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Partitioning of polycyclic aromatic hydrocarbons in the polyethylene/water system

Abstract The suitability of polyethylene sheets as passive samplers of lipophilic contaminants in water bodies was tested. High-density polyethylene (HDPE) and low-density polyethylene (LDPE) sheets were contaminated with PAH. Uncontaminated and pre-contaminated sheets were deployed simultaneously and collected at intervals over 32 days. The exposed sheets and water samples were analyzed for PAH. The initial PAH concentrations in the contaminated and uncontaminated sheets differed by two to three orders of magnitude, but approached a common equilibrium concentration during exposure. The two- to four-ring PAH achieved quasi-equilibrium within the 32-day exposure period, whereas the five- and six-ring PAH did not. The estimated PE/water partition coefficients were approximately three times higher for HDPE than for LDPE, and they were similar in magnitude to the K_{OW} values (the partition coefficients between *n*-octanol and water). The uptake rate constants were approximately four times higher for HDPE than LDPE, which was attributed to the four times higher specific surface area. The uptake and elimination in HDPE followed linear first-order kinetics, whereas for LDPE very slow elimination rates were observed that could not be explained. The results show that PE is a simple, effective, and inexpensive material for sampling trace organic contaminants in water.

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Introduction

Lipid-containing semi-permeable membrane devices (SPMDs) have greatly enhanced the potential to monitor persistent lipophilic organic chemicals in the environment [1, 2, 3]. SPMDs are used as kinetic samplers and the concentration in the sampled environment is usually calculated from a predetermined uptake rate constant and the exposure time. Gale et al. [4] have modeled the uptake of chemicals from water by standard SPMDs and have concluded that for moderate- to high-K_{OW} chemicals (i.e. compounds with large *n*-octanol–water partition coefficients) uptake is limited by the water side resistance (i.e. the aqueous boundary layer on the surface of the polyethylene). Consequently, the uptake rate constant varies with exposure conditions. The use of performance control standards to measure the uptake rate constant during deployment has recently been explored as a method of correcting for this variability [5].

Lefkovitz et al. [6] examined the suitability of polyethylene sheets as an alternative to SPMDs. In laboratory experiments they observed rapid uptake of several three- and four-ring polycyclic aromatic hydrocarbons (PAH) and monochlorinated to trichlorinated biphenyls (PCBs). The accumulation seemed to follow first-order kinetics, suggesting that the kinetic behavior of polyethylene sheets might be simpler than that of the two-phase SPMD samplers, which would simplify the interpretation of the passive sampler results. Several other disadvantages of SPMDs, e.g. difficulties associated with their preparation and with the purification of the extract before analysis could also potentially be circumvented by this alternative method.

This study was undertaken to further evaluate the potential of polyethylene sheets as a sampling phase for lipophilic pollutants in the aquatic or marine environment and to explore the differences between the sampling properties of two distinctly different polyethylene materials. Uncontaminated and pre-contaminated polyethylene sheets were exposed in an estuary and the uptake and elimination

kinetics were followed as the samplers approached a common equilibrium concentration.

Experimental

Preparation of the sampler

Low- (LDPE) and high- (HDPE) density polyethylene bags (Cospak, Brisbane, Australia) were cut open and the seals were removed. The surface area, mass, and thickness were determined for a series of sheets (Table 1). The polyethylene sheets were transferred into a vessel, immersed in dichloromethane (DCM) (Merck, residue-analysis grade), and extracted for 24 h on a shaker. The solvent was discarded and the procedure was repeated with n-hexane (Mallinckrodt, nanograde). After discarding the n-hexane, one half of the sheets were washed again with DCM to remove the hexane and then dried, while the other half was transferred to a DCM solution containing 100 µg of each of fourteen two- to six-ring PAH, and mixed for 24 h on a shaker. The sheets and DCM solution were transferred into a flask which was attached to a rotary evaporator. The DCM was slowly evaporated while the sheets were constantly mixed in the remaining solution. When the DCM was almost completely evaporated (note, the samplers were still coated with DCM) water was added to the flasks. The samplers were then transferred into another clean jar, covered with water, and sealed.

Galvanized iron mesh cages (1 m length and 60 cm diameter with 1 cm×1 cm mesh) were constructed for deploying the samplers at the field site. Nylon ropes were tied to the cages at three levels. The polyethylene sheets were rinsed in the river water to remove residual traces of DCM and attached to the ropes by means of metal clips (Esselte No 1). An equal number of uncontaminated and pre-contaminated samplers were exposed in June–July 1998 at a field site in the estuarine section of Oxley Creek, approximately 1 km from its confluence with the Brisbane River. The water temperature during exposure ranged from 16.4–18.7 °C. For both the HDPE and LDPE two pre-contaminated and two uncontaminated samplers were collected after 0 (blank), 1, 2, 4, 8, 16, and 32 days deployment. On collection, the samplers were cleaned with the creek water at the site, transferred to solvent-washed glass jars, transported to the laboratory, and stored frozen until analysis.

Collection and separation of water samples

To determine organic pollutants in Oxley Creek water, a filter-adsorbent air sampling system consisting of four glass sections fitted with standard glass joints was modified as described elsewhere [7]. Chemicals associated with suspended particulate material were collected on a glass fibre filter (GFF, Whatman GFF, 9 cm diameter) and dissolved compounds which passed through the filter were collected on a resin (XAD-2, Sigma, Melbourne). Before sampling, the center section of the sampler was filled with a bed of approximately 8 cm of XAD-2. The adsorbent was pre-cleaned by

rinsing it with 500 mL each of H_2O, methanol (Mallinckrodt, Chrom AR grade) and acetone (Merck, residue analysis grade). The sampler was then placed in a Soxhlet extractor specifically designed for preconditioning the samplers. The XAD-2 was extracted for 3×10 h, first with DCM, then with n-hexane, and finally with DCM again. The resin was then rinsed with acetone, methanol, and H_2O (500 mL of each). The conditioned resin cartridges were finally filled with H_2O, sealed with fitted ground glass stoppers, and stored in the dark. Before sampling, the top 3–4 cm of the XAD-2 was transferred into a clean container for later analysis as a material blank.

The samples were collected at a flow rate of 1 L min^{-1}. The system was assembled so that the water was drawn through the sampler before it reached the pump. During the final week of sampler exposure two water samples were collected a short distance downstream from the sampler, one at low tide (volume 18 L) and one at high tide (volume 26 L). Depending on the concentration of suspended sediment in the water filters were changed at intervals ranging from 800 mL (low tide) to approximately 1.5 L (high tide). All filters from a given sample were collected in a solvent-rinsed glass jar whereas the adsorbent cartridges were sealed with ground glass stoppers. Samples were transported to the laboratory and stored at 4 °C.

In a preliminary study, sampling artifacts associated with the active sampling system were investigated. Back-up GFFs and back-up XAD-2 traps were installed in series with the respective primary filters and XAD-2 traps and analyzed separately. The concentrations in the back-up filters and back-up XAD-2 traps were below the detection limit [8].

Analysis

For analysis the sealed frozen samples were allowed to warm to room temperature and then spiked with a surrogate recovery standard cocktail containing known amounts of twelve deuterated three- to six-ring PAH ($^2D_{10}$-phenanthrene, $^2D_{10}$-anthracene, $^2D_{10}$-fluoranthene, $^2D_{10}$-pyrene, $^2D_{12}$-benz(a)anthracene, $^2D_{12}$-chrysene, $^2D_{12}$-benzo(b)fluoranthene, $^2D_{12}$-benzo(k)fluoranthene, $^2D_{12}$-benzo(e)pyrene, $^2D_{12}$-indeno(123cd)perylene, $^2D_{12}$-dibenz(ah)anthracene, and $^2D_{12}$-benzo(ghi)perylene) which were used for quantification. Sufficient (approximately 250 mL) DCM was added to cover the sheets, the lids were replaced, and the jars were shaken overnight before the solvent was decanted into a beaker. The sheets were further extracted separately with DCM and n-hexane (100 mL of each), being submerged in each solvent for ~10 min by use of a clean glass rod. The combined extracts were filtered through anhydrous sodium sulfate and concentrated to ~1 mL by rotary evaporation. The extracts were then subjected to adsorption chromatography in a glass column (18 mm i.d.) filled with 20 cm of 5% deactivated Florisil. The PAH were eluted with 110 mL 6% diethyl ether (DEE) in n-hexane followed by 110 mL 10% acetone in n-hexane. The extracts were transferred to DCM and subjected to further cleanup by gel-permeation chromatography (GPC). A 19 mm×150 mm guard column and a 19 mm×300 mm main column were used with Envirogel (100 Å pore size, 15 µm particle size; Waters) as the stationary phase and DCM as the mobile phase. The flow rate was 24 mL min^{-1} and the sample was collected from 14–24 min. The purified extract was concentrated by rotary evaporation to approximately 3 mL and further evaporated, by use of a gentle stream of nitrogen, to a final volume of 1 mL, or less if required for detection of the analytes.

Separation and quantification of the PAH were performed by use of a Varian 3400 GC equipped with a Finnigan A200S liquid autosampler (splitless; injector temperature 295 °C; GC column: HP1MS (Hewlett–Packard), originally 25 m, 0.2 mm i.d., 0.33 µm film thickness; temperature program: 65 °C (isothermal 2 min), 20° min^{-1} to 295 °C (isothermal 10 min.)) and coupled to a Finnigan SSQ 710 single-stage quadrupole mass-selective detector. The quantification criteria included confirmation of the relative retention times and the carbon isotope ratios. The mass fragment with the highest intensity (molecular ion) was routinely used for quantifica-

Table 1 Physical characteristics of the samplers

Property	HD	LD
Weight per sampler (g)	0.680	1.89
Density of polyethylene (g cm^{-3})[a]	0.955	0.91
Surface area per sampler (cm^2)	2768	1769
Specific surface area (cm^2 cm^{-3})	3890	850
Mean sampler thickness (µm)[b]	9	25

[a] from Ref. [9]
[b] specified by Cospack. Measured values were within 20% of these values

Fig. 1 Semilogarithmic plot of the dependence on time of the concentrations measured in HDPE. The lines indicate the linear regressions calculated to determine k_2. Data points in brackets were not included in the data interpretation
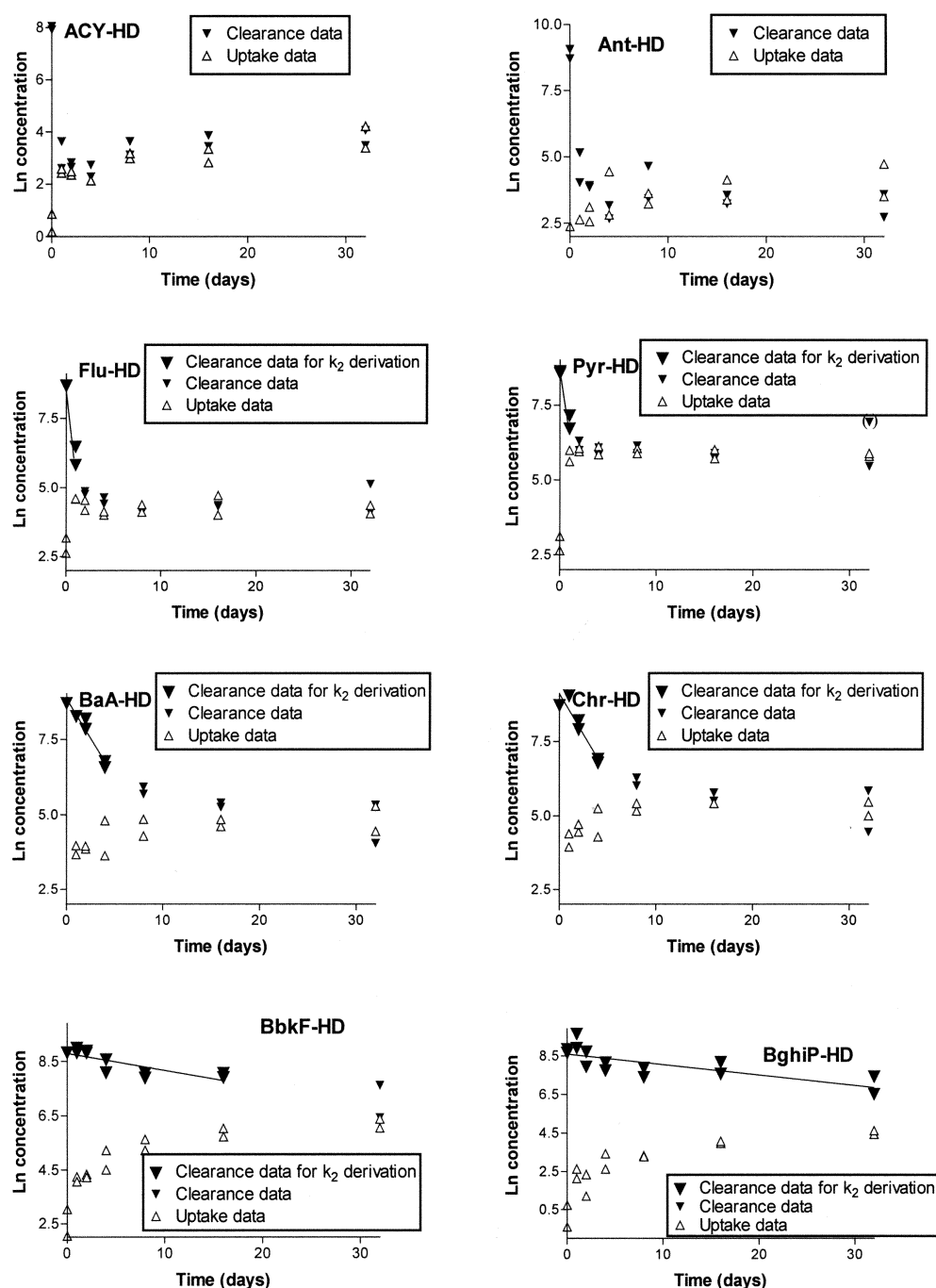
tion. The sample detection limits for individual compounds in a sample were defined by

1. a signal-to-noise ratio greater than three times the average baseline noise in the retention window, and/or
2. analyte concentration in the sample greater than three times the concentration in the sample greater than three times the respective matrix and solvent blank (blank levels were analyzed with each batch of 12 or 18 samples).

The PAH were quantified by isotope dilution using the surrogate recovery standards. The recoveries of the surrogate recovery standards were calculated by use of external calibration. They were consistently greater than 50% for those compounds reported. In this study deuterated phenanthrene was used to quantify PAH with

a molar mass smaller than 178 g. The relative recoveries of these compounds compared with deuterated phenanthrene was determined with a sample which was spiked with both the deuterated PAH and the compounds of interest. With the exception of naphthalene (occasionally the recoveries were as low as 35%) the recoveries were consistently greater than 50% and for PAH with more than two rings greater than 70%.

In addition to the usual quality-control procedures, the accuracy of the PAH method was tested by use of using a soil sample which had previously been used in interlaboratory calibrations in Australia. The mean normalized difference ((value a–value b)/((value a+value b)/2)×100) between the mean value determined by all other laboratories and our result was 30% [8].

Fig. 2 Semilogarithmic plot of the dependence on time of the concentrations measured in LDPE. The lines indicate the linear regressions calculated to determine k_2. Data points in brackets were not included in the data interpretation
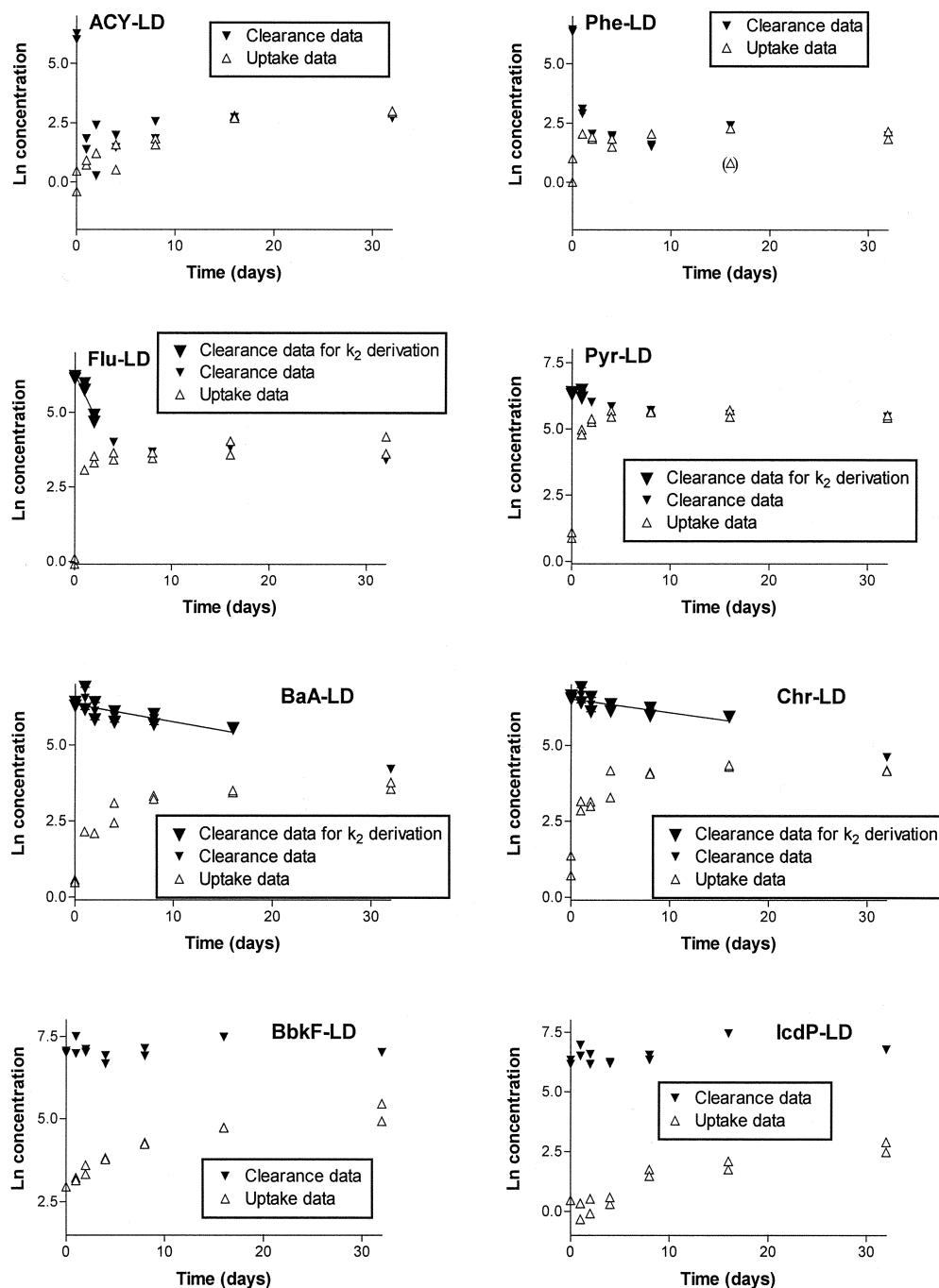
Results and Discussion

The uptake and clearance of PAH in the low- and high-density polyethylene sheets was monitored during the deployment period of 32 days in Oxley Creek. The concentrations were normalized to the volume of polyethylene in the sampler ($\mu g\ L^{-1}$).

Despite the simplicity of the deployment system, the sampler cages remained intact and none of the samplers was lost or significantly damaged during the exposure period. A biofilm on the surface of the sheets was also

clearly visible after exposure for approximately ten days. With increasing exposure time the HDPE sheets showed signs of polymer degeneration, with crack-like patterns developing in the PE structure. The experimental results provided no indication that these changes influenced the sampling properties of the PE, although this was not studied specifically.

Examples of the uptake and clearance behavior of various PAH over the study period are shown in Figs 1 and 2 (note the logarithmic scale of the y-axis and the parallel samples collected at each time point). The PAH concentrations at the start of the exposure period were generally

Table 2 Summary of the experimental results. C_{Seq} is the equilibrium concentration defined as the average concentration achieved after the concentration difference between the contaminated and uncontaminated samplers became within a factor of 2. The PE/water partition coefficients $K_{PE/W}$ were calculated as described in the text. The log K_{OW} values were from Ref. [10]. The k_1C_W values are the slopes from Figs 4 and 5. The k_2 values are the slopes from Figs 1 and 2, the "<" symbols indicate the upper boundary of the 95% confidence interval of the slope

	C_{Seq} HDPE (μg L^{-1})	C_{Seq} LDPE (μg L^{-1})	C_{Water} High/low tide (ng L^{-1})	log $K_{PE/W}$ HDPE (μg L^{-1})/ (μg L^{-1})	log $K_{PE/W}$ LDPE (μg L^{-1})/ (μg L^{-1})	log K_{OW} [10]	k_1C_W HDPE (μg L^{-1} day^{-1})	k_1C_W LDPE (μg L^{-1} day^{-1})	k_2 HDPE (day^{-1})	k_2 LDPE (day^{-1})
Naphthalene	24	2.1	2.8/3.3	3.90	2.83	3.37	–	–	–	–
Acenaphthene	25	9.9	1.4/1.0	4.33	3.92	3.92	–	–	–	–
Acenaphthylene	7.2	2.5	<0.3/<1.3	–	–	4.00	–	–	–	–
Fluorene	20	7.7	<0.22/3.0	4.10 [a]	3.68	4.18	–	–	–	–
Phenanthrene	30	7.0	<0.69/<0.45	–	–	4.57	–	–	–	3.4
Anthracene	43	13	<1.1/<3.0	–	–	4.54	–	–	–	2.7
Fluoranthene	102	43	0.5/2.4	4.85	4.48	5.22	–	–	2.5	0.69
Pyrene	413	250	2.1/11	4.80	4.58	5.18	–	–	1.7	0.13
Benz(*a*)anthracene	147	–	<0.10/0.47	5.71[a]	–	5.91	–	–	0.52	0.058
Chrysene	230	–	<0.17/0.18	6.12[a]	–	5.86	60	10	0.54	0.045
Benzo(*b/k*)fluoranthene	–	–	<0.43/0.40	–	–	5.91[b]	27	5.6	0.062	<0.015
Benzo(*e*)pyrene	–	–	<0.07/<0.9	–	–	6.04[c]	19	5.2	0.047	<0.057
Indeno(*cd*)perylene	–	–	<0.02/<0.5	–	–	6.5[d]	1.9	0.45	0.048	<0.0051
Dibenz(*ah*)anthracene	–	–	<0.04/<0.4	–	–	6.75	0.69	0.17	0.062	<0.01
Benzo(*ghi*)perylene	–	–	<0.01/<3.9	–	–	6.5	2.8	0.63	0.054	<0.0094

[a] the water concentration used to calculate $K_{PE/W}$ was taken as the mean of the measured value at low tide and the detection limit at high tide

[b] mean value from B(b)F and B(k)F
[c] value from B(a)P
[d] value from B(ghi)P

2–3 orders of magnitude higher in the pre-contaminated polyethylene sheets than in the uncontaminated sheets. During exposure the PAH concentrations in the pre-contaminated and uncontaminated samplers approached a common value. We interpret this common value to represent the partitioning equilibrium state between the sampler and the water. The fact that both pre-contaminated and uncontaminated samplers converge to the same concentration suggests that PE is suitable for the passive sampling of hydrophobic organic contaminants in water.

It was assumed that a quasi equilibrium had been achieved if the concentration in the contaminated and uncontaminated samplers were within a factor of two of each other. For naphthalene, acenaphthene, acenaphthylene, fluorene, phenanthrene, and anthracene, uptake and elimination in both the low- and high-density polyethylene sheets was so rapid that quasi equilibrium was achieved after 1–2 days. Within a week, fluoranthene and pyrene had equilibrated, and within a month, chrysene. The five and six-ring PAH, however, were still far from equilibrium at the end of the experiment (32 days). Thus, for a one month exposure period the PE sheets behaved as equilibrium samplers for the smaller PAH and integrating samplers (i.e. the chemical levels in the water were integrated in the sampler throughout deployment) for the larger, more hydrophobic compounds.

For an equilibrium sampler, the key sampler characteristic is the partition coefficient. To determine the polyethylene/water partition coefficients $K_{PE/W}$, the equilibrium concentrations in the samplers C_{Seq} were first estimated as the average of all of the concentrations in both the conta-

minated and uncontaminated samplers collected after the concentration difference dropped to within a factor of two. The equilibrium concentrations in HDPE were about three times higher than in LDPE for most compounds (Table 2). Because the two samplers were exposed in the same environment, this indicates that the affinity of the HDPE for the chemicals studied here was greater than that of LDPE.

$K_{PE/W}$ was approximated by dividing C_{Seq} by the average concentration measured in the dissolved phase (Table 2). As shown in Fig. 3, a positive correlation between log K_{OW} and log $K_{PE/W}$ was observed, and the two partition coefficients were of similar magnitude. The $K_{PE/W}$ values must be viewed as rough estimates, because they were based on two water samples only. One can, nevertheless, conclude that the partitioning properties of the samplers
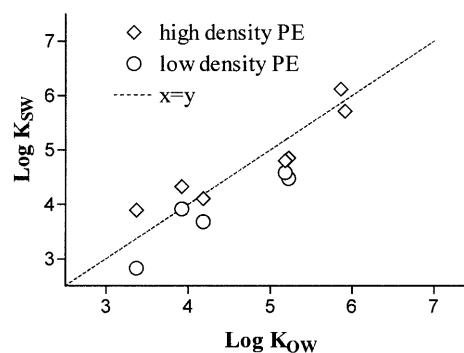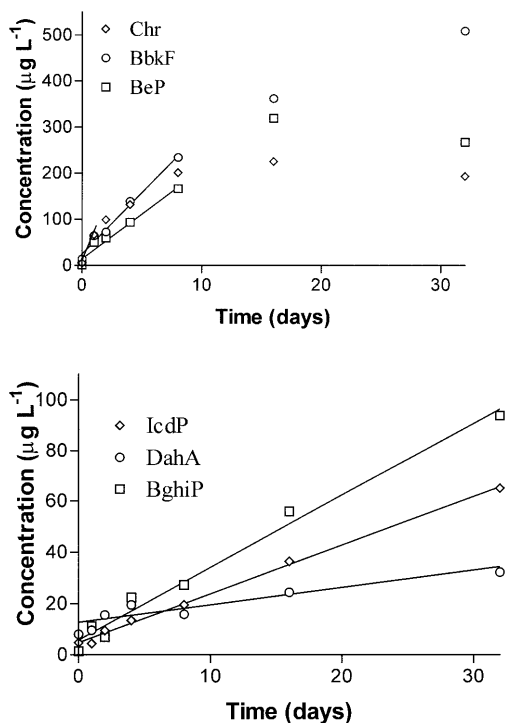

Fig. 3 Log PE/water partition coefficients plotted against log K_{OW} for several PAH

were similar to the partitioning properties of 1-octanol for the two- through four-ring PAH.
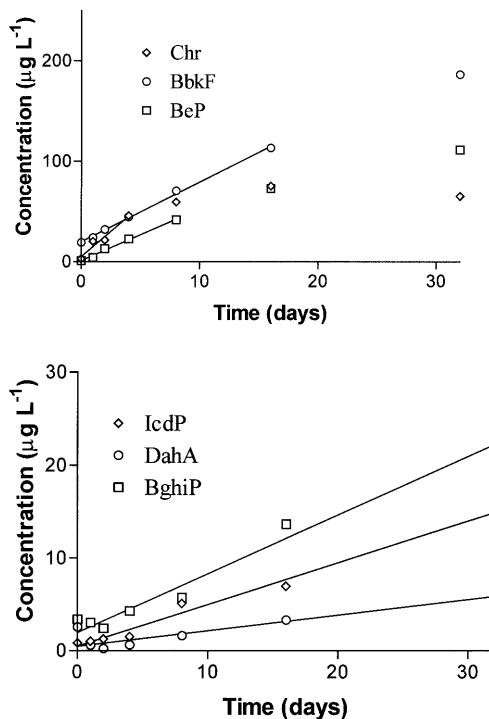
For the five- and six-ring PAH, the PE sheets behaved as accumulating samplers over the 32-day deployment period. To interpret these results a knowledge of the contaminant uptake kinetics is required. The semilogarithmic plots of sampler concentration C_S against time t for the elimination experiments in Figs 1 and 2 yielded straight lines which did not level off until the equilibrium concentration was approached. This suggests that accumulation of the chemical in the sampler can be described by use of first-order kinetics, and implies that both the HDPE and the LDPE behave as a single well-mixed storage compartment.

The time constant for the elimination experiment k_2 was calculated from the slope of the plot of ln C_S against t (Table 2). Because the slope levels off as the equilibrium concentration is approached, only those data points for which C_S was at least 2.7 times (i.e. one ln unit in Figs 1 and 2) greater that the equilibrium concentration were used. Only for the three four-ring PAH were k_2 values available for both LDPE and HDPE. For all three compounds the time constants were markedly higher for HDPE. For the five- and six-ring PAH no elimination was observed in LDPE and the k_2 values were thus highly uncertain. They were, however, at least a factor of 5–10 less than for the same substance in HDPE.

The kinetics during the accumulation phase are illustrated in Figs 4 and 5. The initial slopes of the uptake curve were estimated for each compound by use of linear regression. The slopes are given in Table 2. They are ap-

Fig. 5 Concentration in the LDPE plotted against time for the accumulation experiment. The means of the duplicate samples are shown. The lines indicate the linear regressions calculated to determine the initial uptake rate (k_1C_W)

Fig. 4 Concentration in the HDPE plotted against time for the accumulation experiment. The means of the duplicate samples are shown. The lines indicate the linear regressions calculated to determine the initial uptake rate (k_1C_W)

proximately four times greater for HDPE than for LDPE. Employing a one-compartment model with first-order kinetics gives:

$$\frac{dC_S}{dt} = k_1 C_W - k_2 C_S \tag{1}$$

These slopes represent the product of the uptake time constant k_1 and the dissolved concentration in water C_W. C_W was the same for both sampling materials, so the ratio of the slopes for HDPE and LDPE reflects the ratio of k_1. The approximately four times higher value of k_1 for HDPE is attributed to the approximately four times higher specific surface area of this sampler compared to LDPE.

Because $K_{PE/W}=k_1/k_2$, the ratio of the slopes divided by the ratio of the partition coefficients must reflect the ratio of the k_2 values. The partition coefficients of the smaller PAH were approximately a factor of three higher in HDPE. If this was also true for the five- and six-ring PAH, the elimination time constant k_2 during the uptake experiments was approximately 1.3 times higher for HDPE than for LDPE. This result is in contrast with the large differences between k_2 determined from the elimination experiments.

To resolve this inconsistency, an independent determination of k_2 using only the uptake data and the equilibrium concentration C_{Seq} was undertaken. During the initial uptake Eq. (1) simplifies to:

$$\left[\frac{dC_S}{dt}\right]_{U0} = C_{Seq}k_2 \tag{2}$$

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Using this equation, a k_2 value of 0.26 day^{-1} for chrysene in HDPE (the only example for which both the initial uptake rate and the equilibrium concentration were available) was obtained. This is reasonably close to the 0.52 day^{-1} estimated from the elimination kinetics, especially given that the initial uptake rate was likely underestimated for this compound, because the measured uptake curve was non-linear from the outset (Fig. 4). The fact that both the uptake and the elimination kinetics can be described using the same set of k_1 and k_2 indicates that the first-order model adequately describes the kinetics of contaminant exchange between water and HDPE.

It remains to be explained why the elimination kinetics were much slower for the LDPE, although the initial uptake rate was comparable with that of HDPE. The fact that equilibrium for chrysene in LDPE was achieved within 8 days during uptake, whereas the concentration in the clearance experiment dropped by only 35% during the same period, is evidence that the uptake and elimination kinetics were different for this material. One possible explanation is that the higher residual DCM levels in the contaminated LDPE sheets altered its properties, resulting in different kinetic behavior.

Conclusions

Polyethylene sheets are well suited as passive samplers for monitoring environmental levels of PAH in water. HDPE, in particular, with its first-order uptake kinetics yields data which are easy to interpret. The sampling principle depends on the properties of the chemical. The small, comparatively hydrophilic PAH have rapid kinetics and reach equilibrium quickly. Hence the sampler concentrations reflect the water concentrations towards the end of the exposure period. With increasing size and hydrophobicity of the PAH, the kinetics become slower and the aqueous concentrations towards the beginning of the exposure period have a progressively greater influence on the sampler concentrations. If the PAH is sufficiently hydrophobic, equilibrium is not approached and the sampler simply integrates the water concentrations during the exposure period. The boundaries between these sampling regimes can be shifted to more or less hydrophobic PAH by varying the time of exposure and – most likely – the water turbulence around the sampler.

Sampling materials with distinctly different properties could open new opportunities for monitoring. An example is parallel deployment of an equilibrium sampler and a kinetically limited sampler for the same chemical, the for-

mer as an event sampler to capture unusual peaks, the latter to determine the long-term average concentration. However, the hypothesis that different types of PE have markedly different sampling properties was not definitively answered in this work. The results suggest that the partition coefficients and exchange kinetics were similar for the two materials studied.

For effective implementation of polyethylene sheets in aquatic and marine monitoring, further work is needed. $K_{PE/W}$ values must be determined under controlled laboratory conditions to enable the water concentrations to be back-calculated for chemicals which achieve a partitioning equilibrium in the sampler. A method of calibrating the samplers for kinetically limited uptake is required so that the water concentrations can be derived from the sampler concentrations for chemicals that fall into this sampling regime. Finally, the influence on sampler uptake of chemicals associated with colloids or particles must be investigated. When these issues are resolved PE promises to be a simple and inexpensive method for monitoring persistent hydrophobic organic chemicals in water.

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