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Passive sampling reversed: Coupling passive field sampling with passive lab dosing to assess the ecotoxicity of mixtures present in the marine environment

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ABSTRACT

This study presents a new approach in aquatic toxicity testing combining passive sampling and passive dosing. Polydimethylsiloxane sheets were used to sample contaminant mixtures in the marine environment. These sheets were subsequently transferred to ecotoxicological test medium in which the sampled contaminant mixtures were released through passive dosing. 4 out of 17 of these mixtures caused severe effects in a growth inhibition assay with a marine diatom. These effects could not be explained by the presence of compounds detected in the sampling area and were most likely attributable to unmeasured compounds absorbed to the passive samplers during field deployment.

The findings of this study indicate that linking passive sampling in the field to passive dosing in laboratory ecotoxicity tests provides a practical and complimentary approach for assessing the toxicity of hydrophobic contaminant mixtures that mimics realistic environmental exposures. Limitations and opportunities for future improvements are presented.

1. Introduction

Since the development of semipermeable membrane devices (SPMDs) by Huckins et al. (1990, 1993), passive sampling has become an important tool for the environmental monitoring of aquatic pollutants. Passive sampling methods are low-tech and cost-effective monitoring tools, allowing the determination of freely dissolved contaminant concentrations that are – depending on the used methodology – averaged over the sampling period (Zabiegala et al., 2010). Moreover, nonpolar passive samplers can concentrate hydrophobic compounds (typically present in the water phase at very low concentrations) up to levels that can be easily analysed with standard equipment (Lohmann et al., 2012). Thus, many of the disadvantages associated with active sampling techniques – such as high cost, relatively high detection limits, complex sample preparation – can be avoided by using passive sampling methodologies. Over the past two decades, this has led to the development of a myriad of new passive sampling materials (see for example Zabiegala et al., 2010 for a review).

Passive sampling materials have also been used increasingly as a source of contaminants for (eco)toxicity testing in two types of experiments, the goals of which are markedly different. The first type aims to expose test organisms to environmentally relevant contaminant mixtures. In the earliest of these experiments, SPMDs – of which the extracts were spiked in the (eco)toxicological test medium (e.g. Parrott et al., 1999) or were even directly injected in the test organism (Petty et al., 1998, 2000) – were the most popular, although similar experiments have been conducted with other types of passive samplers (an overview of passive dosing studies is given in Table 1). As these SPMDs had been previously deployed in the aquatic environment, the test organisms were thus exposed to mixtures directly collected in the field. In a number of these studies chemical analysis was performed on the passive sampler extracts in which mostly polycyclic aromatic hydrocarbons (PAHs) (Petty et al., 2000; Rastall et al., 2004; Bopp et al., 2007; Ke et al., 2007; Hillwalker et al., 2010; Emelogu et al., 2013), polychlorinated biphenyls (PCBs) (Petty et al., 2000; Hillwalker et al., 2010; Emelogu et al., 2013) and pesticides (Petty et al., 2000; Shaw et al., 2009; Hillwalker et al., 2010;

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Table 1
Overview of available literature on studies in which a passive sampling device was used as a source of contaminants in ecotoxicity testing.

References	Sampling/dosing device	Origin of contaminants[a]	Assay type[b]	Exposure[c]
Parrott and Tillitt (1997)	SPMD	Environmental	In vitro (F,CL)	Extract spiked
Petty et al. (1998)	SPMD	Environmental	In vivo (F)	Injection of extract
Mayer et al. (1999)	Empore™ disk	Spiked	In vivo (A)	Passive dosing
Parrott et al. (1999)	SPMD	Environmental	In vitro (F,CL)	Extract spiked
Sabaliunas et al. (1999)	SPMD	Environmental	Bacteria	Extract spiked
Petty et al. (2000)	SPMD	Environmental	In vivo (F)	Injection of extract
Sabaliunas et al. (2000)	SPMD	Environmental	Bacteria	Extract spiked
			In vivo (I,Aq)	Extract spiked
			In vitro (H,CL)	Extract spiked
Brown et al. (2001)	PDMS cast in vial	Spiked	Bacteria	Passive dosing
Howsam et al. (2003)	Empore™ disk	Spiked	In vivo (I,Aq)	Passive dosing
Kiparissis et al. (2003)	PDMS cast in vial	Spiked	In vivo (F)	Passive dosing
Koci et al. (2003)	SPMD	Environmental	Bacteria	Extract spiked
			In vivo (I,Aq)	Extract spiked
Gerofke et al. (2004)	Teflon stir bar	Spiked	In vivo (A)	Passive dosing
Johnson et al. (2004)	SPMD	Environmental	Bacteria	Extract spiked
Rastall et al. (2004)	SPMD	Environmental	In vitro (F,CL)	Extract spiked
			Bacteria	Extract spiked
			YES	Extract spiked
Bopp et al. (2006)	Biosilon beads	Spiked	In vitro (F,CL)	Cells attached to PS
Rastall et al. (2006)	SPMD	Environmental	YES	Extract spiked
Bopp et al. (2007)	Biosilon beads	Environmental	In vitro (F,CL)	Cells attached to PS
Breitholtz et al. (2007)	Silica gel	Spiked	In vivo (I,Aq)	Passive dosing
Ke et al. (2007)	SPMD	Environmental	In vitro (M,CL)	Extract spiked
Muller et al. (2007)	Chemcatcher	Environmental	Bacteria	Extract spiked
Mayer and Holmstrup (2008)	PDMS cast in vial	Spiked	In vivo (I,S)	Passive dosing
Bandow et al. (2009a)	Silicone rods	Spiked	In vivo (A)	Passive dosing
Bandow et al. (2009b)	Silicone rods	Spiked	In vivo (A)	Passive dosing
Kwon et al. (2009)	PDMS sheets	Spiked	In vitro	Passive dosing
Liscio et al. (2009)	POCIS	Environmental	YES	Extract spiked
Shaw et al. (2009)	Empore™ disk	Environmental	In vivo (A)	Extract spiked
			Bacteria	Extract spiked
			In vivo (C)	Extract spiked
			In vivo (I, Aq)	Extract spiked
Hillwalker et al. (2010)	Lipid-free tubing	Environmental	In vivo (F)	Extract spiked
Kramer et al. (2010)	PDMS sheets	Spiked	In vitro (F,CL)	Passive dosing
Smith et al. (2010a)	PDMS cast in vial	Spiked	In vivo (I, Aq)	Passive dosing
Smith et al. (2010b)	Silicone O-rings	Spiked	In vitro (H,CL)	Passive dosing
Booij et al. (2011)	PDMS sheets	Spiked	In vitro (M,CL)	Passive dosing
Bougeard et al. (2011)	Silicone O-rings	Spiked	Bacteria	Passive dosing
Engraff et al. (2011)	PDMS cast in vial	Spiked	In vivo (I,S)	Passive dosing
Pesce et al. (2011)	POCIS	Environmental	In vivo (NBF)	Extract spiked
Adolfsson-Erici et al. (2012)	PDMS tubes	Spiked	In vivo (F)	Passive dosing
Morin et al. (2012)	POCIS	Environmental	In vivo (NBF)	Extract spiked
Rojo-Nieto et al. (2012)	PDMS cast in vial	Spiked	In vivo (I,Aq)	Passive dosing
Smith et al. (2012)	Silicone O-rings	Spiked	Bacteria	Passive dosing
Emelogu et al. (2013)	Silicone rubber	Environmental	In vitro (F,CL)	Extract spiked
Seiler et al. (2014)	PDMS cast in vial	Spiked	In vivo (F)	Passive dosing

[a] 'Environmental' indicates the contaminants were collected by deployment of the passive samplers in a contaminated aquatic environment, 'Spiked' indicates the contaminants were spiked on the sampler/dosing device in the laboratory.
[b] F: fish, CL: cell line, A: algae, I: invertebrate, Aq: aquatic, H: human, M: mammal, S: soil, C: coral, NBF: natural biofilm.
[c] 'Extract spiked' indicates the test medium was spiked with passive sampler extract (typically a solvent containing the contaminants absorbed by the sampler).

Pesce et al., 2011; Morin et al., 2012) were the target substances. While in some of these studies a correlation between contaminants and the observed effects was found (e.g. Ke et al., 2007; Liscio et al., 2009), an elaborate interpretation of mixture toxicity – e.g. attempts to explain the observed mixture toxicity based on mixture components – is generally lacking in this type of experiments.

In the second type of study, the main aim was to establish constant exposure concentrations during the entire duration of an (eco)toxicity experiment by partitioning of test substances in the test medium from a solid phase (Mayer et al., 1999). This approach – which is generally referred to as passive dosing – is mainly used for sparingly water-soluble chemicals, as they are difficult to dissolve in water and their aquatic concentrations tend to decline during (eco)toxicity testing due to adsorption of the substance to the test vessel walls, uptake by the test organism, volatilization and biotic and abiotic degradation reactions (Mayer et al., 1999). By placing a dominating solid phase such as polydimethylsiloxane (PDMS) sheets (Kramer et al., 2010) or even Teflon stir bars (Gerofke et al., 2004) loaded with test substance in the (eco)toxicological test medium, dissolved concentrations of the test substance are established as it diffuses into the medium until steady state has been reached. As any test substance that disappears from the test medium (via any of the aforementioned routes) is thus replenished by the solid phase, concentrations are kept constant over time (Mayer et al., 1999). All such passive dosing studies available in literature (Table 1), have been conducted with artificially spiked, nonpolar solid phases. Up to now, these studies have never been conducted with field deployed passive samplers.

The aim of this study is to combine the two approaches described above by using passive samplers previously deployed in the field as a dosing device in a growth inhibition test with a marine diatom. As such, mixtures of micropollutants collected through passive sampling in the field are recreated in laboratory test medium by reversely using the samplers as dosing devices.