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Use of passive samplers for improving oil toxicity and spill effects assessment

Daniel Letinski [a], Thomas Parkerton [b,*], Aaron Redman [a], Ryan Manning [a], Gail Bragin [a], Eric Febbo [c], David Palandro [d], Tim Nedwed [d]

[a] ExxonMobil Biomedical Sciences, Inc., 1545 US Highway 22 East, Annandale, NJ 08801, USA
[b] ExxonMobil Biomedical Sciences, Inc., 800 Bell Street, Houston, TX 77002, USA
[c] ExxonMobil Research Qatar, Maysaloum Street, Doha, Ad Dawhah, Qatar
[d] ExxonMobil Upstream Research Company, 3120 Buffalo Speedway, Houston, TX 77098, USA

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ABSTRACT

Methods that quantify dissolved hydrocarbons are needed to link oil exposures to toxicity. Solid phase microextraction (SPME) fibers can serve this purpose. If fibers are equilibrated with oiled water, dissolved hydrocarbons partition to and are concentrated on the fiber. The absorbed concentration ($C_{polymer}$) can be quantified by thermal desorption using GC/FID. Further, given that the site of toxic action is hypothesized as biota lipid and partitioning of hydrocarbons to lipid and fibers is well correlated, $C_{polymer}$ is hypothesized to be a surrogate for toxicity prediction. To test this method, toxicity data for physically and chemically dispersed oils were generated for shrimp, *Americamysis bahia*, and compared to test exposures characterized by $C_{polymer}$. Results indicated that $C_{polymer}$ reliably predicted toxicity across oils and dispersions. To illustrate field application, SPME results are reported for oil spills at the Ohmsett facility. SPME fibers provide a practical tool to improve characterization of oil exposures and predict effects in future lab and field studies.

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1. Introduction

A critical element of hazard assessment is characterization of test substance exposure in order to establish a concentration–response relationship. In the case of aquatic toxicity tests with crude oil and related petroleum products, test organisms are exposed to water accommodated fractions (WAFs) that are prepared by equilibrating increasing amounts of oil with a known volume of aqueous media (Girling et al., 1992). Traditionally, total petroleum hydrocarbon (TPHs) analyses have been used to characterize test organism exposures to oil for each WAF (i.e., oil to water loading) (Singer et al., 2000; Tsvetnenko and Evans, 2002). However, TPH is a poor predictor of adverse effects on aquatic life since this analysis includes undissolved hydrocarbons that appear much less toxic than hydrocarbons that are dissolved in the aqueous media (Carls et al., 2008; Olsvik et al., 2011). Further, TPH measurements do not account for the concentration and composition of the dissolved phase hydrocarbons that dictate aquatic toxicity (Di Toro et al., 2007). This is an important consideration since the dissolved hydrocarbon composition of WAFs will vary with oil composition, the WAF test system and preparation method, oil loading and extent of weathering (Shiu et al., 1998; Couillard et al., 2005; McGrath et al., 2005; Faksness et al., 2008). Consequently, reliance on TPH measurements in toxicity tests complicates the interpretation and comparison of concentration response relationships with different oils and study designs. In addition, since the dissolved hydrocarbons that contribute to TPH in lab toxicity tests may not be reflective of oil spill exposures in the field, this poses challenges for extending effects characterization in the lab to the field. To improve current practice, dissolved hydrocarbon exposures in oil toxicity tests need to be characterized and linked to a mechanistic understanding of hydrocarbon toxicity.

The target lipid model (TLM) provides a quantitative framework for describing the toxicity of dissolved hydrocarbons. The TLM is based on the hypothesis that aquatic toxicity occurs when the molar concentration in organism lipids exceed a critical threshold which can be expressed as:

$$C_{lipid,toxic} = K_{lipid-water} \times LC_{50} \quad (1)$$

where $C_{lipid,toxic}$, target lipid concentration in organism at which 50% of the test population elicits an adverse effect i.e., mortality, from exposure to a dissolved hydrocarbon (mmol/kg lipid);

* Corresponding author. Tel.: +1 832 954 8089.
E-mail address: thomas.f.parkerton@exxonmobil.com (T. Parkerton).

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$K_{lipid-water}$, lipid–water partition coefficient of the hydrocarbon (L water/kg lipid); LC_{50}, lethal concentration in water at which 50% of the test population elicits an adverse effect (mmol/L water).

Rearranging and taking logarithms yields:

$$\text{Log } LC_{50} = \text{Log } C_{lipid,toxic} - \text{Log } K_{lipid-water} \quad (2)$$

A linear free energy relationship can be used to relate $K_{lipid-water}$ to the octanol–water partition coefficient (K_{ow}) of the hydrocarbon:

$$\text{Log } K_{lipid-water} = a + b \text{ Log } K_{octanol-water} \quad (3)$$

Substituting (3) into (2) yields a convenient log-linear relationship between the acute toxicity of a dissolved hydrocarbon, as expressed by the $LC_{50,}$ and hydrophobicity, as indexed by the octanol–water partition coefficient:

$$\text{Log } LC_{50} = \text{Log } C_{lipid,toxic} - a - b \text{ Log } K_{octanol-water} \quad (4)$$

Eq. (4) has been used in analysis of aquatic toxicity data sets for individual hydrocarbons and test species (Di Toro et al., 2000). Recent application of the TLM framework indicated that the slope of this equation is constant across different test species ($b = 0.94$). Log $C_{lipid,toxic}$ which is derived from the intercept of this regression is used to represent a species-specific hypothetical concentration in lipids at the target site of toxic action that characterizes the sensitivity of the test organism to dissolved hydrocarbons. Based on the analysis of toxicity datasets for 47 test species including bacteria, algae, invertebrates, fish and amphibians, $C_{lipid,toxic}$ ranged from 24 to 500 mmol/kg lipid (McGrath and Di Toro, 2009). These authors report that the "a" term in Eq. (4) was equal to 0, 0.109, and 0.352 for aliphatic, monoaromatic and di/polyaromatic hydrocarbon classes, respectively and accounts for observed differences in toxic potency of these structural classes.

The TLM has been used to assess the toxicity of dissolved hydrocarbon mixtures using the concentration addition model (Altenburger et al., 2003). In this approach, toxic units (TUs) are calculated as follows:

$$TU = \sum_{n=1}^{\infty} \frac{C_{water,i}}{LC_{50,i}} \quad (5)$$

where $C_{water,i}$, dissolved concentration of hydrocarbon i; $LC_{50,i}$, acute effect concentration of hydrocarbon i calculated using Eq. (4).

If hydrocarbons are additive in effect, one TU is expected to result in a 50% response. Previous studies support the use of Eq. (5) for predicting the toxicity of hydrocarbon mixtures and petroleum substances (Herman et al., 1990; Landrum et al., 2003; USEPA, 2003; Barata et al., 2005; McGrath et al., 2005; Olmstead and LeBlanc, 2005; Bellas et al., 2008; Gonçalves et al., 2008). To aid in interpretation of oil toxicity tests, recent research has focused on linking model estimates of dissolved hydrocarbons exposures in WAFs with TLM derived estimates of dissolved hydrocarbon component toxicity using an additive TU model.

The first approach involves detailed characterization of the test oil in terms of hydrocarbon components (e.g., mass distribution of aromatic and aliphatic classes as a function of carbon number). This compositional information has been used to predict the multi-component dissolution and resulting dissolved hydrocarbon concentrations using Raoult's Law (Cline et al., 1991; Verbruggen et al., 2008). More recently, given the oil, water and air volumes in the WAF test system a library of representative hydrocarbon structures and a three phase mass balance model was used to simulate the WAF dissolved phase composition (Redman et al., 2012). The second approach uses speciated total petroleum hydrocarbon analyses of WAFs that provided measured concentrations of aliphatic and aromatic components as input to an oil–water partitioning model that computes the distribution of each component between dissolved and oil droplet phases (Redman et al., 2012a). Both approaches allow WAF exposures to be expressed in terms of TUs which provides a more toxicologically relevant exposure parameter to compare and establish relationships to observed effects than conventional measures of oil exposures, e.g., oil loading, TPH or TPAH.

Another approach for improving characterization of dissolved exposures relies on analytical measurements using passive samplers (Parkerton et al., 2000; Verbruggen et al., 2000; Leslie et al., 2002; Lu et al., 2011; Allan et al., 2012; Mayer et al., 2014). These devices are constructed from different polymers that can be equilibrated with dissolved hydrocarbon constituents in a WAF sample in a single step prior to analysis. In addition to enhanced analytical sensitivity, if a high volume ratio of water to polymer is employed during equilibration, the polymer will not significantly deplete hydrocarbons from the WAF sample during equilibration. In this case, the concentration of individual hydrocarbons on the polymer can be directly related to the dissolved concentration based on the $K_{polymer-water}$ of the hydrocarbon constituent. The $K_{polymer-water}$ can in turn be related to K_{ow} in the same manner as $K_{lipid-water}$ using Eq. (3) (USEPA, 2012). These partitioning relationships allow the $C_{lipid,toxic}$ to be translated into an equivalent polymer concentration of total hydrocarbons:

$$C_{polymer,toxic} = C_{lipid,toxic} \frac{K_{polymer-water}}{K_{lipid-water}} \quad (6)$$

The objectives of this study were to (1) apply passive samplers in laboratory acute toxicity tests with a range of physically and chemically dispersed oils; (2) test if theoretically derived polymer concentrations of total hydrocarbons derived using Eq. (6) are predictive of observed shrimp toxicity; and (3) demonstrate the use of passive samplers in assessing dissolved hydrocarbons and predicted effects of a simulated oil spill with and without addition of chemical dispersant.

2. Materials and methods

2.1. Preparation of water accommodated fractions

Physically dispersed (PDWAFs) solutions were generated for six crude oils and a No. 2 fuel oil with API gravities ranging from 11 to 38. Individual WAFs were prepared at different loadings by delivering a known amount of oil from a glass syringe to natural, 3.5 L of filtered seawater in a 4 L glass aspirator bottle. The solutions were placed on stir plates and mixed with Teflon coated stir bars for 24 h. The vortex was set to 20–25% of the static liquid depth. At the end of mixing, solutions were allowed to settle for 2 h before WAF removal from an outlet port at the bottom of the mixing vessel for use in toxicity testing. Chemically dispersed (CDWAFs) solutions were prepared for each crude oil as described above but with the addition of either Corexit 9500 or 9527 following oil addition to yield a 1:10 dispersant to oil ratio. At the end of mixing, a 6 h settling period was provided for CDWAFs before sampling for toxicity tests.

2.2. Acute toxicity tests

Americamysis bahia (formerly *Mysidopsis bahia*) were obtained from an in-house culture that were the fed brine shrimp, *Artemia*. Five animals that were 5–6 d old were placed in 3 or 4 replicate 500 mL glass jars for each WAF treatment and control (seawater with no oil). Test chambers were completely filled with WAF solution and sealed with lids to minimize volatilization losses. Mortality observations for each test chamber were recorded after 24 and 48 h. Tests were conducted using a 16:8 h light:dark cycle under ambient laboratory light at a temperature of 24–27 °C.

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