboratory (left), and about 1 litre of free-water drained off from one of the cans (right).

Figure 4-1 Left: 1 of the 8 cans with IM-5 Wakashio. Right: Free-water, approx.1 litre of seawater, drained off from one of the cans.

4.2 Evaporation

The standardized evaporation procedure is a simple one-step distillation to vapour temperatures of 150 °C, 200 °C and 250 °C (Stiver and Mackay, 1984). The IM-5 Wakashio fuel oil was distilled to 250°C+, agreed with NCA and Cedre based on the very low content of lighter compounds.

4.3 True boiling point (TBP) curve

The true boiling point (TBP) or distillation curve is obtained by measuring the vapour temperature as a function of the amount of oil distilled, shows the relative distribution of volatile and heavier components in the oil. The boiling point of a chemical component depends on its vapour pressure, which is a function of its molecular weight and chemical structure. Hence, the distillation curve is an indicator of the relative amount of different chemical components, principally as a function of molecular weight, but also as determined by the chemical composition. The TBP of IM-5 Wakashio was analysed by use of "simulated distillation of marine fuel oils" in accordance with ASTM D7169 (Intertek UK).

The TBP (wt.%) of IM-5 Wakashio is shown in Figure 4-2 in comparison of selected other low sulfur fuel oils (VLSFOs and ULSFOs), in addition to two traditional heavy fuel oil (IFO 180 /380). In comparison with other residual fuel oils, IM-5 Wakashio is neither a heavy nor a light fuel oil. The black vertical lines in Figure 4-2 show the b.p > 500°C that reflects heavy residuals, and b.p.> 250°C+ that indicates the tentative evaporative loss over some days at sea.

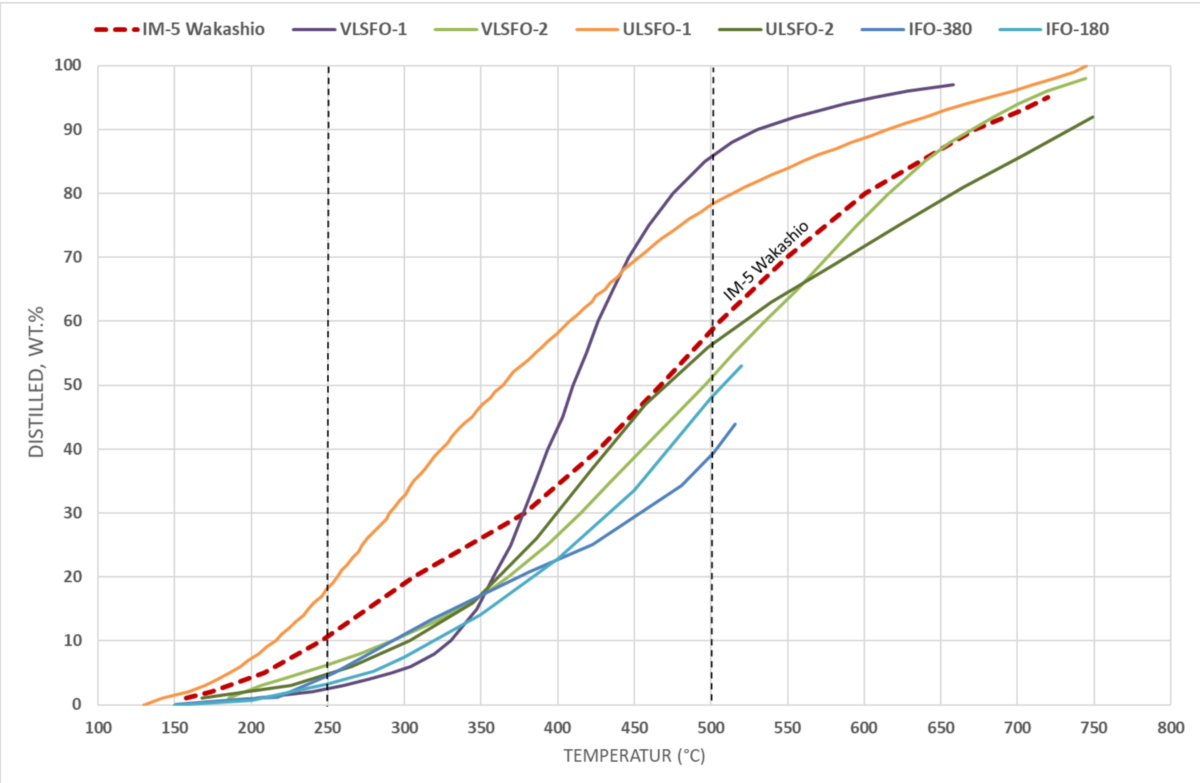


Figure 4-2 TBP of IM-5 Wakashio (red line) in comparison with other VLSFOs, ULSFOs and IFOs. The black vertical lines show the b.p > 500°C that reflects heavy residuals, and b.p.> 250°C+ that indicates the tentative evaporative loss after typically 0.5-1 week at sea.

4.4 Gas chromatographic (GC-FID) characterization

The gas chromatograms show the n-alkanes (paraffins) as systematic narrow peaks. The first peaks in the chromatogram represent components with the lowest boiling points (b.p). Some of the more complex components, such as resins and naphthenes, shown as a broad and poorly defined bump below the sharp peaks, are often described as "Unresolved Complex Mixture" (UCM). Heavier compounds such as asphaltenes (> nC40) are not possible to analyse with this technique. Figure 4-3 shows the gas chromatographic characterization of the fresh IM-5 Wakashio and its 250°C+ residue. The lightest compounds have been evaporated, e.g., the nC14 (253 °C) peak is clearly reduced upon evaporation.

The GC-chromatogram of the fresh oil IM-5 Wakashio is compared with other previous tested VLSFOs and ULSFOs, as shown in Figure 4-4. The marine residual fuels oils in comparison exhibit different hydrocarbon profiles reflecting a great wide span in the chemical composition. IM-5 Wakashio has a broad range of n-alkanes in nC9-nC36, similar as for the two batches of ULSFOs but they still exhibit different hydrocarbon profiles. The VLSFO-1 shows n-alkanes in the range of nC20 to nC30 with minor content of compounds lower than nC17 (300 °C). The VLSFO-2 exhibits high peaks of naphthalenes shown as irregular compounds relative to the n-alkanes (the n-alkanes are almost absent).

By combining TBP (Figure 4-2) to the GC, the percent (%) of mass above C36 (boiling point, b.p.>500° C) was estimated. The vertical lines in Figure 4-4 at nC36 illustrate the mass % of residual components with b.p.>500 °C that is discriminated (none-chromatographable compounds) in the GC-analysis. IM-5 Wakashio has mass of components (approx. 42%) above 500 °C (Figure 4-2) that is in the same range as VLSFO-2 and ULSFO-2.

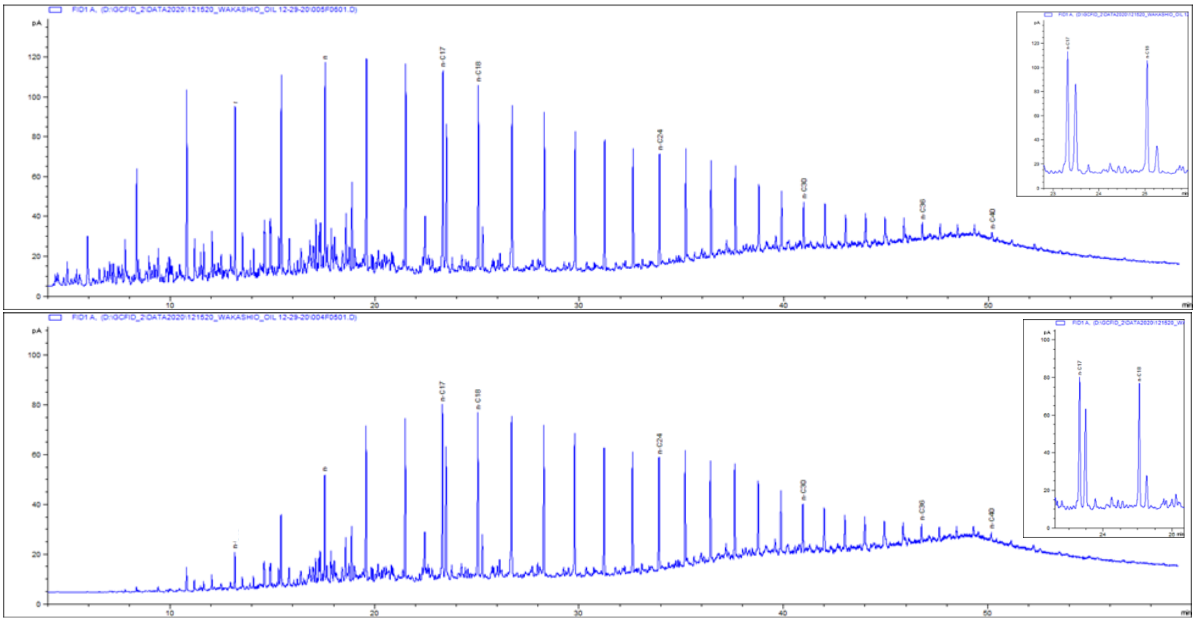


Figure 4-3 GC-FID chromatograms of fresh oil and evaporated 250°C+ residue of IM-5 Wakashio.

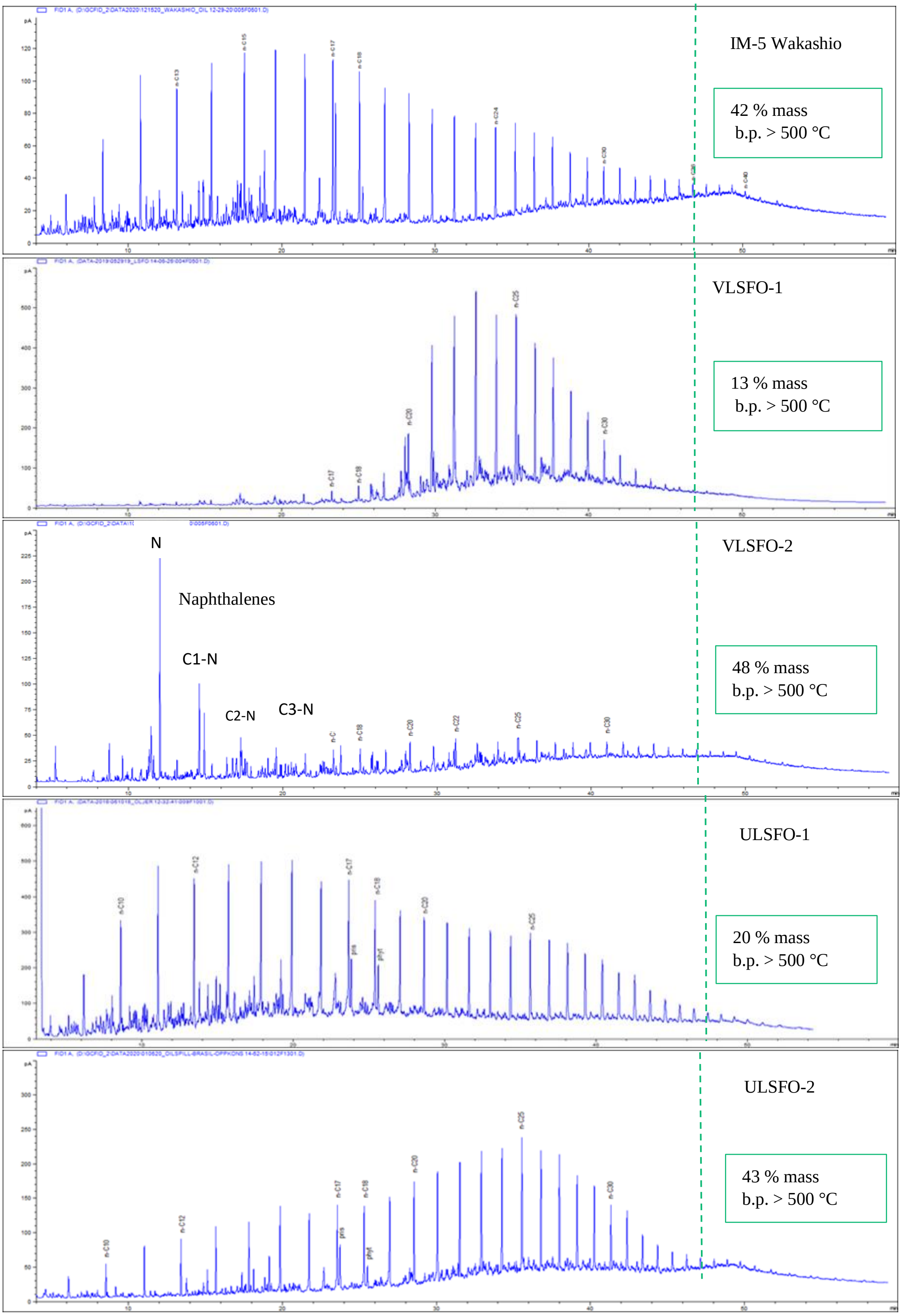


Figure 4-4 *GC-FID chromatograms of fresh samples of IM-5 Wakashio in comparison with VLSFO-1, VLSFO-2, ULSFO-1 and ULSFO-2. The green vertical lines at nC36 illustrate the mass % of residual components with b.p.>500 °C (see Figure 4-2) that is discriminated (none-chromatographable compounds) in the GC-analysis.*

4.5 Physico-chemical properties

Generally, both wax and asphaltenes contribute to stabilize water-in-oil emulsion. Asphaltenes have a surface-active property that form a "skin layer" at the water-oil interface that stabilizes the water droplets, whilst the waxes contribute to stabilize the asphaltenes near the water-oil interface.

The contents of asphaltenes and wax for the IM-5 Wakashio are given in Table 4-2, in comparison with other VLSFOs and ULSFOs. The results show a high variation of asphaltenes and wax content among the fuel oils. IM-5 Wakashio has a medium content of wax (5.4 wt.%) and is in the same range as the other VLSFOs. The asphaltene content (0.52 wt.%) of IM-5 Wakashio is in the same lower range as VLSFO-1, whilst VLSFO-2 has the highest content of asphaltenes 4.8 wt.%. The two batches of ULSFOs have low and similar content of asphaltenes (0.14-0.15 wt.%) compared to the VLSFOs but exhibit very high wax contents of 13-21 wt.%.

Table 4-2 Asphaltene ("hard") and wax content

Oil type	Residue	Asph. * (wt. %)	Wax (wt. %)
IM-5 Wakashio	Fresh 250°C+	0.52 0.59	5.4 6.0
VLSFO-1	Fresh	0.44	4.5
VLSFO-2	Fresh 250°C+	4.8 5.2	4.9 5.3
ULSFO-1	Fresh 250°C+	0.15 0.18	13.1 15.5
ULSFO-2	Fresh 200°C+ 250°C+	0.14 0.15 0.15	20.7 21.1 21.6

**n-heptane (nC_7) precipitation*

The physical properties of IM-5 Wakashio are listed in Table 4-3 in comparison with other VLSFOs and ULFSOs. The evaporative loss of IM-5 Wakashio is relatively low (12 wt.%) and is in the same range as the ULSFO-1with densities 0.87-0.91 g/mL. However, the other oils in comparison had either insignificant or even lower evaporative loss (5.1-7.9 vol.% reflecting more high-density fuel oil (0.92-0.99 g/mL. IM-5 Wakashio has pour points of +9 (fresh oil) to +15 °C (250°C+) which is in the same range as the two other VLSFOs but is lower than the two batches of ULSFO (+30 °C for 250°C+ residues).

Table 4-3 shows that all the residual oils behave as non-Newtonian fluids with decreasing viscosity with increasing shear rate, (s^{-1}) due to the wax lattice structure that breaks up with increasing applied shear rate. The oils have therefore higher viscosities at a lower shear rate (e.g. $10s^{-1}$) compared to the viscosities measured at higher shear rates (e.g. 100 s^{-1}). IM-5 Wakashio has significantly higher viscosities at 2 °C than measured at 15 °C, and the viscosities decrease with increasing shear rates. However, compared with all the other oils in Table 4-3, the viscosities measured at 2 °C (6402 mPa.s, $10s^{-1}$) and 15°C (1199 mPa.s, $10s^{-1}$) are considerably lower for IM-5 Wakashio.

Figure 4-5 shows a comparison of IM5-Wakashio with other fuel oils tested at SINTEF. The figure clearly shows that IM-5 Wakashio does to differ from the other oils on one way or another, i.e. IM-5 Wakashio is not consider to be an extreme of different fuel oil with respect to density, pour point, and asphaltenes and wax contents.

Table 4-3 *Physical properties of IM-5 Wakashio in comparison with other VLSFOs and ULSFOs.*

Oil types	Residue	Evap. (vol. %)	Res. (wt. %)	Dens. (g/mL)	Flash point (°C)	Pour point (°C)	Visc. (mPa.s) 2°C $10s^{-1}$	Visc. (mPa.s) 2°C $100s^{-1}$	Visc. (mPa.s) 15°C $10s^{-1}$	Visc. (mPa.s) 15°C $100s^{-1}$
IM-5 Wakashio	Fresh 250°C+	0 12	100 89	0.908 0.917	84* -	9* 15*	6402 18 613	1954 6258	1199 4977	582 1682
VLSFO-1	Fresh	0	100	0.989	109[a]	9	71 236	28 399	5550**	3948**
VLSFO-2	Fresh 250°C+	0 7.9	100 93	0.990 0.996	100[a] NA	3 12	132 46 878 540	77 638 136 400	19 450* 106 130**	16 507** 68 041**
ULSFO-1	Fresh 250°C+	0 14.6	100 86	0.872 0.878	75 112	24 30	13 106 77 782	- -	4300** 33 169**	- -
ULSFO-2	Fresh 200°C+ 250°C+	0 2.7 5.1	100 98 96	0.917 0.920 0.922	85[b] 87[c] 89[c]	24 27 30	111 800 205 220 350 250	21 017 - -	33 564** 53 251** 91 496**	5986** 9903** 14 826*

a: Certificate of Analysis (COA) b: Data from SL Ross; c: Estimated data:Data from Cedre ** Viscosities measured at 13 °C.*
-: No viscosity at the specified temperature

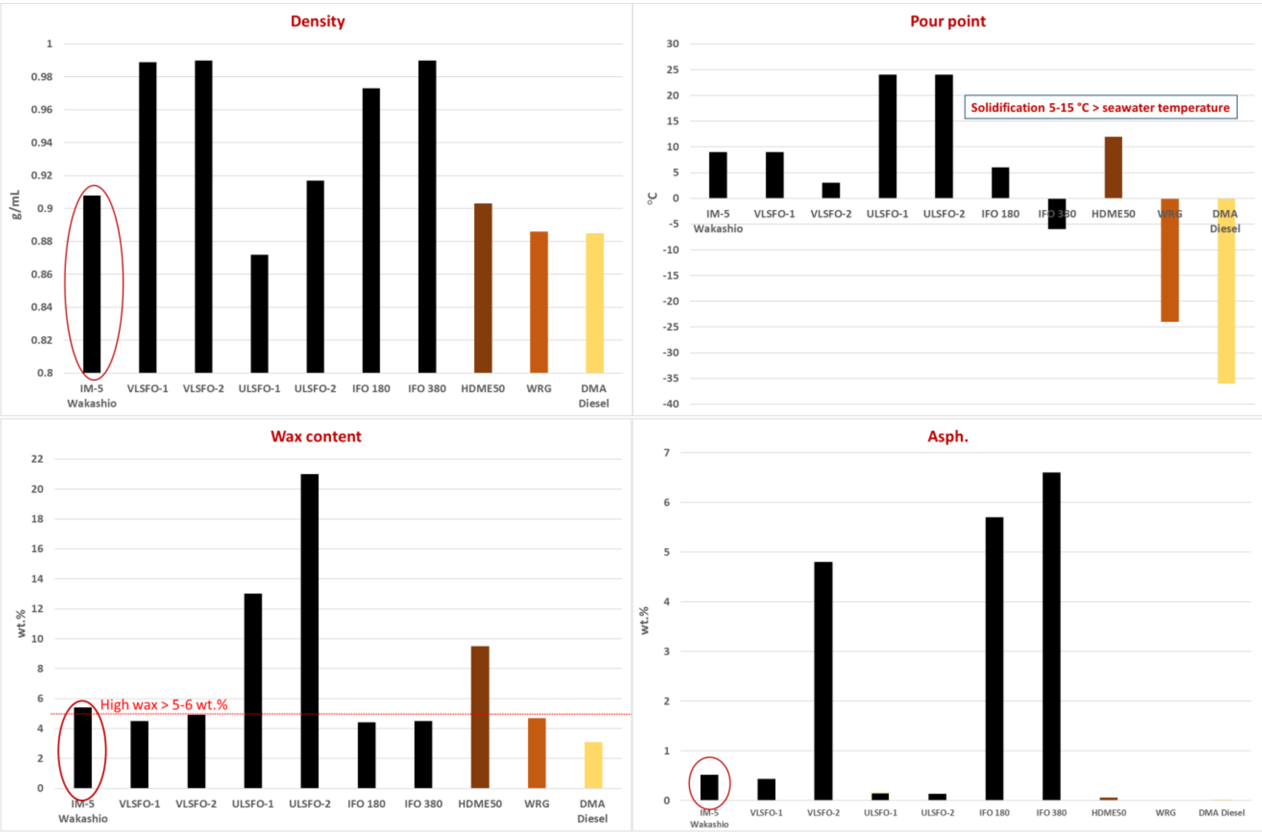


Figure 4-5 *Comparison of IM-5 Wakashio with previous tested fuel oils at SINTEF.*

Temperature-sweep (viscosity) with a temperature range from 50-0 °C was measured at shear rate $10s^{-1}$ (Figure 4-6). The temperature-sweep of IM-5 Wakashio is compared with other VLSFOs, ULSFOs, and IFO-380 and DMA diesel. The temperature-sweeps clearly show that the viscosities of all the oils increase significantly with decreasing temperature (except the DMA diesel). However, IM-5 Wakashio exhibits the lowest viscosity in the temperature range of 2-15 °C among the residual fuel oils, reflecting the chosen seawater temperatures for testing. IM-5 Wakashio has a viscosity of ~33 mPa.s at 50 °C that is in the same range as previous intermediate fuel oils (IFO-30), and in the lower range than previous tested residual fuels oils (except from ULSFO-1). However, IM-5 Wakashio has significantly higher viscosity than DMA-distillate marine fuel oil.

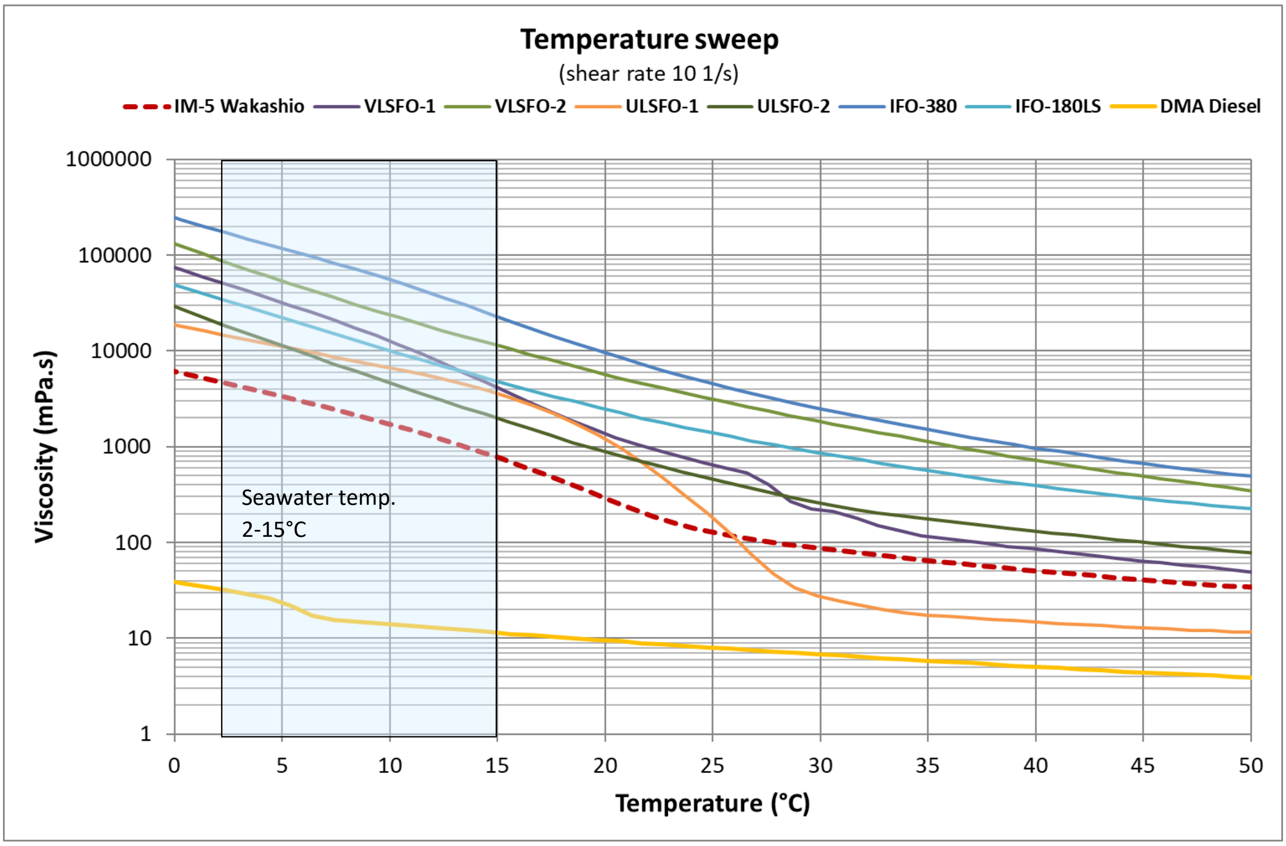


Figure 4-6 Temperature-sweep measurements (viscosities) of IM-5 Wakashio in comparison with other marine fuel oils.

4.6 Emulsifying properties

The emulsifying properties were studied using the rotating cylinders method as detailed in Hokstad et al. 1993. The parameters for kinetics (rate of water uptake) and maximum water uptake were studied to define the emulsification characteristics of oils selected for this study as described in Appendix A.

The emulsification testing was carried out on the non-evaporated (i.e. fresh) oil, in addition to the evaporated residue (250°C+). Experiments of the fresh oil and residue were made to produce data for stability, viscosity, maximum water uptake, and the effectiveness of the emulsion breaker application. Four cylinders of fresh oil /residue of each oil were prepared to study in parallel: stability testing and water uptake (rotating cylinder 1); viscosity / water uptake (rotating cylinder 2); effectiveness of emulsion breaker at dosage of 500 ppm (wt.%) (rotating cylinder 3); and effectiveness of emulsion breaker at dosage of 2000 ppm (wt.%) (rotating cylinder 4).

4.6.1 Water-in-oil emulsification

Figure 4-7 and Figure 4-8 show images from the emulsification testing of IM-5 Wakashio after 24-hour rotating time for the fresh oil and 250°C+ residue, at 2 and 15 °C. Table 4-4 and Table 4-5 show the maximum water uptake, viscosity, and stability at 2 and 15 °C, respective. The maximum water uptake was lower at 2 °C compared with 15 °C (fresh oil; 55 vol% vs. 65 vol.%, respective). However, the results show higher viscosities at 2 °C (11 054 mPa.s,) than 15 °C (5503 mPa.s), and higher viscosity for the emulsified 250 °C+ residue compared with the emulsified fresh oil at both temperatures. It should be emphasized that the water uptake particularly at low temperature (2 °C) can be underestimated in rotating cylinders (bench-scale) for high viscous fuel oils, and meso-scale flume testing (section 5) was conducted as a supplement to achieve the most reliable weathering data for oil weathering predictions (section 6).

The emulsion stability of IM-5 Wakashio was studied by quantifying the amount of water released from the emulsion for 24 hours settling time. The emulsions at both temperatures were very stable, i.e. no or minor release of free-water were measured upon standstill in 24 hours after the rotation time.

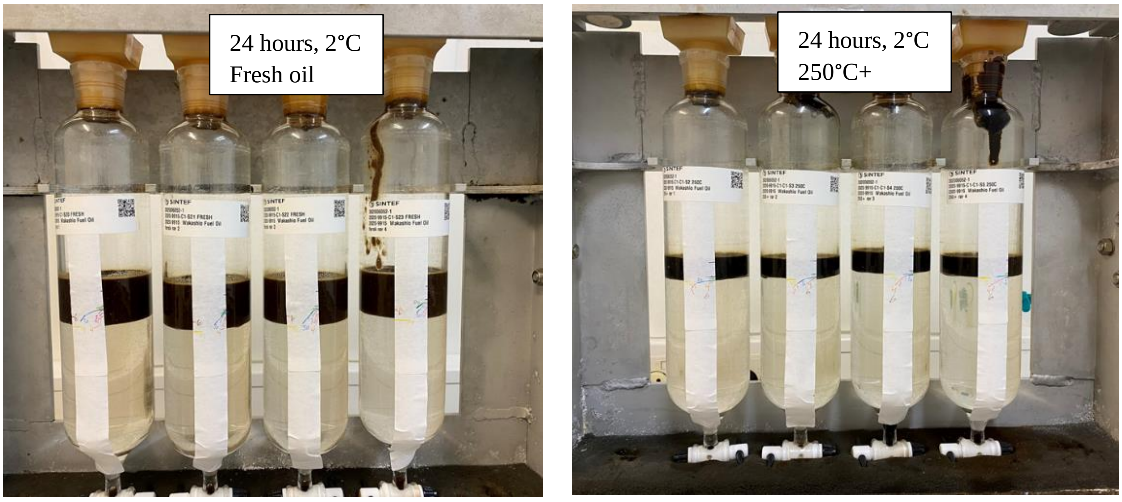


Figure 4-7 The rotating cylinders of water-in-oil emulsion of IM-5 Wakashio after 24 hours of rotation at 2 °C. Left: Fresh oil. Right 250°C+ residue.

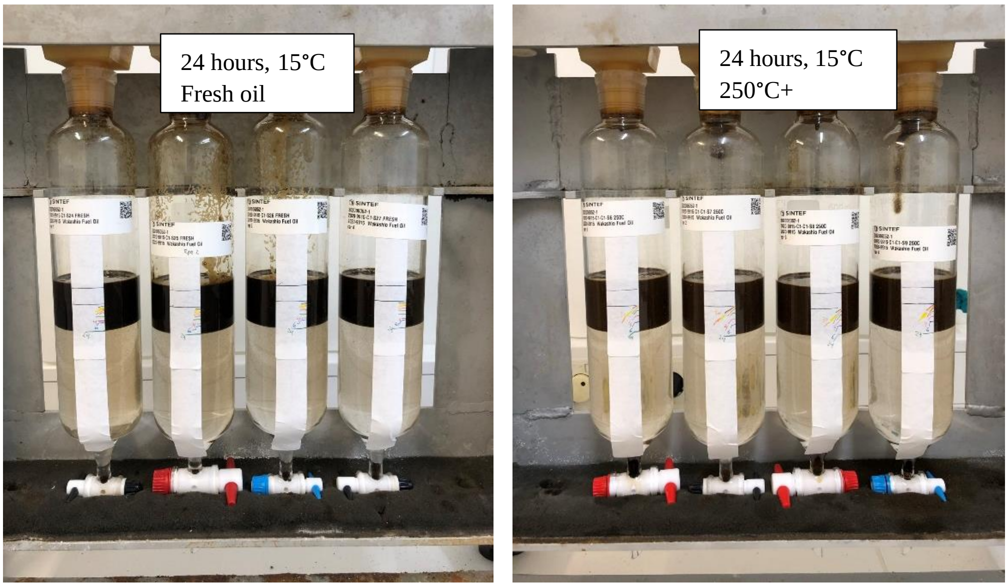


Figure 4-8 The rotating cylinders of water-in-oil emulsion of IM-5 Wakashio after 24 hours of rotation at 15 °C. Left: Fresh oil. Right 250°C+ residue.

Table 4-4 Emulsification at 2 °C of IM-5 Wakashio.

Water-in-oil emulsification	**Fresh oil, 2 °C**	**250°C+ residue, 2 °C**
Max. water content (vol.%)	55	12
Viscosity (mPa.s/ $10s^{-1}$)	11 054	17 060
Stability ratio *	1	1

**Stability ratio of 1 implies a totally stable emulsion for 24 hours settling. Stability ratio of 0 (zero) implies a totally unstable emulsion; all the water is settled out for 24 hours settling.*

Table 4-5 Emulsification at 15 °C of IM-5 Wakashio.

Water-in-oil emulsification	**Fresh oil, 15 °C**	**250°C+ residue, 15 °C**
Max. water content (vol.%)	65	65
Viscosity (mPa.s/ $10s^{-1}$)	5 503	15 258
Stability ratio*	0.97	0.95

**Stability ratio of 1 implies a totally stable emulsion for 24 hours settling. Stability ratio of 0 (zero) implies a totally unstable emulsion; all the water is settled out for 24 hours settling.*

4.6.2 Effectiveness of emulsion breaker

Generally, the effectiveness of emulsion breakers can be important in a mechanical recovery situation because separating the oil from water enables optimal use of available storage facilities/tankers.

At 2 °C, there was observed a negligible or no effect with use of emulsion breaker (2000 ppm) to release water from the emulsion prepared from the fresh oil and 250°C +residue.

At 15 °C, about 87 % dehydration (release of water) was quantified on the emulsified fresh oil with emulsion breaker (2000 ppm), and similar about 47 % dehydration on the emulsified 250°C+ residue. The lower concentration of 500 ppm had however minor effect on both emulsions. The use of emulsion breaker (high dosage, minimum 2000 ppm) could be an option at 15 °C to reduce the total volume emulsion in storage tanks.

5 Meso-scale flume study results

The weathering behaviour of IM-5 Wakashio was studied in the meso-scale flume basin at SINTEF oil spill laboratory. The experiments in the flume were conducted at 2 and 15 °C. At 15 °C, sunlight was simulated with use a solar simulator during the experiment. This report includes the main findings and a selection of images from the two experiments. A description of the meso-scale flume is given in Appendix A. In addition, project memos (Sørheim et al. 2021) were outlined with extent descriptions and images from the experiments at 2 and 15 °C. A total of 8-9 litres of IM-5 Wakashio were applied onto the flume surface water in each experiment. The fuel oil was weathered for 7 days (168 hours) under standard breaking waves conditions at both temperatures, before the experiments were ended by repeating application of dispersant (*in-situ*). The weathering duration of each experiment was decided with the NCA to harmonize with the standard test condition at Cedre. Dasic Slickgone NS was used as dispersant that also agreed with the NCA. The meso-scale flume data were used in combination with the bench-scale weathering data (section 4) as input to the SINTEF Oil Weathering Model (OWM) to give the most reliable weathering predictions (see section 6 for predictions). This chapter shows the flume data plotted together with the weathering predictions of evaporative loss, water content and emulsion viscosity at 2 and 15 °C.

5.1 Meso-scale flume experiment at 2 °C

An overview of the results from the meso-scale flume testing of IM-5 Wakashio (2 °C) is given in Table 5-1.

Table 5-1 Results from the meso-scale flume testing of IM-5 Wakashio at 2 °C.

Sample no.	Time (hours)	Water-content (vol%)	Evap. (wt.% %)	Viscosity (mPa.s) $10s^{-1}$	Oil-in-water- (ppm)	Oil-in-water % of original amount	FET-test
1	0.5	6	0.0	7211	0.4	0.0	n.a
2	1	15	0.1	8910	n.a	n.a	Good dispersible
3	2	28	0.6	9785	n.a	n.a	Good dispersible
4	4	44	1.1	12160	n.a	n.a	Good dispersible
5	6	54	1.9	13450	n.a	n.a	Reduced dispersible
6	24	64	3.8	25620	n.a	n.a	Poor dispersible
7	48	66	5.5	28870	n.a	n.a	Poor dispersible
8	72	66	5.6	33580	n.a	n.a	n.a
9	120	67	6.8	35740	n.a	n.a	n.a
10	168	66	7.4	39960	1.3	0.1	Poor dispersible
1. Application of dispersant: 401.6 g Dasic NS (DOR = 6.04 wt.%)							
3 min. disp 1					159	0.9	n.a
10 min. disp 1					24	1.5	n.a
30 min. disp 1		60	6.7	21670	25	1.4	Poor dispersible
2. Application of dispersant: 354.7 g Dasic NS (DOR= 5.34 wt. %)							
3 min. disp 2					44	2.7	n.a
10 min. disp 2					68	4.0	n.a
30 min. disp 2		56	7.5	14310	71	4.2	Reduced dispersible
3. Application of dispersant: 355.1 g Dasic NS (DOR= 5.34 wt.%)							
3 min. disp 3					93	5.3	n.a
10 min. disp 3					140	8.0	n.a
60 min. disp 3		47	7.9	12930	148	9.1	Reduced /Good dispersible
4. Application of dispersant:304.4 g Dasic NS (DOR= 4.58 wt.%)							
10 min. disp 4		43*	8.2	10560*	758	49	n.a

*Total dispersant: 1415.8 g (DOR 1:5 and DER 1:14), n.a: not analyzed, *sample taken at the end of the experiment.*

Weathering, 2 °C

The results from the meso-scale testing at 2 °C show consistent trends as increasing viscosities and water contents of the emulsified oil by time. After 0.5 hour the viscosity was to 7211 mPa.s but increased up to 39 960 mPa.s after 7 days of weathering (the viscosities were measured at shear rate $10s^{-1}$). Similarly, the water content was low 6 vol. % at 0.5 hour but increased subsequently to 66 vol. % after 7 days of weathering. As expected, the evaporation loss was relatively low during the experiment and ended with 7.4 wt.% after one week of weathering, which is also reasonable based on the TBP (Figure 4-2) of IM-5 Wakashio. The oil-in-water concentrations were very low (0-1.3 ppm, parts per million) from the first sampling to the last sampling after 7 days of weathering, meaning that most of the oil (emulsion) remined on the surface, and natural dispersion was therefore negligible. The FET (field dispersants effectiveness test) was used to test the potential for dispersant use on the surface oil emulsion. The FET-test showed that the weathered oil was good dispersible up to four hours, reduced dispersible after 6 hours, and found poor dispersible after 24 hours.

In-situ dispersant application, 2 °C

After 7 days of weathering, the meso-scale flume experiment was finalized by repeating *in-situ* dispersant application strategy on the remaining surface oil-emulsion. Due to high surface thickness (2-4 cm) of the emulsified oil, a total of 4. rounds (repeats) of dispersant were applied with 401.6 g, 354.7 g, 355.1 g and 304.4 g respectively (cumulative amounts 1415.8 g). This gives a cumulative DOR (dispersant to oil ratio) of about 1:5 and DER (dispersant to emulsion ratio) of 1:14. After the 1. round with dispersant treatment the water content of the emulsion decreased from 66 to 60 vol.% and the viscosity was reduced from 39 960 mPa.s to 21 670 mPa.s. Oil in the water phase was measured to about 25 ppm 30 min. after treatment. After the 2. and 3. rounds with dispersant application, the viscosities were reduced to 14 310 and 12 930 mPa.s, respectively, and the water contents were in the range of 56-47 vol. %. The FET-test indicated that the oil (emulsion) was reduced dispersible after the 2. round, but was good dispersible after the 3. round with dispersant application. The oil-in-water was measured to 140-148 ppm after the 3. round with dispersant application. After the 4. round, the viscosity had decreased to 10 560 mPa.s and water content was reduced to 43 vol.%. The concentration of oil in the water column was subsequently increased to 758 ppm, indicating a significant effect to break up the surface emulsion after the fourth round with dispersant application.

From an operational point of view, the flume experiment at 2 °C shows that IM-5 Wakashio is not dispersible after one-week at sea. IM-5 Wakashio is however expected to be good dispersible during the first hours (4-6 h) after a spill at 2 °C, based on the simplified FET-test.

Mass balances, 2 °C

Figure 5-1 shows the estimated mass balance after one-week weathering before application of dispersant. Figure 5-2 shows the mass balance of dispersed oil into the water column after application of dispersant (Dasic Slickgone NS). About 62 % of available oil was dispersed in the water column after the fourth round of dispersant application.

Flume data compared with oil weathering predictions, 2 °C

Prediction of evaporative loss, water uptake and viscosity are shown in Figure 5-3 together with the flume data. By combining results from the bench-scale study and meso-scale flume experiment the predicted weathering properties and flume data harmonized well at 2 °C.

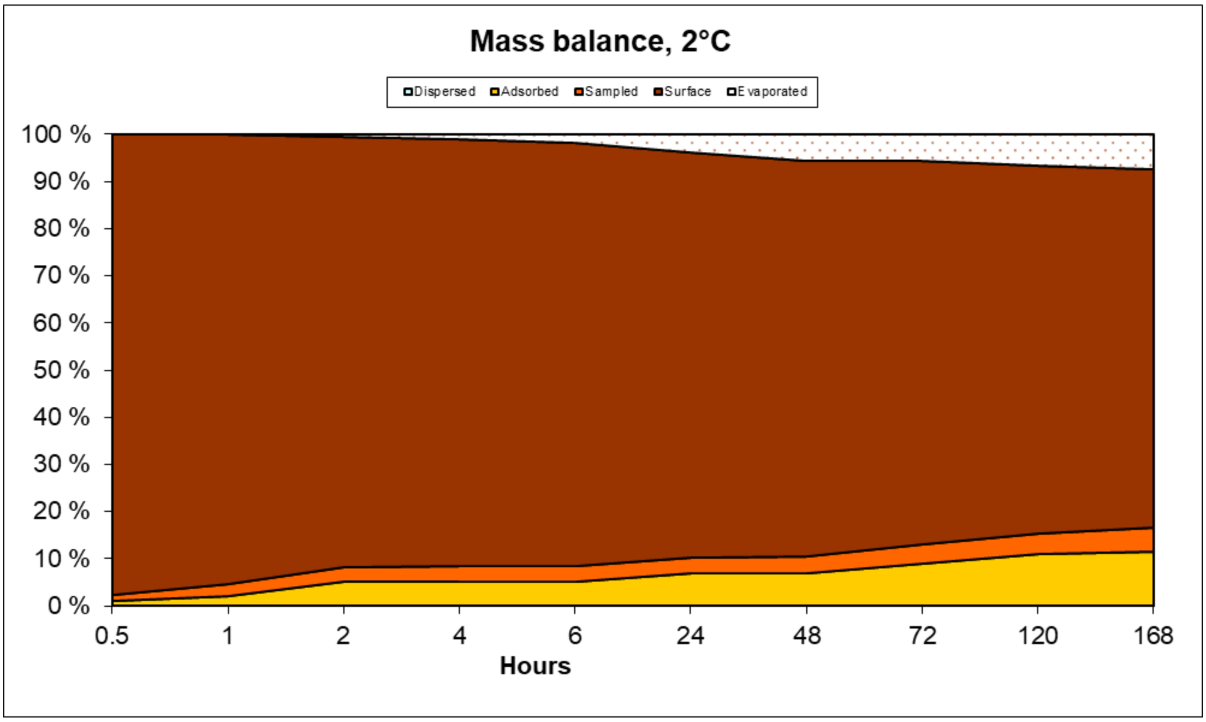


Figure 5-1 Mass balance of IM-5 Wakashio in the meso-scale flume basin at 2 °C, before dispersant application.

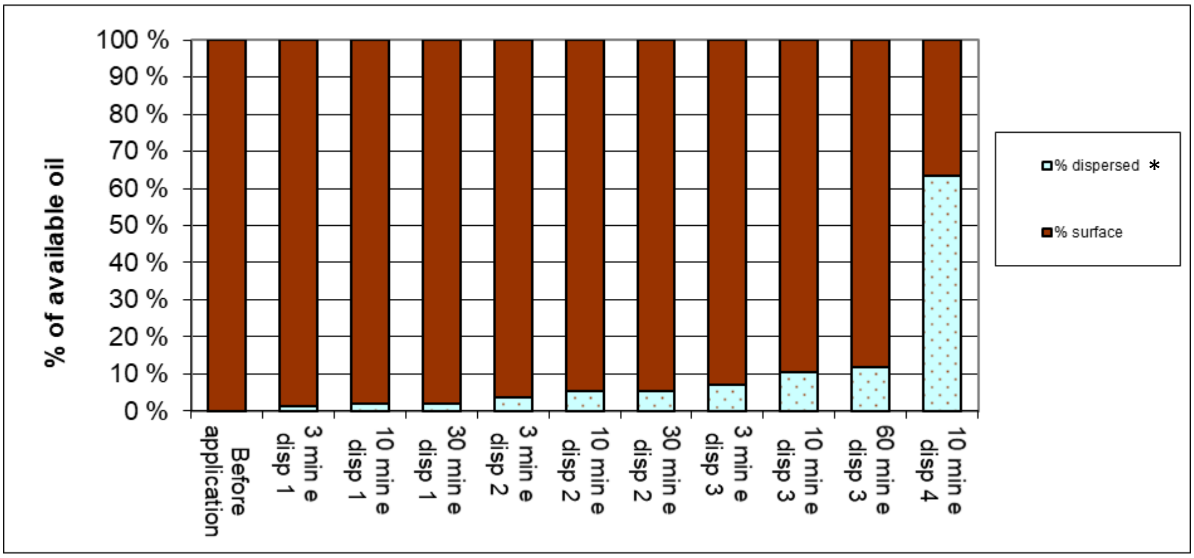
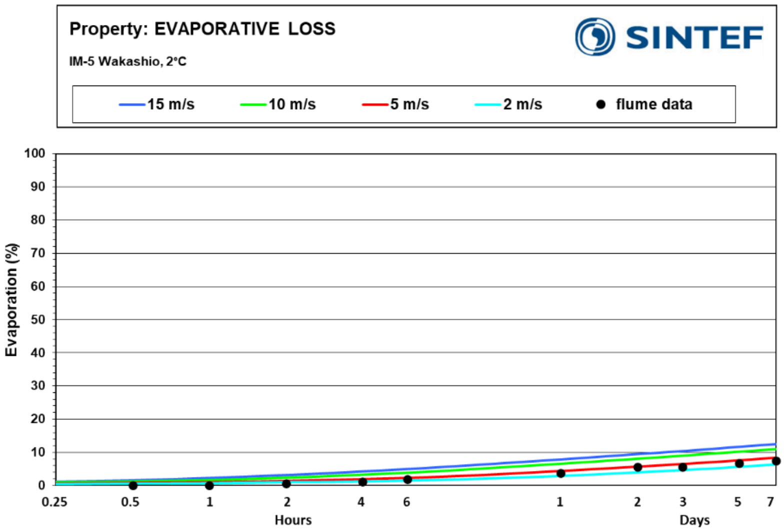
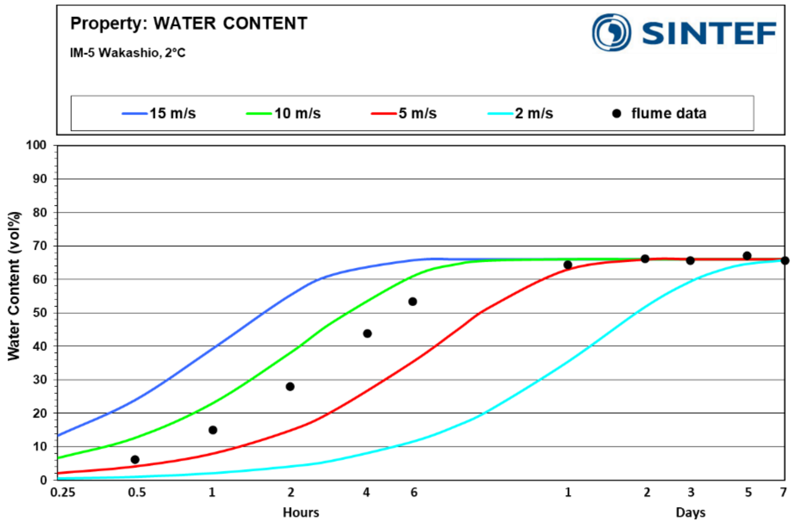


*Figure 5-2 Mass balance of IM-5 Wakashio after application of Dasic Slickgone NS as percentage of available surface oil emulsion at 2 °C. *Large droplets/lumps in the water phase (not small, dispersed oil droplets).*





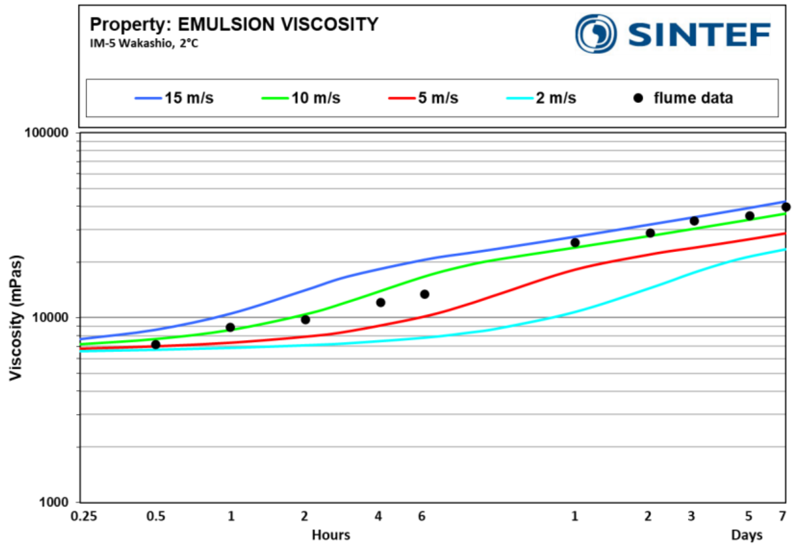


Figure 5-3 *Predicted evaporative loss (above), water uptake (middle) and viscosity (below) and the dots that present the experimental data from the meso-scale flume experiments of IM-5 Wakashio at 2 °C.*

Images, 2 °C

Examples of photo-documentation straight after the application of IM-5 Wakashio onto the water surface and after 7 days of weathering (Figure 5-4 and Figure 5-5). Example after the fourth round with dispersant application is shown in Figure 5-6.

Figure 5-4 Application of IM-5 Wakashio onto the water surface at 2 °C.

Figure 5-5 Surface at 168 hours (7 days) of IM-5 Wakashio at 2 °C. Water content: 66 vol.%. Viscosity: 39 960 mPa.s. Images taken prior to dispersant application.

Figure 5-6 Surface 10 minutes after 4. round of application of dispersant on IM-5 Wakashio at 2 °C. The structure in the emulsion is broken and the effect of the dispersant is apparent. About 49 % of the original amount of oil was dispersed into the water phase.

5.2 Meso-scale flume experiment at 15 °C

An overview of the results from the meso-scale flume testing of IM-5 Wakashio at 15 °C is given Table 5-1.

Table 5-1 Results from the meso-scale flume testing of IM-5 Wakashio at 15 °C.

Sample no.	Time (hours)	Water-content (vol%)	Evap. (wt.% %)	Viscosity (mPa.s) $10s^{-1}$	Oil-in-water-(ppm)	Oil-in-water % of original amount	FET-test
1	0.5	43	1.7	2036	21.2	1.1	n.a
2	1	58	2.7	2716	n.a	n.a	Good dispersible
3	2	67	3.4	4241	n.a	n.a	Good dispersible
4	4	73	3.7	4420	n.a	n.a	Good / Reduced dispersible
5	6	72	5.9	5769	n.a	n.a	Reduced dispersible
6	24	81	8.1	11102	n.a	n.a	Reduced /poor dispersible
7	48	81	9.6	14556	n.a	n.a	Poor dispersible
8	72	78	10.6	15232	n.a	n.a	Poor dispersible
9	120	80	12.0	16617	n.a	n.a	Poor dispersible
10	168	80	13.1	22083	3.1	0.2	Poor dispersible
1. Application of dispersant: 352.7g Dasic NS (DOR = 5.1 wt.%)							
3 min. disp 1		81			14	0.7	n.a
10 min. disp 1		81			24	1.3	n.a
30 min. disp 1		77		18345	33	1.9	Poor dispersible
2. Application of dispersant: 352.8g Dasic NS (DOR= 5.1 wt. %)							
3 min. disp 2		77			51	3.0	n.a
10 min. disp 2		77			86	5.1	n.a
30 min. disp 2		77			106	5.8	n.a
60 min. disp 2		74		7077	127	6.8	Reduced dispersible
3. Application of dispersant: 334.0 g Dasic NS (DOR= 4.8 wt.%)							
3 min. disp 3		74			161	9.0	n.a
10 min. disp 3		74			176	10.5	n.a
30 min. disp 3		74		10369*	232	13.6	Reduced / good dispersible

*Total dispersant: 1039.5 g (DOR 1:7 and DER 1:33), n.a: not analyzed, *= sample taken at the end of the experiment.*

Weathering, 15 °C

The results from the meso-scale testing at 15 °C show consistent trends as increasing viscosities and water contents of the emulsified oil by time. After 0.5 hour the viscosity was to 2036 mPa.s but increased up to 22083 mPa.s after 7 days of weathering (the viscosities were measured at shear rate $10s^{-1}$). Similarly, the water content was 43 vol. % at 0.5 hour but increased subsequently to 80 vol. % after 7 days of weathering. The evaporation loss was relatively low during the experiment and showed to increase from 1.7 from the start to 13 wt.% after one week of weathering, which is also reasonable based on the TBP (Figure 4-2) of IM-5 Wakashio. The oil-in-water concentration was 21 ppm (parts per million) after 0.5 hour i.e., some larger oil droplets were observed in the water phase, but these were re-surfaced within in short time (1 h). After 7 days the oil-in-water content was only 3.1 ppm, meaning that most of the oil (emulsion) still was remined on the surface, and the natural dispersion was very low. The FET (field dispersants effectiveness test) was used to test the potential for dispersant use on the surface oil emulsion. The FET-test showed that the weathered oil was good dispersible up to four hours, reduced to poor dispersible after 6 hours, and found poor dispersible after 24 hours.

In-situ dispersant application, 15 °C

After 7 days, the flume experiment was finalized by repeating in-situ dispersant application on the remaining surface oil emulsion. Due to the high surface thickness (2-4 cm) of the emulsified oil, a total of three rounds (repeats) of dispersant were applied with 352.7 g, 352.8 g, and 334 g respectively (cumulative 1039.5 g). This gives a cumulative DOR of 1:7, and DER of 1:33. After the 1. round with dispersant treatment the water content of the emulsion decreased from 80 to 77 vol.% and the viscosity was reduced from 22 083 mPa.s to 18 345 mPa.s. Oil in the water phase was measured to about 33 ppm 30 min. after treatment. After the 2 and 3. rounds with dispersant application, the viscosities were reduced to 7 077 and 10 369 mPa.s, respectively, and the water contents were in the range of 77-74 vol. %. The FET-test indicated that the oil (emulsion) was reduced dispersible after the 2. round, but was good /reduced dispersible after the 3. round with dispersant application. The oil-in-water was measured to 232 ppm after the 3. round with dispersant application. A 4. round with application of dispersant was decided not to be performed, but as it is assumed that the emulsion would be broken even more into patches as shown at 2 °C.

From an operational point of view, the flume experiment at 15 °C shows that IM-5 Wakashio is not dispersible after one-week at sea. IM-5 Wakashio is however expected to be good dispersible during the first hours (4-6 h) after a spill at 15 °C, based on the simplified FET-test.

Mass balances, 15 °C

Figure 5-7 shows the estimated mass balance after one-week weathering before application of dispersant. Figure 5-8 shows the mass balance of dispersed oil into the water column after application of dispersant (Dasic Slickgone NS).

Flume data compared with oil weathering predictions, 15 °C

Prediction of evaporative loss, water uptake and viscosity are shown in Figure 5-9 together with the flume data. By combining results from the bench-scale study and meso-scale flume experiment the predicted weathering properties and flume data harmonized well at 15 °C.

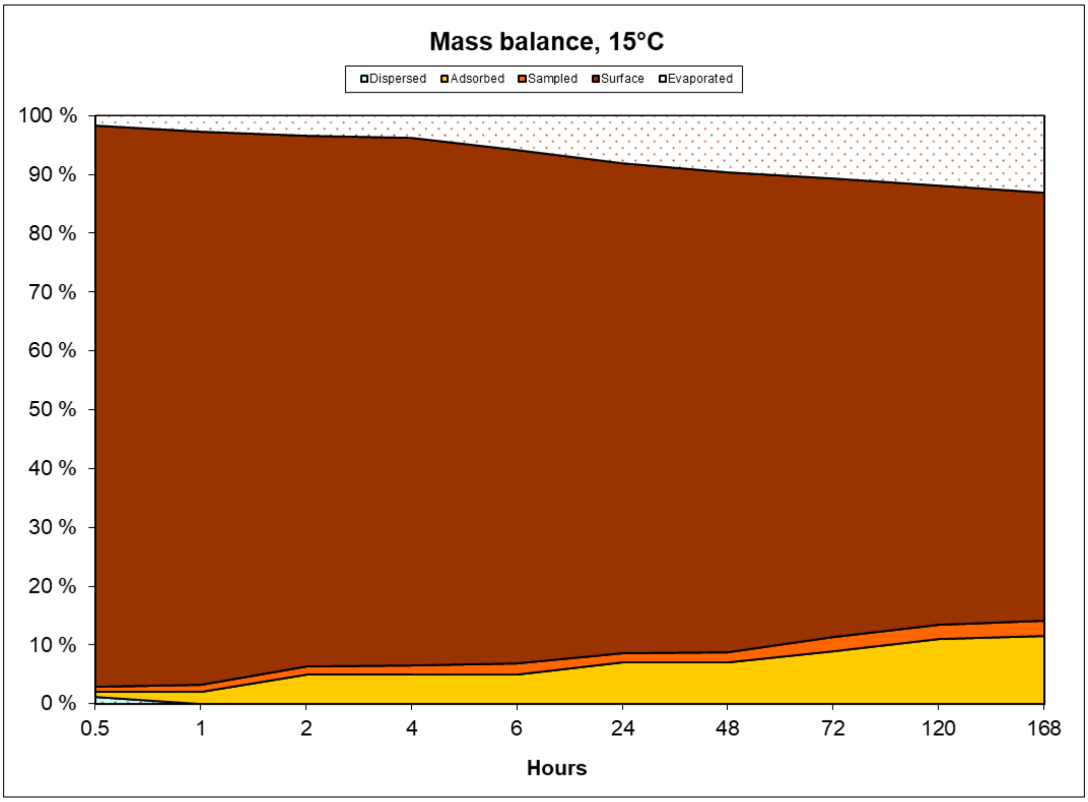


Figure 5-7 *Mass balance of IM-5 Wakashio in the meso-scale flume basin at 15 °C, before dispersant application.*

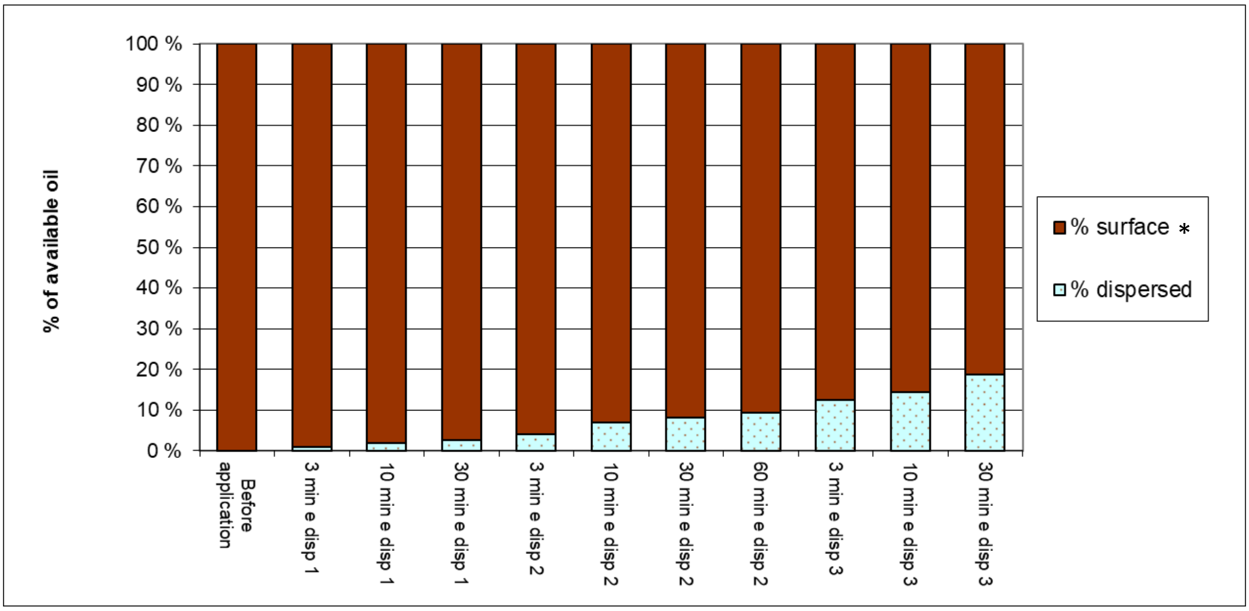


Figure 5-8 *Mass balance of IM-5 Wakashio application of Dasic Slickgone NS as percentage of available surface oil emulsion at 15 °C. *Large droplets/lumps in the water phase (not small, dispersed oil droplets).*

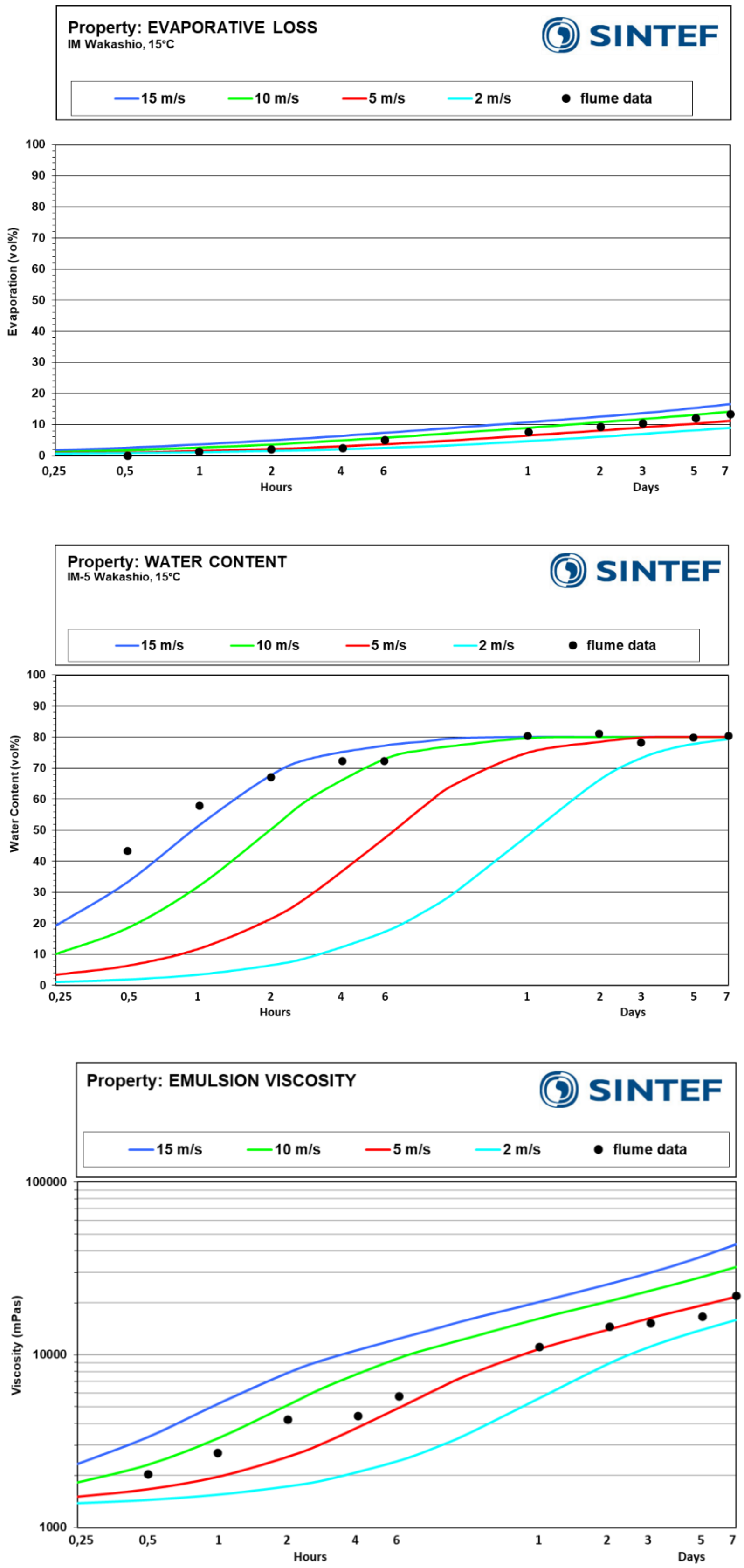


Figure 5-9 *Predicted evaporative loss (above), water uptake (middle) and viscosity (below) and the dots that present the experimental data from the meso-scale flume experiments of IM-5 Wakashio at 15 °C.*

Images, 15 °C

Examples of photo-documentation after application of oil onto the surface and after 7 days of weathering are given in Figure 5-10 and Figure 5-11. Example after the third round with dispersant application is shown in Figure 5-12.

Figure 5-10 Application of Wakashio fuel oil onto the water surface.

Figure 5-11 Surface at 168 hours (7 days). Water content: 80 vol.%. Viscosity: 22 083 mPa.s. Images taken prior to dispersant application.

Figure 5-12 Surface 30 minutes after 3. round of application of dispersant. The structure in the emulsion is broken into patches and the effect of the dispersant is apparent. About 14 % of the original amount of oil was dispersed into the water phase.

5.3 Summary meso-scale flume testing.

- The results from the meso-scale testing of IM-5 Wakashio at 2 and 15 °C showed consistent trends as increasing evaporative loss, viscosities and water contents of the emulsified oil by time.
- At 2 °C, the water content reached 66 vol.% and a viscosity of 39 960 mPa.s.
- At 15 °C, the water content reach 80 vol.% and a viscosity of 22 083 mPa.s.
- The natural dispersion and the evaporative loss were fund to be negligible at both 2 and 15 °C.
- IM-5 Wakashio was shown to be easily dispersible the first 4 to 6 hours after a spill at 2 and 15 °C, based on the simplified FET-test.
- After 7 days of weathering, repeated application of dispersant (Dasic Slickgone NS) 3-4 times and large amounts of dispersant slightly broke up the surface slick into smaller patches and reduced the emulsion viscosity and slightly the water content.
- The results from the meso-scale flume testing were also used as input to the SINTEF Oil Weathering model (OWM) in combination with results from the bench-scale testing to give most reliable predictions of the oil weathering properties of IM-5 Wakashio.
- The flume basin testing in larger scale was found to be an important supplement to bench-scale testing in smaller cylinders for the IM-5 Wakashio fuel oil.

Figure 5-13, Figure 5-14 and Figure 5-15 summaries the increasing evaporative loss, water content and emulsion viscosity of IM-5 Wakashio during 7 days of weathering time in the meso-scale flume at 2 and 15 °C.

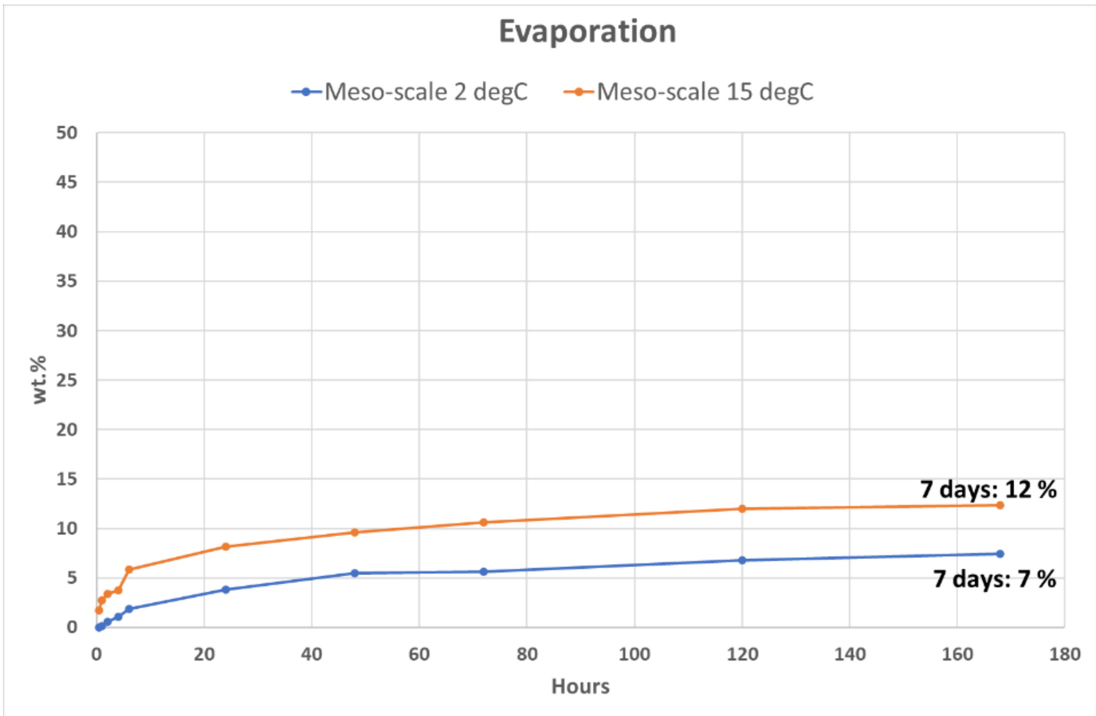


Figure 5-13 Evaporative loss during 7 days of weathering 2 and 15 °C.

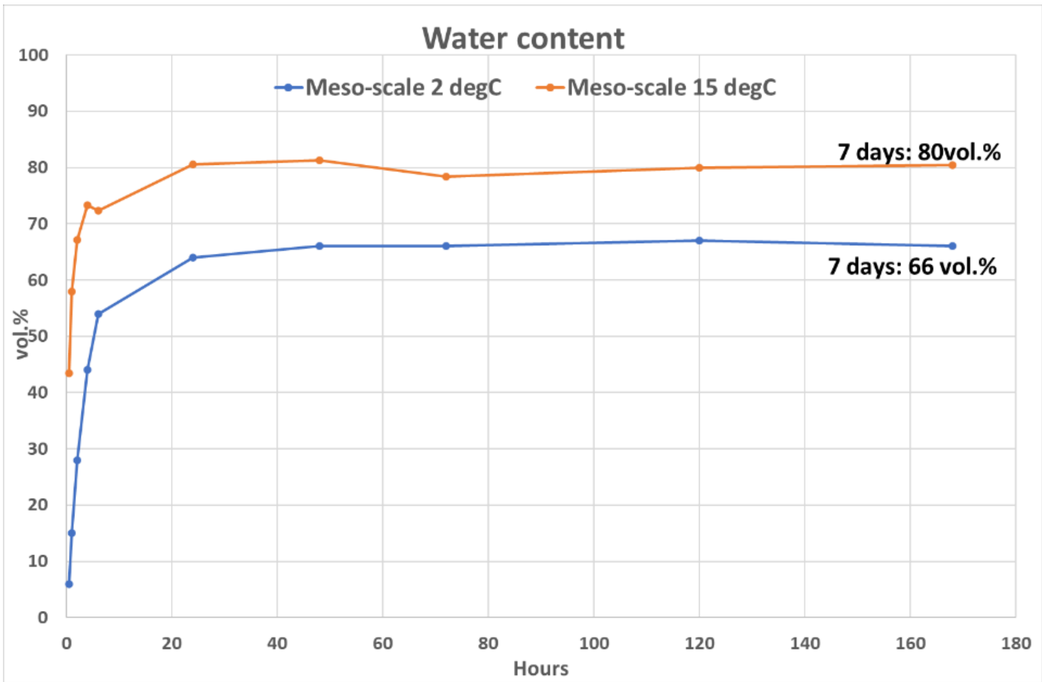


Figure 5-14 Water content during 7 days of weathering 2 and 15 °C.

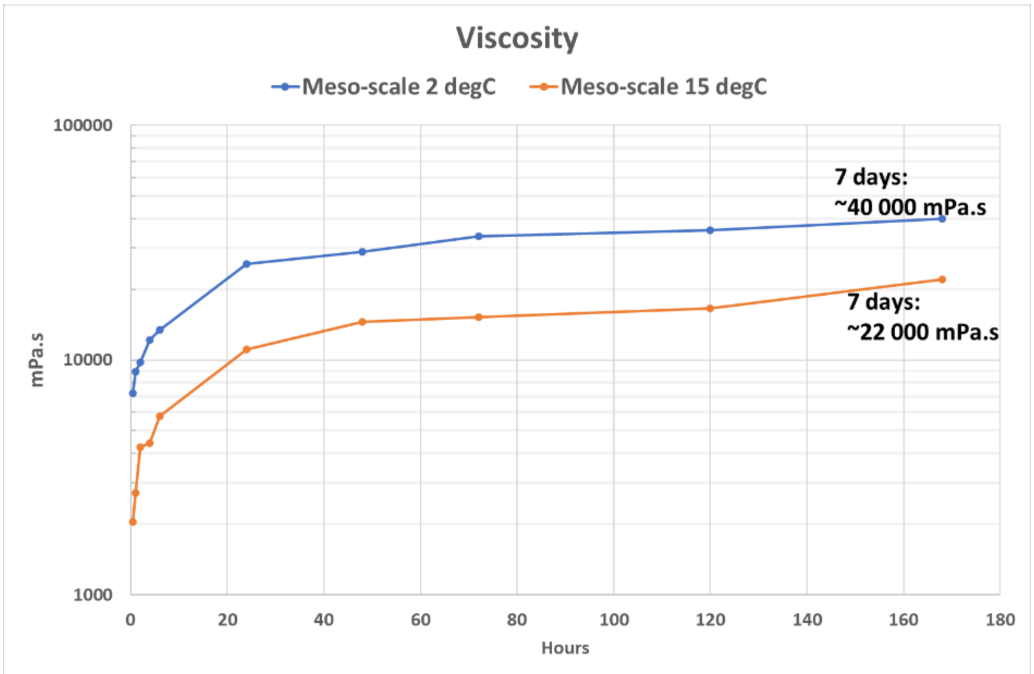


Figure 5-15 Viscosity during 7 days of weathering 2 and 15 °C.

6 SINTEF Oil Weathering Model (OWM)

The SINTEF Oil Weathering Model (OWM) relates oil properties to a chosen set of conditions (oil/emulsion film thickness, wind speeds and sea temperature) and predicts the change rate of the oil's properties on the sea surface with time. The SINTEF OWM is schematically shown in Figure 6-1. The predictions obtained from the SINTEF OWM are useful tools in the oil spill contingency planning related to the expected behaviour of oil on the sea surface, and to evaluate the time window for operational response strategies in a spill operation. The SINTEF OWM is described in more details in e.g. Johansen (1991), and in the user's guide for the model.

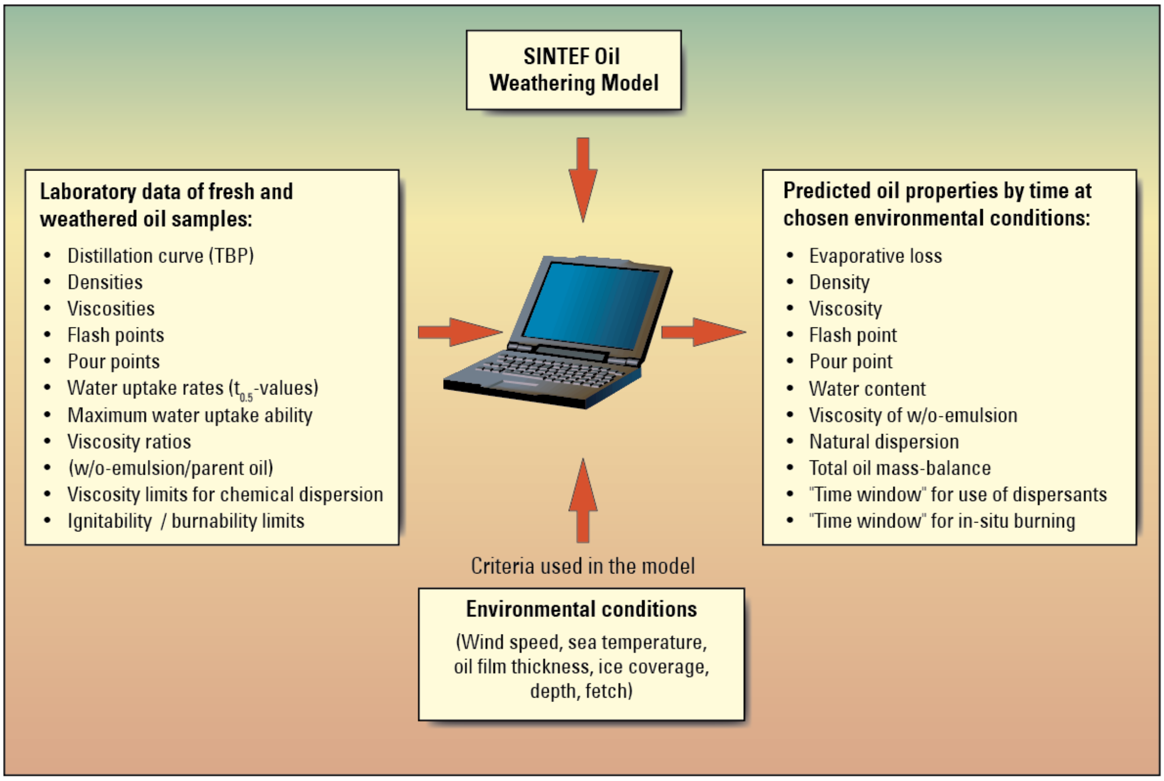


Figure 6-1 ***Schematic input data to the SINTEF OWM and the predicted output oil weathering properties***

6.1 Oil weathering predictions of IM-5 Wakashio

In this report, the presented OWM predictions span a period from 15 minutes to 7 days after an oil spill has occurred. The input laboratory data of IM-5 Wakashio is summarized in Appendix B. A comparison of OWM predictions of IM-5 Wakashio and other residual fuel oils are given in section 6.2.

A standard surface release scenario (80 m^3/h) was chosen to give OWM predictions of the weathering properties of the tested oils. The residual marine fuel oils are expected to reach a terminal oil film thickness of 2 mm from a surface (batch) release. The seawater temperatures chosen for the OWM predictions were 2 and 15 °C reflecting relevant sea temperatures from the laboratory testing. The relationship between the wind speeds and significant wave heights used in the oil weathering predictions are given in Table 6-1. An overview of the scenario OWM parameters is given in Table 6-2.

Table 6-1 Relationship between wind speed and significant wave height used in the SINTEF OWM.

Wind speed [m/s]	Beaufort wind	Wind type	Wave height [m]
2	2	Light breeze	0.1 - 0.3
5	3	Gentle to moderate breeze	0.5 - 0.8
10	5	Fresh breeze	1.5 - 2.5
15	6 – 7	Strong breeze	3 - 4

Table 6-2 Scenario input parameters to SINTEF OWM.

Parameters	Value
Release scenario	Surface release
Release rate (m^3/h)	80
Duration of spill (minutes)	15
Volume spilled (metric tons)	20
Terminal oil film thickness (mm)	2
Wind speed (m/s)	2, 5, 10 and 15
Seawater temperature (°C)	2 and 15
Prediction period	15 min. to 7 days

6.1.1 Predictions at 2 °C

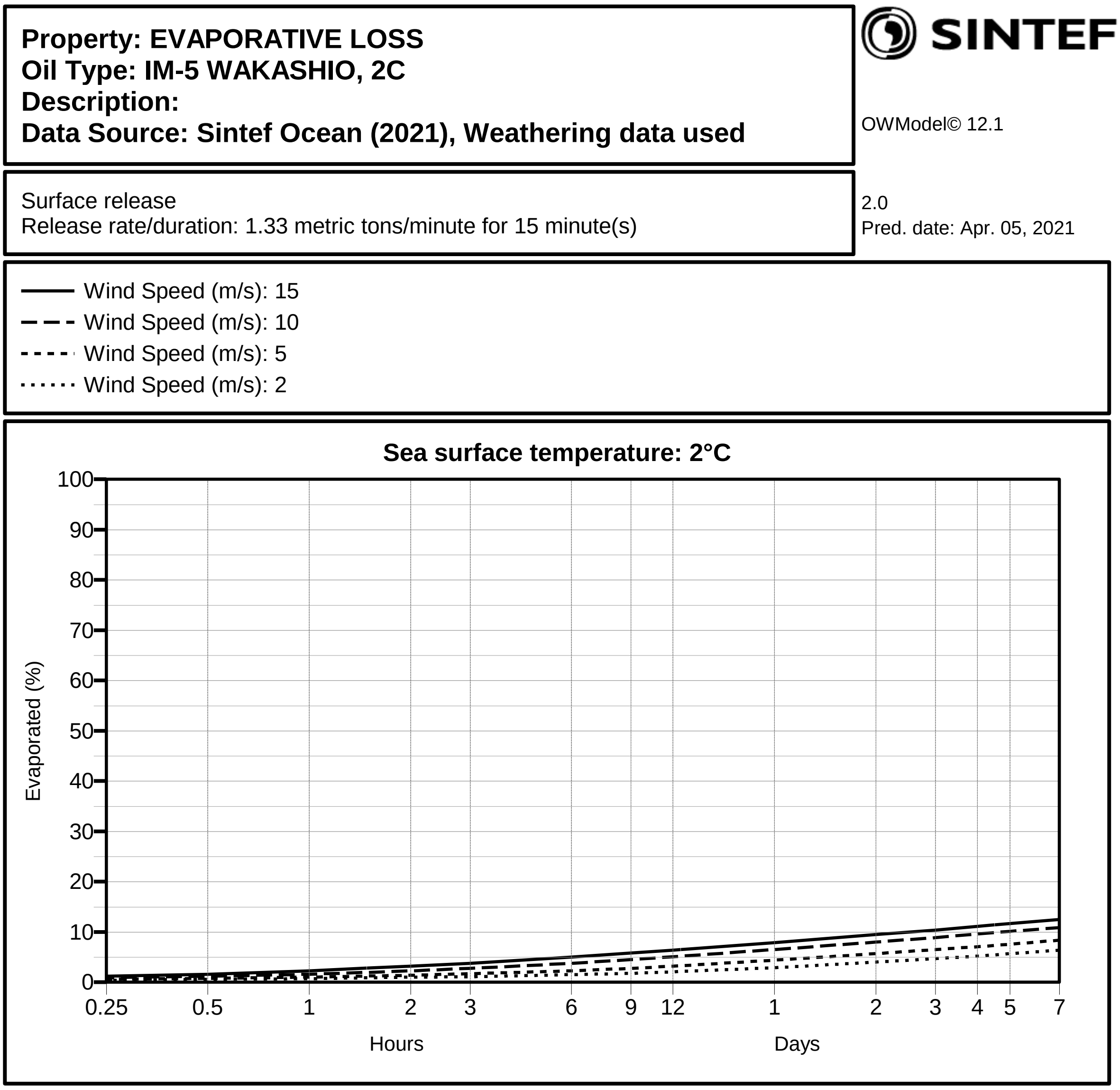


Figure 6-2 Evaporative loss of IM-5 Wakashio predicted at sea temperature of 2 °C.

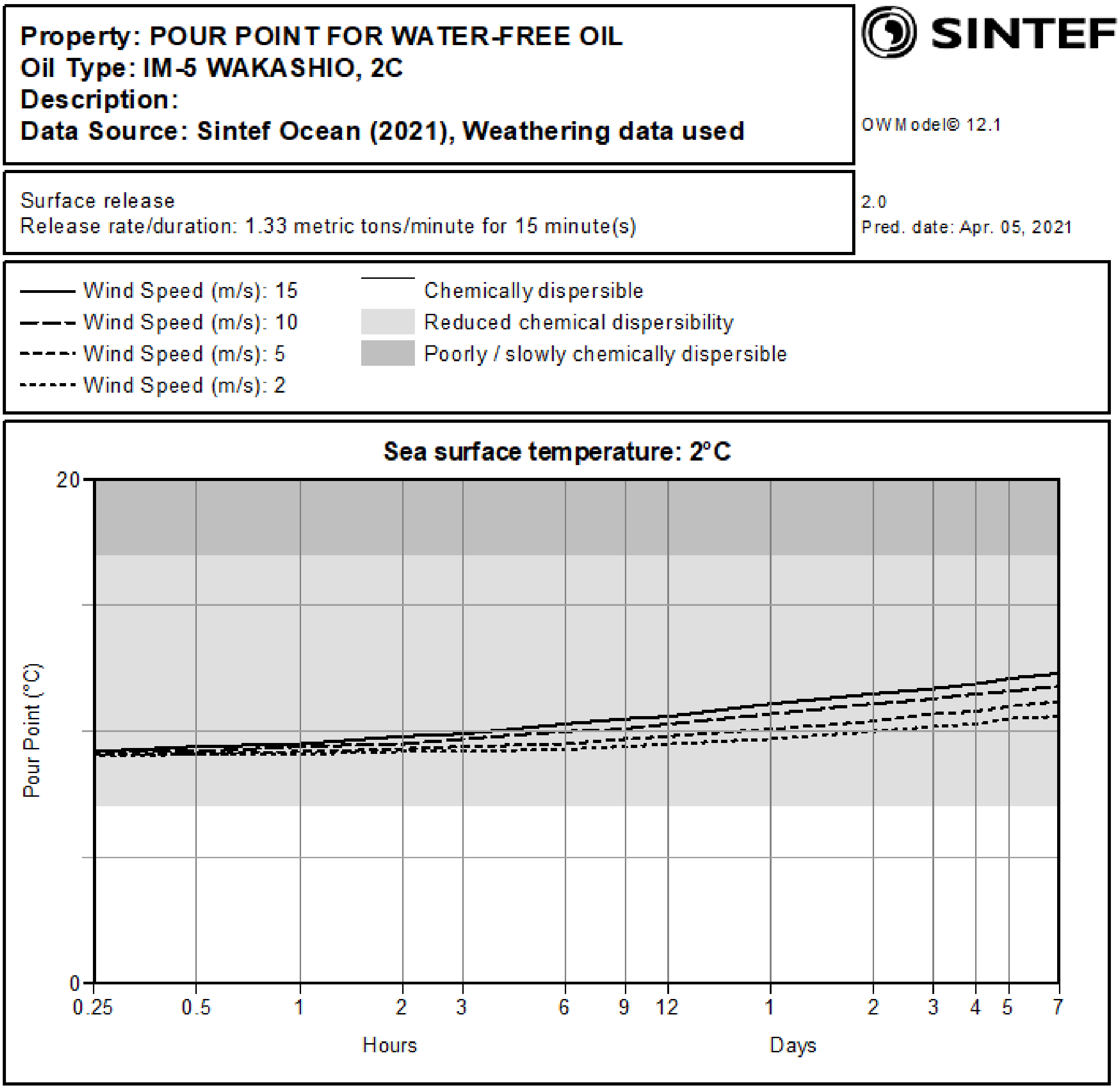


Figure 6-3 Pour point of IM-5 Wakashio predicted at sea temperature of 2 °C.

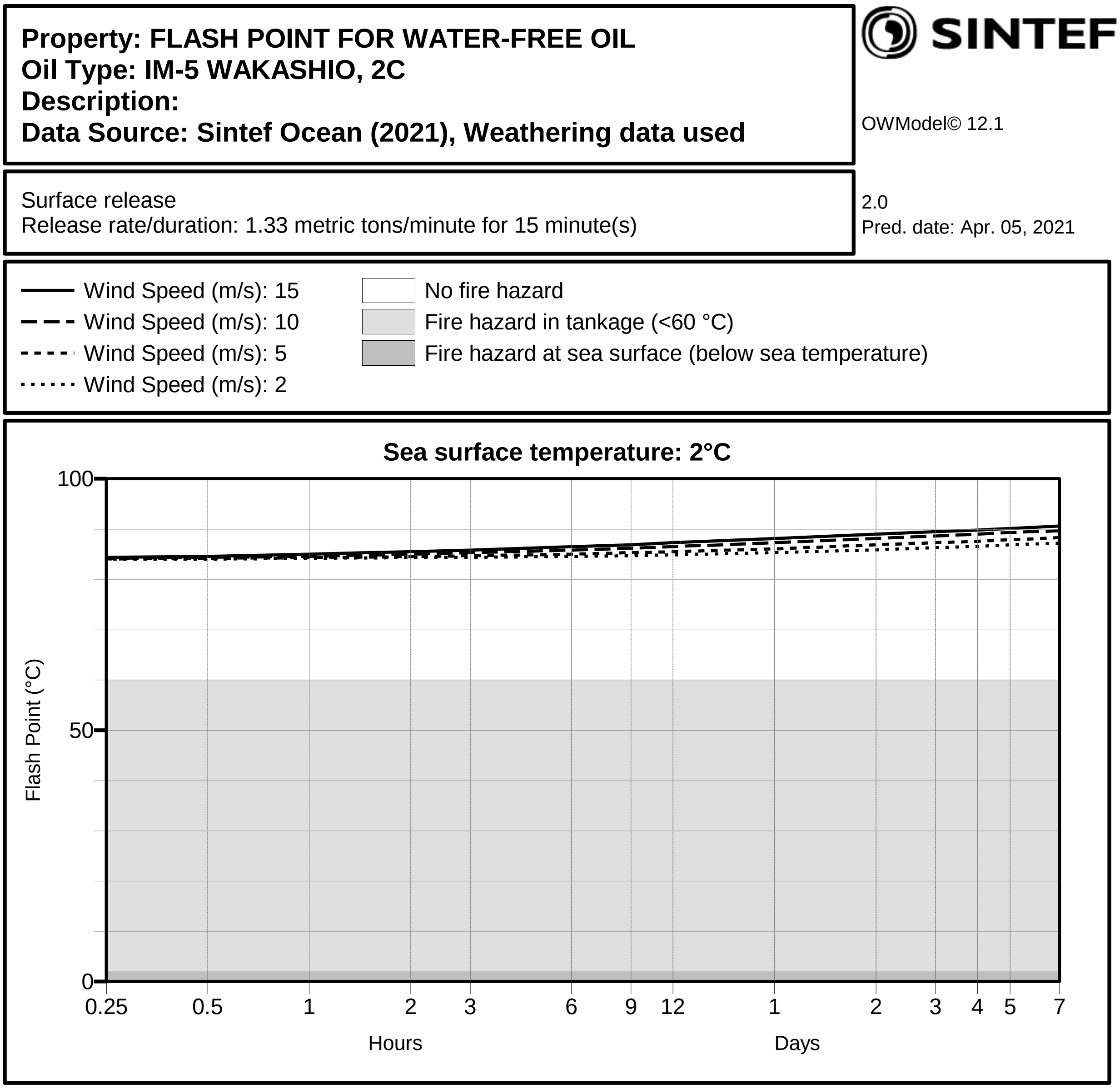


Figure 6-4 Flash point of IM-5 Wakashio predicted at sea temperature of 2 °C.

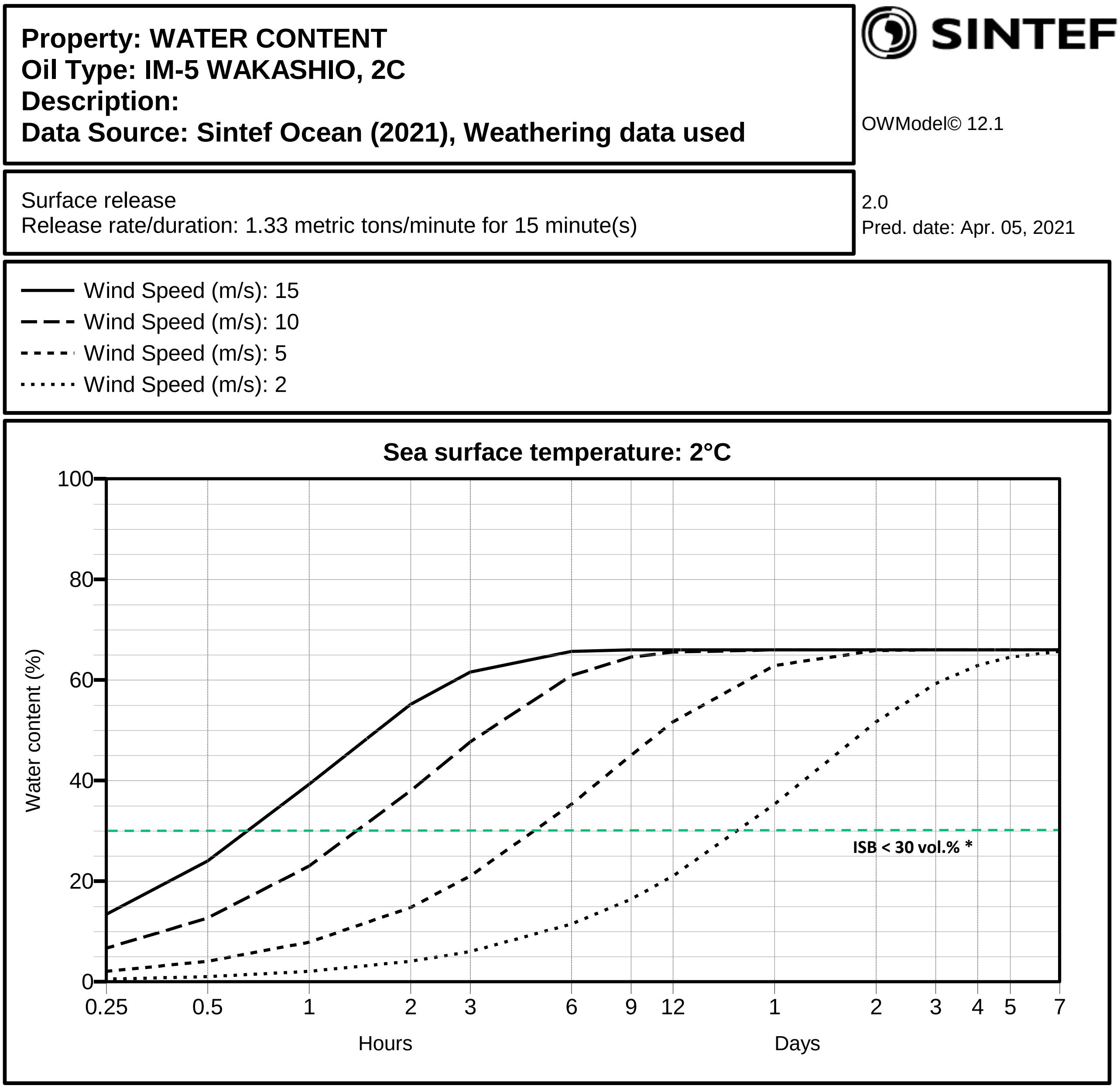


Figure 6-5 *Water content of IM-5 Wakashio predicted at sea temperature of 2 °C. *Rule of thumb: Water content < 30 vol.% for ISB/ignitability.*

Property: VISCOSITY OF EMULSION
Oil Type: IM-5 WAKASHIO, 2C
Description:
Data Source: Sintef Ocean (2021), Weathering data used

OWModel© 12.1

Surface release
Release rate/duration: 1.33 metric tons/minute for 15 minute(s)

2.0
Pred. date: Apr. 05, 2021

Wind Speed (m/s): 15
Wind Speed (m/s): 10
Wind Speed (m/s): 5
Wind Speed (m/s): 2

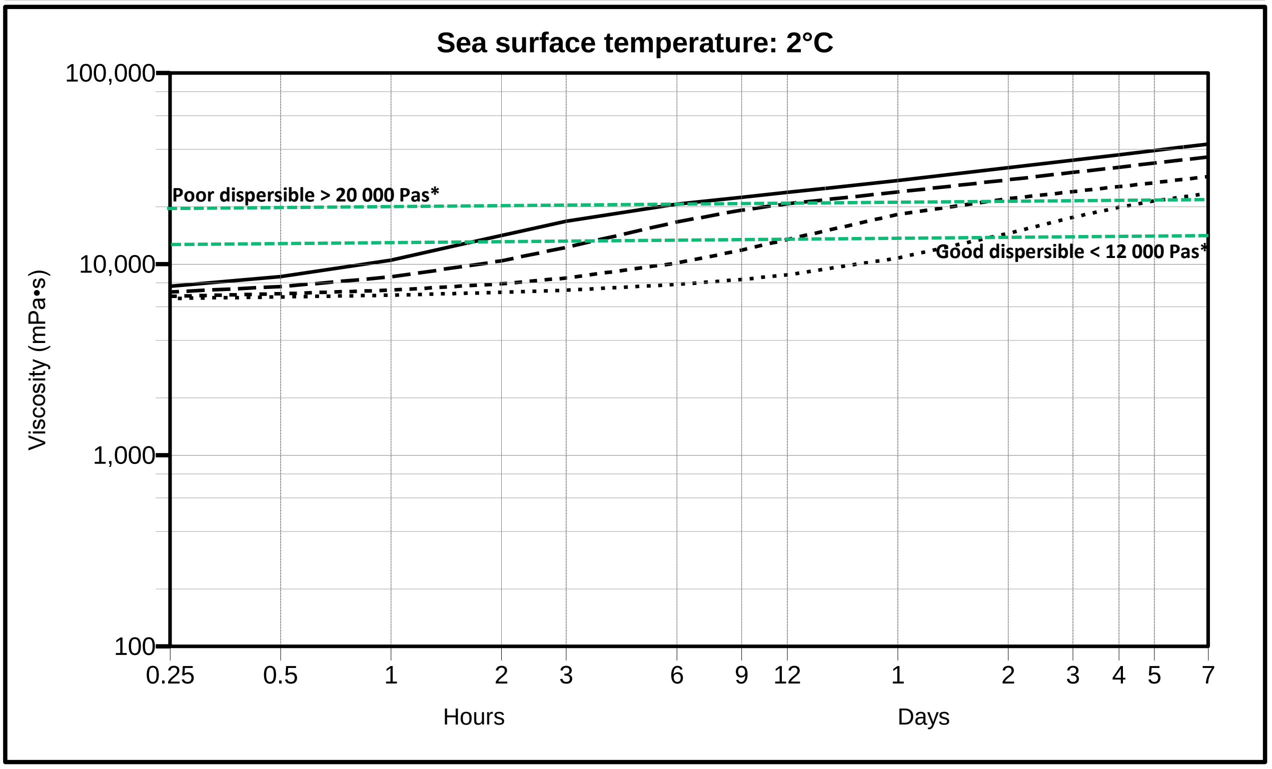


Figure 6-6 *Viscosity of IM-5 Wakashio emulsion predicted at sea temperature of 2 °C. Prediction is based on measurements of emulsions performed at a shear rate of 10 s^{-1}. * Estimated dispersibility limits is based on the FET-test at 2 °C from the meso-scale flume testing.*

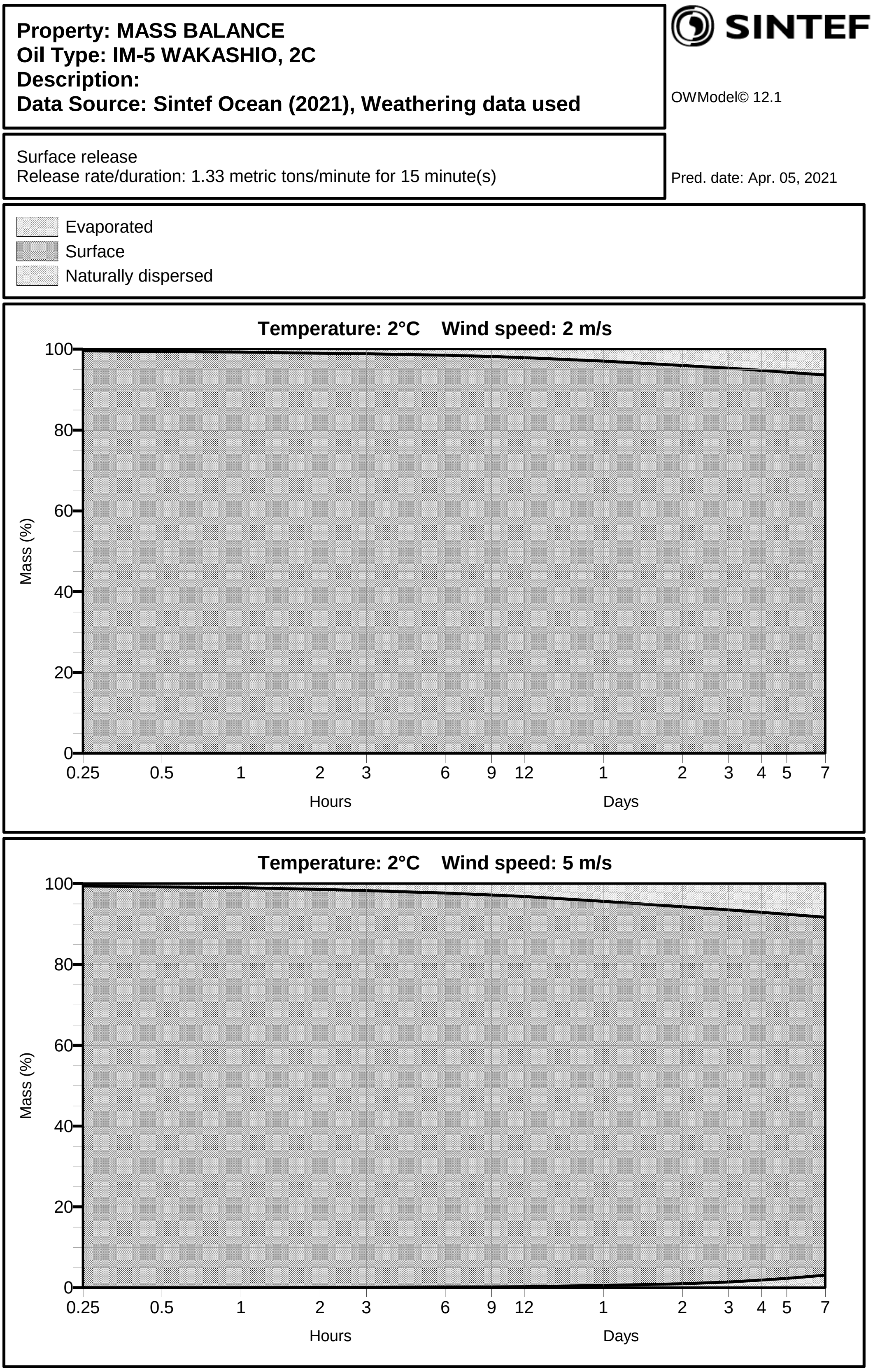


Figure 6-7 *Predicted mass balance of IM-5 Wakashio at 2 °C and wind speeds of 2 and 5 m/s.*

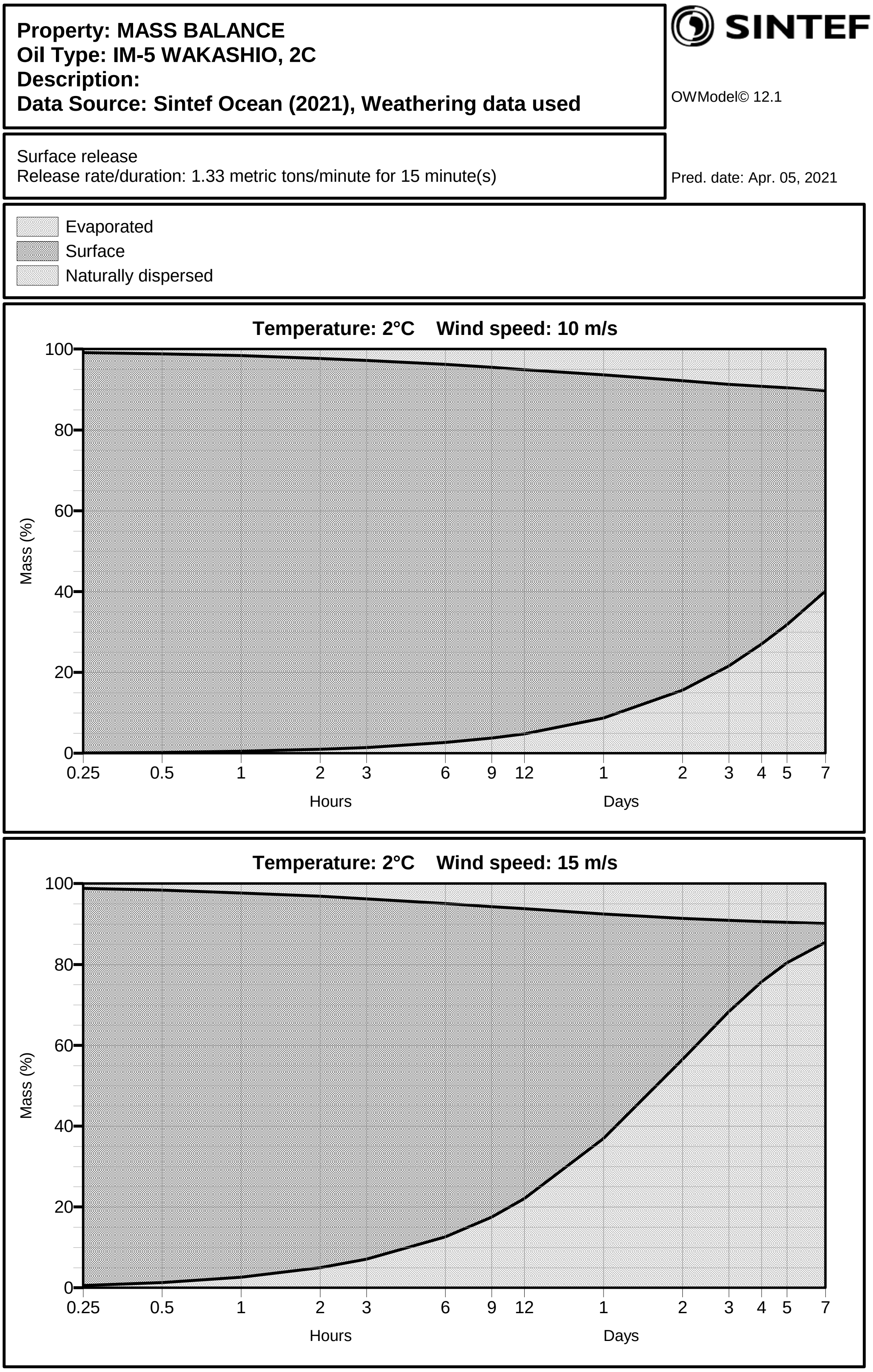


Figure 6-8 Predicted mass balance of IM-5 Wakashio at 2 °C and wind speeds of 10 and 15 m/s.

6.1.2 Predictions at 15 °C

Property: EVAPORATIVE LOSS
Oil Type: IM-5 WAKASHIO, 15C
Description:
Data Source: Sintef Ocean (2021), Weathering data used

SINTEF

OWModel© 12.1

Surface release
Release rate/duration: 1.33 metric tons/minute for 15 minute(s)

2.0
Pred. date: Apr. 05, 2021

Wind Speed (m/s): 15
Wind Speed (m/s): 10
Wind Speed (m/s): 5
Wind Speed (m/s): 2

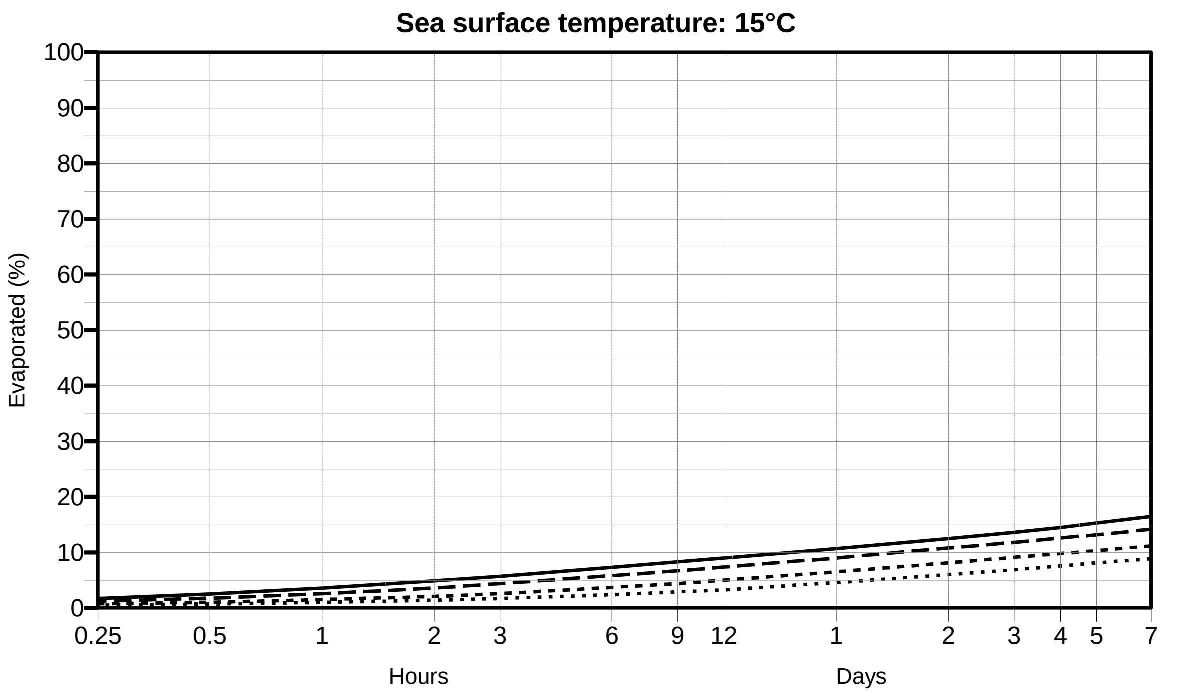


Figure 6-9 Evaporative loss of IM-5 Wakashio predicted at sea temperature of 15 °C.

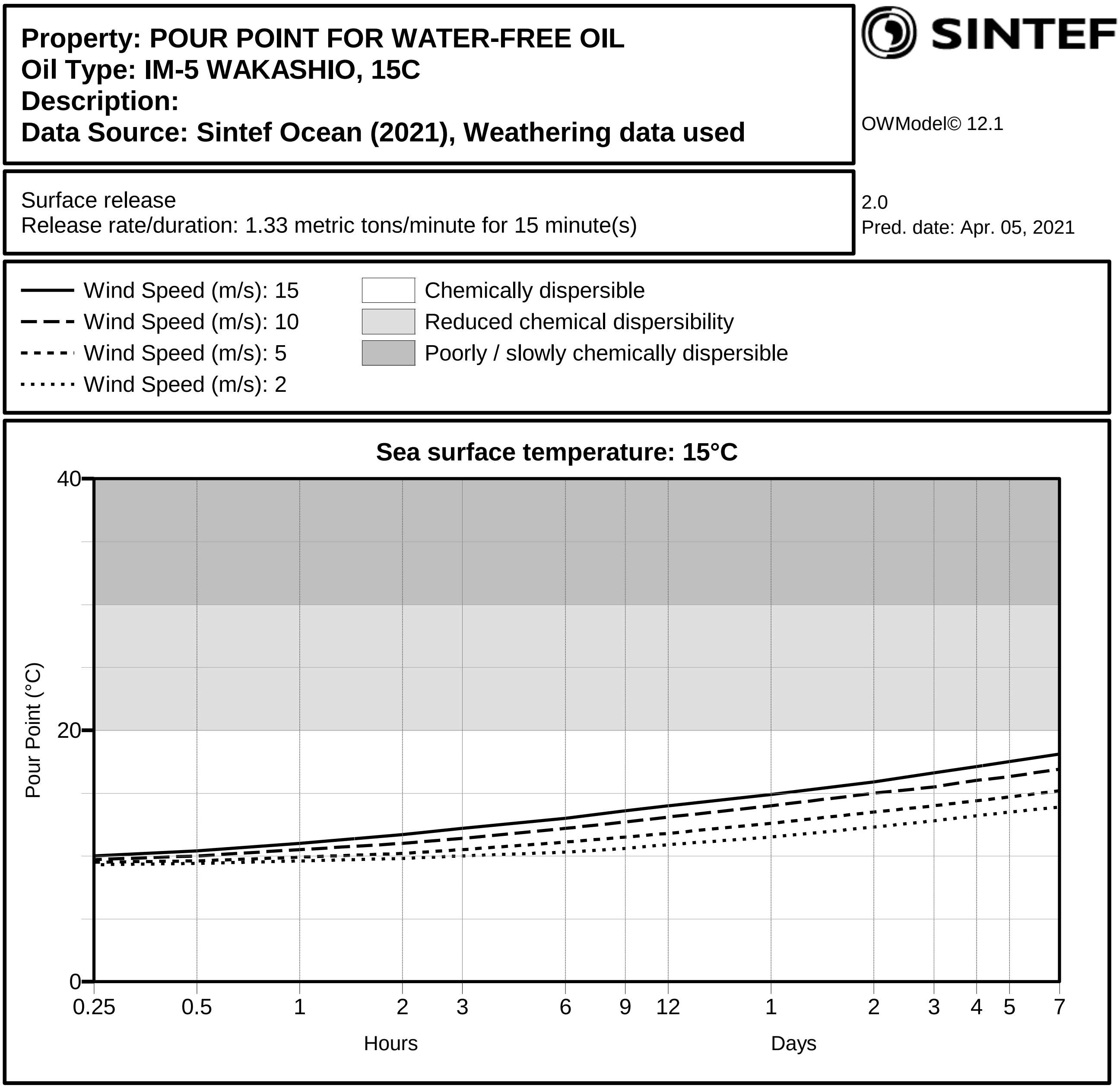


Figure 6-10 Pour point of IM-5 Wakashio predicted at sea temperature of 15 °C.

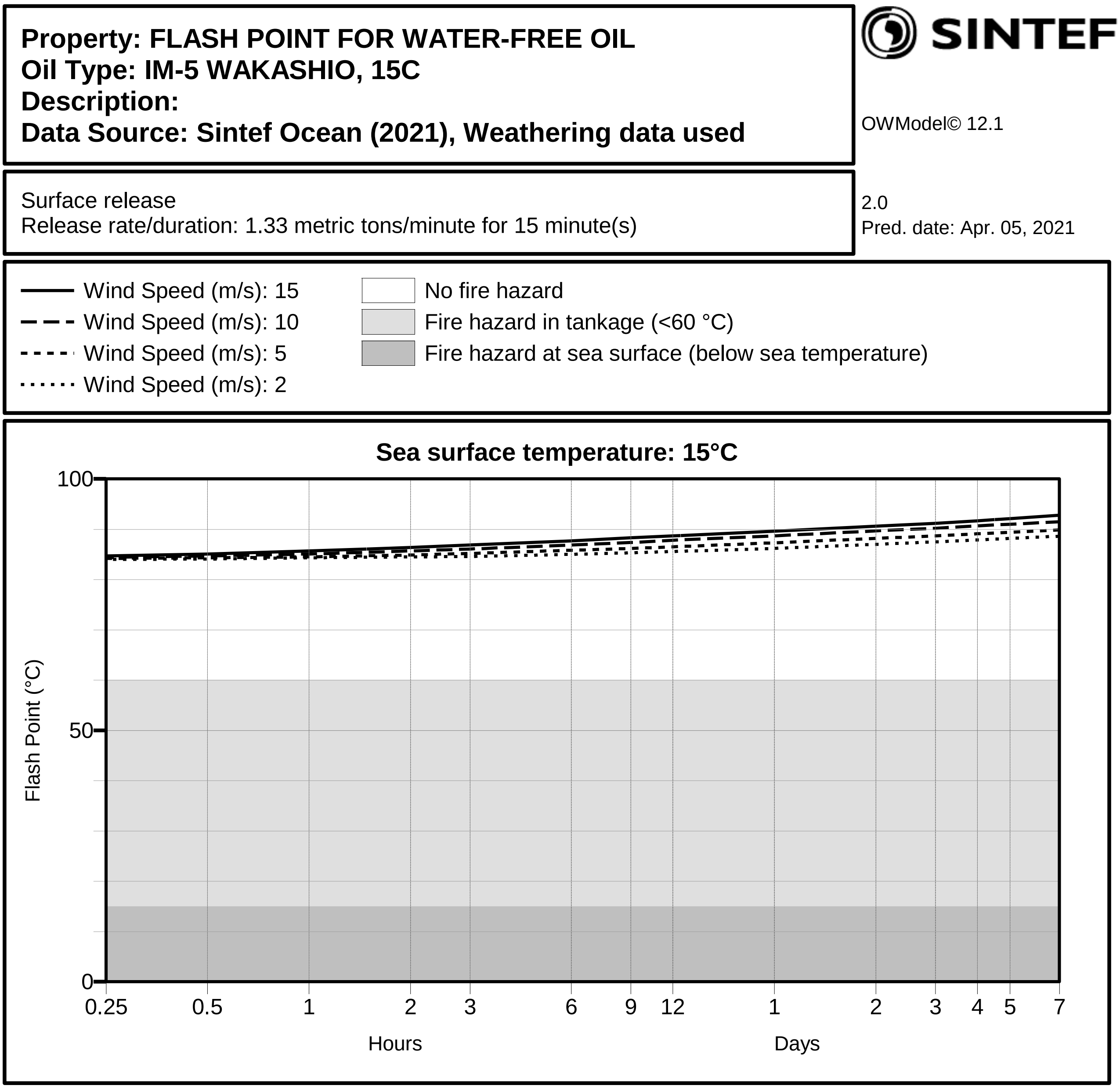


Figure 6-11 Flash point of IM-5 Wakashio predicted at sea temperature of 15 °C.

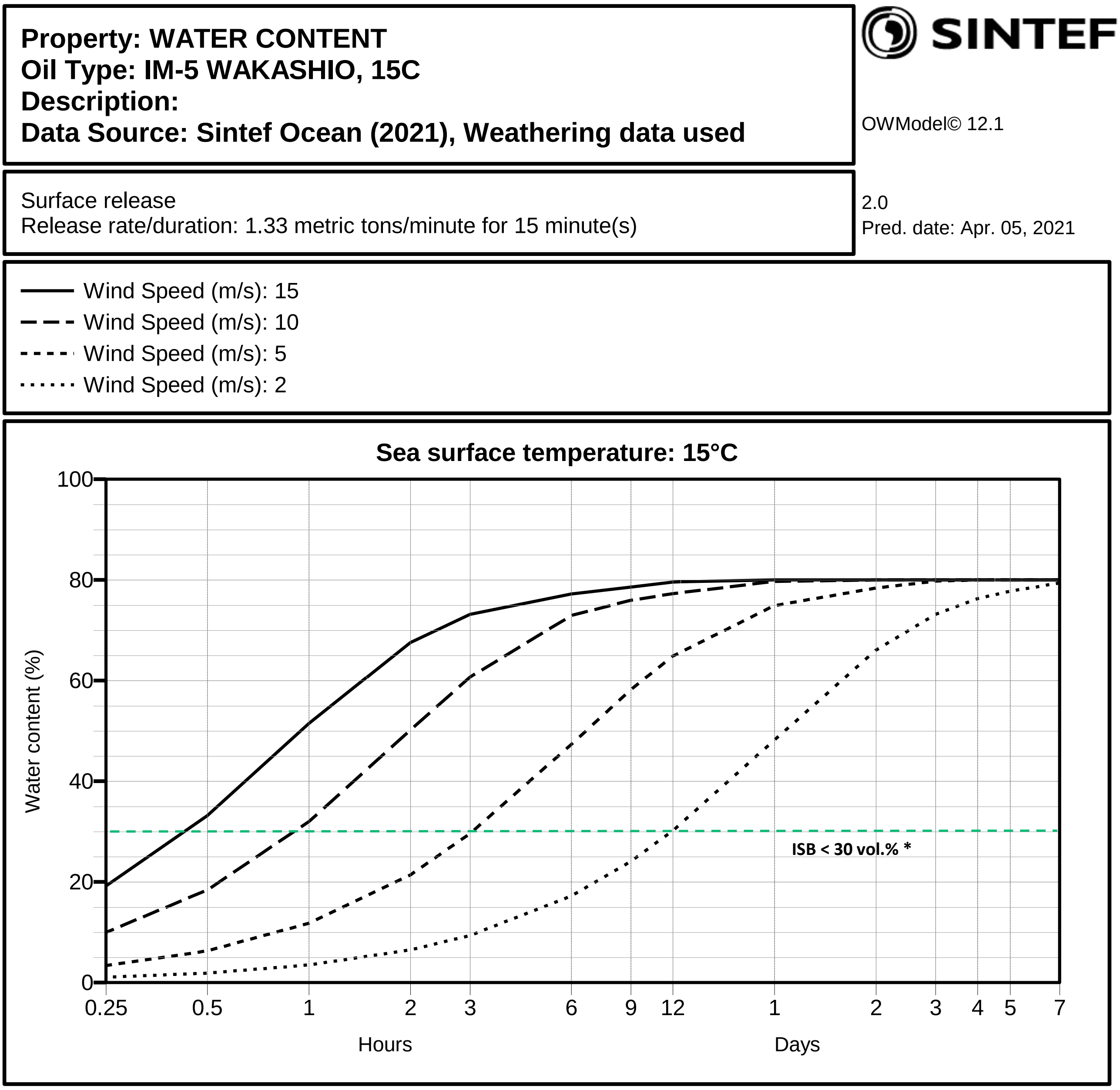


Figure 6-12 *Water content of IM-5 Wakashio predicted at sea temperature of 15 °C. *Rule of thumb: Water content < 30 vol.% for ISB/ignitability.*

Property: VISCOSITY OF EMULSION
Oil Type: IM-5 WAKASHIO, 15C
Description:
Data Source: Sintef Ocean (2021), Weathering data used

SINTEF

OWModel© 12.1

Surface release
Release rate/duration: 1.33 metric tons/minute for 15 minute(s)

2.0
Pred. date: Apr. 05, 2021

Wind Speed (m/s): 15
Wind Speed (m/s): 10
Wind Speed (m/s): 5
Wind Speed (m/s): 2

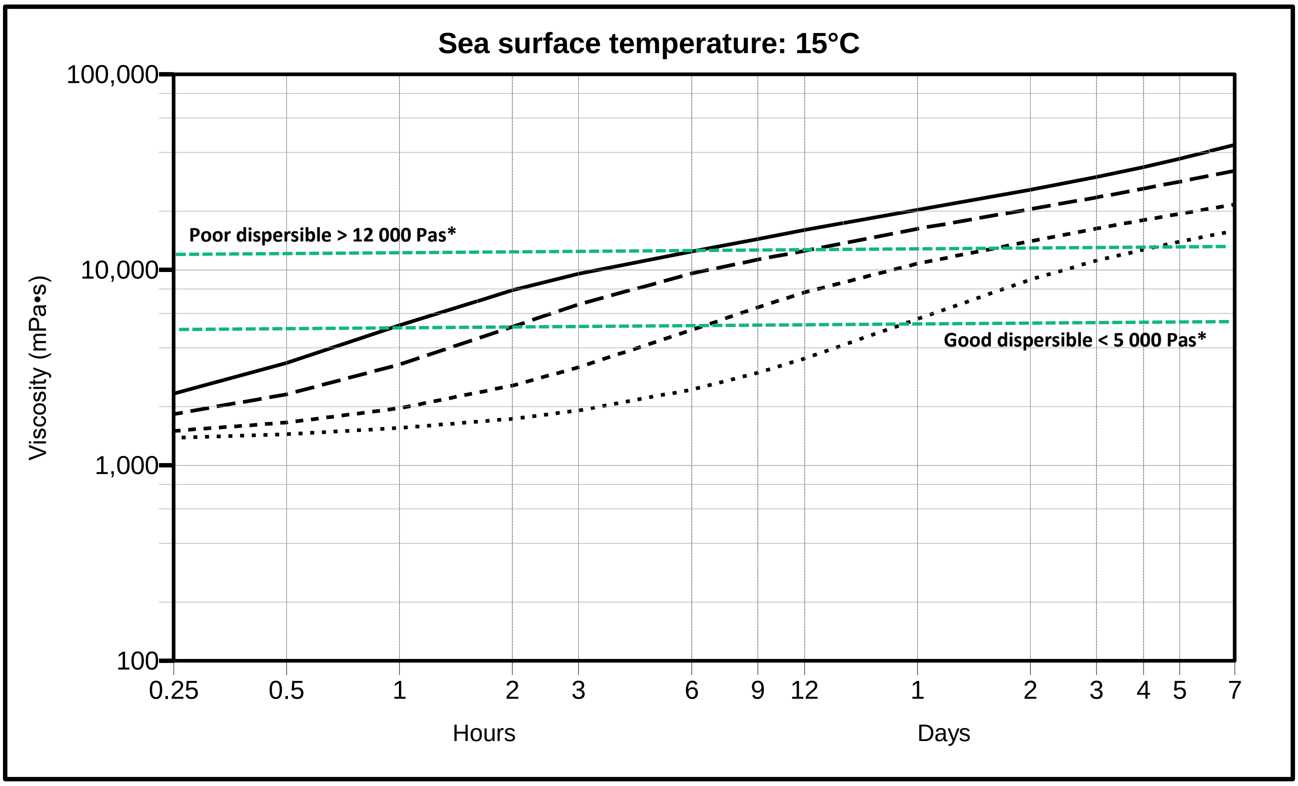


Figure 6-13 *Viscosity of IM-5 Wakashio emulsion predicted at sea temperature of 15 °C. Prediction is based on measurements of emulsions performed at a shear rate of 10 s^{-1}. * Estimated dispersibility limits is based on the FET-test at 15 °C from the meso-scale flume testing.*

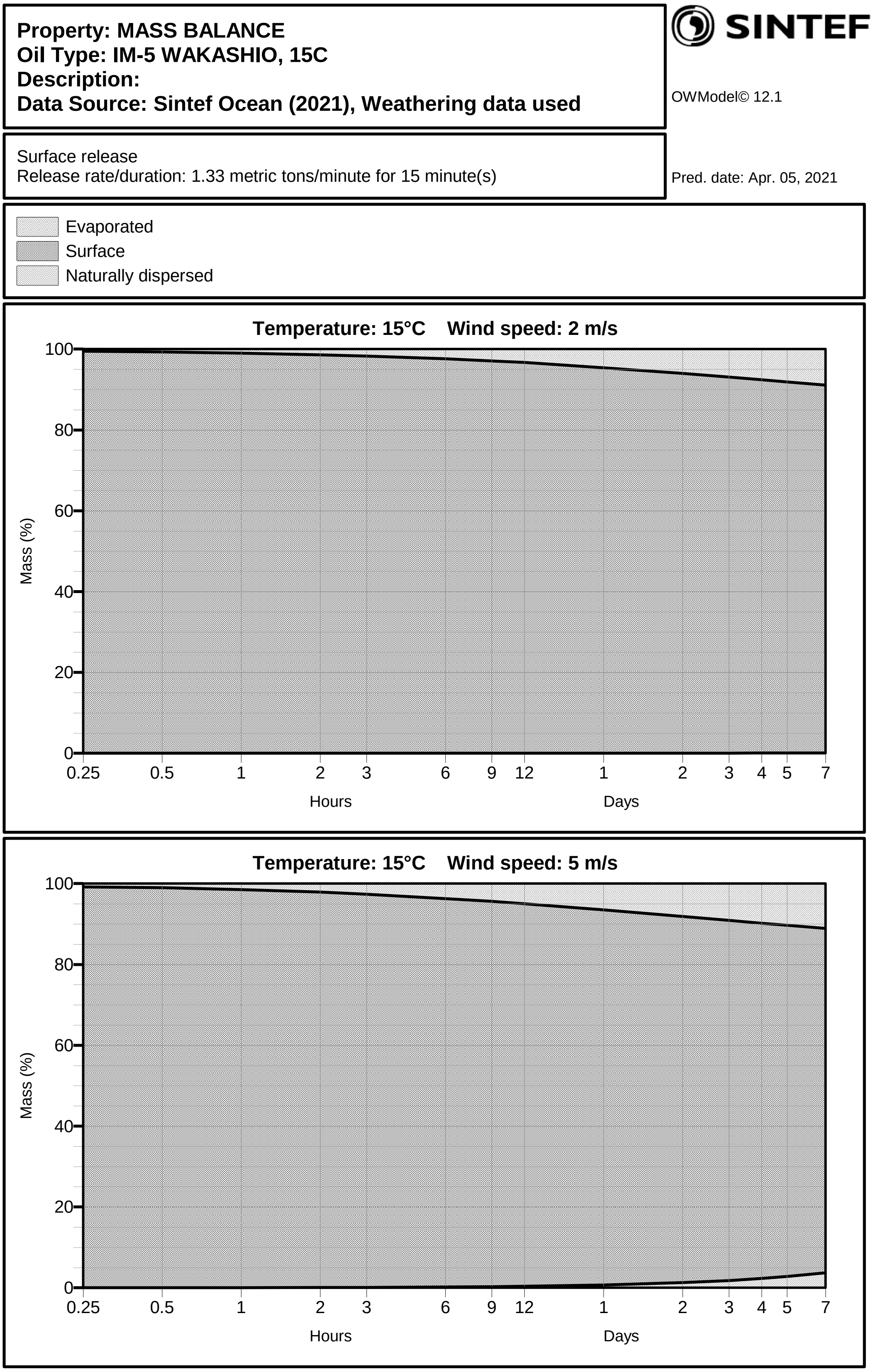


Figure 6-14 Predicted mass balance of IM-5 Wakashio at 15 °C and wind speeds of 2 and 5 m/s.

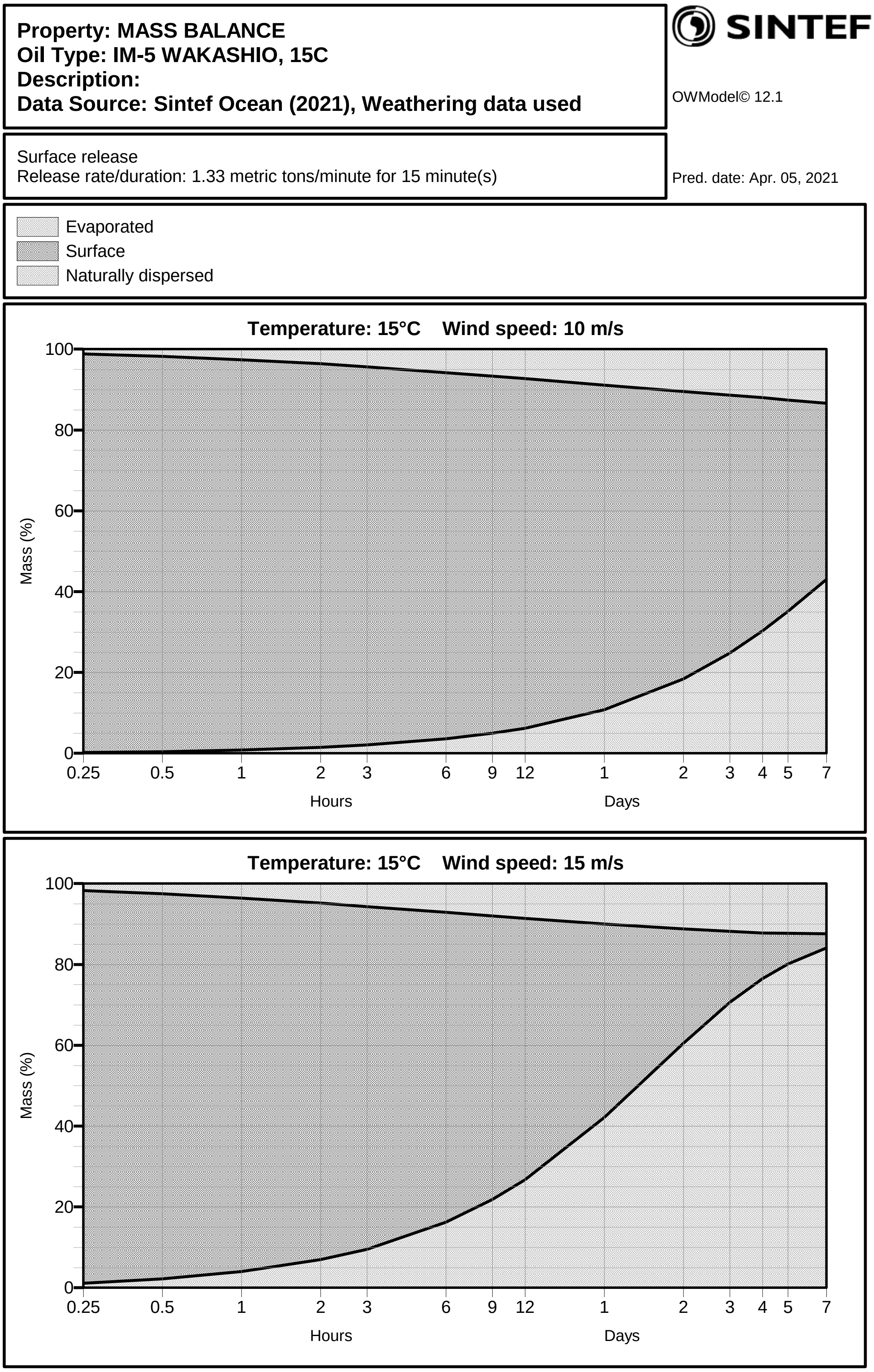


Figure 6-15 Predicted mass balance of IM-5 Wakashio at 15 °C and wind speeds of 10 and 15 m/s.

6.2 Comparison of OWM predictions of IM-5 Wakashio with other residual fuel oils

In this sub-section, the predicted weathering properties of IM-5 Wakashio are compared with VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2. The presented comparisons are based on predictions using a temperature of 2 °C and wind speed of 10 m/s, reflecting arctic/cold water and breaking waves conditions.

6.2.1 Evaporative loss

The predicted evaporative loss is shown in Figure 6-16. Evaporation is one of the natural process that promotes removing spilled oil from the sea surface. The low sulfur marine fuel oils tested in this project have low evaporative loss (< 20 wt. %) after 7 days of weathering, reflecting low content of light components in the oils similar as for the other oils in comparison.

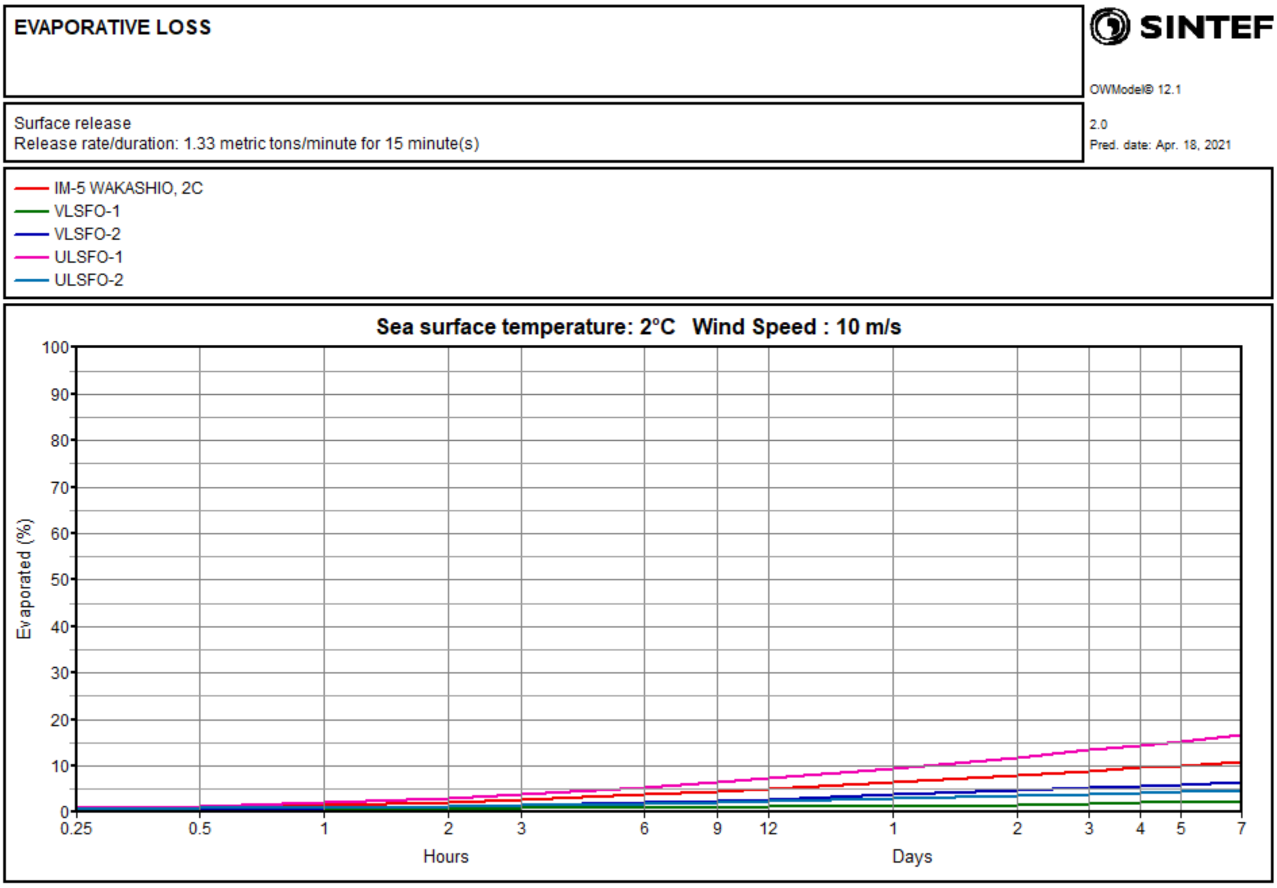


Figure 6-16 Comparison of predicted evaporative loss at 2 °C and 10 m/s IM-5 Wakashio, VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2.

6.2.2 Water uptake

The predicted maximum water uptake of IM-5 Wakashio (66 wt.%) is higher compared with the other oils and its most similar with the ULSFO-1 (54 wt.%). VLSFO-1, VLSFO-2 and ULSFO-2 have water uptake in the range if 25-40 wt.%. It should be emphasized that the VLSFOs and ULSFO-2 have been not tested in the flume, and somewhat higher water uptake for these oils cannot be excluded, however, these oils have much higher viscosities, and it is therefore expected that water uptakes would be lower than IM-5 Wakashio and ULSFO-1 (Daling et al.1990).

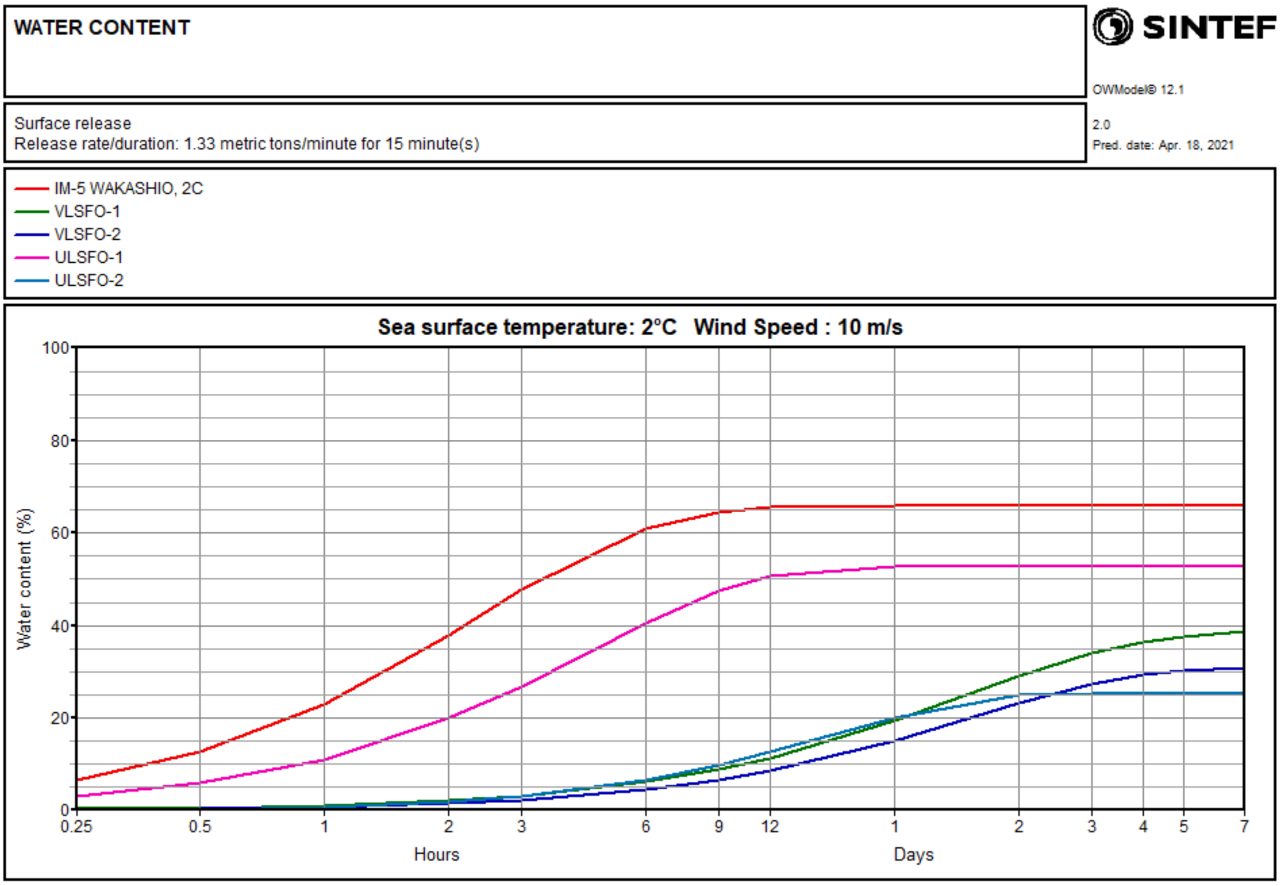


Figure 6-17 Comparison of predicted water uptake at 2 °C and 10 m/s IM-5 Wakashio, VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2.

6.2.3 Emulsion viscosity

The predicted emulsion viscosities at 2 °C of IM-5 Wakashio and the other fuel oils are shown in Figure 6-18. Among these oil, IM-5 Wakashio exhibits the lowest emulsion viscosity by time reaching about 40 000 mPa.s., which is in the range of medium-viscous emulsions (10-50 000 mPa.s) after 5-7 days at sea. In comparison, VLSFO-2 has a very high emulsion viscosities and can reach about 600 000 mPa.s. High viscosities influence of strategy for oil spill response.

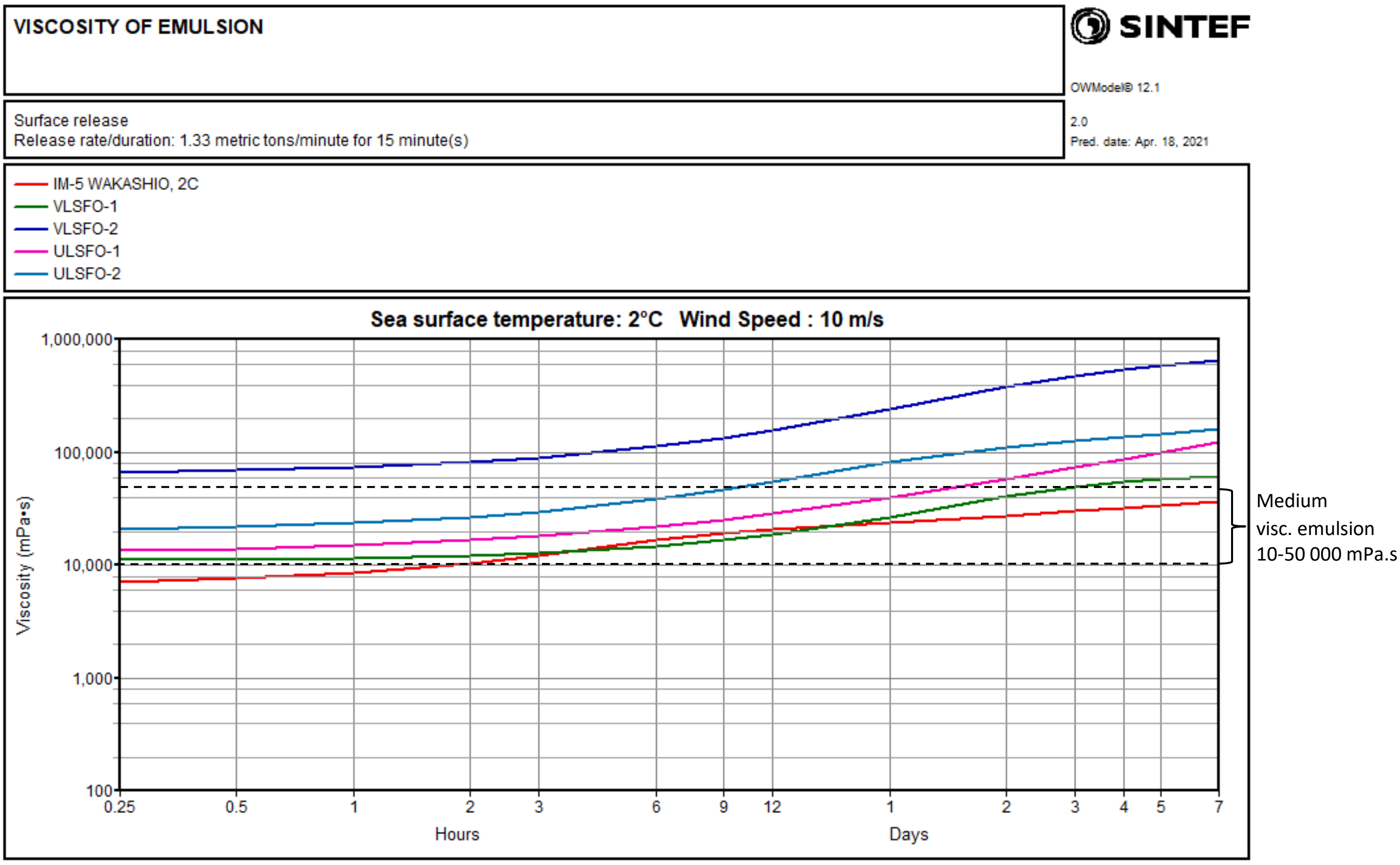


Figure 6-18 Comparison of predicted emulsion viscosity at 2 °C and 10 m/s IM-5 Wakashio, VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2.

6.2.4 Flash point

In general, oils spilled on the sea surface will be cooled to the ambient water temperature within a short period. The probability of fire hazard will be high if the flash point of the oil is below the sea temperature. Moreover, fire hazard is dependent upon the concentration of volatile components in the oil, and the potential for fire is usually surpassed within the first few minutes of a spill due to the rapid evaporation of those components.

The flash point of IM-5 Wakashio is most similar with ULSFO-2 as shown in Figure 6-19. All the fuel oils on comparison have initially high flash points, and no fire or exploration hazard occurs after a release, as the flash points are well above sea temperature, and above 60 °C as limit for vessels not permitted as cargo for flashpoint < 60 °C. The slow increase of flash points reflects also the low evaporative loss.

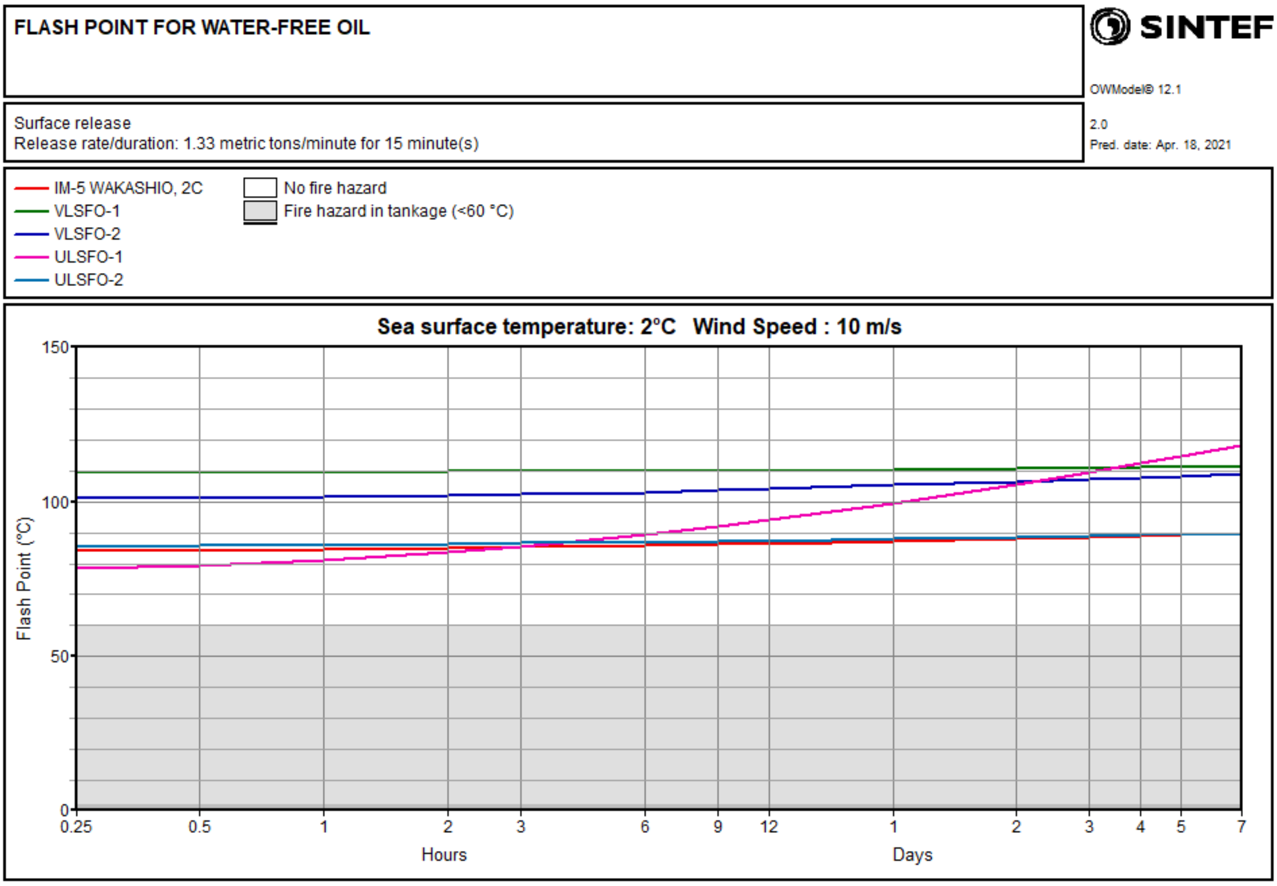


Figure 6-19 Comparison of predicted flash point at 2 °C and 10 m/s IM-5 Wakashio, VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2.

6.2.5 Pour point

Pour point depends on the oil's wax content and the amounts of light components that can keep the wax components dissolved in the oil phase. In addition, contents of asphaltenes prevent or reduce precipitation and lattice formation and hence lowers the pour point. High pour points may prevent the dispersant to soak into the oil slick and influence the dispersant effectiveness and may also reduce the potential for flowability towards weir skimmers. High pour points may cause solidification (elastic properties) when oil is spilled on the sea surface. High pour point may therefore imply solidification on the sea surface immediately after the release, and this is pronounced when the pour point is typically 5-15 °C above sea temperature and in cold temperatures (Daling et al. 1990). High pour point may reduce the dispersant effectiveness.

The predicted pour points of the tested oils are given in Figure 6-20. IM-5 Wakashio and VLSFO-1 have very similar pour points and are lower than the two ULSFOs with very high pour points (reflects poorly /low dispersibility). VLSFO-2 has the lowest pour points among these oils.

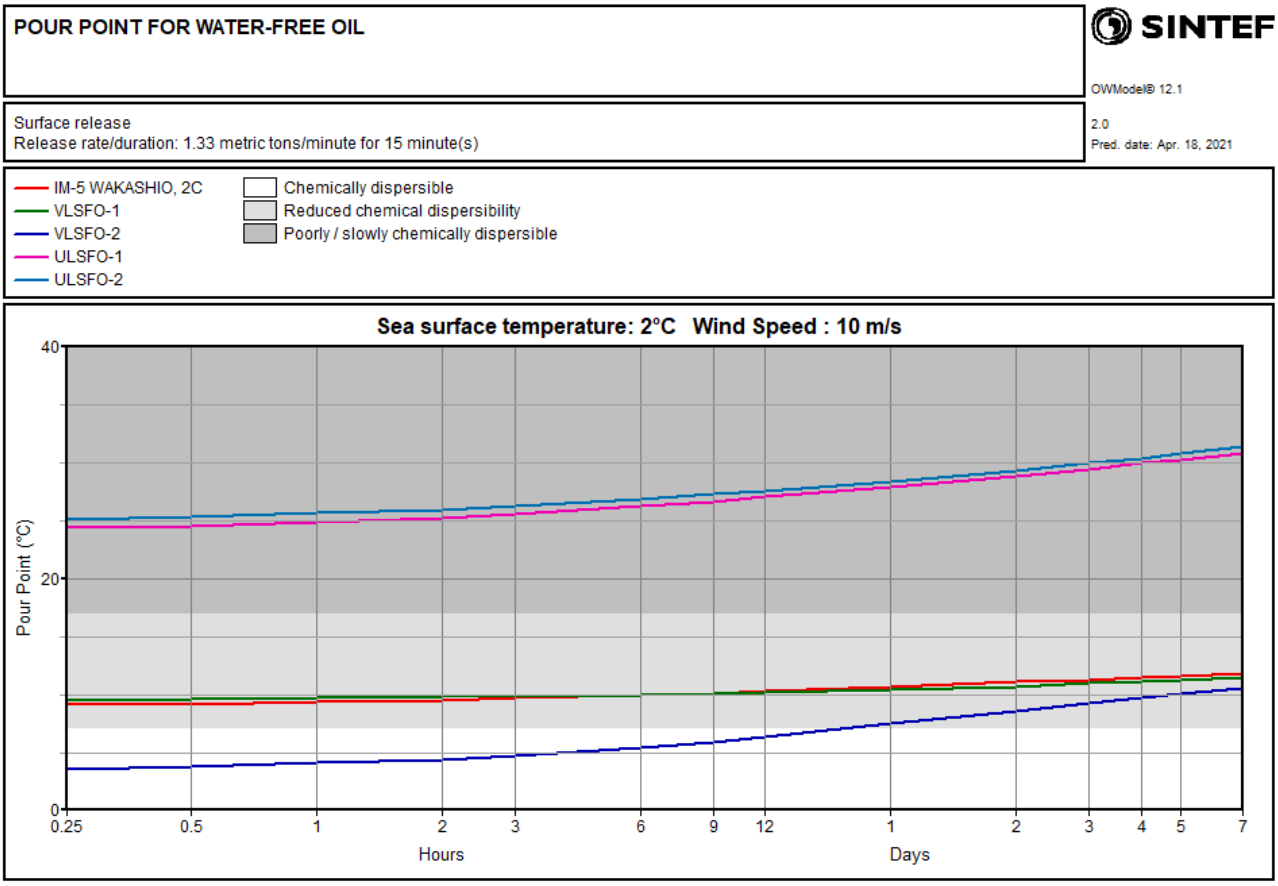


Figure 6-20 Comparison of predicted pour point at 2 °C and 10 m/s IM-5 Wakashio, VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2.

6.2.6 Volume of surface emulsion

In general, the total volume of surface oil will for most oil types be reduced with time due to evaporation and natural dispersion in the initial stages of weathering. However, the volume of water mixed into the oil may increase the total volume of the surface emulsion considerably. Increasing surface emulsions should be considered in a spill operation, for example, when evaluating skimmer capacity based on the total volume of emulsified oil. For the residual marine fuel oils studied in this project, the evaporation is very low.

The predicted volumes of oil emulsion on the sea surface (relative to the amount of oil released) are shown in Figure 6-21 for IM-5 Wakashio in comparison with VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2. IM-5 Wakashio has the highest increase of the total emulsion volume (about 2.5 times), compared with the other oils, and reflecting the higher water uptake. The VLSFOs and ULSFO-2 exhibit low and slow emulsification rate (water uptake) that reflect the low increase of the total volume.

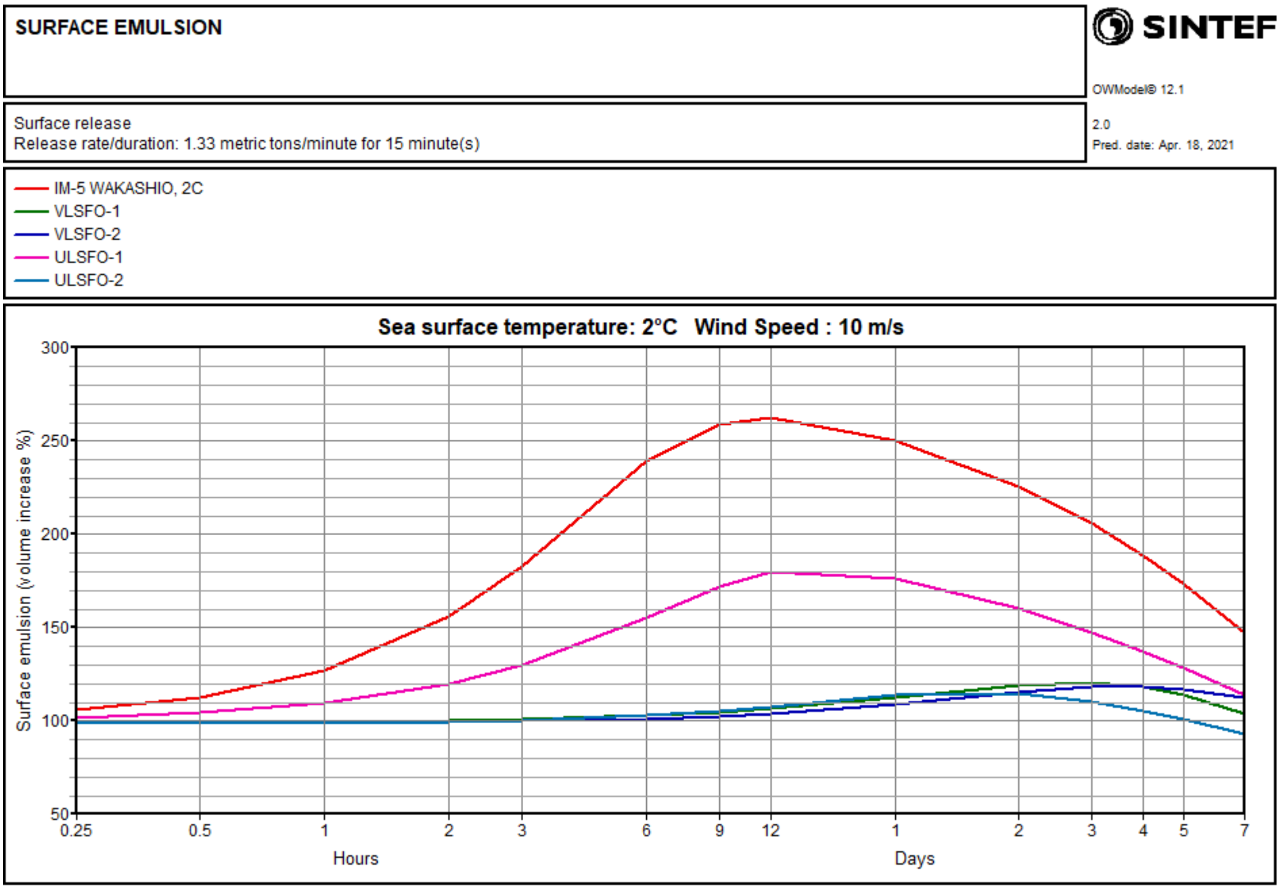


Figure 6-21 Comparison of predicted surface emulsion (emulsification) at 2 °C and 10 m/s IM-5 Wakashio, VLSFO-1, VLSFO-2, ULSFO-1, and ULSFO-2.

7 Oil properties related to oil spill response in cold water conditions

The oil weathering properties will influence on the evaluation of response options such as mechanical recovery, use of dispersants and *in-situ* burning (ISB). Based on the present study, this section briefly discusses the possible response options for IM-5 Wakashio if spilled in cold climate region or arctic conditions (2 °C).

Mechanical recovery

Previous studies at SINTEF have shown that weir skimmers may reduce recovery rates (m^3/h) when skimming oils with viscosities above 15-20 000 mPa.s (Leirvik et al. 2001). NOFO (Norwegian Clean Seas Association for Operating Companies) is operating with viscosity limits for skimmer efficiency as followed: primary use of weir skimmers (<20 000 mPa.s), combination of weir and high-visc. skimmer (20-50 000 mPa.s), and primary high visc. skimmer (>50 000 mPa.s). The NCA, however, have other skimmer types in their stock that are suitable for a range of emulsion viscosities, as used the following criteria's: Low viscous oils/emulsions<10 000 mPa.s, medium viscous oil/emulsions: 10-50 000 mPa.s and high viscous oils/emulsions >50 000 mPa.s. Other oils spill responders may have other skimmer /equipment in stock.

For IM-5 Wakashio, mechanical recovery should be considered as the main strategy for oil spill response in cold climate regions or arctic conditions. The emulsion viscosity may reach to about 40.000 mPa.s after one-week of weathering at sea, and such viscosities are considered at as medium viscous oil /emulsions by the NCA (<50 000 mPa.s.) In addition, the pour point of IM-5 Wakashio is not that high to expect any server solidification at sea that will influence negatively to the mechanical response strategy.

Dispersibility

The results from the meso-scale flume testing indicate that IM-5 Wakashio can be dispersible during the first hours after a spill in cold climate /arctic conditions. However, after 7 days of weathering the use of dispersants may break up the slick into smaller patches and lumps that may be entrained into the water phase by waves, but not forming a good dispersion as small oil droplets. Moreover, breaking up the slick by dispersant application requires breaking wave conditions and successive application of dispersants with high dosages, particularly if the emulsion layer is thick. Therefore, use of dispersants is very depended on the weathering time and is not recommended as a main strategy for IM-Wakashio after several days of weathering at sea.

***In-situ* burning / ignitability**

In-situ burning (ISB) is often considered as a primary response operation in arctic and ice-covered areas. The ice can be used to confine the oil and increase the film thickness to increase the ignitability. However, ISB is dependent on other factors such as wind speeds and a water contents (degree of emulsification). This means that ISB could be an option during the in the first hours for IM-Wakashio in low wind speeds (<5 m/s), however, the oil may require prolonged time to be heated by burning gelled gasoline /diesel mixture shown for other VLSFOs and ULFOs (Sørheim et al. 2020, Hellstrøm et al. 2017). As a rule of thumb, low efficacy for ISB is expected for residual fuel emulsions with water content > 30 vol.%.

8 Intercalibration comparison

As a part of this project, SINTEF and Cedre have performed some similar testing on the fresh oil of IM-5 Wakashio for comparison. The laboratories are using similar or comparable analytical methods for evaporative loss, density, viscosity, asphaltenes and waxes. The results are recognized as sufficient comparable data between the two laboratories, as summarized in Table 8-1.

Table 8-1 Physico-chemical parameters of IM-5 Wakashio measured at SINTEF and Cedre.

IM-5 Wakashio Fresh oil	**Evaporative loss vol.%**	**Density g/mL**	**Viscosity 15°C mPa.s $100s^{-1}$**	**Asphaltenes wt.%**	**Waxes wt.%**
SINTEF	12.0	0.91	582	0.52	5.4
Cedre	10.5	0.92	375	0.72	5.7

9 References

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Mackay, D. and Zagorski, W. 1982. "Studies of W/o Emulsions". Report EE-34: Environment Canada, Ottawa, Ontario.

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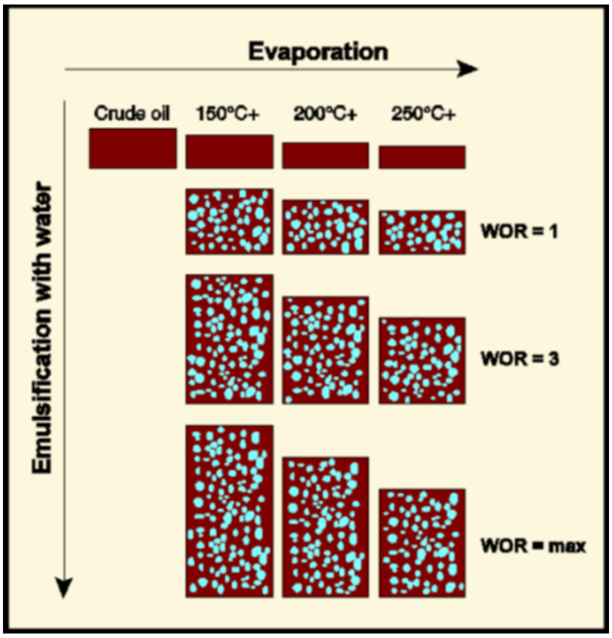
Appendix A Experimental setup

A.1 Bench-scale laboratory methodology

To isolate and map the various weathering processes at sea, the 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 Bench-scale laboratory weathering flow chart of oil.

A.2 Evaporation

The evaporation procedure used is described in Stiver and Mackay (1984). Evaporation of the lighter compounds from the fresh condensate was carried out as a simple one-step distillation to vapor temperatures of 150 °C, 200 °C and 250 °C, which resulted in 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.

A.3 Physical and chemical analysis

The viscosity, density, pour point and flashpoint of the fresh and water-free residues was analysed. In addition, wax content and "hard" asphaltenes was measured for the 250°C+ residue. Viscosity for all the w/o emulsions was determined. The analytical methods used 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	-
Flash point	ASTM D 56-82	Pensky-Martens, PMP1, SUR

Table A-2 Analytical methods used to determine the chemical properties.

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

A.4 Chemical characterization by GC-FID and GC-MS

- The distribution of hydrocarbons (nC_5-nC_{40}) was analysed using a Gas Chromatograph coupled with a Flame Ionisation Detector (GC-FID). The Gas Chromatograph used was an Agilent 6890N with a 30m DB1 column.
- The analysis and quantification of PAHs, phenols, and alkylated phenols (C_0-C_4) were completed using an Agilent 6890 Gas Chromatograph coupled with a 5973 MSD detector (GC-MS) operating in SIM mode (Selected Ion Monitoring).
- The volatile components were in the range of nC_5-nC_{10} and were quantified by use of PT-GC-MS (Purge and Trap Gas chromatograph Mass Spectrometer operating in full-scan mode and using a modified version of the EPA 8260 analysis method).

A.5 Emulsification properties

The w/o emulsification studies 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
- 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.

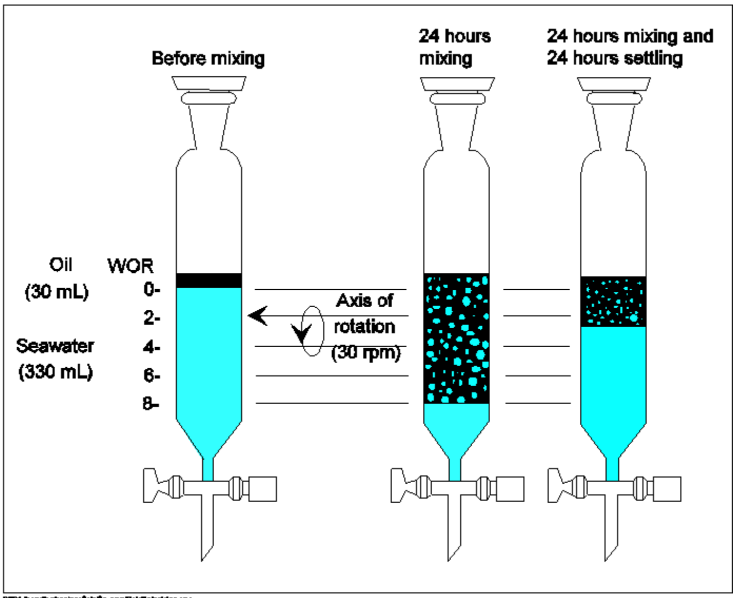


Figure A-2 Principle of the rotating cylinder method

A.6 Description and weathering in the meso-scale flume basin

A schematic drawing of the SINTEF meso-scale flume (Singsaas et al. 1993) is given in Figure 4 1. Approximately 5 m3 seawater circulates in the 10 metres long flume. The flume basin is stored in a temperature-controlled room (0 °C-20 °C). Two fans are placed in a covered wind tunnel, controlling the wind speed. The fans are calibrated to simulate an evaporation rate corresponding to a wind speed of 5-10 m/s on the sea surface.

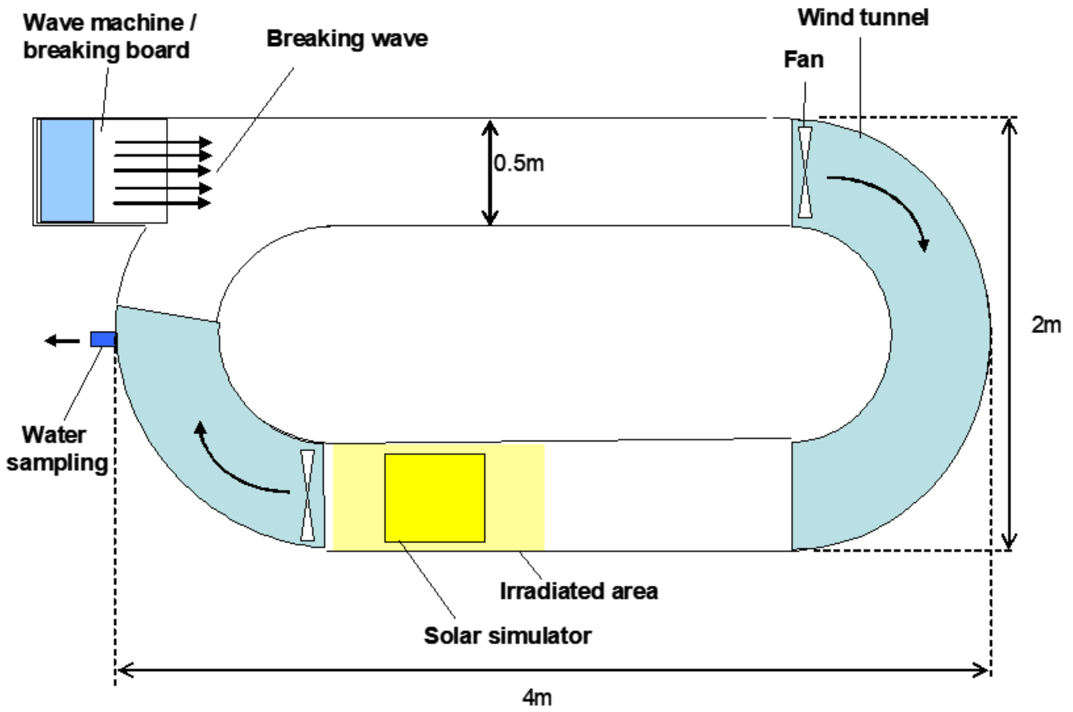


Figure A-3 Schematic drawing of the meso-scale flume basin at SINTEF.

A standard volume sample (9 L) of the relevant oil is carefully released onto the seawater surface under calm conditions. The wave machine and fans are then started, and the experiment begins. A reference water sample is collected before the beginning of the experiment.

Samples of the surface oil/emulsion were in this project collected after 0, 0.5, 1, 2, 4, 6, 24 hours, and 2, 3, 5 and 7 days. Free-water was drained off after settling in the climate room and handled for further analysis of viscosity, water content, density, evaporative loss, and emulsion stability. The water samples were analyzed for oil concentration. Samples were taken at a depth of 50 cm through a tap in the basin wall into a Pyrex glass bottles (1 L). Samples are extracted by liquid-liquid extraction with dichloromethane (DCM) and quantified by Ultraviolet (UV) spectrophotometry.

Sunlight is simulated with a solar simulator from Gmbh Steuernagel. The solar lamp (4 KW) emits a wavelength spectrum calibrated to fit natural sunlight at high noon and in the absence of clouds. Figure A-4 shows the measured spectrum from the solar simulator compared to one of the most widely used standard spectra for solar irradiance (CIE publication 85, 1989).

Appendix B Input data to SINTEF Oil Weathering Model (OWM)

The obtained laboratory data are customized for input to SINTEF OWM (weathering model). The tabulated laboratory oil data for IM-5 Wakashio are given Table B-1 to Table B-4. The input data were based on the weathering data at 2 and 15 °C.

Table B-1 Physical and chemical parameters of IM-5 Wakashio.

Summary properties fresh oil	**IM-5 Wakashio 2 °C**	**IM-5 Wakashio 15 °C**
Density (60 F/15.5°C) (g/mL)	0.9079	0.9079
Pour point (°C)*	9	9
Reference temperature (°C)	2	15
Viscosity at ref. temp. (mPa·s = cP) **	1199	6402
Asphaltenes (wt. %)	0.52	0.52
Wax content (wt. %)	5.4	5.4
Flash point (°C)*	84	84

** Measured at shear rate 100 s^{-1} ** Data from Cedre -:no data*

Table B-2 True boiling point (TBP) IM-5 Wakashio (TBP based on simulated distillation).

Temp. (°C)	**IM-5 Wakashio (vol. %)**
157	1.3
174	2.5
209	5.9
246	11.4
306	22.2
379	32.6
427	42.7
467	52.7
506	62.5
550	72.3
601	81.9
672	91.4
704	94.2
720	96.0

Table B-3 Summary lab weathering and customized data (italic) of IM-5 Wakashio, 2 °C.

Properties	**Fresh**	**150°C+**	**200°C+**	**250°C+**
Vol. Topped (%)	0	0	*8*	12
Weight Residue (wt. %)	100	100	*97*	92.7
Density (g/mL)	0.9079	0.9079	*0.915*	0.9171
Pour point (°C)	9	9	*12*	15
Flash Point (°C)	84	84	*86*	90
*Viscosity of water-free residue (mPa.s =cP)	6402	6402	*10000*	18613
**Viscosity of 50% emulsion (mPa.s = cP)	-	12997	*15000*	18316
**Viscosity of 75% emulsion (mPa.s = cP)	-	-	-	-
**Viscosity of max water (mPa.s = cP)	-	11054	*25000*	35000
Max. water cont. (vol. %)	-	66	*66*	66
(T1/2) Halftime for water uptake (hrs)	-	0.5	*0.2*	0.11
Stability ratio	-	1.0	*1.0*	1.0

** Measured at shear rate 100 s^{-1}*
*** Measured at shear rate 10 s^{-1}*
- No data

Table B-4 Summary lab weathering and customized data (italic) of IM-5 Wakashio, 15°C.

Properties	**Fresh**	**150°C+**	**200°C+**	**250°C+**
Vol. Topped (%)	0	0	*8*	12
Weight Residue (wt. %)	100	100	*97*	92.7
Density (g/mL)	0.9079	0.9079	*0.915*	0.9171
Pour point (°C)	9	9	*12*	15
Flash Point (°C)	84	84	*86*	90
*Viscosity of water-free residue (mPa.s =cP)	1199	1199	*3000*	4977
**Viscosity of 50% emulsion (mPa.s = cP)	-	2673	*8000*	10106
**Viscosity of 75% emulsion (mPa.s = cP)	-	-	-	-
**Viscosity of max water (mPa.s = cP)	-	5958	*12000*	15000
Max. water cont. (vol. %)	-	65	*80*	80
(T1/2) Halftime for water uptake (hrs)	-	0.2	*0.2*	0.2
Stability ratio	-	1.0	*1.0*	1.0

** Measured at shear rate 100 s^{-1}*
*** Measured at shear rate 10 s^{-1}*
- No data

Appendix C Chemical characterization on GC-MS (OSCAR oil profile)

The OSCAR oil profile is based on the quantification of semi-volatile organic hydrocarbons (SVOC) and volatile organic hydrocarbons (VOC) by GC-MS. The composition is divided into individual pseudo-component groups (OSCAR groups) representing the oil from the TBP (True Boiling Point) fractions.

Table C-1 Chemical characterization ("oil profile") of IM-5 Wakashio fresh oil derived from GC-MS analysis and TBP oil fraction.

IM-5 Wakashio wt.%	Groups	Pseudo-Components
0.000	1	C1-C4 gasses (dissolved in oil)
0.000	2	C5-saturates (n-/iso-/cyclo)
0.045	3	C6-saturates (n-/iso-/cyclo)
0.005	4	Benzene
0.050	5	C7-saturates (n-/iso-/cyclo)
0.005	6	C1-Benzene (Toluene) et. B
0.396	7	C8-saturates (n-/iso-/cyclo)
0.033	8	C2-Benzene (xylenes; using O-xylene)
1.414	9	C9-saturates (n-/iso-/cyclo)
0.053	10	C3-Benzene
2.000	11	C10-saturates (n-/iso-/cyclo)
0.011	12	C4 and C4 Benzenes
1.989	13	C11-C12 (total sat + aro)
0.000	14	Phenols (C0-C4 alkylated)
0.193	15	Naphthalenes 1 (C0-C1-alkylated)
5.807	16	C13-C14 (total sat + aro)
N/A	17	Unresolved Chromatographic Materials (UCM: C10 to C36) 0 0 0
0.345	18	Naphthalenes 2 (C2-C3-alkylated)
4.655	19	C15-C16 (total sat + aro)
0.288	20	PAH 1 (Medium soluble polyaromatic hydrocarbons (3 rings-non-alkylated;<4 rings)
4.712	21	C17-C18 (total sat + aro)
4.000	22	C19-C20 (total sat + aro)
7.500	23	C21-C25 (total sat + aro)
0.500	24	PAH 2 (Low soluble polyaromatic hydrocarbons (3 rings-alkylated; 4-5+ rings)
66.000	25	C25+ (total)

N/A: Not analyzed. UCM included in group 11-25.

The SINTEF OSCAR model is a 3-dimensional Oil Spill Contingency And Response model system that calculates and records the distribution (as mass and concentrations) of contaminants on the water surface, on shore, in the water column and in sediments. The model allows multiple release sites, each with a specified beginning and end to the release. This allows time-variable releases at a given location, as well as throughout the study area. The model computes surface spreading, transport, entrainment into the water column, evaporation, emulsification and shore interactions to determine oil drift and fate at the surface. In the water column, horizontal and vertical transport by currents, dissolution, adsorption, settling and degradation are simulated. By modelling the fate of individual pseudo-components, changes in the oil composition due to evaporation, dissolution and degradation are accounted for.