

Report

Weathering Properties and Toxicity of Marine Fuel Oils

Summary Report

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ABSTRACT

Weathering properties of six marine fuel oils have been investigated; four diesel qualities (three DMA-qualities, one DMZ/RMA-quality) and two so-called hybrid fuel oils. The diesel fuel oils are often used in Norwegian waters and in arctic areas, and the weathering behaviours at sea of these qualities in representative summer and winter temperatures were investigated. Hybrid fuel oils are new fuel products designed to be used in heavy fuel oil machinery/engines, and to meet recent restrictions in regulations regarding sulphur emission. To our knowledge, comprehensive testing of Hybrid fuel oils have not previously been performed.

The chemical composition and emulsifying properties as well as dispersibility (both natural and dispersant enhanced) and potential for *in-situ* burning (ISB) of the fuel oils have been investigated, in addition to the water accommodated fractions (WAF) of the fuel oils and their toxicity.

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Preface from the Norwegian Coastal Administration (NCA)

The Norwegian Coastal Administration has a strategic focus on R&D activities related to arctic oil spill response. Increased knowledge as well as technology development contributes to better preparedness, in case of an oil spill in the vulnerable arctic environment.

Regulations to ban heavy fuel oil in the national parks and nature reserves of Svalbard are in place. However, also countermeasures to spills of lighter distillate fuels need to be addressed in order to minimize the environmental impacts of a potential spill.

Furthermore, this report also presents data on another emerging topic: New generation fuel oils that are entering the markets as a response to the efforts to prevent air pollution from ships. These new fuel oils allow ships to comply with the revised MARPOL Annex VI regulations with regard to sulphur emissions. However, so far little attention has been drawn to their characteristics in a spill situation.

This report and the supporting material contributes significantly to NCAs continuous work to narrow the knowledge gaps that are emphasized in the white Paper: Meld. St. 35 (2015–2016) «På rett kurs — Forebyggende sjøsikkerhet og beredskap mot akutt forurensning». Together with tests performed at the National Centre for testing of oil spill equipment this work adds to the knowledge of behaviour, toxicity and response options for distillate marine fuel oils and new generation low sulphur fuel oils, and leaves us better prepared in case of a future spill in the Svalbard region or along the coast of mainland Norway.

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1 Executive summary

The weathering and toxicity properties with relevance for operative oil spill response of six different marine fuel oils have been investigated through this project. Response operations such as application of oil spill dispersants, mechanical recovery and *in-situ* burning (ISB) has been evaluated. The six fuel oils involved three DMA-qualities (called Gas Oil (GO), Marine Gas Oil (MGO) and "Rotterdam" diesel) and a Wide Range Gas oil (WRG). DMA-qualities (as defined by ISO 8217:2017) are allowed used within the nature reserves and national parks around Svalbard, while the WRG is not allowed within this region. These fuel oil qualities are otherwise commonly used in Arctic regions and along the Norwegian coast (Kystverket, 2017). In addition to these distillate fuel oils, two hybrid fuel oils, ExxonMobil HDME 50 (Heavy Distillate Marine ECA 50) and Shell ULSFO (Ultra Low Sulphur Fuel Oil), have been investigated. The abbreviation ULSFO has recently been used as a collective name for Hybrid fuel oils in discussions related to the new ISO 8217:2017 standards. In this report, ULSFO has been used solely to describe the Shell ULSFO product, while hybrid fuel oils has been used as a general term.

Hybrid fuel oils is a term used for fuel oils that cannot easily be classified within the ISO 8217 standards, and exhibit properties from both light distillate fuels and heavier fuel qualities. Hybrid fuel oils are produced and used in increasing rate as a consequence of stricter regulation regarding sulphur emission from marine vessels, both within and outside Sulphur Emission Control Areas (SECA). Figure 1-1 depict the changes in regulations regarding sulphur content in marine fuels, and show both past and future developments. The use of hybrid fuel oils is only one of several measures available for meeting the new sulphur emission regulations. Other measures can be installation of scrubbers on existing machinery for continued use of traditional heavy fuel oils (HFO) with sulphur content higher than the allowed limits in the respective marine areas. Modification of machinery for use of lighter fuel oils that have lower sulphur content is another possible solution to meet the sulphur emission regulations.

Hybrid fuel oils are created with a purpose of replacing sulphur rich fuel oils (> 0.1 % S), such as residual fuel oils, without demanding modifications of machinery, and are likely to become more common in years to come (Kystverket, 2017). Due to their recent introduction to the fuel marked, information regarding the properties and behaviour of hybrid fuel oils have not been available, and this study is one of the first to look at the weathering properties, dispersibility and ignitability of hybrid fuel oils.

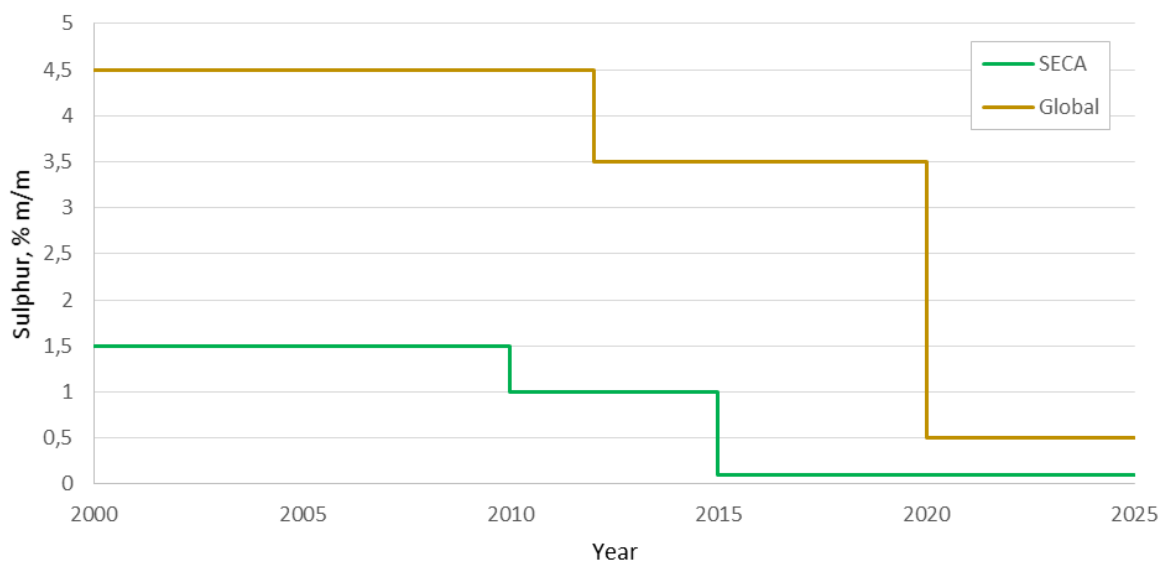


Figure 1-1: Previous and future changes in regulations regarding sulphur content in marine fuel oils within SECA and globally

The obtained data material has been extensive, and each activity has been reported with full detail in separate memos. These memos are collected in a separate report (Hellstrøm *et al.*, 2017), and a solitary WAF-report (Faksness and Altin, 2017), which can be used as supporting material. This summary report aims to provide an overview of the behavioural traits of the different fuel qualities, as well as for the individual oils. The consequences of the observed behaviour is also discussed from an operational oil spill response view.

Performed tests and results

Bench-scale studies where chemical composition, physical properties and emulsifying properties were investigated were performed for all six oils. The testing was performed at two temperatures, representing arctic conditions (2 °C) and summer conditions in the North Sea (10/13 °C). Of the tested oils, WRG, HDME 50 and Shell ULSFO expressed emulsifying properties (investigated by using rotating cylinders; Hokstad *et al.*, 1993).

A screening of six different oil spill dispersants (Dasic Slickgone NS, Corexit 9500, Finasol OSR-52, Superdispersant, Gamlen OD 4000 and Radiagreen) were performed for all the fuel oils, using the IFP-methodology (low energy conditions). In addition, the natural dispersion (no dispersant) of the DMA-qualities and WRG were investigated with the same methodology. The results indicated that Dasic Slickgone NS overall showed the highest effectiveness, followed by Corexit 9500. Radiagreen was the most effective dispersant for HDME 50. The effectiveness of application of oil spill dispersant was highest for the lighter DMA-qualities, and lowest of HDME 50. Dosage testing and more extensive dispersibility studies were also performed. Shell ULSFO and HDME 50 showed limited dispersibility particularly in winter temperatures, mainly due to their high pour points. This was also observed in the meso-scale flume experiments.

Weathering in SINTEF's meso-scale flume allow evaporation, natural dispersion and emulsification to occur simultaneously, and testing was performed for four of the six fuel qualities. MGO was tested at 13 °C and 2 °C, and it is expected that the other two DMA-qualities, GO and Rotterdam diesel, will behave similarly to this oil. WRG, HDME 50 and Shell ULSFO were all tested at both 2 °C and 13 °C, in order to determine their properties in winter and summer conditions.

Obtained results from the bench-scale and meso-scale testing were used as input to SINTEF's Oil Weathering Model (OWM), and the individual oils behaviour in a standardised scenario has been predicted with this tool. Results of this modelling is presented in the memo-report, Hellstrom *et al.*, 2017.

The chemical composition of the water soluble components, water accommodated fraction (WAF), was investigated for each oil, and the toxicity of the WAF was investigated on two marine organisms, the algae *Skeletonema sp.* and the copepod *Calanus finmarchicus*. These organisms represent two different trophic levels in the marine ecosystems. The results showed that Rotterdam diesel produced the WAF that caused the highest toxic effect of the tested oils, likely due to a high content of light polycyclic aromatic (and toxic) hydrocarbons (PAHs). GO and MGO did not express the same chemical composition, and did not cause similar effects. The hybrid fuel oils caused little toxic effect, and had in general low content of water soluble components.

The ignitability of the six tested fuel oils were tested in a bench scale testing (in a "Burning cell") at SINTEF and an up-scaled testing at Falck Nutec's facilities in Trondheim. The scale of both tests are considered small in comparison to full-scale field tests. The testing revealed that MGO and Shell ULSFO ignited most easily of the tested oils, followed by GO and Rotterdam diesel. These fuels contained a minor amount of lighter components (below C₁₀), and had flash points between 60-85 °C. WRG and HDME 50 are heavy distillate cuts, and the tested batches did not contain significant amounts of lighter components (<C₁₅). Their flash points were 116 and 186 °C, respectively, and the ignitability testing showed that these fuel oils were difficult to ignite. However, with sufficiently long ignition time, all oils ignited in the up-scaled testing.

In addition to the tests mentioned above, a limited study on the spreading potential of GO and WRG was performed. These tests have been used as supplementary experiments to strengthen the obtained data from experiments within the Petromaks 2 project, Thin oil films (TOF), with the aim to generate a relevant and robust data set for spreading of oils with varying rheological properties. The obtained data from these studies have been used to develop an improved spreading model for modelling oil in both the Newtonian and non-Newtonian range, computation of a terminal film thickness, and ceasing of spreading, which is currently under testing in an unreleased (research) version of the OSCAR (Oil Spill Contingency And Response) model. The improved spreading model will be available in future versions of OSCAR and will allow more accurate modelling of the surface signature of oil spills and spills of other fluids, e.g. polymers or petroleum oils. The extra data also supported publishing the theory behind the model, which has been submitted to Marine Pollution Bulletin.

The performed experiments showed clear differences in the film thicknesses that formed for GO and WRG, with GO forming a thinner film than WRG. The results support the use of a thicker terminal film thickness in OWM for WRG compared to the GO.

Relevance for operative oil spill response

The following findings can be of importance in relation to oil spill response operations.

In calm conditions (< 5 m/s wind speed), the spreading of oil and formation of thin oil film thicknesses of DMA-qualities will limit the time window for mechanical recovery and application of oil spill dispersant. A minimum film thickness requirement for mechanical recovery is 100-200 µm, while film thicknesses > 50-100 µm can be considered for application of oil spill dispersants. The initiation of mechanical recovery should therefore be done as soon as possible after a spill of DMA-qualities. According to OWM prediction, DMA-qualities may, in calm weather conditions, have potential for mechanical recovery and application of oil spill dispersant within the first 1-2 days (24-48 hr) after release.

If the diesel oil is confined within fire-proof booms or other meet other barriers (e.g. ice edge or shoreline), in-situ burning may also be considered.

The effectiveness for use of mechanical recovery on free-drifting DMA-diesels is considered to have a limited effectiveness, due to low film thickness and very low viscosities, which can easily lead to boom leakage. In rougher, breaking wave conditions, the lifetime on the sea surface for the DMA-diesels is considered to be limited (< 1 day) due to natural dispersion and evaporative loss.

Application of oil spill dispersant to WRG, HDME 50 or Shell ULSFO should be performed while emulsion viscosities are low. The lowest viscosities are expected directly after the spill has occurred for WRG, regardless of temperature. For HDME 50 and Shell ULSFO, the lowest emulsion viscosities are expected directly after the release in summer temperatures, while in winter temperatures the initial viscosities may become high a short time after release due to the high pour point of these oils. However, these emulsion viscosities may decrease slightly with time. When taking response time for response operation vessels into consideration, application of dispersant should be performed as soon as possible, regardless of temperature. Repeated application of dispersant will be more effective than a single high-dose application for these fuel oils. Application of additional energy (e.g. use of high dosage water flushing) in low energy conditions will likely enhance the dispersion of the treated oil/emulsion.

Mechanical recovery of oil/emulsion on the sea surface is expected to be possible for WRG, HDME 50 and Shell ULSFO. In cold temperatures, HDME 50 and Shell ULSFO may form solidified, non-dispersible oil lumps. A previous study performed at SINTEF with a weir skimmer indicated that the tested skimmer model would be less effective for recovery of emulsion, when the emulsion viscosity reached 15 000 - 20 000 mPa·s (Leirvik *et al.*, 2001). NCA, however, has a wide range of different oil skimmers and operate with

general viscosity limits that specify that oils/emulsions with viscosities below 10 000 cP (= 10 000 mPa·s) are deemed low viscous, emulsion viscosities between 10 000-50 000 mPa·s have medium viscosity, and high viscous oils have viscosities above 50 000 mPa·s (Kystverket, 2014b).

The emulsion viscosities of Shell ULSFO and HDME 50 may surpass 20 000 mPa·s over time, and ULSFO in particular may obtain emulsions with viscosities above 50 000 mPa·s after a few days of weathering, especially in colder temperatures. The choice of skimmers must be made based on the expected or measured emulsion viscosity, and high viscosity skimmers can in some cases be required for effective recovery of the oil/emulsion. WRG is expected to form emulsions within the low viscous range (< 10 000 mPa·s).

2 Sammendrag

Toksisitet og forvittringsegenskaper med relevans for operativ oljevernberedskap ble kartlagt for seks forskjellige marine drivstoffoljer. Bruk av dispergeringsmidler, mekanisk oppsamling og *in-situ* brenning (ISB) ble evaluert. De seks drivstoffoljene inkluderte tre DMA-kvaliteter, i denne rapporten omtalt som "Gas Oil" (GO), "Marine Gas Oil" (MGO), og "Rotterdam diesel", samt en såkalt "wide range gas oil" (WRG). I naturreservatene på østsiden av Svalbard og i de tre store nasjonalparkene på vestsiden er det kun tillatt å benytte og medbringe drivstoff av DMA-kvaliteter, mens WRG oljen ikke er tillatt innenfor disse områdene. Disse marine drivstoffoljene benyttes også ellers i arktiske områder og langs norskekysten (Kystverket, 2017). I tillegg til disse lettere oljene inkluderte prosjektet studium på to drivstoffoljer av såkalt "hybrid-kvalitet", eller hybridoljer; ExxonMobil HDME 50 (Heavy Distillate Marine ECA 50) og Shell ULSFO (Ultra Low Sulfur Fuel Oil). I denne rapporten refereres det kun til produktet Shell ULSFO når forkortelsen ULSFO forekommer. Den samme forkortelsen er nylig tatt i bruk om drivstoffgruppen hybridoljer, i diskusjoner relatert til de nye ISO 8217:2017-standardene for drivstoffoljer, men i denne rapporten benyttes hybridoljer til generelle diskusjoner.

Hybridoljer er et begrep benyttet til å beskrive drivstoffoljer som ikke kan klassifiseres i henhold til ISO-standardene (ISO 8217), og har gjerne egenskaper fra både lettere destillatoljer og tyngre drivstoffkvaliteter. Hybridoljer produseres i økende grad som følge av strengere krav til svovelutslipp fra marine fartøyer, både innenfor og utenfor svovelutslippkontrollområder (SECA – Sulphur emission control areas). Tidligere og fremtidige endringer i maksimum tillatt utslipp av svovel er gitt i Figure 1-1. Bruk av hybridoljer er kun en av flere metoder som kan benyttes for å tilfredsstille disse kravene. Andre mulige tiltak kan være installasjon av skrubber i eksisterende maskineri om bord i fartøy som vil fortsette å benytte tyngre drivstoffoljer (HFO) med høyere svovelinnhold. Endringer i maskineri som tilrettelegger for bruk av lettere drivstoffoljer med lavere svovelinnhold er et annet mulig tiltak for å møte kravene til redusert svovelutslipp.

Hybridoljer fremstilles for å erstatte tyngre, svovelrike drivstoffoljer, som bunkersoljer, og må derfor ha egenskaper som er egnet til bruk i maskineri som er beregnet for tyngre drivstoffoljer. Produksjonen og tilgjengeligheten av hybridoljer har økt de siste årene og er forventet å fortsette å øke i årene som kommer. Ettersom hybridoljer er nye på markedet er forvittringsegenskapene til disse produktene lite kjent. Det vil derfor være et behov for å kartlegge disse egenskapene i fremtiden, og dette studiet har vært et av de første som fokuserer på dette området.

Prosjektet har vært omfattende og den produserte datamengden har vært stor. Resultater fra hver delaktivitet har blitt rapportert separat i notater, og disse notatene er samlet i en egen notatrapport (Hellstrøm *et al.*, 2017), og i en selvstendig toksisitetsrapport (Faksness og Altin, 2017). Disse rapportene anbefales som støttemateriale og for mer detaljert informasjon om de ulike aktivitetene. Målet med denne sluttrapporten er å gi en oversikt over de viktigste egenskapene til de testede drivstoffoljene, med spesielt fokus på relevans for oljevernberedskap.

Oljevernberedskap

De følgende funnene er vurdert som av interesse i forbindelse med oljevernberedskap.

Drivstoff av DMA-kvalitet vil spres raskt på havoverflaten under rolige værforhold (< 5 m/s) og danner tynne oljefilmer. Tynne oljefilmer begrenser muligheten for mekanisk oppsamling og for bruk av dispergeringsmiddel, i tillegg til å forhindre antenning. Dersom det er mulig, bør spredningen av oljer av denne kvaliteten forhindres så raskt som mulig, og det bør gjøres forsøkes på å samle oljen til en tykkere oljefilm. Bruk av dispergeringsmiddel vil være mulig på oljefilmer med en tykkelse på 50-100 μm og tykkere. Dersom et frittdrivende oljeflak samles i brannsikre lenser eller møter en annen brannbestandig barriere (strand eller iskant), kan *in-situ* brenning (ISB) også være et aktuelt beredskapstiltak kort tid etter et akutt utslipp.

Effektiviteten av mekanisk oppsamling av oljer av DMA-kvalitet anses som begrenset dersom oljen driver fritt, grunnet lav filmtykkelse og lave viskositeter, og lav viskositet kan bidra til lekkasje fra lenser. Ved brytende bølger anses levetiden for DMA-kvaliteter på havoverflaten for å være lav, mindre enn 1 dag, grunnet naturlig dispergering og høy grad av avdampning.

Påføring av dispergeringsmiddel på emulsjoner av WRG, HDME 50 eller Shell ULSFO bør utføres mens emulsjonsviskositetene ennå er lave. Lavest emulsjonsviskositet for WRG forventes de første timene etter utslipp av oljen, uavhengig av temperatur. Ved sommertemperaturer ($13+^{\circ}\text{C}$) er det forventet at HDME 50 og Shell ULSFO også vil ha lavest emulsjonsviskositet umiddelbart etter utslipp av oljen, tilsvarende som for WRG. Under kaldere temperaturer (vintertemperaturer) kan emulsjonsviskositetene bli svært høye for begge disse oljene kort tid etter utslipp, grunnet de høye stivnepunktene til HDME 50 og Shell ULSFO. Emulsjonsviskositetene kan deretter reduseres i en kortere periode som følge av opptak av vann og ødeleggelse av voks-gitterstrukturer, før ytterligere emulgering bidrar til en ny økning i emulsjonsviskositet. Med tanke på responstiden til skip, fly og helikopter for påføring av dispergeringsmiddel, anbefales det å gjennomføre påføring av dispergeringsmiddel så snart som mulig for alle disse oljene, uavhengig av temperatur. Gjentatt påføring av dispergeringsmiddel vil trolig gi en høyere effektivitet enn bruk av høyere doseringer for disse drivstoffoljene. Tilførsel av ekstra energi, f.eks. ved bruk av vannkanoner, under rolige værforhold vil også trolig bidra til å øke dispersjon av oljene og deres emulsjoner.

Både vannfri olje og emulsjoner av WRG, HDME 50 og Shell ULSFO forventes å kunne samles opp mekanisk. Ved lave temperaturer kan HDME 50 og Shell ULSFO stivne på overflaten og danne klumper av olje, og disse klumpene vil ikke kunne dispergeres. I et tidligere studium utført ved SINTEF med en overløpsskimmer indikerte at den aktuelle skimmeren var mindre effektiv på emulsjoner over 15 000 - 20 000 $\text{mPa}\cdot\text{s}$ (Leirvik et al., 2001). Kystverket har derimot en rekke ulike type oljeskimmere med varierende kapasitet ved ulike viskositeter, og opererer med andre grenser for lav, middels og høy viskositet: Oljer med viskositet under 10 000 $\text{mPa}\cdot\text{s}$ regnes som lavviskøse oljer, viskositeter mellom 10 000 – 50 000 $\text{mPa}\cdot\text{s}$ regnes som middels høye, og oljer med viskositet over 50 000 $\text{mPa}\cdot\text{s}$ regnes som høyviskøse oljer (Kystverket, 2014b).

Emulsjonsviskositetene til HDME 50 og Shell ULSFO kan overskride 20 000 $\text{mPa}\cdot\text{s}$, og ULSFO kan oppnå emulsjonsviskositeter over 50 000 $\text{mPa}\cdot\text{s}$, spesielt ved lave temperaturer. Valg av skimmer må bli gjort basert på de forventede eller målte emulsjonsviskositeter, og høy-viskositet skimmere kan bli nødvendig for effektiv mekanisk oppsamling av olje/emulsjon. WRG forventes ikke å danne emulsjoner med viskositeter i denne størrelsesordenen.

3 Introduction

An oil spill preparedness analysis (EPA) of Jan Mayen and Svalbard (Kystverket, 2014a) revealed a need for characterisation of chemical-physical properties and emulsifying behaviours of different diesel qualities commonly used in Arctic conditions. The Norwegian Coastal Administration (NCA, Kystverket) engaged SINTEF in 2014 to perform a preliminary study on five different diesel qualities, where the aim was to map the most relevant properties. The results from this study have been presented in a separate report (Sørheim and Daling, 2015), and were used as a basis for selecting oils for this extensive study on other, operationally relevant properties such as emulsifying properties, dispersibility and toxicity.

In January 2015, new regulations regarding sulphur emission in Sulphur Emission Control Areas (SECA) were enforced, reducing the allowed content of sulphur in marine fuel oils from 1 % m/m (mass/mass) to 0.1 % m/m. In addition, changes in sulphur emission regulations outside SECA areas are announced to occur in 2020, reducing allowed sulphur content in fuel oils from 3.5 % m/m to 0.5 % m/m. With these changes, new low-sulphur fuel qualities entered the market and their demand was (and is) expected to increase in the coming years. These new fuel oils were given the nickname Hybrid fuel oils, since they have been designed to have low sulphur content and diesel-like qualities while still having properties suitable for combustion in heavy fuel oil engines (CIMAC, 2017). Very little information regarding these new fuel oil types are available, and a need characterisation of the physical-chemical properties and weathering behaviour of these oil types were evident.

On request from NCA, SINTEF Environmental technology has performed an extensive study with relevance for oil spill response operations on a wide spectre of marine diesel oils and hybrid fuel oils in the period 2015-2017. Three DMA diesel qualities (MGO from Esso Slagen, GO from Mongstad and Rotterdam Diesel from the Netherlands) were chosen, as was a DMB/DMC quality fuel (WRG from Mongstad). In addition, the Hybrid fuel oils Shell ULSFO (Ultra Low Sulphur Fuel Oil from Shell) and HDME 50 (Heavy Distillate Marine ECA from ExxonMobil) have been included in the study. These fuel types were relevant as they are used on vessels in Norwegian and/or European waters. Several activities have been performed in the study, and these are listed in Table 3-1.

Table 3-1: Overview of the different activities in the project listed for each fuel oil.

Activity\oil	GO	MGO	Rotterdam Diesel	WRG	ULSFO	HDME 50
Bench-scale testing, (2) 13 (20) °C	(x) x* (-)	(x) x* (-)	x	(x) x* (-)	(x) x (x)	(x) x (x)
Dispersibility study	IFP/MNS (13 °C)	x	x**	x	x	x
	FET-test (2)13(20) °C	-	-	-	(x) x (x)	(x) - (x)
Meso-scale flume testing (2) 13 °C	-	(x)x	-	(x)x	(x)x	(x)x
Ignitability: Small scale testing	x	x	x	x	x	-
Ignitability: up-scaled testing	x	x	x	x	x	x
Spreadability	x	-	-	x	-	-
WAF and toxicity study	x	x	x	x	x	x
OWM – oil weathering model: Predicted behaviour	x	x	x	x	x	x

*Emulsifying properties were tested at 10 °C

** Only a screening study was performed

This report aims to summarise the most important results from the various activities and link these to provide comprehensive notion of the properties of each of the tested oils. Results from each activity has previously been reported in project memos, and these have been collected in a separate memo report (Hellstrøm *et al.*, 2017) and a toxicity report (Faksness and Altin, 2017). A summary of the experimental methods applied are given in Appendix A, while the detailed results are found in the memo report.

4 Description of test oils and definitions

Six fuel oils of different qualities have been studied in this project. These six fuel oils consist of three DMA-qualities, here called GO, MGO and Rotterdam diesel, a heavier marine fuel oil, Wide Range Gas oil (WRG), and two hybrid fuel oils, HDME 50 (Heavy Distillate Marine ECA 50) and Shell ULSFO (Ultra-Low Sulphur Fuel Oil). The following section lists the different oils with further details regarding their usage areas, properties and characterisation within existing classification charts (ISO 8217:2017). Some details regarding the classification categories and sources of the test oils can be found in Table 4-2. The test oils were procured by NCA.

A revised ISO 8217 standard for marine fuels was issued in June 2017, and stricter regulations for sulphur content was implemented, compared to the 8217:2012 standard. The old 2012 and new 2017 standards form the basis for the discussions and comparisons in this report, with some reference to the 8217:2005 standard also being made. Table 4-1 provide the key parameters and limits for distillate fuel oils as given in ISO 8217:2017, and includes also the previous limits for sulphur content from ISO 8217:2012.

Table 4-1: Classification criteria for marine distillate fuel standards according to the current standard (ISO 8217, 2017), including former limits for sulphur content from the previous version (in italics) (ISO 8217:2012)

Characteristics	Unit	Limit	Category ISO-F-						
			DMX	DMA	DFA	DMZ	DFZ	DMB	DFB
Kinematic viscosity at 40 °C	mm ² /s	Max.	5.500	6.000		6.000		11.00	
		Min.	1.400	2.000		3.000		2.000	
Density at 15 °C	kg/m ³	Max.	—	890.0		890.0		900.0	
Cetane index	—	Min.	45	40		40		35	
<i>Sulphur ISO 8217:2012</i>	<i>mass %</i>	<i>Max.</i>	<i>1.00</i>	<i>1.50</i>		<i>1.50</i>		<i>2.00</i>	
Sulphur	mass %	Max.	1.00	1.00		1.00		1.50	
Flash point	°C	Min.	43	60		60		60	
Hydrogen sulfide	mg/kg	Max.	2.00	2.00		2.00		2.00	
Acid number	mg KOH/g	Max.	0.5	0.5		0.5		0.5	
Total sediment by hot filtration	mass %	Max.	—	—		—		0.10	
Oxidation stability	g/m ³	Max.	25	25		25		25	
Fatty acid methyl ester (FAME)	volume %	Max.	—	—	7.0	—	7.0	—	7.0
Carbon residue: micro method on the 10 % volume distillation residue	mass %	Max.	0.30	0.30		0.30		—	
Carbon residue: micro method	mass %	Max.	—	—		—		0.30	
Cloud point	winter	°C	Max.	-16	report		report		—
	summer	°C	Max.	-16	—		—		—
Cold filter plugging point	winter	°C	Max.	—	report		report		—
	summer	°C	Max.	—	—		—		—
Pour point (upper)	winter	°C	Max.	—	-6		-6		0
	summer	°C	Max.	—	0		0		6
Appearance	—	—	Clear and bright						
Water	volume %	Max.	—	—		—		0.30	
Ash	mass %	Max.	0.010	0.010		Ash		mass %	
Lubricity, corrected wear scar diameter (wsd 1.4) at 60 °C	µm	Max.	520	520		520		520	

Any reference to GO, MGO, Rotterdam diesel, WRG, Shell ULSFO and HDME 50 are directed towards the tested oils individually, and not the fuel quality categories in general. Full-length descriptions such as gas oil, marine gas oils, DMA-qualities, heavy distillate fuel oils and hybrid fuel oils have been used when describing properties of fuel qualities in general.

Table 4-2: Descriptions of the tested oils regarding sources and classification category.

Oil product	Description	Source	Sulphur content	Received	SINTEF ID
GO	Gas Oil with max 10 ppm sulphur, DMA-quality. Dyed with 40 mg/L green dye	Mongstad, Norway	10 ppm S	4.12.2014	2014-0552
MGO	Marine Gas Oil with maximum 500 ppm sulphur, DMA-quality	Esso Slagen, Norway	500 ppm S	4.12.2014	2014-0551
Rotterdam diesel	Marine gas oil (dyed) from Rotterdam, DMA-quality	Shell, the Netherlands	< 1000 ppm S	12.4.2016	2016-0232
WRG	Wide range gas oil, DMZ-RMA quality	Mongstad, Norway	2500 ppm S	4.12.2014	2014-0553
			<500 ppm S	24.4.2017	2017-1880
HDME 50	Heavy Distillate Marine ECA 50 (Emission Commission Control Area)	ExxonMobil, the Netherlands	< 1000 ppm S	12.4.2016	2016-0231
			< 1000 ppm S	24.4.2017	2017-1879
ULSFO	Ultra Low Sulphur Fuel Oil, RMA-quality oil	Shell, the Netherlands	< 1000 ppm S	12.4.2016	2016-0233

4.1 DMA diesels – GO, MGO and Rotterdam diesel

Three different diesel fuels qualifying for DMA-quality were studied in this project. These have been called GO, MGO and Rotterdam Diesel (DMA-diesel) in this report and the memo reports, in order to separate the results easily. Key properties of these diesel oils are listed in Table 5-1. The MGO and GO oils were chosen for analysis based on the results from the preliminary study (Sørheim and Daling, 2015), while a limited number of analysis were included in the study for the Rotterdam diesel. These three diesel fuels all have sulphur content that allow use within SECA areas (<0.001 % S), but this is not universal for DMA fuel types. DMA diesel are often used on board large vessels such as cruise ships, and supply and general cargo vessels, but qualities with very low sulphur content, such as the GO diesel (also called Autodiesel), are also used in land based furnaces and smaller vessels (Kystverket, 2017). DMA-qualities are also used in fishing boats and for the help engines aboard larger vessels using HFO as bunker fuel oil for the main engines.

4.2 DMB/DMC/RMA– WRG

Wide Range Gas oil (WRG, 2014) is a heavy distillate cut. This oil has been used in the area around Svalbard and outside SECA areas due to a high sulphur content.

The batch of WRG received in 2014 and used in this study qualified as a DMB/DMC fuel oil under the former ISO 8217:2005 criteria. However, between 2005 and 2012, the DMC-classification was removed from the list of distillate fuels in order to form the basis for the RMA-quality in 2012. Based on the 2012 ISO standards, the WRG batch from 2014 would be too viscous to be classified as a DMB, thus moving up to a RMA classification. There were no reported changes in the classification of residual marine fuels between the 8217:2012 and 8217:2017, and the 2014 batch of WRG would fall under RMA classification under the present standards as well, and not classified among the distillate fuel oils (Table 4-1).

DMB/DMC-qualities are commonly used by larger fishing boats, general cargo ships and Hurtigruta (Norwegian Coastal Express). WRG has been described as having IFO 10-qualities and was chosen for further studies since it is used frequently along the Norwegian coast and in the northern areas (Kystverket, 2017).

Batch variations of WRG are further discussed in Chapter 5.4.6 on page 32.

4.3 Hybrid fuel oils

Fuel oils that cannot be categorized according to the present ISO standards are entering the market as a result of higher demand for low sulphur fuel oils, as a result to new regulations for sulphur content in SECA areas. These new fuel types have properties that allow use in heavy bunker fuel engines, while at the same time having the low sulphur content associated with lighter diesel qualities. Having properties like a hybrid of diesel and heavy fuel oil, these new oils are commonly called Hybrid fuel oils. Presently, several different hybrid fuel oils may be available commercially, and two different types have been studied in this project. These two oils are Shell ULSFO (Ultra Low Sulphur Fuel Oil) and HDME 50 (Heavy Distillate Marine ECA). Both hybrid fuel oils are produced in the Netherlands, Shell ULSFO is produced by Shell while HDME 50 is produced in Rotterdam by ExxonMobil. Hybrid fuel oils are commonly used on larger vessels such as container ships, ro-ro ships and general cargo ships in traffic within SECA areas (Kystverket, 2017).

Variations between different batches of HDME 50 are further discussed in Chapter 5.5.5 on page 37.

5 Experimental results

A small selection of the experimental results are presented here. These results are shown and discussed in detail in the memo-report, but the main findings are provided here as well for the readers benefit. The oils have been given a colour code comparable to the appearance of the fresh oil, as shown on the front picture of this report, and this colour coding is used throughout the report. Green colouring have been added to the GO 10 ppm S diesel oil prior to analysis, and has been given a green colour code.

The interfacial tension (IFT) of HDME 50 and Shell ULSFO 250°C+ residue were not possible to measure with the applied method due to the oils viscous properties. Interfacial tension describe the tension in the interface between two immiscible liquids (here oil and water). As little surface area as possible is ideal between immiscible fluids, and this is seen between oil and water in the preference of forming separate phases or large oil droplets. IFT is a measurement of the tension between the oil and water. Of two oils of similar viscosity, the oil with lowest IFT have a higher potential for natural dispersion into oil droplets. Surfactants from oil spill dispersants will position themselves in the interface between the liquids and contribute to a dramatic decrease in interfacial tension, thus enhancing dispersion by stabilising the smaller oil droplets that form.

Table 5-1: Key results for physical and chemical properties for all tested oils

Oil type	Residue	Evaporation (vol. %)	Residue (wt. %)	Density (g/ml)	Flash point (°C)	Pour point (°C)	Viscosity (mPa·s) 2°C (10 s ⁻¹)	Viscosity (mPa·s) 13°C (10 s ⁻¹)	Viscosity (mPa·s) 50°C (*40°C)**	IFT _{in} (mN/m)	IFT _{eq} (mN/m)	Asphaltenes "hard" (wt. %)	Wax (wt. %)
GO 10 ppm S	Fresh	0	100	0.833	71.5	<-36	5	0	2.1	18.2	8.2	0.01	0.01
	250°C+	59.5	41.2	0.846	107.5	-33	10	7	-	22.7	5.8	0.03	0.01
MGO 500 ppm S	Fresh	0	100	0.852	62.5	<-36	8	3	3.7	23.7	10.4	0.02	0.81
	250°C+	30.6	70.8	0.868	110	<-36	20	12	-	22.2	8.3	0.03	1.14
Rotterdam diesel	Fresh	0	100	0.885	82.5	<-36	43	12	5.4*	30.6	14.6	0.02	3.1
	250°C+	6.1	94	0.887	110.5	-27	56	14	-	32	16.4	0.02	3.3
WRG	Fresh	0	100	0.886	115.5	-24	179	59	12.6*	17.2	7.9	0.01	4.74
HDME 50	Fresh	0	100	0.903	186	12	11002	1005	36	-	-	0.06	9.5
ULSFO	Fresh	0	100	0.872	75	24	13106	4300	11	14	11.7	0.15	13.1
	250°C+	14.6	86	0.878	112	30	77782	33169	-	-	-	0.18	15.5

*measured at 40 °C, not 50 °C

**data provided from received certificates and data sheets for the individual oils

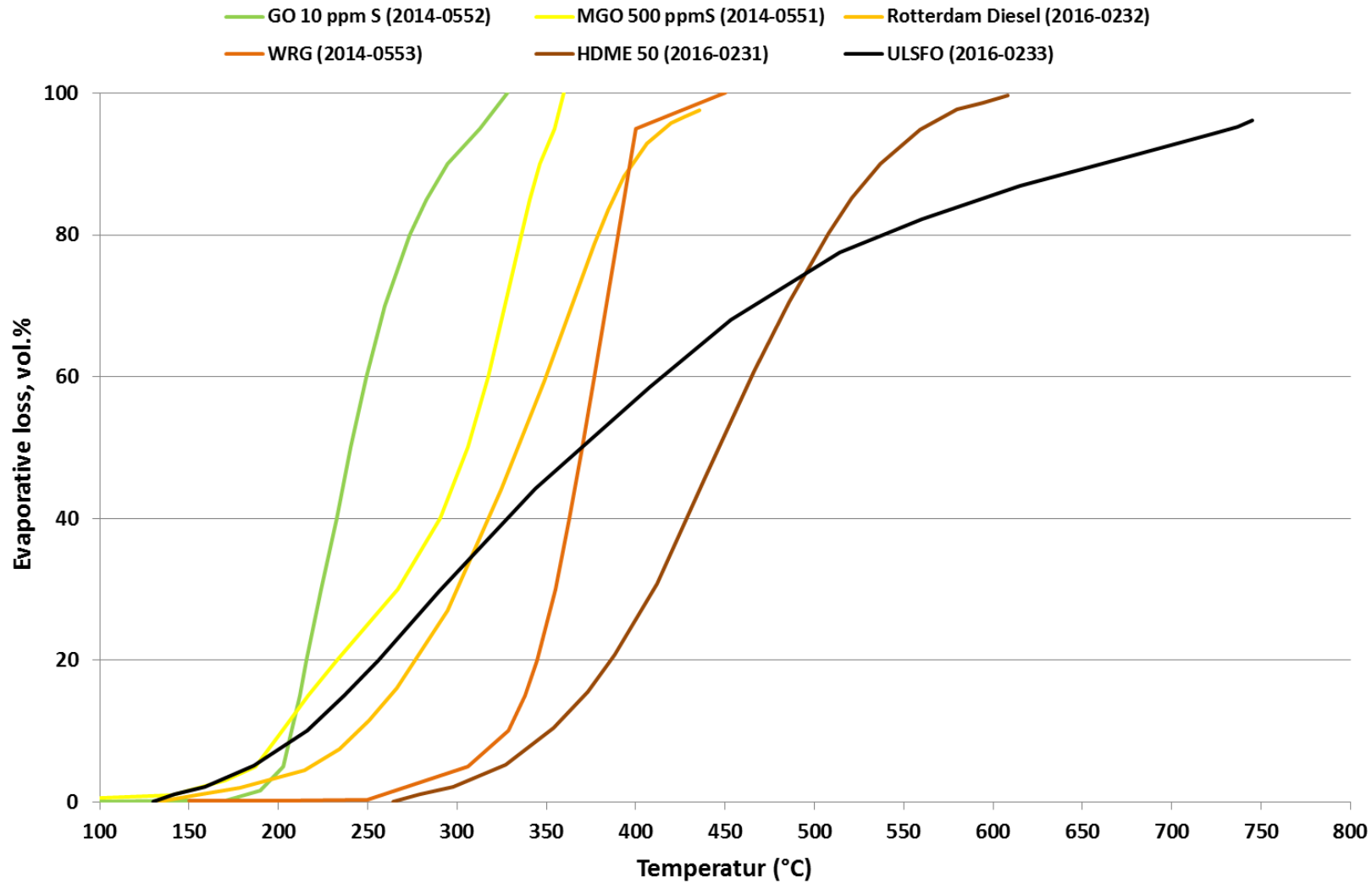


Figure 5-1: Boiling point curves for the tested oils

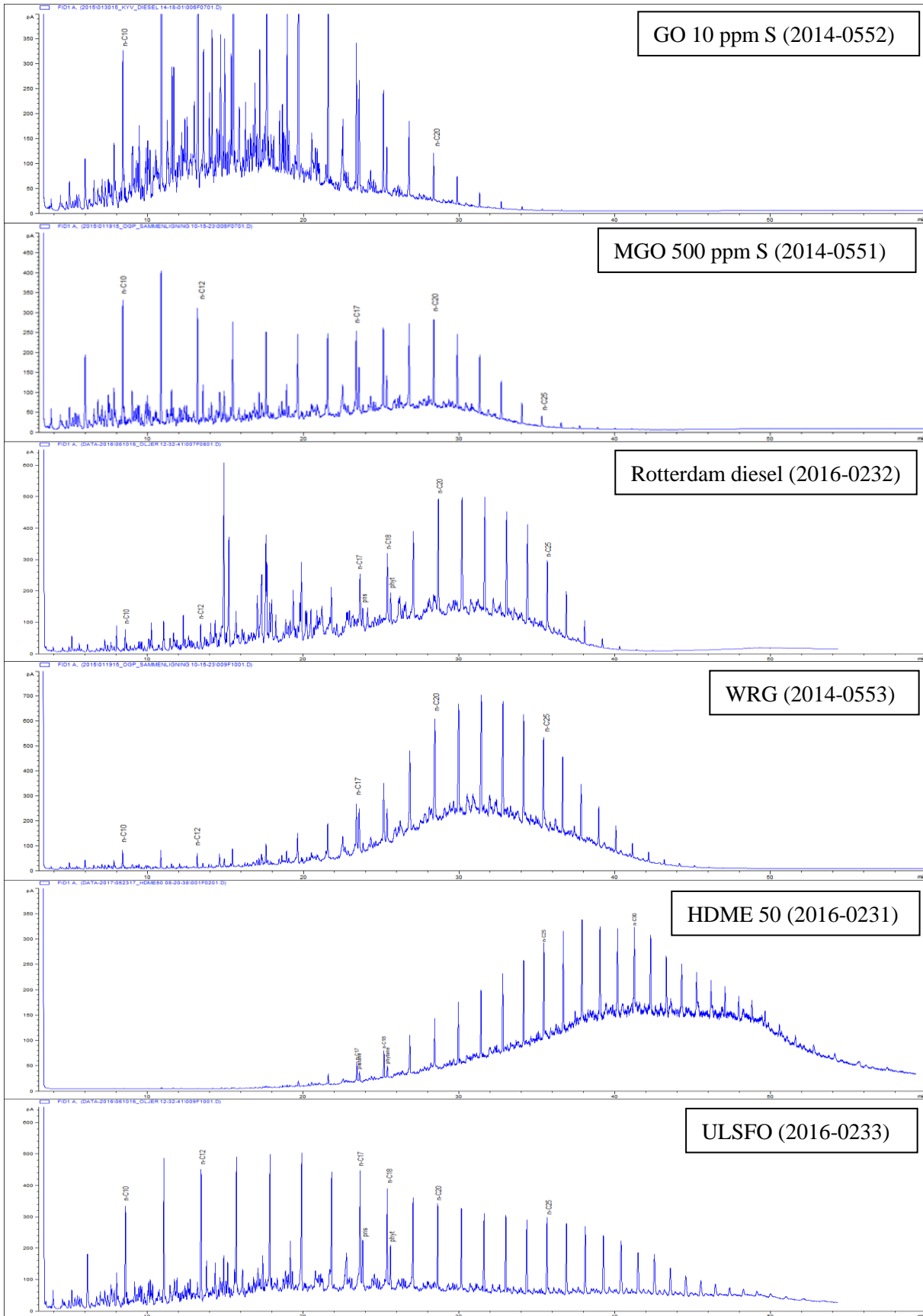


Figure 5-2: Chromatograms of the tested oils.

5.1 GO 10 ppm sulphur (2014-0552)

5.1.1 Bench-scale study

The preliminary testing performed on GO in 2014 showed that neither the oil or the weathered 250°C+ residue of GO had emulsifying properties (Sørheim and Daling, 2015). The chromatogram of GO shown in Figure 5-2 reveal the this oil has very little, if any, content of compounds above C₂₅, and the lack of wax and asphaltenic components explain the lack of emulsifying properties.

A special fuel dye was added to GO prior to artificial weathering and other analysis. This dye is added to the fuel type in a concentration of 40 ppm (40 mg/L) before commercial sale but was received separately at SINTEF, and received the SINTEF ID (2014-0554). For the bench-scale testing, the same concentration of dye was added to the fuel, while in the spreadability study, described in Chapter 5.1.3, an excess of dye was applied to the fuel to be sure the oil film would be visible on camera.

During the artificial weathering (topping) the colour of the dyed fuel oil changed from bright green to brown, and the difference between fresh oil and residue can be seen in Figure 5-3, which also demonstrate the lack of emulsifying properties. Other analysis from the bench-scale study are presented in Table 5-1, and showed that GO had:

- Low pour point for both fresh oil and 250°C+ residue (<-36 and -33 °C, respectively)
- Very low viscosity for fresh oil and residue (< 10 mPa·s at 2 °C)
- High evaporative loss (59.5 vol. % were volatile components with boiling point below 250 °C)
- Relatively low density (0.833 g/ml)

All measured properties were within the ISO standards for DMA-quality, given in Table 4-1.

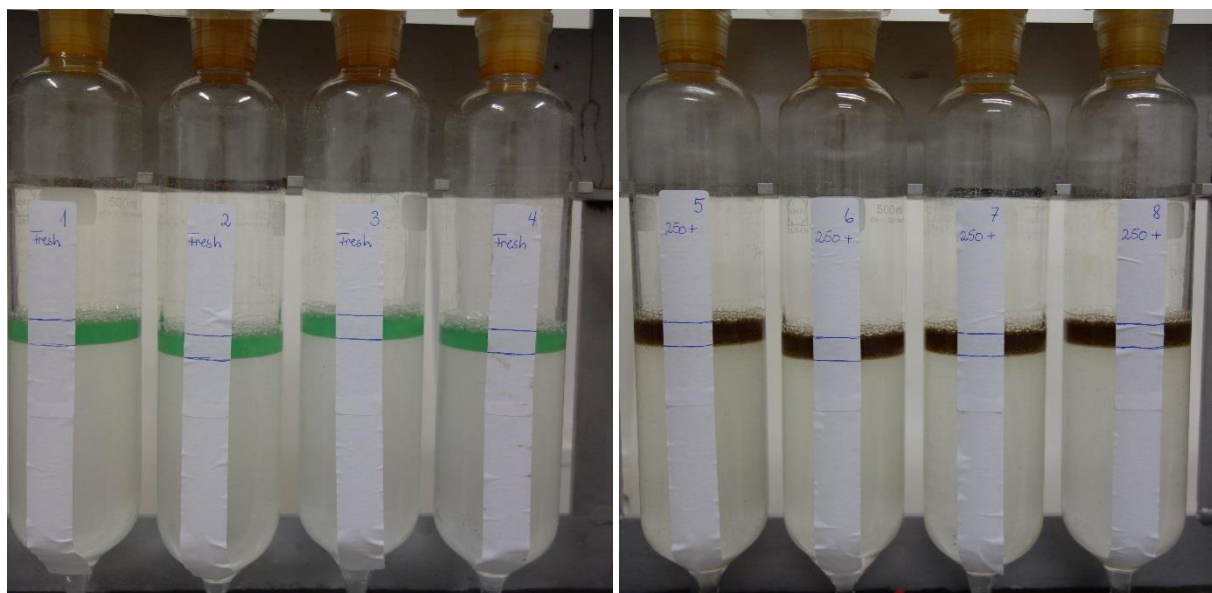


Figure 5-3: Rotating cylinders containing fresh GO (left) and its weathered 250°C+ residue (right) after 24 hours of rotation

5.1.2 Dispersibility

A screening of six different oil spill dispersants was performed on the water-free fraction of weathered (250°C+) GO using the IFP methodology (further described in Appendix A.2) at 13 °C. Corexit 9500 and Dasic Slickgone NS were found to be the most effective oil spill dispersants for GO in the screening testing (Figure 7-1), with effectiveness of 79 and 73 %, respectively. Dasic Slickgone NS was chosen for further dosage testing, and for this testing both IFP and MSN methodology (see Appendix A.2) were used. The tests

were performed on water-free fractions of both fresh and weathered GO at 13 °C. The results indicated that GO has potential for use of chemical dispersion, presuming scenarios where the oil film is thick enough (50-100 µm) allowing the dispersant to soak into the film and preventing the chemical dispersant from breaking through and enter the water-phase.

The main results from the dispersibility testing were that GO had:

- High natural dispersion (without dispersant) in breaking wave conditions (65 % effectiveness in MNS test)
- Poor natural dispersion in non-breaking wave (swell) conditions (< 1 % effectiveness, IFP test)
- Good potential for use of chemical dispersion
 - Complete dispersion in breaking wave conditions (DOR = 1:25, MNS)
 - Up to 73 % effectiveness in swells conditions (DOR = 1:25, IFP)
- Dosage dependant dispersibility in non-breaking wave (swell) conditions (IFP test)
 - 15-73 % effectiveness for dispersant to oil ratios (DOR) of 1:200-1:25, respectively
- No upper dispersibility limit for reduced dispersibility was obtained for this oil

5.1.3 Spreadability

A specific spreadability study was performed with GO and WRG (see Chapter 5.4.4), as supplementary experiments to the TOF-project (thin oil films) in progress. The TOF-project is a part of Petromax 2, and is funded by RCN and selected oil companies project. The aim of the study was to generate relevant and robust data on the spreadability of oils with varying rheological properties, and among these oils were GO and WRG. Obtained results have been used in the development of improved spreading model which will be available in future versions of OSCAR.

The results showed that GO formed an oil film that gradually decreased in thickness and became too thin for measurement using ultra sound (approx. < 200 µm) a short distance from the release point (approx. 60 cm away from centre). The measured film thickness can be seen in Figure 5-5, where the blue line draws an approximation of film thickness based on the results. The film thickness could not be measured further away than 60 cm from the centre, but GO was gradually spread over the entire area of the test pool (5.5 m in diameter, 23.76 m²), showing high spreadability. Figure 5-4 shows three snapshots taken during the experiment after 10, 60 and 180 seconds after initiation, and the dark green colour was caused by an excessive amount of dye added to the diesel oil before testing. Due to the dark colour of the oil, the film may be perceived as thicker than it truly was.

OWM was used to predict the emulsion film thickness of the different fuel oils tested in this project, and the predictions are presented in Figure 6-7.

In an acute incident, GO is expected to spread and form thin oil films. The formation of thin oil films shortens the lifetime of the oil on the sea surface by preventing the formation of emulsions and enhancing removal of light components through evaporation. With thin oil films, 0.05-0.1 mm, both application of chemical dispersant and mechanical recovery are challenging, as dispersant droplets may break through the oil film, and the collection of such oil films in booms is time consuming and ineffective.

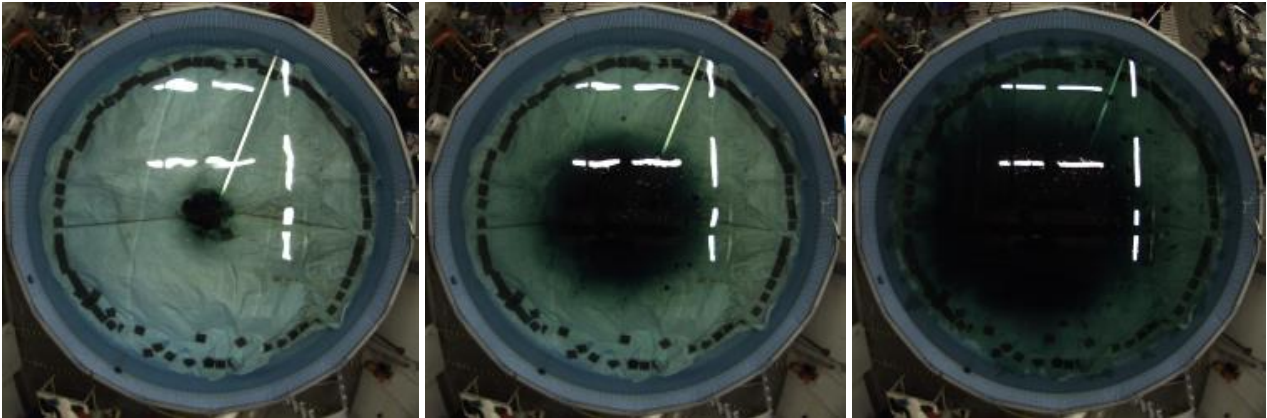


Figure 5-4: The development of a film of GO in the spreadability study after 10 seconds (left), 60 seconds (middle) and 180 seconds (right) after initiation.

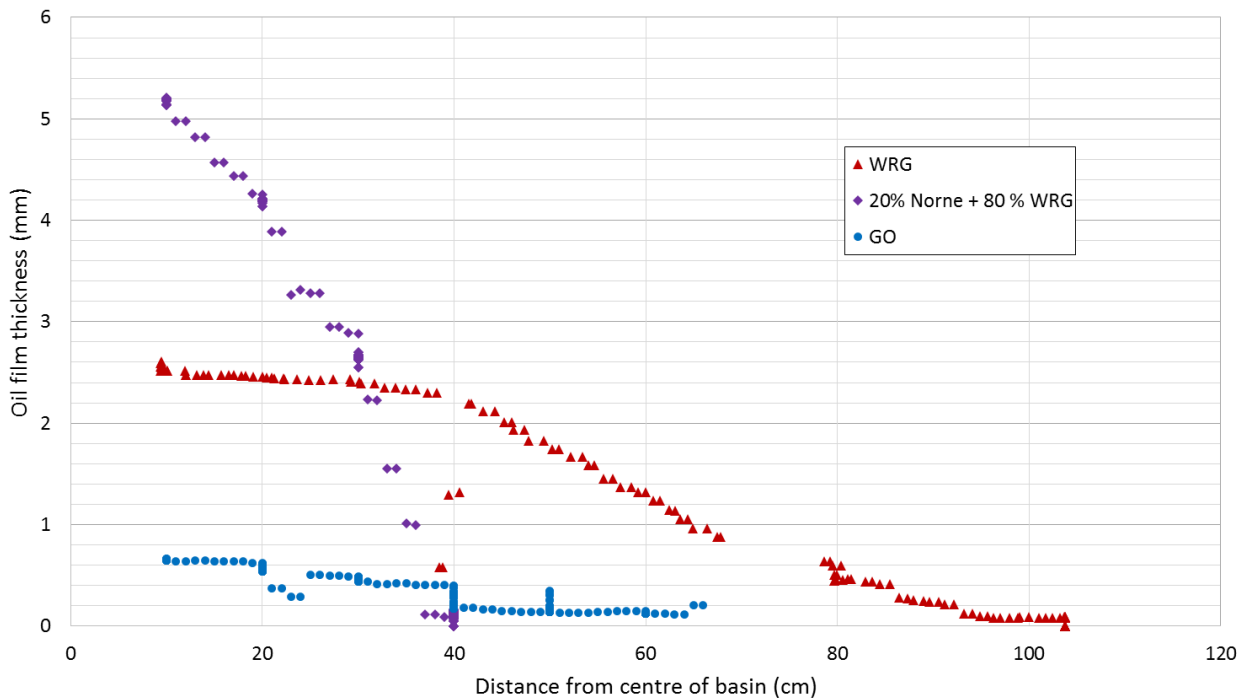


Figure 5-5: Measured film thicknesses for WRG, GO and a mixture of WRG and Norne crude oil

5.1.4 Ignitability

The potential use of in-situ burning (ISB) as a response operation for GO was investigated by testing the ignitability of GO. Both a bench-test and an up-scaled test was applied (Appendix A.4) and the results showed that:

- Fresh GO was found to be ignitable in the bench-scale testing with prolonged ignition time (20 sec.)
- The weathered 250°C+ residue of GO also ignited after prolonged ignition time (20 sec.)
- In the up-scaled testing, fresh GO ignited after 12 minutes and 40 seconds of exposure to burning gelled gasoline, and thus expressed a long ignition time
- The burning efficiency of GO was high in both test scenarios (> 80 %)

The results indicate that ISB can be a potential response operation, and that the burning efficiency can be expected to be high. However, a sufficiently thick film would be essential for ignition of oil, in order to prevent heat loss to the water-phase. The OWM was used to predict the ignitability of GO in a scenario where spreading of the diesel oil has been prevented, the wind conditions were calm and the diesel oil film thickness was high (5 mm). Under these conditions, GO was predicted to have a prolonged time window for potential use of ISB. ISB is further discussed in Chapter 7.3.

5.2 MGO 500 ppm sulphur (SINTEF ID: 2014-0551)

5.2.1 Bench-scale study

Bench-scale laboratory testing using rotating cylinders have previously shown that neither MGO nor its weathered residue (250°C+) had significant emulsifying properties (Sørheim and Daling, 2015). The lack of emulsifying properties were due to the low content of emulsion stabilizing components such as asphaltenes and wax, see Table 5-1. Figure 5-6 show the rotating cylinders containing MGO after 24 hours of rotation and depict no water uptake for the fresh oil (left) and insignificant water uptake for the weathered residue.

Results from other bench-scale analysis are also presented in Table 5-1 and show that MGO has:

- Low pour point for both fresh oil and 250°C+ residue (<-36 °C)
- Low viscosity for both fresh oil and 250°C+ residue (< 20 mPa·s at 2 °C)
- Intermediate evaporative loss (30.6 vol. % at 250 °C)
- A predominance of *n*-alkanes (C₉-C₂₅) (see Figure 5-2)

All measured properties were within the ISO standards for DMA-quality.

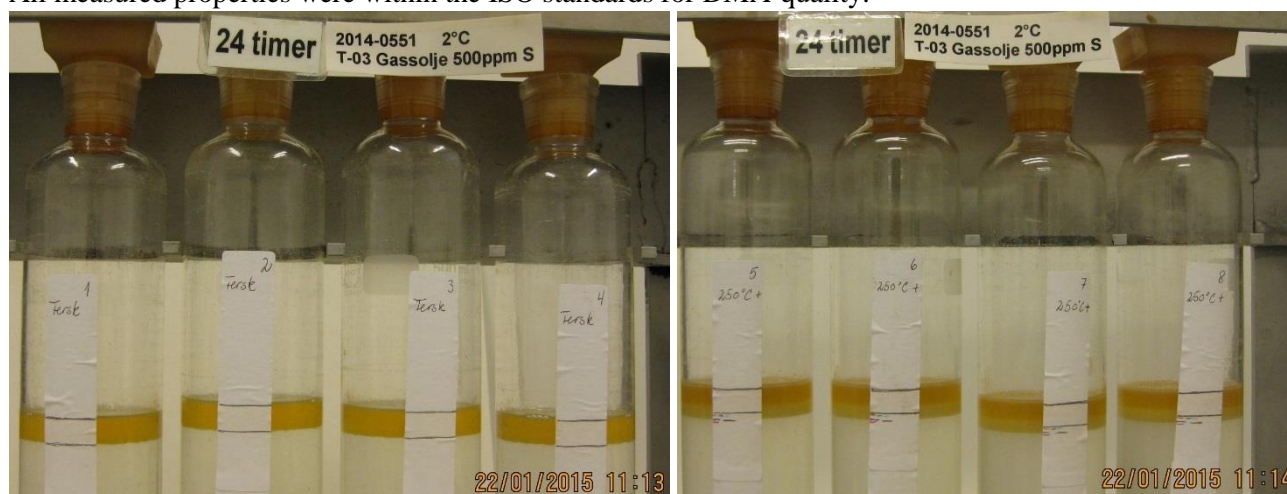


Figure 5-6: shows the rotating cylinders used in testing the emulsifying properties of MGO after 24 hours of rotation. Four cylinders containing fresh MGO is shown to the left and four cylinders containing the weathered 250°C+ residue is shown to the right. No significant emulsification was observed. The black lines on the white marks indicate the volume of water-free oil used in the test, and coloured lines show changes during the 24 hours of rotation.

5.2.2 Dispersibility

A screening of chemical dispersants and a dispersibility study were performed on the water-free fractions of both fresh and weathered (250°C+) MGO using both the MNS and IFP methodology. See memo-report (Hellstrøm *et al.*, 2017) and Appendix A.2 for details. Dasic Slickgone NS and Corexit 9500 were found to be the most effective chemical dispersants for MGO in the screening testing (see Figure 7-1). Dasic Slickgone NS was chosen for further dispersibility testing and MGO was also found to have:

- Relatively high natural dispersion in breaking wave conditions (> 50 % effectiveness, MNS-test), though slightly less than for GO
- Poor natural dispersion in non-breaking wave (swell) conditions (< 1 % effectiveness, IFP-test)
- Dosage dependant dispersibility in non-breaking wave (swell) conditions
 - 22-66 % effectiveness for dispersant to oil ratios (DOR) of 1:200-1:25, respectively
 - Relatively effective at low dosages
- Good potential for use of chemical dispersion
 - Up to 82 % effectiveness in breaking wave conditions (DOR = 1:25, MNS)
 - Up to 66 % effectiveness in swells conditions (DOR = 1:25, IFP)

The results from the dispersibility study show that MGO has a potential for use of chemical dispersion. However, the use of chemical dispersion requires a sufficiently thick film ($> 0.5-0.1$ mm) for the dispersant to soak into and not break through the film ("herding"). The use of chemical dispersant may thus be most relevant if the diesel oil has been trapped or confined. No upper viscosity limit for reduced or poor dispersibility was obtained for this oil.

5.2.3 Meso-scale flume testing

Contrary to the results from the small-scale testing, testing in the meso-scale flume at 2 and 13 °C showed that the MGO diesel oil formed a type of loose and unstable emulsions that broke when handled. The formation of this emulsion-like layer occurred more rapidly at 13 °C than at 2 °C, and the viscosities remained very low, beneath 20 mPa·s, throughout both the tests. It must be emphasized that the loose emulsion that was formed cannot be compared to emulsions formed from crude oil or heavier fuel oils in regards to appearance, stability and viscosity. This loose unstable MGO diesel emulsion was light-coloured (yellow), and had a foam-like appearance despite being formed from oil and water only. The stability of the loose emulsion was low and normal handling or mechanical stress resulted in a rapid separation of the oil and water phase.

Application of chemical dispersant (Dasic NS) had very good effect, and total dispersion (>95 %) was observed in both the meso-scale flume tests 30 minutes after application of dispersant (only one application was required). The meso-scale flume is a closed system with limited surface space that restrict the spreading area of the diesel oil, which will have resulted in a relatively thick film. In an event where spreading is not hindered, the MGO would likely form a film thinner over time than the film that was formed in the meso-scale flume. This is also indicated in Figure 6-7, which shows the predicted emulsion thicknesses.

The formation of emulsions also require a minimum film thickness (>100 μm), and the emulsification will not occur in scenarios where the film thickness is beneath this limit.



Figure 5-7: The emulsion like layer formed by MGO at 13 °C in the meso-scale flume experiment

5.2.4 Spreadability

A limited spreadability study was performed for another DMA-quality diesel, the GO diesel (see chapter 5.1.3). MGO and GO have very similar chemical and physical properties, and it is assumed that the spreading properties also are similar, and that MGO thus will with time form thin film thicknesses in scenarios where no barriers are present. Figure 5-5 show that the GO diesel reached a film thickness that was

undetectable by the instrumentation (approximately $< 200 \mu\text{m}$) only 60+ cm from the release point. Assuming MGO and GO behave similarly, this indicates that MGO will have high spreadability and a rapid decrease in diesel oil film thickness in an acute event (see Figure 6-7). A thin oil film may enhance evaporation and prevent emulsification, resulting in a generally shortened life time of the diesel oil on the sea surface. The thin oil film ($< 50\text{-}100 \mu\text{m}$) will also hinder mechanical recovery and limit successful application of chemical dispersants due to dispersant droplets breaking through the oil film (herding) and dissolving in the sea water. A thin diesel oil film will also prevent the use of in-situ burning due to rapid loss of lighter components and heat loss between the water and oil phases.

5.2.5 Ignitability

The ignitability of MGO was tested in a bench-scale study as well as in an up-scaled study, in order to reveal the potential use of in-situ burning (ISB) as a response operation (see Chapter 7.3, and memo-report for full detail). The results showed that:

- Fresh MGO was found to be ignitable in the bench-scale testing under the standard test conditions (3 attempts with 10 sec. ignition time)
- The weathered residue ignited after prolonged ignition time (30 sec.).
- In the up-scaled testing, fresh MGO ignited within 4 minutes.

In conditions where spreading of the diesel oil is prevented, the wind conditions are calm and the diesel oil film thickness is high ($> 5 \text{ mm}$), MGO is predicted to have a prolonged time window for potential use of ISB.

5.3 Rotterdam diesel – DMA diesel (2016-0232)

5.3.1 Bench-scale testing

A reduced bench-scale analysis was performed at 13 °C for the Rotterdam diesel. Based on tests previously performed on similar diesel oils like GO and MGO, fresh Rotterdam diesel was assumed to not express significant emulsifying properties. However, Rotterdam diesel had a higher content of wax (3.1 wt. %) compared to the other tested DMA-qualities, and an enrichment of wax content with evaporation of the oil might have given the weathered oil some emulsifying properties. For this reason, the emulsifying properties of the weathered 250°C+ residue was tested using rotating cylinders (Hokstad *et al.*, 1993). Figure 5-8 shows the rotating cylinders after 24 hours of rotation. No emulsification was observed, but the oil formed a foam on the oil phase surface.

Table 5-1 show the key results from the bench-scale testing, and reveal that Rotterdam diesel had:

- Low evaporative loss, 6.1 vol. % of components with boiling point below 250 °C
- High content of lighter polycyclic aromatic hydrocarbons (PAHs)
- Relatively high density (0.885 g/ml) compared to the other DMA-qualities
- Relatively high flash point (82.5 °C) compared to the other DMA-qualities
- Low pour point for both fresh oil (<-36 °C) and weathered 250°C+ residue (-27 °C)
- Low viscosities for fresh oil and weathered residue (<60 mPa·s at 2 °C)
- High content of lighter aromatic compounds



Figure 5-8: Rotating cylinders containing weathered 250°C+ residue of Rotterdam diesel after 24 hours of rotation. The black lines on the white marks indicate the volume of water-free oil used in the test, and coloured lines show changes during the 24 hours of rotation. No significant water uptake was observed.

5.3.2 Dispersibility

A screening study with six chemical dispersants was performed at 13 °C on the water-free fraction of the weathered 250°C+ residue, using the IFP-methodology. The results can be seen in Figure 7-1 and showed that Dasic Slickgone NS was the most effective dispersant, followed by Corexit 9500. In general, the effectiveness of chemical dispersants were slightly lower for Rotterdam diesel than for the other DMA-qualities, ranging from 41-56 % effectiveness. Without the application of dispersant, Rotterdam diesel was not dispersible under the low-energy conditions of the test (<1 % effectiveness, IFP-test), similar to the other DMA-qualities. The slightly reduced dispersant effectiveness compared to the other DMA-qualities are

likely due to the difference in chemical composition. No upper viscosity for reduced or poor dispersibility was obtained for this oil.

5.3.3 Spreadability

Like MGO, the Rotterdam diesel is expected to behave in the same way as GO, rapidly spreading and forming thin oil films in a spill situation where spreading is not prevented. The predicted emulsion thickness of Rotterdam diesel is shown in Figure 6-7.

5.3.4 Ignitability

When the ignitability of fresh Rotterdam diesel was tested in the bench-scale study, the diesel oil ignited after 25 seconds of prolonged ignition time. The oil produced a characteristic black smoke, likely due to the high content of PAH, and in order to avoid exposure, the experiment was quickly ended. The black smoke was also observed in the up-scaled testing where the oil ignited after 10 minutes and 40 seconds, requiring two packs of gelled gasoline. See Chapters 5.1.4 and 7.3, and the memo-report (Hellstrøm *et al.*, 2017) for full detail.

In conditions where spreading of the diesel oil is prevented (confined against ice or shore, or by use of fire resistant booms), the wind conditions are calm and the diesel oil film thickness is high (5 mm), Rotterdam diesel is predicted to have a prolonged time window for potential use of ISB (Hellstrøm *et al.*, 2017).

5.4 WRG (2014-0553)

5.4.1 Bench-scale study

The bench-scale study performed in the preliminary study in 2014, showed that WRG (then called WRD) had emulsifying properties (Sørheim and Daling, 2015) at both 2 °C and 10 °C, as shown in Figure 5-9. The analysis also showed that:

- The water uptake happened very rapidly
- The emulsions that formed were unstable at both temperatures
 - The emulsions contained large droplets of water and broke apart rapidly
 - The stability ratio (ability to retain water during settling) was 0.2, indicating a very unstable emulsion (see A.1.3 for further details)
- The emulsions had a light yellow colour
- The emulsion viscosities were lower than the viscosity of the water-free oil (105 and 179 mPa·s at 2 °C and shear rate 10 s⁻¹, respectively)

Results from other physical and chemical analyses are given in Table 5-1, and show that the WRG had:

- No significant evaporative loss beneath 250 °C (no volatile components with boiling point beneath this temperature)
- Low pour point (-24 °C)
- High flash point (115.5 °C)
- A relatively high wax content (4.7 wt. %)
- Low content of asphaltenes (0.01 wt. %)
- Emulsion viscosities were higher at 2 °C than at 13 °C
- "Oversaturation" of water in emulsion at 13 °C. Oversaturation occur when water is forced into the oil in higher volumes than realistically achievable in an acute spill. The phenomenon is known to occur in the emulsification testing with rotating cylinders, for certain oils.

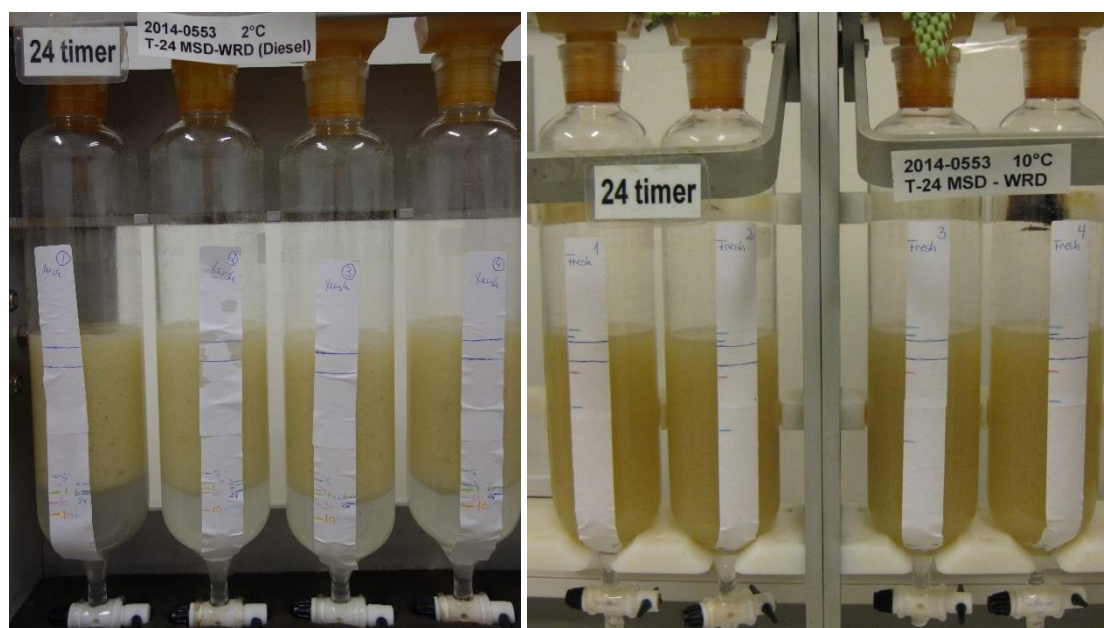


Figure 5-9: Show the emulsions of WRG formed at 2 °C (left, 87 vol. % water) and 10 °C (right, 91 vol. % water) in the rotating cylinders. The black lines on the white marks indicate the volume of water-free oil used in the test, and coloured marks indicate changes during the 24 hours of rotation.

5.4.2 Dispersibility

The dispersibility of WRG was tested using both the IFP and MNS-methodology. The screening study on six dispersants showed that Dasic Slickgone NS was the most effective dispersant, followed by Corexit 9500. Further dispersibility studies were performed at 2 °C and 13 °C and the results from these showed that:

- WRG had poor or reduced natural dispersibility (no dispersant), even in high-energy (breaking wave) conditions (< 30 % effectiveness, MNS)
- The oil showed a effectiveness when using dispersant dependant on the dispersant to oil ratio (DOR) in low-energy conditions (IFP), with higher effectiveness for higher DOR
 - With DOR = 1:25 the effectiveness was measured to 88 %
 - With DOR = 1:200 the effectiveness was measured to 7 %
- The chemical dispersibility was reduced at the lower temperature
 - The dispersibility was poor in low energy conditions at 2 °C
 - In breaking wave conditions, the effectiveness of chemical dispersion was good, > 50 % for all tested emulsions
- At 13 °C, WRG expressed good dispersibility in both high (MNS) and low (IFP) energy conditions.
- The emulsion viscosities varied between temperatures, as seen in Table 5-2
 - At 2 °C the emulsion viscosities surpassed 1000 mPa·s
 - At 13 °C the highest emulsion viscosity was measured to 205 mPa·s
- A limit for reduced dispersibility was set at 300 mPa·s based on the results from both test temperatures. This limit is considered conservative for summer temperatures (>10 °C)

No upper limit for poor dispersibility was obtained for WRG in the bench scale tests.

Table 5-2: Emulsion viscosities and their respective effectiveness in the dispersibility testing at both 2 °C and 13 °C

Wide Range Gas oil (SINTEF ID: 2014-0553)				
Temperature	Water content in emulsion (Vol. %)	Viscosity (mPa·s, 10 s ⁻¹)	Effectiveness (wt. %)	
			IFP	MNS
2 °C	0	179	13	99
	50	1244	2	53
	75	1057	8	58
	83	1081	12	65
13 °C	0	59	67	82
	50	205	80	100
	75	-	79	88
	91*	-	90	73

- emulsion viscosity was not possible to measure

*oversaturated emulsion

5.4.3 Meso-scale flume testing

Three meso-scale flume tests were performed on WRG, two tests were performed at 13 °C and one at 2 °C. The initial study was performed with varying energy settings at 13 °C, in order to assess the energy level necessary for emulsification to occur. Three energy levels were tested representing different sea states. The lowest energy setting presented swells, the intermediate setting presented non-breaking waves, while the highest energy setting created breaking waves in the flume. The results from this testing showed that:

- No water uptake or significant natural dispersion was measured at the lowest energy setting (swells)
- A change in colour and slightly increased dispersion was observed during the use of the intermediate energy setting (non-breaking waves)

- Emulsification occurred immediately when the highest energy setting was applied
 - The emulsion had a yellow tending to brown colour
 - The viscosity increased from 48 mPa·s (water-free oil) to 194 mPa·s (emulsion, 69 vol. %)
- Large droplets of emulsified oil (2-7 mm) and "oil-covered" water droplets (1.5-2 cm wide) were observed in the water phase (see Figure 5-10). The oil droplets rose rapidly, while the covered water droplets had lower buoyancy.
- Dispersion of oil/emulsion droplets increased with the highest energy setting
- Application of chemical dispersant was effective and total dispersion was observed

The two other meso-scale tests that were performed used standard (breaking wave) conditions, and used the same procedure as for other tests described for other oils in this report. The results can therefore be compared more directly between oils. The temperature dependant behaviour seen in the bench-scale study was also observed in the meso-scale testing. For the 13 °C, the results showed that:

- WRG emulsified rapidly and formed a light coloured foam-like emulsion, similar to the emulsion that was formed in the initial study
- The volume of water that was incorporated into the oil was high (approx. 80 vol. %)
- The emulsion viscosities were low (<600 mPa·s)
- Natural dispersion of oil increased throughout the test period
- Repeated application of chemical dispersant showed improved dispersion
 - The observed effectiveness was approx. 95 % of available oil
 - Measured oil-in-water indicated only dispersion of > 40 % of available oil
- Low dosage to oil ratio (DOR) was used in the testing (1:100)



Figure 5-10: Example of structure observed in the water column, assumed to be a water droplet covered by an oil/emulsion "skin". The large bulb was estimated to have a diameter of 2-3 cm.

For the 2 °C test, the results showed that:

- The emulsification occurred slower than at 13 °C
- The emulsion stability appeared to be higher at 2 °C than at 13 °C, and the stability seemed to increase during the test
- The emulsion viscosities were higher at 2 °C than at 13 °C, but decreased during the test period, from 1112 mPa·s initially to 548 mPa·s prior to dispersion
- The first application of chemical dispersant resulted in an increased emulsion viscosity (to 1536 mPa·s)
 - The second application gave a new decrease in emulsion viscosity (to 734 mPa·s)
- After 72 hours of weathering in the flume, the chemical dispersant Dasic Slickgone NS was applied three times, while Corexit 9500 was applied once as the fourth application. The application of dispersant occurred at 30 minutes intervals and the results showed that:
 - Dasic Slickgone NS had poor effect on the dispersibility; in general less than 20 % of available oil was dispersed. The applied DORs were low.
 - Corexit 9500 showed good effectiveness and was measured to remove more than 60 % of the available oil. The applied DORs were low.

It is difficult to say if the type of dispersant (Corexit) caused this observed change in effectiveness, or if it was caused by the significant dispersant concentrations that had been reached in the flume after four applications of dispersants.



Figure 5-11: Show the emulsion of WRG after 72 hours of weathering in the flume, prior to dispersion. Emulsion from testing at 2 °C is shown to the left, and emulsion from testing at 13 °C is shown to the right

5.4.4 Spreadability

WRG was used in the spreadability study previously presented in Chapter 5.1.3. Figure 5-5 shows the measured results from the testing. The red line draws an approximation of film thickness based on the results. The results reveal that WRG formed a thicker film compared to GO, and that the film was nearly 2 mm thick in the centre, decreasing gradually until it became too thin to measure approximately 110 cm from the centre. Figure 5-12 shows the progress of the experiment with WRG. Contrary to the behaviour of GO, WRG did not spread over the entire area of the test pool, and these result indicate that WRG will form thicker diesel oil film than GO in an acute event where spreading is not prevented.

Since WRG consist of a heavy distillate cut, the oil will not lose volume through evaporation. The formation of a thick film promote the likelihood of emulsification, and this weathering process may dominate the behaviour of WRG. With emulsification, the film thickness may increase. Figure 6-7 on page 52 show the predicted film thicknesses of all the tested oils.

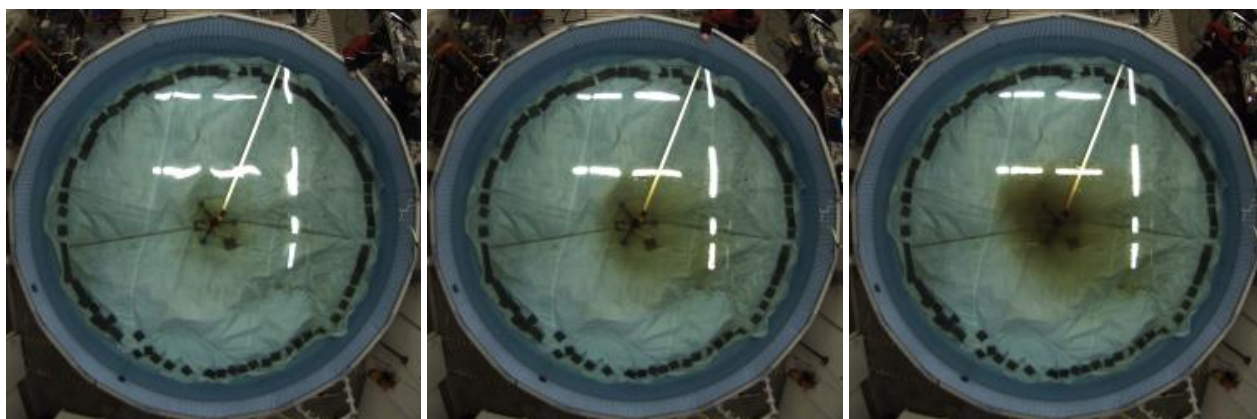


Figure 5-12: release of 4 Litres of WRG, shown after 20 seconds (left), 60 seconds (middle) and 120 seconds (right) after initiation

5.4.5 Ignitability

In the bench-scale ignitability study, WRG did not ignite despite the use of prolonged ignition time. WRG has a high flash point, 115.5 °C, which accounts for the difficulty of ignition. Though the oil did not ignite in the bench-scale study, WRG was attempted ignited in the up-scaled testing, and this was successful after 19 minutes and 20 seconds of ignition time. The oil burned for a little less than 8 minute, and the burning efficiency was calculated to 49 %.

The emulsifying properties of WRG, and its high flash point, will likely reduce the potential for use of ISB. Though the emulsions that formed were unstable, and little additional energy may be required for breaking up the emulsion, ISB will likely only be applicable in a short period before the oil emulsifies. An ignitor with higher temperature may reduce the ignition time, and may contribute to a longer window of opportunity.

The ignitability studies are described in detail in the memo-report (Hellstrøm *et al.*, 2017).

5.4.6 Batch variations for WRG

Variations between batches have been evaluated based on the measured values for the 2014 batch used in the presented experiment and measured results for a few parameters for a newer batch, produced and received in 2017, procured by NCA. The 2014 batch had a sulphur content of <0.25 % m/m, limiting it for use only outside SECA areas. The 2017 batch, however, has been produced with the aim to meet the sulphur requirements set for SECA areas, and has a low sulphur content (<0.05 % m/m). The reduction in sulphur content has resulted in an increased wax content, which has increased the oils' viscosity and pour point.

The parameters that were measured were density and viscosity of the 2017 batch, presented in Table 5-3, and GC-FID, shown in Figure 5-13. In Figure 5-13, the chromatograms of the 2014 batch and the 2017 batch have been overlapped, with the 2014 batch shown in blue and the 2017 batch shown in red. The chromatogram support the reported increase in wax content, and show a slightly heavier cut than the 2014 batch. The densities of the 2014 and 2017 batches were very similar. However, the viscosity of the 2017 batch is significantly higher than the viscosity measured for the 2014 WRG batch, at both test temperatures.

An increase in viscosity was expected based on the reported increase in wax content. However, the observed differences indicate that the 2017 batch can have significantly different weathering properties compared to the 2014 batch. An increased wax content can contribute to more stable emulsions and higher emulsion viscosities, while higher emulsion viscosities may further influence on the dispersibility of the oil. Based on the obtained data, the results presented in this project for the 2014 WRG batch cannot be assumed to be valid

for the 2017 batch. Assuming the composition of future WRG batches will be similar to the 2017 WRG batch, it is strongly recommended to perform more extensive characterisation of the 2017 WRG batch with respect to emulsifying properties, emulsion viscosities, and dispersibility and ignitability.

Table 5-3: Measured values for density and viscosity for two different batches of WRG, one from 2014 and another from 2017

Oil	Batch year	SINTEF ID	Density	Viscosity, 10 s ⁻¹	
				2 °C	13 °C
WRG	2014	2014-0553	0.886	179	59
	2017	2017-1880	0.883	844	123

As previously discussed in Chapter 4.2, the classification of the 2014 WRG batch would likely fall under the RMA-quality according to the current ISO 8217:2017 classification. With the increase in viscosity and pour point seen and reported for the 2017 batch, this new batch may not meet the criteria set for viscosity for RMA-qualities. The RMA category requires the kinetic viscosity to be below 10 mm²/s at 50 °C (ISO 8217, 2017), while the 2017 batch is reported to have kinetic viscosity of 18.7 at 40 °C (according to its Certificate of analysis).

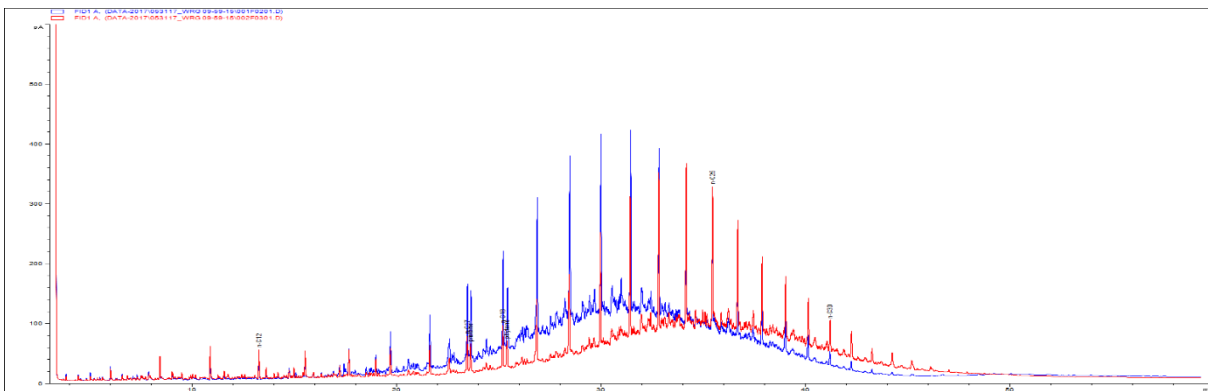


Figure 5-13: Overlapping gas chromatograms of the 2014 WRG batch (blue) and the 2017 WRG batch (red). Note that the new batch has a higher content of waxy components.

5.5 HDME 50 (2016-0231)

5.5.1 Bench-scale study

HDME 50 is a heavy distillate cut with physical and chemical properties that cannot be categorized within the ISO standards. It is one of two hybrid fuel oils tested in this project. The chromatogram in Figure 5-2 show that HDME has a large UCM (unresolved complex mixture) hump and *n*-alkanes in the range between C₁₅-C₃₅. The bench-scale study also showed that HDME 50 has:

- High wax content (9.5 wt. %)
- Some asphaltenic content ("Hard asphaltenes"; 0.06 wt. %)
- High density (0.903 g/ml)
- High flash point (186 °C)
- No significant content of components with boiling point below 250 °C
- High viscosity at low temperatures
- High pour point (12 °C)

Other key results from the bench-scale testing is presented in Table 5-1.

The emulsifying properties of HDME 50 was tested at three different temperatures, 2, 13 and 20 °C. Figure 5-14 show the emulsifying properties of HDME 50 after 24 hours of rotation. The results showed that HDME 50 emulsified at all three temperatures, and that the water uptake varied with the test temperature, increasing with higher temperatures. The results also showed that:

- HDME 50 incorporated small volumes of air into the emulsions, particularly at lower temperatures
- HDME 50 formed emulsions with a light brown colour
- The rate of water uptake ($t_{1/2}$) varied with the test temperature, being more rapid at higher temperatures, and slower at lower temperatures
- The emulsions were stable regardless of temperature
 - The use of demulsifier was effective and dosage dependant at 13 and 20 °C
 - The use of demulsifier was not effective at 2 °C
 - The stability ratio ranged from 0.96-1, indicating very stable emulsions (see Appendix A.1.3)
- The emulsion viscosities of HDME 50 increased with higher water content
- The emulsion viscosities were temperature dependant
 - Pour point properties influenced the viscosities at lower temperatures resulting in high viscosities

The emulsion viscosities of HDME 50 are presented for all test temperatures in Table 5-4. The viscosity of water-free oil at 2 °C was more than 10 times the viscosity measured at 13 °C. HDME 50 had a pour point of 12 °C, and high viscosity at 2 °C is likely influenced by the pour point.

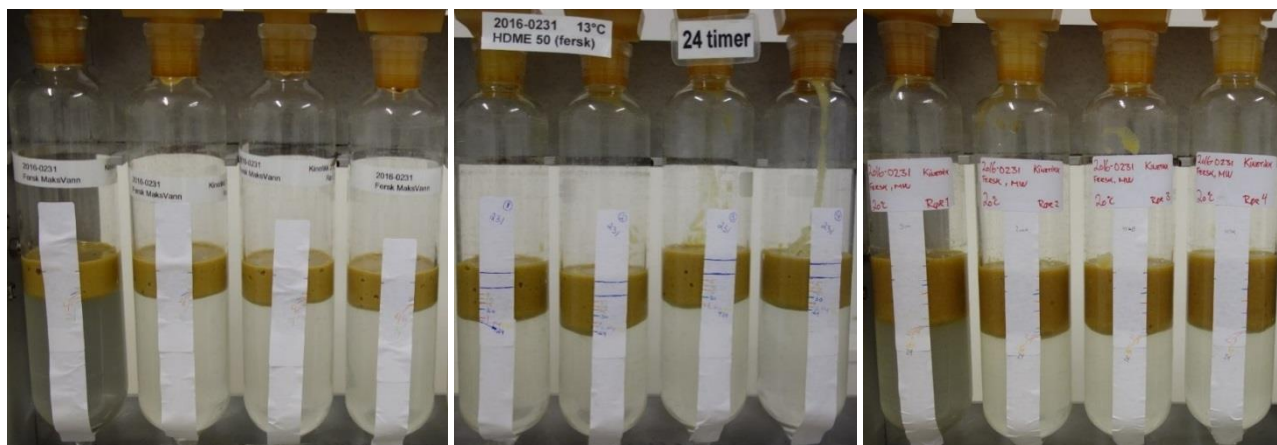


Figure 5-14: The emulsifying properties of HDME 50 at 2 °C (left, 52 vol. % water), 13 °C (middle, 68 vol. % water) and 20 °C (right, 77 vol. % water) after 24 hours of rotation. The emulsions are formed with an excess of seawater, meaning that the available volume of water is higher than realistically possible for the oil to incorporate into an emulsion (> 90 vol. % water). The emulsions incorporate the maximum possible water content and are so-called max water emulsions

Table 5-4: Emulsion viscosities for HDME 50 at 2 °C, 13 °C and 20 °C, given as mPa·s measured at shear rate 10 s⁻¹. Emulsions that were not formed are marked with –, while untested emulsions are marked with greyed cells. The water content of max water emulsions are listed for 2/13/20 °C.

Water content (vol. %)	Emulsion viscosity of HDME 50		
	2 °C	13 °C	20 °C
	mPa·s, 10 s ⁻¹	mPa·s, 10 s ⁻¹	mPa·s, 10 s ⁻¹
0	11002	1005	369
50	-		2020
75	-		5252
52/68/77	19719	9529	6111

5.5.2 Dispersibility study

A screening of chemical dispersants were performed on an emulsion of HDME 50, containing 50 vol. % sea water, at 13 °C using the IFP methodology. The results are presented in Figure 7-1 and show generally low effectiveness, with Radiagreen and Dasic Slickgone NS as the most effective chemical dispersants (34 and 26 % effectiveness, respectively). A dosage and dispersibility study was performed at 13 °C using Dasic Slickgone NS and both MNS and IFP methodology. The results showed:

- In low-energy conditions the effectiveness was reduced (< 30 %), even for the highest dosage (DOR = 1:25, 26 % effectiveness, IFP-test)
 - Lower dosages (1:50, 1:100 and 1:200) had no significant effect (< 5 %)
- In high-energy (breaking wave) conditions (MNS), the effectiveness of the highest dosage (DOR = 1:25) was good (effectiveness of 62 %), but lower dosages (1:50, 1:100 and 1:200) showed poor effectiveness (< 20 %)
- Water-free oil and emulsions with low water content had higher effectiveness.

Based on the results HDME 50 is considered to have reduced or poor dispersibility when dispersant is used in calm weather conditions (IFP). In breaking wave conditions (MNS), reduced dispersibility can be expected for emulsions with viscosities >3000 mPa·s. Emulsions viscosities above 10 000 mPa·s are expected to have poor dispersibility, even in high-energy conditions.

The dispersibility of HDME 50 was tested at 2 and 20 °C using the Field Effectiveness Tests (FET) in order to observe any temperature dependant behaviours regarding dispersibility of HDME 50. The results from the FET-testing showed that:

- Emulsions of HDME 50 and the water-free fraction were dispersible at 20 °C
- Water-free HDME 50 showed reduced dispersibility at 2 °C, while emulsions were not dispersible
 - Poor dispersibility at 2 °C were caused by solidification of oil and emulsion, due to high pour point (12 °C)

5.5.3 Meso-scale flume testing

The weathering properties of HDME 50 were tested in two meso-scale flume experiments at 2 °C and 13 °C, and the results are presented in detail in the memo report (Hellstrøm et al., 2017). The meso-scale flume experiment allow weathering processes such as evaporation and emulsification to occur simultaneously, and can give valuable information regarding an oils' properties at different temperatures. Based on the results from the bench-scale and dispersibility studies, HDME 50 was expected to show some temperature dependent properties. The meso-scale testing showed that:

- HDME 50 expressed temperature dependant behaviour
 - HDME 50 solidified upon application at 2 °C but not at 13 °C
- At 13 °C HDME 50 emulsified rapidly and formed homogeneous emulsion with entrained water droplets (as seen in Figure 5-15 (left))
 - Water content in emulsion after 72 hours was 61 vol. %
 - The emulsion viscosity increased over time (from 6864 mPa·s to 20232 mPa·s)
 - Application of chemical dispersant resulted in a change in appearance, giving the oil a light yellow, creamy colour
- At 2 °C the emulsification occurred slowly, and only on the surface of solidified oil lumps
 - Water uptake was low, 33 vol. % after 72 hours
 - Emulsion viscosity decreased over the first days of testing, from 49275 mPa·s to 18730 mPa·s
- There was no significant natural dispersion at either test temperature
- Applications of chemical dispersant resulted in a reduced emulsion viscosity at both temperatures
 - Repeated application gave higher effectiveness
 - The effectiveness of chemical dispersant was highest at 2 °C
 - The emulsion dispersed
 - Oil lumps remained unaffected

Figure 5-15 shows the emulsions of HDME 50 after 72 hours of weathering at 13 °C (left) and 2 °C (right), prior to application of chemical dispersant. Solidified oil lumps can be perceived at 2 °C, while the emulsion at 13 °C and entrained large pockets of water.



Figure 5-15: Emulsions of HDME 50 after 72 hours of weathering, prior to dispersion. The homogenous emulsion (20232 mPa·s) that formed at 13 °C is shown to the left, while the right hand picture show the emulsion (18730 mPa·s) at 2 °C, which contained solidified oil.

5.5.4 Ignitability

HDME 50 was not tested in the bench-scale ignitability study due to its high flash point (186 °C) and based on failed ignitability of oils with lower flash points (WRG; 115.5 °C). However, HDME 50 was attempted ignited in the up-scaled testing, and though the ignition time required was long (18 minutes), this hybrid fuel oil eventually ignited. Three packs of gelled gasoline was necessary for the ignition of HDME 50, and the burning efficiency was the lowest of the six tested oils, only 34 %.

The uptake of water will rapidly decrease the potential for use of ISB since additional energy will be required for breaking the emulsion and removal of the water. In the up-scaled testing, only water-free oil was used. Since HDME 50 has shown emulsifying properties (though slow at low temperatures), and has a very high flash point, ISB can only be expected applicable in very short period of time or under special circumstances where emulsification has been prevented.

The use of an ignitor with a high burning temperature may allow prolonged window of opportunity for ignition of the oil. However, since the oil consist of a heavy distillate cut the burning efficiency will likely be poor and a very high burning temperature would be necessary for improved burning efficiency.

5.5.5 Batch variation for HMDE 50

Since the HDME 50 is a distillate product, some variation may be expected between batches. A new batch of HDME 50 produced in 2017 has been compared with the 2016 batch used in the present study. The comparison has been made with a very limited number of analyses; density (g/ml), viscosity (2 and 13 °C) and GC-FID analysis. The results for the 2017 batch is given in Table 5-5 and Figure 5-16 along with similar, previously obtained data for the 2016 batch. The chromatograms indicate that the 2017 batch has a smaller UCM hump especially in the heavier end, and has less of the heavier *n*-alkanes. The peak of the 2017 batch is approximately at C₂₅, while for the 2016-batch the peak was approximately at C₂₇. These result could

indicate that the 2017 batch would have a lower density than the 2016 batch, but the results show that the newer 2017-batch of HDME 50 has a higher density than the 2016-batch. However, the difference is not considered big. The viscosities measured for the 2017 batch were significantly lower than the viscosities measured for the 2016 batch. Such variations can be expected to cause different weathering properties of the 2017 batch of HDME 50 compared to the 2016 batch. A lower viscosity may influence the water uptake and the dispersibility of the oil. HDME 50 is a distillate cut, and less variation is expected for this type of fuel oil compared to fuel oils that are mixed from different cuts, such as Shell ULSFO. In order to make a well based evaluation of the degree variation between batches, key properties such as emulsifying properties, density, viscosity, and wax and asphaltene content of 3-5 different batches should be analysed. Further analysis and testing are recommended in order to follow up if the batches of HDME 50 vary significantly over time.

Table 5-5: Measured values for density and viscosity for two different batches of HDME 50, one from 2016 and another from 2017

Oil	Batch year	SINTEF ID	Density	Viscosity, 10 s ⁻¹	
				2 °C	13 °C
HDME 50	2016	2016-0231	0.903	11002	1005
	2017	2017-1879	0.910	4500	409

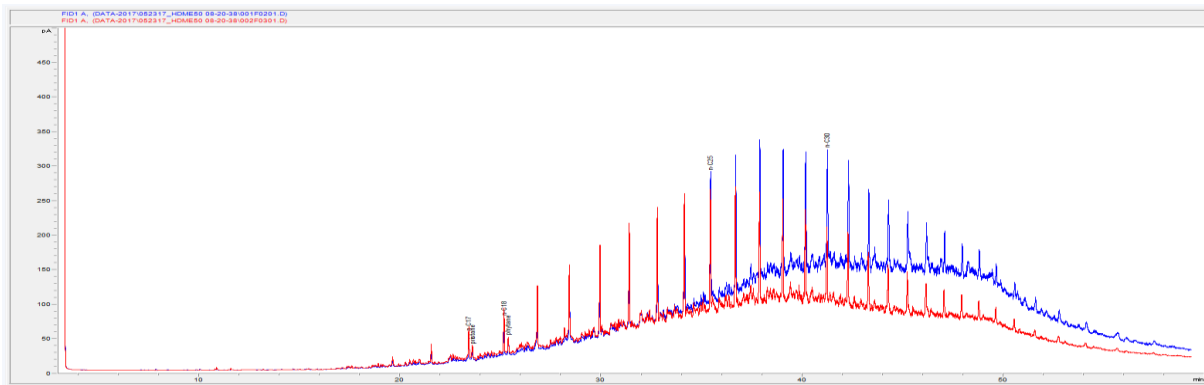


Figure 5-16: Overlapping gas chromatograms for two batches of HDME 50, one from 2016 (blue) and one from 2017 (red).

5.6 Shell ULSFO, 2016-0233

5.6.1 Bench-scale study

Shell ULSFO is a hybrid fuel oil that consist of a residual fuel oil containing a broad spectre of lighter and heavier components, and the chromatogram in Figure 5-2 show that Shell ULSFO contain *n*-alkanes in the range between C₉-C₄₀. Other bench-scale analyses revealed that Shell ULSFO (non-weathered) had:

- Very high wax content (13.1 wt. %)
- High pour point (24 °C)
- Relatively high asphaltenic content compared to the other tested oils (0.15 wt. %)
- High viscosity at test temperatures (13106 and 4300 mPa·s at 2 and 13 °C, respectively)
- Flash point (75 °C) within the range observed for tested DMA-qualities (62.5-82.5 °C)
- Solidification issues at room temperature
- Low evaporative loss (14.6 vol. % of components had boiling point below 250 °C)

The obtained results from the bench-scale analyses are presented along with similar results for the other tested oils in Table 5-1. Testing of emulsifying properties with rotating cylinders was performed on Shell ULSFO at three different test temperatures, 2, 13 and 20 °C, and Figure 5-17 shows the rotating cylinders after 24 hours of rotation at the three test temperatures. The testing revealed that Shell ULSFO had emulsifying properties that depended on the test temperature. The results are presented in further detail in the memo-report (Hellstrøm et al., 2017), but a short summary is given here. The results showed that:

- Fresh and weathered Shell ULSFO had emulsifying properties at all test temperatures
 - Weathered Shell ULSFO (250°C+) solidified at 2 °C, and had only a low water uptake at this temperature
 - Fresh Shell ULSFO solidified initially, but emulsified over time
- Fresh Shell ULSFO had a higher water uptake than its weathered residue, independent of temperature
- Total water uptake was lower at colder temperatures and higher at warmer temperatures
 - At 2 °C the water uptake was 65 and 14 vol.% for fresh and weathered Shell ULFSO, respectively
 - At 13 °C the water uptake was 84 and 71 vol.% for fresh and weathered Shell ULFSO, respectively
 - At 20 °C the water uptake was 91 vol. % (oversaturated) and 82 vol.% for fresh and weathered ULFSO, respectively
- Formation of waxy lumps occurred at 13 °C
- The rate of water uptake ($t_{1/2}$) was temperature dependant, being more rapid at higher temperatures, and slower at lower temperatures
- The emulsions were overall stable regardless of temperature and fraction (fresh or weathered Shell ULSFO)
 - The stability ratio ranged from 0.88-1, indicating little loss of water content and stable emulsions (see Appendix A.1.3 for definition)
 - Use of demulsifier was effective at high dosage (2000 ppm) for fresh oil emulsions at all temperatures
- Emulsion viscosities of Shell ULSFO varied with temperature and water content, see Table 5-6
 - Higher emulsion viscosities were observed with higher water content for fresh Shell ULSFO emulsions
 - In general, emulsion viscosities were higher at lower temperatures

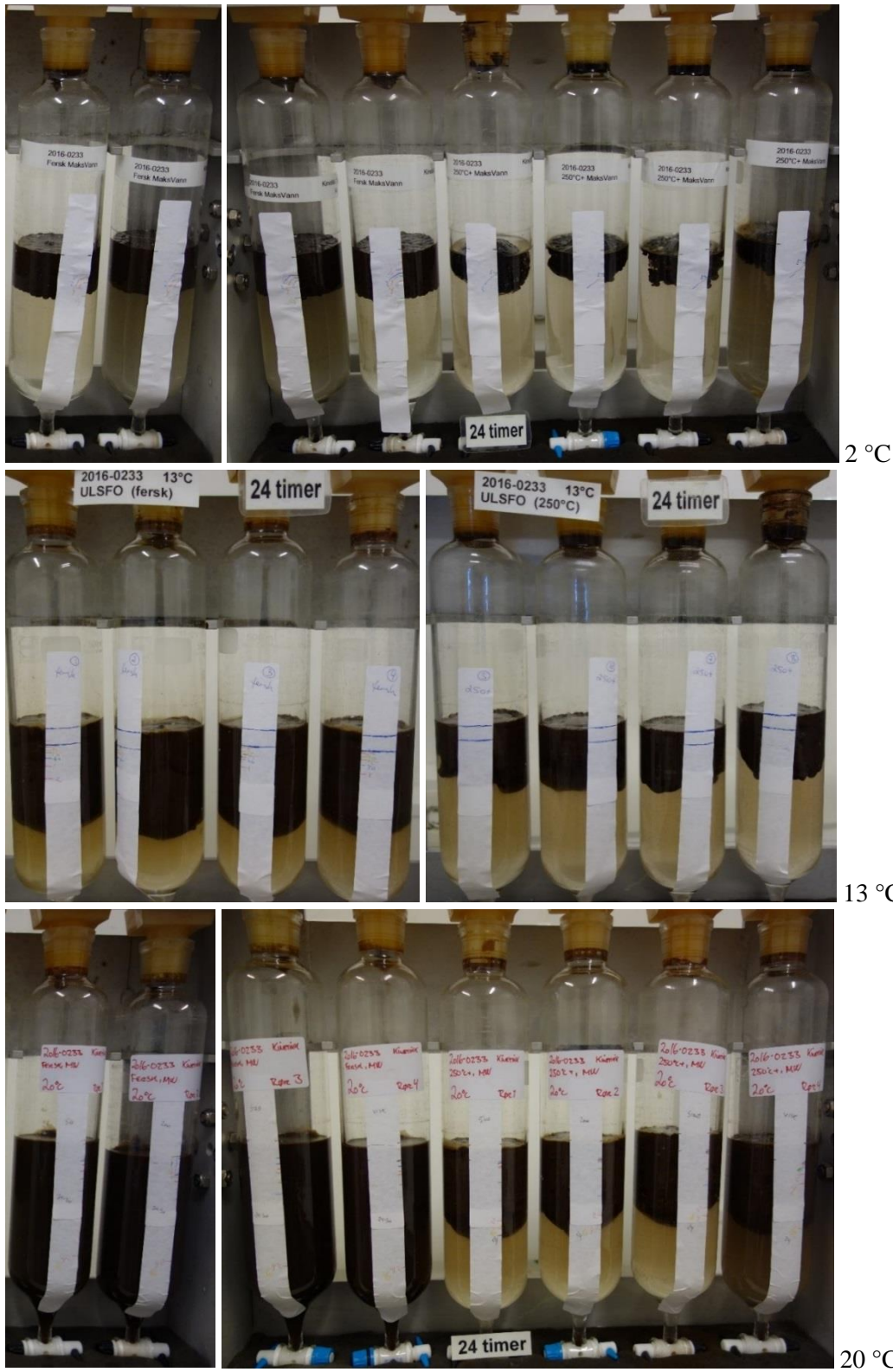


Figure 5-17: Rotating cylinders from testing of emulsifying properties of Shell ULSFO at 2 °C (upper pictures), 13 °C (middle pictures) and 20 °C (lower pictures). Fresh oil emulsions are shown in the four left cylinders, while emulsions from the weathered fraction are shown in the four right cylinders.

The viscosities for Shell ULSFO emulsions are listed in Table 5-6. The large differences in viscosity between temperatures were caused by influence from the pour point properties of the oil. When oil solidifies, stable wax lattice formations are formed, and these require energy in order to be broken. The wax lattices affect the viscosity of the oil by increasing the liquids resistance to flow, and this influence is stronger with lower temperatures. For the water-free oil and 250°C+ residue, viscosities were measured at share rate of 100 s⁻¹, in order to assure that the wax lattices had been broken and did not influence the viscosity. For all emulsions, their viscosities were measured at share rate of 10 s⁻¹, since the incorporation of water will have broken the wax lattices prior to the viscosity measurement.

Table 5-6: Emulsion viscosities of Shell ULSFO emulsions at three test temperatures, 2, 13 and 20 °C, measured at share rate 10 s⁻¹ for the emulsions and 100 s⁻¹ for the water-free residues

Fraction	Water content (vol. %)	Viscosity (mPa·s) 2 °C	Viscosity (mPa·s) 13 °C	Viscosity (mPa·s) 20 °C
ULSFO Fresh	0	1617*	663*	278*
	50	15664		593
	75	-		2624
	68/84/91	15931	7770	5490
ULSFO 250°C+	0	10152*	4285*	1604*
	50	-		2596
	75	-		8674
	14/71/82	31602	17061	14480

*measured at share rate 100 s⁻¹

5.6.2 Dispersibility

The screening of chemical dispersants performed on emulsions and water-free fractions of Shell ULSFO at 13 °C showed that Corexit 9500 and Dasic Slickgone NS were the most effective chemical dispersants (see Figure 7-1, and the memo report for full details). Dasic Slickgone NS was chosen for further use in a dosage and dispersibility study at 13 °C. The results showed:

- A clear dosage dependent effectiveness at low-energy (swells) conditions (IFP)
 - 5-52 % effectiveness for dispersant to oil ratios (DOR) of 1:200-1:25, respectively
- High effectiveness at breaking wave conditions (MNS) independent of dosage
 - >90 % effectiveness at all DORs
- Reduced dispersibility of emulsions with viscosities >4000 mPa·s
- Poor dispersibility of emulsions with viscosities > 9000 mPa·s

In addition to the dispersibility study performed with the MNS and IFP-methodologies, Field Effectiveness Tests (FET) were performed on water-free fractions and maximum water emulsions of fresh and weathered Shell ULSFO at 2 and 20 °C, in order to obtain an indication of the dispersibility of this hybrid fuel oil at different temperatures. The results from this testing showed that:

- Fresh Shell ULSFO emulsions were dispersible at 20 °C, but had reduced dispersibility at 2 °C
- Weathered 250°C+ emulsions with low water content were dispersible at 20 °C, but the dispersibility was reduced with increased water content
- At 2 °C, emulsions of the weathered 250°C+ residue were not dispersible
 - The poor dispersibility at 2 °C was caused by the solidification of the oil

5.6.3 Meso-scale flume testing

In order to obtain more information regarding the behaviour and dispersibility of Shell ULSFO at different temperatures, meso-scale flume testing were performed at 2 and 13 °C. As expected, the behaviour of Shell ULSFO varied between these two test temperatures, and the significant difference in appearance after 72 hours is shown in Figure 5-18.



Figure 5-18: The appearance of Shell ULSFO weathered for 72 hours at 13 °C (left) and 2 °C (right)

At 13 °C, a homogeneous slick of emulsion was slowly formed over the first four hours before it broke into thicker patches. The emulsion viscosities increased during the 72 hours of testing from 5522 mPa·s to 24635 mPa·s, prior to dispersion. There was no natural dispersion, and the application of chemical dispersion was seen to have limited effect. The application of chemical dispersant (Dasic Slickgone NS) were performed three times, and the observed and measured effects were:

- Decreased emulsion viscosity (24 635 mPa·s to 13 845 mPa·s)
- Some oil was dispersed (< 20 %) after repeated treatment with dispersant
- The emulsion patches seen prior to dispersion were broken into smaller lumps of emulsion.

At 2 °C, the oil solidified immediately upon application, and formed lumps that slowly emulsified on the surface. The emulsion viscosities were reduced with increasing water uptake over the first 24 hours from 18347 mPa·s to 9800 mPa·s, and the emulsifying lumps adhered to each other. Throughout the test, large volumes of the oil and emulsion stuck to the flume walls. After 72 hours, less than 20 % of the originally applied oil was left in the flume (approx. 60 % had adhered to the flume walls), and large lumps of solidified oil were present within the emulsion. The application of chemical dispersant had a good effect on the emulsion but no effect on the remaining lumps. Approximately 30 % of the available surface oil dispersed.

Based on the obtained results, the following conclusions from the flume testing were made:

- The behaviour of Shell ULSFO was temperature dependent and solidification was a major challenge for this oil due to its high pour point.
 - Development of emulsion viscosities differed between temperatures
- Independently of temperature, Shell ULSFO rapidly formed emulsion viscosities that were above the lower limit for dispersibility (4000 mPa·s), previously established in the bench-scale dispersibility study
- The effectiveness of chemical dispersants was low in both flume experiments
 - The effectiveness was higher for the 2 °C emulsion (30 %) compared to the 13 °C emulsion (20 %)
 - Solidified oil lumps at 2 °C were not affected by application of chemical dispersant
- Adsorption to solid surfaces, such as the flume wall, was a challenge at both test temperatures

Based on the findings from the bench-scale study, the dispersibility study and the meso-scale study, Shell ULSFO is considered to have a poor potential for use of chemical dispersants, especially in winter conditions.

5.6.4 Spreadability

The spreadability of Shell ULSFO was not tested. However, the high pour point of the oil will limit the spreading of the oil, and solidification of the oil will occur at winter temperatures.

5.6.5 Ignitability

The results from the bench-scale and up-scaled ignitability testing (described in detail in the memo-report, and summarised in chapter 5.2.5) showed that:

- Fresh Shell ULSFO was found to be ignitable in the bench-scale testing (burning cell) under the standard test conditions (3 attempts with 10 sec. ignition time)
- The weathered residue ignited after prolonged ignition time (30 sec.)
- An emulsion of fresh Shell ULSFO and 10 vol. % water was also ignited after prolonged ignition time (20 sec.)
- Fresh Shell ULSFO ignited within one minute in the up-scaled testing

Due to the emulsifying properties of the oil, and the stability of the emulsion that form, Shell ULSFO is not expected to have a long time window for in-situ burning.

6 Comparison of predictions related to oil spill response

In this section, the predicted weathering properties of the six tested fuel oils are compared and related to challenges in oil spill response. The presented comparisons are based on predictions limited to a temperature of 5 °C and wind conditions of 10 m/s (breaking wave conditions), with the exception of remaining surface oil and emulsion film thickness, which are presented at 2, 5, 10 and 15 m/s wind conditions. The properties are presented with the assumption that the oils will remain on the sea surface for 5 days. However, at breaking wave conditions (10-15 m/s wind) the DMA-qualities will all have been naturally dispersed within the first day (see Figure 6-6).

6.1 Evaporative loss

The predicted evaporative loss of the tested marine fuel oils are shown in Figure 6-1. The behaviour reflect the content of lighter, easily evaporated components in the oil, with the light GO oil expressing the highest evaporative loss (approx. 95 % after 5 days of weathering). Although they are within the same fuel classification (DMA), both MGO and Rotterdam diesel are predicted to have a significantly lower evaporative loss compared to GO. This shows that diesel oils within the same classification may have a wide range in degree of evaporation. The Shell ULSFO hybrid fuel oil had low evaporative loss, and less than 20 vol. % are predicted lost during the first five days of weathering. The HDME 50 and WRG oils both have low content of lighter components and their evaporative losses are predicted to be very similar (< 2 vol. % within 5 days), and overlap in the figure below, hence the visual lack of WRG.

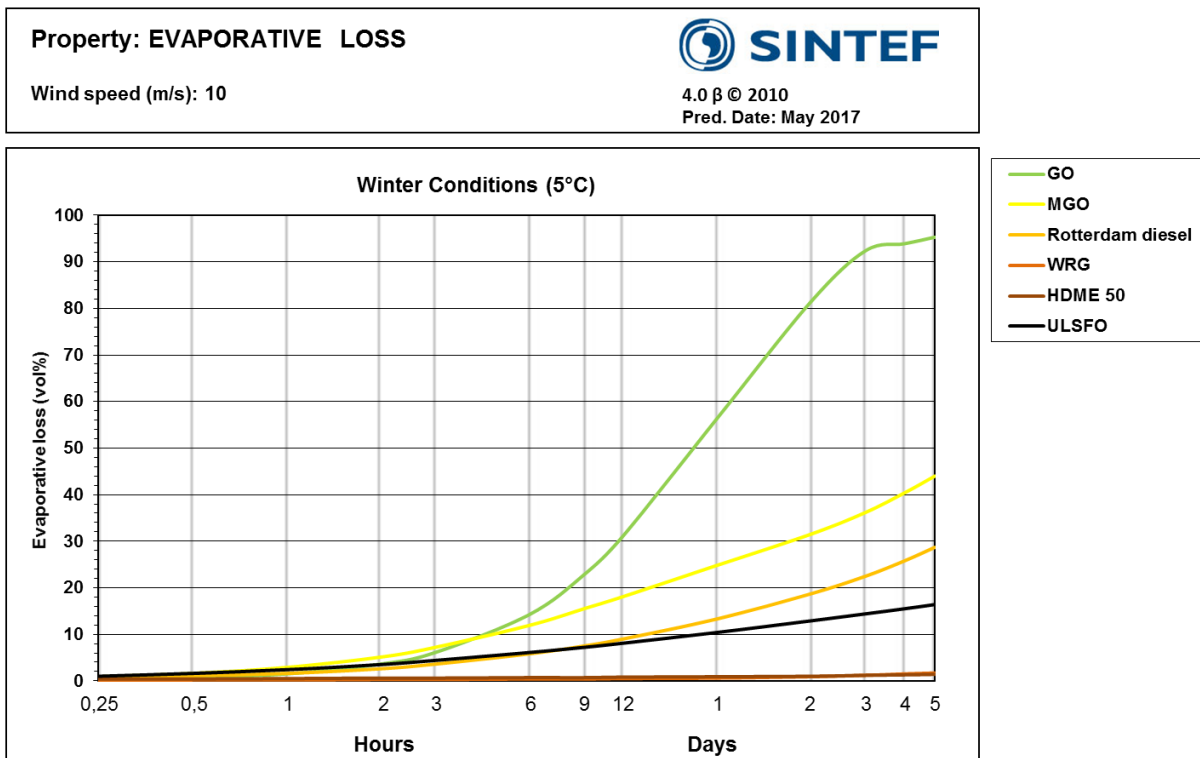


Figure 6-1: Comparison of predicted evaporative loss at 5 ° and 10 m/s wind for six tested marine fuel oils

6.2 Flash point

Marine fuel oils of DMA-quality and heavier qualities are required to have a flash point above 60 °C, according to the ISO 8217 standard (Table 4-1). All the tested oils meet this requirement, with MGO having the lowest flash point (62.5 °) and HDME 50 having the highest (186 °C) flash point. With the evaporation of lighter, more easily ignitable components, the flash point of an oil (whether crude or fuel oil) will increase. Figure 6-2 show the predicted development of the flash points for the six tested oils. For the HDME 50 and WRG, the change in flash point is small, due to the low evaporative losses of these oils. The flash points of MGO and GO are predicted to develop similarly over the first five days of weathering, while the Shell ULSFO hybrid fuel oil show a general increase. The biggest change is seen for Rotterdam diesel, which show a large increase in flash point despite expressing a relatively low evaporative loss. A higher relative content of heavier distillate cut compared to MGO and GO can explain this behaviour.

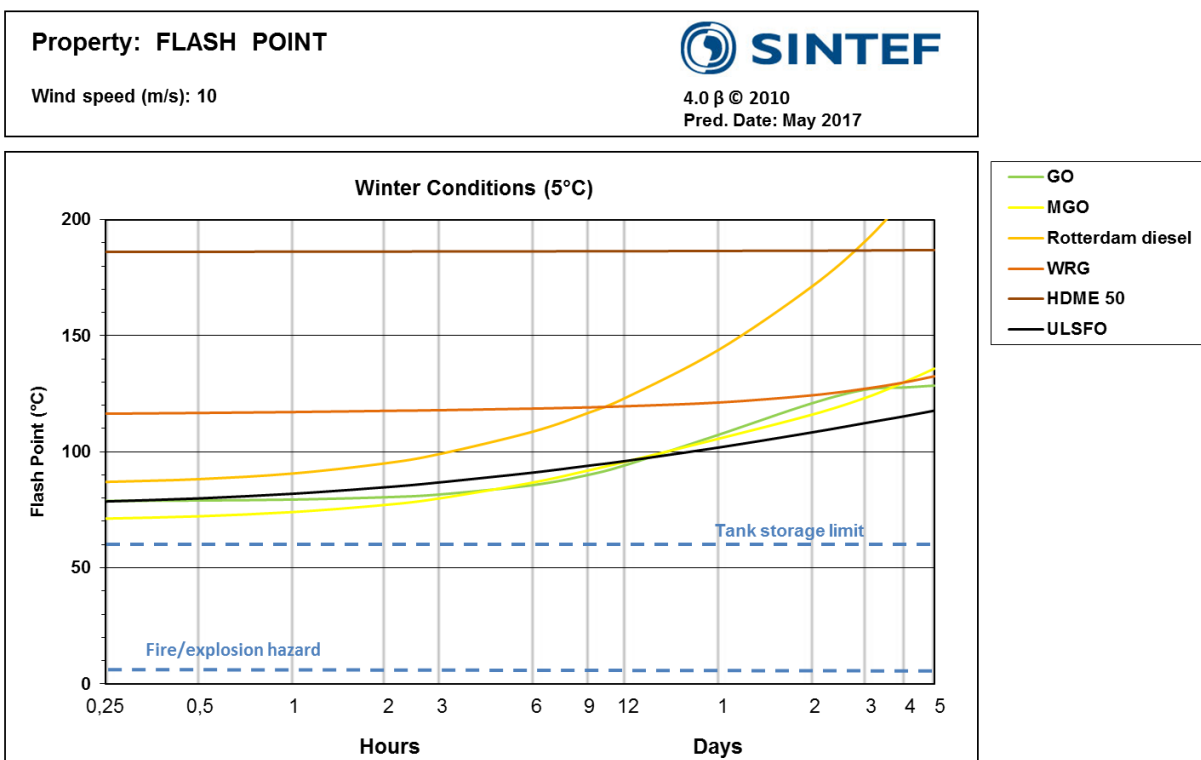


Figure 6-2: Predicted flash point for the tested marine fuel oils at 5 °C and 10 m/s wind

6.3 Pour point

An oil's pour point depends on the chemical composition of the oil. A high wax content may cause precipitation of wax and the formation of wax lattice structures within the oil. This would cause the oil to solidify. However, the presence of lighter components may contribute to keep the present wax dissolved in the oil, thus preventing solidification of the oil and lower the pour point. A high asphaltenic content will also contribute to a lower pour point as the asphaltenes prevent the formation of wax lattice structures and precipitation of wax. When the lighter components are removed from the oil due to evaporation, the pour point will increase, thus increasing the risk of solidification.

The pour point of oil may influence the dispersant effectiveness since a high pour point may prevent the dispersant to soak into the oil slick, and thus change the interfacial tension between the oil and the water phase. If prevented from soaking into the oil, the dispersant is likely to be washed into the water phase and have no effect. Previous studies have shown that such solidification of the oil on the sea surface takes place typically when the pour point becomes higher than 5-15 °C above the sea temperature (Daling *et al.*, 1990).

Figure 6-3 shows the predicted pour point of the tested oils. The predictions express similar trends as seen for the predicted flash point, with very little change for HDME 50 and WRG, a slight increase for Shell ULSFO, and similar predicted behaviour for GO and MGO. The largest change is seen for the Rotterdam diesel, which is predicted to show an increase in pour point from -36 °C to + 8 °C within 5 days at sea. This development is likely caused by the loss of the lighter components that initially keep the wax content dissolved. Rotterdam diesel had a high wax content than the other two DMA-qualities MGO and GO (see Table 5-1), and with the evaporation of the lighter components in the oil, the relative content of wax increases enough to influence the pour point of the oil. Based on the presented prediction, only Shell ULSFO would be expected to solidify on the sea surface at 5 °C, while HDME 50 would be close to solidifying.

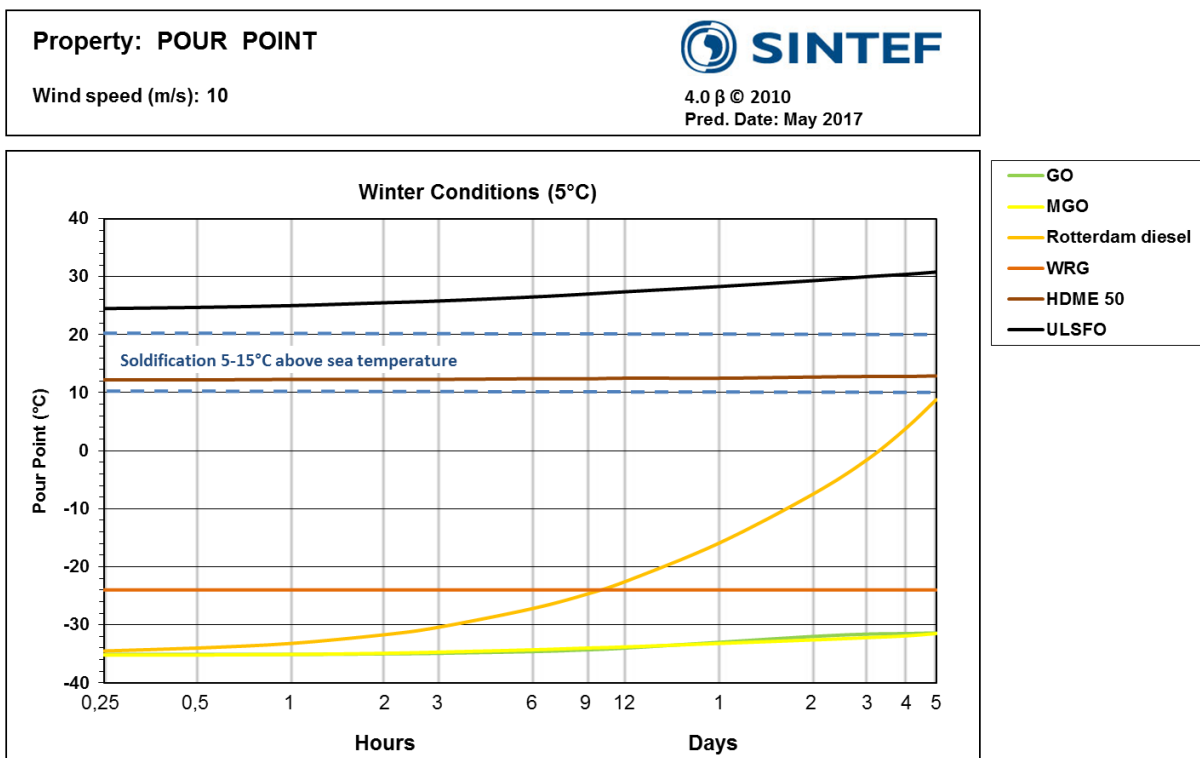


Figure 6-3: Predicted pour point for the tested marine fuel oils at 5 ° and 10 m/s wind. The dotted lines indicate the pour point that must be achieved before solidification is likely to pose an issue.

6.4 Water uptake

The predicted water uptake of the tested oils at two temperatures, 5 and 15 °C, are presented in Figure 6-4. Of the tested DMA-quality fuels, only MGO was observed to have some form of emulsifying properties, and the predicted water uptake shows a predicted water content of 10 vol. % for MGO after 2 days at sea at both the presented temperatures. In comparison, GO and DMA diesel show insignificant (< 5 vol. %) water uptake.

Wide Rage Gas oil (WRG) was seen to have a high water uptake in both the bench-scale study and the meso-scale study, but expressed different behaviour with varying temperatures. To reflect these differences, two different sets of data input were used in OWM to better reflect the oils behaviour at the relevant temperatures. WRG is predicted to have a high water uptake, up to 80 vol. % after 1 day on the sea surface at 5 °C, and up to 72 vol. % at 15 °C after 6 hours of weathering.

Of the hybrid fuel oils Shell ULSFO and HDME 50, Shell ULSFO is predicted to have the highest water uptake, stabilising just below 60 vol. % after 1 day of weathering at 5 °C, and nearly 80 vol. % water at 15 °C. Two different input data sets were used for HDME 50, in order to better reflect this oils behaviour at the various temperatures, similarly to WRG. HDME 50 is predicted to have a water uptake of 40 vol. % after 3 days of weathering at 5 °C, but at 15 °C this oil is predicted to emulsify more rapidly and obtain a higher water content than at 5 °C. After 6 hours of weathering at 15 °C, HDME 50 is predicted to have a water content of approx. 60 vol. %.

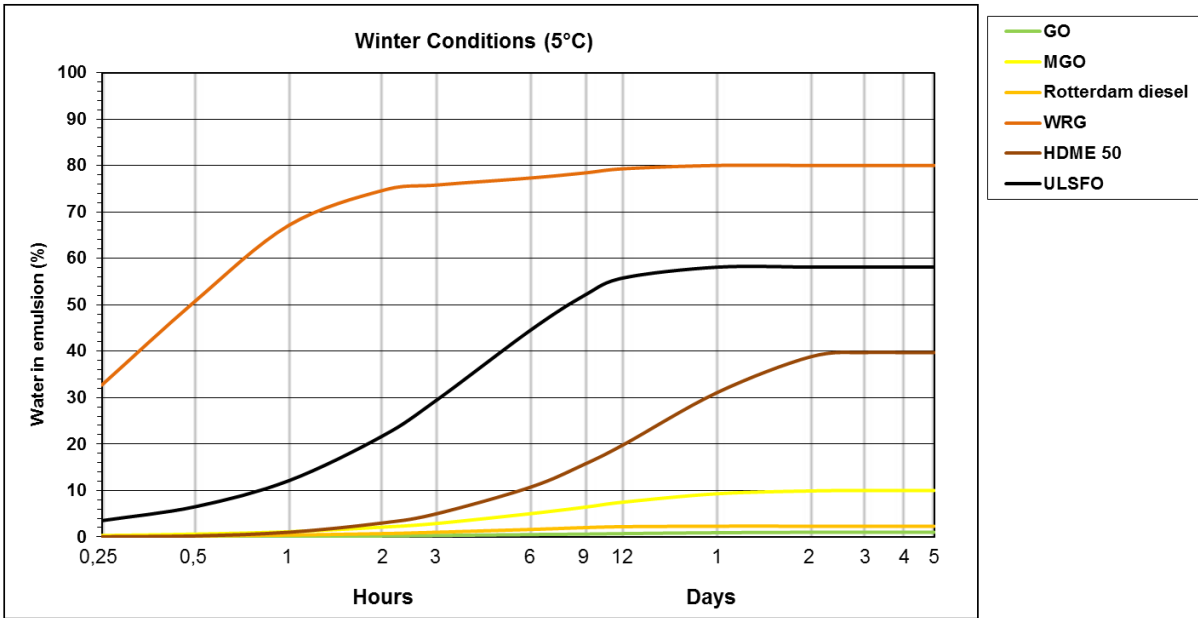
The rate of water uptake vary for the tested oils with WRG showing a rapid water uptake at both temperatures, Shell ULSFO expressing a slower rate of water uptake at both temperatures and HDME 50 showing a varying rate of water uptake depending on temperature.

Property: WATER CONTENT

Wind speed (m/s): 10



4.0 β © 2010
Pred. Date: May 2017



Property: WATER CONTENT

Wind speed (m/s): 10



4.0 β © 2010
Pred. Date: June 2017

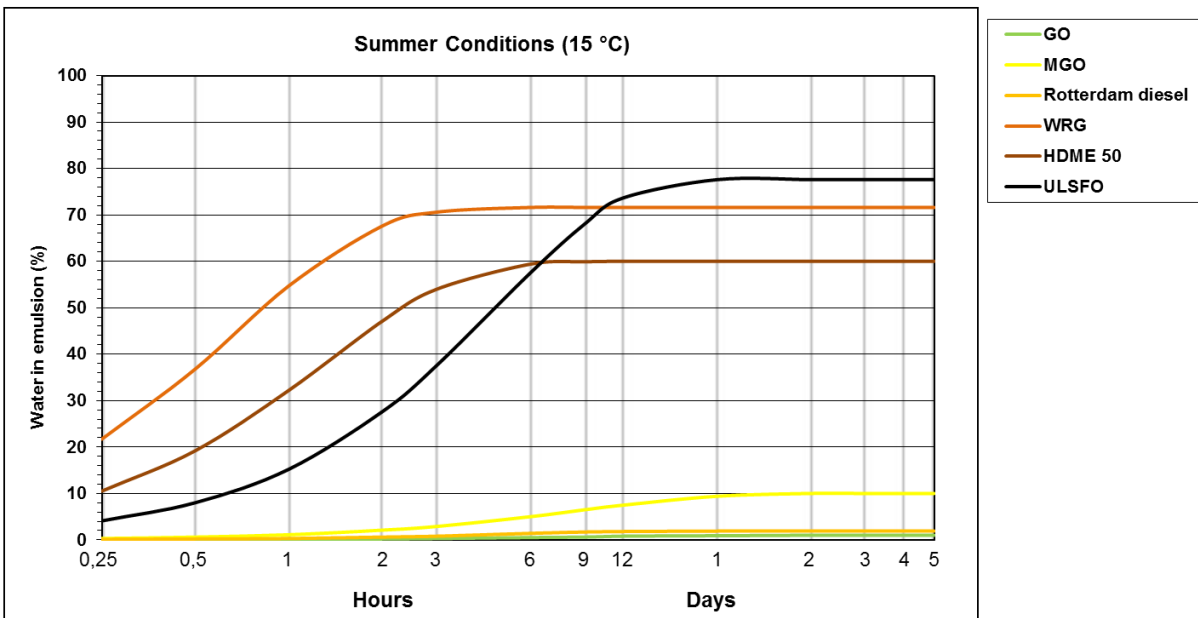


Figure 6-4: Predicted water uptake for the tested marine fuel oils at 10 m/s wind and 5 °C and 15 °C

6.5 Emulsion viscosity

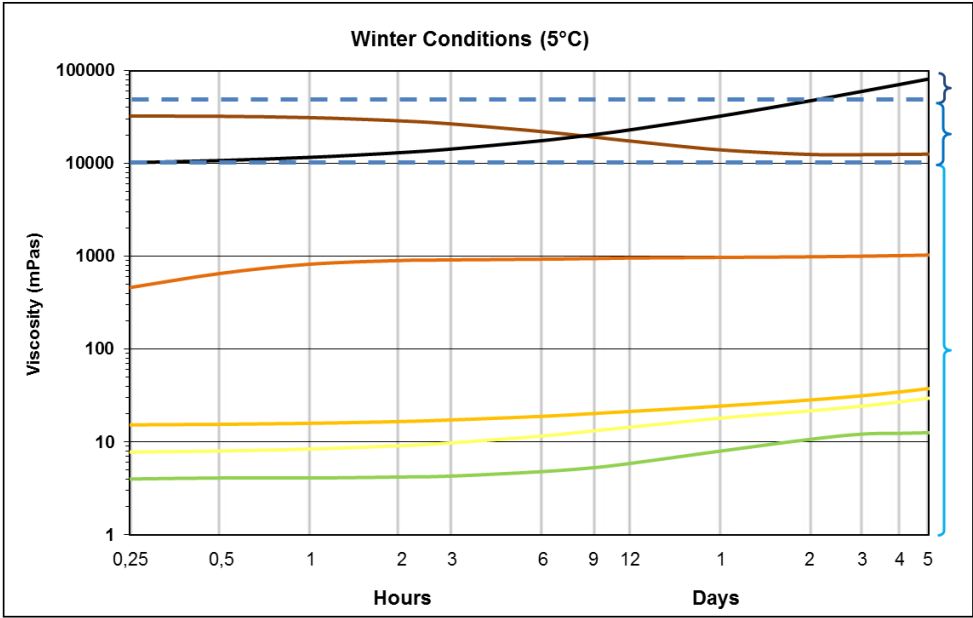
The predicted emulsion viscosities of the tested marine fuel oils are shown in Figure 6-5, and clearly show a large variation between the tested oils at both predicted temperatures. The DMA-quality oils MGO, GO and Rotterdam diesel are all predicted to have emulsions viscosities below 50 mPa·s for the first 5 days of weathering at 5 °C. At such low viscosity, trapping oil within a boom will be difficult due to high risk of boom leakage. The WRG is predicted to have viscosities that reach 1000 mPa·s within the first 2 days of weathering at 5 °C. With this viscosity, some boom leakage will be expected, but use of low speed and careful surveillance of potential boom leakage may enhance the trapping of WRG emulsion within a boom. At 15 °C, the emulsion viscosity of WRG is predicted to remain below 500 mPa·s for the first few days of weathering.

The predicted development of HDME 50 show that the emulsion viscosity is expected to reduce slightly during the first 3 days of weathering at 5 °C. This behaviour was also observed in the meso-scale flume testing at 2 °C. The predicted reduction in emulsion viscosity may allow enhanced skimmer efficiency after 9 hours of weathering. At 15 °C, the emulsion viscosity is predicted to increase steadily over the first five days of weathering.

Shell ULSFO is predicted to show a continuous increase in emulsion viscosity at both 5 and 15 °C, and may surpass the viscosity limit set by NCA for high viscous oils (> 50 000 mPa·s, see Chapter 7.1) after 3 days of weathering at 5 °C. However, results from the meso-scale testing at 2 °C showed a decrease in emulsion viscosity (from 18347 mPa·s to 9800 mPa·s), similarly to HDME 50, indicating that the Shell ULSFO emulsions may remain within the medium viscous oil limit for a longer time than predicted. As Figure 6-3 shows, solidification caused by Shell ULSFOs high pour point may also pose a challenge for the mechanical recovery of Shell ULSFO.

For mechanical recovery of the DMA-quality oils at the WRG, skimmers suitable for low viscous emulsions are recommended. Skimmers suitable for medium viscous are recommended for removal of HDME 50 and Shell ULSFO for the first 3 days.

Property: **EMULSION VISCOSITY**
 Wind speed (m/s): 10
SINTEF
 4.0 β © 2010
 Pred. Date: May 2017

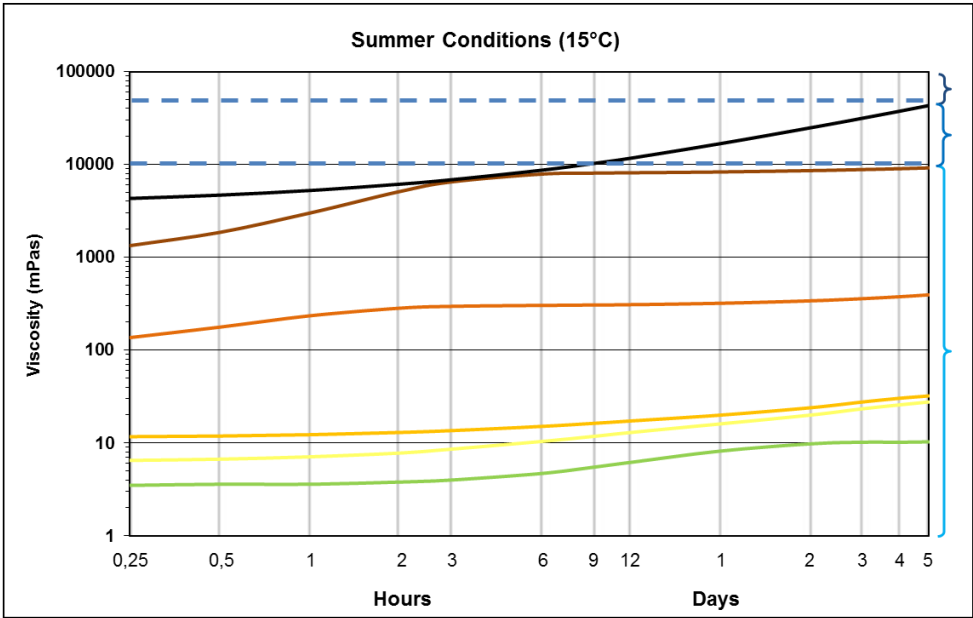


High viscous emulsion
 Viscosity > 50 000 mPa·s

Medium viscous emulsion
 Viscosity between
 10 000 - 50 000 mPa·s

Low viscous emulsion
 Viscosity < 10 000 mPa·s

Property: **EMULSION VISCOSITY**
 Wind speed (m/s): 10
SINTEF
 4.0 β © 2010
 Pred. Date: May 2017



High viscous emulsion
 Viscosity > 50 000 mPa·s

Medium viscous emulsion
 Viscosity between
 10 000 - 50 000 mPa·s

Low viscous emulsion
 Viscosity < 10 000 mPa·s

Figure 6-5: Predicted emulsion viscosity for the tested marine fuel oils at 5 °C and 15 °C, and 10 m/s wind

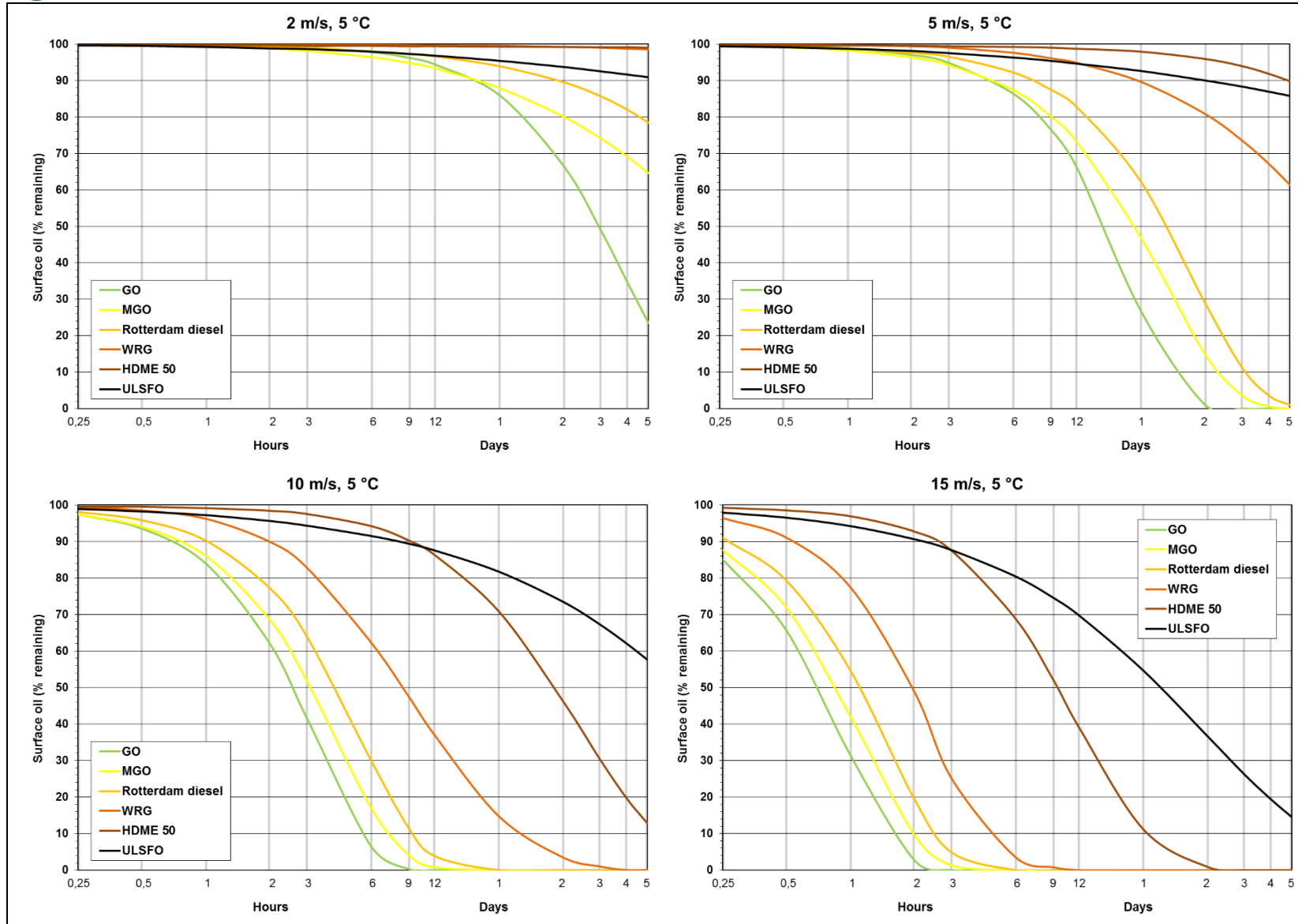


Figure 6-6: Predicted volume of remaining surface oil (%) for the six tested fuel oils at 2, 5, 10 and 15 m/s wind conditions

Due to evaporative loss and natural dispersion the oil on the sea surface will gradually be reduced. Figure 6-6 compares the predicted mass balance of the tested fuel oils by comparing remaining surface oil (water-free) in percentage relative to released volume.

The high evaporative loss and natural dispersibility of the DMA-qualities compared to the heavier WRG and hybrid fuel oils is shown as more rapid removal from the sea surface at all wind conditions. HDME 50 is predicted to have a slower removal than Shell ULSFO for the first days in calm weather conditions, likely due to the lower evaporation. However, the emulsification of Shell ULSFO contribute to a prolonged lifetime at sea in breaking wave conditions compared to the HDME 50.

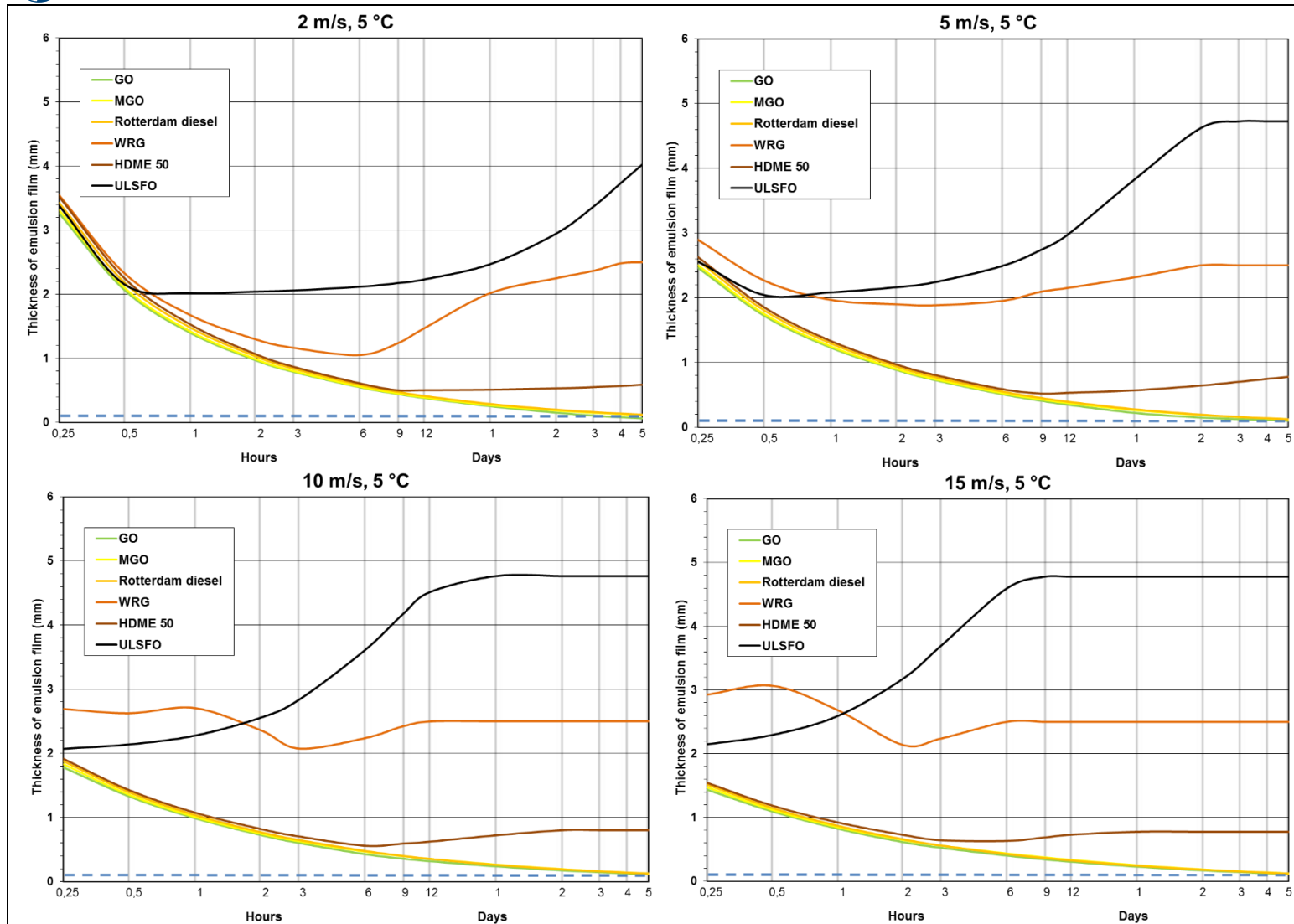


Figure 6-7: Predicted emulsion film thickness for the six tested oils at 2, 5, 10 and 15 m/s wind conditions. The blue line indicate the film thickness required for emulsification to occur

The predicted emulsion film thickness of the tested oils at different weather conditions are presented in Figure 6-7. The predictions show similar behaviour for the three DMA-qualities GO, MGO and Rotterdam diesel. The HDME 50 also express similar behaviour to the DMA-qualities initially, but the emulsion thickness is predicted to increase with time, corresponding to the oils slow uptake of water.

In calm conditions, WRG is also predicted to behave similarly to the HDME 50 hybrid fuel oil and DMA-diesels in the initial phase after release. However, the water uptake of WRG result in a different predicted behaviour with time and in breaking wave conditions.

Shell ULSFO is predicted to have the thickest emulsion film, influenced by the uptake of water and the low spreadability of the oil.

In breaking wave conditions (10-15 m/s wind) the expected lifetime on the sea surface is predicted to be <1 day for the DMA-qualities.

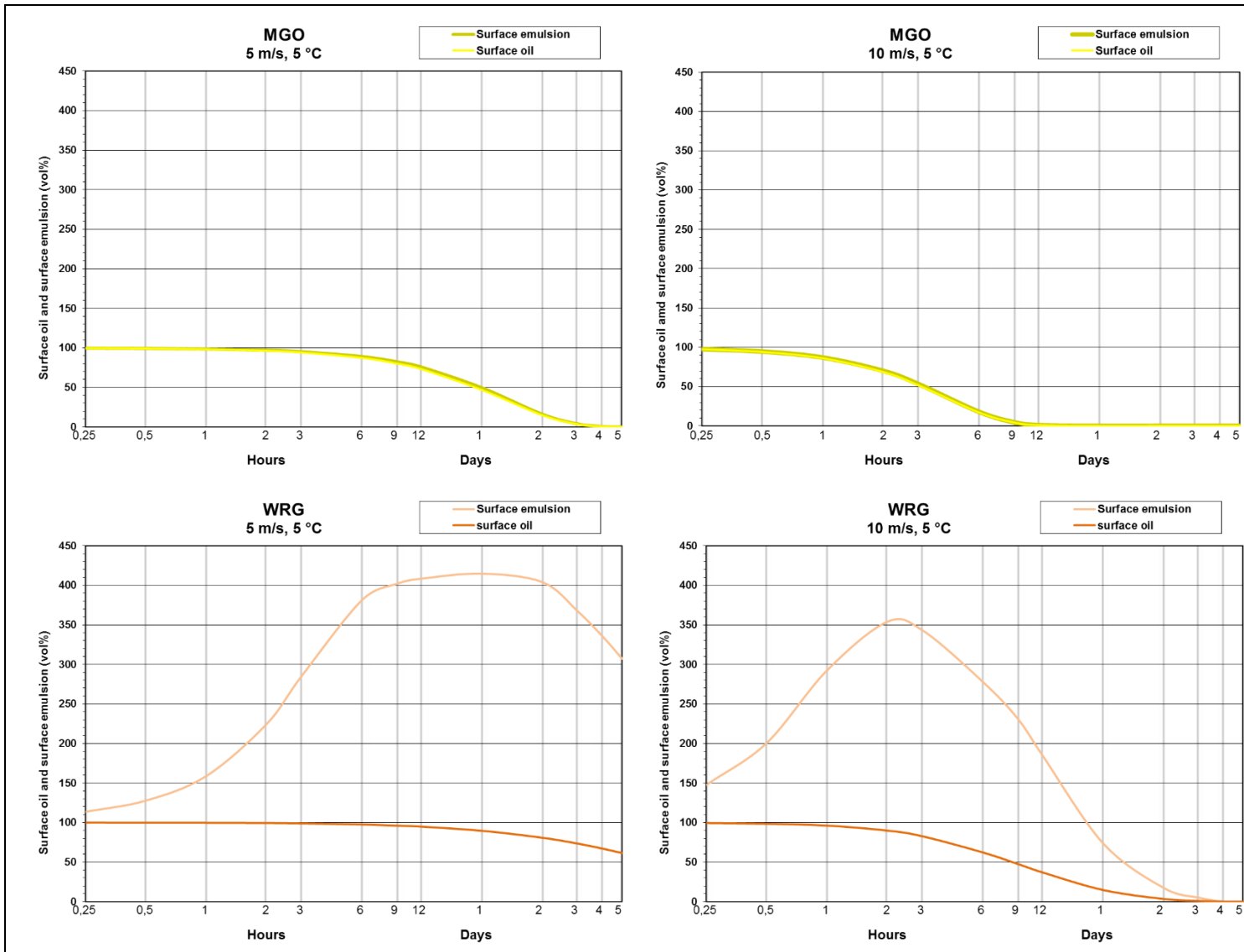


Figure 6-8: Comparison of predicted surface oil and surface emulsion for MGO (upper figures) and WRG (lower figures) at 5 m/s (left) and 10 m/s (right) wind at 5 °C

The predicted volume of surface emulsion compared to surface oil for WRG and MGO at two different wind conditions, 5 m/s and 10 m/s, at 5 °C are shown in Figure 6-8. MGO is not predicted to have a significant water uptake, and the line for the surface emulsion is hidden behind the surface oil line. WRG is predicted to have a very high water uptake, obtaining 80 vol. % water, resulting in a quadrupled volume of emulsion compared to released volume after 1 day at sea. It must be noted that the emulsions of WRG are likely to be very unstable, and likely will break easily when handled.

The observed differences in emulsion volume between the wind conditions reflect the variation in water uptake and natural dispersion between MGO and WRG. At wind speeds of 5 m/s, the natural dispersion is low and both oils have a longer lifetime at sea compared to the predictions for 10 m/s conditions. The higher wind conditions contribute to a significantly quicker removal of WRG from the sea surface, and though the predicted emulsion volume is more than tripled after 2 hours at sea, the removal of oil is predicted to occur rapidly over the next hours.

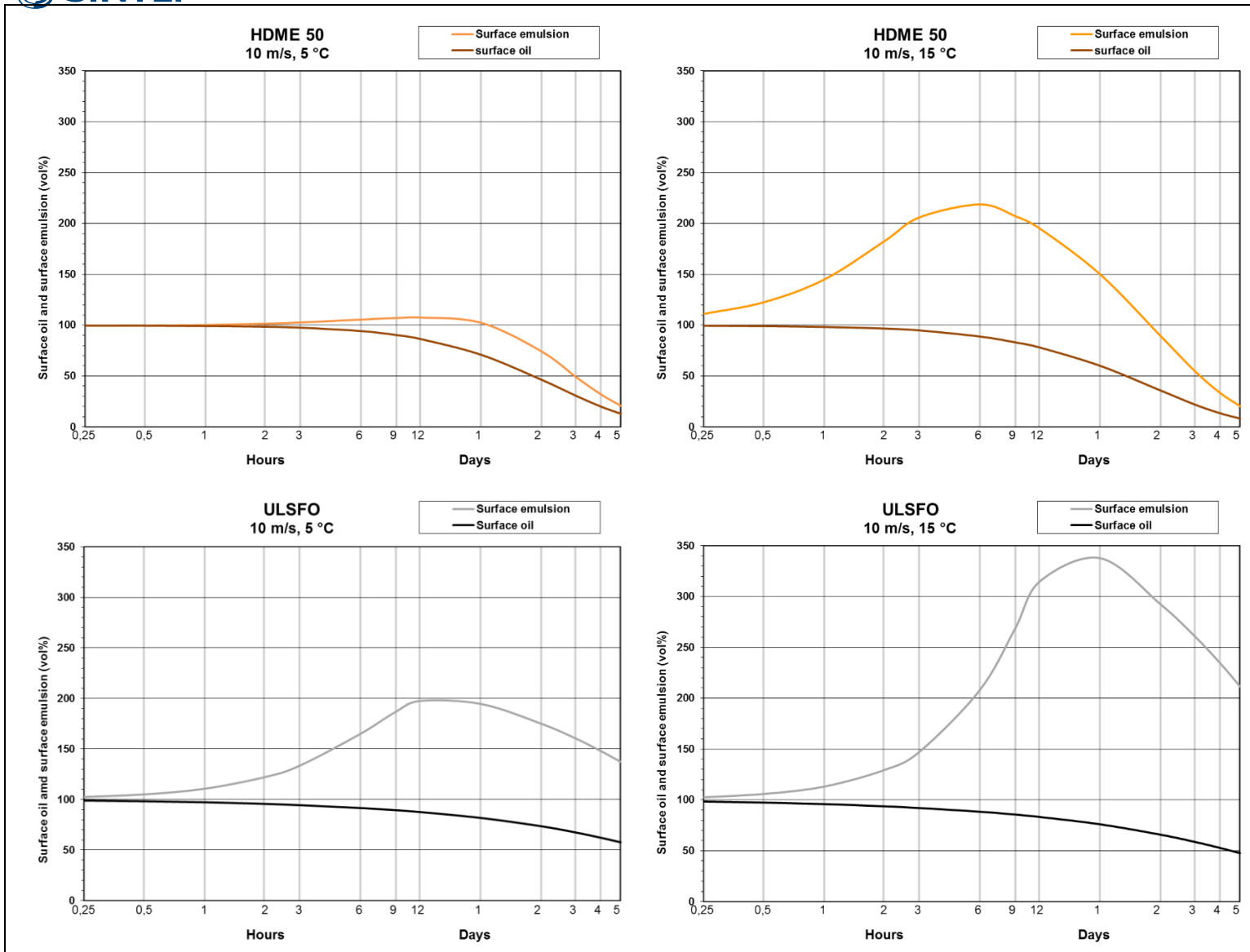


Figure 6-9: Comparison of predicted surface oil and surface emulsion for HDME 50 (upper figures) and Shell ULSFO (lower figures) at 10 m/s wind at 5 °C (left) and 15 °C (right)

The predicted volume of surface emulsion compared to surface oil for HDME 50 and Shell ULSFO at two temperatures are shown in Figure 6-9. The observed differences in emulsion volume between warmer and colder temperatures reflect the increased water uptake at higher temperatures observed for both hybrid fuel oils.

Shell ULSFO is predicted to have a high water uptake at warmer temperatures, and to produce emulsions volumes nearly 3.5 times the released amount during the first day of weathering at 15 °C. At 5 °C, the emulsion volume is predicted to be double the release volume after the first day of weathering.

For HDME 50, delayed emulsification at 5 °C will result in only a minor volume increase. However, at 15 °C, HDME 50 is predicted to emulsify more rapidly and to form emulsion volumes more than twice the volume of the released oil. For both HDME 50 and Shell ULSFO, even higher emulsion volumes is expected at warmer temperatures (e.i. 20 °C).

7 Properties of different fuel oils related to oil spill response

The weathering properties of the tested oils will influence the evaluation of best response action in a spill situation. In this chapter, the results from the tests will be discussed in relation to different response actions and toxicity of the oils.

7.1 Mechanical recovery

Experiences from Norwegian field trials have demonstrated that the effectiveness of various mechanical clean-up operations may be reduced due to leakage of the confined oil or w/o emulsion from the oil spill boom. This leakage was found to be particularly pronounced if the viscosity of the oil or the w/o emulsion is lower than 1000 mPa·s (Nordvik et al., 1992). Based on this, the lower viscosity limit for an optimal mechanical clean-up operation has been set at 1000 mPa·s, as shown in Figure 6-5. However, it should be emphasized that boom leakage is also influenced by other factors such as the operational speed of recovery vessel and weather conditions, and that NCA do not operate with this same limit for lower viscosity. Counter measurements can be made in order to reduce boom leakage, for instance by reducing the operational speed. A study performed at SINTEF on the recovery rate of weir skimmer primarily used by NOFO showed a reduced recovery rate (m³/t) at viscosities above 15 000-20 000 mPa·s (Leirvik *et al.*, 2001). The Norwegian Coastal Administration (NCA), however, have several different types of skimmers in stock that each are suitable for a range of emulsion viscosities, and the NCA operate with three different viscosity limits:

- Low viscous oils/emulsions: < 10 000 cP
- Medium viscous oils/emulsions: 10 000 cP - 50 000 cP
- High viscous oils/emulsions: > 50 000 cP (1 cP = 1 mPa·s)

DMA-quality fuel oils such as the GO, MGO and Rotterdam diesel have, in general, a high spreadability and will rapidly form thin film thicknesses if the oil is allowed to drift at sea (see Figure 6-7). Free drifting, thin oil films are difficult and time consuming to confine in booms to a film thickness suitable for mechanical recovery. However, in scenarios where the spreading of the oil has been prevented e.g. by shoreline, ice cover or booms, these diesel oils may form film thicknesses with potential for mechanical recovery (> 100 µm).

The low viscosity of the diesel oils may result in boom leakage, and this may be a challenge particularly in conditions with wind and waves. In calm weather conditions, mechanical confinement of the oil should be attempted as soon as possible. However, mechanical recovery of DMA-qualities may not be effective unless the spilled diesel oil has sufficiently thick film thicknesses for mechanical recovery (>100-200 µm).

Heavier marine distillate fuels, such as the WRG, may emulsify and form emulsions with viscosities that are better suited for confinement in booms, but in the case of WRG, some boom leakage would be expected due to low emulsion viscosity, particularly shortly after the release and in early stages of emulsification. The WRG formed thicker films than the GO diesel, as seen in the spreadability study, and a thicker film is more advantageous for collecting within booms. The rapid water uptake of WRG also increase the film thickness rapidly, and the predictions presented in Figure 6-7 indicate that WRG may form emulsion film thicknesses well above 1 mm.

The two tested hybrid fuel oils, HDME 50 and Shell ULSFO, both form emulsions with viscosities well above 1000 mPa·s in cold temperatures, and boom leakage is not expected to pose a challenge for these oils in arctic areas. In cold environment, the emulsion viscosities for these oils have been predicted to approach or even surpass the viscosity limit for high viscous emulsions used by NCA (> 50 000 mPa·s), risking reduced efficiency of mechanical recovery. Thus, the choice of skimmer should be made to meet the requirements for efficient recovery of these oils in cold climates.

HDME 50 showed significantly lower emulsion viscosities at warmer temperatures (13 °C and 20 °C) compared to at 2 °C. It is expected that the emulsion viscosity will rapidly surpass 1000 mPa·s at these

higher temperatures, thus limiting the risk of boom leakage. In higher temperatures, the emulsification will occur faster, and the total water content will be higher than at 2 °C. This is again expected to cause an increase in the emulsion viscosity. However, it is not believed that emulsion viscosities of HDME 50 will rapidly surpass 20 000 mPa·s during the first days of weathering at 20 °C, and a wide range of different skimmers will likely be effective for the mechanical recovery of these emulsions. Due to the increased water uptake, larger volumes are expected at warmer temperatures than at lower, and a larger storage capacity on board recovery vessels may be required for efficient recovery. Figure 6-9 show the predicted emulsion volumes compared to the remaining surface oil (water-free) at two temperatures, and gives an indication of the variation in emulsion volume depending on temperature. For HDME 50, this variation can be significant.

Similar to HDME 50, Shell ULSFO also showed lower emulsion viscosities at higher temperatures, but not so low as to expect that boom leakage will be a challenge. Shell ULSFO, similar to HDME 50, also expressed a significantly higher water uptake at warmer temperatures, and predicted volumes at two temperatures are shown in Figure 6-9. The weathered residue of Shell ULSFO produced significantly higher emulsion viscosities than the un-weathered Shell ULSFO oil in the bench-scale and dispersibility study. Thus, the emulsion viscosities of Shell ULSFO is expected to increase significantly with weathering, and is predicted to surpass 50 000 mPa·s within the first three days of weathering at 5 °C (Figure 6-5). For Shell ULSFO, solidification may be an issue even at relatively high temperatures (10 °C+), due to the high pour point of the oil (24 °C). Solidification influence the viscosity of the oil, as the formation of wax lattice structures stabilise a solid structure, resulting in reduced flow of the oil and a significant increase in viscosity. The reduce of flow of an oil/emulsion can be critical for the effectiveness of a skimmer, and skimmers with good effectiveness on high viscosity oils/emulsions should be used if there is a potential for solidification of Shell ULSFO.

NCA has performed tests in their National Centre for Testing of oil spill response equipment with three different skimmer types on the MGO, WRG, HDME 50 and Shell ULSFO oils. Their results show that mechanical recovery of Shell ULSFO at low temperatures (2 °C) was challenging, due to the oils high pour point and low flowability towards the skimmers. The skimmers recovery efficiency was found to be good for the other tested oils (Holt et al., 2017).

7.2 Dispersibility

The results from the performed dispersibility studies have been presented for the individual oils in Chapter 5. In this chapter, the behaviour and properties of the oils in relation to dispersibility will be discussed from an operational view.

7.2.1 Effectiveness of different dispersants

Screening studies of six different dispersants were performed using the IFP-methodology for all the tested oils. The choice of fraction and emulsion for these studies were made in order to provide a range of effectiveness for different dispersants. The Pollution Regulation, Chapter 19, (the current Norwegian regulation) state that an emulsion of a 200°C+ fraction (representing 1 day of weathering) containing 50 vol. % water should be used for such screening testing of chemical dispersants. In this project, this requirement was not easily met due to lacking emulsifying properties of the DMA-qualities, lack of weathered residues for WRG and HDME 50, and a strong pour point influence on the properties of the weathered Shell ULSFO. The choices of oil, fraction and emulsion (water content) for each oil used for the screening study are listed in Table 7-1. These choices are deemed to be the best possible approximations for satisfying the current regulations for each oil.

Table 7-1: Lists the fractions and water content of potential emulsions used for screening study of chemical dispersants.

SINTEF ID	Oil	Fraction	Water content (vol. %)
2014-0552	GO	250°C+	0
2014-0551	MGO	250°C+	0
2016-0232	Rotterdam diesel	250°C+	0
2014-0553	WRG	Un-weathered/fresh oil	0
2016-0231	HDME 50	Un-weathered/fresh oil	50
2016-0233	ULSFO	Un-weathered/fresh oil	50

The natural dispersion (effectiveness without use of chemical dispersion) was tested for the DMA-qualities (GO, MGO and Rotterdam diesel) and the WRG, but not for the HDME 50 and Shell ULSFO emulsions. The results from the screening is presented in Figure 7-1, and show a clear advantage for use of chemical dispersant for the DMA-qualities and WRG oil when comparing the effectiveness of no dispersant to the use of dispersant. For all oils, except HDME 50, the Dasic Slickgone NS and Corexit 9500 were found to be the most effective dispersants. For HDME 50, Radiagreen was the most effective dispersant, followed by Dasic Slickgone NS.

Based on the obtained results from the screening testing, Dasic Slickgone NS can be considered the most versatile dispersant for the tested oils, followed by Corexit 9500, which also showed good effectiveness for most of the oils. Corexit 9500 has previously been found to be more effective on heavy bunker fuels compared to Dasic Slickgone NS (Sørheim *et al.*, 2014), and good effectiveness would be expected when used on DMA-qualities and Shell ULSFO. The effectiveness of Corexit 9500 was low for HDME 50, and for this oil use of Dasic Slickgone NS, or Radiagreen, is recommended based on the results from the screening test.

It must be noted that results from the flume testing with WRG at 2 °C indicated that Corexit 9500 may be more effective than Dasic Slickgone NS on emulsions of this oil at low temperatures. Corexit 9500 was applied to the emulsion as a last attempt of dispersion, when three applications of Dasic NS had yielded little effect (< 20 % effectiveness). The application of Corexit 9500 resulted in the dispersion of 60 % of the available oil/emulsion in the flume test. It is uncertain whether this increased dispersion was due to the general increase in DOR of chemical dispersant, or caused by Corexit 9500 having advantageous properties compared to Dasic Slickgone NS at this temperature. Additional testing would be required for documenting this.

The poor dispersibility of WRG at 2 °C (using Dasic Slickgone NS) indicate that mechanical recovery of WRG in cold temperatures may be more beneficial than attempted dispersion.

7.2.2 Dispersibility limits

Extended bench scale dispersibility studies were performed for WRG, HDME 50 and Shell ULSFO in order to obtain dispersibility limits, presented in Table 7-2. The dispersibility limits are found by analysing the dispersibility of emulsions with varying emulsion viscosity, and can be used to describe the expected dispersibility of any emulsion based on its viscosity. The use of chemical dispersant requires a certain film thickness, in order to prevent herding, droplet of dispersant breaking through the oil film instead of soaking into the oil. As a rule of thumb, 50-100 µm oil film thickness is the minimum thickness for application of chemical dispersant (Singsaas *et al.*, 2017). Low viscous oils, such as the tested DMA-qualities, can easily form thinner oil films if drifting freely on the sea surface. The spreadability of these oils can be limiting factor of higher importance than viscosity, for the successful dispersion of these oils.

For WRG and HDME 50, insufficient dispersibility data were available for the determination of an upper viscosity limit above which the oil could be assumed to not be dispersible. However, an estimation of an upper limit for HDME 50 was made, as seen in Table 7-2. HDME 50 showed some dispersibility at emulsion viscosities above 10 000 mPa·s in the meso-scale flume tests, and the estimated upper limit may be low for this oil. However, retaining the estimated value provides a conservative limit.

Figure 7-2 shows the predicted time window for use of chemical dispersant for all the tested oils. Dispersible emulsions are marked in green, emulsions with reduced dispersibility are marked with orange, and emulsions that are predicted not dispersible are marked with red. The viscosities of the DMA-qualities have been predicted to remained low (< 50 mPa·s) under the set conditions, wind speed of 5 m/s and temperature of 5 and 15 °C, and are all deemed dispersible during the first 5 days of weathering.

The application of chemical dispersant may not give an instant increase in dispersion of the oil/emulsion, but may change the properties of the emulsion in a way that promote dispersion after repeated application of chemical dispersant.

Table 7-2: Dispersibility limits for WRG, HDME 50 and Shell ULSFO obtained through dispersibility testing using MNS and IFP-methodology

Oil	Dispersibility limits based on viscosity (mPa·s = cP)	
	Chemically dispersible below	Not chemically dispersible above
WRG	300	N.E.
HDME 50	3000	10 000*
ULSFO	4000	9 000

*Estimated limits based on available results

N.E. – Not estimated (poor dispersibility was not observed)

A reduction in water content and reduced emulsion viscosities are potential effects of applying chemical dispersants, and have previously been observed for emulsions of bunker fuel oils (Sørheim *et al.*, 2014). Similar behaviour was also observed for HDME 50 and Shell ULSFO in the meso-scale flume experiments (see memo-report for details). The reduction in emulsion viscosity may result in emulsion viscosities within the dispersible range, even though emulsion viscosities prior to application would be deemed not dispersible. This can explain the dispersibility of HDME 50 in the flume tests, even at high emulsion viscosities, and underline the potential advantages of repeated application of chemical dispersant.

WRG showed an unusual behaviour in the meso-scale flume test at 2 °C since the emulsion viscosity increased after the application of chemical dispersant. This behaviour had not previously been observed, and the reasons for this behaviour is uncertain.

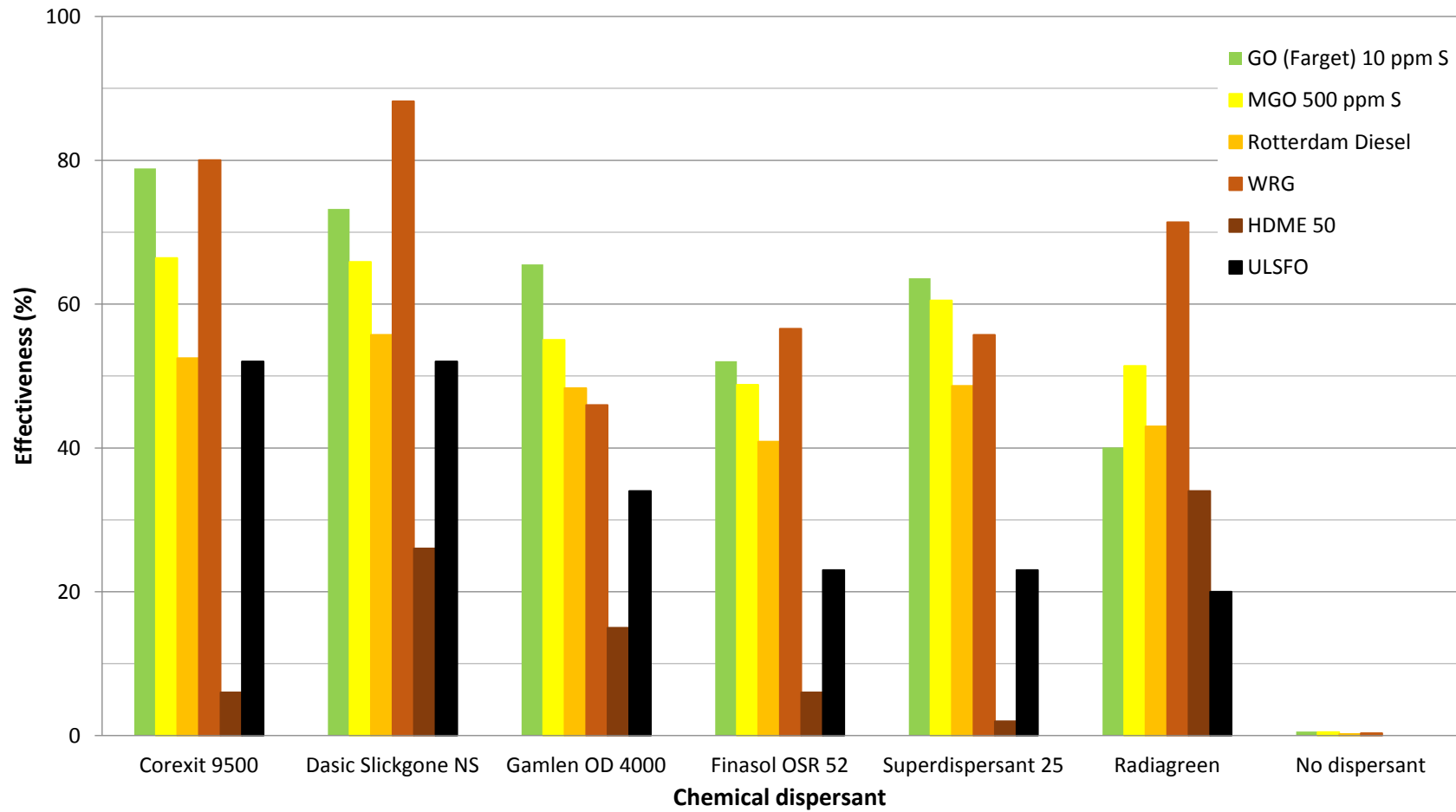


Figure 7-1: Shows the effectiveness of the applied chemical dispersants in the screening testing performed on all oils. The test was performed at 13 °C using the IFP methodology.



Figure 7-2: Predicted dispersibility over time at 5 and 15 °C for 5 m/s wind, for all tested oil. Green = dispersible, orange = reduced dispersibility and red = not dispersible.

7.2.3 Variations in dispersibility with temperature

Figure 7-2 shows the dispersibility of the tested oils for the first 5 days of weathering at 5 m/s at winter and summer temperatures (5 and 15 °C). Emulsions that have viscosities that are deemed dispersible are marked with green, while emulsions with viscosities that have surpassed the individual dispersibility limit for reduced dispersibility are shown in orange. Emulsions that have predicted viscosities that exceed the limit when the emulsion is deemed not dispersible are marked in red. Shell ULSFO and HDME 50 are seen to have predicted time windows for use of chemical dispersant at 15 °C, but not at 5 °C. For details regarding predicted emulsion viscosities, please see the memo-report (Hellstrøm *et al.* (2017).

Significant variations in dispersibility was observed between test temperatures for WRG, HDME 50 and Shell ULSFO. The observed behaviour varied between the oils, and for HDME 50 and Shell ULSFO high pour points influenced the emulsification and the emulsion viscosities at the lower temperature.

WRG had a low pour point of -24 °C, and this property was not believed to influence on the emulsification or emulsion viscosities for this oil. WRG formed low (<600 mPa·s) emulsion viscosities at 13 °C, and somewhat higher (< 1500 mPa·s) emulsion viscosities at 2 °C. Dispersion of emulsions with viscosities in this range is often expected to result in high effectiveness. However, WRG did not disperse well, especially at 2 °C, and a dispersibility limit of 300 mPa·s was set based on results from the bench scale testing, seen in Table 7-2. This may have been caused by low dosage to oil ratio (DOR) or unsuccessful application strategy.

At 13 °C, HDME 50 emulsified rapidly and formed a homogenous emulsion that showed increased effectiveness of repeated application of dispersant. At 2 °C, HDME 50 solidified and formed lumps that very slowly emulsified. The emulsion dispersed relatively easily, but the lumps were unaffected by the application of chemical dispersant. Mechanical recovery of such lumps would be necessary for the successful removal of the oil from the sea surface. Their total volume is not expected to be higher than the volume of spilled oil.

The behaviour of Shell ULSFO was similar to HDME 50, with the formation of homogenous emulsion at 13 °C but solidification and formation of lasting oil lumps at 2 °C. Repeated application of chemical dispersant had slightly better effect on the emulsion at 2 °C than at 13 °C but was not effective at either temperature. Similar to HDME 50, the solidified oil lumps remained at 2 °C throughout the testing and was not affected by chemical dispersant.

In general, chemical dispersion is not deemed an optimal response for Shell ULSFO nor HDME 50, particularly in cold conditions. The WRG may also have a limited time window for use of oil spill dispersant at low temperatures.

7.3 Ignitability

In general, *in-situ* burning (ISB) is an alternative response operation which require little infrastructure or large amounts of special recovery equipment, and if the oil properties permit ISB. For this reason, ISB is often considered as a primary response operation in remote areas with limited accessibility. In the Arctic, the use of mechanical recovery or chemical dispersants may be challenged by the presence of ice and a long response time for necessary equipment. Since cold temperatures can prevent extensive evaporation, and the presence of ice may prevent spreading and may limit waves from reaching a spill, cold conditions and a cover of ice may prolong the window of opportunity for ISB. Without the energy from waves an oil is less likely to emulsify, and this prolong the time window for use of ISB further (Brandvik *et al.*, 2010).

ISB is a relatively low-cost method, but is likely to produce potentially smoke and soot particles. For assessment of the possibility for use of ISB as a response operation, information regarding the ignitability of an oil is of interest. Knowledge regarding the burning efficiency of the oil is also important since this can be a measurement for the effectiveness of ISB as a response operation. In this project, the ignitability of the selected fuel qualities was tested in both bench-scale testing at SINTEF and in an up-scaled testing at Falck Nutec, by SINTEF personnel. Results from this testing is described in detail in the memo-report, and have

been summarised for the individual oils in Chapter 5. A short description of the methodologies used are also given in Appendix A.4.

Un-weathered MGO and Shell ULSFO ignited in the bench-scale testing within the standard ignition time (3x10 sec.), while GO and Rotterdam diesel ignited with prolonged ignition time after standard ignition had been attempted (20 and 25 sec., respectively). WRG did not ignite in the bench-scale test, and HDME 50 was not tested since its flash point was even higher than that of WRG. In the up-scaled testing, all oils ignited showing that even high cut distillates such as WRG and HDME 50 ignite if a heat source of the necessary temperature is available for sufficient time. The ignition times that were required in the up-scaled testing were in accordance with the results seen in the bench-scale testing. Figure 7-3 show a comparison of the required ignition times from the two test scenarios.

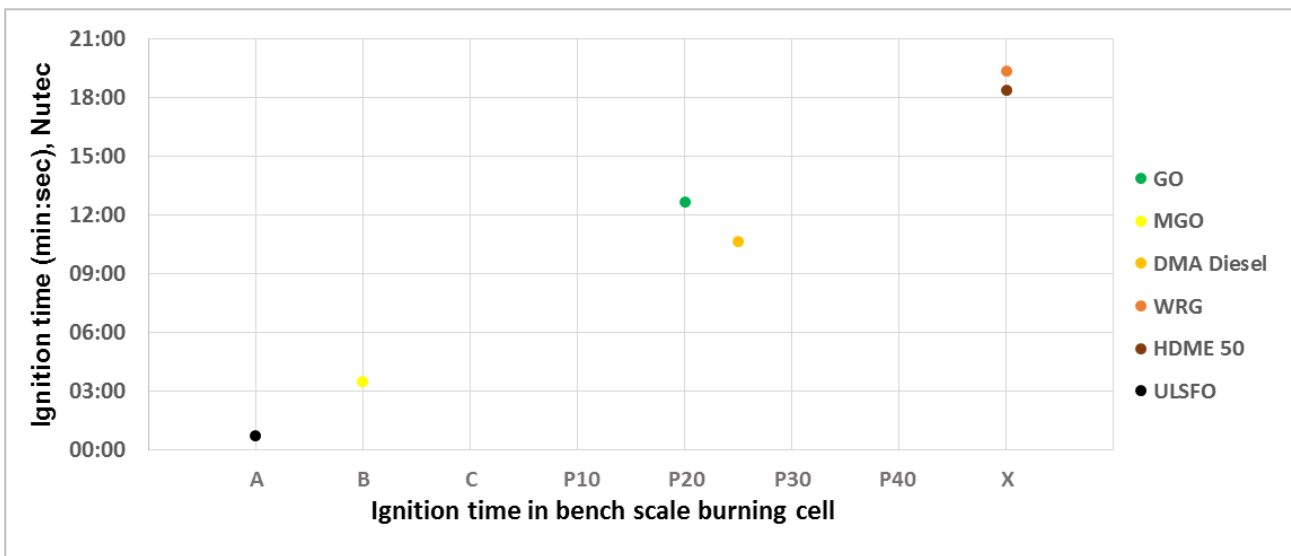


Figure 7-3: Comparison of ignition time in up-scaled testing at Nutec and ignition time required in the bench scale study. For the bench scale study, A, B and C are the standard three attempted ignitions of 10 seconds each, while P10-P40 stand for the Prolonged ignition of 10-40 seconds. X show the fuel oils that did not ignite in the bench scale study

The effectiveness of ISB can be measured by the Burning Efficiency (BE %) of an oil. The BE % for the tested fuel oils were measured gravimetrically in both the bench scale and the up-scaled studies, and the results are presented in Figure 7-4. Unfortunately, results from the bench-scale study were not available for WRG and HDME 50 due to failed and unperformed ignition, nor for Rotterdam diesel, which produced black smoke when ignited, which indicate a high content of PAH, causing the experiment to be terminated in order to prevent exposure to potential harmful gasses. The BE % is highly dependent on the scaling, increasing with larger scale, and in a full-scale field test BE % can be expected to be higher than those achieved in the performed tests. The results for BE % from the performed tests must therefore be viewed as relative values.

The burning efficiencies from the bench scale study show similar patterns as seen in the up-scaled testing. As expected due to their higher content of heavy components, the heavier fuel oils, WRG, HDME 50 and Shell ULSFO, had lower burning efficiencies than the DMA-qualities. The burning efficiencies of the DMA-qualities were high, well above 60 % in both the bench scale test and the up-scaled test.

Of the heavier fuel qualities, Shell ULSFO had the highest burning efficiency, likely due to a higher content of lighter components. In contrary to WRG and HDME 50, which are heavy distillate oils, Shell ULSFO is a residual oil that contain both lighter and heavier fractions. The presence of the lighter fraction also explain the rapid ignition of Shell ULSFO, which was seen in both tests.

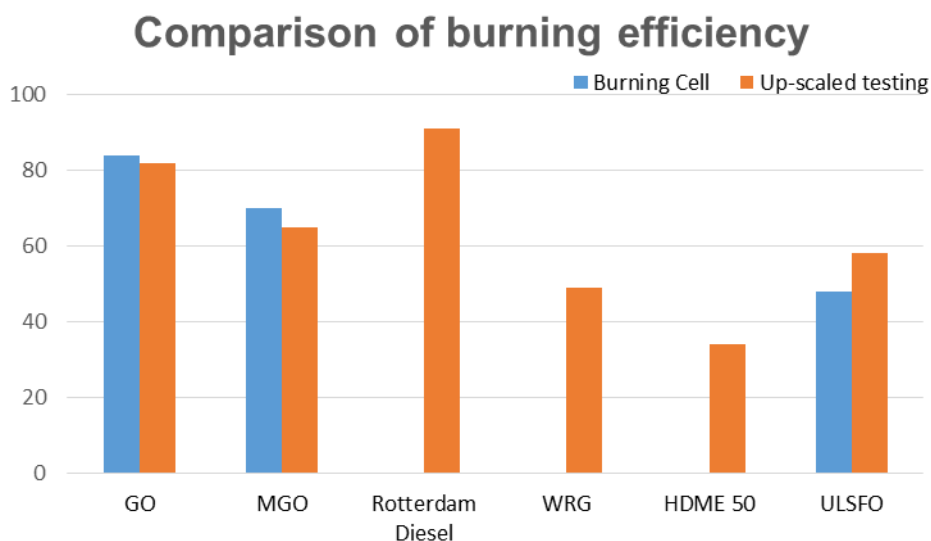


Figure 7-4: Burning Efficiency (%) for the tested oils in the bench-scale studies (blue) and the up-scaled testing (orange). Burning efficiency from the bench scale study was not available for Rotterdam diesel, WRG or HDME 50.

Predicting ignitability with OWM

SINTEF's Oil Weathering Model (OWM) contains a module for predicting the ignitability of an oil (Brandvik *et al.*, 2010). The module was developed based on tests performed with crude oils and crude oils emulsions, and consider the following properties of the oil/emulsion:

- Water content
- Viscosity
- Wax and asphaltene content
- Flash point

These properties vary greatly between different fuel oil categories, as well as in comparison to crude oils. For instance, DMA-qualities have low or insignificant emulsifying properties, a property that the ignitability module emphasises. For these reasons, the ignitability module has some limitations for predicting ignitability of refined products. Despite these limitations, the ignitability of the tested oils were attempted predicted for a controlled scenario. The chosen scenario for the predictions was designed to represent a situation where spilled oil had been trapped or contained immediately after release and had formed a film thickness of 5 mm. It was assumed that natural dispersion was prevented, and that the oil film thickness remained thick throughout the predicted time line. Two temperatures were used for the scenario, 0 °C and 13 °C, representing conditions found in the Arctic and in Norwegian waters, and two wind speeds, 2 m/s and 5 m/s. Experience has shown that ignition of oil spills at higher wind speeds would be difficult. The time line for the predictions were set at 5 days, and ice was not present in the chosen scenario.

The results from the predictions are summarised in Table 7-3, and show that the DMA-qualities are predicted to remain ignitable for more than 5 days. Lack of water uptake and absence of natural dispersion will likely retain these oils as pure oils films. The thick oil film (5 mm) also slows the evaporative loss, keeping lighter components more easily ignitable in the oil.

The emulsifying oils, WRG, HDME 50 and Shell ULSFO express shorter windows of opportunity for ignition, due to the uptake of water. In general, the oils are predicted to be ignitable for a longer time in cold temperatures, due to lower evaporative loss. However, for oils that form more stable emulsions in low temperatures, such as WRG, the window of opportunity for ignition may be shorter in low temperatures

compared to higher temperatures, since more energy will be required to break the stable emulsions before ignition.

Table 7-3: Predicted ignitability, "time window", for a controlled scenario, using OWM.

Oil	Predicted ignitability			
	13 °C		0 °C	
	2 m/s	5 m/s	2 m/s	5 m/s
GO	> 5 days	> 5 days	> 5 days	> 5 days
MGO	> 5 days	> 5 days	> 5 days	> 5 days
Rotterdam Diesel	> 5 days	< 5 days	> 5 days	> 5 days
WRG	< 12 hours	< 3 hours	< 2 hours	< 0.5 hours
HDME 50	< 2 hours	< 0.5 hour	> 5 days	< 2 days
ULSFO	< 1 day	< 6 hours	< 2 days	< 9 hours

Conclusions regarding ignitability

Based on the results from the ignitability studies and on the acquired knowledge regarding each oils properties and behaviour, some conclusions can be made regarding application of ISB.

DMA-qualities (GO, MGO and Rotterdam Diesel) are likely to spread and disperse easily, leaving oil films that are too thin for ignition. This limit the applicability of ISB, but use of ISB is expected to be possible in scenarios where the diesel has been confined and has formed thicker films and is not subject to dispersion. The tested oils showed high burning efficiencies, indicating that ISB would be effective for some time if the conditions are right.

WRG was found to require a long ignition time and showed a burning efficiency of approx. 50 %. The fuel oil emulsified rapidly form unstable emulsions that may break apart easily. Extra energy for the breaking of the emulsion may be necessary before the oil ignites. The use of a high temperature ignitor may promote breaking of the emulsion and reduce the ignition time. Since the burning efficiency is intermediate/low, mechanical recovery of the remaining oil will likely be necessary.

HDME 50 also consist of a heavy distillate cut, similar to WRG, and has a very high flash point (186 °C). With such high flash point, an ignitor with very high temperature will be necessary for successful and relatively rapid ignition. HDME 50 was found to emulsify at 13 °C but in cold conditions (2 °C) the oil solidified and emulsification occurred only slowly. The burning efficiency of HDME 50 was the lowest of the tested fuel oils, less than 40 %. With this low BE, mechanical recovery will likely be the preferred response method.

Shell ULSFO has a flash point in the same range as the DMA-qualities, which is reflected in the rapid ignition of this hybrid fuel oil in the performed tests. Since Shell ULSFO has emulsifying properties, the window of opportunity for use of ISB may be shortened, but in scenarios where water uptake is prevented, the potential for use of ISB may be prolonged. This can be the case in cold temperatures, as Shell ULSFO solidify rapidly due to a very high pour point (24 °C). The solidification would delay the emulsification of the oil, thus prolonging the window of opportunity for use of ISB in cold conditions.

7.4 WAF and toxicity

In acute spill situation, components in the spilled oil that have some solubility in water will migrate from the oil phase to the water phase. The solubility potential is primarily relevant when the oil has been dispersed as smaller oil droplets, and diffusion from oil to water is increased. Aquatic organisms can then be exposed to these oil compounds, and the potential of toxic effects occur. Knowledge of the toxicity of an oil is of importance for the evaluation of response operations, and estimations of the negative effects on the environment resulting from an acute spill situation.

The water-soluble fraction (water accommodated fraction, WAF) of the tested six oils and their weathered 250°C+ residues have been tested. The aim has been to identify the chemical composition of the water accommodated fraction (WAF), and determine the toxicity of each oils WAF on two marine species, the marine algae *Skeletonema* sp. and the copepod *Calanus finmarchicus*. These two species represent different trophic levels in the marine ecosystem. Two oil-to-water ratios, or loading concentrations, have been used; 1:40 and 1:10 000. The highest concentration, 1:40, is unlikely to occur in a situation where dilution is allowed, but has been used as a representative for a worst-case scenario. A 1:10 000 concentration is likely to occur in a spill situation, and represent a scenario where the oil has been dispersed, to a concentration of 100 ppm in the water column. All WAFs were produced at 13 °C, with an additional WAF for WRG being produced at 2 °C. Full detail regarding the method and detailed results are given in Faksness and Altin (2017).

7.4.1 Chemical composition of oil vs. WAF

The chemical composition of the different aromatic component groups in the tested oils and their 250°C+ residues are presented in Figure 7-5 (not WAF-composition). Similarities in composition can be seen in between MGO and GO, though GO has a higher decalin content. Rotterdam diesel, in the figure referred to as DMA, has a surprisingly high content of naphthalenes and 2-3 ringed PAHs (polycyclic aromatic hydrocarbon). It is worth noting that GO, MGO and Rotterdam diesel (DMA in the figure) are all of DMA-quality, according to the ISO 8217 standard (Table 4-1), but still express significant variations in their chemical compositions. The composition of WRG is dominated by naphthalenes and 2-3 ringed PAHs, while Shell ULSFO has a higher content of 4-6 ringed PAHs and decalines. HDME 50 had the lowest concentration of aromatics, and the composition consisted mainly of PAHs.

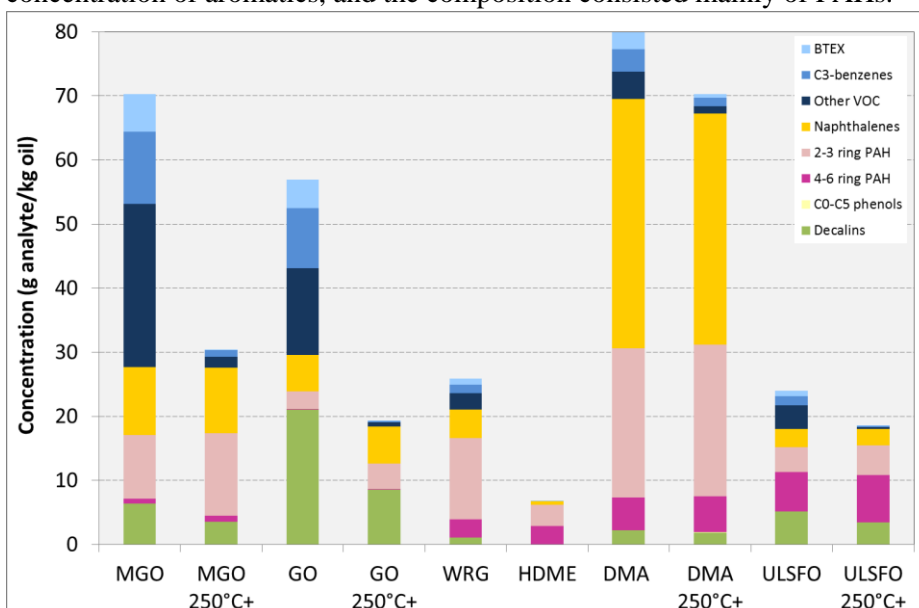


Figure 7-5: Chemical composition in the tested oils of aromatic component groups for six marine fuel oils. DMA = Rotterdam diesel

The chemical profile of the water soluble fraction (WAF) of an oil differs from that of the parent oil due to differences in water solubility for the various oil components. The variation can be seen for the Rotterdam diesel (DMA in the figure) and the hybrid fuel oils by comparing the composition in the oils (Figure 7-5) to the chemical composition in their WAFs (Figure 7-6). The presentation of the chemical compositions of WAF (Figure 7-6) present the Unresolved Complex Material (UCM), which was not seen in the chemical composition of the oil.

The solubility of a compound depend on its size and its polar properties, and small components and components with polar groups or elements (such as sulphur, oxygen and/or nitrogen) usually have higher solubility than larger, non-polar components. The removal of compounds containing sulphur in the process of producing low sulphur fuel oils may thus result in a lower total WAF concentration.

VOCs and SVOCs are small and have relatively high solubility in water, and the WAFs of the fresh fuel oils contain more of these component groups than their respective weathered 250°C+ residues, simply because the majority of these lighter components are removed from the oil during the artificial weathering.

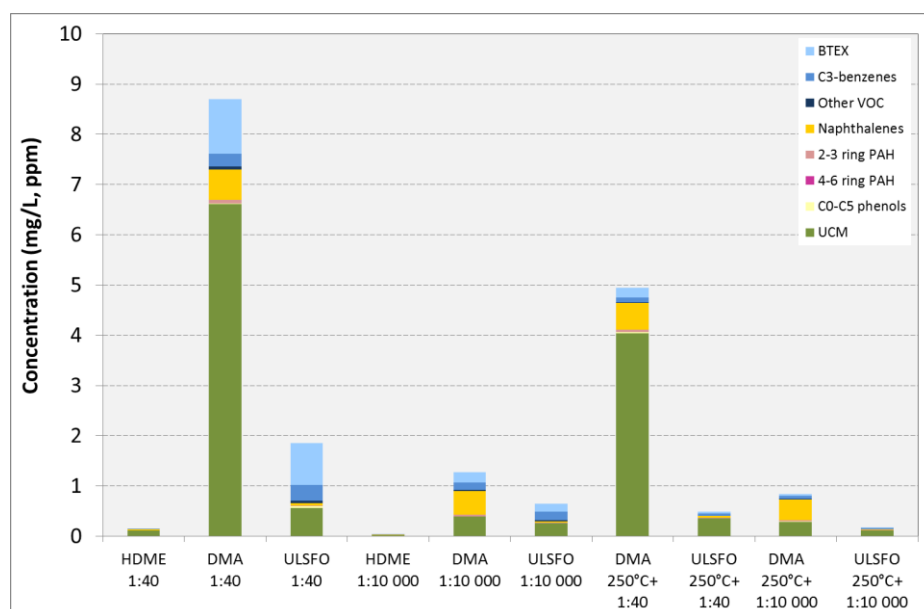


Figure 7-6: Chemical composition of selected components groups in the WAFs from HDME 50, Shell ULSFO and Rotterdam diesel (represented as DMA diesel).

7.4.2 Toxicity

The toxicity of the tested fuel oils have been described by using so-called LC₅₀ and EC₅₀ values. These values describe the concentration that is necessary for 50 % of the test organisms to die (Lethal Concentration = LC) or express significant negative effect (Effect Concentration = EC) after being exposed to the chemical that is tested, in this case WAF, for a limited time (Rand et al., 1995). The negative effect that was measured for algae was inhibition of growth, while lethal immobilisation was used for the copepods in order to determine the toxicity.

LC₅₀ and EC₅₀ are given in percent dilution of 100 % WAF (undiluted). If less than 50 % of the test organisms were affected, the LC₅₀ and EC₅₀ values could not be calculated, as a concentration stronger than the 100 % WAF (undiluted) would be necessary for death or growth inhibition to occur in ≥ 50 % of the test organisms. These cases are marked with * in Figure 7-7, which shows the relative toxicity of the tested oils. High bars indicate low toxicity (high concentration necessary to meet the effect requirements) and low bars indicate high toxicity (low concentration necessary to meet the effect requirements). Of the WAFs that did

not produce LC₅₀ and EC₅₀ values, negative effects were still observed for less than 50 % of the test organisms, with the exception of 1:10 000 loading of HDME 50, where no effect was observed.

The results show that Rotterdam diesel (DMA in the figure), both fresh and weathered, were the most toxic of the tested oil fractions followed by MGO 250°C+ and GO 250°C+. HDME 50 was the least toxic of the 1:40 loadings. In 1:10 000 loadings of MGO, HDME 50, Shell ULSFO and Shell ULSFO 250°C+, less than 50 % of the test organisms were affected, and no LC₅₀ and EC₅₀ values were calculated.

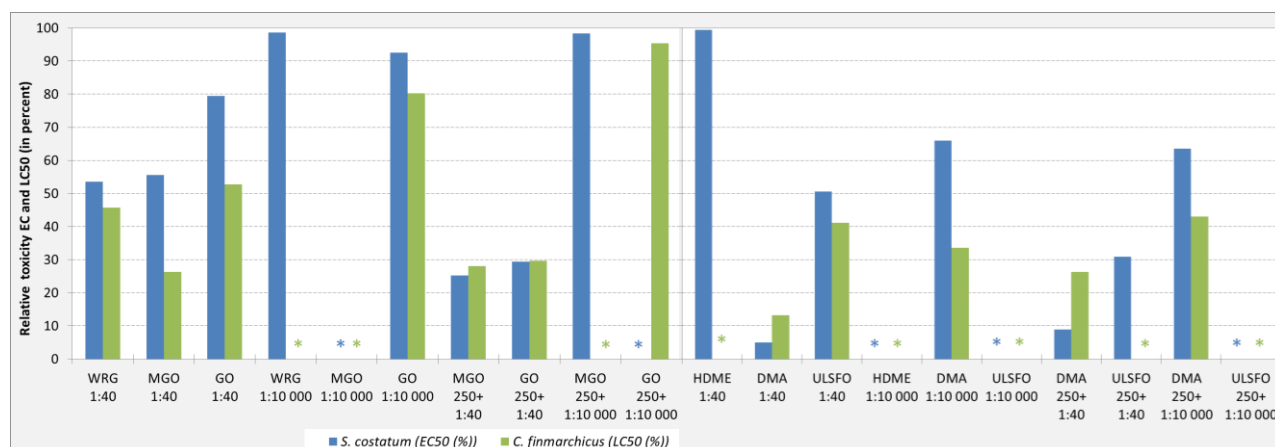


Figure 7-7: Relative toxicity of the WAFs shown as EC₅₀ and LC₅₀ given in percent of dilution of 100% WAF (undiluted). Low bars indicate higher toxicity, but no bars indicate absence of an observed effect on the test organisms (*). DMA = Rotterdam diesel

Toxic Unit (TU) express the acute toxicity of a WAF and can be predicted based on the chemical composition of the WAF and the individual components K_{ow} (octanol-water partition coefficient; ratio between solubility in octanol relative to solubility in water for individual component). A Toxic Unit higher than 1 imply that 50 % of test organisms will die. The calculation of TU does not include the UCM fraction of the WAF, since the composition of individual components in the UCM is unknown. Calculated TUs for the tested oils are presented in Figure 7-8, and show that fresh Rotterdam diesel (DMA in the figure) was the most toxic of the tested oils, with a TU of 3.8. MGO 250°C+ and the weathered fraction of Rotterdam diesel (DMA 250°C+) also express TU above 1, indicating mortality in 50 % or more of the test organisms. All other fuel oils and residues had TU below 1, with HDME 50 having the lowest TU, along with Shell ULSFO 250°C+ and WRG. These results correspond well with the relative toxicity previously discussed.

A comparison of the calculated Toxic Units for the tested marine fuel oils with toxic units of a range of other oils and oil products is presented in Figure 7-9. Computed TU for Rotterdam diesel was similar to Sleipner and comparable to TUs of other North Sea light crude oils. The other tested marine fuels had TU < 1, similar to IFO 180, Bittern, Grane, Boardale and Heidrun Åre.

The results show a strong variation in toxicity between marine fuel oils from different sources. This is further supported by a previously tested Marine diesel, which had TU of approximately 1.5, intermediate to the results found for Rotterdam diesel (TU = 3.8) and the other tested marine fuels (TU < 1). With such variation in toxicity between fuel oils, individual evaluations of the chemical composition of marine fuels would be necessary for a good evaluation of the potential effect of a spill.

WAFs of Shell ULSFO and HDME 50 had the lowest toxic effects of the tested WAFs, and were not found to be acutely toxic. WRG showed similar toxic effects as fresh MGO and GO, while the weathered 250°C+ residues of these latter oils were seen to have higher toxic effects and TUs than their corresponding fresh oils. The results for Rotterdam diesel is of particular concern as both toxic effect and toxic unit were high.

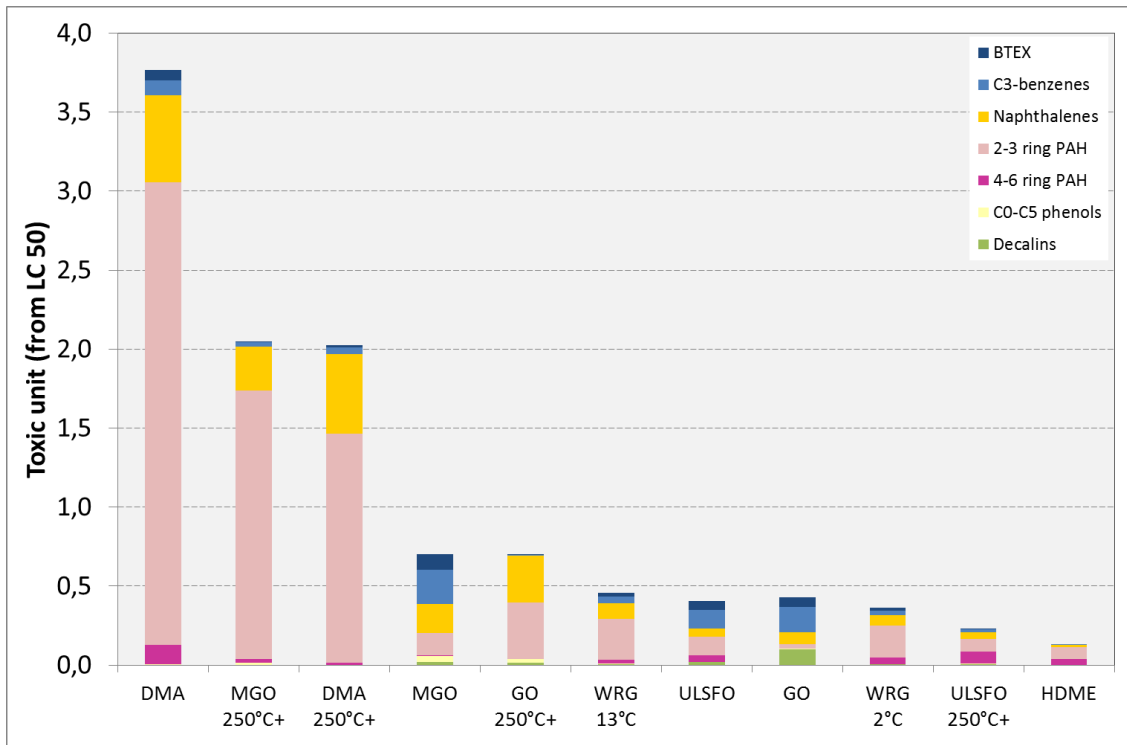


Figure 7-8: Toxic Unit calculated for WAFs of 1:40 loading for the tested oils and weathered residues. TU > 1 indicate that 50 % of the test organisms dies. DMA = Rotterdam diesel

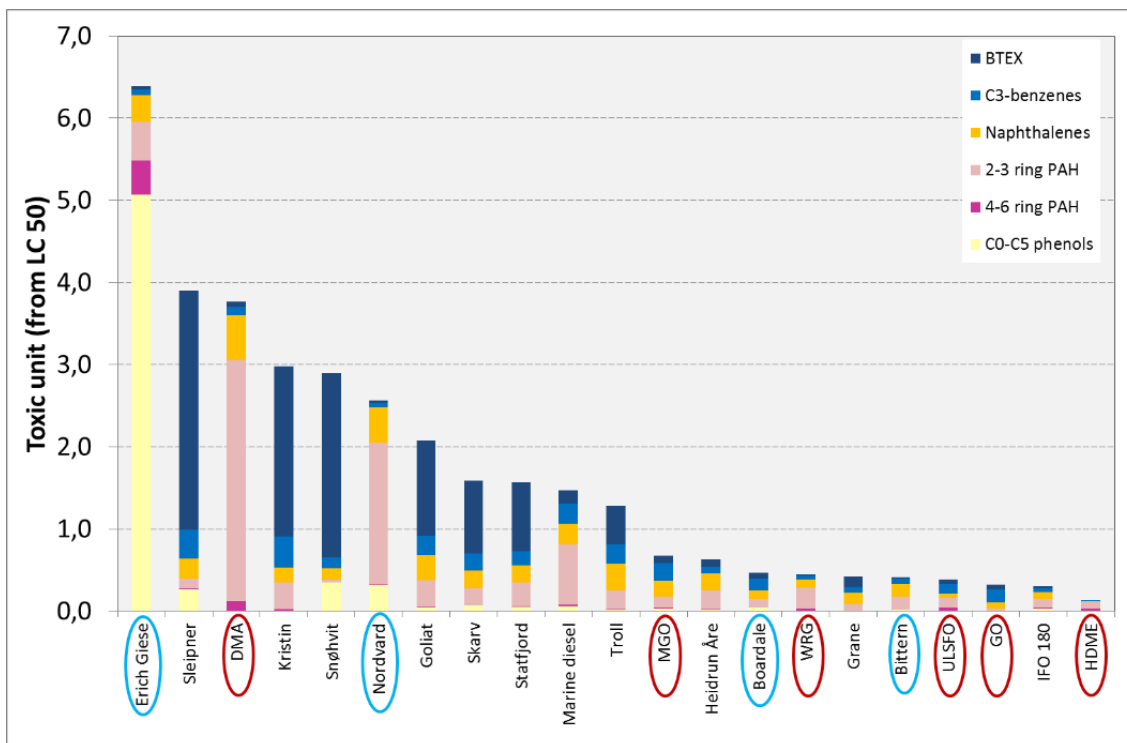


Figure 7-9: Calculated Toxic Units (TU), as an expression of acute toxicity, for WAFs with an oil-to-water loading of 1:40. The marine fuel oils presently tested are marked with red ellipses, and are compared with North Sea crude oils, other refined products and shipwreck oils from WW2 (blue ellipses, from Faksness *et al.*, 2015). TU > 1 indicate mortality in 50 % of the tested organisms. DMA = Rotterdam diesel

8 Concluding remarks and recommendations

This study has included extensive testing on six different marine fuel oils, and the aim has been to reveal relevant weathering properties of these different fuel types with relevance to oil spill response mitigation methods. The main findings are summarised in this section.

8.1 Diesel fuels

Little variation was observed in the physical properties of the DMA-qualities MGO, GO and Rotterdam diesel. None of these oils was seen to have emulsifying properties; they had similar pour points and densities, and are predicted to have similar weathering properties, also with regard to use of chemical dispersants and ignitability. However, the chemical compositions of the oils varied, and this cause different rates of evaporation as well as differences in toxicity. Thus, the impact on the environment, in form of toxicity to organisms, may vary significantly between oils of DMA-quality.

The WRG showed significant differences compared to the DMA-qualities, both in physical and chemical properties. This oil emulsified and formed unstable emulsions with low viscosities that were not easily dispersible at low temperatures. WRG was in general less toxic than the DMA-qualities, and showed little variation in WAF composition at different temperatures. Due to a high flash point, ISB is considered to have limited potential for this oil (i.e. need a long/extended ignition time and has a short "time window" for ISB after release), and mechanical recovery is the recommended primary response operation, especially in cold environments.

Significant variations between the tested WRG batch (2014) and a newly produced batch (2017, SECA-quality) show that this product has change over the last years, and the obtained results for the 2014 batch cannot be considered valid for this new batch. The reported increase of wax content in the 2017 batch may have given the 2017 batch other emulsifying properties compared to the 2014 batch, as well as differences in emulsion stability. However, the observed chemical and physical variations are not likely to have enhanced the potential for use of dispersants, nor influenced the ignitability of the oil significantly.

8.2 Hybrid fuel oils

The results from the present testing showed that both HDME 50 and Shell ULSFO solidified at low temperatures, and that high pour points influenced strongly on the oils behaviour at 2 °C. Different response operations can be relevant at different temperatures.

In cold temperatures (2 °C):

- Emulsification may occur at a slow rate
- Solidification and high emulsion viscosities limited the effectiveness of the use of dispersants.
 - Repeated application of dispersant was more effective, but solidified lumps were not affected
- Choice of skimmer should reflect the expected emulsion viscosities and pour point influence for successful and effective recovery
- ISB is expected to have a limited potential due to high flash point and long expected ignition time

In warmer temperatures (13 °C+):

- Emulsification begin immediately
- Chemical dispersion had low effectiveness on emulsion, but repeated application gave an increased positive effect
- ISB is expected to have a limited potential due to high flash point and long expected ignition time and a limited "time window" for ISB after release

Viscosity increases with reduced temperature with a logarithmic dependency, and for oils with low pour point, this relationship is both predictable and the likely limiting factor for use of chemical dispersants. For oils with high pour points, the formation of wax lattice structures at temperatures (5-15 °C) below the pour point disrupt the temperature/viscosity relationship. If measured at low shear rates, the viscosity of the oil can then be seen to have increased drastically. However, measuring the viscosity at higher shear rates may not reveal any significant increase, since the energy provided by the higher shear during measurement rate destroy the wax lattice. In the same way, presence of energy from waves and currents may prevent an oil from solidifying even at temperatures up to 15 °C below the oils pour point, allowing applied dispersants to soak into the oil, and thus contribute to increased dispersibility.

8.3 Response operations

Due to high spreadability and formation of thin films (<100-200 µm, see Chapter 6.7), mechanical recovery of free drifting DMA-diesels is considered ineffective, unless spreading of the diesel oils has in some way been prevented (confined by a barrier like shore or ice edge). WRG, HDME 50 and Shell ULSFO all emulsify. The WRG formed emulsions with a high water content (80 %) and large storage volume capacity would be necessary for the mechanical recovery of this oil, if all oil was retained in the emulsion. Since WRG was found to form very unstable emulsions, some water may be removed from the emulsion during recovery handling. The emulsion viscosities are expected to be sufficiently high for collection with booms at low temperatures, while some leakage may be expected at higher temperatures due to lower viscosities (<1000 mPa·s).

Mechanical recovery of emulsions of HDME 50 and Shell ULSFO is expected to be possible with less risk of boom leakage. However, high volume of water in emulsion at summer temperatures (15 °C) may require large storage capacity. Figure 6-9 show the predicted differences of water uptake at different temperature conditions for HDME 50 and Shell ULSFO, and reveal that HDME 50 at 15 °C may obtain an emulsion volume that is twice as high as it is predicted to be at 5 °C. Shell ULSFO is predicted to form emulsions with enough water to double the volume of the released water-free oil at 5 °C, while at 15 °C, Shell ULSFO may produce an emulsion volume that is tripled that of the released water-free oil. For Shell ULSFO, solidification may pose a challenge for mechanical recovery at low temperatures, as the flow of the oil towards skimmers may be reduced, which is estimated to occur approximately 5-15 °C below the oils' pour point. Thus, the high pour point of ULSFO may be a more significant limiting factor for mechanical recovery than viscosity at low temperatures.

Use of chemical dispersion can be challenging for all tested fuel oils from different operative aspects. For DMA-qualities, the thin film thickness may result in herding (dispersant breaking through oil film) rather than migration into the oil. A film thickness of 50-100 µm is in general considered as a minimum for recommending chemical dispersion. If application of dispersant is successful, the effectiveness is expected to be good. WRG has a good potential for dispersant use in summer conditions.

For the HDME 50 and Shell ULSFO fuel oils, application of dispersant may have limited effect, particularly in winter conditions. Repeated application will likely increase the effectiveness, but the oils are not expected to be completely dispersed. HDME 50 and Shell ULSFO may solidify at low temperatures and the solid oil lumps will not be dispersible. The applications of dispersant should ideally be performed before emulsion viscosities become too high, in effect as rapidly as possible after the release.

Due to relatively low flash points and lacking emulsifying properties, DMA-qualities may have potential for *In-situ* burning, provided spreading of the oils have been prevented. A film thickness of 5-10 mm is considered necessary for reducing heat loss to the underlying water phase sufficiently for this fuel quality. For heavier distillates and bunker fuel oils, a 10 mm film thickness may be necessary (Buist, 2003). For emulsifying oils, potential for ISB is considerably lower, and for Shell ULSFO the predicted window of opportunity will be short after release. Due to the combination of high flash points and emulsifying

properties of WRG and HDME 50, ISB is not recommended as a response option with the current ignition procedures. Introduction of high temperature ignitors may enhance the possibility for successful ISB for these oils.

Studies regarding the behaviour of hybrid fuel oils in scenarios when these fuel types reach the shoreline was not included in this study. In order to determine the optimal response for shoreline remediation, further studies is recommended. The significant differences in properties and behaviour for the hybrid oils tested in this reported study may also have significance for choice of shoreline response actions.

8.4 Future challenges with Hybrid fuel oils

This project has included two different types of hybrid fuel oils. Recent information indicate that the number of different marine fuel oils that can classify as Hybrid qualities are increasing. This increase in availability of hybrid oils has occurred rapidly within the past few years, and the increase will likely continue over the years to come. This study has revealed some similarities but also significant differences between the two tested hybrid fuel oils, ExxonMobil HDME 50 and Shell ULSFO, and the obtained results are only relevant for these specific oils. In addition, comparisons of different batches of HDME 50 and WRG indicate that the obtained results may potentially be relevant only for the specific production batches used in these experiments. This may also be the situation for Shell ULSFO.

Since weathering properties of the new hybrid fuel oils are mainly unknown, these types of fuel oils pose an uncertainty regarding its behaviour in different spill situations. Individual weathering studies would provide good information for each oils' properties and expected behaviour in an acute situation. A mapping of the properties and behaviour of several different hybrid fuel oils from various refineries from different countries may provide an indication of the range of properties these fuel types have. Such information may reveal oil types that ought to be tested further, and may form a basis for some general guidelines or recommendations for hybrid fuel oils.

In addition, the extent of batch variations within individual hybrid fuel oils should be further investigated. The impact of these variations in trajectory models should also be investigated, if data from a single batch is to form the basis for such modelling in the future.

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A Experimental setup

The experimental setup is described for crude oils but the methodologies also apply for marine distillate fuel oils and hybrid fuel oils in this project.

A.1 Bench-scale laboratory testing

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

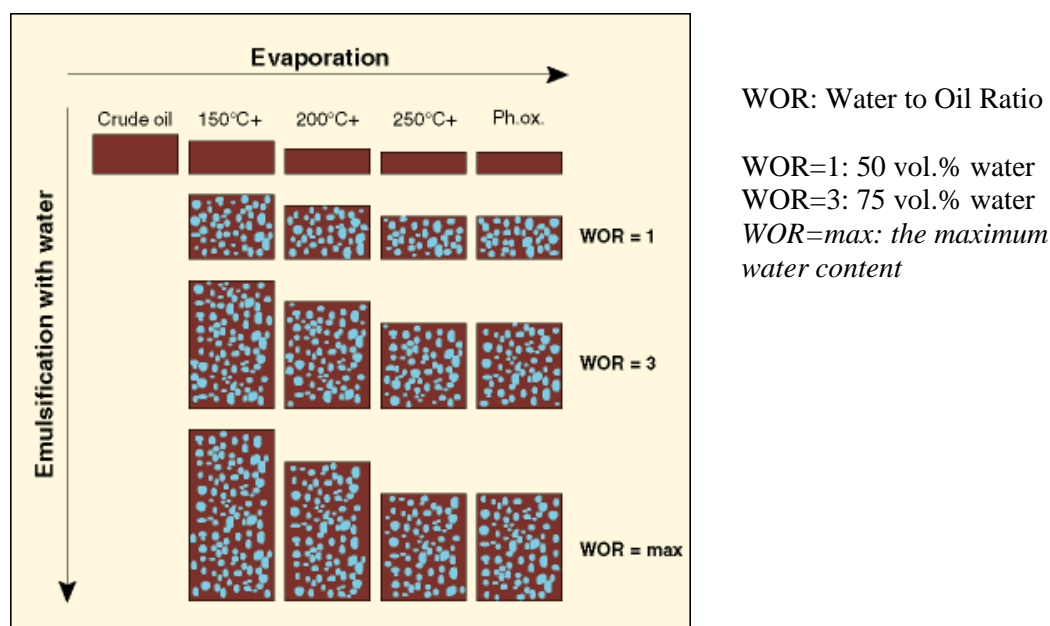


Figure A-1: Small-scale laboratory weathering flow chart of oil

A.1.1 Evaporation

The density of the oil was monitored during the degassing. This was performed before evaporation by standard procedure.

The evaporation procedure used is described in Stiver and Mackay (1984). Evaporation of the lighter compounds from the fresh oil was carried out as a simple one-step distillation to vapour temperatures of 150°C, 200°C and 250°C, which resulted in oil residues with an evaporation loss corresponding to approximately 0.5-1 hour, 0.5-1 day and 0.5-1 week of weathering on the sea surface. These residues are referred to as 150°C+, 200°C+ and 250°C+, respectively. For the marine fuels tested in this project, the DMA-qualities GO, MGO and Rotterdam diesel as well as the hybrid fuel oil Shell ULSFO were weathered to one residue, the 250°C+ residue. Neither WRG nor HDME 50 had significant content of compounds with boiling point below 250 °C, and this residue was not formed for either oil.

A.1.2 Physical and chemical analysis

The viscosity, density, pour point and flash point of the fresh oil and water-free residues were analysed. In addition, wax content and "hard" asphaltene content were measured for the 250°C+ residue where this was available, and for the fresh oil when necessary (WRG and HDME 50). 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
Interfacial tension (IFT)	-	Pendant Drop method, KRÜSS Germany

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

Chemical characterization by GC/FID and GC/MS

The distribution of hydrocarbons (nC_{10} - $nC_{36/40}$) was analysed using a Gas Chromatograph coupled with a Flame Ionisation Detector (GC/FID). The system comprised of an Agilent 6890N GC fitted with an Agilent 7683B Series autosampler.

Gas chromatography–mass spectrometry (GC/MS), used for analysis and quantification of PAHs, phenols and alkylated phenols (C_0 - C_4) is operated in selected ion monitoring (SIM) mode. The system comprised of an Agilent 6890N GC with an Agilent 5975B quadrupole mass-selective detector (MSD).

The volatile components in the range of nC_5 - nC_{10} were quantified by use of Chromatography/Mass Spectrometry (P&T GC/MS), using a modified EPA-Method 8260, with a 50 m (0.20 mm id, 0.50 μ m filmthick ness) Supelco Petrocol capillary column. Target analyses were detected with an Agilent 5973B mass selective detector (MSD) and the data acquired using the Agilent EnviroQuant Chemstation software.

A.1.3 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.

The stability of an emulsion can be tested by comparing the water content of the emulsion after 24 hours of rotation in the rotating cylinders, to the water content after subsequent 24 hours of settling. The effectiveness

of emulsion breakers can be analysed in a similar way, by adding emulsion breaker after emulsification (24 hours of rotation) but prior to 24 hrs settling.

The stability on an emulsion, with or without added emulsion breaker, is presented as a fractional dehydration, which provide a number between 0 and 1, where:

Stability ratio = 1: The emulsion is completely stable and has lost no water content

Stability ratio = 0: The emulsion is completely unstable and totally broken having lost all its water content

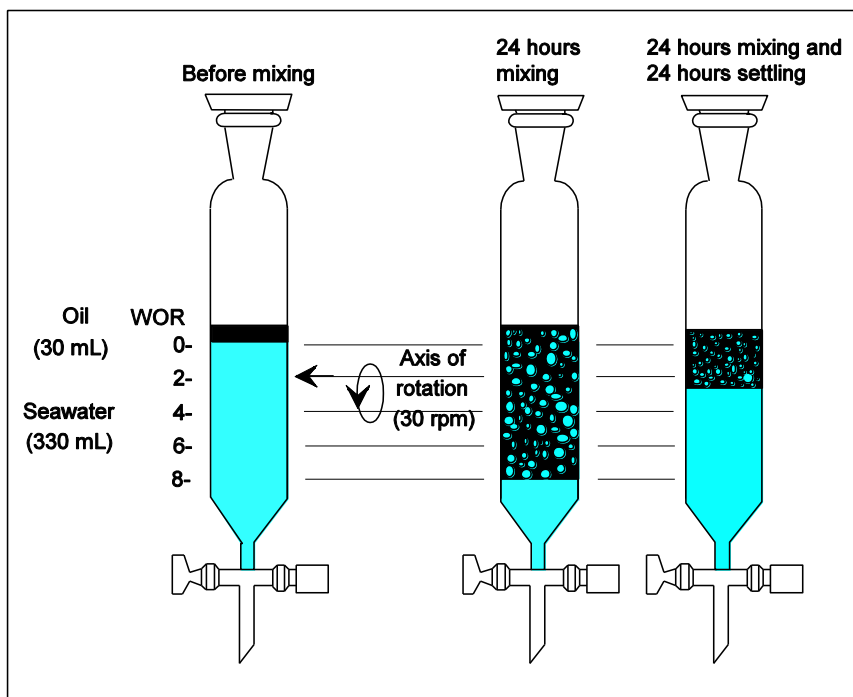


Figure A-2: Principle of the rotating cylinder method

A.2 Chemical dispersibility testing

As a screening test, the oils dispersibility properties were tested using five different dispersants at a dosage of 1:25 (4 wt. %). The dispersants tested were; Dasic Slickgone NS, Corexit 9500, Gamlen OD 4000, Finasol OSR-52, SuperDispersant 25 and Radiagreen. For the dispersant showing greatest dispersibility, different dosages were studied. The screening was performed using the IFP method (discussed below) on the water-free 250°C+ residues of the DMA-diesels and the WRG, and on a 50 vol. % emulsion of seawater and fresh oil for HDME 50 and Shell ULSFO. For the dosage testing, both the IFP and the MNS tests were used. The viscosity of all the weathered samples was determined.

There are several different tests for evaluating the effect of chemical dispersants. The energy input will differ in the different tests, and the obtained efficiency will be representative of different wave energies. At SINTEF, the IFP and MNS test is used in dispersibility testing.

IFP (Institute Francais du Petrole test, Bocard *et al.*, 1984) is a low energy test estimated to represent low wave energies (2-5 m/s wind speed). A surge beating up and down in the test vessel at a given frequency, gives energy input to the seawater column. The water column is continuously diluted, which gives a more realistic approach to field conditions, compared to other tests.

MNS (Mackay and Szeto, 1980) is estimated to correspond to a medium to high sea-state condition. The energy input in this system, applied by streaming air across the oil/water surface, produces a circular wave motion. The sample of the oily water is taken under dynamic conditions after a mixing period of 5 min. Both IFP and MNS test apparatus is shown in Figure A-3.

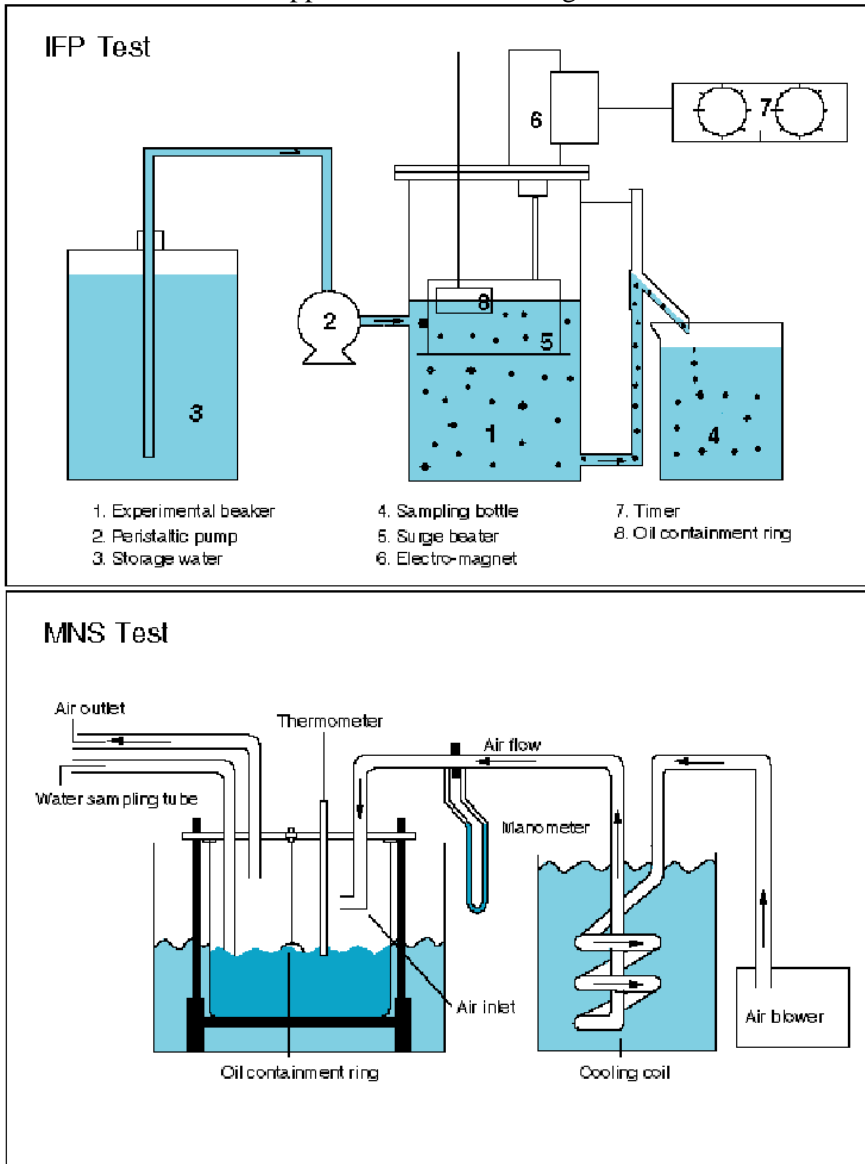


Figure A-3: IFP and MNS test apparatus.

A.3 Meso-scale laboratory testing

In an oil spill situation at sea, the weathering processes will occur simultaneously and affect each other. In the small-scale laboratory testing, these processes are studied individually. By using the meso-scale flume the weathering and effect of dispersants can be studied under more realistic conditions, having the weathering processes to occur at the same time, giving a better image of the behaviour of the oil at sea.

A.3.1 Description of the flume basin

A meso-scale flume basin (Singsaas et al., 1993), located at SINTEF's Sealab, is routinely used to simultaneously study the weathering processes under controlled conditions. The meso-scale flume basin was

rebuilt in 2006, having state of the art instrumentation. A schematic drawing of the flume is given in Figure A-4.

Approximately 5 m³ seawater circulates in the 10 metres long flume. The flume basin is located 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.

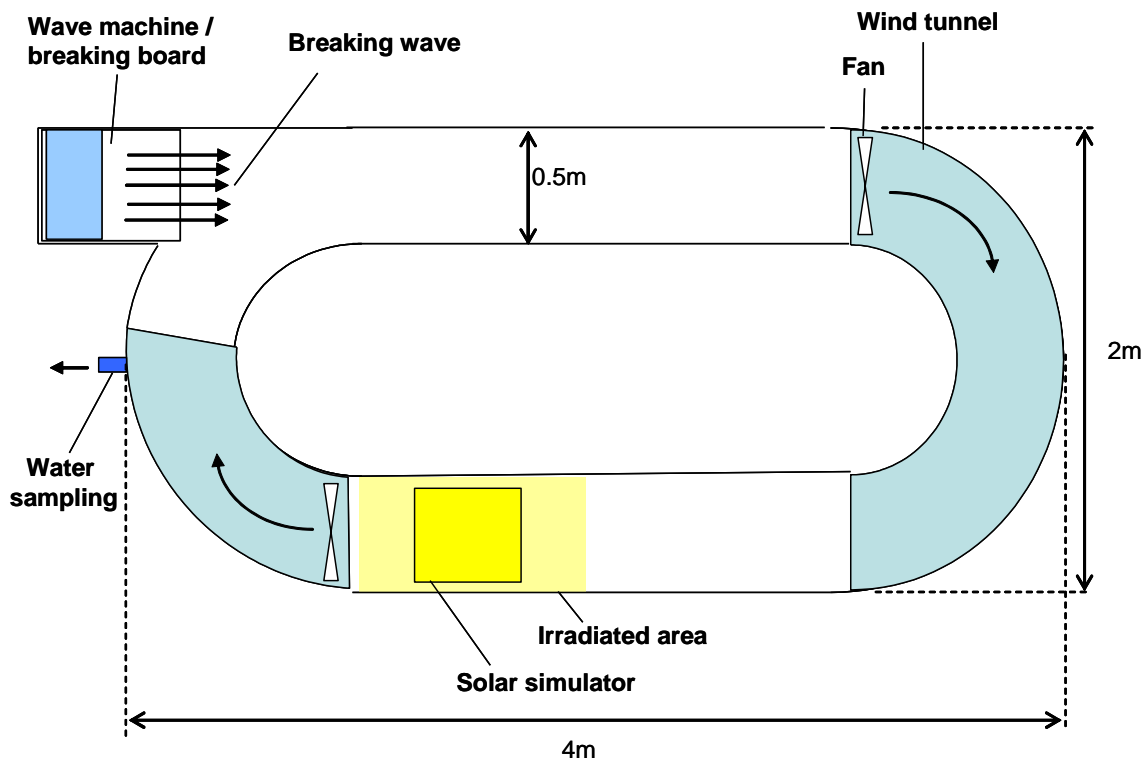


Figure A-4: Schematic drawing of the meso-scale flume

A.3.2 Oil weathering in the flume basin

In a meso-scale flume study, a standard volume sample (9 L) of the relevant oil is carefully released on 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. Both surface oil/emulsion and water column are sampled frequently the first 6 hours, then after 12 hours and subsequently every day.

After three days (72 hour) dispersant is applied to the surface emulsion. Water samples are then collected more frequently. The addition of dispersant may be repeated once or twice, in which case water samples are also collected.

Analysis of surface oil/emulsion

Samples of the surface oil/emulsion are taken using an aluminum tray and transferred to a 0.5 L separating funnel. The free water is removed after settling for 10 minutes in the climate room. The oil phase is further handled for an analysis of its physical properties.

The physical properties determined for all surface oil samples during the experiments are:

- Viscosity
- Water content
- Density
- Evaporative loss

- Emulsion stability (for a limited amount of samples)

Analysis of water samples

Water samples are analysed for oil concentration in the water column (droplets and dissolved components). The samples are taken at a depth of 50 cm through a tap in the basin wall into a Pyrex glass bottle (1 L). The sampled water is acidified with some droplets of 10% HCl (pH < 2). The sampling position is shown in Figure A-4. Samples are then extracted by liquid-liquid extraction with dichloromethane (DCM) and quantified by Ultra Violet (UV) spectrophotometer.

Solar simulation in the meso-scale flume

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-5 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).

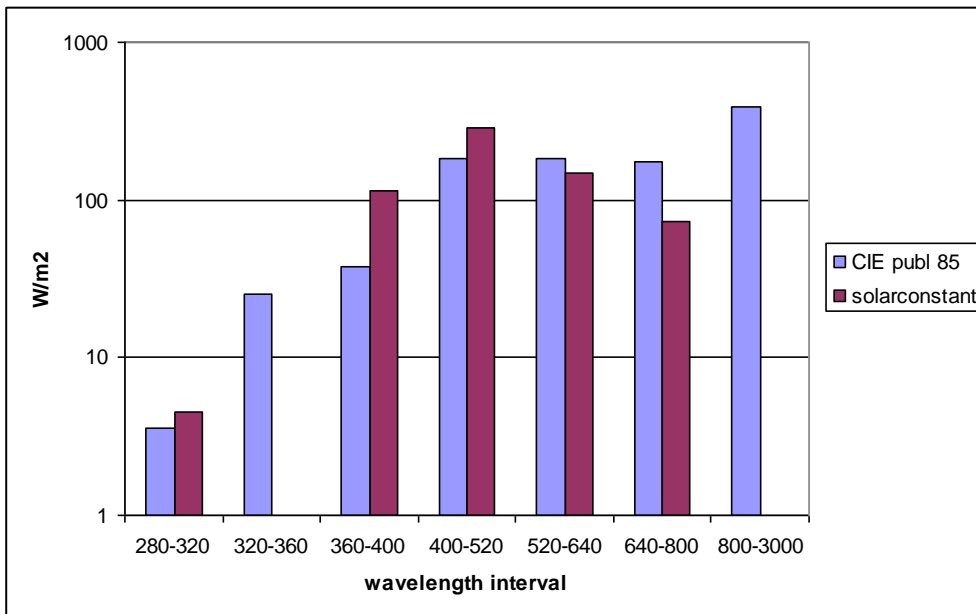


Figure A-5: Measured wavelength compared to standard spectrum suggested in CIE publication 85

The exact exposure of solar irradiance on the oil in the meso-scale flume is somewhat difficult to calculate. Since the oil is moving, it will only be within the irradiated area periodically. The exposure will be highly dependent on the distribution of the oil in the flume and the thickness of the emulsion on the water surface. An estimated irradiation pr. day using the solar simulator is compared with the average irradiation pr. day for selected Norwegian cities in Figure A-6.

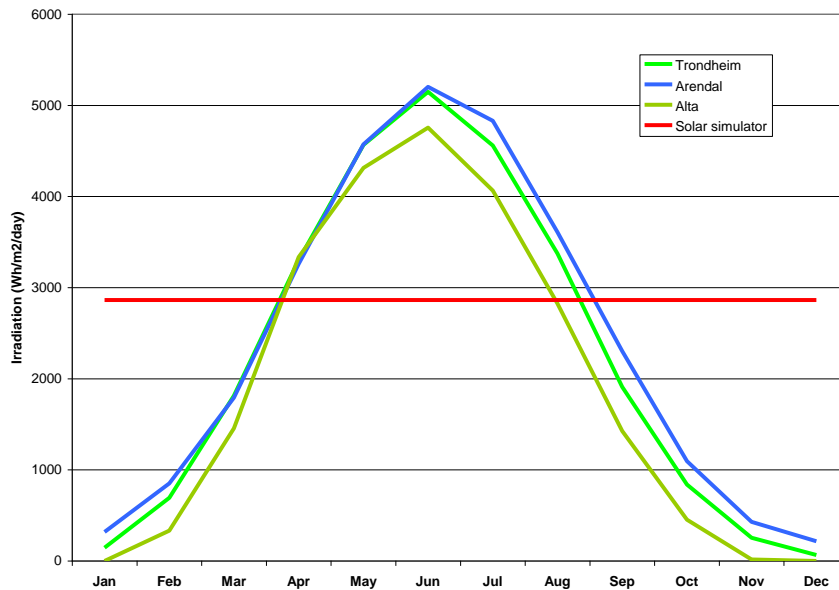


Figure A-6: Daily Irradiation for some Norwegian cities throughout the year compared with the estimated daily irradiation in the meso-scale flume

The simulated irradiance seems to be the approximate average of the daily irradiance throughout the year. It should be noted, however, that the simulations are not taking clouds into consideration. The real irradiation will be lower than that predicted in Figure A-6.

A.4 Ignitability testing

A.4.1 Bench-scale testing

SINTEF has previously conducted several field trials with the aim to study ISB of crude oil in ice. Field experiments have been conducted both at Svea on Svalbard and in the Barents Sea (Stand-Rasmussen *et al.*, 2010; Stand-Rasmussen and Brandvik, 2011). As a part of these experiments a bench-scale Burning cell was developed in order to evaluate the ignitability of oil samples of various degrees of weathering before the remaining oil slick was attempted ignited. The ignitability an oil tested with the Burning cell has been verified through field trials with crude oil (Brandvik *et al.*, 2010).

The Burning cell consists of a cup, a cooling apparatus, several thermo-elements for the measuring of oil, water and flame temperatures, and has an inbuilt ventilation. The cup is filled with sea water where upon an oil sample (approx. 150 ml) is added. A sketch of the Burning cell is given in **Error! Reference source not found.** below.

For the ignition of the selected oils and residues, a propane flame held at 30° angle from the surface was used. The ignition was attempted for 10 seconds at the time, with a pause of 2 minutes in between attempts. Three attempts were made with this ignition. If the oil did not ignite during these three attempts, a final attempt was made with a prolonged ignition time of 40 seconds. The time it took before the oil ignited was documented.

The methodology used for the Burning cell was originally designed for the ignition of crude oils where the presence of light components contribute to lower flashpoints than the flash points usually seen for the diesel and oil types used in this project. Since diesel oils often lack the lightest components found in crude oil, a fourth attempt with prolonged ignition time was performed in order to achieve high enough temperature for ignition to occur.

The burning efficiency was estimated gravimetrically by comparing the weight of the oil before and after burning.

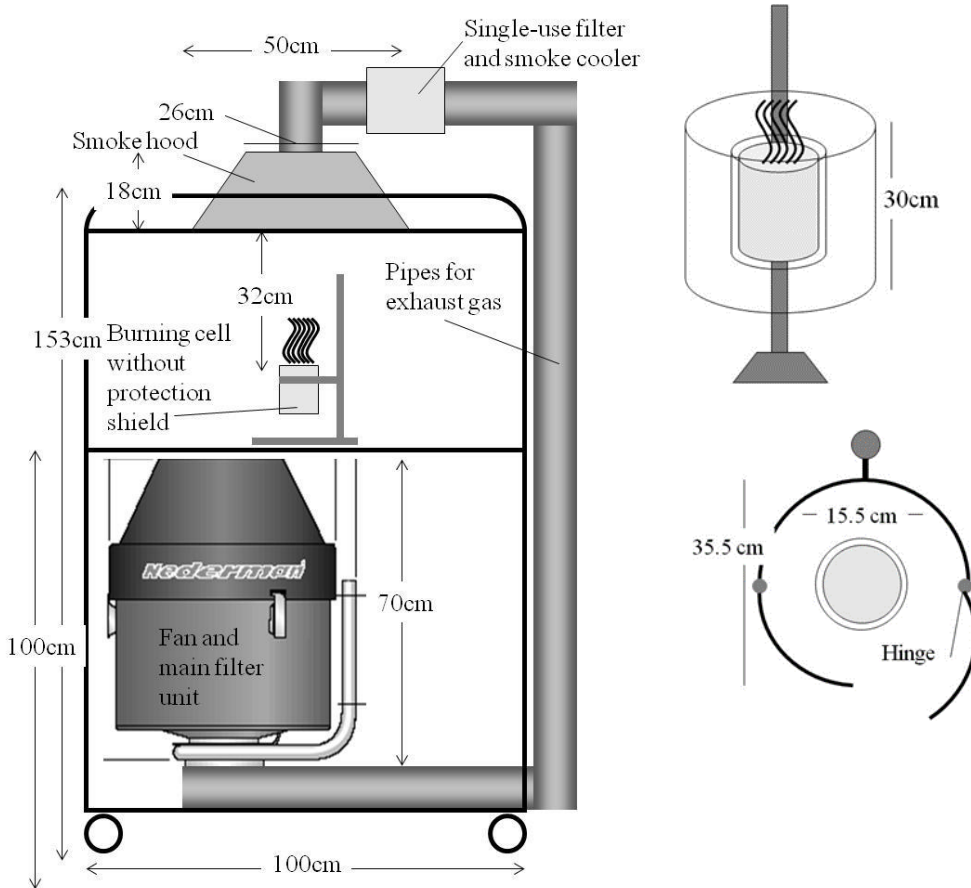


Figure A-7: Sketch of the SINTEF Burning cell

A.4.2 Up-scaled testing at Falck Nutec

Up-scaled ignitability testing was conducted at Falck Nutec in Trondheim. Falck Nutec is the largest supplier of quality research-based safety and emergency services to the maritime industry in Norway. They have specialized facilities for experiments and training with fires, and have all required release permits.

The tests were performed in a square basin (approximately 60 x 60 cm) filled with fresh water, as seen in Figure A-8. Approximately 5 L of oil were used in each test, providing a film thickness of 1.4 cm. Special thermometers were used to log the oil temperature, the flame temperature and potential temperature variations in the water column caused by heating from the fire.

For ignition, packs containing 500 ml gasoline gel were used. One pack was placed in the middle of the oil film and ignited. If the oil had not ignited by the near end of the packs' burning time (typically 8-10 minutes), another pack was placed in the same spot and ignited. This was repeated twice, giving a total of 3 packs of gelled gasoline per test (if required).

Ignition of the oil was manually registered, and determined by the spreading of flames across the oil slick. The time before the first pack of gel was ignited to the oil caught fire was registered, and the number of gel packs required was counted. The burning time of the oil was also documented, and the burning efficiency

was determined gravimetrically by comparing the collected remnants after burning relative to the originally applied amount of oil.



Figure A-8: Basin used for up-scaled testing, shown with sensors for flame temperature, oil temperature and water temperature.



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