



Chemical and toxicological characterisation of residues from offshore in-situ burning of spilled fuel oils

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ABSTRACT

Burn residues collected after large scale experimental in situ burns performed in the North Sea were characterised with emphasis on chemistry and acute toxicity. Low-energy water accommodated fractions (WAFs) of three marine fuels (Ultra Low Sulphur Fuel Oil (ULSFO), bunker oil (IFO180), and marine gas oil (MGO) and their field-generated residues from in situ burning (ISB) were prepared to evaluate the potential impact of ISB residue to the environment. The toxicity effects on primary consumers were assessed by testing on early life stage (nauplii) of the marine copepod *Calanus finmarchicus*. Toxicity studies showed that ISB decreased the acute toxicity of the WAFs compared to the initial oils. WAF of MGO had highest toxicity, and ISB residue of MGO seems to be more toxic than WAFs of fresh ULSFO and IFO180. Additive toxicity expressed as toxic unit (TU) based on the chemical composition also indicated that the toxicity of WAFs from ISB residues were lower than for the initial oils. The 2–3 ring PAHs seem to contribute most to the TU. Overall, the three offshore burns reduced the total mass of PAHs in the water accommodated fractions by >90 % compared to the released unburned oils and caused a reduction of the acute toxicity to copepod nauplii.

1. Introduction

In-situ burning (ISB) is an oil spill response technique that involves the controlled ignition and burning of the oil at or near the spill site on the water surface (e.g. [1]). Controlled ISB has proven effective for oil spills in ice conditions and has been used successfully to remove oil from spills in ice-affected waters in several large-scale field experiments since the 1970s (summarized in Ref. [2]). ISB is a response option that has rarely been used on marine oil spills in open water previously. However, after its successful use during the Gulf of Mexico Deepwater Horizon response in 2010 where approximately 400 burns were completed [3], ISB is also considered used in other areas than the Arctic.

Oil removal efficiency by ISB is primarily a function of three main factors (more details in Ref. [2]): The initial thickness of the slick, the thickness of the residue remaining after extinction, and flame coverage of the slick. Other secondary factors include environmental effects such as wind and current herding of slicks against barriers (e.g. booms or ice edge) and temperature effects. ISB will always leave a burn residue, and the fate, behaviour and effects of the residues are of environmental concern. The characteristics of the burn residue will vary with several

factors, such as oil type, weather conditions and burn efficiency. Several studies have looked into the chemical composition of the ISB residues, and a mutual observation was a depletion of lower boiling point components and an increase in the typically pyrogenic high-ring PAHs (e.g. [4–9]). There are relative few studies that have investigated the toxicity of ISB residues according to a review by Fritt-Rasmussen et al. [10], and none of them had studied the effects of residues from ISB of fuel oils. However, in a recent paper by Johann et al. [11], the toxicity of ISB residues of IFO180 to early life stages of zebrafish was investigated. They concluded that ISB residues did not induce greater toxicity in zebrafish embryos compared with the initial oil. The Newfoundland Oil Burn Experiment (NOBE, summarized in e.g. [12]) studied potential toxic effect on marine organisms of ISB residues and unburned crude oil (Alberta Sweet Mix Blend). Samples of the underlying water after burning, both from the laboratory and NOBE, as well as weathered oil and ISB residue were tested and it was concluded that ISB does not generate a residue that is more toxic than the weathered oil (Blenkinsopp et al. [13] and Daykin et al. [14]). In a paper by Gulec and Holdway [15], acute toxicity of seawater after ISB and ISB-residue were studied. They used two marine organisms (amphipod and snail) on two

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development stages in lethal and sub-lethal bioassays and found very low toxicity in all studies.

In Norway, the Norwegian Clean Seas Association for Operating Companies (NOFO) and the Norwegian Coastal Administration (NCA) are cooperating closely to operationalize ISB as a possible response method in Norwegian waters. As a part of the "Oil-on-water" experiments in the North Sea in 2018 (OOW2018) and 2019 (OOW2019), seven experimental ISB with different oil types were performed. In these field experiments testing of ignitors (hand-held or from drone), validation of fire booms, and estimation of burning efficiency were included [16]. Photo from one of the burns is shown in Figure S5 in the Supplementary Information (SI5). Several samples of burned residues from these burns were collected at different positions within the boom. These residue samples are used in the present study. A more extensive characterization of the physical-chemical properties of the burned residues and the smoke emissions are discussed in Faksness et al. [16] and the potential for human exposure during ISB in Szwangruber et al. [17]. The operational aspects of these ISBs are described and discussed in Jensen et al. [18].

Early life stage (nauplii, N3) and late copepodite stage (CV) of *Calanus finmarchicus* have earlier been exposed to LE-WAF of fresh ULSFO to compare potential toxic effects [19]. It was observed that the nauplii were more sensitive to the WAF than the late copepodite stage which may be related to both size related kinetics and lipid content [20,21].

The objective of the present study was to evaluate impact to the marine environment of ISB residues after offshore experimental burning of three marine fuel oils: ULSFO, IFO180 and MGO. Low-energy water accommodated fractions (LE-WAF) were prepared from fresh oils and their burn residues. Potential toxic effects on primary consumers were assessed by testing on nauplii (N3, early life stage) of the marine copepod *Calanus finmarchicus*, which is one of the key ecological species in northern boreal to Arctic oceans.

2. Materials and methods

2.1. The field experiments

The offshore in situ burning experiments took place at the former Frigg field in the Norwegian part of the North Sea in mid-June [16]. About 6 m³ of each oil were released and contained into a fire-boom before being ignited by use of a "Pyro-drone" (DESMI) with a gelled ignitor consisting of diesel (80 %) and gasoline (20 %). ISBs with artificially pre-weathered Oseberg Blend crude (200°C+, corresponding to approximately 0.5–1 day of weathering on sea), marine gas oil (MGO), an Ultra Low Sulphur Fuel Oil (ULSFO) and a heavy fuel oil (IFO 180) were performed.

2.2. Oil properties

The properties of the initial (unburned) oils and their ISB residues are given in Table 1. The GC chromatograms of the oils and their ISB residues are given in Supplementary Information (SI2), Figures S1 to S3. A more detailed characterization of the physical-chemical properties of the burned residues is reported in Faksness et al. [16]. The ISB residues of MGO and IFO180 had very different physical properties, which was

Table 1

Physical properties of the oils used in in-situ burning [16]. Density was measured at 15.6 °C and viscosity at 10 °C (shear rate 10 s⁻¹).

SINTEF ID	Oil	Density (g/kg)	Viscosity (cP)
2018-3881 S1-S2	ULSFO fresh	0.917	17700
2018-3881-S12-S1	ULSFO ISB residue	0.945	131 000
2019-5233-S1	IFO180 fresh	0.960	12 600
2019-5233-S2	IFO180 ISB residue	1.001	1 010 000
2019-5235-S1	MGO fresh	0.847	6
2019-5235-S3	MGO ISB residue	0.886	259

reflected in their viscosities. The MGO residue was still liquid after burning (viscosity of 259 cP), while the residue of IFO180 was very sticky with a viscosity of more than 1 mill cP and a density of 1.001 g/kg. A density above one indicates that the residue may have the potential to sink, especially if exposed to for example sand particles, but as natural seawater with a density of 1.025 was used in the present study, this was not a problem.

2.3. WAF preparation

Preparation of low energy WAF (LE-WAF) was performed under controlled conditions following the guidelines established by the Chemical Response to Oil Spills: Ecological Research Forum (CROSERF). These guidelines were developed to standardize WAF preparation for laboratory exposures to aquatic organisms, and analytical chemistry measurements used to determine the acute toxicity of the water-soluble components in the oil [22]. LE-WAF can be defined as a water solution of dissolved oil components prepared in closed vessels, with calm mixing of oil and water without the formation of any vortex. LE-WAFs were chosen in this study in order to avoid generation of oil droplets.

Sterile filtered (0.22 µm) natural seawater (1.75 L) collected from 90 m depth in the Trondheimsfjord was added to 2 L bottles giving a water to air headspace ratio of 4 to 1. The loss of masses during ISB relative to the initial oil masses were not accounted for when preparing WAFs of the ISB residues, as all WAFs were prepared with the oil-to-water loading of 1–40 (25 g oil/L seawater). The oil-to-water ratio of 1:40 is assumed to be a "saturated" system and therefore represents a "conservative" estimate of the concentrations foreseeable during an oil spill. The ISB residues were heated to 50 °C for approximately 2 h to get them as homogenous as possible before weighting and applied warm to the water surface to get it as evenly distributed as possible. The WAFs were generated with a contact time between water and oil for three days at room-temperature (approximately 22 °C) before the water was collected for chemical characterization and toxicity tests. Samples for chemical analysis was acidified (pH < 2) after sampling and stored at 5 (±2) °C. Toxicity testing was initiated the same day as sampling. Photos of WAF systems with fresh and ISB residue of MGO are shown in SI5 (Figure S6).

2.4. Sample preparation and chemical analysis

An aliquot of the initial oils and the ISB residues was weighted directly into graduated volumetric flasks (10 mL and dissolved in dichloro methane (DCM)). Water samples from the generated WAFs were processed using liquid-liquid extraction with DCM (modified [23]).

All samples were added internal standards for quantitative analysis on gas chromatograph with flame ionisation detector (GC/FID) and gas chromatograph with mass spectrometer (GC/MS). For GC/FID *o*-terphenyl and 5 α -androstane were added as internal standards, and for the GC/MS analysis naphthalene-*d*₈, phenanthrene-*d*₁₀, chrysene-*d*₁₂, fluorene-*d*₁₀ and acenaphthene-*d*₁₀ were added.

The samples were analysed for semi volatile organic compounds (SVOC; decalins, naphthalenes, PAHs and hopane) using GC/MS and for total petroleum hydrocarbons (TPH) using GC/FID. In addition, the oil and water samples were analysed for volatile organic compounds (VOC; C₅-C₁₀), including BTEX (benzene, toluene, ethylbenzene, and xylenes), by use of P&T GC/MS (Purge and Trap Gas Chromatography Mass Spectrometry). A list of all target analytes is shown in Table S1 (SI1). This list includes the recommended analytes given by Singer et al. [24] and is a typical standard list for the target compounds used during post-oil spill damage assessments.

The GC/FID analyses were performed according to a modification of EPA Method 8015D [25]. TPH (resolved plus unresolved TPH) was quantified by the method of internal standards using the baseline corrected total area of the chromatogram and the average response factor for the individual C₁₀ to C₄₀ n-alkanes

The semi-volatiles were quantified by modifications of EPA Method 8270D [26]. The mass spectrometer was operated in the selective ion monitoring mode to achieve optimum sensitivity and specificity. The quantification of target compounds was performed by the method of internal standards, using average response factors (RF) for the parent compounds. The PAH and phenol alkyl homologues were quantified using the straight baseline integration of each level of alkylation and the RF for the respective parent PAH compound. The response factors were generated for all targets and surrogates versus fluorene-*d*₁₀.

A total of 35 target volatile analytes in the C₅ to C₁₀ range were determined by P&T GC/MS using a modification of EPA method 8260C [27]. The samples were added the internal standards toluene-*d*₈, ethylbenzene-*d*₈ and chlorobenzene-*d*₅. The quantification of individual compounds was performed by using the RFs of the individual compounds relative to the internal standards. All standards and samples were analysed in a full scan mode.

2.5. Toxicity of WAF samples

2.5.1. Acute toxicity to nauplii of *Calanus finmarchicus*

Potential effects on primary consumers were assessed by testing on the early nauplii (N3) life stage of the marine copepod *Calanus finmarchicus*, which is one of the key ecological species in northern boreal to Arctic oceans. The acute toxicity testing was performed as a modification of ISO 14669:1999 [28] with lethal immobilization (LC₅₀) as the endpoint. The original ISO protocol is not designed for testing of solutions containing volatiles and was modified by using glass vials with screw caps as exposure vessels to preserve volatiles during exposure. To accommodate for testing with nauplii stages of *C. finmarchicus* (Photo in SI5, Figure S7), compared to the listed larger species and stages in the ISO protocol [28], 5 mL vials were used. The exposure time was increased to 72 h at a set temperature of 10 ± 2°C.

The WAF samples were diluted in a series of seven concentrations with a spacing factor of 1.7 between dilutions with each exposure concentration made in quadruple. Seawater only was used as negative control in duplicate of the exposure concentrations, i.e. eight vials. For positive control, a single concentration of 3,5-dichlorophenol (0.5 mg/L) was used in same replication as for the exposure dilutions. The exposure vials were filled with minimal headspace to keep potential evaporative loss to a minimum during exposure, and the respective exposure vessel was stocked with nominally 25–30 nauplii at onset exposure. Mortality was monitored at end of exposure after 72 h under a low-power dissecting microscope. The test animals were not fed during exposure. The calculated values are corrected for mortality in the control series and the effect is calculated within the span 0–100 % effect by constraining the top and bottom of the concentration-effect curve to 100 and 0.

2.5.2. Predicted toxicity using toxic units

One toxic unit (TU = 1) is the reciprocal of the contaminant concentration that causes 50 % effect or mortality to organisms for acute exposures (Concentration/LC₅₀). Thus, for TU's >1 the effect exceeds 50 %, whereas for TUs below 1 the effect is less than 50 %. The TU thus represent the number of times a contaminant must be diluted or concentrated to give 50 % effect on the test species (e.g. [29]).

TU models are specialized cases of concentration addition that are used to assess the toxicity in mixtures of components with a similar mode of toxic action based on chemical analyses [30,31]. In the WAFs from petrogenic products, the compounds of concern for toxicity assessment are typically restricted to the characterized VOCs and SVOCs, which are structurally classified as causing Type I narcotics. The target lipid model of narcotic toxicity demonstrates that the acute toxicities of these chemicals vary and are correlated with the octanol-water partition coefficient (K_{ow}) such that LC₅₀ decreases with increasing K_{ow} [31]. A toxic unit (TU) for the individual compounds is computed by dividing the measured concentration in the WAF by the compound's

water effect concentration using regression models. It appears to be a linear negative relation between log LC₅₀ of the marine organisms and log K_{ow} of the components that may cause toxic effects [32,33] and Di Toro et al. [31]:

$$\log LC_{50} = m \log (K_{ow}) + b \quad (1)$$

The slope (m), log K_{ow}, and the intercept (b) for different component groups (e.g. MAH (monoaromatics), PAH and phenols) are given in McCarty et al. [33] and Neff et al. [34]. The LC₅₀ (mg/L) is calculated for each individual component by use of equation (1). Different regression coefficients can be used to calculate TU relative to different species [35], but since no regression is available for the current test organisms a generic regression was used [32,33]. The same regression was used for all WAFs and this will thus provide comparable data for exposure solutions, but it may not represent the TU for the copepod nauplii. To compare the theoretical toxicity and the contribution of the various characterized constituents in the WAFs, TUs of 62 individual VOC and SVOC components (given in Table S1 (SI1)), excluding "Other VOCs" and hopane) are summed up to compute the TUs of the WAFs.

3. Results and discussion

The fuel oils ULSFO, IFO180 and MGO were used in this study. Both the initial, unburned oils and their corresponding ISB residue were from the large-scale offshore burns during the "Oil on water" field trial in the North Sea in 2018 (ULSFO) and 2019 (IFO180 and MGO). Chemical characterisation of the WAF systems prepared of the crude oil Oseberg 200 °C + and its ISB can be found in Keitel-Gröner et al. [36]. More detailed results are provided in Supporting Information.

3.1. Chemical composition of the oils, ISB residues and their WAFs

All GC chromatograms are shown in Supplementary Information (SI) Figure S1 to S3 (SI2). The GC chromatograms of the ISB residues illustrated that there were still lighter components (<C₁₀) present after ISB, but that there were less of them. As detailed in Table S3 (SI3), total WAF concentrations of fresh oils were 1.1, 3.4 and 10 mg/L for ULSFO, IFO180 and MGO, respectively. In WAFs of ISB residues, the WAF concentrations were reduced to 0.33, 0.21 and 2.1 mg/L for ULSFO, IFO180 and MGO, respectively.

Fig. 1 summarizes the composition of the main groups of aromatics in the oils (Fig. 1A) and their corresponding WAFs (Fig. 1B). The data are also provided in Table S2 and Table S3 (SI3). The "chemical profile" of a WAF is unlike that of its parent oil. Decalins, 4–6 ring PAH and some of the 2–3 ring PAH have low solubility in water, and this is also reflected in the chemical composition of the WAFs.

The contribution of the different component groups indicates the effect ISB has on the chemical composition of the oil with loss of most of the volatiles and some of the naphthalenes and 2–3 ring PAHs. All ISB residues studied here contained volatiles (Table S2, SI3). However, it has been assumed that residues after ISB do not contain water soluble oil components, but the results from our studies, using ISB residues from the offshore large-scale burns, have shown that the residues still had components that could dissolve into the water as the burning efficiency (BE) could vary within a slick. E.g., for ISB residues sampled at three different locations in the boom after ISB of IFO180 [37], the BE was estimated to vary from approximately 30–60% (sample with highest BE was used in this study). Estimated BE for ULSFO was 57 % and more than 95 % for MGO [16].

Compared with the initial, unburned oils, the depletion in the total concentration of PAHs (including decalins and naphthalenes) in the ISB residues were approximately 40 % for ULSFO, 90 % for MGO, and 70 % for IFO180 during the OOW field trial. Unburned ULSFO contained less low boiling point components, such as decalins and naphthalenes than the other oils, which may cause the depletion to be lower in the ULSFO

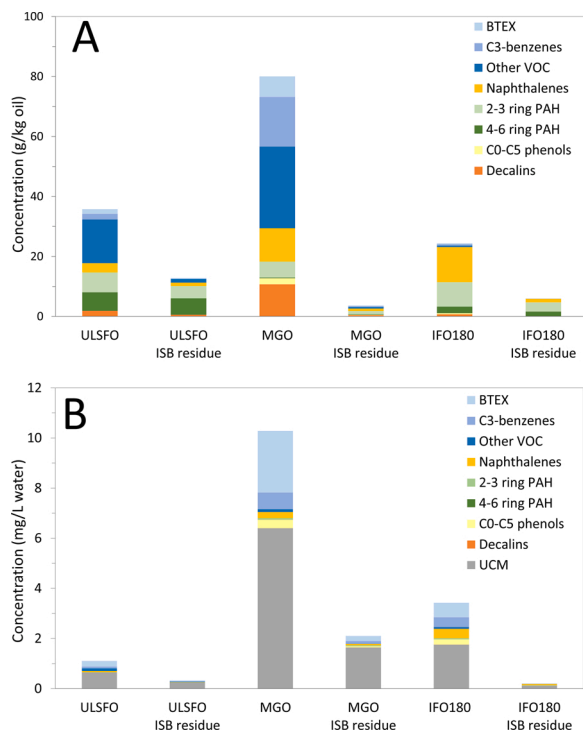


Fig. 1. Chemical composition of selected component groups (aromatics) in the unburned oils and ISB residues (Figure A). The corresponding WAF systems for ULSFO, MGO and IFO180 are shown in Figure B. Note that there are different scale and titles on the y-axis.

ISB residue. The concentrations of the most bioavailable and water soluble components in the oils, such as naphthalenes and 2–3 ring PAHs, were reduced during ISB, but the percentage of more heavy, typically pyrogenic, 5–6 ring PAH, increased. An increase in heavy, high-ring numbered PAHs as a result of burning has also been reported by others (e.g. [43–8]). Van Gelderen et al. [42] observed up to 90 fold increase in some pyrogenic PAHs in the residue after an experimental burn of a heavy oil. However, these are heavy PAHs and are not expected to be readily dissolved in water. We recorded a slight increase in the concentrations of 4–6 ring PAHs in the WAFs of burn residues compared the fresh oils of ULFO and MGO (Fig. 1, Supplementary information; Table S7), but this did not appear to have a strong impact effect on the toxicity (Fig. 3). The final chemical composition of the residue and the resulting WAFs will, however, depend on the initial oil type and the efficiency of the burning.

Garrett et al. [38] studied pyrogenic PAHs after burning a crude oil in the laboratory with 85 % BE. They observed that the concentrations of several of the pyrogenic PAHs were somewhat enriched in the residue, but these increases were outweighed by the mass of oil consumed in the

burn as the ISB substantially reduced the total amounts of PAHs left on the water surface after the spill. Stout and Payne [35] studied the chemistry of the residues generated after ISB during the Deepwater Horizon oil spill and estimated that ISB reduced the total mass of PAH by 89 %. These observations are in accordance with our results, where the total amount of SVOC were reduced from 556 kg in the released oils to 39 kg in the ISB residues, i.e. 93 % reduction (Table S4, SI3). Several other studies have also seen a reduction in total PAH content after ISB (e.g. [43,5,8]).

The SVOC concentrations in WAFs were lower in the WAFs of ISB residues. The loss of SVOC components varied from 65 % for ULSFO to 90 % for IFO180 and Oseberg (80 % reduction for MGO). The largest loss was among the naphthalenes, phenols, and the 2–3 ring PAHs with lowest boiling point. This is illustrated in Fig. 2, where the distribution of SVOC components in WAF of fresh and ISB residue of MGO is shown as an example.

3.2. Toxicity

3.2.1. Acute toxicity of WAFs to nauplii of *Calanus finmarchicus*

The acute toxicity, expressed as LC₅₀, can be given in percent dilution of the undiluted (or 100 %) WAF (relative toxicity, LC₅₀ in percent (% WAF)), or as normalized to the total WAF concentration (specific toxicity, LC₅₀ in mg/L or ppm). Low values of LC₅₀ indicate a high toxicity, while a high value of LC₅₀ corresponds to lower toxicity [39]. Whereas relative toxicity takes into account the combined solubility (maximum dissolution) of oil components, the specific toxicity is only related to the mass of oil components in the WAF. Results presented both as relative and specific toxicity are given in Table S6 (SI4).

In Fig. 3, copepod nauplii survival is plotted as a function of WAF concentration (in % diluted) at test endpoint (72 h). The graphs compare the survival exposed to WAFs of fresh oil (blue line) and WAFs of ISB residue (black line). The graphs show that the percent dead copepod nauplii increase with the WAF concentration and that the observed mortality was approximately 100 % in the undiluted WAF in all systems (Table S6, SI4).

Most of the previous studies have not reported any toxic effects when different organisms have been exposed to ISB residues. However, comparing toxicity results from different laboratories with different exposure media, species and test protocols are challenging. E.g. Blenkinsopp et al. [13] investigated toxicity of the weathered crude oil used at NOBE and the resultant burn residue using LE-WAFs (no vortex). Toxicity of WAFs with seawater were tested on three-spine stickleback and the gametes of the white sea urchin. All samples were found to be non-toxic to the species tested. TPH concentration in the WAF of weathered oil was 1.1 mg/L, which was higher than the WAFs in our study (ULSFO (0.71 mg/L), but lower than IFO180 (2.4 mg/L) and MGO (7.05 mg/L)). In our study, testing the toxicity on the nauplii stage of a copepod, LC₅₀ values for the non-burned oils were in the range of 12 % (MGO) to 24 % (ULSFO). For the TPH concentration in the WAF with

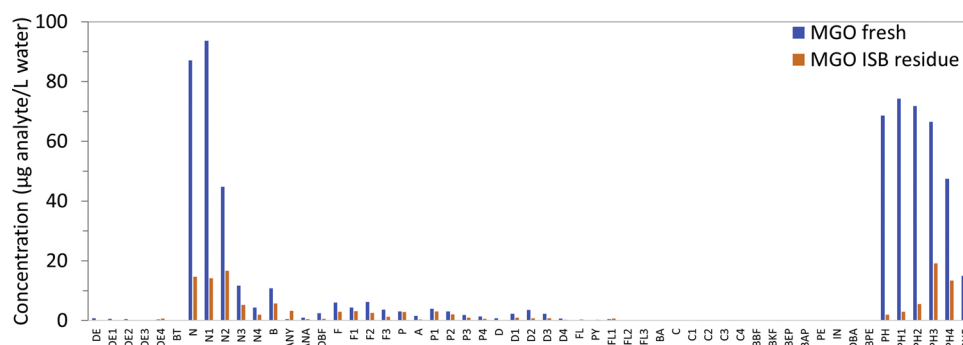


Fig. 2. Distribution of SVOC components in WAFs of MGO: Fresh, unburned oil and ISB residue. PAH abbreviations are given in Table S1, SI1.

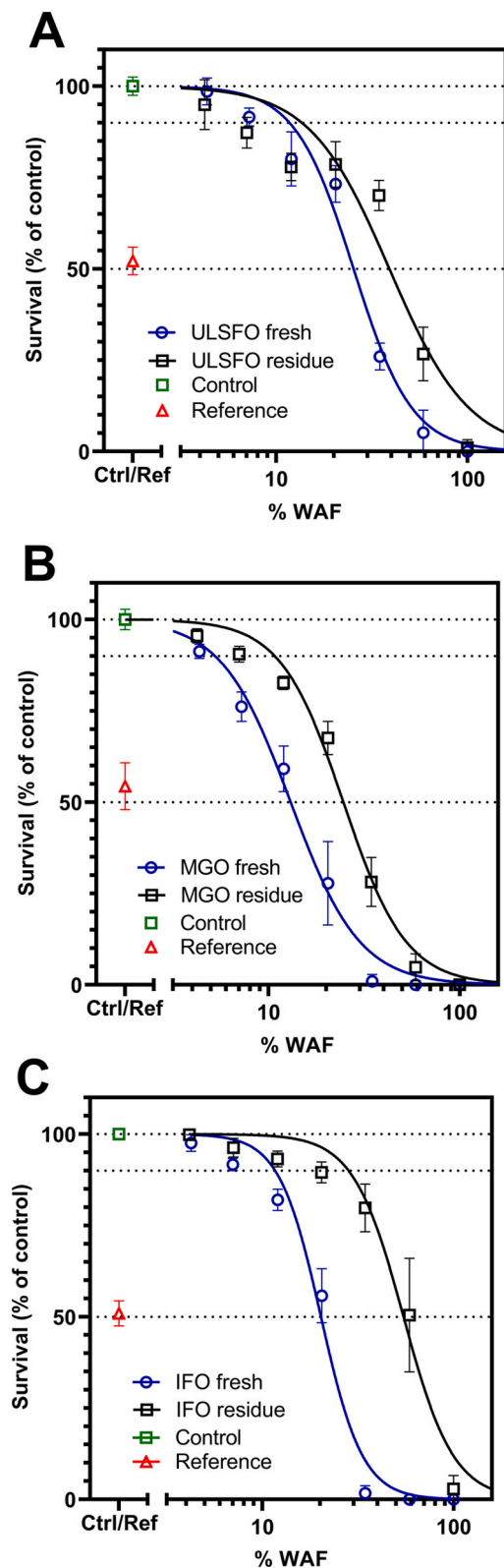


Fig. 3. Survival of *Calanus finmarchicus* nauplii relative to controls (green) after 72 h exposure to low energy WAFs of oil:water ratios of 1:40 of fresh oils (blue) and corresponding burn residue (black) as a function of WAF dilutions (%). A: ULSFO; B: MGO; C: IFO180. The reference (red) is a positive control (0.5 mg/L dichlorophenol) verifying the sensitivity of the test tested population. Vertical bars represent 95 % confidence interval. The number of replicates for each concentration was 4 for the exposed groups and 8 for the controls (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

burned residue, Blenkinsopp et al. [13] reported 0.13 mg/L, which was lower than in our WAFs with ISB residues of IFO 180 (0.18 mg/L), ULSFO (0.27 mg/L), and MGO (1.78 mg/L). We observed nearly 100 % mortality at test endpoint for the *Calanus* nauplii when exposed for undiluted WAFs of ISB residues of ULSFO (LC₅₀ = 36 % WAF), MGO (LC₅₀ = 23 % WAF) and IFO180 (LC₅₀ = 52 % WAF). WAF of fresh MGO had highest toxicity (LC₅₀ of 12 % WAF) of the tested systems. WAF of ISB residue of MGO seems to be more toxic (LC₅₀ of 23 % WAF) than ULSFO (LC₅₀ of 36 % WAF). Lowest toxic effect was observed in WAF of ISB residue of IFO180 (LC₅₀ of 52 % WAF). A comparison based on the TPH concentrations of the exposure media suggest that the species used in our toxicity tests, with mortality as endpoint, were more sensitive to water soluble oil components than the species tested by Blenkinsopp et al. [13].

In a recent study, Johann et al. [11] prepared LE-WAFs with IFO180 and ISB residue of IFO180 for exposure experiments using zebrafish in a fish acute embryo toxicity test (120 h post fertilization). The WAF concentrations were quantified by 18 unsubstituted PAHs, but neither TPH nor volatiles were analysed. The PAH concentrations were 0.185 mg/L in WAF of initial oil, and 0.052 mg/L in WAF of burned oil which for the same 18 PAHs is in the same range as in our study which was 0.165 mg/L and 0.030 mg/L, respectively. In line with our results Johann et al. [11] observed higher acute mortality for the fresh oil WAF than for the burnt residue WAF. However, based on the sublethal effects they concluded that there were no overt differences in the acute toxicity of the WAFs of IFO180 and burned IFO180 to zebrafish embryos.

Camus [40] investigated the environmental effects of ISB residues in mesocosms systems installed in the fjord ice on Svalbard. The ISB residue was generated in the laboratory and frozen into the mesocosms in February and the experiments were terminated in July. No effects on the sea ice community were observed on adult copepods, but there were also performed effect studies in the laboratory of ISB residue on copepods (*Calanus glacialis*) and polar cod (*Boreogadus saida*) [41]. Camus [40] summarized that no quantified effect was observed for adult copepods, but that there were indications on effects of the development of nauplii. This supports our observation that early stages of copepods may be among the most sensitive marine organisms to oil components.

Bender et al. [41] studied the potential long-term physiological effect on maturing polar cod after acute exposure (48 h) to WAF of burnt oil residue. Overall, no significant long-term effects were seen in growth or mortality for adult polar cods, but the reproduction system to the female polar cod seems to be influenced if exposed to ISB residue.

Keitel-Gröner et al. [36] studied acute and sublethal effects on WAF of Oseberg 200 °C + and its ISB residue to Northern shrimp larvae. The ISB residue of Oseberg used, was from a large scale burn on the OOW field trial in 2018. No mortality when exposing the larvae to WAFs of ISB residue was observed, however, the WAF concentration was 0.075 mg/L, which is between 3 and 28 times lower than in our studies and slightly below the lowest LC₅₀ of 0.11 mg/L (IFO burnt residue) observed for the *Calanus* nauplii. WAF of unburned Oseberg was acutely toxic to the shrimp larvae with a LC₅₀ of 37.4 % (0.57 mg/L total WAF). They observed no significant long-term effects of either of the WAFs.

In the studies mentioned above, the ISB residues have been used to prepare exposure solutions for toxicity testing. Faksness et al. [5] burned a pre-weathered crude oil in the laboratory (approx. 80 L water, 0.8 L water, film thickness of 2.7 mm) and sampled the underlying water before and after ISB to study acute toxicity to *Calanus finmarchicus*. The water soluble oil components were of interest, and the results showed that there were no increase in toxicity in the underlying water after ISB when compared with the toxicity of water of the initial weathered oil.

3.2.2. Calculation of toxic units

The acute TUs of the various WAFs from the toxicity tests with *Calanus* nauplii were based on total WAF concentration of the "saturated" (100 %) WAFs and the recorded LC₅₀s (Fig. 4B). Additionally, a theoretical acute toxic unit (TU) for the same WAFs was predicted based on

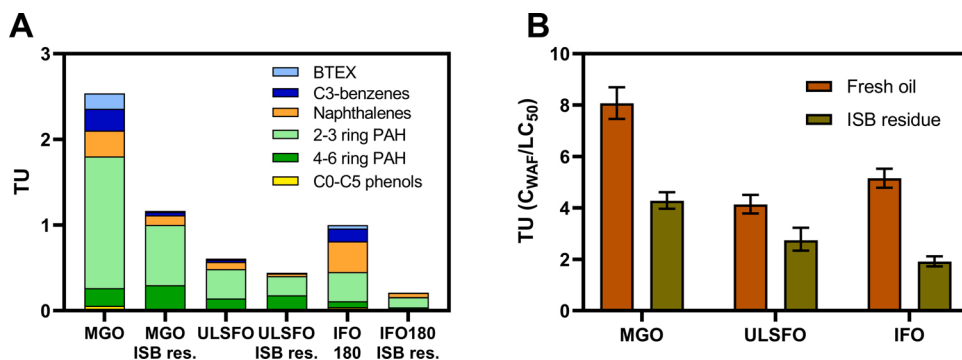


Fig. 4. Comparison of toxicity of undiluted low energy WAFs from oil:water ratios of 1:40 of fresh oils and corresponding burn residues expressed as toxic units (TU). A: Estimate based on additive toxicity of characterized volatile and semi-volatile components. B: Based on toxicity tests with *Calanus finmarchicus* nauplii related to the total WAF concentration. TU corresponds to the number of times the solution must be diluted to cause 50 % mortality (TU = 1) of the tested organisms. Vertical bars in Figure B represent 95 % confidence interval.

their chemical composition by concentration addition and regressions between LC_{50} and $\log K_{ow}$ for the individual components. Both approaches show the same pattern with the WAFs of ISB residues being less toxic than the corresponding fresh oil WAFs. Furthermore, the ranking of the toxicity of the various WAFs is the same, however, the TUs based on the toxicity test are consistently higher. This is expected since the theoretical calculation based on characterized components only covers a fraction of the oil mass (SVOC and VOC) whereas the unresolved fraction (UCM) which could include thousands of unidentified components, is also expected to contribute to the toxicity. Furthermore, the *Calanus nauplii* have previously been shown to be sensitive to oil contaminants [19] and may be more sensitive than the "average" organism represented by the generic regressions used in the present calculations. The results, however, confirm that TU models based the composition of SVOC and VOC components can be used to estimate the relative toxicity of WAFs of petroleum products and their residues. $TU > 1$ for the total WAF implies that it is expected to cause more than 50 % mortality in the test organisms. Predicted TUs for the WAFs based on chemical composition are shown in Fig. 4A (also provided in Table S7, SI4) and show that all ISB residues have lower TU than the WAFs of fresh, unburned oils. TU was below 1 for WAF of fresh and ISB of ULSFO (0.63 and 0.44, respective), and for ISB residue of IFO180 (0.21), suggesting that their WAFs result in mortality for less than 50 % of the tested organisms. WAFs of fresh IFO180 and fresh and ISB residue of MGO had TU higher than 1 (1.0, 2.54 and 1.16, respective), indicating a mortality for more than 50 % of the tested organisms. Based on the additive method PAHs (2–6 ring PAH) seem to contribute most to the toxicity in the WAFs, and the concentration of PAHs were higher in the WAFs of MGO than the other WAFs.

3.3. Comparison with WAFs from other oil products

WAF concentrations and TU for the WAFs were compared with WAFs of other batches of the fresh fuels that have been studied previously (Table 2). Two batches of MGO are shown, the one studied here ("MGO

OOW" (unburned MGO)) had a higher WAF concentration (4.6 mg/L vs 10.2 mg/L) and different chemical composition than the "MGO 2016" studied previously [42]. This resulted in a $TU < 1$ for "MGO 2016", while "MGO OOW" had a TU of 2.5. The calculations indicated that especially the PAHs contributed to the toxicity. Results from two batches of ULSFO are also given, "ULSFO 2016" [42] and "ULSFO OOW" (present study). The previously tested ULSFO contained more volatile components than "ULSFO OOW" and had a TU lower than the WAFs of unburned and burned "ULSFO OOW" studied here (0.39 vs 0.61 and 0.44, respectively). A previously tested IFO180 [43] had a lower total WAF concentration than "IFO180 OOW" (1.2 mg/L vs 3.4 mg/L) and less volatiles and naphthalenes, resulting in a lower TU (0.3 vs 1.0). These examples illustrate that different batches of fuels have different chemistry and therefore may not have the same impact on the environment following a spill scenario. Especially the difference in the TU for the two batches of MGO are considered to be significant. Among previously studied WAFs presented in Table 2, none had a $TU > 1$. In the present study, WAFs of both fresh and ISB residue of MGO and WAF of fresh IFO180 had $TU > 1$.

4. Conclusions

In situ burn residues (ISB from a large experimental offshore burning of three fuel oils (ULSFO, IFO180 and MGO) were collected and their water accommodated fractions were characterized, tested for toxicity and compared to the corresponding fresh oils in laboratory studies. The potential toxicity effects on primary consumers were assessed by testing on nauplii (N3, early life stage) of the marine copepod *Calanus finmarchicus*. The nauplii have limited fat reserves and are therefore more sensitive and less protected than the grown organisms where heavier components (PAHs) will be stored temporary in the fat reserves and in that way be physiological unavailable in short term studies.

In the WAFs of ISB residues, the concentrations were reduced due to loss of components during the burn. However, the ISB residues and their corresponding WAFs still contained volatiles, as the burn efficiency will vary within a burn, resulting in a residue with different properties.

Table 2

Total WAF concentrations and predicted acute toxicity expressed as TU of oil products tested in previous studies at SINTEF, compared with the WAF studied here: Unburned ULSFO (ULSFO OOW), MGO (MGO OOW) and IFO180 (IFO180 OOW), and their ISB residues. In addition to total TU, the contribution to TU from the component groups are given. A $TU > 1$ indicated a mortality of 50 % for the tested organisms (ND: Not detected).

	MGO 2016	MGO OOW	MGO ISB residue	ULSFO 2016	ULSFO OOW	ULSFO ISB residue	IFO180 2008	IFO180 OOW	IFO180 ISB residue
WAF conc (mg/L)	4.60	10.2	2.09	1.80	0.92	0.32	1.22	3.36	0.33
Total TU	0.68	2.54	1.16	0.39	0.61	0.44	0.31	1.00	0.21
BTEX	0.095	0.179	0.012	0.056	0.014	0.002	0.025	0.040	0.001
C3-benzenes	0.218	0.255	0.039	0.118	0.026	0.005	0.048	0.151	0.002
Naphthalenes	0.185	0.302	0.111	0.049	0.083	0.031	0.083	0.357	0.050
2–3 ring PAH	0.137	1.536	0.704	0.120	0.341	0.224	0.106	0.340	0.119
4–6 ring PAH	0.005	0.205	0.287	0.040	0.144	0.180	0.008	0.069	0.038
C0-C5 phenols	0.038	0.059	0.012	0.004	ND	ND	0.037	0.042	0.001

Compared with the other fuels, MGO had the highest total WAF concentration, both from fresh, initial oil (10 mg/L) and from ISB residue (2.1 mg/L). WAF of initial, fresh IFO180 had higher WAF concentration than fresh ULSFO (3.4 mg/L vs 1.1 mg/L) and WAF of ISB residue of IFO180 had lower WAF concentration (0.21 mg/L vs 0.33 mg/L)

The acute toxicity of the WAFs was expressed as the dilution needed to reach 50 % survival of the test species (% WAF) or as toxic units (TU). This showed that the toxicity of the WAFs of all three burnt residues was reduced compared that of the corresponding fresh fuel oils. The major cause of the reduction in toxicity was reduced dissolution of components from the burn residues compared to the fresh oils. Despite that the combined specific toxicities (LC₅₀ as mg/L) of the WAFs from burnt residues were slightly higher than that of the corresponding fresh oils, the decreased dissolution from the ISB residues by far outweighed this difference. Theoretical TU calculation indicated a small increase in the toxic contribution of the 4–6 ring PAHs in the ISB residue WAFs of ULSFO and MGO which may be attributed to the generation of pyrogenic PAHs. However, this had no major impact on the overall toxicity of the ISB WAFs.

MGO WAFs of fresh oil and ISB residue were more toxic than the corresponding other fuel oils based on relative toxicity (LC₅₀ of 12 % and 23 % WAF, respectively). WAF of fresh IFO180 was more toxic to the nauplii than ULSFO (LC₅₀ of 19 % vs 24 % WAF). However, WAF of ISB residue of IFO180 was less toxic than the ISB residue of ULSFO (LC₅₀ of 52 % vs 36 % WAF). The predicted TUs indicate that the PAHs (2–6 ring PAH) contribute most to the toxicity in the WAFs, and that the PAH concentrations were higher in WAFs of MGO than the other WAFs, and higher in the ISB residue of ULSFO compared with IFO180. The predicted TUs show the same relative trend as TU based on measured toxicity, indicating that predictions based on the chemically resolved oil fractions in WSFs can be used to estimate toxicity of ISB residues and their parent oils.

The present study gives insight to potential harmful effects of ISB residues on the early nauplii life stage of the marine copepod *Calanus finmarchicus*, which is a primary consumer and one of the key ecological species in northern boreal to Arctic oceans. The results may be used as a part of Net Environmental Benefit Analysis (NEBA) or Spill Impact Mitigation Assessment (SIMA) of oil spill responders to evaluate the most beneficial oil spill mitigation method.

CRedit authorship contribution statement

Liv-Guri Faksness: Conceptualization, Methodology, Validation, Formal analysis, Data curation, Investigation, Resources, Writing - original draft, Writing - review & editing, Visualization, Project administration, Funding acquisition. **Dag Altin:** Conceptualization, Methodology, Validation, Formal analysis, Data curation, Investigation, Writing - review & editing, Visualization. **Hilde Dolva:** Conceptualization, Resources, Funding acquisition. **Trond Nordtug:** Conceptualization, Methodology, Validation, Formal analysis, Data curation, Investigation, Writing - original draft, Writing - review & editing, Visualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.toxrep.2022.01.006>.

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