



# SINTEF REPORT

## SINTEF Materials and Chemistry

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TITLE

**Weathering properties of the Linerle crude**

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### ABSTRACT

A limited weathering study is performed on the Linerle crude.

Linerle is an extremely heavy asphaltenic crude. The oil is heavily biodegraded and holds almost no light end components.

Linerle has a slow emulsification rate compared to other Norwegian crudes. Combined with a low evaporation this will give a slow rise in viscosity for the oil on the sea surface. As the initial viscosity for the crude oil is high (1470cP) the emulsion viscosity will however reach viscosities of above 10.000 mPas under 24 hours at wind speeds above 10 m/s. A maximum water content of 70% is recorded in laboratory testing at 13°C. The emulsions formed are stable, and use of demulsifying products will be necessary to break the emulsions quantitatively.

Because of the low evaporation, a relative high lifetime on the sea surface is predicted compared to other Norwegian crudes.

KEYWORDS	ENGLISH	NORWEGIAN
GROUP 1	Environment	Miljø
GROUP 2	Oil	Olje
SELECTED BY AUTHOR	Weathering	Forvitring
	Linerle	Linerle

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## **1 Introduction**

New oil types are continuously coming into production on the Norwegian Continental Shelf. Because of large variations in the physical and chemical properties of the crude oils, their behaviour and fate when spilled at sea may vary greatly. The accident with the “Braer” on the Shetlands and the “Sea Empress” in Wales have shown how important it is to be able to predict the efficiency of different cleanup methods (mechanical, burning, dispersant treatment etc.) and the drift and spreading. It is therefore important to have good knowledge about each oils' expected behaviour at sea, in case of an accidental oil spill. According to new regulations from The Norwegian Pollution Control Authorities (SFT) a characterisation of the oil or condensate with respect to weathering properties and fate in the marine environment should be performed during both exploration and for every oil coming into production.

A limited weathering study of Linerle has been performed on one temperature (13°C). The data from the laboratory investigations has been used as input in the SINTEF Oil Weathering Model for prediction of the oils behaviour at sea under different weather conditions.

## 2 Weathering of oils

Crude oil is a complex mixture of thousands of chemical components. The relative compositions vary, giving rise to crude oils with different chemical and physical properties.

The components found in crude oil are classified into two main chemical groups; hydrocarbons and heteroatomic organics, see Figure 2.1.

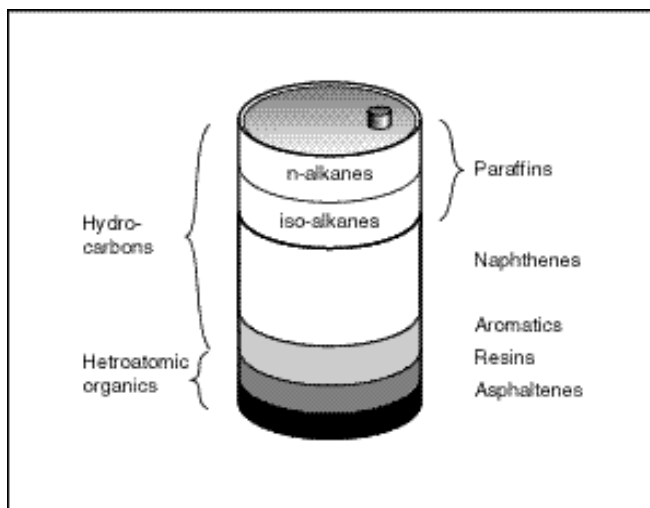


Figure 2.1: The chemical composition of crude oil.

When a crude oil is spilt at sea a number of natural processes take place, which change the volume and the chemical properties of the oil. These natural processes are evaporation, water-in-oil (w/o) emulsification, oil-in-water (o/w) dispersion, release of oil components into the water column, spreading, sedimentation, oxidation, and biodegradation. A common term for all of these natural processes is weathering. The relative contribution of each process varies during the duration of the spill. Figure 2.2 illustrates the various weathering processes and Figure 2.3 shows their relative importance with time.

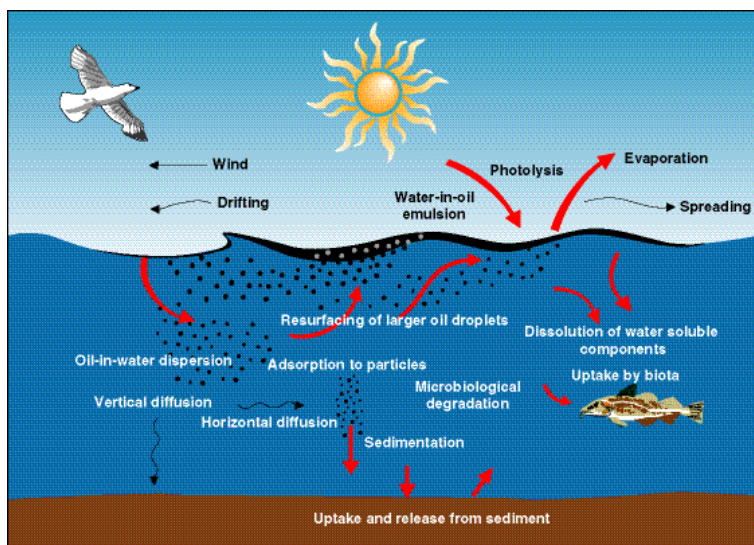


Figure 2.2 The weathering processes taking place when an oil is spilt on the sea surface.

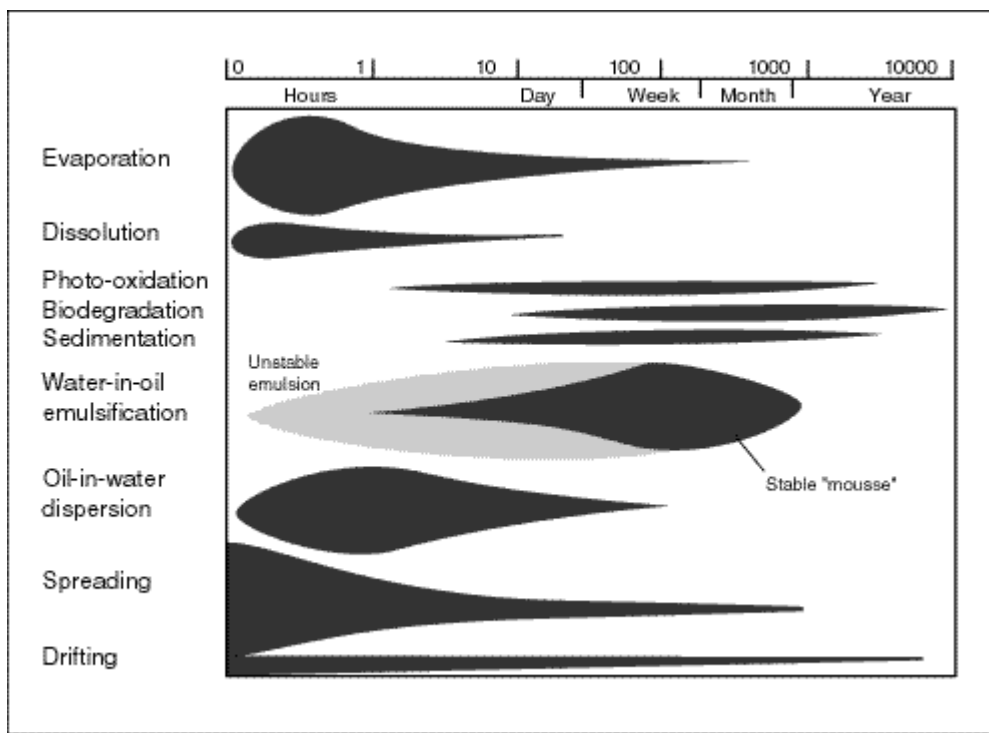


Figure 2.3 Weathering processes' relative importance with time.

The weathering of oil depends on the oil type (chemical and physical properties), the weather conditions (wind, waves, temperature and sunlight) and the properties of the seawater (salinity, temperature, bacterial flora etc.).

For more details about weathering processes see NRC (1985).

### **3 Laboratory weathering testing**

#### **3.1 Experimental methods for bench-scale weathering testing**

##### **3.1.1 Oil Samples**

The oil samples from Statoil were received by SINTEF March 30. 2005.

20 liters of Linerle was received.

The sample was labeled:

Statoil's Prøvenummer: 050128003

Registrert Statoil: 28.01.2005

Site 6608/11-4

The sample is given SINTEF Id 2005-0391

##### **3.1.2 Test temperatures**

The testing of weathering properties was performed at 13°C for Linerle. 13°C is considered a typical summer temperature in the Norwegian Sea. For waxy crudes, the high pour point will complicate modeling of temperature dependent physical properties. As Linerle will have a low pour point even after days of weathering on the sea surface, changes in physical properties due to change in temperature can be readily modeled, and studies on one temperature is considered sufficient.

### 3.1.3 Bench-scale laboratory testing

In order to isolate and map the various weathering processes that take place when an oil is spilled on the sea surface, the weathering of the oils is carried out using a systematic, stepwise procedure developed at SINTEF (Daling *et al.*, 1990). The standard artificial weathering process is illustrated in Figure 3.1. As the Linerle oil shows no significant evaporation at 150°C, and 200°C, the test setup is slightly changed. The oil is evaporated only to the 250°C+ residue, and the fresh oil is used as a representative for the lower boiling residues (150°C+ and 200°C+), and thus used for emulsification testing in the standard setup.

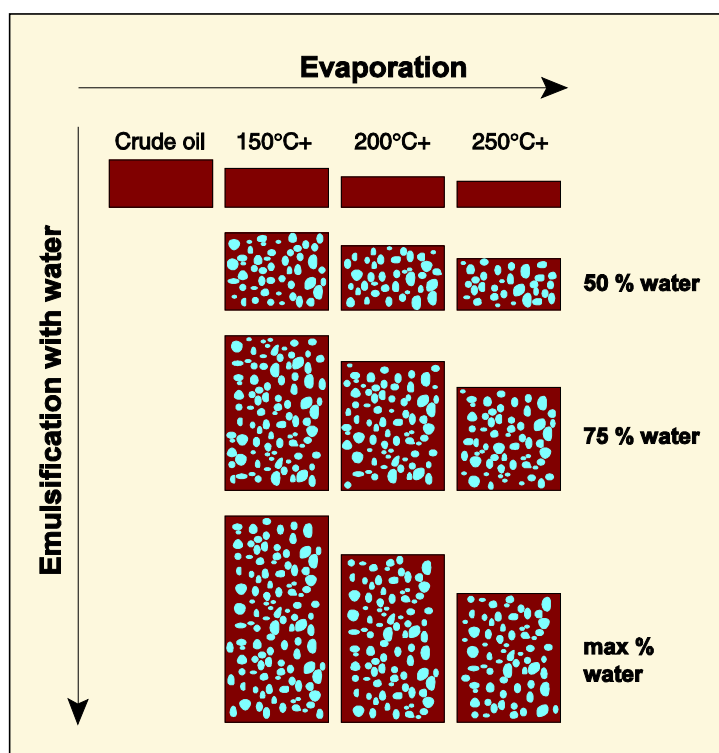


Figure 3.1: Flow chart for the bench-scale laboratory weathering of a crude oil.

#### Evaporation

Evaporation of the lighter compounds from the fresh crude oil is carried out according to a modified ASTM-D86/82 distillation procedure (Stiver and Mackay, 1984). The fresh crude oil is distilled, in a simple one step distillation, to a vapor temperature of 250°C. This will give an oil residue with an evaporation loss typically corresponding to 2-5 days of weathering of an oil slick on the sea surface.

### Water-in-oil (w/o) emulsification

The procedures used in the w/o-emulsification studies are described in detail by Hokstad *et al.*, 1993.

The w/o-emulsification of the fresh crude oil is carried out based on the rotating cylinder method developed by Mackay and Zagorski, 1982. Oil (30 mL) and seawater (300 mL) are rotated (30 rpm) in a separating funnel (0,5 L), see Figure 3.2. The emulsification kinetics are mapped by measuring the water content at fixed rotation times. The maximum water content is determined after 24 hours of rotation.

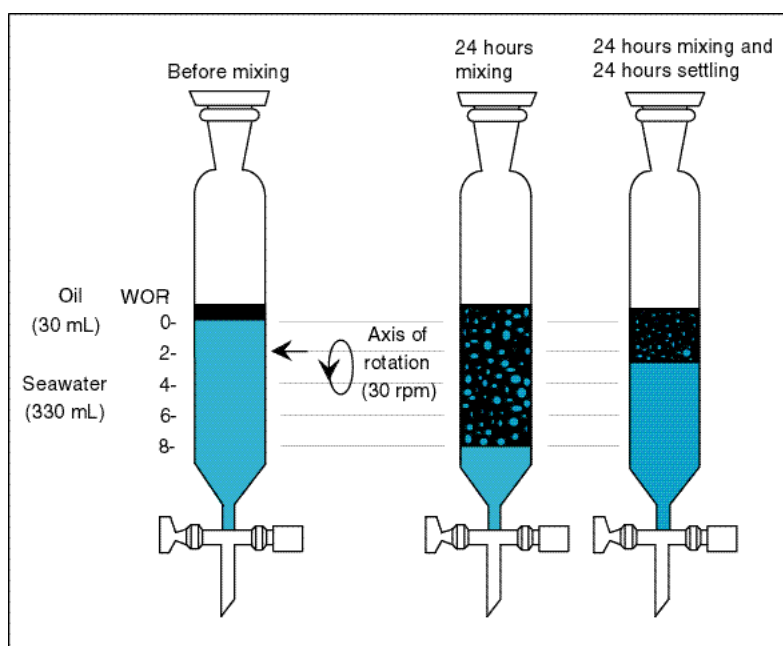


Figure 3.2: Principle of the rotating cylinder method.

To test the effectiveness of the emulsion breaker Alcopol O 60 %, the agent were added drop wise to the w/o-emulsion at a dosage of 2000ppm. After a contact period of 5 minutes and a rotation time of 5 minutes (30 rpm), the treated emulsion stood for 24 hours before the water drained from the emulsion was determined.

The distilled residues were emulsified with 50 vol% and 75 vol% water in addition to the maximum water content w/o-emulsion. Three parallel runs were performed to map the w/o-emulsion kinetics and one run were performed with the addition of Alcopol O 60 %. Viscosity at a range of shear rates were measured for all the emulsions prepared.



### Physical and chemical analysis

The viscosity, density, pour point and interfacial tension of the water free residues were determined. The analytical procedures used are given in Table 3.1.

*Table 3.1: Summary of the analytical methods used in the determination of the physical properties.*

Physical property	Analytical method	Instrument
Viscosity	McDonagh and Hokstad, 1995	Physica MCR 300
Density	ASTM method D4052-81	Anton Paar, DMA 4500
Pour Point	ASTM method D97	-
Interfacial tension	De Nouy Ring method	Kruss tensiometer

The wax content and “hard” asphaltene content are determined using the analytical procedures given in Table 3.2.

*Table 3.2: Summary of the analytical methods used in the determination of the chemical properties.*

Chemical property	Analytical method
Wax content	Bridiè <i>et al.</i> , 1980
“Hard” asphaltene	IP 234/84

## 4 Results of physical and chemical weathering properties

The experimental methods used are described in chapter 3.

### 4.1 Chemical and physical properties

Results from physical and chemical analysis of the fresh crude and the evaporated residue (250°C+) are shown in Table 3.1 and Table 4.2. The gas chromatograms are given in Figure 4.1

Table 4.1 Chemical properties for the fresh crude and evaporated residues of Linerle.

Oil	Residue	Asphaltenes "hard" (wt%)	Wax (wt%)
Linerle	Fresh	0,77	0,27
	250°C+	0,78	0,27

Table 4.2 Physical properties for the fresh crude and evaporated residues of Linerle.

Oil	Residue	Evaporated (vol.%)	Residue (wt. %)	Density (g/mL)	Pour Point (°C)	Viscosity 10 s <sup>-1</sup> (cP, 13°C)	Interfacial tension (mN/m)
Linerle	Fresh	0	100	0,951	-15	1470	10,2
	250°C+	0,4	99,5	0,953	-15	1410	12,1

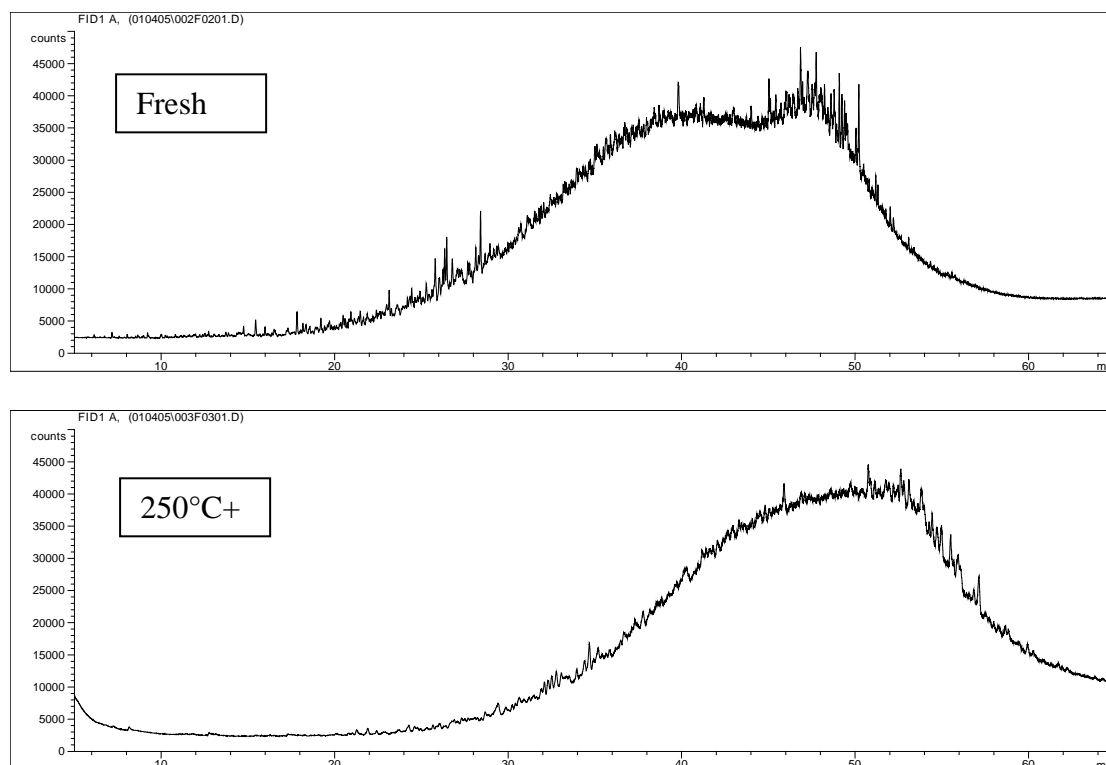


Figure 4.1: Gas chromatogram for fresh Linerle crude oil and the evaporated residue.

SINTEF has developed a concept for tentative categorisation of oils into 4 categories. These categories are:

- **Naphtenic oils**, characterised by a disrupted n-alkane pattern in the gas chromatogram due to biodegradation of the oil in the reservoir. The content of paraffines is therefore normally low.
- **Paraffinic oils**, often characterised by a low density which reflects a high content of light components (paraffines).
- **Asphaltenic oils**, with a high content of heavier components like asphaltenes and resins. The content of lighter components is correspondingly low, reflected by high density and low evaporation.
- **Waxy oils**, often exhibit high pour points due to a large content of wax components. At low temperatures these oils can have a tendency to solidify at the sea surface, especially if the sea water temperature is 10-15°C below the pour point.

A tentative categorisation of the Linerle oil in comparison with several other Norwegian crude oils is shown in Figure 4.2.

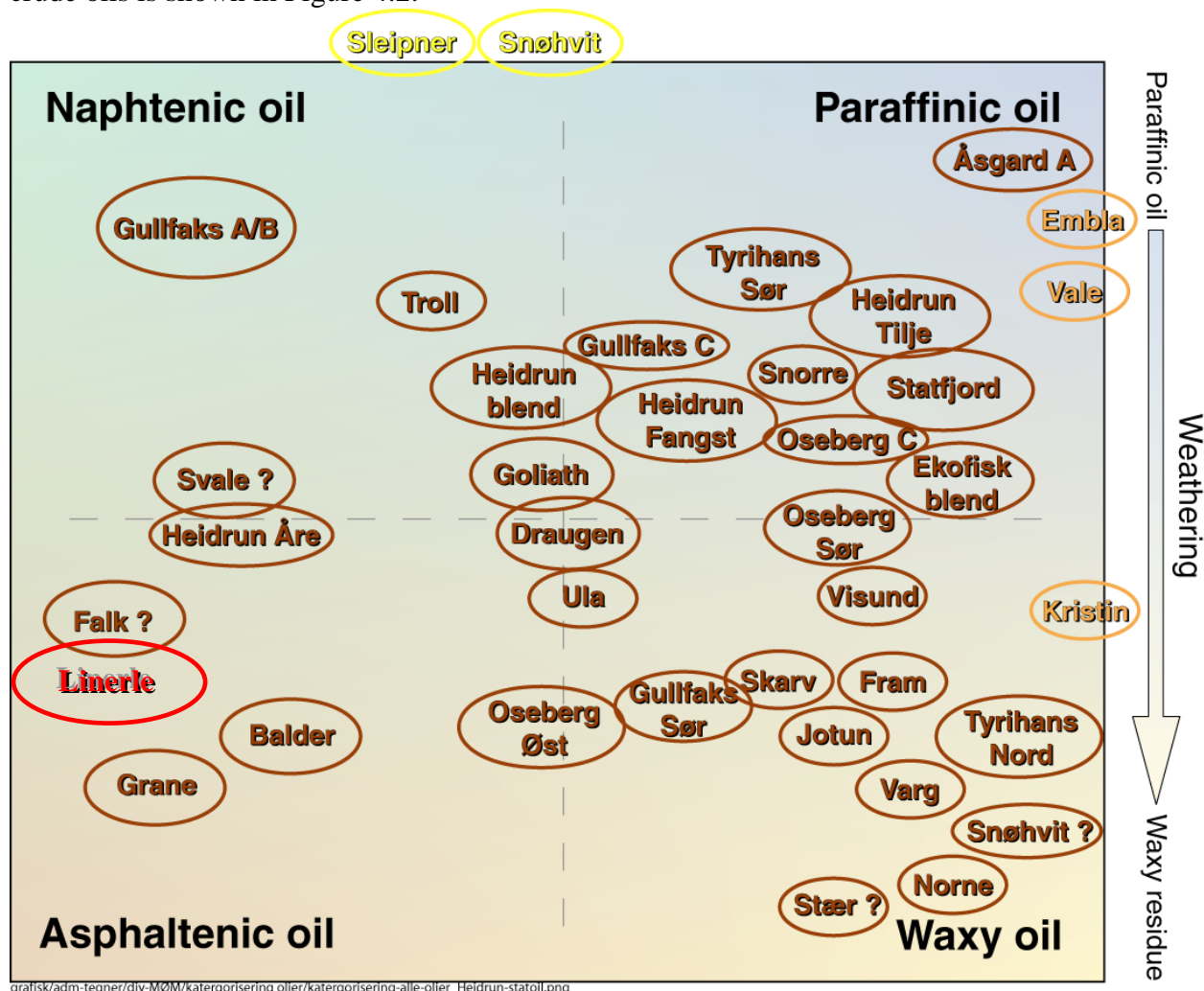


Figure 4.2: Categorisation of some Norwegian crude oils.

Linerle is categorised among the asphaltenic crudes. The asphaltene content (0,77 wt%) is lower than for Grane and Balder but higher than for Heidrun Åre. The chromatograms show that Linerle is highly biodegraded with no sign of n-alkanes whatsoever. The wax content is low at 0,27wt%.

Following the low content of light end components, Linerle also is extremely heavy at a density for the fresh oil at 0,95 g/ml. The pour point is low at -15°C for the fresh crude, and due to the low evaporative loss barely rising with weathering.

#### 4.2 W/o emulsification properties

The formation of w/o-emulsions on the sea surface delay the evaporation and the natural dispersion weathering processes increasing the lifetime for the oil on the sea surface. The emulsification properties of the Linerle oil are studied and the results form a basis as input for simulations of weathering behaviour

The  $t_{1/2}$  (time to emulsify half of maximum water content) is reported in table 3.7, the stability and effect of emulsion breaker in table 3.8, and the viscosities of residue and w/o-emulsions in table 3.9.

*Table 4.3 The calculated  $t_{1/2}$ -values and the maximum water content for Linerle w/o-emulsions at 13°C.*

Oil type		13°	
		$t_{1/2}$ [h]	Maximum water content [vol%]
Linerle	fresh	1,9	45
	250°C+	0,71	67

*Table 3.8 Water content (%) for w/o-emulsions of Linerle with and without the application of Alcopol O 60% (500 ppm and 2000 ppm) after 10 min settling and 24 hours settling with and without rotation at 13 °C.*

Residue	Emulsion breaker	Water in emulsion at 13°C (vol. %)		
		24 hours rotation	24 hours rotation and 10 min settling	24 hours rotation and 24 hours settling
fresh	none	46	46	42
250°C+	none	58	58	58
fresh	Alc. O 60 % 2000 ppm	46	33	6
250°C+	Alc. O 60 % 2000 ppm	62	48	6

Linerle forms stable emulsions, even at a short time of weathering on the sea surface. The emulsions are readily broken when treated with a 2000ppm dosage of the emulsion breaker Alcopol O 60%

*Table 4.4 Viscosities of the Linerle crude oil, residues and w/o-emulsions (0 vol%, 50 vol% and maximum water content) measured at 13°C.*

Residue	Water content 5°C/13°C (vol.%)	Viscosity at 13°C (cP)
		10 s <sup>-1</sup>
Fresh	0	1470
250°C+	0	1410
Fresh	50	8710
250°C+	50	7050
Fresh	45	7050
250°C+	67	16000

## 5 Prediction of physical properties

### 5.1 SINTEF Oil Weathering Model – the models and the input

#### 5.1.1 The SINTEF Oil Weathering Model

The laboratory data is used as input to SINTEF's Oil Weathering Model (OWM) in order to predict the behavior of the oil at sea. The SINTEF OWM relates oil properties to a chosen set of conditions (oil/emulsion film thickness, sea state and sea temperature) for a surface release and predicts the rate in change of an oil's properties and behavior on the sea surface. The SINTEF OWM is schematically shown in Figure 5.1

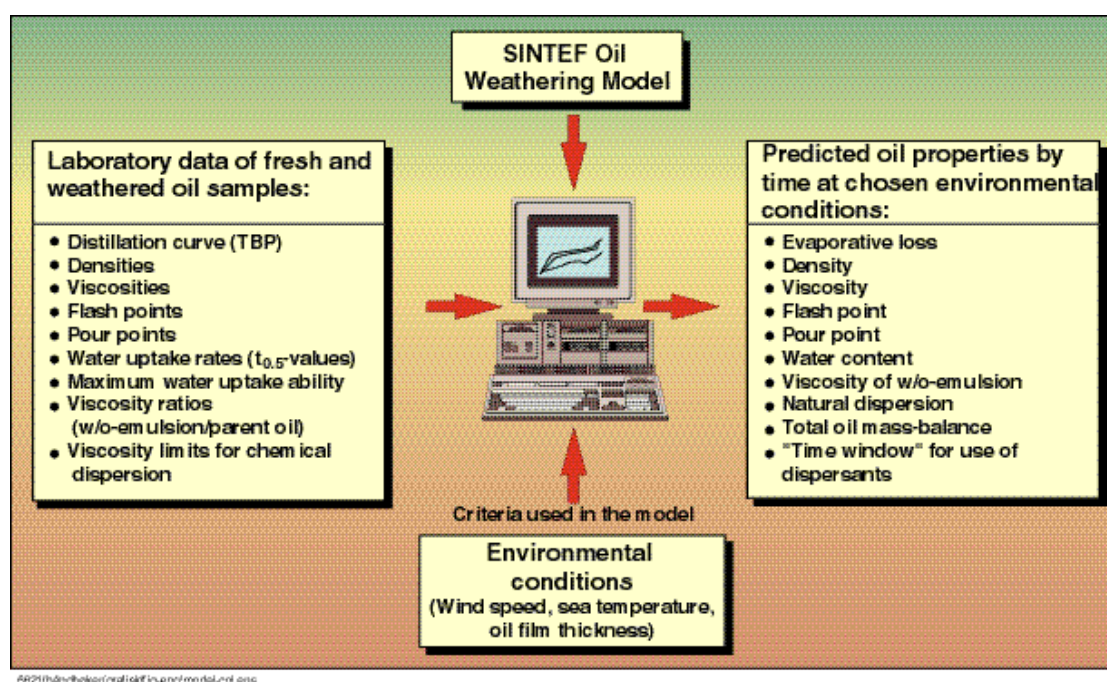


Figure 5.1: The SINTEF Oil Weathering Model.

The OWM is described in detail in Aamo *et al.*, 1993. Verification of the model with field experiments is described in Daling and Strøm, 1999.

#### Oil film thickness

In the SINTEF OWM the oils are categorised into condensates, emulsifying crudes, low emulsifying crudes, heavy bunker fuels or refined distillates based on experimental results obtained in the bench-scale testing. The terminal film thickness is set according to these categories based on experimental field experience. As Linerle is an emulsifying crude oil a terminal film thickness of 1 mm is used for Linerle. In the predictions an initial film thickness of 20mm is used. The OWM can also predict the initial film thickness based on release condition data (release rate, gas/oil-ratio and release depth).

**Sea temperature**

The prevailing weather conditions greatly influence the weathering rate of oil on the sea surface. The temperature used in this report is 5°C and 15°C. 5°C is a typical minimum winter temperature in the North sea, while 15°C is considered a typical maximum summer temperature.

**Wind speed**

The relation between the wind speed and the significant wave heights used in the prediction charts obtained from the SINTEF OWM are shown in Table 5.1.

*Table 5.1: The relation between the wind speed and the significant wave heights 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



### 5.1.2 Input to SINTEFs Oil Weathering Model

Geographical area:	<b>Linerle</b>
Initial oil film thickness:	20 mm
Terminal oil film thickness:	1 mm
Release rate:	1,33 metric tons/minute
Sea temperature:	5°C and 13°C
Wind speed:	2 m/s, 5 m/s, 10 m/s and 15 m/s

The data used as input to the SINTEF OWM for Linerle crude oil are given in Table 5.2 to Table 5.4.

Table 5.2: Physical and chemical data obtained for the fresh Linerle crude..

<b>Properties of fresh oil:</b>	
Gravity (°API)	
Specific Gravity (60 F/60 F)	0,951
Density correction factor	
Total Sulphur (wt. %)	
Mercaptan Sulfur (ppm wt.)	
Total Nitrogen (wt. %)	
Pour Point (°C)	-15
Reference temperature #1 (°C)	13
Viscosity at ref. temp.#1 (cP)	
Reference temperature #2 (°C)	
Viscosity at ref. temp.#2 (cP)	
Vanadium (ppm wt.)	
Nickel (ppm wt.)	
Conradson Carbon (wt. %)	
Asphaltenes (wt. %)	0,77
n-Pentane Insolubles (wt. %)	
Reid Vapor Pressure (psia)	
Flash Point (°C)	
Hydrogen Sulfide (ppm wt.)	
Neutralization Num.(mg KOH/g)	
Bottom Water & Sediment (LV %)	
Ash Content (wt. %)	
Salt as NaCl (lbs/1000 bbls)	
Wax Content (wt. %)	0,27
Dispersable for visc. <	
Not dispersable for visc. >	
Reference temperature #3 (°C)	
Reference temperature #4 (°C)	
Maximum water uptake (%)	

*Table 5.3 The true boiling point curve used for the Linerle Crude. The TBP used is obtained from the Crude Assay of the oil dated 30.08.04*

Temperature	Volume(%)
15	0.14
65	0.14
90	0.14
150	0.14
180	0.14
240	1.99
320	14.51
375	34.57
420	48.61
525	81.38
565	87.68

*Table 5.4 Laboratory weathering data at 13°C.*

<b>PROPERTY</b>	<b>Fresh</b>	<b>150°C+</b>	<b>200°C+</b>	<b>250°C+</b>
Boiling temp. (°C)	0	200	250	300
Volume topped (%)	0	0	0	0,4
Residue (wt. %)	100	100	100	99,5
Specific gravity (g/l)	0,951	0,952	0,952	0,953
Pour point (°C)	-15	-15	-15	-15
Flash point (°C)				
Viscosity at 13°C (cP)	1470	1470	1470	1470
Viscosity of 50% emulsion at 13°C (cP)		8710		7050
Viscosity of 75% emulsion at 13°C (cP)				
Viscosity of max water at 13°C (cP)		7050	10000	16000
Max. water content at 13°C (%)		45	60	67
Halftime for water uptake at 13°C (hrs)		1,9	1,9	0,71
Stability ratio at 13°C		0,14	0,14	0,05

## 5.2 The weathering properties of Linerle

### 5.2.1 How to use the prediction charts, an example

If Linerle has drifted for a period of time on the sea surface the prediction charts can be used to determine the remaining oil's chemical, physical and emulsifying properties. Table 5.5 shows examples for the following scenario:

- Drift time: 24 hours
- Temperature: 5°C / 15°C
- Wind speed: 10 m/s

*Table 5.5: Weathering properties for Linerle obtained from the prediction charts.*

Property	Temperature [5°C]	Temperature [15°C]
Evaporation	2%	4%
Pour point	15°C	15°C
Water content	51 %	62 %
Viscosity of the emulsion	15900 mPas	10700 mPas

### 5.2.2 Prediction charts for Linerle

The predictions shown for Linerle are :

- Evaporation* (Figure 5.2)
- Pour point* (Figure 5.3)
- Water content* (Figure 5.4)
- Viscosity of emulsion* (Figure 5.5)
- Mass Balance Figure* (Figure 5.6-5.9)

The weathering behaviour of Linerle is compared with Norne, Åsgard, Heidrun blend and Heidrun Åre in Figure 5.10 – 5.13.

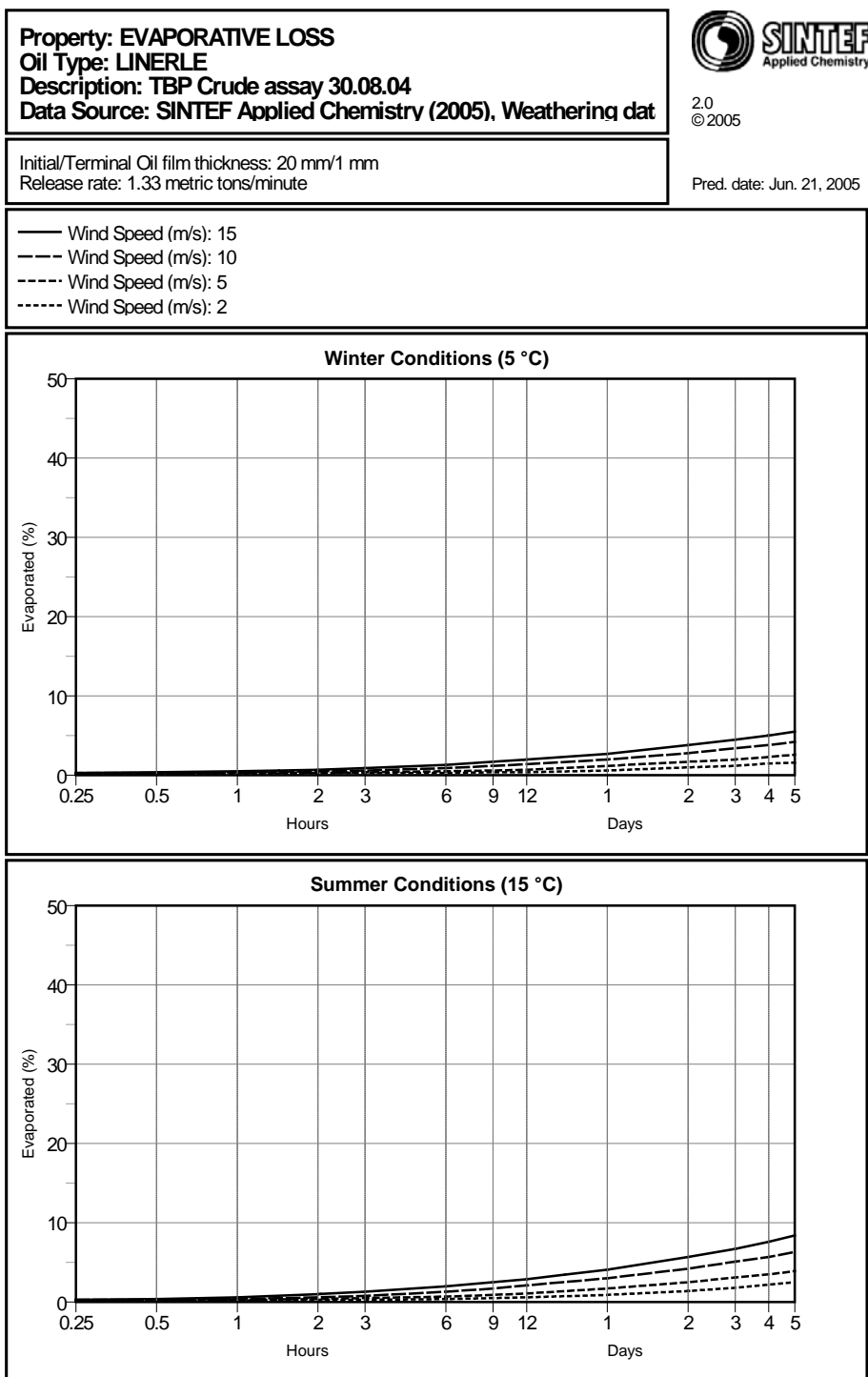


Figure 5.2 Predicted evaporation of Linerle at 5°C and 15°C sea temperature.

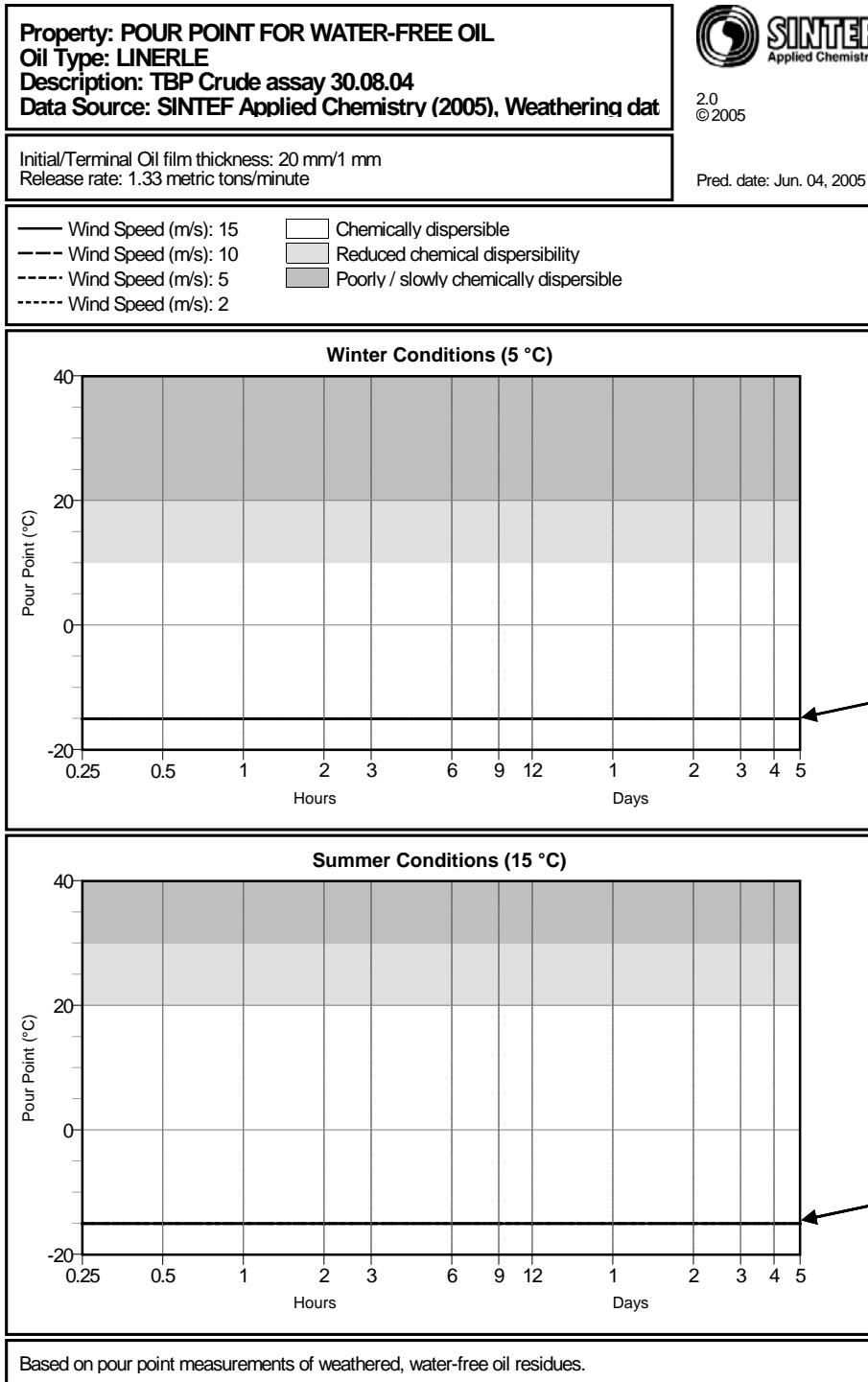


Figure 5.3 Predicted pour point of Linerle at 5°C and 15°C sea temperature.

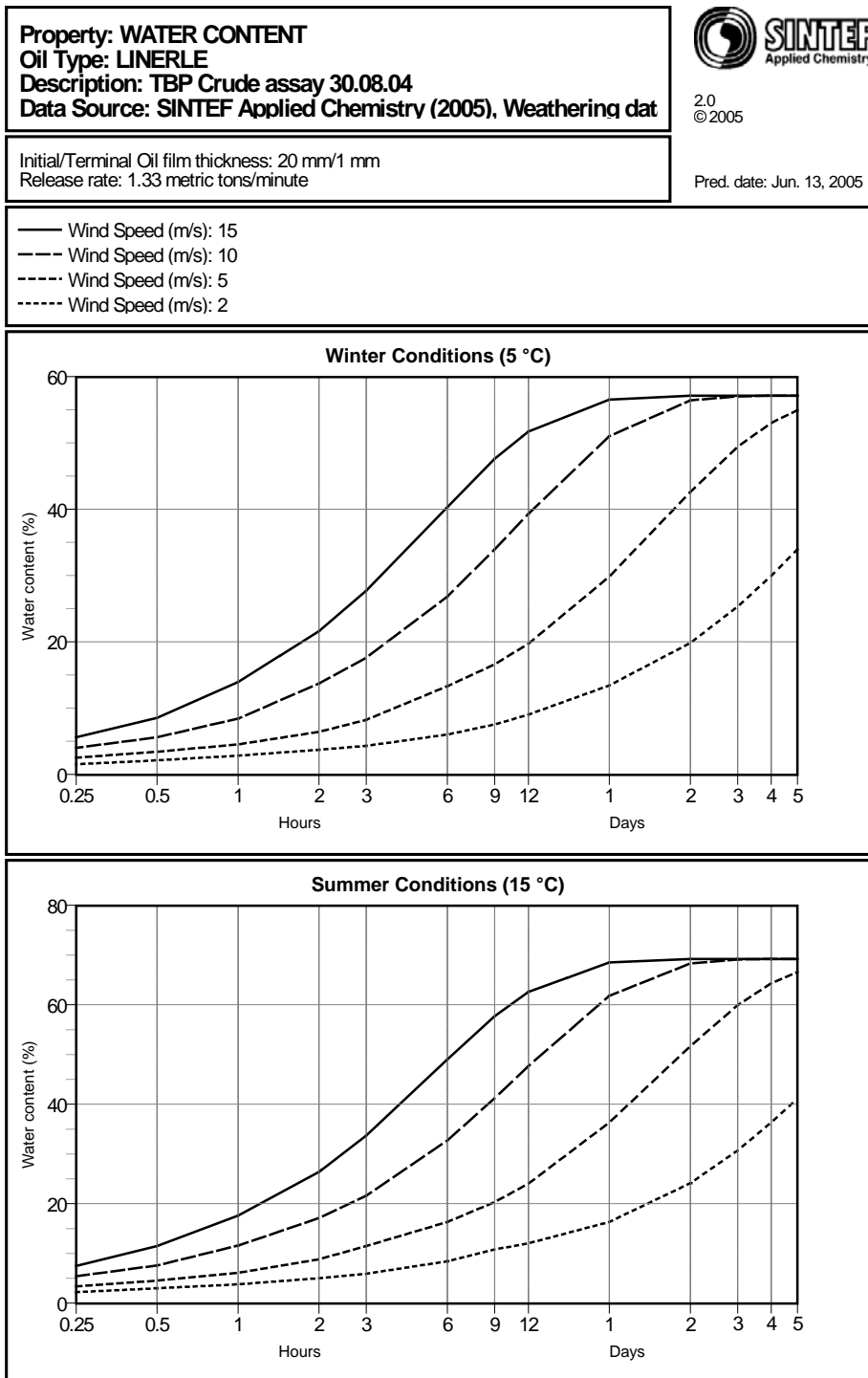


Figure 5.4 Predicted water content of Linerle at 5°C and 15°C sea temperature.

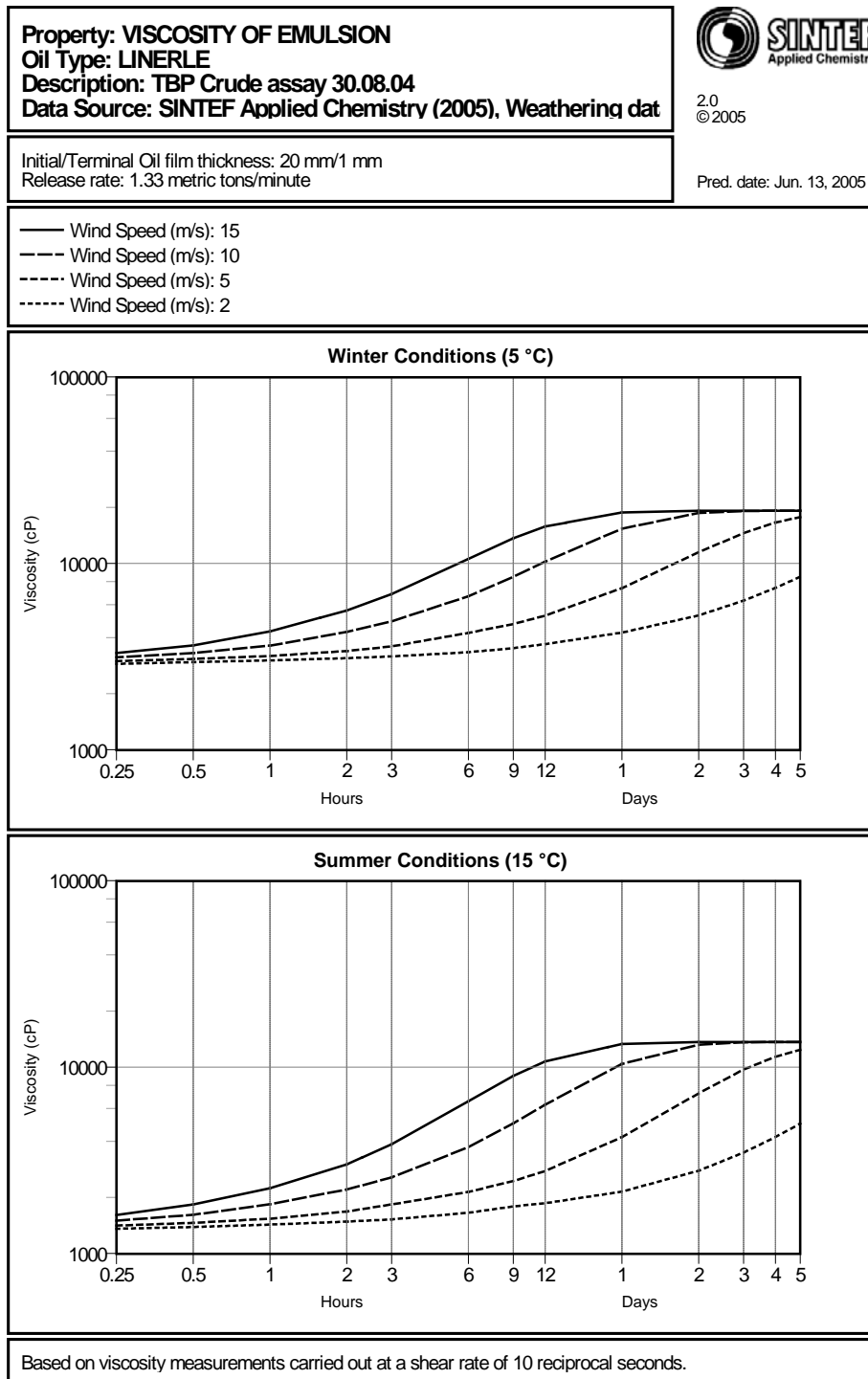
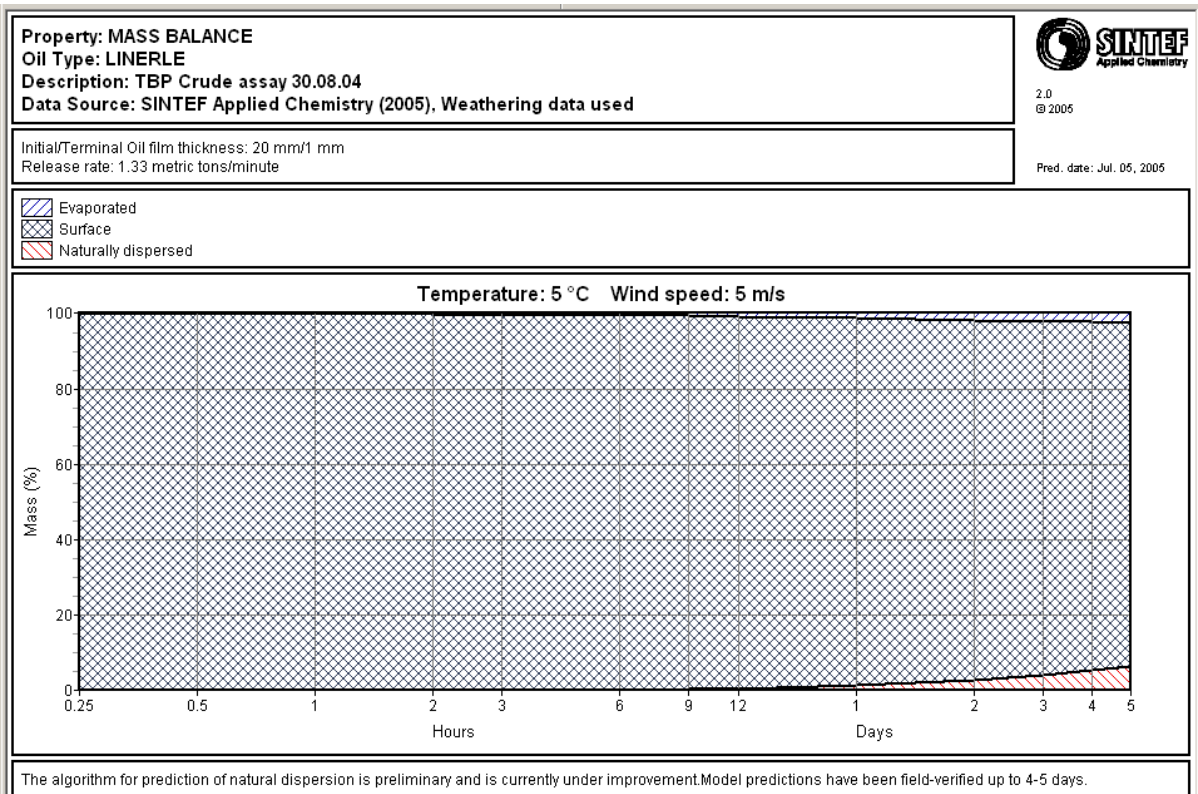
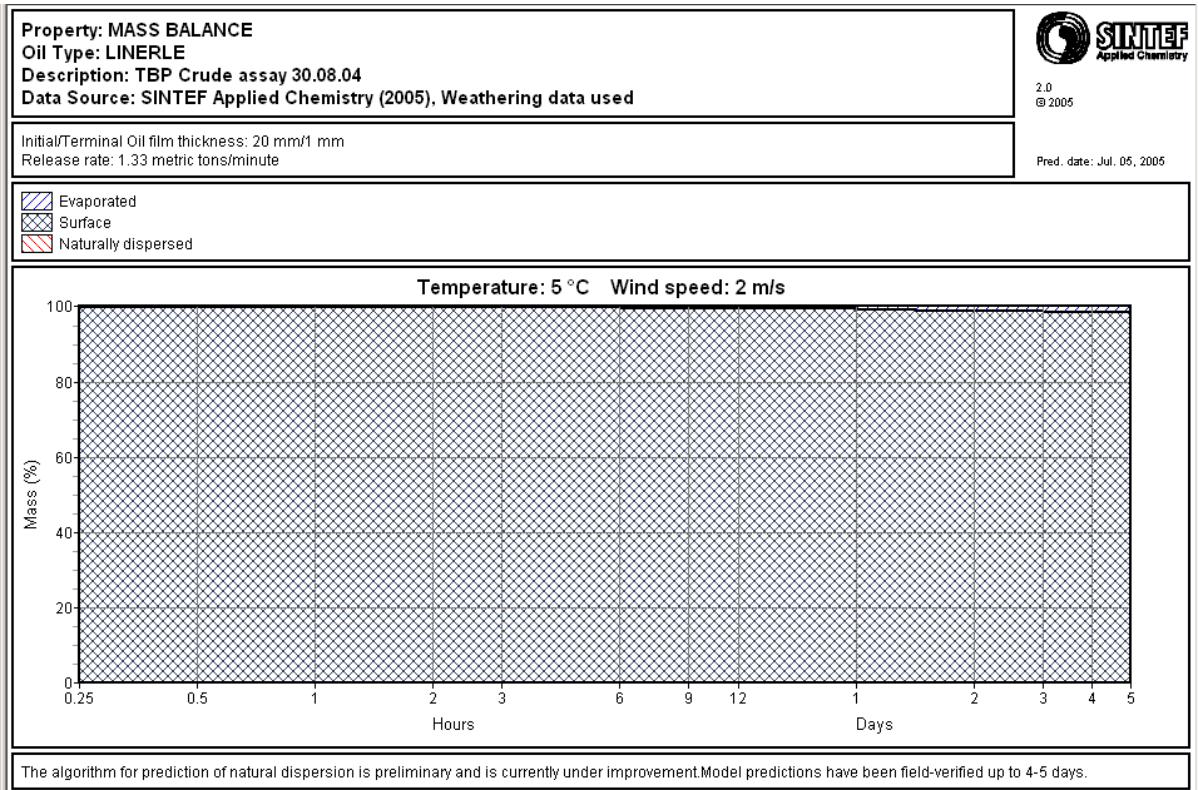


Figure 5.5 Predicted emulsion viscosity of Linerle at 5°C and 15°C sea temperature. Viscosities are measured at a shear rate of  $10 \text{ s}^{-1}$ .



*Figure 5.6 Predicted mass balance for Linerle at 5°C and wind speeds at 2 m/s and 5 m/s, respectively.*



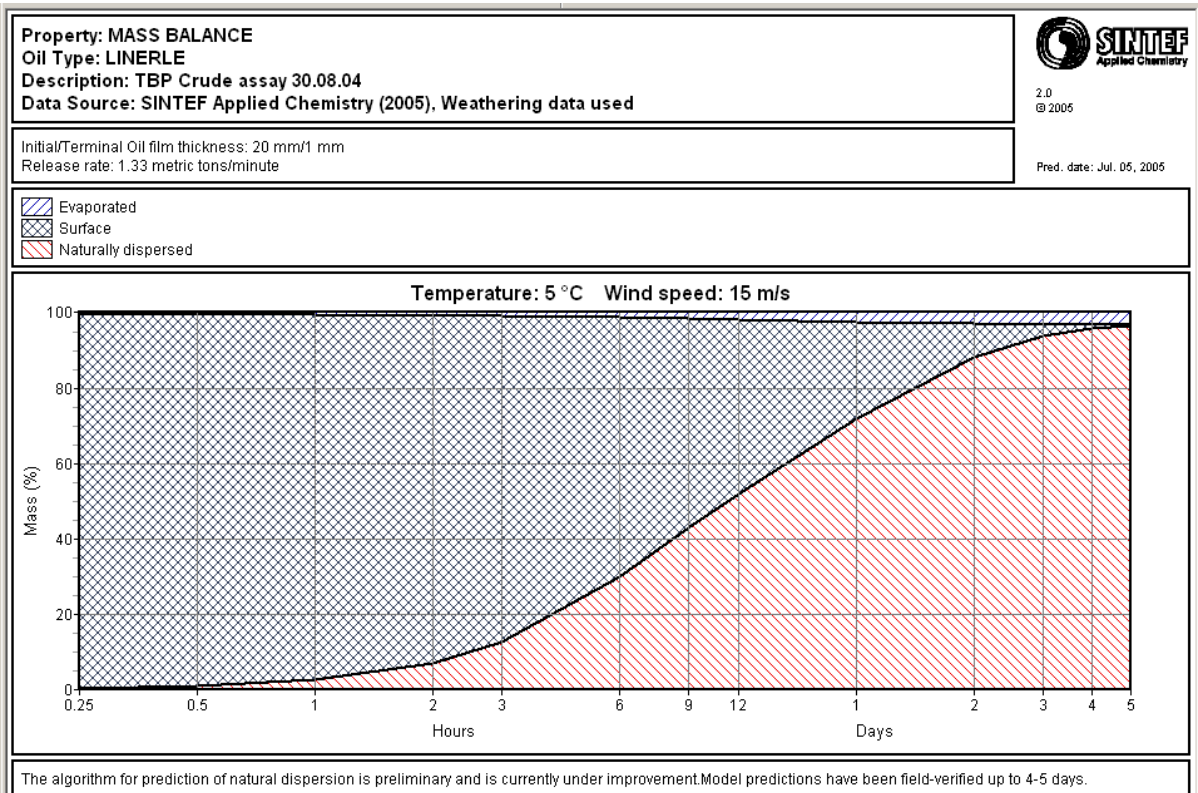
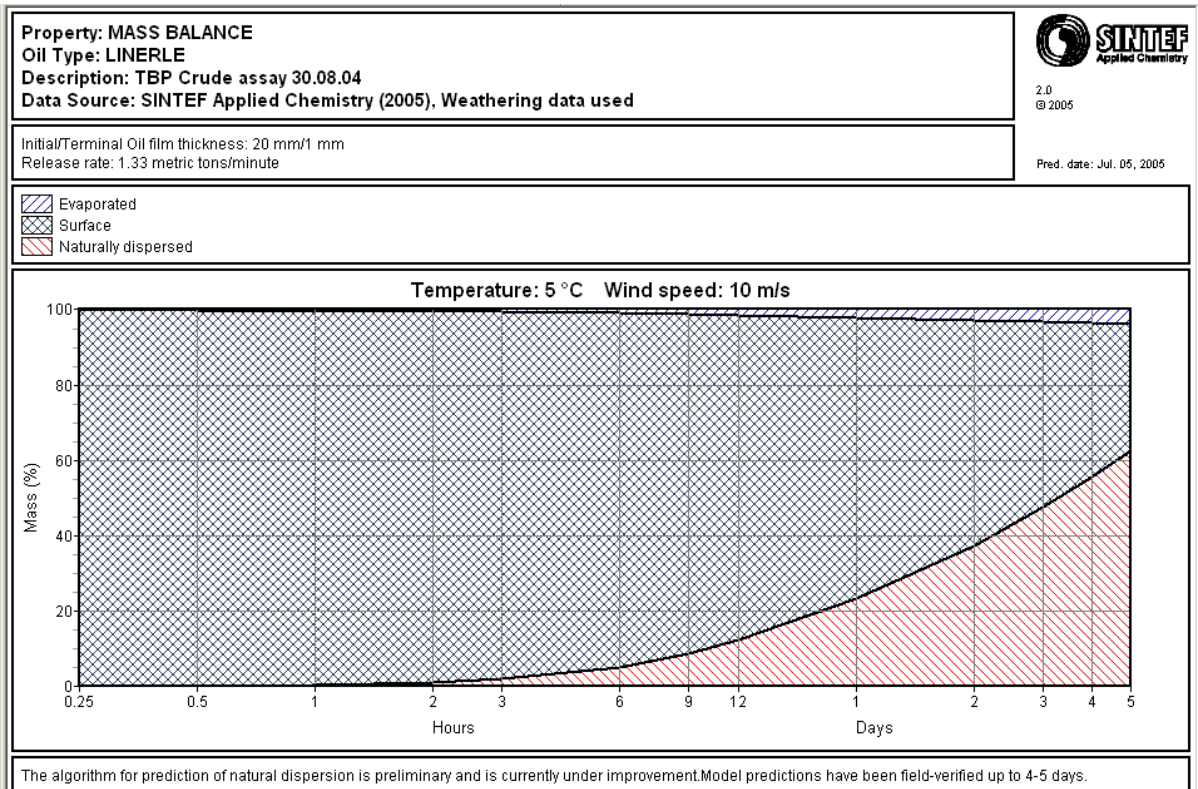


Figure 5.7 Predicted mass balance for Linerle at 5°C and wind speeds at 10 m/s and 15 m/s, respectively.

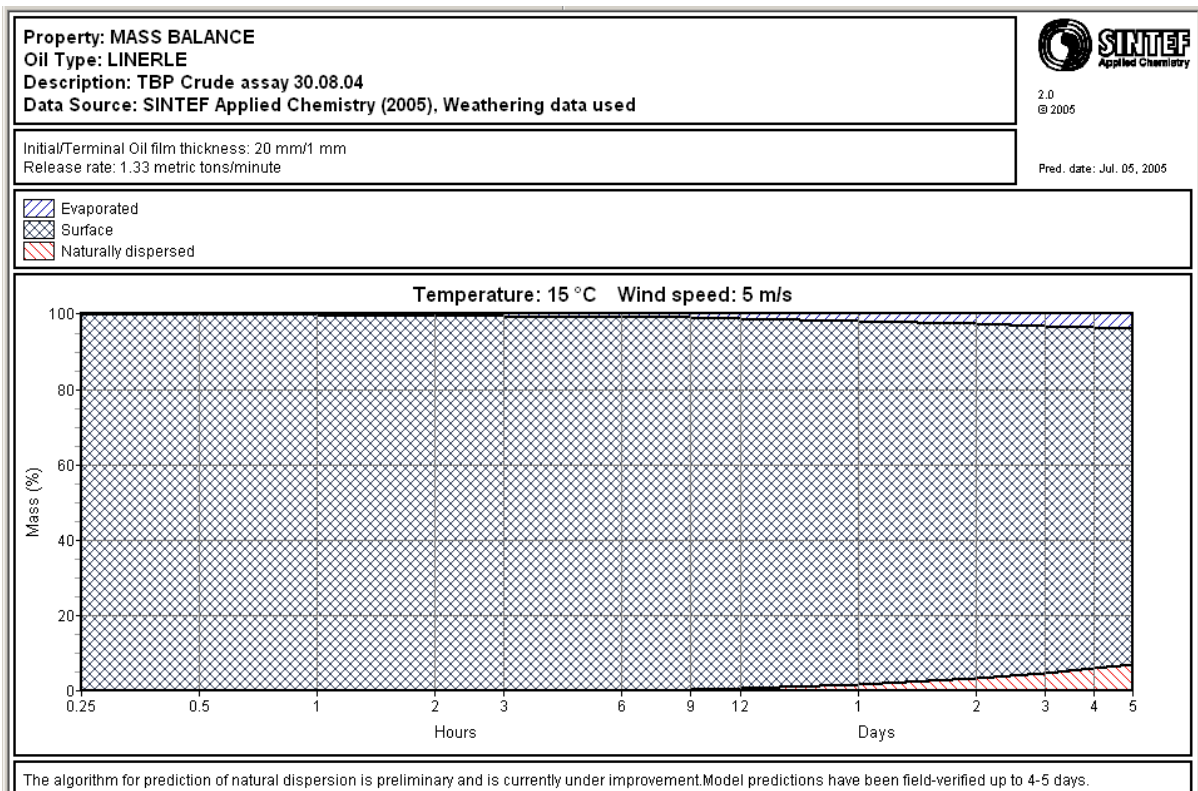
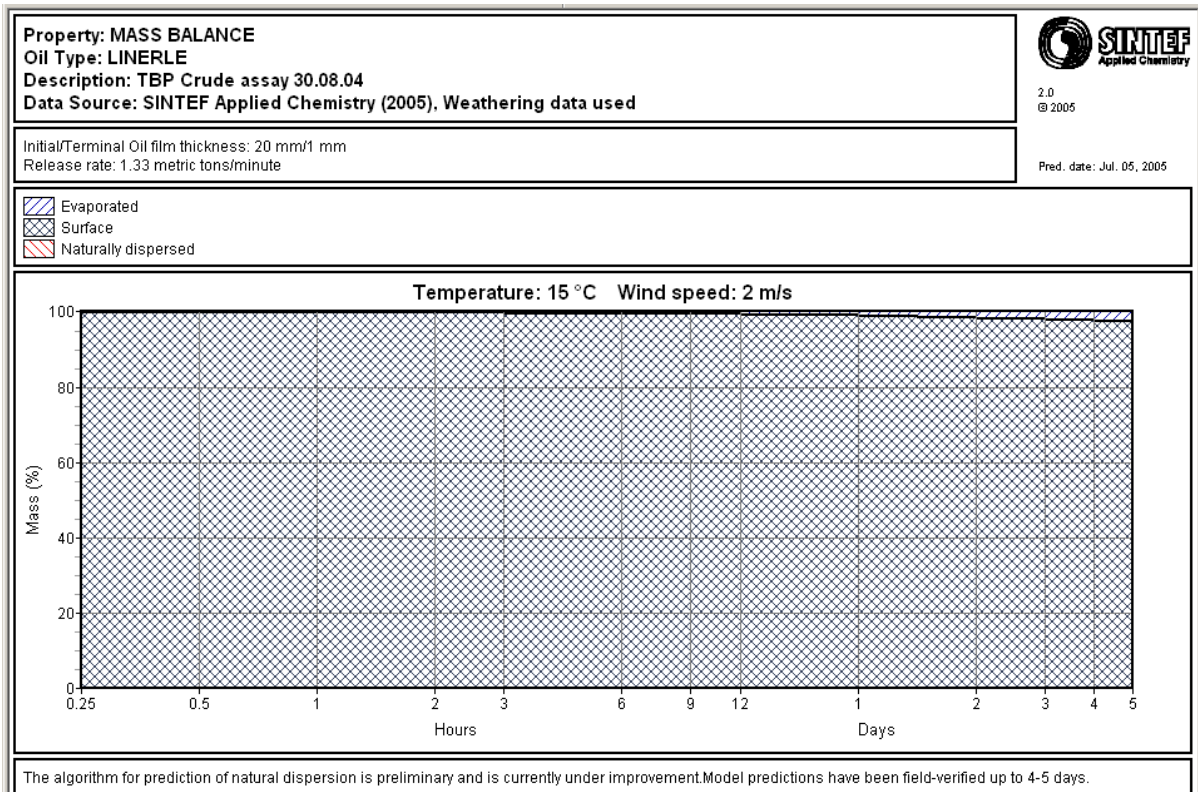


Figure 5.8 Predicted mass balance for Linerle at 15°C and wind speeds at 2 m/s and 5 m/s, respectively.

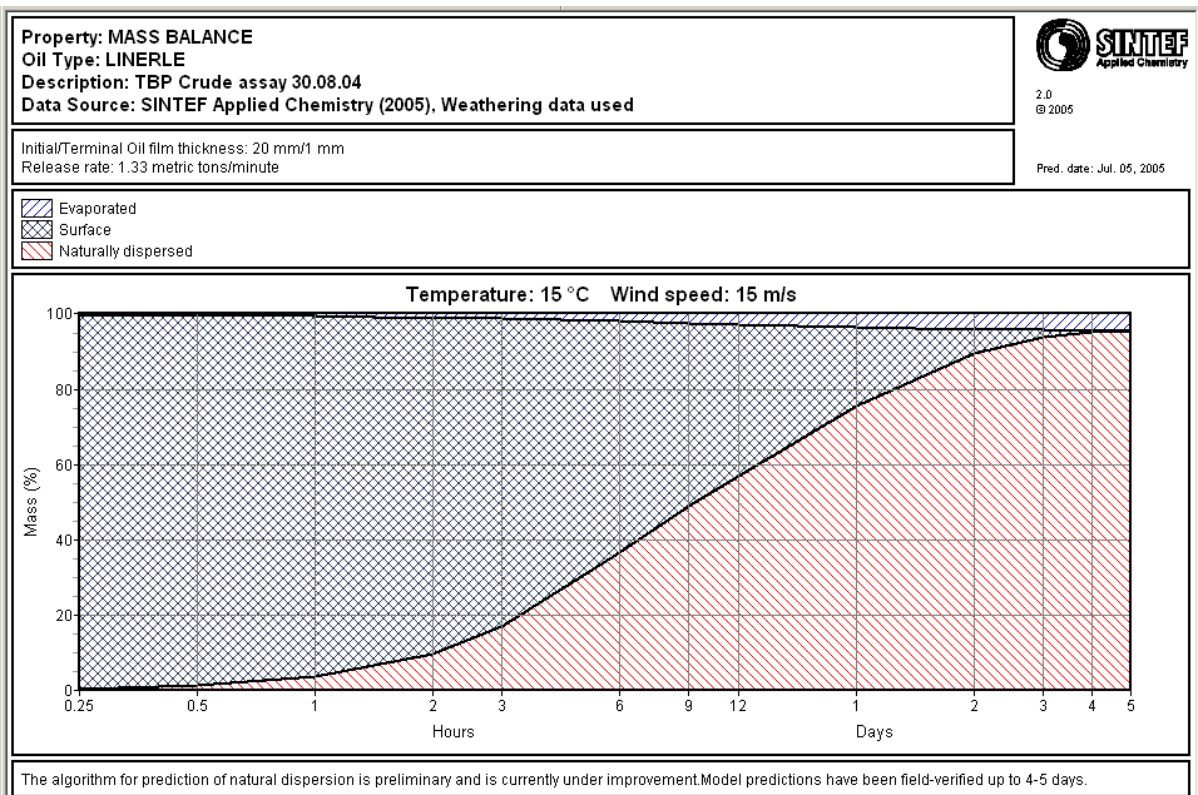
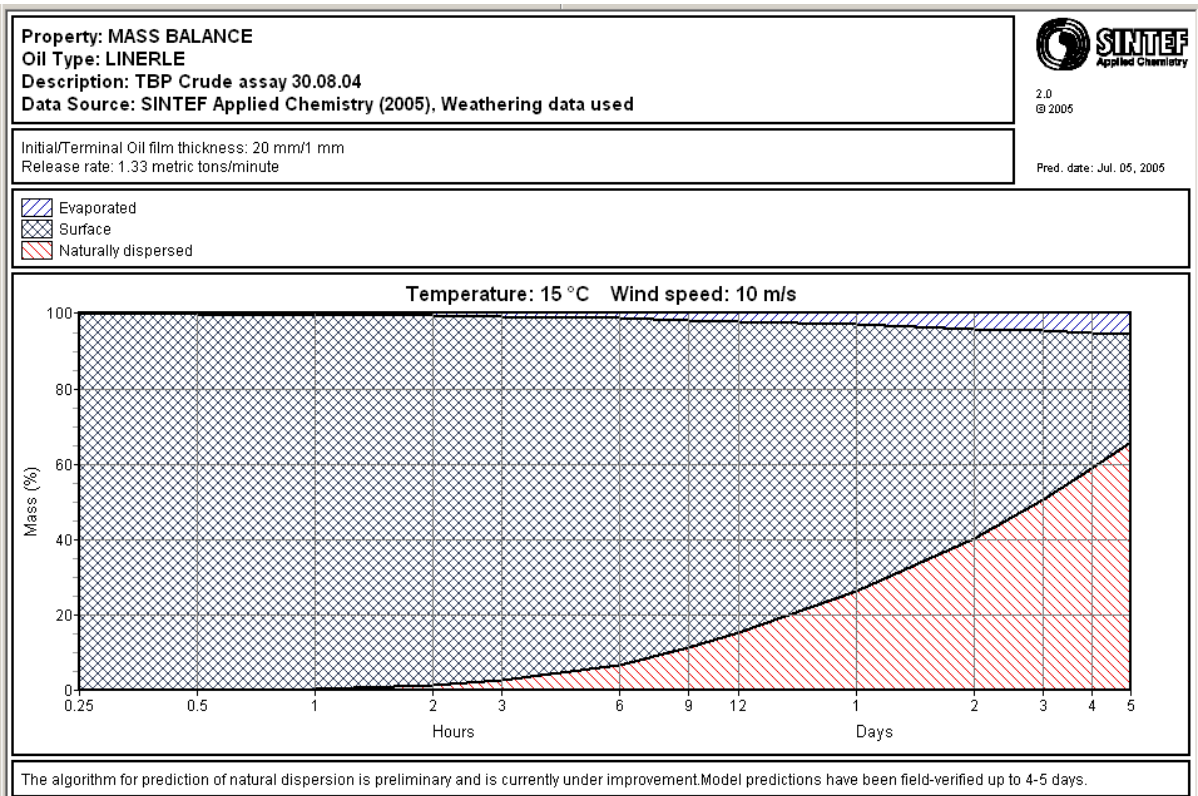


Figure 5.9 Predicted mass balance for Linerle at 15°C and wind speeds at 10 m/s and 15 m/s, respectively.

### 5.2.3 Comparison between weathering properties for different Norwegian crudes

A comparison between the predicted physical properties for different Haltenbanken crudes is given in the figures below. All predictions are performed for a sea temperature of 15°C and a wind speed of 10 m/s.

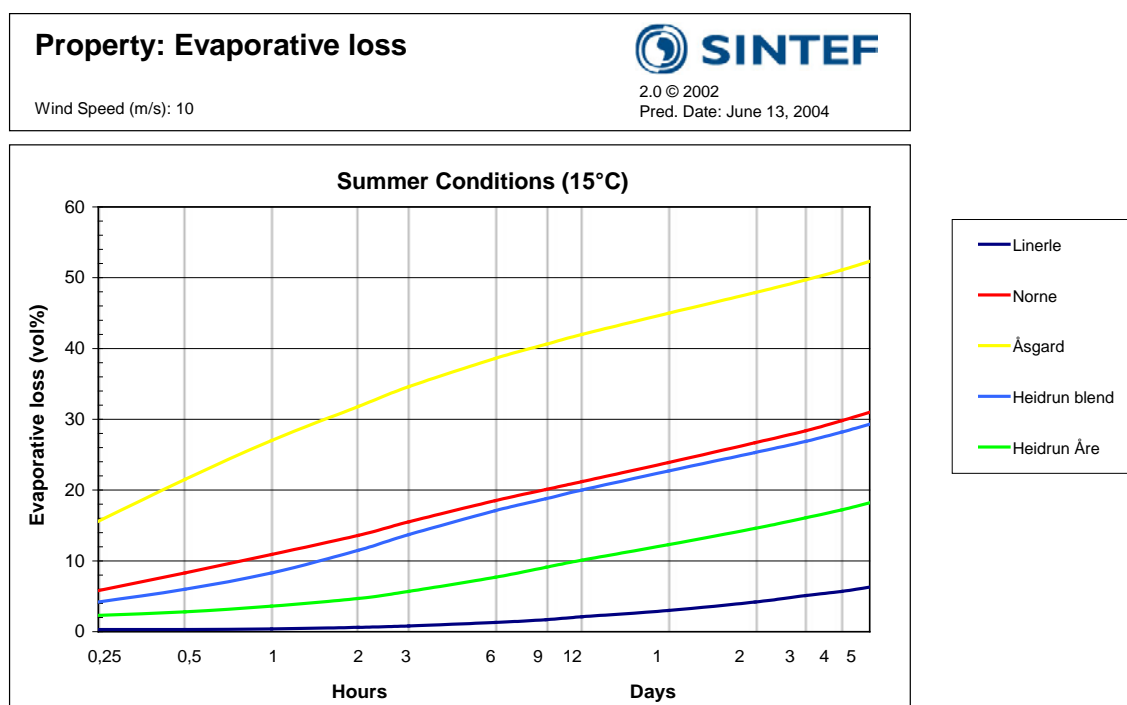


Figure 5.10 Predicted evaporative loss for Linerle compared with Norne, Åsgard, Heidrun blend and Heidrun Åre. Predictions are done for a sea temperature of 15°C and a wind speed of 10 m/s.

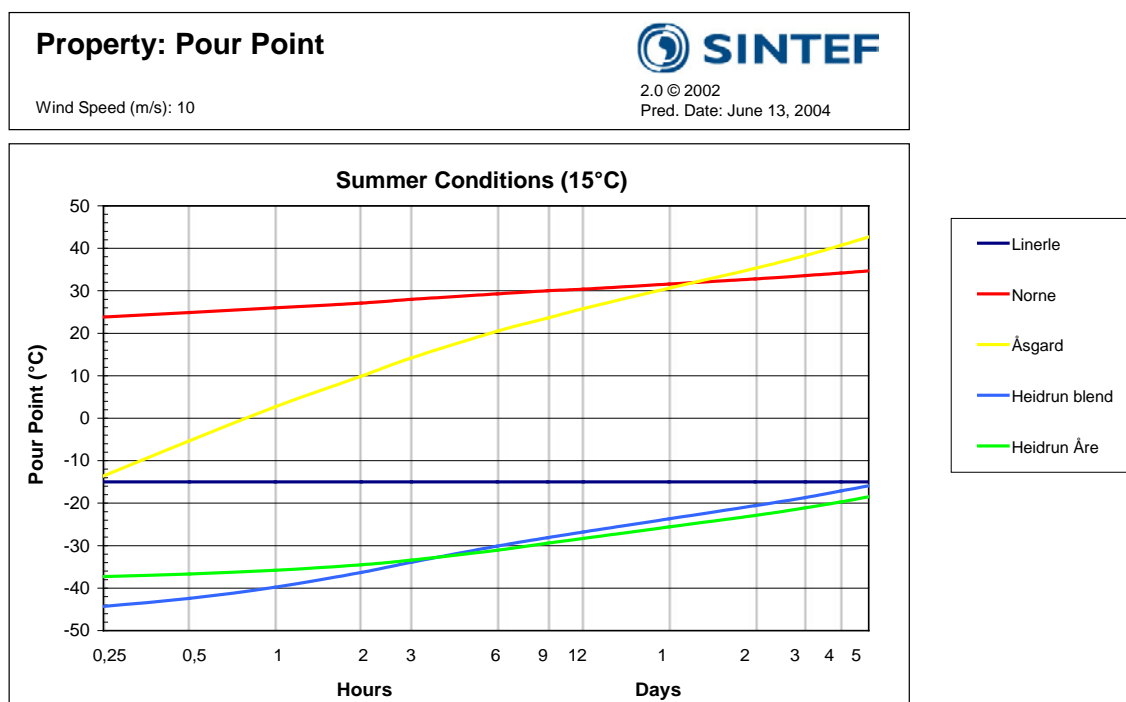


Figure 5.11 Predicted pour point for Linerle compared with Norne, Åsgard, Heidrun blend and Heidrun Åre. Predictions are done for a sea temperature of 15°C and a wind speed of 10 m/s.

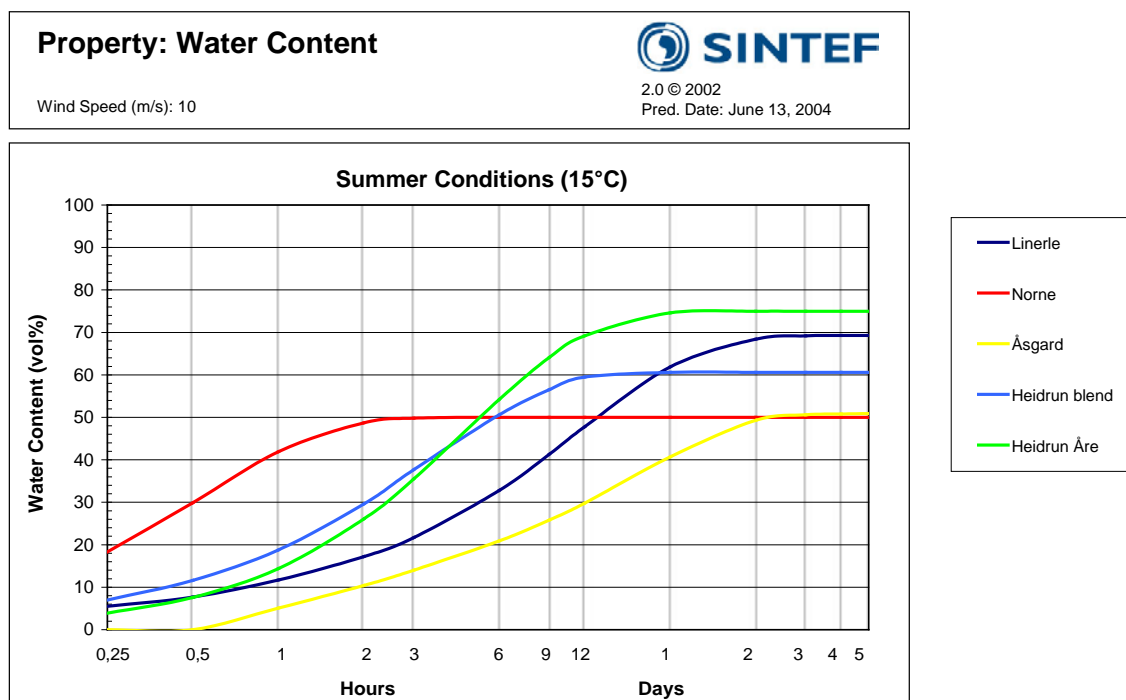
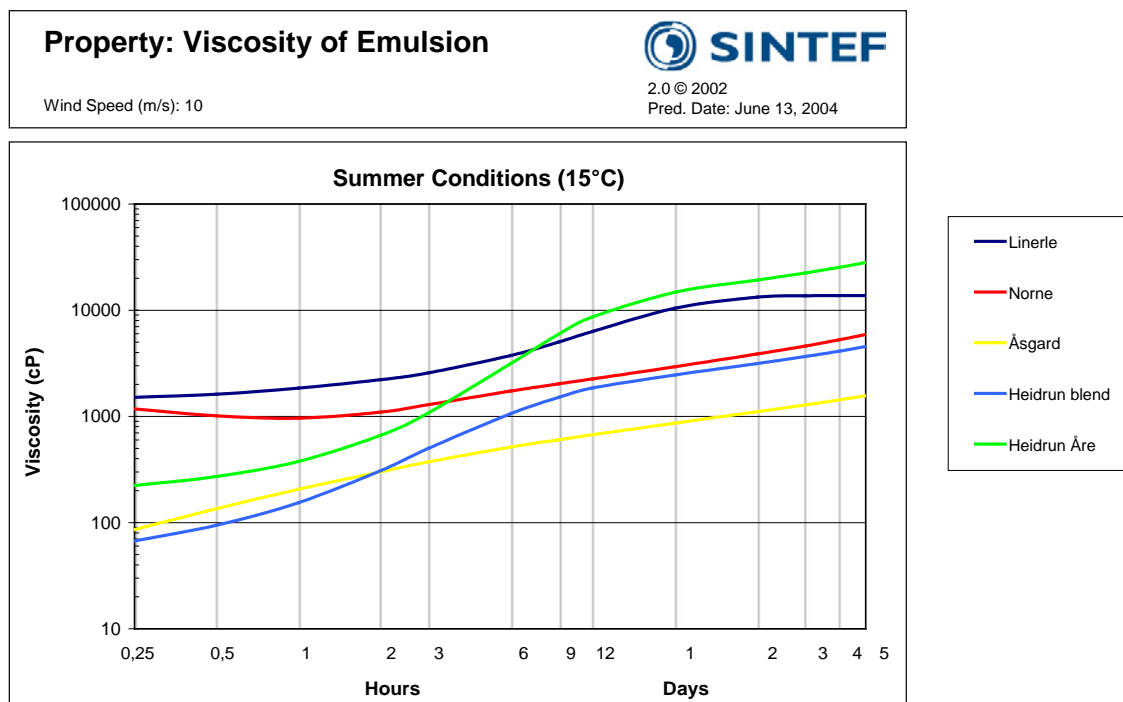


Figure 5.12 Predicted water uptake for Linerle compared with Norne, Åsgard, Heidrun blend and Heidrun Åre. Predictions are done for a sea temperature of 15°C and a wind speed of 10 m/s.



*Figure 5.13 Predicted emulsion viscosity for Linerle compared with Norne, Åsgard, Heidrun blend and Heidrun Åre. Predictions are done for a sea temperature of 15°C and a wind speed of 10 m/s. The viscosity is predicted for a shear rate of  $10s^{-1}$ .*

Due to the extremely low content of light end components the evaporative loss of Linerle is low compared to the other crudes in the comparison. As evaporation is low changes in the properties of the water free residue will also be low. This means changes in the viscosity and pour point of the water free residues will be almost non-existing.

Linerle has a slow water uptake, but reaches a maximum water content of 70% which is relatively high compared to the other oils in the comparison. Combined with the initial high viscosity of the Linerle crude, the emulsion viscosity reaches a viscosity above 10.000 after days of weathering, only surpassed by Heidrun Åre in the comparison.

## 6 Weathering properties of Linerle

Linerle is a highly biodegraded asphaltenic crude. Linerle is extremely heavy compared to other Norwegian crude oils, and will only lose a few percents of its mass due to evaporation on the sea surface.

The Linerle oil has shown a maximum water uptake of 67% in laboratory studies at summer temperatures, and a predicted maximum water content of 56% at winter temperatures. The emulsification rate of Linerle is slow, and the maximum water content is not reached until 2 days at a wind speed of 10 m/s. The emulsions formed are stable, and addition of emulsion breaker will be necessary to break the emulsion.

A lower viscosity limit for efficient boom recovery is stated at 1000 mPas (Norvik *et. al.* 1997). Due to the high viscosity of the fresh oil the Linerle oil / emulsion will never be below this limit.

The pour point of the Linerle emulsions are low (-15°C), and solidification or flowability problems is not considered a problem for Linerle. The emulsions formed do, however, have a high viscosity. Basin trials at SINTEF (Leirvik *et. al.* 2001), and field studies (Daling *et. al.* 2005) have shown limitations in recovery efficiency at viscosities as low as 10.000 mPas. As an example, this limit is reached after respectively 12 hours and 1 day at summer and winter temperatures at a wind speed of 10 m/s. The higher viscosities at summer temperatures are due to the higher and faster water uptake of the emulsion.

For crude oils with a low initial viscosity and a high content of light end components, a high degree of natural dispersion and evaporative loss is expected. As Linerle has a high initial viscosity and almost no volatile components, a longer lifetime on the sea surface is predicted compared to other Norwegian crudes.

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