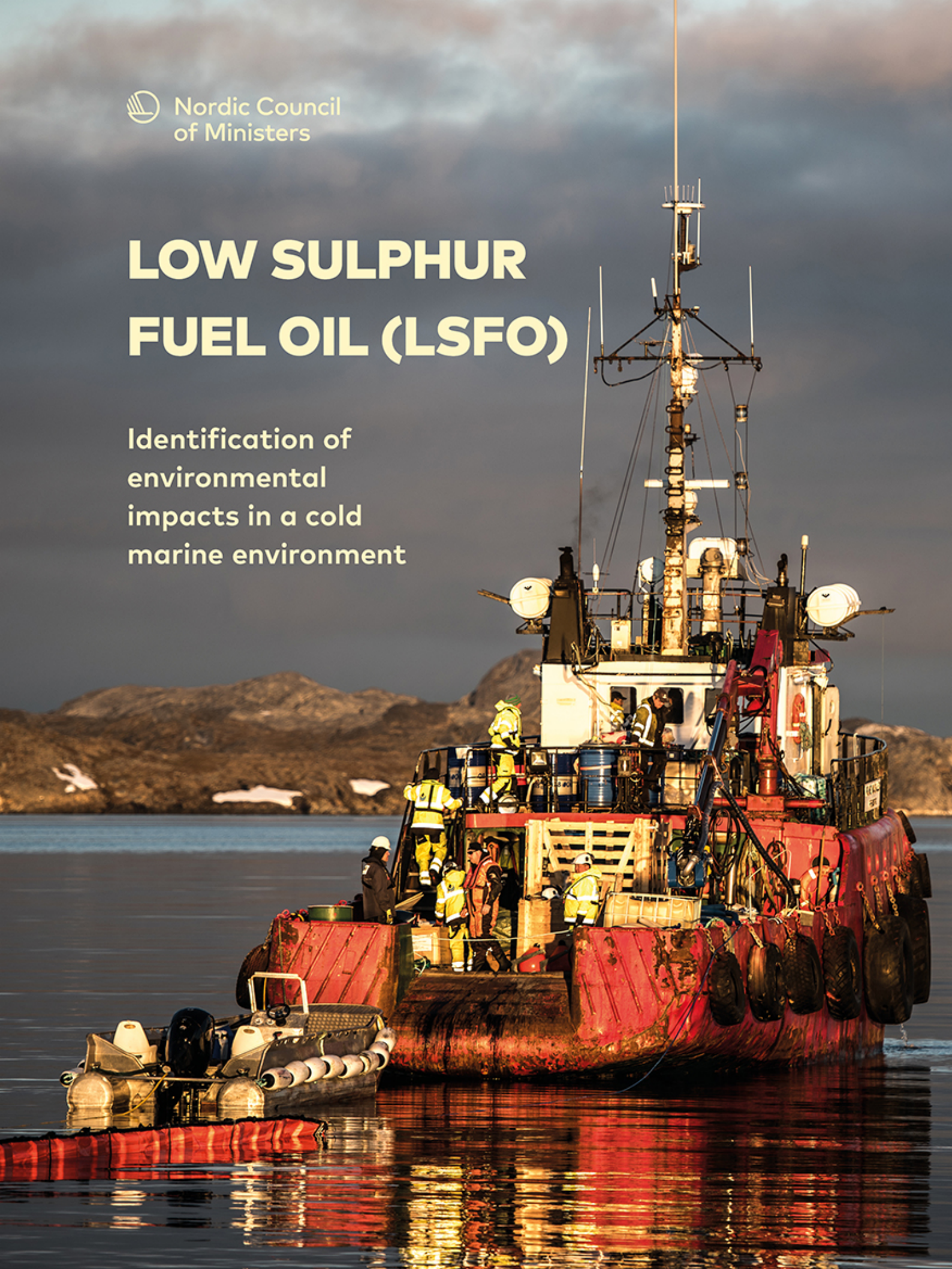




Nordic Council
of Ministers

LOW SULPHUR FUEL OIL (LSFO)

Identification of
environmental
impacts in a cold
marine environment



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This publication is also available online in a web-accessible version at <https://pub.norden.org/temanord2022-558>.

Preface

In connection with new international rules for the sulphur content of ship fuels, new low sulphur fuel oils (LSFO) have been developed. The present project aims to gather and strengthen the knowledge base on the environmental fate and behaviour of LSFO spills in cold seawater. The project includes laboratory experiments to investigate the effectiveness of combating LSFO spills in cold seawater by in-situ burning and chemical dispersion, respectively, and the potential for biodegradation of LSFOs and dispersed LSFOs.

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<https://www.youtube.com/watch?v=yA9DJleljdq>

Summary

New international regulations on the sulphur content of ship fuels (IMO 2017) came into force in 2015 within the Sulphur Emission Control Areas (SECAs); thus, ships must use fuels with a maximum sulphur content of 0.1%S, called ultra low sulphur fuel oil (ULSFO), and 0.5%S called very low sulphur fuel oil (VLSO), globally outside SECA from 2020. Thus, an increasing number of “new generation” low sulphur fuel oils (LSFO, both ULSFO and VLSFO) are currently being marketed for replacement of the conventional fuel types, such as heavy fuel oil (HFO) with a high content of sulphur (1–3.5%S). LSFO is a wide group of different oil products with varying chemical compositions. The very different physical and chemical properties will have major influence on the distribution and fate of spill of the various LSFOs in the sea.

The present project aims to gather and strengthen the knowledge base on the environmental fate and behaviour of LSFO spills in cold seawater and to overall asses which key organisms are at risk when spills are combatted by in-situ burning or chemical dispersion. The project includes laboratory experiments to investigate the effectiveness of combating LSFO spills in cold seawater by in-situ burning and chemical dispersion, respectively, and the potential for biodegradation of LSFOs and dispersed LSFOs.

In the project, three different LSFOs were included: wide range diesel (WRD, a distillate fuel), ultra low sulphur fuel oil (ULSFO 2018, a residual fuel oil) and heavy distillate marine ECA 50 (HDEM50, a distillate fuel oil made from heavy distillation cut). Additionally, to be able to make a relative assessment of “old” fuel types, the project also included a heavy fuel oil (IFO 180) and an arctic diesel type oil (AGO). The study only includes fresh oils and not weathered (i.e. evaporated and emulsified) oils. Oil weathering will further reduce the potential for combatting an oil spill.

The results from the laboratory dispersion tests indicated that the tested LSFOs were difficult to disperse. This was mainly due to the high pour points of the oil in combination with the low water temperatures (2 and 5 °C). Increasing the test temperature from 2 to 5 °C resulted in a small increase of dispersion effectiveness. WRD was somewhat dispersible and highest for dispersant:oil ratio (DOR) 1:10 and at 5 °C, whereas HDME50 was poorly dispersible even for successive dispersant applications. The PAH (polyaromatic hydrocarbon) composition in the water was similar to the composition in the fresh oil. The PAHs were dominated by naphthalenes and phenanthrenes that are partly dissolved in the water, but the majority of PAHs from the dispersion tests were found in the dispersed oil.

Small-scale in-situ burning tests were completed for WRD and ULSFO 2018 using different film thicknesses, and AGO was included for the purpose of comparison. Even though all the oils tested were ignitable, ULSFO 2018 was most difficult to ignite, suggesting it would be even more difficult to ignite in a real spill. The burn efficiencies were >50% and highest for WRD as a 20 mm film reaching 85%. None of the burn residues sank, and burn residues from the tested oils are therefore expected to be of highest environmental concern for surface living organisms. As also observed for burn residues in earlier studies including crude oils and HFOs, there was an increase in the 4-6 ring PAHs in the residues of all oil types.

Biodegradation of WRD and ULSFO and dispersant treated WRD and ULSFO was tested with fresh oil. Approximately 20% and 50% of the ULSFO and WRD, respectively, were degraded in 5 °C Baltic Sea seawater during the experiment (28 days). Experiments with and without dispersant had similar biodegradation rates.

LSFOs show a wide span in properties relevant for behaviour and fate when spilled at sea. The results of the dispersion test indicated a limited potential of natural and chemical dispersion of LSFO spills in cold seawater. However, it should be noted that these oils are difficult to handle in small amounts, and a large variation between replicates was found. The burn tests indicated that the tested LSFOs were ignitable and that the burn residues did not sink.

Overall, our results indicate that the LSFOs tested likely have a high degree of persistence on the sea surface and shoreline even when chemical dispersion or in-situ burning is attempted.

Sammenfatning

Nye internationale regler omkring indholdet af svovl i skibsbrændstof (IMO 2017), trådte i kraft i 2015. Dette betyder at indenfor Sulphur Emission Control Areas (SECAer), må skibe kun anvende brændstoffer med et svovlindhold på maksimum 0,1 %S, benævnt ultra low sulphur fuel oil (ULSFO) og globalt udenfor SECA et svovlindhold på maksimum 0,5 %S benævnt very low sulphur fuel oil (VLSFO), fra 2020. Således markedsføres et stigende antal af nye olier med et lavt svovlindhold (LSFO, både ULSFO og VLSFO) til erstatning for de konventionelle brændstoffer, såsom tunge bunkerolier (HFO) med et højt indhold af svovl (1–3,5 %S). LSFO'er er en omfattende gruppe af forskellige olieprodukter med varierende kemiske sammensætninger. De meget forskellige fysiske og kemiske egenskaber vil have stor indflydelse på fordelingen og skæbnen i havet i tilfælde af spild.

Nærværende projekt har til formål at samle og styrke vidensgrundlaget om LSFO-spilds skæbne og opførsel i miljøet og i koldt havvand og overordnet vurdere, hvilke nøgleorganismer der er særligt udsatte, når udslip bekæmpes ved in-situ afbrænding eller kemisk dispergering. Projektet omfatter laboratorieforsøg for at undersøge effektiviteten af bekæmpelse af LSFO-udslip i koldt havvand ved henholdsvis in-situ afbrænding og kemisk dispergering samt potentialet for biologisk nedbrydning af LSFO'er og dispergerede LSFO'er.

Projektet inkluderede tre forskellige LSFO'er: 'wide range diesel' (WRD, et destillatbrændstof), 'ultra low sulphur fuel oil' (ULSFO 2018, en residualbrændselsolie) og 'heavy distillate marine ECA 50' (HDEM50, en destillatbrændselsolie fra et tungt destillationsprodukt). For at kunne foretage en relativ vurdering af "gamle" brændstoftyper omfattede projektet også en tung bunkerolie (IFO 180) og en arktisk dieselolie (AGO). Projektet omfatter kun forsøg med friske olier og ikke forvitrede olier (dvs. fordampede og emulgerede). Olieforvitring vil yderligere reducere potentialet for at bekæmpe et olieudslip.

Resultaterne fra laboratorieforsøg med dispergering viste, at de testede LSFO'er var svære at dispergere. Dette skyldtes primært oliernes høje hældepunkter i kombination med de lave vandtemperaturer (2 og 5 °C). En øgning af testtemperaturen fra 2 til 5 °C resulterede i en lille stigning i dispersionseffektiviteten. WRD var let dispergerbar og højest for dispergent:olie-ratio (DOR) 1:10 og ved 5 °C, hvorimod HDME50 var svært dispergerbar, selv efter flere applikationer af dispergeringsmiddel. Sammensætningen af PAH'er (polyaromatiske kulbrinter) i vandet svarede til sammensætningen i den friske olie. PAH'erne var domineret af naphthalener og phenanthrener, som er delvist opløselige i vand, men størstedelen af PAH'erne fra dispergeringsforsøgene var fra den dispergerede, men stadig uopløste, olie.

Småskala in-situ afbrændingsforsøg blev gennemført for WRD og ULSFO 2018 og med forskellige oliefilmstykker. AGO var også inkluderet med henblik på sammenligning. Alle olierne kunne antændes, men ULSFO 2018 var sværest at antænde. Dette tyder på, at denne olie vil være endnu sværere at antænde i forbindelse med et virkeligt oliespild. Afbrændingseffektiviteten var >50 % og højest

for WRD, hvor en 20 mm film resulterede i en effektivitet på 85 %. Afbrændingsresterne sank ikke, og derfor forventes den største miljømæssige påvirkning fra de testede olier at være i relation til overfladelevende organismer. Som også beskrevet i tidligere studier med både råolier og bunkerolier, ses der en stigning i 4-6-ring-PAH'er i afbrændingsresterne.

Biologisk nedbrydning af frisk WRD og ULSFO samt WRD og ULSFO behandlet med dispergeringsmiddel blev testet. Omtrent 20 % og 50 % af henholdsvis ULSFO og WRD blev nedbrudt i 5 °C havvand fra Østersøen (over 28 dage). Forsøg med og uden dispergeringsmiddel havde tilsvarende biologiske nedbrydningsrater.

LSFO'er har et vidt spænd i egenskaber, der er relevante for oliernes opførsel og skæbne ved spild i havet. Resultaterne fra dispergeringsforsøgene indikerede et begrænset potentiale for både naturlig og kemisk dispergering ved spild i koldt havvand. Det skal dog bemærkes, at disse olier er svære at håndtere i små mængder, og at der blev fundet stor variation mellem replikater. Afbrændingsforsøgene viste, at de testede LSFO'er var antændelige, og at afbrændingsresterne ikke sank.

Samlet set indikerer vores resultater, at de testede LSFO'er sandsynligvis har en høj grad af persistens på havoverfladen og kystlinjen, selv ved kemisk dispergering og in-situ afbrænding.

1. Introduction

As a result of the decreasing sea ice cover in the Arctic Ocean, new shipping routes will become available, and shipping activities in Arctic waters are thus expected to increase significantly. The greatest increase in traffic is cargo ships sailing in and out of the Arctic area. This enhanced destination traffic has taken place over at least two decades (2000–2020). Increased shipping activities, combined with undesirable climate effects with more rapid storms and stronger winds, are expected to augment the frequency of oil spills connected to shipping activities. For instance, DNV GL (Det Norske Veritas), in PAME II (2016), estimated that “an incident leading to an oil spill is likely to happen every second year within the Bering Sea”.

New regulations of the sulphur content of ship fuels (IMO 2017) came into force in 2015 within the Sulphur Emission Control Areas (SECAs); thus, as from 2020 the maximum sulphur content of fuels used by ships is 0.1% or 0.5% worldwide in areas outside the SECAs. Hence, a growing number of low sulphur fuel oils (LSFOs) are currently being marketed to replace the traditional fuel types such as heavy fuel oil (HFO) with a high content of sulphur (1–3.5%S). LSFOs are a wide group of different oil products with varying chemical compositions, and they exhibit a wide span in properties such as viscosity, density and pour point (e.g. Sørheim et al. 2020), PAME/EPPR 2022).

Knowledge about the fate and behaviour of an oil spill in the marine environment is necessary to understand the ability to respond to and combat such an oil spill. The choice of response technique(s) with respect to operational applicability must be analysed together with the potential impacts on organisms in different environmental compartments (sea surface, sea column, sea bed and shoreline) in a Spill Impact Mitigation Assessment (SIMA) or a strategic environmental analysis using e.g. the EOS tool (Environment & Oil spill Response) (Wegeberg et al. 2020). Thus, detailed knowledge about the effects on key organisms in the environmental compartments as well as knowledge about the fate and behaviour of oils in the environment, also when treated as part of an oil spill response, is important for selecting the most efficient countermeasures to handle and combat spills.

Several factors influence the fate and behaviour of an oil spill in seawater. Besides air and seawater temperature, the physical and chemical properties of different oil types and derived compounds from a spill response operation are pivotal. One of the major challenges is the high pour point that many of these LSFOs have, resulting in solidification of the oil if it is accidentally spilled on a cold seawater surface (temperature below the pour point of the oil). A recent report from the ongoing PAME/EPPR project shows a large span in the pour points of both VLSFOs and ULSFOs. The variation in the pour points of LSFOs used in the Arctic are presented in Figure 1 and shows that the majority of the bunker fuels have a pour point above 0 °C. Solidification of the oil, will negatively impact the effectiveness of oil spill response equipment. For instance, many mechanical recovery methods rely on the flow of oil to skimmers; however, this will be limited due to the high pour point, resulting in reduced recovery effectiveness. Regarding the chemical dispersion of oil, the high pour point may inhibit the penetration of the dispersant into the oil, resulting in possible wash-off of the dispersant, and the method will therefore have no effect as also found for high viscous oils (Brandvik and Faksness 2009).

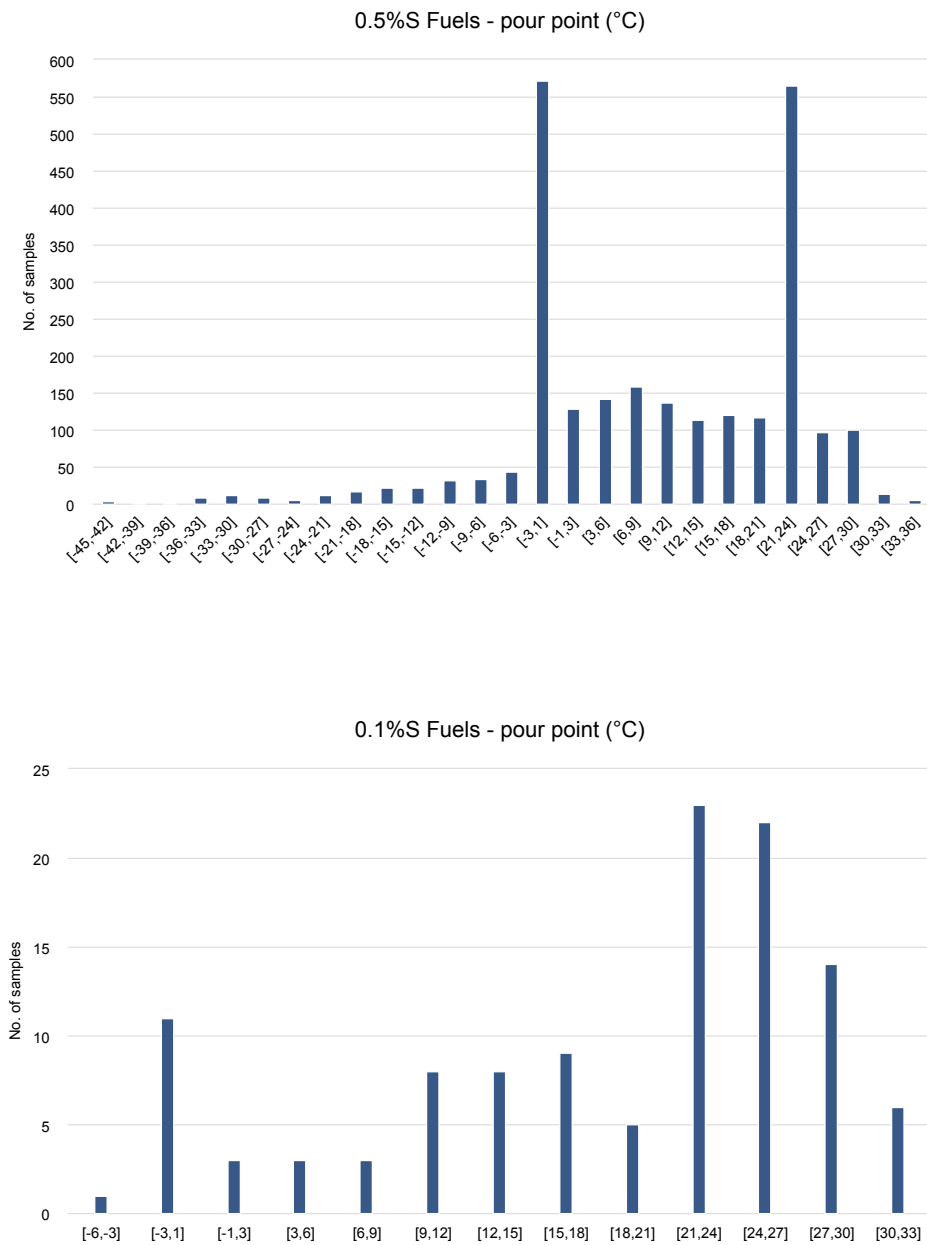


Figure 1: Number of bunker fuels used in the Arctic region grouped relative to pour point ranges. Top figure: 0.5% S content fuels (VLSFOs) and bottom figure: 0.1% S content fuels (ULSFOs).

Source: Figures from PAME/EPPR (2022).

In the longer run, e.g. natural biodegradation will impact the fate of the oil, but only if exposed to oil-degrading bacteria, either in the water column or in sediment (Fritt-

Rasmussen et al. 2018). Fritt-Rasmussen et al. (2018) found that the potential for natural biodegradation, assuming absence of nutrient limitation, of HFO was reduced at cold temperatures and limited by the amount of oil that can dissolve in the water phase. The potential for natural biodegradation of LSFOs under corresponding conditions may thus be even more reduced than that of HFO due to the limited bioavailable surface of the oil as a result of the high pour point.

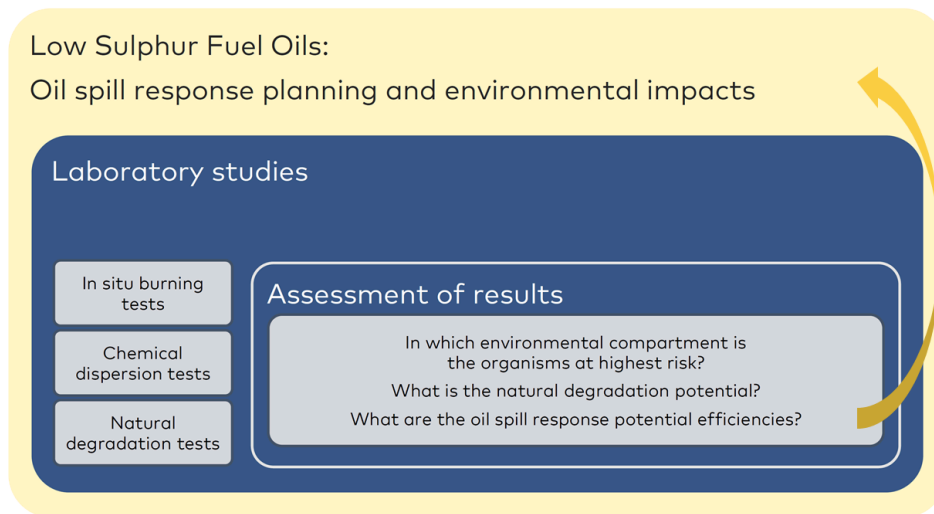
Oil itself may have significant adverse environmental impact on the marine environment, and the oil spill response techniques may have environmental side effects (Wegeberg et al. 2017). For the assessment of the impacts (the SIMA) on the environment, the possible impacts from an oil spill and from the treated oil as part of the response are assessed with focus on the relevant organisms and ecosystems for the different environmental compartments: sea surface, seawater column, seabed and shoreline (Wegeberg et al. 2020). For oil or residues from an in situ burning operation at the sea surface, the organisms considered most vulnerable are seabirds. Seabirds rely on their feather for insulation, flying and buoyancy (Stephenson 1997), and even small amounts of oil can have lethal effects by destroying the waterproofing of their plumage (Fritt-Rasmussen et al. 2016, Leighton 1993). Marine mammals, e.g. whales and seals, go to the sea surface when breathing. The potential risk to whales is related to smothering of the blow hole and baleen plates, although this effect has not yet been observed (AMAP 2010). As for the seawater column, if oil is naturally or chemically dispersed, plankton, fish larvae/fry and other pelagic organisms may be exposed to toxic concentrations if the dispersed oil is not diluted into non-toxic concentrations (Wegeberg et al. 2017). However, oil types with a low density, a high content of asphaltenes and paraffins and a high viscosity may only disperse to a low degree (Fritt-Rasmussen et al. 2018), and such oil types may have a high degree of persistence on the sea surface and hence also a higher risk of beaching. As such, organisms associated with the sea surface and the shoreline (e.g. birds and marine mammals as well as benthic organisms in the tidal zone) may be at higher risk of exposure to oil components (Wegeberg et al. 2020).

Therefore, here we aim at investigating the effect and efficiency of oil spill response methods, the fate of the oil and the treated oil in the environment and which environmental compartment(s), and hence which organism(s), may be at highest risk for exposure, even after potential oil spill response methods have been applied. This information will be used to focus and design exposure tests and to select the most relevant test organisms in a separate study. Note that our study only considers fresh oils and does not take into consideration the effect of weathering (e.g. evaporation of volatiles and emulsification) after the spill, which may have a very important effect on, for instance, the efficiency of the different response methods.

Hence, the questions to be answered in the project are (Figure 2):

- How efficient is combat of oil spill by LSFO by in-situ burning and chemical dispersion in a cold environment?
- What is the expected fate and biodegradation of LSFO in a cold environment in the case of natural and chemical dispersion?
- Which key organisms are at most risk from an untreated and treated (burned, chemically dispersed) LSFO spill based on the results from this study?

Figure 2: Overview of project questions.



2. Physical and chemical properties of the oils included in the project

In the project, three different LSFOs were included. As mentioned in the introduction, LSFOs are a wide group of different oil products with varying chemical compositions that differ in properties such as viscosity, density and pour point. This is central for their fate and behaviour in the sea as well as their potential combat at sea in the case of an oil spill. To be able to make a relative assessment for conventional fuel types previously tested, the project also included a heavy fuel oil (IFO 180) and an arctic diesel type oil (AGO). Selected properties of relevance for oil spill response apart from the sulphur content of the oil types are shown in Tables 1–5, and GC-FID chromatograms of the oils are shown in Figure 3. The GC-FID chromatograms present the composition of the oil from nC5-alkane to nC35-alkane as systematic narrow peaks, with the lowest boiling point in the first peaks. Waxes (n/iso paraffins) are typically found from n-C20-alkane and forward (Øksenvåg et al. 2021).

Viscosity of a crude oil is a measure of its resistance to flow, and it decreases with increasing temperature. Viscous and waxy crude oils as well as water-in-oil emulsion may exhibit non-Newtonian behaviour (i.e. viscosity change under force), especially close to, or below, their pour-point (Øksenvåg et al. 2021). This means that in a spill situation under sea turbulence conditions, the water-in-oil emulsions may be liquid, but under calmer conditions or at the shoreline the oil can become much more viscous (Øksenvåg et al. 2021). Thus, measurements of viscosity are given for two shear rates.

Wide Range Diesel Oil (*WRD, other names Wide Range Gas Oil/ Marine Distillate Oil*)

WRD is a somewhat heavier diesel fraction (<30% residual oil and >70% diesel), with a high boiling range/curve (approx. nC12–nC33), and it is considered to be a distillate fuel. Selected properties are given in Table 1. The oil is in the ISO8217 category RMA and IMO Resolution definition ULSFO-RM (Øksenvåg et al. 2021).

Table 1: Selected properties for Wide Range Diesel Oil – WRD¹.

Properties ³	
Density	0.872 g/ml
Pour point	-15 °C
Flash point	>100 °C ²
Sulphur content	0.042%
Viscosity at 5 °C (10 s ⁻¹)	102 mPa/s
Viscosity at 5 °C (100 s ⁻¹)	92 mPa/s
Asphaltenes	0.01 wt. %
Wax	4.74 wt. %

Note: ¹Marine distillate oil, 23 February 2021, ExxonMobil. This oil was also included in the fate and behaviour studies of sediments and bedrock in Øksenvåg et al. (2021) with the internal SINTEF ID 2021-361-S2 FRESH (WRG/MSD).

Source: ³All properties from Øksenvåg et al. (2021), except ²from Sørheim and Daling (2020).

ULSFO (Ultra Low Sulphur Fuel Oil), Shell 2018

ULSFO 2018 is a residual fuel oil with a high density and pour point. ULSFO 2018 has a broad range of n-alkanes (Figure 3, approx. nC10–nC35) as it is a mix of distillate and a residual fraction of heavier compounds. This is also reflected as a wide range of the boiling point curve. From nC-20, it reflects a high wax content (Sørheim et al. 2020). Selected properties of ULSFO 2018 are given in Table 2. The high wax content is reflected in the pour point.

Table 2: Selected properties for ULSFO Shell 2018¹.

Properties ³	
Density at 15 °C	0.917 g/ml
Pour point	24 °C
Flash point	85 °C
Sulphur content	0.1%
Viscosity at 5 °C (10 s ⁻¹)	42,029 mPa/s ²
Viscosity at 5 °C (100 s ⁻¹)	9,678 mPa/s ²
Asphaltenes	0.14 wt. %
Wax	20.7 wt. %

Note: ¹Ultra low sulphur fuel oil (ULSFO), RMD80 0.1%, from 18 February 2018, Rubis terminal, Rotterdam, Netherlands. This oil was also included in studies by Sørheim et al. (2020) with the internal SINTEF ID: 2019-11170

Source: ³All properties from Sørheim et al. (2020) except ²from Øksenvåg et al. (2021).

HDME 50 (Heavy Distillate Marine ECA 50)

HDME50 is considered as a distillate fuel oil made from heavy distillation cut with minor content of heavy compounds such as asphaltenes (Sørheim et al. 2020) and n-alkanes > nC-17 (Figure 3). Selected properties of the HDME50 are given in Table 3. As the material datasheet only provided limited information, the properties for another batch of HDME50 are given.

Table 3: Selected properties for Heavy Distillate Marine ECA 50 (HDME 50)¹.

Properties ³	
Density at 15 °C	0.903 g/ml
Pour point	12 °C
Flash point	186 °C
Sulphur content	0.1%
Viscosity at 5 °C (10 s ⁻¹)	5,045 wt.% ²
Viscosity at 5 °C (100 s ⁻¹)	2,472 wt.% ²
Asphaltenes	0.06 ²
Wax	9.5 ²

Note: ¹Heavy Distillate Marine ECA 50 – ExxonMobile Premium HDME 50. This oil was also included in studies by Hellstrøm (2017), Hellstrøm et al. (2017) and Faksness and Altin (2017), with the internal SINTEF ID: 2016-0231.

Source: ³All properties from Hellstrøm (2017) except ²from Øksenvåg et al. (2021).

Intermediate Fuel Oil 180 (IFO180)

The IFO180 used in this project comes from Greenland and was supplied by Polar Oil. IFO180 is a blend of IFO380 and marine diesel mixed by Polar Oil in a ratio meeting the IFO180 requirements (viscosity < 180 cS at 50 °C). No certificate of analysis is available for this specific oil. Hence, the values presented in Table 4 are for the oils included in the blend IFO380 and marine diesel and should therefore only be seen as an indication of the levels in the IFO180.

Table 4: Selected properties required for Polar Oil for Intermediate Fuel Oil 380 and marine diesel. Note that the values given are indicative and not absolute properties.

Properties	IFO380	MGO
Density at 15 °C	0.979 g/ml	0.820-0.860 g/ml
Pour point	-6 °C	n.a.
Flash point	114 °C	61 °C
Sulphur content	1.90%	n.a.
Viscosity at 40 °C	n.a.	2 – 4 mm ² /s

Source: Values from the product specification requirements provided by Polar Oil

Arctic Gas Oil (AGO)

The AGO used in this project comes from Greenland and is supplied by Polar Oil. Selected product specification requirements of the Polar Oil AGO are given in Table 5. It is an oil dominated in the lower boiling point range (n-alkanes approx. < nC-22) (Figure 3).

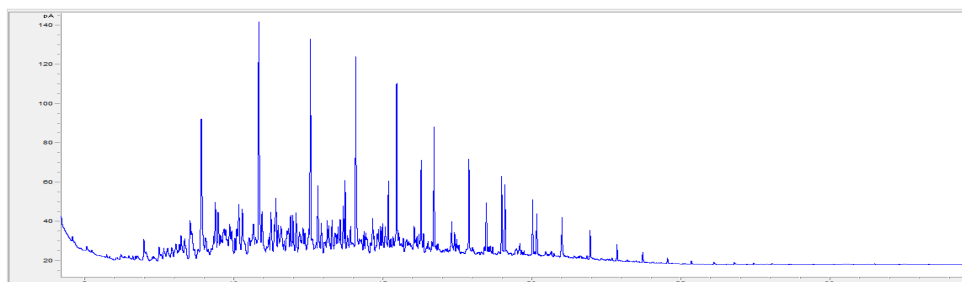
Table 5: Selected properties for Arctic Gas Oil (AGO).

Properties	
Density at 15 C	0.81 – 0.84 g/ml
Cloud point*	- 22 °C
Flash point	61.0 °C
Sulphur content	10 mg/kg
Viscosity at 40 °C	1.5 – 3.0 mm ² /s

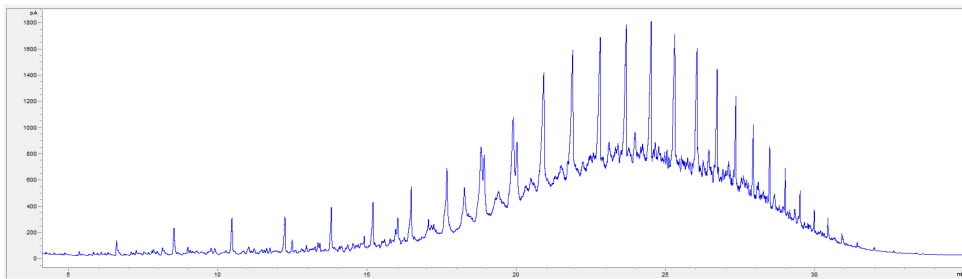
*Note: *Cloud point is the lowest temperature at which crystal formation can be observed as a cloudy suspension (Gouveia et al. 2017). The pour point is the minimum temperature below which a liquid loses its flow characteristics and as such is lower than the cloud point (Babu & Anand 2019).*

Source: Values from the product specification requirements provided by Polar Oil

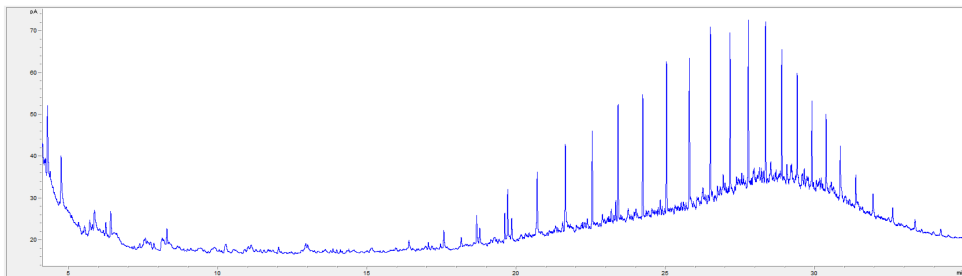
AGO



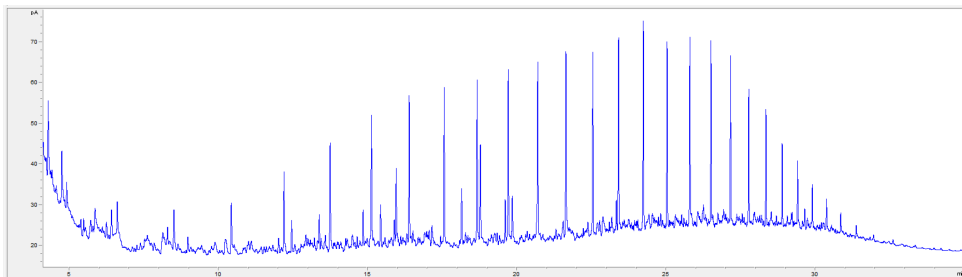
WRD



HDME



ULSFO 2018



IFO180

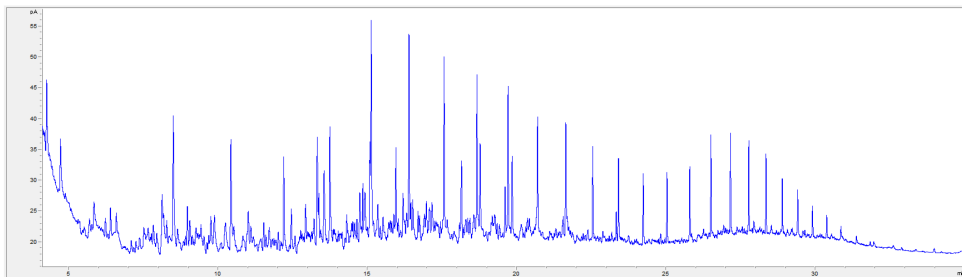


Figure 3: GC-FID chromatograms for AGO, WRD, HDME50, ULSFO 2018 and IFO180.

Note: For HDME50, ULSFO and IFO180, the first part of the chromatograms – up to 7 minutes – represents unknown impurities in the solvent that are not part of the oil. The chromatograms show n-alkanes as systematic narrow peaks, where the first peaks represent the compounds with the lowest boiling point. More complex components are shown as unclear bumps below the peaks (often denoted 'Unresolved Complex Mixture').

3. Dispersion tests

3.1 Experimental set-up of dispersion tests

Four different oils were included in the experiments; two LSFOs and – for comparison – also a conventional heavy fuel oil and a diesel type oil. The oils were: WRD, HDME50, AGO and IFO180. The chemical dispersant used in the dispersion tests was Finasol OSR 52. Saltwater with a salinity of 30.3‰ was used in the experiments.

The experiments were conducted in a climate room at 2 and 5 °C. Table 6 gives an overview of the experimental key parameters. Previous studies have been conducted at 2 and 13 °C and have shown that the temperature increase in many cases resulted in improved dispersion effectiveness. We conducted our tests at 2 °C to be able to compare our results with those of previous studies and included 5 °C in our experiments to investigate if such a small increase would improve the dispersion effectiveness, 5 °C being a typical summer surface water temperature in Arctic waters ([Carvalho & Wang 2020](#)). The dispersion effectiveness was tested at dispersant:oil ratios (DOR) of 1:10 and 1:25. However, for the HDME50, 2°C experiments, successive dispersion (2 x 1:10) was included as the oil was not dispersible at 2°C using a single 1:10 application.

Table 6: Seawater temperature and dispersant:oil ratios (DOR) in the dispersion testing.

Oil type	Water temperature [°C]	Dispersant	DOR
WRD	2 and 5	Finasol 52	1:10, 1:25
HDME50	2 and 5	Finasol 52	1:10, 1:10x2
AGO	2	Finasol 52	1:10, 1:25
IFO180	2	Finasol 52	1:10, 1:25

3.2 Dispersion test procedure

120 ml seawater was added to a 250 ml separatory funnel. 100 µl oil was carefully added to the seawater surface by a pipette. The dispersant was then added to the centre of the oil. Either 10 or 4 µl dispersant was added to a DOR of 1:10 or 1:25, respectively. The flasks were placed on an orbital shaker and mixed for 10 min at a rotation speed of 200 rpm. In some of the experiments, successive dispersion was included whereby additional dispersant (10 µl) was added after the first 10 min of shaking, followed by additional 10 min of shaking. The flasks were to remain stationary for 10 min before extraction. The extraction procedure was as follows: The first 2 ml of the sample was drained and discarded. Thereafter 50 ml of the sample was collected in a cylinder and transferred to a 500 ml separatory funnel

and extracted three times with 5 ml dichloromethane (DCM). The extract was adjusted to a final volume of 20 ml and stored at 5 °C until fluorescence/GC analyses. For reference, a number of controls followed the same procedure, i.e., oil without added dispersant as well as pure seawater. The number of replicates and controls can be found in Table 7, and photos from the laboratory are shown in Figure 4.

The dispersion test procedure was modified from Venosa et al. (2002) and Srinivasan et al. (2007), but it varies from these as separation funnels were used instead of baffled flasks. This most likely results in differences in the agitation turbulence, and direct comparison of results should keep this in mind. In general, it is difficult to conduct laboratory dispersion tests as the results rely on the surface area relative to the oil amount, the mixing energy and contact between oil and the glass wall. The test methods in the present study are challenged by a large surface area relative to a little amount of oil, possibly creating difficulties with dispersant-oil contacts and possible large contact between the oil and the glass wall during rotation.

The content of oil in each extract, calibrated with external standards, was determined using a Cary Eclipse Fluorescence Spectrophotometer, Agilent Technologies. The standard curves were prepared from measuring the fluorescence intensity of a number of external standards with known oil concentrations (see Appendix 1).

The dispersion effectiveness (DE %) was calculated as follows:

$$Dispersion\ effectiveness(\%) = \frac{(Total\ oil\ dispersed)}{V_{oil}} * 100 \quad \text{Equation 1}$$

where V_{oil} is the total volume of oil added to the funnel (100 µl) and total oil dispersed is from eq.2.

$$Total\ oil\ dispersed(\mu l) = concentration\ of\ oil * V_{DCM} * \left(\frac{V_{sw}}{V_{es}}\right) \quad \text{Equation 2}$$

where V_{DCM} is 20 ml, V_{sw} is the total volume of seawater in the funnel (120 ml), V_{es} is the total volume of seawater extracted (50 ml), and the concentration of oil is from eq. 3.

$$Concentration\ of\ oil\ \left[\frac{\mu l\ oil}{DCM\ ml}\right] = A * Fluorescence\ intensity - B \quad \text{Equation 3}$$

where A and B are the constants from the standard curve regression lines. A standard curve was prepared for each oil type (see Appendix 1).

The PAH (polyaromatic hydrocarbon) concentration in the extract was measured by GC-MS: Before analysis, samples were filtered via a funnel with glass wool + Na₂SO₄. The samples were evaporated and the solvent changed to toluene, which was transferred to GC-vials after which internal standards were added (5 deuterated PAHs). GC-MS: Analysis in SIM mode. Column: HP-5MS, length: 30 m, diameter: 0,250 mm, film thickness: 0,25 µm. Temperature programme: 90°C 1 min,

7°C/min to 240°C, 240°C 4 min, 20°C/min to 270°C, 270°C 18.5 min.

The PAH target compounds measured: Naphthalene, C1-naphthalene, C2-naphthalenes, C3-Naphthalenes, Acenaphthylene, Acenaphthene, Fluorene, Dibenzothiophene, Phenanthren, C1-phenanthrenes, C2-phenanthrenes, C3-Phenanthrenes, Anthracene, Benz(a)fluorine, Fluoranthene, Pyrene, 1-methylpyrene, Benz(a)anthracene, Chrysene/Triphenylene, Benz(b+j+k)fluoranthenes, Benz(e)pyrene, Benz(a)pyrene, Perylene, Indeno(1.2.3-cd)pyrene, Benzo(ghi)perylene, Dibenzo(ah)anthracene.

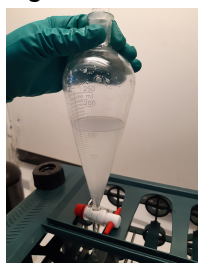
Table 7: Number of replicates and controls for the dispersion experiments.

Sample ID	Temp. °C	Sea water ml	Oil µl	Dispersant µl	V Extract ml	V DCM ml
WRD 1, 1:10*	2	120	100	10	50	20
WRD 2, 1:10	2	120	100	10	50	20
WRD 3, 1:10	2	120	100	10	50	20
WRD Control, A	2	120	100	0	50	20
WRD 1, 1:25	2	120	100	4	50	20
WRD 2, 1:25	2	120	100	4	50	20
WRD 3, 1:25	2	120	100	4	50	20
WRD Control, B	2	120	100	0	50	20
HDME50 1, 1:10	2	120	100	10	50	20
HDME50 2, 1:10	2	120	100	10	50	20
HDME50 3, 1:10	2	120	100	10	50	20
HDME50 A	2	120	100	0	50	20
HDME50 1, 1:10x2	2	120	100	10+10	50	20
HDME50 2, 1:10x2	2	120	100	10+10	50	20
HDME50 3, 1:10x2	2	120	100	10+10	50	20
HDME50 B	2	120	100	0	50	20
AGO 1, 1:10	2	120	100	10	50	20
AGO 2, 1:25	2	120	100	4	50	20
AGO Control, A	2	120	100	0	50	20
IFO 1, 1:10	2	120	100	10	50	20
IFO 2, 1:25	2	120	100	4	50	20
IFO Control, B	2	120	100	0	50	20

Sample ID	Temp.	Sea water	Oil	Dispersant	V Extract	V DCM
	°C	ml	µl	µl	ml	ml
Water	2	120	100	0	50	20
Water	2	120	100	0	50	20
WRD 1, 1:10	5	120	100	10	50	20
WRD 2, 1:10	5	120	100	10	50	20
WRD 3, 1:10	5	120	100	10	50	20
WRD Control, A	5	120	100	0	50	20
WRD 1, 1:25	5	120	100	4	50	20
WRD 2, 1:25	5	120	100	4	50	20
WRD 3, 1:25	5	120	100	4	50	20
WRD Control, B	5	120	100	0	50	20
HDME50 1, 1:10	5	120	100	10	50	20
HDME50 2, 1:10	5	120	100	10	50	20
HDME50 3, 1:10	5	120	100	10	50	20
HDME50 Control, A	5	120	100	0	50	20
HDME50 1, 1:10x2	5	120	100	10+10	50	20
HDME50 2, 1:10x2	5	120	100	10+10	50	20
HDME50 3, 1:10x2	5	120	100	10+10	50	20
Control, Water	5	120	0	0	50	20
Dispersant	5	120	0	10	50	20
Dispersant	5	120	0	4	50	20

Note: *Refilled with 70 ml seawater

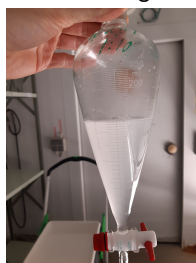
Figure 4: Photos of oil dispersant mixture just after mixing.



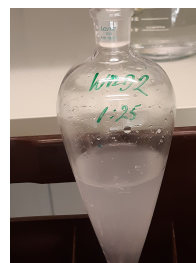
**WRD, 1:10
2°C**



**WRD, 1:25,
2°C**



**WRD, 1:10
5°C**



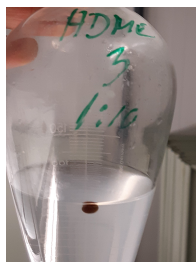
**WRD, 1:25,
5°C**



**HDME50,
1:10, 2°C**



**HDME50,
1:10x2, 2°C**



**HDME50,
1:10, 5°C**



**HDME50,
1:10x2, 5°C**



**AGO, 1:10, 2
°C**



IFO180, control, 1:10, 1:25



**AGO, 1:25, 2
°C**

3.3 Dispersion test results and discussions

As indicated above, the oil dispersed in the seawater was extracted with dichloromethane (DCM), and the content was measured using a fluorescence spectrophotometer. In addition, a number of samples were analysed for the content of polyaromatic hydrocarbons (PAHs) by GC-MS. PAHs are a group of components in oil which is of concern in relation to bioaccumulation and toxicity effects on aquatic organisms.

The dispersion effectiveness was calculated for each oil type, and the results are presented in Table 8. In Figure 5, the dispersion effectiveness for WRD and HDME is presented as average values ($n=3$). In Figure 6 and 7, the concentration of PAHs dispersed in the seawater and normalised PAH concentrations are given, respectively. The LSFOs are dominated by phenanthrenes, whereas AGO and IFO180 are dominated by naphthalenes. This difference is due to the composition of the original oils.

Table 8: Dispersion effectiveness (%).

Sample ID	Temperature [°C]	Oil added [μl]	Dispersant added [μl]	Dispersion effectiveness [%]
WRD 1, 1:10 ¹	2	100	10	11
WRD 2, 1:10	2	100	10	8
WRD 3, 1:10	2	100	10	16
WRD Control, 1:10	2	100	0	-1
WRD 1, 1:25	2	100	4	70
WRD 2, 1:25	2	100	4	1
WRD 3, 1:25	2	100	4	49
WRD Control, 1:25	2	100	0	-1
HDME50 1, 1:10	2	100	10	0.4
HDME50 2, 1:10	2	100	10	0.4
HDME50 3, 1:10	2	100	10	0.4
HDME50 Control, 1:10	2	100	0	-0.1
HDME50 1, 1:10x2	2	100	10+10	1.8
HDME50 2, 1:10x2	2	100	10+10	3.5
HDME50 3, 1:10x2	2	100	10+10	2.7
HDME50 Control, 1:10x2	2	100	0	-0.1
AGO 1, 1:10	2	100	10	78
AGO 2, 1:25	2	100	4	0 ²
AGO Control, 1:10	2	100	0	4
IFO 1, 1:10	2	100	10	0.53
IFO 2, 1:25	2	100	4	0.43
IFO Control, 1:10	2	100	0	-0.01
WRD 1, 1:10	5	100	10	55
WRD 2, 1:10	5	100	10	65
WRD 3, 1:10	5	100	10	27
WRD Control, 1:10	5	100	0	0
WRD 1, 1:25	5	100	4	36
WRD 2, 1:25	5	100	4	5
WRD 3, 1:25	5	100	4	36
WRD Control, 1:25	5	100	0	-1
HDME50 1, 1:10	5	100	10	1.1
HDME50 2, 1:10	5	100	10	1.5
HDME50 3, 1:10	5	100	10	1.8
HDME50 Control, 1:10	5	100	0	-0.1
HDME50 1, 1:10x2	5	100	10+10	5.3
HDME50 2, 1:10x2	5	100	10+10	6.5
HDME50 3, 1:10x2	5	100	10+10	5.2

Note:¹Refilled with 70 ml seawater.

²This result is most likely an error in the fluorescence measurements as the PAH analyses reveal a high content of dispersed PAHs.

Figure 5: Average dispersion effectiveness (%) for WRD (blue) and HDME50 (yellow) oils with standard error bars (n=3).

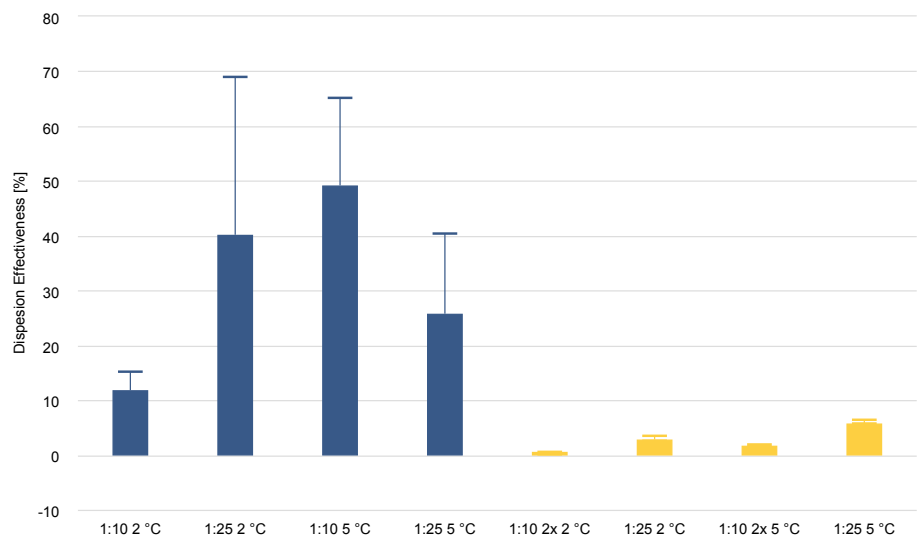


Figure 6: Concentration of PAHs (µg/l) in water samples after dispersion tests.

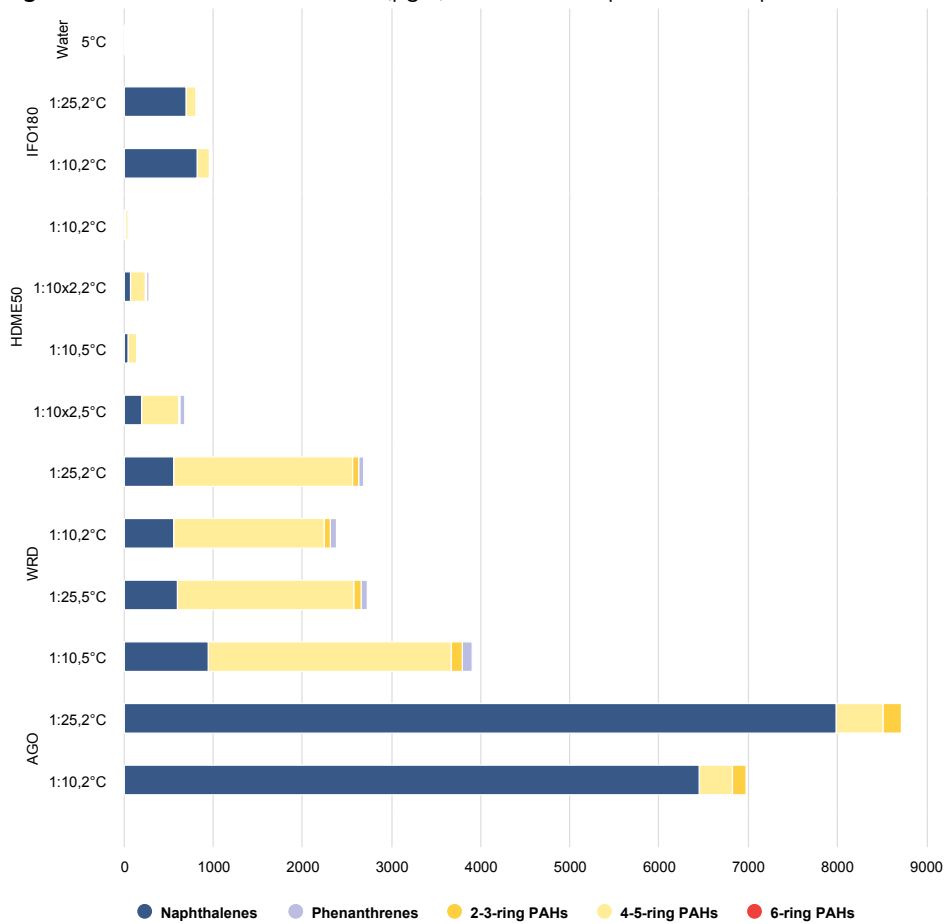
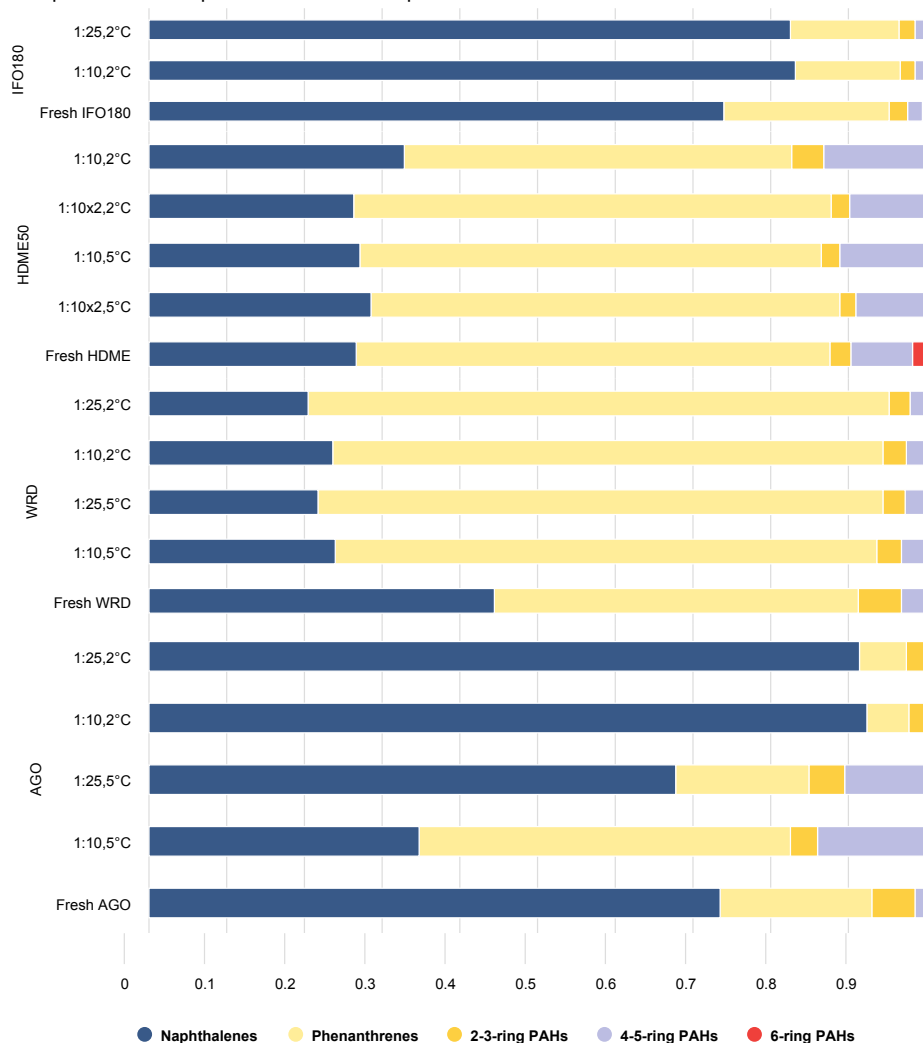


Figure 7: Relative distribution of PAHs (normalised to the sum of PAHs) in water samples from dispersion tests compared to fresh oils.



As shown in Figure 5, HDME50 is very poorly, if at all, dispersible. There is a slight increase in the dispersion effectiveness when successive dispersant application is included at raised temperature; thus, the dispersion effectiveness is slightly higher at 5°C than at 2°C. However, the effectiveness is generally very low. The results therefore suggest that HDME50 is not dispersible at the temperatures tested and not when treated with successive dispersant applications either.

Hellstrøm et al. (2017) also tested the dispersion effectiveness at bench-scale and found that HDME50 (50% emulsions at 13 °C) has reduced or poor dispersibility under calm weather conditions and reduced dispersibility under breaking wave conditions. In larger scale laboratory (meso-scale) testing, it was found that successive applications increased the dispersion effectiveness.

For WRD, the dispersion effectiveness was considerably higher than for HDME50; thus WRD was dispersible at 2 °C up to, on average, 40% (Figure 5). Somewhat surprisingly, for the 2°C experiments, the dispersion effectiveness of the DOR 1:25 experiments was higher than for DOR 1:10. There is a large variation between the replicates in the results for the DOR 1:25 2°C experiment. This may be due to the

challenges involved in securing contact between the oil and the dispersant. The oil appeared as a confined droplet on the water surface due to its high pour point and the low experimental test temperatures. Examining the funnels after the shaking process revealed that oil may stick to the inside of the glass wall (Figure 4). Even though we were careful to avoid such sticking, it is most likely the reason for the variations in the results. The effects of the above explanations may cause even larger variations due to the small scale of the experimental set-up and the low temperatures.

Hellstrøm et al. (2017) also tested WRD (named WRG, but assumed to be similar to WRD) and found that at 2 °C and relatively low mixing energy, dispersibility was low, whereas at relatively high mixing energy ("breaking waves") it was >50% and assessed as good (Hellstrøm et al. 2017).

We did not include ULSFO 2018 in our experiments. Results from other studies have shown that ULSFO 2018 is not dispersible at 2 °C due to its high pour point and thereby solidifying properties; and its dispersibility at 13 °C is thus low (Sørheim et al. 2020).

Different methods exist for testing dispersion effectiveness, where in particular mixing energy is a parameter and considered to influence the results. Various laboratory tests have found different effectiveness values also due to different test conditions (energy, sampling, static/dynamic etc.). Generally, it is assessed that small-scale test methods underestimate the dispersion effectiveness compared to large-scale test facilities (SL Ross 2011), which should be kept in mind when analysing the results of the present study.

The relative distribution of the PAHs in the water samples from the dispersion tests (Figure 7) shows that the PAH composition in water is similar to the composition in the fresh oil, indicating that the PAHs found in the water are mainly a result of dispersion and only to a minor degree dissolution. The PAHs in the water samples are dominated by naphthalenes and phenanthrenes. Naphthalenes and phenanthrenes are the PAHs with the highest water solubility, but they are also the dominant PAH components in the fresh oil. As for the oil types tested, the "old type" fuel oils (IFO180 and AGO) are dominated by naphthalenes, whereas the LSFOs are dominated by phenanthrenes. Hence, the concentration of PAHs dispersed in the water (Figure 6) reflects, to a large extent, the dispersion effectiveness.

3.4 Summary of the dispersion tests of LSFOs

Based on the small-scale dispersion tests on non-weathered LSFOs, WRD and HDME50, it was found that:

- WRD was somewhat dispersible (up to an average of DE 50%), the highest values being found for DOR 1:10 and at 5 °C.
- HDME50 was poorly dispersible even in the case of successive dispersant applications.
- Increasing the test temperature from 2 to 5 °C had a positive impact on the dispersion effectiveness. For WRD DOR 1:10, the effectiveness increased from < 20% to > 50% dispersion effectiveness.

- The concentration of PAHs dispersed in the water was associated with dispersion effectiveness. The PAHs were dominated by the naphthalenes and phenanthrenes, the PAHs with the highest water solubility, but also by the dominant PAH components in the fresh oil (Figure 7).

4. Burn tests

4.1 Experimental set-up

The laboratory burn tests were based on a set-up developed for small-scale in-situ burning tests (Fritt-Rasmussen & Brandvik 2011).

Burn efficiency, mass burning rate/regressions rate and possible sinking of burn residue were determined in small-scale in-situ burning tests for two LSFOs – WRD and ULSFO 2018 – and, for the purpose of comparison, an Arctic diesel (AGO) was included.

4.1.1 Burn procedure

The burn tests were completed in open air, and the wind was between 1–5 m/s during the test period. Air temperatures were measured during the burn experiments and are given in Table 9.

The burn tests were performed in Pyrex glass containers (diameter = 19 cm, area = 0.028 m²) with seawater (30.3 psu) and placed in the middle of a water bath of 100 cm x 100 cm and 20 cm high for cooling. In the Pyrex glass container, 11 shale tiles (2.5 x 2.5 cm) were added to the bottom to prevent the glass from floating. The oil sample was carefully added to the seawater surface of the Pyrex glass container. The oil was ignited by a butane torch up to 3 times of 10 s, if necessary. After flame out, the glass container was placed on a table for studying the possible sinking of the residue after cooling. To be able to calculate the efficiency of the burn (see 4.1.2), the weight of the oil before the burn and the weight of the residue after the burn were measured on a scale. Although sinking was not expected for the tested oil types with densities between 0.8 and 0.92 g/ml for each experiment, a control was included where the oil was not ignited but left on the water surface to allow sinking. The key parameter for the small-scale burns can be found in Table 9.

The residues were analysed for THCs (total hydro carbons) and PAHs.

For THC analyses: All the samples were weighed, 350 mg, and 10 ml DCM/pentane was added, 1:1. Shaken until all the oil had dissolved. Purified on SPE: Strata®FL-PR Florisil 2g/12 mL, Phenomenex, (10 ml MEOH, 10 ml pentan, 1000 µl sample, 20 ml DCM/pentan, 50/50). The samples were transferred to GC-vials and internal standard added (squalane), and the samples were then analysed on GC-FID as follows: GC Column HP db5, 0,25 µm. Column length: 20 m, diameter: 0.18 mm, film thickness: 0.18 µm. Temperature programme: 35°C 0.5 min; 7.5°C/min to 110°C; 10°C/min to 260°, 13°C/min to 325°C, 325°C 5 min.

For PAH analyses: All the oils were weighed, 350 mg, and 10 ml DCM/pentane was added, 1:1. Shaken until all the oil had dissolved. The dissolved oil was analysed directly on GC-MS, no internal standards were added. GC-MS: Analysis in SIM mode. Column: HP-5MS, length: 30 m, diameter: 0,250 mm, film thickness: 0,25 µm. Temperature programme: 90°C 1 min, 7°C/min to 240°C, 240°C 4 min, 20°C/min to 270°C, 270°C 18.5 min. The PAH target compounds measured: Naphthalene,

C1-naphthalene, C2-naphthalenes, C3-Naphthalenes, Acenaphthylene, Acenaphthene, Fluorene, Dibenzothiophene, Phenanthren, C1-phenanthrenes, C2-phenanthrenes, C3-Phenanthrenes, Anthracene, Benz(a)fluorine, Fluoranthene, Pyrene, 1-methylpyrene, Benz(a)anthracene, Chrysene/Triphenylene, Benz(b+j+k)fluoranthenes, Benz(e)pyrene, Benz(a)pyrene, Perylene, Indeno(1.2.3-cd)pyrene, Benzo(ghi)perylene, Dibenzo(ah)anthracene.

Table 9: Key parameters of the experimental small-scale burns.

Burn ID	Date	Oil type	Total oil amount [g]	Initial oil film thickness [mm]	Air temperature [°C]	Water temperature [°C]	Ignition [no. of 10 sec attempt]
WRD_20_1	27.04.21	WRD	439	20	11.2	12.6	2
WRD_20_2	27.04.21	WRD	510	20	10	21	2
WRD_20_3	27.04.21	WRD	510	20		27	2
WRD-20-control	27.04.21	WRD		20			NO
WRD_10_1	27.04.21	WRD	259	10	13	25	2
WRD_10_2	27.04.21	WRD	275	10	12.7	25	2
WRD_10_3	27.04.21	WRD	270	10		25	2
WRD-10-control	27.04.21	WRD		10			NO
ULSFO_20_1	28.04.21	ULSFO 2018	510	20	12.6	14.5	2
ULSFO_20_2	28.04.21	ULSFO 2018	508	20		14.5	2
ULSFO_20_3	28.04.21	ULSFO 2018	509	20	11	21	2
ULSFO-20-control	28.04.21	ULSFO 2018		20			NO
ULSFO_10_1	28.04.21	ULSFO 2018	257	10	11	22	2
ULSFO_10_2	28.04.21	ULSFO 2018	223	10	12.2		2
ULSFO_10_3	28.04.21	ULSFO 2018	253	10	12.2		2
ULSFO-10-control	28.04.21	ULSFO 2018		10			NO
AGO_20_1	28.04.21	AGO	439	20	12		1
AGO_20_2	28.04.21	AGO	423	20	12	21	1
AGO_20_3	28.04.21	AGO	449	20	10	24	1
AGO_20_4	28.04.21	AGO	219	10	11	22	1
AGO_20-control	28.04.21	AGO		20			NO

4.1.2 Calculations of burn efficiency and mass burning rate/regressions rate

Burn efficiency and regression rates were calculated for all burns and are presented in Table 11.

Burn efficiency (BE%) is a gravimetric estimation of the amount of oil consumed during the burn and was calculated from the initial oil amount and burn residue (Table 9):

$$BE \left[\% \right] = 100 \left(1 - m_f / m_o \right) \quad \text{Equation 4}$$

m_o amount of the initial amount of oil; m_f mass of oil residue

The regression rate, which is the oil thickness reduction with time, is another often used measure to express the burning efficiency (SL Ross 1998):

$$\dot{r} \left[mm / min \right] = \frac{\dot{m}}{A\rho_o} \quad \text{Equation 5}$$

ρ_o is the initial oil density of 0.850 g/cm³, A is the oil slick area, $\dot{m}$ is the mass burning rate defined as the mass lost per unit time of burning.

Here we calculate the overall mass burning rate:

$$\dot{m}_{global} \left[g / s \right] = \frac{(m_o - m_f)}{t_f} \quad \text{Equation 6}$$

t_f time of burn, m_o mass of initial amount of oil, m_f mass of oil residues.

4.2 Burn test results and discussions

The results from the burn experiments are shown in Table 10, and photos of the different oil type and their residues can be seen in Figure 8.

Generally, for all the oils, they ignited and burned after one or two attempts. The burning efficiency (i.e. how much oil was removed by the burn) was above 50% in all cases. The wind conditions were overall calm, but on day 2 (28.04.21) some minor casts occasionally occurred. This resulted in bent flames but not blowout, and the wind conditions somewhat supported the burn by securing oxygen to the combustion process.

After flame-out, the burn residues were kept on the water surface for investigation of potential sinking of residue after cooling to ambient temperatures. None of the burn residues sank as can be seen from Figure 8.

The WRD oils had the highest burn efficiencies for the 20 mm oil slicks (Table 10). The 20 mm WRD oil slicks burned for a relatively longer time than the other oils, which is also reflected by the lower regression rates compared to the 10 mm WRD oil slick experiments. The 10 mm WRD oil slick experiments only burned for around 10 minutes, yielding high regression rates and only slightly lower burning efficiencies.

The ULSFO 2018 oils were the most difficult to ignite and required two ignition attempts. For the burns of the ULSFO 2018 10 mm slick, each burn ended in a boil-over phase, which expectedly is the reason for the higher burning efficiencies of the 10 mm compared to 20 mm burns (Table 10). During the boil-over, oil droplets were also ejected outside of the glass container. This oil/residue was collected and included in the total amount of residue. The amount of oil/residue ejected as a result of boil-over constituted approximately 50 g.

Overall, it seems that the combustion of WRD is more efficient than that of ULSFO 2018 for the 20 mm oil slick experiments. The burn times are comparable, but the burn efficiency and the regression rate are lower for the ULSFO 2018 than the WRD experiments (Table 10). For the 10 mm oil slick experiments, the differences between the burning efficiencies for the WRD and ULSFO 2018 experiments are smaller, likely due to a boil-over phase resulting in an increase in the burn efficiency. However, the WRD 10 mm oil slicks burned for a shorter period of time, resulting in a higher regression rate, which supports the suggestion that the WRD combustion is more efficient than the ULSFO 2018 combustion. All of the four AGO 20 mm oil slick burns had very high burn efficiencies as well as regression rates, leaving only an oil sheen.

Sørheim et al. (2020) and Hellstrøm et al. (2017) studied the ignitability of HDME50, WRG and ULSFO 2018. WRG outdoor meso-scale burns had low burning efficiencies, whereas WRG did not ignite in the bench-scale tests. Taking into consideration the differences in experimental set-up and potential variations between oil batches (physical and chemical variations), the results were comparable for ULSFO 2018.

Table 10: Results from the experimental small-scale burns.

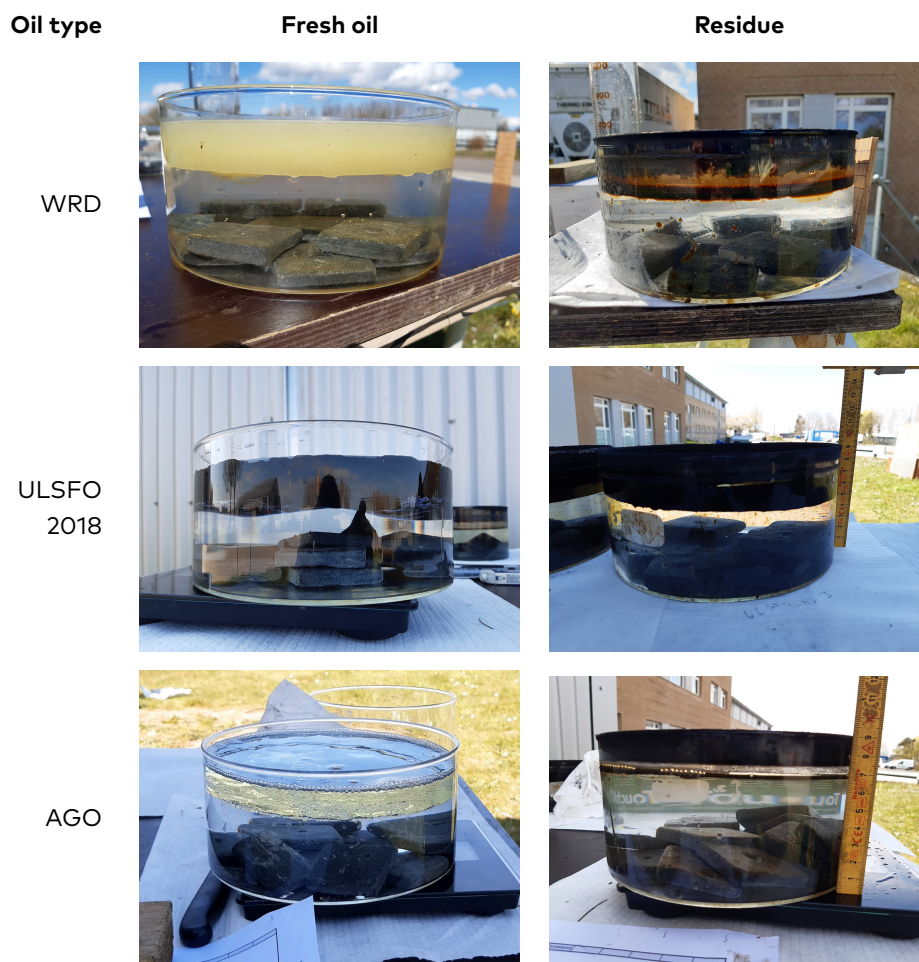
Burn ID	Burn time [min]	Total residue mass [g]	Burn efficiency [%]	Regression rate [mm/min]	Residue sinking	Boil-over ¹
WRD_20_1	00:29:50	65	85	0.52	No	Yes
WRD_20_2	00:31:40	75	85	0.57	No	Yes
WRD_20_3	00:32:40	80	84	0.57	No	Yes
WRD_10_1	00:09:55	68	74	0.79	No	Yes
WRD_10_2	00:08:55	75	73	0.92	No	Yes
WRD_10_3	00:10:45	89	67	0.68	No	Yes
ULSFO_20_1	00:27:35	257	50	0.38	No	No
ULSFO_20_2	00:28:27	253	50	0.37	No	No
ULSFO_20_3	00:27:37	243	52	0.40	No	No
ULSFO_10_1	00:13:01	68	74	0.60	No	Yes ²
ULSFO_10_2	00:14:55	75	66	0.41	No	Yes ²
ULSFO_10_3	00:12:36	71	72	0.60	No	Yes ²
AGO_20_1	00:21:32	23	95	0.80	No	Yes
AGO_20_2	00:19:30	23	95	0.85	No	Yes
AGO_20_3	00:16:57	32	93	1.02	No	Yes
AGO_20_4	00:10:11	1	100	0.90	No	Yes

Note: All WRD experiments were performed on 7 April 2021 and all ULSFO 2018 and AGO experiments on 28 April 2022.

¹⁾ Oil/residue ejected outside the glass container was collected and included in the total amount of residue.

²⁾ The boil-over was significant for the ULSFO 2018 10 mm experiments. Oil/residue ejected outside the glass container was collected and amounted to approximately 50 g.

Figure 8: Photos of the three oils included in the burn experiments and their burn residue.

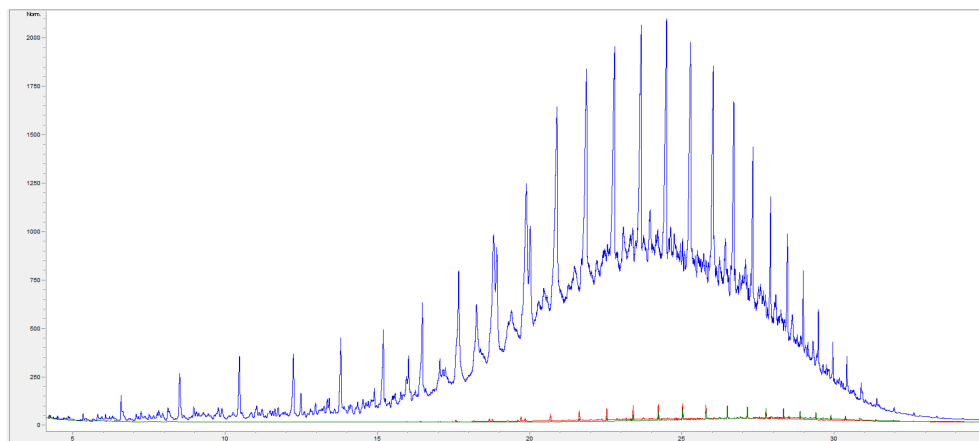


Chromatograms of fresh oils and corresponding burn residue are shown in Figure 9 and Figure 10. Note that the fresh oils in Figure 10 were diluted to ease comparison of the chromatograms. The chromatograms show n-alkanes as systematic narrow peaks where the first peaks represent the compounds with the lowest boiling point. More complex components are shown as unclear bumps below the peaks. The high burning efficiencies of all the experiments (from 50–100%, Table 10), in particular the AGO and the WRD experiments, can be observed in the chromatograms as a marked removal of the oil components (Figure 9). Lighter compounds up to nC15 for AGO and to nC16 for WRD are totally removed. For ULSFO 2018 (20 mm) for which the burn did not end by boil-over, more low components are found in the residue. In general, for all three oil types, there is a shift towards a higher content of the heavier compounds after combustion (Figure 10). The higher burn efficiency for the WRD 20 mm oil slick compared to the WRD 10 mm oil slick is also reflected in the chromatograms (Figure 10) as a slightly more pronounced shift towards a higher content of heavier oil compounds.

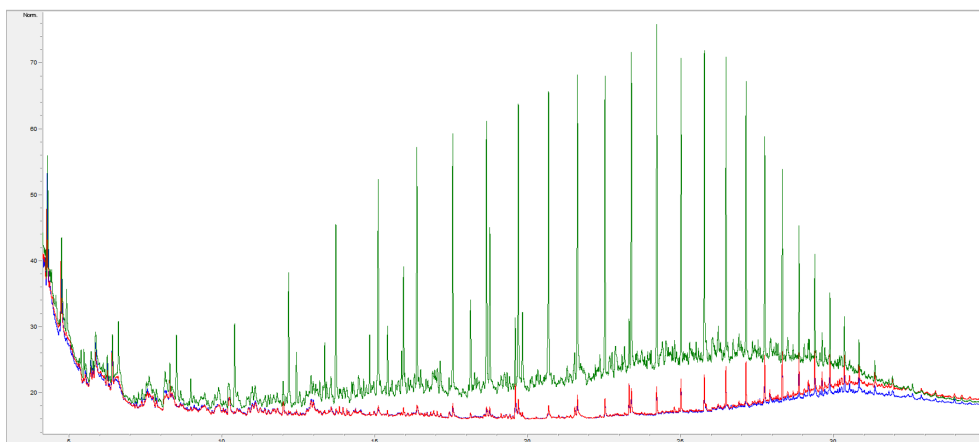
The relative composition of PAHs (normalised to the sum of PAHs) is exhibited in Figure 11. In general, burning resulted in a reduction of naphthalenes and phenanthrenes but a relative increase of 4-6-ring numbered PAHs when comparing

the analyses of fresh oil samples with the burn residue samples. This is most likely due to production of PAHs during the burn (pyrogenic PAHs) as also found in other studies (Fritt-Rasmussen et al. 2013; Garrett et al. 2000; Wang et al. 1999). In Figure 12, the burn efficiency is multiplied to the composition of PAHs, highlighting that the total amount of PAHs is reduced by the burn.

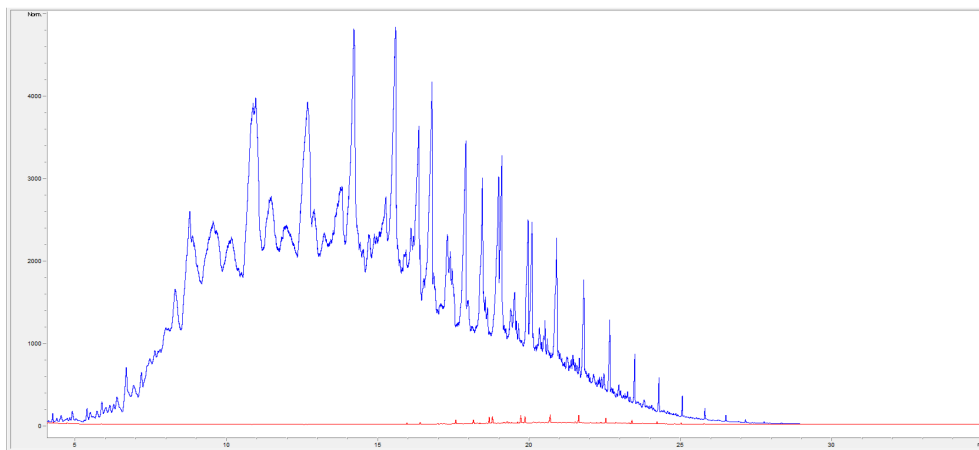
Figure 9: GC-Chromatograms of burn residue and corresponding fresh oils. Detailed information on the burn residues is displayed in the GC-Fid chromatograms in Figure 10.



Note: WRD fresh (blue), WRD 10 mm (red) and WRD 20 mm (green).

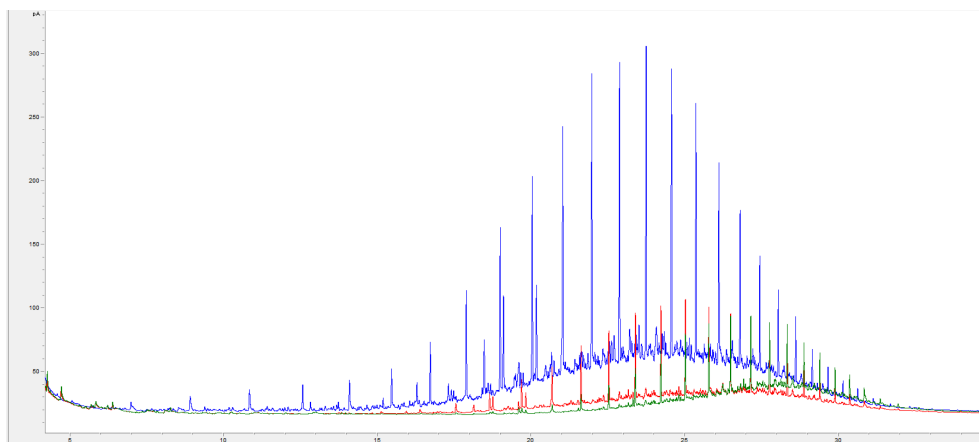


Note: ULSFO 2018 fresh (green), ULSFO 2018 10 mm (blue) and ULSFO 2018 20 mm (red). The first 7 minutes represent unknown impurities in the solvent and not part of the oil/residue.

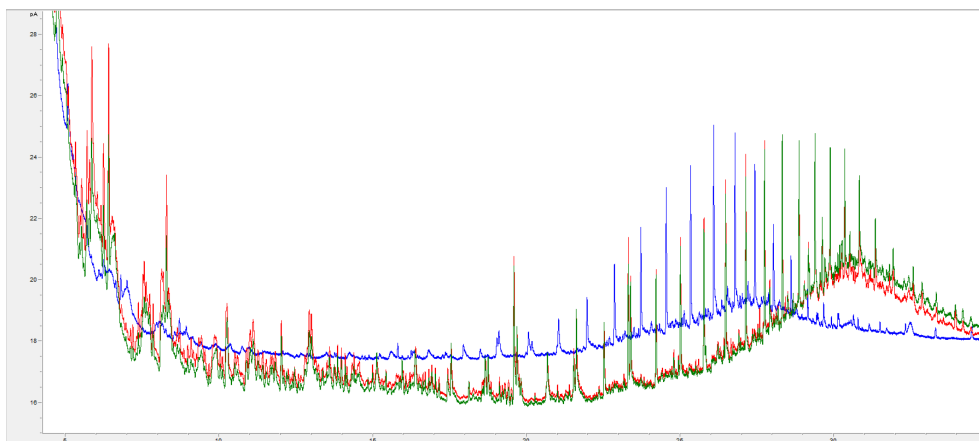


Note: AGO fresh (blue) and AGO 20 mm (red). The AGO fresh (blue) chromatogram appears 'overloaded' but is included to illustrate the removal of the residue (red).

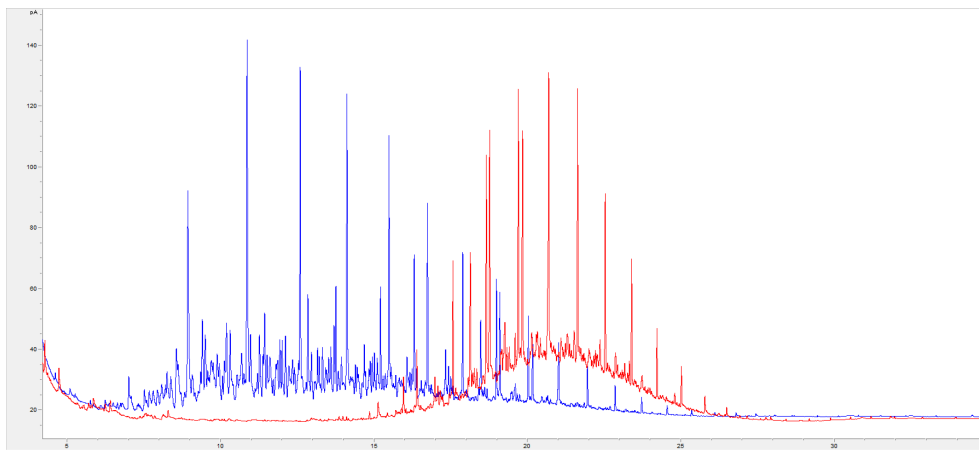
Figure 10: GC-Chromatograms of burn residue and corresponding fresh oils. The fresh oils have been diluted to allow a relative comparison of the chromatograms.



Note: WRD fresh (blue) (FF=10x), WRD 10 mm (red) and WRD 20 mm (green).



Note: ULSFO 2018 fresh (blue) (FF=10x), ULSFO 2018 10 mm (red) and ULSFO 2018 20 mm (green). For the first part of the chromatograms up to 7 minutes represent unknown impurities in the solvent and not part of the oil.



Note: AGO fresh (blue) (FF=100x) and AGO 20 mm (red).

Figure 11: Relative composition of PAHs (normalised to the sum of PAHs) for fresh oil samples and residue samples.

Note: Note that the group "2-3-ring PAHs" is without naphthalenes and phenanthrenes. These are shown separately due to their high proportions.

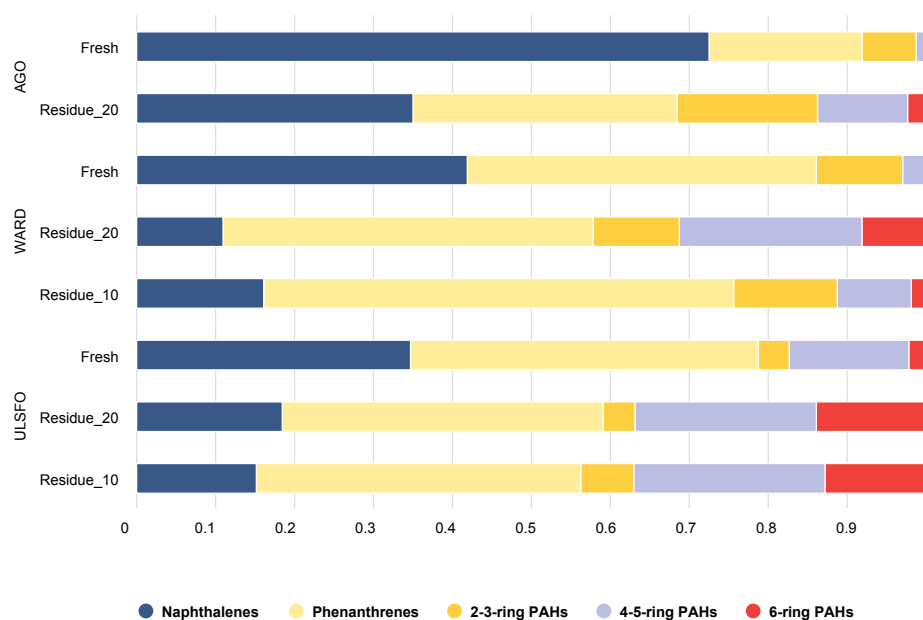
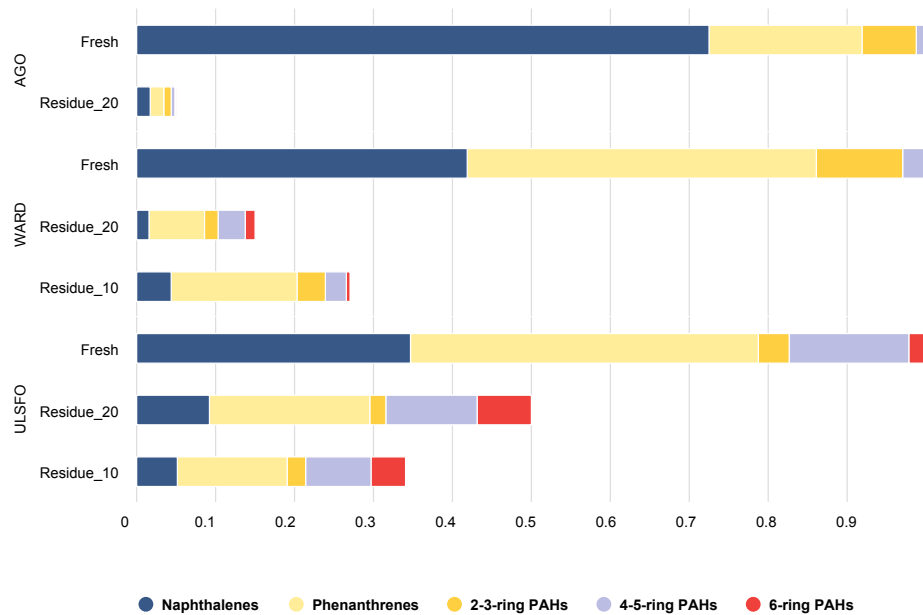


Figure 12: Composition of PAHs (normalised to the sum of PAHs and multiplied with the burning efficiency (%)) for fresh oil samples and residue samples.

Note: Note that the group "2-3-ring PAHs" is without naphthalenes and phenanthrenes; these are shown separately due to their high proportions.



4.3 Summary of burn tests of LSFO

Based on the small-scale burn tests with two different non-weathered LSFOs, WRD and ULSFO 2018, it was found that:

- All of the oils were ignitable. However, ULSFO 2018 was the most difficult to ignite.
- The burn efficiencies for WRD were up to 85% and for ULSFO 2018 74%. The regressions rates were 0.92 mm/min and 0.6 mm/min for WRD and ULSFO 2018, respectively.
- None of the burn residues sank after cooling to ambient temperatures.
- There was an increase in the 4-6-ring PAHs in all residues, most likely due to pyrogenic production.

5. Biodegradation tests

5.1 Biodegradation test set-up

Biodegradation of low sulphur fuel oils and dispersed low sulphur fuel oils was investigated in cold climate microcosm experiments. The microcosm experiments consisted of 1 L glass bottles containing seawater and oil immobilised in absorbent fabric. Immobilising oil in absorbent fabric mimics microbial oil–seawater interface processes in natural seawater (Lofthus et al. 2018). Sterile treatments were included to compare biotic and abiotic degradation and contained sterilised seawater. The protocol is based on methodology used by SINTEF (2020, unpublished), and a similar method was used by Brakstadt and Bonaunet (2006) and Lofthus et al. (2018).

Hence, in the first experiment, biodegradation of WRD was studied with application of dispersant spiked to the fresh oil (without absorbent fabric). In the second experiment, biodegradation of WRD and ULSFO 2018 oils (Figure 14) was studied without dispersant (Table 11). Seawater for the first experiment was sampled in May 2021 on-board RV Aranda at 1 m depth from the JML sampling station (59°34.91' 23°37.61') located in the Gulf of Finland. Seawater for the second experiment was sampled in August 2021 from the Vuosaari boat harbour (60°11.93', 025°08.43') at 0.5–1 m depth. The sampled seawater was stored at 5 °C until initiation of the experiments.

Table 11: Number of replicates and controls in the biodegradation experiments.

Sample ID	Number of replicates	Temp. °C	Seawater origin	Oil applied* µl	Absorbent fabric	Dispersant DOR
ULSFO 0h	3	5	Vuosaari (Coastal)	100	Yes	-
ULSFO 14d	3	5	Vuosaari (Coastal)	100	Yes	-
ULSFO 28d	3	5	Vuosaari (Coastal)	100	Yes	-
ULSFO 0h sterile control	3	5	Vuosaari (Coastal)	100	Yes	-
ULSFO 28d sterile control	3	5	Vuosaari (Coastal)	100	Yes	-
WRD 0h	3	5	Vuosaari (Coastal)	100	Yes	-
WRD 14d	3	5	Vuosaari (Coastal)	100	Yes	-
WRD 28d	3	5	Vuosaari (Coastal)	100	Yes	-
WRD 0h sterile control	3	5	Vuosaari (Coastal)	100	Yes	-
WRD 28d sterile control	3	5	Vuosaari (Coastal)	100	Yes	-
WRD oh dispersant	3	5	JML (Open sea)	2	No	1:25
WRD 14d dispersant	3	5	JML (Open sea)	2	No	1:25
WRD 28d dispersant	3	5	JML (Open sea)	2	No	1:25
WRD 0h dispersant sterile ctrl	3	5	JML (Open sea)	2	No	1:25
WRD 28d dispersant sterile ctrl	3	5	JML (Open sea)	2	No	1:25

*Note: *per petri dish in absorbent fabric containing treatments, per bottle in treatments with dispersant.*

First, 100 µl of each oil was applied to the surface of a petri plate containing 15 ml autoclaved deionised water (pre-heated in an oven to 60 °C) and heated at 60 °C for 30 minutes (Figure 13). After application, the oil generated a thin film on the surface of the water. Prior to application, ULSFO oil was heated for 30 min at 80 °C in a heating block to decrease viscosity and allow pipetting. WRD diesel was applied at room temperature.

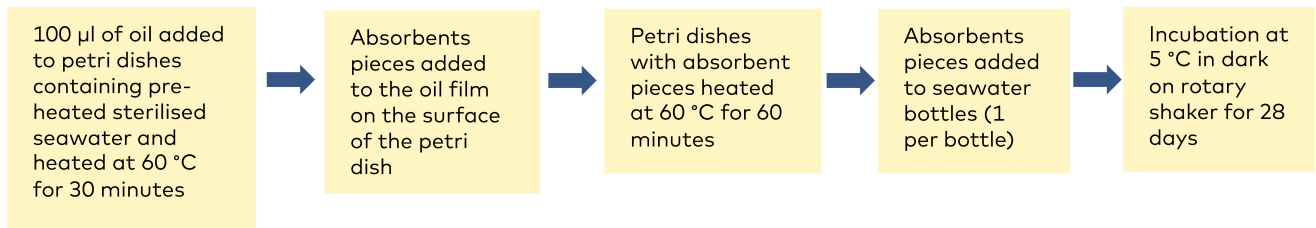
The oil film was immobilised on 1 cm² (1 x 1 cm) pieces of absorbent fabric (Sefar Inc., Tal; Switzerland; 09-150/36). The fabric squares were washed in dichloromethane and rinsed in sterile seawater before being placed on the oil film. A Maximum 15 absorbent pieces were added to one petri dish, and the petri dishes were kept at 60 °C for 60 min. The absorbent pieces were then removed from the petri dish (see Figure 15) and rinsed with sterile seawater twice to remove excess oil before being transferred into 1 L glass bottles filled with normal or sterile seawater. One absorbent piece was added per bottle and typically sank to the bottom. The

experimental bottles were then placed on an orbital shaker and incubated in the dark at 5 °C. No oil droplets were observed in the ULSFO treatment bottles. Absorbent fabric was used to obtain more reliable replicates and because the method prevents absorption of free oil to the glass surfaces of the bottles.

WRD oil was also applied with dispersant Finasol OSR 52 to assess the degradation of dispersed oil. Dispersant was added to the oil at a ratio of 1:25 (dispersant:oil) in an Eppendorf tube, mixed by inverting the tube by hand and heated in an oven at 60 °C for 30 min. Then 2 µl dispersant and oil mixture was added directly to the seawater 1 L glass bottles, and the bottles were incubated in the dark at 5 °C on an orbital shaker. No visible oil droplets were observed on the surface of the WRD dispersant in the treatment bottles during the experiment.

Figure 13: Flow-chart of the experimental set-up.

Oil treatment



Dispersant treatment

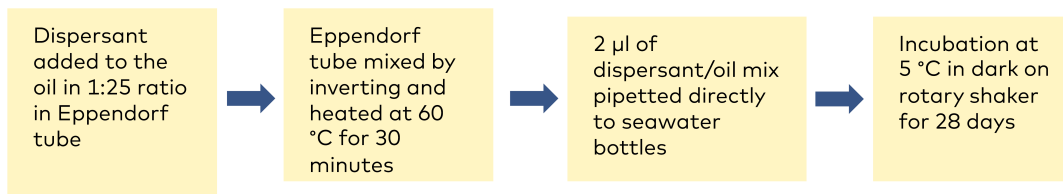
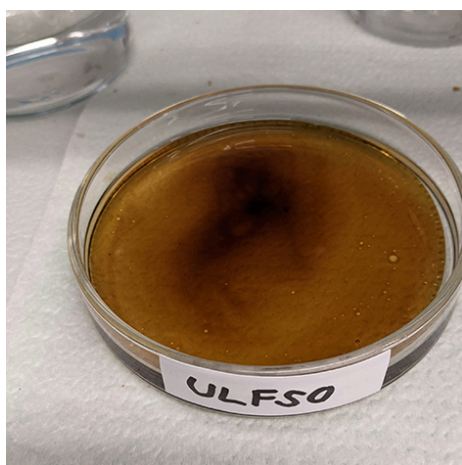


Figure 14: Oils used in the biodegradation tests. Left WRD and right ULSFO 2018.



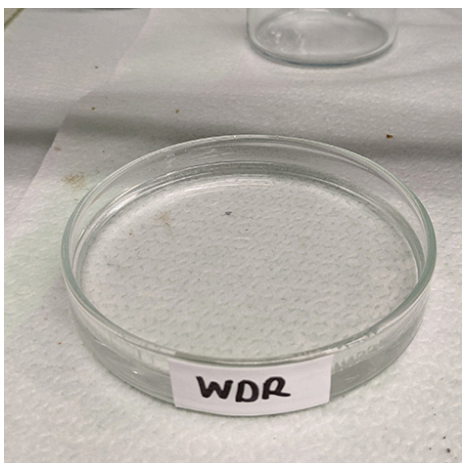
Figure 15: Preparation of oil immobilisation on adsorbent pieces in petri dishes in the experiment.



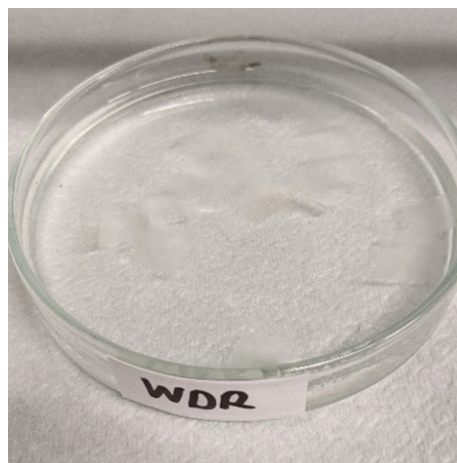
ULFSO 2018 before application of adsorbent pieces.



ULFSO 2018 after adsorbent application and incubation.



WDR before application of absorbent pieces.



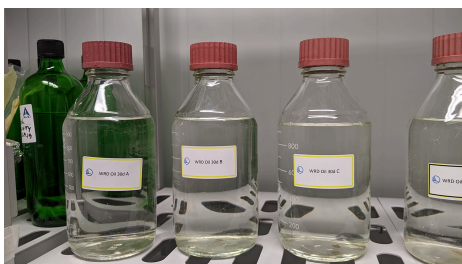
WDR after absorbent application and incubation.

5.2 Chemical analysis

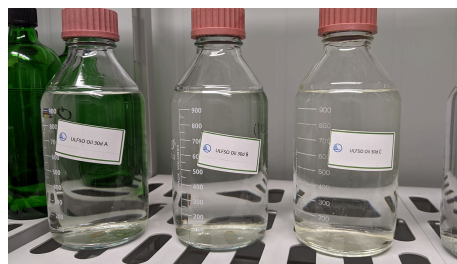
During both experiments, microcosm bottles were sacrificed for chemical analysis at three time points 0h, 14d and 28d (see Figure 16). Biodegradation rates during the experiment were assessed by analysing concentrations of petroleum hydrocarbon C_{10} – C_{40} in the water phases and in extracts from the fabric absorbent squares by analysing the whole contents of the bottle, including the fabric pieces.

The chemical analyses were done by an external accredited laboratory using the internal GC-MS modified method SFS-EN ISO 9377-2 (hydrocarbons C_{10} – C_{40}) (ISO, 2000). The uncertainty of measurement was 20% for concentrations above 0.5 mg/L.

Figure 16: Microcosm bottles at the end of the experiment. The adsorbent pieces have sunk to the bottom of the bottles, and there is no visible oil film on the water surface.



WRD bottles after 30d.



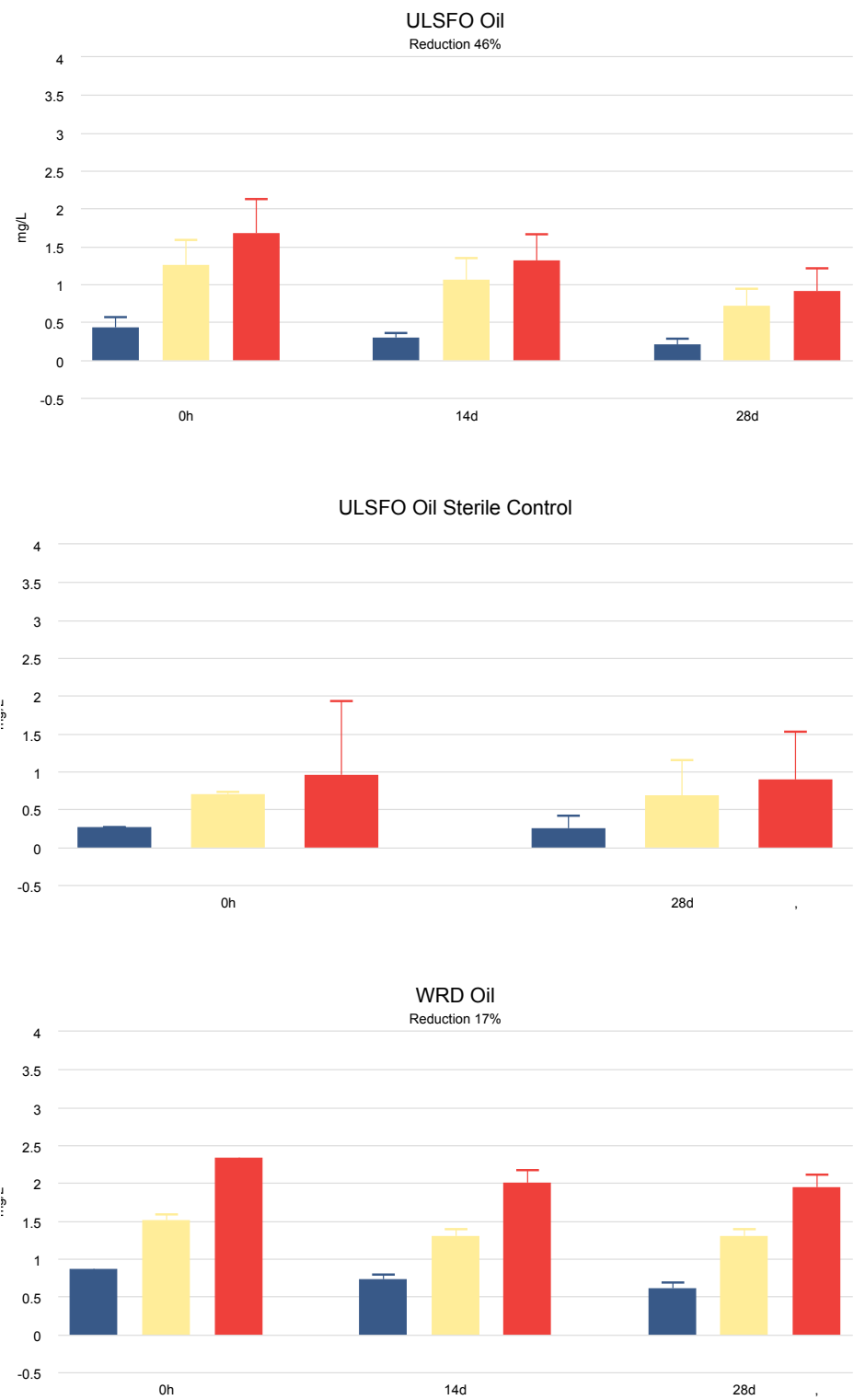
ULSFO 2018 bottles after 30d.

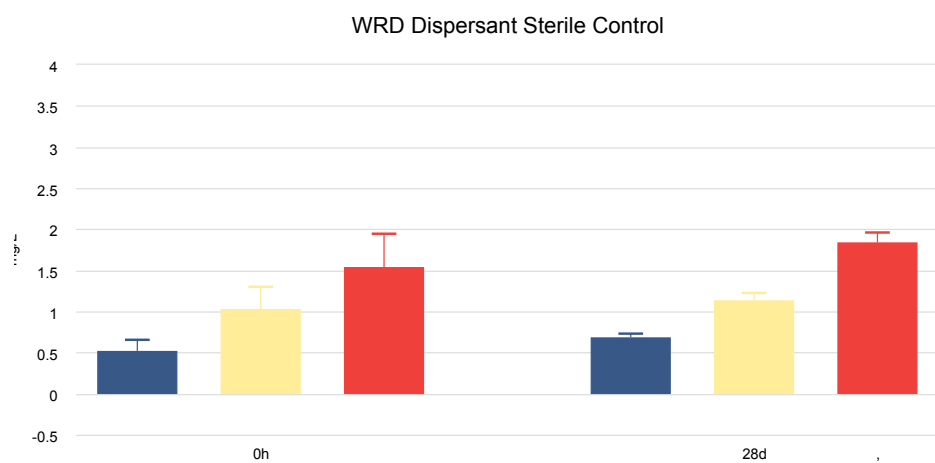
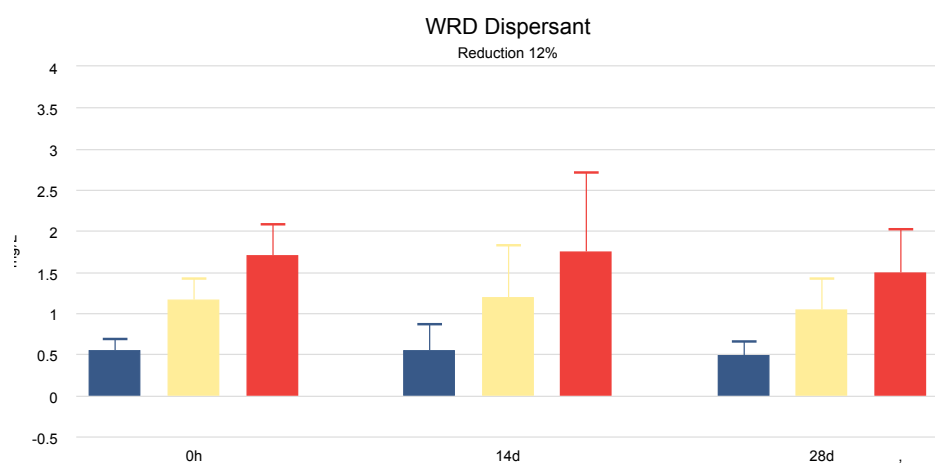
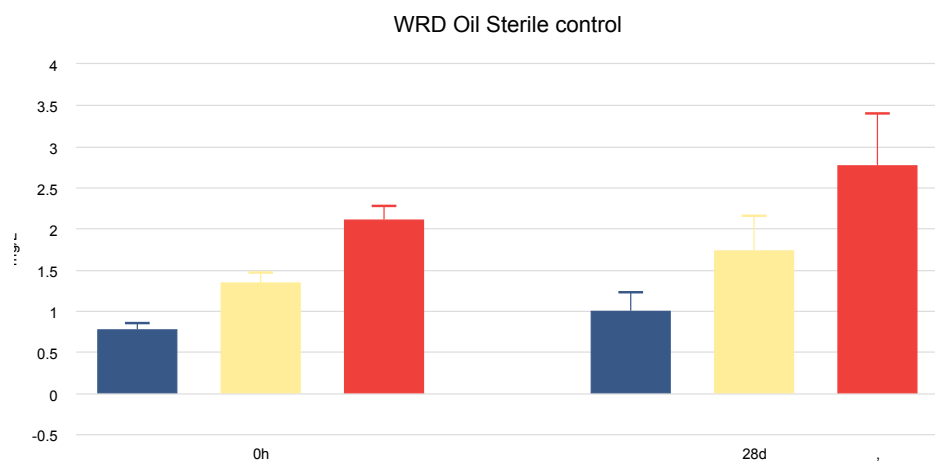
5.3 Biodegradation test results and discussions

The results from the chemical analysis (hydrocarbon fractions $C_{10}-C_{40}$) are shown in Figure 17. For both ULSFO and WRD, $C_{10}-C_{40}$ concentrations decreased during the experiment. Without dispersant addition, ULSFO decreased from 1.7 mg/L to 0.9 mg/L (46%), and WRD decreased from 2.3 mg/L to 1.9 mg/L (17%) after 28 days based on the average of three replicates. The lighter fractions, $C_{10}-C_{20}$, decreased more than the heavier fractions, $C_{20}-C_{40}$, for both oils. These rates are comparable to or slightly lower than degradation rates earlier reported for heavy fuel oil at low temperatures (Brown et al. 2016, Venosa and Holder, 2007, Fritt-Rasmussen et al. 2018). Sterile control treatments for both oils showed a slight or no decrease, suggesting low abiotic degradation of the oil during the experiment. Dispersed WRD oil exhibited a relatively slighter decrease of $C_{10}-C_{40}$ from 1.7 to 1.49 mg/L (12%) compared to untreated oil. There might have been an effect of the seawater used for WRD with and without dispersant, as it was derived at different times and locations and thus may differ slightly in nutrient concentrations. The sterilised dispersed WRD treatment demonstrated a slight increase of $C_{10}-C_{40}$ at the end of the experiment. A possible explanation of this is that the mixture of dispersant and dispersed oil has a toxic effect on the microbes, leading to a reduction of the degradation rate.

Overall, both ULSFO and WRD showed relatively slow biodegradation in 5 °C Baltic Sea seawater during the duration of the experiment compared to degradation rates earlier reported for heavy fuel oil at low temperatures. Due to the large variation between replicates, the observed decrease was not statistically significant. Even though the results with and without dispersant cannot be directly compared due to the different experimental setup, which was necessary in order to perform the test. However, the decreasing trend was visible both in ULSFO and WRD. From the rate of the biodegradation, it seems that use of dispersant with WRD oil did not increase degradation. Similar results have been obtained in studies with dispersed crude oils, where also a possible toxic effect of the dispersed crude oils on microbes could be observed (Tonteri et al. 2017).

Figure 17: Petroleum hydrocarbon (C10–C40) concentrations in the experimental bottles during the laboratory experiments. The error bars indicate the standard deviation of three replicates.





● Fractions C10–C21 ● Fractions C21–C40 ● Fractions C10–C40

5.4 Summary of biodegradation tests with LSFOs

Based on the biodegradation tests of WRD and ULSFO 2018, it was found that:

- ULSFO 2018 and WRD had degraded 46% and 17% after 28 days.
- An improved method for adding high pour point oils was tested.
- Dispersant addition did not increase biodegradation.
- Dispersant addition inhibited biodegradation, possibly due to toxic effects on microbes.

6. Environmental assessment of potential impact and identification of key organisms at risk

Several factors may influence the fate and behaviour of an oil spill in seawater and, consequently, the potential exposure of organisms to the oil and its impact on these in the marine environment. Besides air and seawater temperature, the physical and chemical properties of different oil types and the derived compounds from a spill response operation are pivotal. A high content of water-soluble compounds and natural/chemical dispersion may increase the likelihood of exposure of organisms in the water column (e.g. phyto- and zooplankton and fish) to the oil with subsequent adverse impacts. A low degree of natural/chemical dispersion, a low density, a high content of asphaltenes and paraffins and a high viscosity and pour point suggest an oil with a high degree of persistence on the sea surface that may increase the likelihood of exposure to and impact of oil on organisms associated with the seawater surface and the tidal zone (e.g. birds and marine mammals as well as benthic organisms in the tidal zone). The test oils had densities < 1 g/ml and would thus float on the water surface. High viscosity and high pour point are more important properties for the persistence of the oils than their densities.

6.1 Fate of LSFO spills and derived effect of in situ burning and chemical dispersion of an LSFO spill

In this project, a number of small-scale tests on in situ burning and chemical dispersion of an LSFO spill were performed as well as studies of the potential of biodegradation (of oil and chemically dispersed oil). The results from the tests are, along with other relevant literature values, summarised in the following.

6.1.1 In-situ burning, response possibilities and fate of burn residue

Our small-scale laboratory studies demonstrated that all the oils tested (fresh oils: WRD, ULSFO 2018) were ignitable, with burning efficiencies >50%. None of the burn residues sank, even after cooling. Hence, the potential environmental impacts of a successful burn of the tested LSFOs are expected to be associated with the smoke plume and the remaining residue in the environmental compartments, such as the sea surface, and/or if residue reaches shorelines and to organisms associated with the sea surface and the intertidal zone.

The oils tested were fresh and water-free LSFOs. However, in the sea, oil weathers and its properties change within a relatively short time, implying that the oil becomes more difficult to ignite, primarily due to uptake of water (water-in-oil

emulsification) and evaporation of volatile compounds (Fritt-Rasmussen and Brandvik 2011). In particular for ULSFO 2018, the oil was most difficult to ignite, the window of opportunity for burning is expectedly short (hours/a few days).

Sørheim et al. (2020) and Hellstrøm (2017) concluded that all their tested water-free LSFOs were ignitable, but their potential use for in-situ burning might be limited due to low contents of volatiles and thus a prolonged ignition time. For emulsified oils, Sørheim et al. (2020) observed that ignition was not possible without using a significant amount of primers (e.g. diesel). They visually observed the residue after it had cooled down after the burn and found that it did not sink.

6.1.2 Chemical dispersion, response possibilities and fate of dispersed oil

The small-scale dispersion tests with non-weathered LSFOs, WRD and HDME50 found that WRD was somewhat dispersible and highest for DOR 1:10 and at 5 °C. Contrary to this, HDME50 was poorly dispersible even in successive dispersant applications. The tests were conducted at 2 and 5 °C, and albeit the temperature increase to 5 °C did result in enhanced dispersibility, it was still very low (Table 8). The low sulphur fuel oils tested had a high pour point, which is considered to be the main reason for the low dispersibility. When such oils are spilt at sea in the Arctic, or in other cold waters, the oil will solidify. Weathering will also change the properties of the oil, turning it into an oil that in some cases may be even more difficult to disperse (Wegeberg et al. 2017). However, Sørheim et al. (2020) found that some of the test emulsions were dispersible, whereas the water-free oil was not.

Our results suggest that chemical dispersion is not an effective method to combat LSFO spills on cold sea surfaces. Also, Hellstrøm (2017) concluded that natural dispersion is minimal as they found low dispersibility at low mixing energy at 2 °C. Higher mixing energy, higher temperatures and successive application somewhat improved dispersibility (as seen in our study and that of Hellstrøm 2017). Still, untreated LSFOs (as well as water-in-oil emulsions due to weathering of the LSFOs) as well as LSFOs treated with chemical dispersants are assessed to be a risk mainly for organisms associated with the sea surface and the shoreline if the oil is beaching. Low effectiveness of dispersant use was also found by Sørheim et al. (2020) and Hellstrøm (2017), and Sørheim et al. (2020) suggested that the low chemical dispersibility may be due to either high viscosity of the emulsions and/or a high pour point of the oils.

6.1.3 Potential for biodegradation of oil and dispersed oil

Based on the small-scale test investigating the potential for natural degradation, it was found that both ULSFO 2018 and WRD had fairly slow biodegradation rates in cold Baltic Sea water during the 28-day experiment. Correspondingly, the results display that biodegradation of LSFO in film on the sea surface would be rather slow. The use of dispersant on WRD did not increase the degradation potential; on the contrary, it seemed to inhibit biodegradation. Hence, biodegradation of LSFOs, fresh as well as treated oil, is not expected to play a major role in the short-term fate of these oils if spilled on the sea surface. However, on the long term, biodegradation will continue with a slow rate.

6.1.4 Potential for mechanical recovery

Mechanical recovery was not part of this study. However, several basin tests and field experiments using different mechanical recovery techniques are currently undertaken, e.g. in the Norwegian Coastal Administration's test basin and as part of the Norwegian Clean Seas Association for Operating Companies (NOFO) oil in water 2022 tests. In the literature, for instance Sørheim et al. (2020) found that "the effectiveness of mechanical recovery is dependent on the choice of skimmer system that forces contact between the oil and the recovery unit. Oils with high pour points will e.g. need an "active" high viscosity or belt skimmer designed for solidified oils at sea". In other words, the available conventional skimmers will most likely have large difficulties in recovering LSFOs from the sea surface.

6.2 Assessment of key organisms at risk

LSFOs may have a significant adverse environmental impact on the marine environment, and oil spill response techniques may have environmental side effects. For the assessment of the impacts on the surrounding environment, we divided the marine environment into different spatial compartments: sea surface, seawater column, seabed and shoreline (Wegeberg et al. 2020). For each of these compartments, the possible impacts of an LSFO spill and of treated oil as part of the response were assessed with focus on the relevant organisms and ecosystems based on the results from this study.

6.2.1 Sea surface

Based on the oils tested in this limited study, the results suggest that chemical dispersion may not be an effective method for combatting LSFO spills at cold sea surfaces. Burning the oil is a possibility for fresh oils; however, the ignitability is likely inhibited with increasing weathering of the oil. In our study, the burn residue did not sink, indicating high degree of persistence on the sea surface.

In the case of oil at the sea surface, the organisms most vulnerable are seabirds, and even small amounts of oil can have lethal effects (e.g. Piatt 1990). Oil on seabird feathers will destroy the waterproofing of the plumage, resulting in loss of insulation and buoyancy (Fritt-Rasmussen et al. 2015). As most seabirds spend their entire non-breeding season at sea and rely on feathers for flight, insulation and buoyancy (Stephenson 1997), feather fouling from oil could be a major cause of mortality (Leighton 1993).

Marine mammals are less vulnerable to oil spills. The potential risk to whales of LSFO spills is smothering of blow hole and baleen plates, but this effect has not yet been found recorded (AMAP 2010).

6.2.2 Seawater column

Based on the oils tested in this limited study, chemical dispersion seems not to be an effective method for combatting LSFO spills on cold sea surfaces as the burn residue did not sink. Hence, the oils tested in this study indicate a very low degree of dispersion or dissolution in the water column. Only WRD was somewhat dispersed at 5 °C and may thus enter the water column. Therefore, based on the results from this small-scale study, the risk of exposure of bacteria, plankton, fish, fish larvae/fry and other pelagic organisms to oil or treated oil is low. As for WRD at a temperatures of 5 °C, however, exposure of the mentioned organisms to dispersed oil and therefore also to toxic effects involves moderate risk.

However, as shown in Figure 1, there is wide variation in the pour point and also the viscosity of the LSFOs, and some of the LSFOs therefore hold a potential of being dispersed.

A side effect of in-situ burning of an LSFO spill may be soot and hereby generation of high ring-numbered PAHs that may bioaccumulate in zooplankton and fish if mixed into the water column (Fritt-Rasmussen et al. 2015).

6.2.3 Seabed

As the results suggest that fresh and treated LSFOs are persistent on the seawater surface, it is assessed that there is only a minor risk for fresh oil, dispersed oil or burn residues reaching the seabed. However, the density of the burn residues and weathered oil may increase also as a result of sediment build-up (Fritt-Rasmussen et al. 2015). This is particular of concern in shallow waters, where the oil may also reach the seabed and hence potentially impact the benthic community.

6.2.4 Shoreline

As the oils tested in this study seem to have a high degree of persistence on the sea surface, there is an evident risk of oil/burn residue beaching with resulting smothering of the shoreline community due to high viscosity of the LSFOs; thus, the natural cleaning by sea wash may be reduced (Wegeberg et al. 2020, Gustavson et al. 2020). Further, coastal fish species spawning nearshore, e.g., capelin, may experience toxic effects from exposure to oil compounds (Tairova et al. 2019). As for oil spills reaching the shoreline, the oil can also be contained in ice, particularly winter spills.

6.3 Perspective and summary of assessment of key organisms at risk

The main challenge for many of the new generation of LSFOs (both VLSFOs and ULSFOs) is that many (but not all) have a high pour point and viscosity and therefore high persistence in the marine environment, which may limit the effectiveness of the different response options. Our results indicate that the LSFOs tested likely have a high degree of persistence on the sea surface and shoreline even when chemical dispersion or in-situ burning is attempted. Hence, overall the results of this small-scale study suggest that the next step in investigating potential toxic effects should focus on organisms associated with the sea surface, e.g., seabirds, and shoreline organisms such as blue mussels (*Mytilus edulis*) and tidal vegetation (*Fucus* spp.).

7. References

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8. Appendix

8.1 Standard calibration curves for dispersion tests

The content of oil in each extract, calibrated with external standards, was determined using a Cary Eclipse Fluorescence Spectrophotometer, Agilent Technologies. The standard curves were prepared by measuring the fluorescence intensity of a number of external standards with known increasing concentrations. The standard curves are given below (Figures 18–21), including regression lines with A and B values for calculation of the dispersion effectiveness.

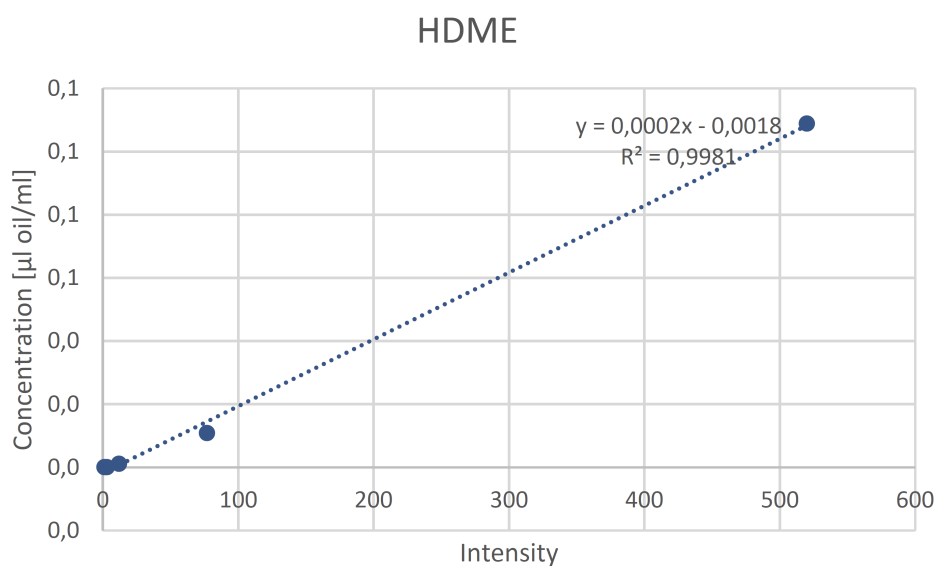


Figure 18: Standard calibration curve and regression equation for HDME.

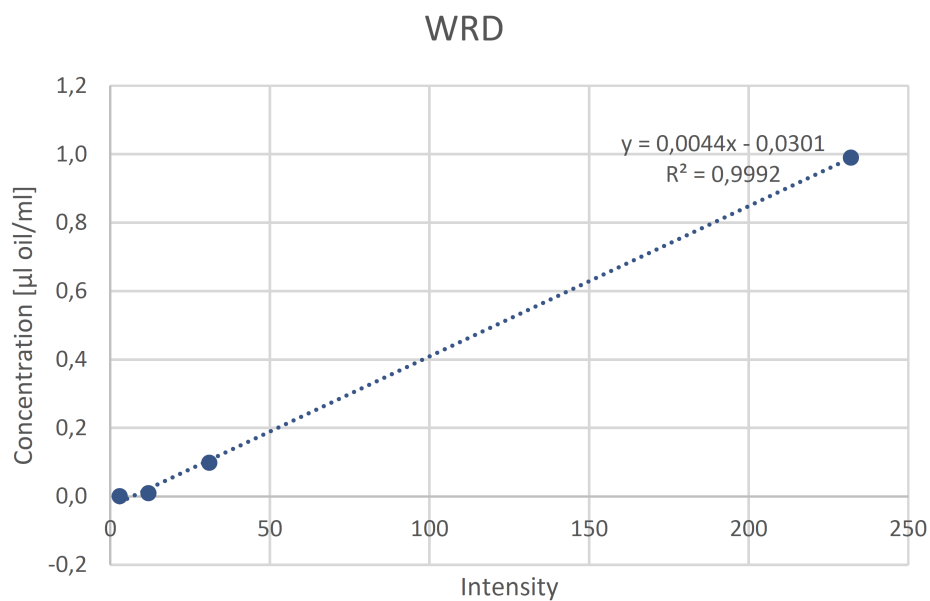


Figure 19: Standard calibration curve and regression equation for WRD.

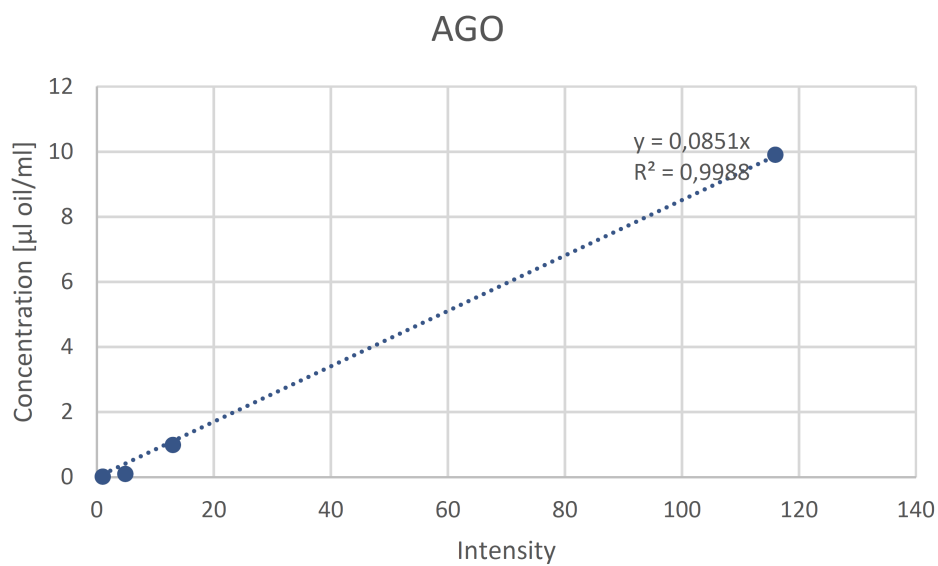


Figure 20: Standard calibration curve and regression equation for AGO.

IFO180

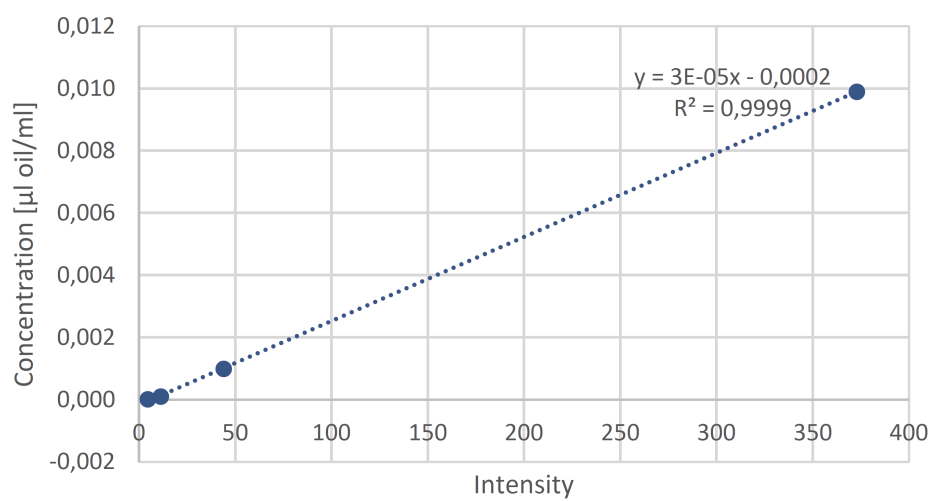


Figure 21: Standard calibration curve and regression equation for AGO.

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