Finding 19

Guidelines on oil characterization to inform spill response decisions

Oil Spill Response Joint Industry Project

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About this report

In response to the Deepwater Horizon incident at the Macondo Prospect off the Gulf of Mexico In April 2010, the International Association of Oil and Gas Producers (OGP) formed the Global Industry Response Group (GIRG). This Group was tasked with identifying ways to prevent the recurrence of such an incident and to identify learning opportunities both with respect to the cause of, and response to, the incident. Part of this effort involved the formation of a subgroup on Oil Spill Response (OSR). This group was comprised of nominees from OGP member companies, from the IPIECA Oil Spill Working Group (OSWG), from Oil Spill Response Limited (OSRL), and from other industry organizations, associations and spill response cooperatives, as appropriate.

The OGP GIRG-OSR task force reported on its findings to both the OGP Management Committee and the IPIECA Executive Committee at a joint session in February 2011. While certain actions recommended by the GIRG-OSR report fell within the remit of existing organizations, it was recognized that the most efficient way to execute the resultant work was for the industry to establish a limited duration Joint Industry Project (JIP), governed by the funding companies.

This report addresses Finding 19 of the OGP Global Industry Response Group (GIRG) report and is intended to set out the importance of 'knowing your oil'. A definition of characterization and the various parameters and methods commonly used is presented. Oil characterization allows for more efficient emergency planning and better informed response decisions in the event of an oil spill.

The assistance of CEDRE in the production of this document is gratefully acknowledged.

What is oil characterization, and why is it important?

Oils can act very differently once released/spilled in the marine environment, however an understanding of an oil's physical and chemical characteristics will indicate its likely behaviour. Characterization is, in basic terms, a description of an oil's distinctiveness in relation to other oils. It commonly involves the analysis of the source oil's physical and chemical properties, which can be followed by laboratory and mesoscale studies (or, much more rarely, field studies) focusing on the changes in the oil's properties once it is released to the marine environment and subjected to weathering processes. Characterization is a key tool in operational practice and contingency planning, for example in the case of an accidental release to the environment.

Oil characterization is widely used in production operations and, in particular, is essential for effective reservoir management. While these aspects are not discussed in detail within this document, some of the parameters obtained in operational management may be of use in oil characterization.

Oil characterization is important in the case of a release to the environment because:

- a properly characterized oil may be utilized to prove or disprove a suspected spill source;
- the determination of an oil's properties can help to prevent delays in decision making for an oil spill response;
- information on the properties of oil can help to ensure the effectiveness of decisions made with regard to response measures;
- knowledge of the physical and chemical properties of an oil can be used to predict the severity of environmental damage and the oil's persistence in the environment; and
- characterization data is a requirement for spill trajectory and fate models.

How often should oils be characterized?

Oil characterization is an important aspect in the development of contingency plans for oil spill response. When developing a response plan, it is vital to understand the range of response options available and which of those options are most suited to the properties of the oil in question. The data obtained from characterization assessments is therefore extremely useful for guiding planning decisions.

Crude oils or condensates

For crude oils or condensates, the samples tested should be representative of the oil being produced, handled, stored or transported at that time. During production, the properties of the oil have the potential to change over a period of years. The parameters could therefore be analysed every few years, depending on the rate of physical and chemical changes over time. However, the weathering tests carried out in the laboratory would not need to be repeated as frequently provided that the physical and chemical characterization assessments do not illustrate a significant change in key parameters, such as viscosity and density.

Refined products

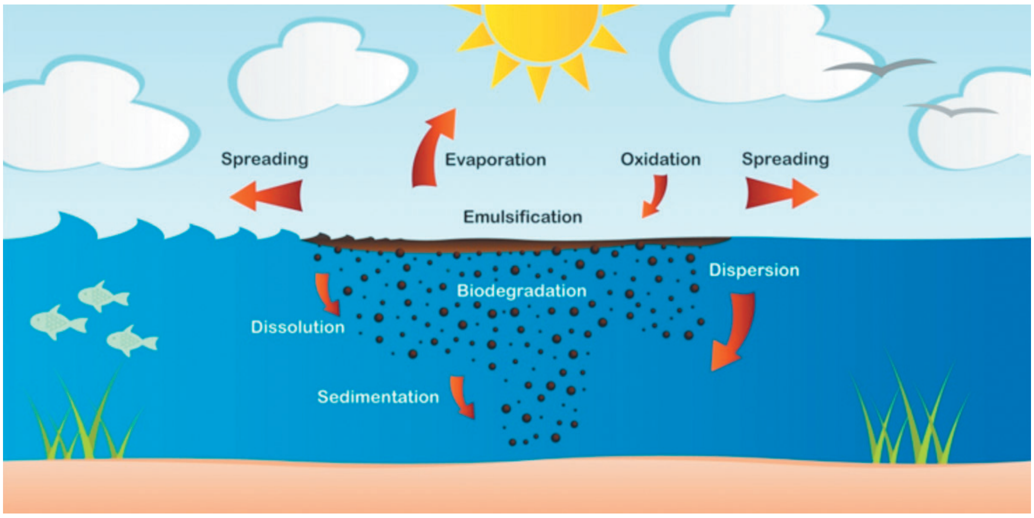
Samples of refined products (such as bunker fuel oil or distillation products transported in bulk) can be characterized using the same methods as those used for crude oils, although some of the characterization methods for lighter/heavier products have been modified to cope with the differences in viscosity. Characterization of refined products should not be a frequent requirement as long as the properties of the final product remain unchanged.

Environmental processes at work on oil released to the marine environment

Whenever an oil spill occurs, questions are raised relating to the potential fate and behaviour of the crude oil, condensate or refined product involved. Spilled oil undergoes a number of physical and chemical changes, some of which lead to its removal from the marine environment while others cause it to persist. These changes depend on factors such as the quantity of oil spilled, the oil's initial physical and chemical characteristics, the prevailing climatic and sea conditions, and whether the oil remains at sea or is washed ashore.

Spilled oil is subjected to weathering processes such as spreading, evaporation, emulsification, dispersion, dissolution or photo-oxidation (as illustrated in Figure 1). These processes occur under natural conditions due to agitation at the sea surface by wind, waves and currents and the exposure of the oil to the sun. As weathering progresses, the physical state of the oil is continuously changing; the light fractions evaporate gradually, its density increases, and part of the oil disperses naturally into the water column while the remaining oil on the water surface may become emulsified with sea water (which tends to increase the volume of the oil and its persistence) and is oxidized by ultraviolet radiation. Some components of the oil partially dissolve and may evaporate. Finally, residues (or high density oils to begin with) can be subjected to the process of sedimentation.

Figure 1 Overview of the weathering process



Source: ITOPF, www.itopf.com/information-services/publications/documents/TIP2FateofMarineOilSpills.pdf

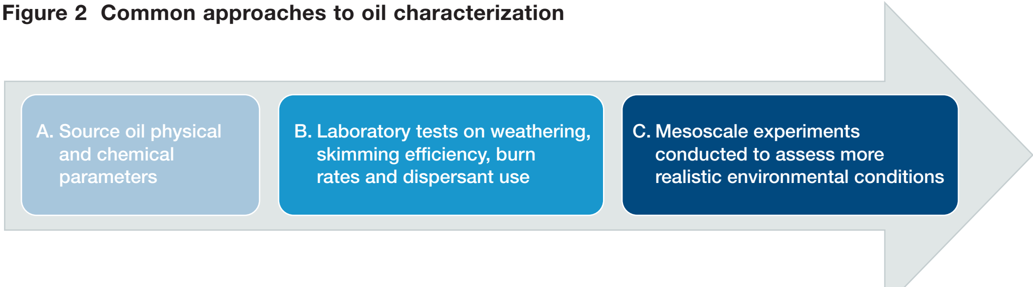
The behaviour of weathered oil is often different from the behaviour of the source oil. Oils generally become increasingly viscous over time and can therefore become a more persistent pollutant. Weathering can also have an impact on the physical and chemical characteristics of an oil to the point where the oil is no longer easily identifiable when compared to the source oil.

Understanding these transformations is a key element in: evaluating the potential impacts of the spilled oil; optimizing the response options according to the effects of weathering on the oil; and implementing the emergency response plan.

Characterization strategies

In relation to spill response, an oil is commonly characterized by its properties as a source oil, in addition to its behaviour once spilled and its subsequent amenability to response techniques. The most commonly adopted strategies to oil characterization are described in detail below.

Figure 2 Common approaches to oil characterization



Source oil physical and chemical characterization parameters

Some of the more basic characteristics of an oil can provide a reliable view of its behaviour when spilled without considering interactions with the environment. Examples of standardized methods for characterizing the basic physical and chemical parameters of oil are provided in Appendix 1 on page 13. The parameters which can provide useful insight into the fate of spilled oil are discussed below.

- **Distillation characteristics:** The basic distillation characteristics (boiling point distribution) are expressed as the proportions of the parent oil that distil within given temperature ranges. Some oils contain bituminous, asphaltenic or waxy residues which do not readily distil, even at high temperatures, and which are likely to persist in the marine environment for extended periods of time. The basic distillation characteristics can also give an indication of the maximum evaporation expected (which is generally considered to correspond with the fraction that distils below 250°C).

- **Wax content and pour point:** The pour point, which is directly related to the wax and asphaltene content, will mark the difference between the solid and liquid state of the oil. If the ambient temperature is below the pour point, the oil will essentially behave as a solid and will not spread over the sea surface as a film but fragment into discrete patches (Figure 3). This has the potential to limit the effectiveness of dispersants, and care will be needed when choosing appropriate recovery equipment.

  The wax content refers to the component of paraffin hydrocarbon within the oil. When the temperature of a waxy crude oil is lowered, the heavier fractions may begin to crystallize.

Figure 3 A heavy fuel oil spill fragments into patches

Source: ITOPF

- **Flash point:** The flash point (based on source oil data) represents a reliable parameter when assessing the risks of ignition. This has obvious implications for the health and safety of response personnel and may be particularly useful if considering an in-situ burn (ISB).

- **Vapour pressure:** The vapour pressure provides a further indication of the volatility of the oil. A vapour pressure greater than 3 kPa is the criteria for evaporation to occur under most conditions. Above 100 kPa, the substance behaves like a gas. For example: gasoline has a vapour pressure of 40–80 kPa; Cossack crude of 44 kPa; and the much less volatile Boscan crude oil of 1.7 kPa.

- **Asphaltene content:** These heavy molecules have active properties and play a significant role in the formation of water-in-oil emulsions. They are qualified as 'hard' or 'soft' according to testing. It is generally considered that emulsions can be formed when the asphaltene content, which is measured as heptane insolubles, is greater than 0.5%, with the resultant emulsions containing up to 80% water.

- **Viscosity:** The viscosity of an oil is its resistance to flow, and will provide information in terms of spreading and whether the application of dispersants would be appropriate. High viscosity oils flow less easily than low viscosity oils, and are less susceptible to the effects of dispersant chemicals. All oils become more viscous as the temperature falls (some more than others) and as the volatile component evaporates over time.

- **Specific gravity:** The specific gravity, or relative density, is the density in relation to water. Most oils are less dense (lighter than) sea water which has a specific gravity of approximately 1.025 at 20°C. Heavy or very dense oils may sink, partially or fully, as illustrated in Figure 4. The American Petroleum Institute gravity (°API) is commonly used to describe the specific gravity of crude oils and petroleum products.

  In addition to determining whether or not the oil will float, the specific gravity can also give a general indication of other properties of the oil. For example, oils with a low specific gravity (high °API) tend to contain a high proportion of volatile components and be of low viscosity.

Figure 4 Sunken oil recovery

Source: ITOPF

Fingerprinting

The term 'fingerprinting' refers to the examination of a sample of spilled oil, to determine whether or not it originates from the suspected source, rather than a parameter to be used in response decisions. It involves the quantification of the organic constituents of an oil sample, most commonly by the use of capillary column gas chromatography (GC) coupled with flame ionization detection (FID). For a more accurate and detailed analysis, mass spectrometry (MS) may also be used. The term 'biomarker' is used in fingerprinting and refers to the calculation of peak area ratios for a range of the more stable hydrocarbon compounds including hopanes, isoprenoids and steranes. When conducting fingerprint characterization of weathered oils, the distribution patterns and ratios of biomarkers can be extremely useful in comparing samples with suspected source oils despite the weathering effects (differences in the oil's appearance and properties).

Laboratory tests on weathering and dispersant effectiveness

Weathering studies

Laboratory simulation of the main weathering processes at sea can be carried out for crude oils, condensates or refined products by exposing them to a systematic, stepwise procedure involving distillation, emulsification processes and, occasionally, photo-oxidation.

- **Topping:** The evaporation process is simulated by distilling the oil at various temperatures (typically 150°C, 200°C and 250°C) for periods of 6, 12 and 36 hours[i]. This provides information on the true boiling point (TBT) as well as the asphaltene and wax content. The residues can be analysed to determine the density, viscosity, flash point and pour point.

- **Emulsification:** After evaporation, the residues can be emulsified with sea water at various water contents (50%, 75% and the maximum) and characterized by recording a number of physical measurements (e.g. viscosity, water content, emulsion stability, etc.). For example, a modified version of the Mackay-Zagorsky method[ii], using rotary funnels, can be used to simulate the emulsification process. The rotation simulates the mixing energy of wave activity at sea over the first 6, 12 or 72 hrs, starting with a 50% water content.

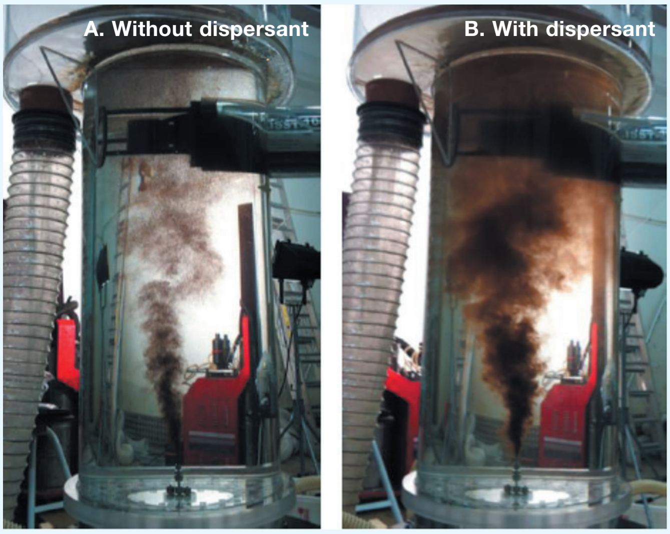
- **Photo-oxidation:** Finally, and rarely, samples may be exposed to ultraviolet light to recreate the process of photo-oxidation if there is a particular emphasis on longer-term weathering processes. As this method provides data for just one test temperature, modelling software can then be used to extrapolate the data for various temperatures and wind speeds, which can then be utilized in fate and trajectory models.

Dispersant effectiveness

It has been reported[iii] that approximately 50 different laboratory test methods are currently available for determining the effectiveness of dispersants on oil, although not all are currently in use. They can be further categorized into four general groups:

1. **Laboratory tests** include both those that use standard laboratory equipment, such as the warren springs rotating flask test[iv]; the labofina test; the swirling flask test (later updated by the baffled flask test[v]); the Exxon wrist action shaker method test[vi] (EXDET); and those involving specifically constructed laboratory equipment, such as the Mackay-Nadeau-Steelman test[vii] (MNS); the Revised Standard Dispersant Effectiveness test[viii] (RSDET); the Institut Français du Pétrole (IFP) test[ix]; the oscillating hoop test[x]; and the flowing cylinder test[xi].
2. **Mesoscale tests**, such as flume tests, the cascading weir test[xii] and the delft flume test.
3. **More recent tests** designed to understand specific aspects of dispersant use, such as subsea injection of dispersants using the SINTEF/CEDRE MiniTower (see Figure 5), are currently being developed. These do not comprise part of a standard approach to oil characterization but can be useful to understand specific aspects of oil spill response options.

Figure 5 The SINTEF/CEDRE MiniTower bench scale test to investigate subsea injection of dispersants



The test methods commonly focus on agitation/mixing, and record the success or failure to disperse the oil, thereby providing an indication of the conditions in which it would be effective during an actual spill response. To confirm the accuracy of laboratory testing, the results need to be correlated with dispersant effectiveness in field tests and at real incidents.

The oil samples tested for dispersibility using these test methods should be 'weathered' in the laboratory to provide a range of viscosities that correspond to different weathering times; this will help to define the 'window of opportunity' for dispersant use in the field. Previous studies (e.g. Guyomarch et al., 1999[xiii] and 2002; and Daling et al.,1997[xiv] have illustrated the limits of dispersibility with increasing viscosity over time for a range of crudes; the effectiveness of the applied dispersant does not decrease progressively but drops quickly to a value close to zero when the oil reaches its viscosity limit for dispersant use.

Mesoscale experiments and calibration with field tests

Mesoscale experiments simulate different weathering conditions simultaneously and can be conducted in a purpose-built facility. To simulate the various weathering processes as realistically as possible, hydraulic canals (wave tanks) or flumes are used to recreate different wind, current, wave and solar conditions. Evaporation and emulsification are recreated as the oil weathers as a slick at the water surface with the addition of realistic mixing energy.

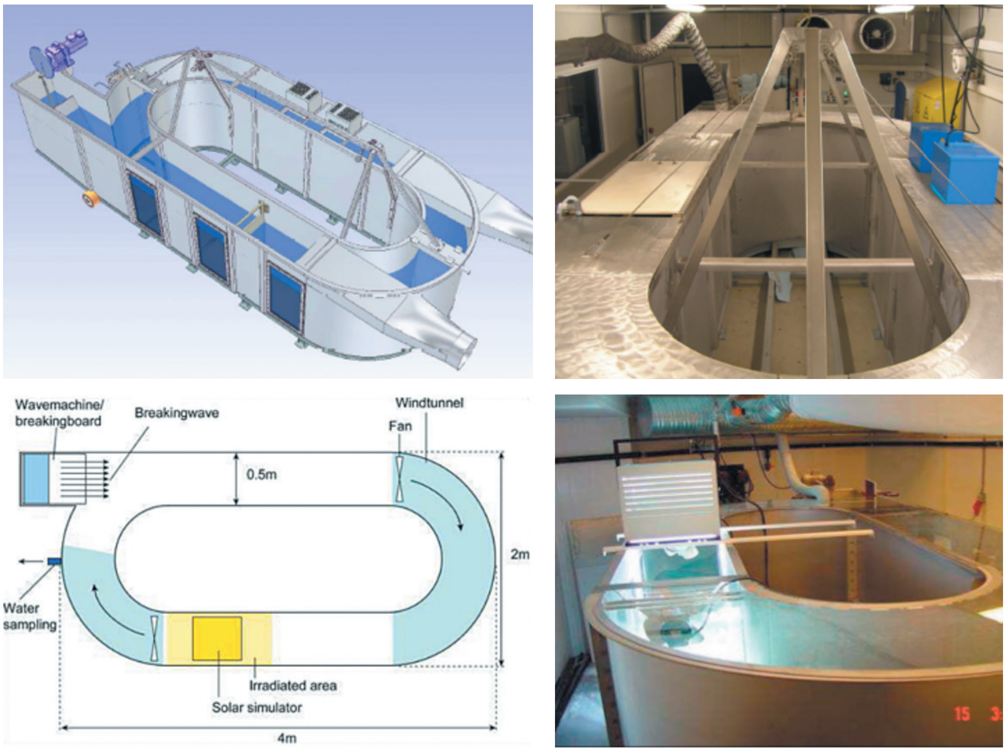
There are a number of wave tanks, including: the Texas A&M SERF tank at Corpus Christi (previously called COSS or Coastal Oil Studies System); the EPA/BIO tank at the Bedford Institute of Oceanography in Dartmouth, Nova Scotia; the Ohmsett[xv] (national oil spill response test facility) large wave tank in New Jersey; and the smaller S. L. Ross (SLR[xvi]) wave tank.

Figure 6 The Texas A&M SERF tank (left) and the Ohmsett[xviii] test tank (right)

A flume, such as the facilities at CEDRE and SINTEF, is a looped tank in which water is continuously circulated and waves can be generated (see Figure 7). Some flume tanks have facilities for specialized testing, for example the analysis of long-term behaviour of oil in cold environments. All environmental parameters can be controlled to ensure reliable comparisons between tests. Test runs can last for up to a week using around 10 litres of oil. Oil samples taken throughout the test runs can be used to determine the physical and chemical properties (see pages 4–6) and to evaluate the likely effectiveness of available response options. For example, a dispersability test[xvii] might be carried out in the flume to provide a realistic assessment of the likely efficacy.

Mesoscale tests can also be carried out in the open environment, by containing an area in which the experiments are to be performed. A major disadvantage of such testing is that it does not provide any control of the environmental conditions.

Figure 7 Examples of flumes used for mesostudies



Source: top, Cedre; bottom: SINTEF

Field tests are less commonly carried out due to the cost and difficulty obtaining the necessary authorizations, and hence would not be part of a standard oil characterization. However, calibration of laboratory and mesoscale experiments against field experiments provides valuable information on the scalability of the lab and mesoscale tests. Most recent field testing has focused on oil in ice environments, such as those conducted by SINTEF[xviii]. Some field-based dispersant effectiveness tests that have been undertaken include the US EPA field dispersant effectiveness test, American Petroleum Institute (API) field dispersant effectiveness test, the Fina spill test kit, the Mackay simple field test, and the Pelletier screen test[xix].

Response options

The results of laboratory scale or mesoscale experiments performed on fresh or weathered samples of oil can help to guide decisions on the most appropriate choice of spill response options. For example, emulsions and heavily weathered oils cannot normally be chemically dispersed or ignited; a well-characterized oil will therefore enable confident decisions to be made with regard to the effectiveness of the response options in such cases.

Importantly, the oil characterization may indicate that the particular oil spilt is not persistent under the prevailing environmental conditions at the time of the spill. Hence, an oil characterized as non-persistent and of low viscosity may not require an active response if, through monitoring, it is predicted that there is little risk of the spilt oil having an impact on sensitive resources before it dissipates at sea. In such a case, the primary response option would be to closely monitor the spill. It should be borne in mind that, as environmental conditions vary, the behaviour of the oil may alter, for example in extreme cold a usually non-persistent oil may exhibit persistent traits.

Dispersants

It is important to differentiate between natural and chemical dispersion. Natural dispersion occurs when wave energy breaks up a slick (as occurred during the *Braer* oil spill in 1993). Chemical dispersants greatly increase the rate of natural dispersion of oil into the water column by breaking up the oil slick on the surface into small droplets that disperse more easily through the water column.

The effectiveness of chemical dispersants depends on the oil’s initial chemical composition, its weathering stage, and the intrinsic efficiency of the dispersant when used with that particular source oil. The prevailing environmental conditions will also have significant influence, not only in terms of the extent of weathering but also with regard to providing sufficient mixing energy required to achieve active dispersion; calm seas are not ideal in this respect.

Laboratory tests for efficacy should be representative of the real conditions at sea. As mentioned previously, the dispersibility of the source oil in question should be assessed at the laboratory scale when evaluating a contingency plan (and when planning for the choice of dispersant) prior to an incident. However, smaller-scale tests on subsequently weathered samples and a test spray run on site are more practical and can serve as a useful planning tool before full-scale use of dispersants at sea. Samples tested in the laboratory should reflect the various stages of weathering, so that the most effective ‘window of opportunity’ for dispersant application can be determined. Chemical dispersants are generally more effective when applied to oils that have low viscosity and are unweathered/unemulsified.

In-situ controlled burning

In-situ burning of spilled oil can be an effective response option under certain conditions. However, the window of opportunity for a burn depends on the oil's 'ignitability' which, in turn, is limited by weathering and emulsification of the oil. While more research is still needed, tests have been developed to determine the ignitability of oil under different conditions. By performing these tests on a fresh crude oil and on weathered samples of the same oil it is possible to specify a window of opportunity as a function of weathering. In-situ burning has been studied extensively for arctic conditions, where the limited water uptake due to ice coverage, combined with the slow evaporation of light fractions due to low temperatures, provides a longer window of opportunity for burning. Conversely, if the water content of the oil increases, for example due to emulsification, its ability to ignite will decrease and the window of opportunity may be lost.

Containment and recovery

The efficacy rates of skimmers and pumps are generally published by the respective manufacturers, and are normally dependent on viscosity. As the viscosity of a heavy oil increases it will become more difficult to pump. This is because an increase in viscosity is generally accompanied by an increase in adhesion. Waxy oils, for example, can clog pipes and valves, and have a significant impact on pump flow rates; a heavily emulsified oil will be similarly difficult to pump, although the entrainment of water by emulsification may reduce adhesion[xx]. Some types of skimmers rely on oleophilic attraction to collect the spilled oil, and the effectiveness of this specific type of skimmer can be reduced significantly for the same reasons.

Tests which measure the degree of oil adhesion to an oleophilic plate are a reliable means of assessing whether the use of oleophilic skimmers would be an effective response option. These tests are generally simple and can easily be performed in combination with weathering experiments. They are particularly relevant in mesoscale experiments as they can be performed directly on the slick at various times and degrees of weathering.

Characterization parameters used in fate and trajectory models

Various modelling software has been developed by different organizations (for example ASA, BMT, NOAA and SINTEF) and the characterization data required is fairly standard. The most basic trajectory models require information regarding specific gravity (°API), pour point, viscosity and density. Other properties such as distillation information, and asphaltene and trace metal concentrations may also be useful to the operator. Models that can accommodate characterization data for weathered samples provide increased reliability for longer-term predictions. Some models can also combine data on the weathering of oil slicks at the sea surface with information on the solubilization of certain compounds in the water column.

Conclusion

The characterization of oils is considered to be a necessary step in both preparing for, and responding to, an oil spill. Adequate knowledge regarding the likely behaviour of the oil, and the most effective response strategies according to changes in the oil over time, is invaluable for both the responsible party and the oil spill response personnel involved in the clean-up operations.

Appendix 1:
Characterization methods

Characterization	Examples of Appropriate Standardized Methods	
Physical and chemical parameter tests	ASTM D6560	Standard Test Method for Determination of Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products
	ASTM D1298 - 12b	Standard Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
	ASTM D5002 - 99(2010)	Standard Test Method for Density and Relative Density of Crude Oils by Digital Density Analyser
	ASTM D287 - 12b	Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
	ASTM D97; D5853	Standard Test Methods for Pour Point of Petroleum Products / Crude Oils
	ASTM D323; D5191; D6377	Standard Test Methods for Determination of Vapour Pressure of Petroleum Products / Crude Oils
	ASTM D445	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
	ASTM 95 - 05	Standard Test Method for Water content in Petroleum Products and Bituminous Materials by Distillation
	ASTM D3278 - 96(2011); ASTM D7215 - 08	Standard Test Method for Flash Point of Liquids by Small Scale Closed-Cup Apparatus / Standard Test Method for Calculated Flash Point from Simulated Distillation Analysis of Distillate Fuels
	ASTM D7169	High Temperature Simulated Distillation
	ASTM D5739	Standard Practice for Oil Spill Source Identification by Gas Chromatography and Positive Ion Electron Impact Low Resolution Mass Spectrometry
	CEN/TR 15522-2:2012	Oil spill identification - Waterborne petroleum and petroleum products - Part 2: Analytical methodology and interpretation of results based on GC-FID and GC-MS low resolution analyses
Dispersant efficacy tests	ASTM F2059	Swirling flask test (SFT)
	EPA Appendix C to part 300, 40 CFR: section 2.0	EPA Swirling flask dispersant effectiveness test
ISB tests	ISO 9038:2013	Determination of sustained combustibility of liquids
Toxicity tests	ISO 10253:2006	Marine algal growth inhibition test with Skeletonema costatum and Phaeodactylum tricornutum
	ISO 14669:1999	Determination of acute lethal toxicity to marine copepods
	ISO 16712:2005	Determination of acute toxicity of marine or estuarine sediment to amphipods
	OECD Guidelines for the Testing of Chemicals, Section 2, Test No. 203	Fish, Acute Toxicity Test

Appendix 2: References and further reading

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