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Experimental results on effects of capping on fluxes of persistent organic pollutants (POPs) from historically contaminated sediments

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Abstract

One option proposed for the remediation of Oslo harbour, Norway, has been dredging of up to 700 000 m^3 of contaminated sediments and deposition at 60 m depth in a nearby intermittently anoxic fjord basin. Contaminant retention in the deposit area will be favoured by metal sulphide precipitation and the current absence of sediment dwelling organisms. In addition capping will be performed to protect against future fjord recovery and recolonisation with benthic animals. In order to assess the risk of contaminant leakage from the deposit area a long-term mesocosm experiment was performed to simulate the three expected successive phases: (1) anoxic basin water, (2) oxic water and (3) oxic water and presence of bioturbators. Fluxes of dissolved, bioavailable contaminants (PAHs, PCBs and DDTs) were measured by uptake in passive membrane samplers (SPMDs) located in the source seawater and downstream 0.25 m^2 boxes with capped and non-capped harbour sediments. The experiment showed that pyrene and fluoranthene was released from no-cap treatments at high rates (2545 and 830 pmol m^{-2} day^{-1}, respectively) during the initial, anoxic phase. The majority of components showed, however, maximum fluxes during the final, bioturbation phase: 243 pmol m^{-2} day^{-1} sumKPAH (six of the most cancerogenous PAHs), 19.6 pmol m^{-2} day^{-1} of sumPCB_7 and 13.6 pmol m^{-2} day^{-1} DDT. Cap layers reduced fluxes and bioaccumulation of PAH in the gastropode *Hinia reticulata* by 89–100%. Fluxes and bioaccumulation of PCBs was not reduced to the same extent suggesting that external sources may be relatively more important for PCB than for PAH levels in this organism. A minor release of PAH-components from one of the cap treatments indicated slight contamination from fossil fuel or explosives applied during production of the machined sand used as cap material for this treatment. Differences due to cap thickness variations between 10 and 50 cm were not found throughout the experimental period of 34 months.

Keywords: Fluxes; Sediments; Bioaccumulation; PAH; PCB; DDT

1. Introduction

In many estuaries and fjords surrounded by large populations, harbours and a variety of potentially polluting anthropogenic activities, the top layer of marine sediments has developed into major reservoirs for a diverse mixture of contaminants. The post-industrial decline of most other discharge sources has left recycling from historically contaminated sediments to overlying water and marine organisms a major potential risk for the fjord environment (Larsson, 1985). In Norway, as in many other countries, environmentally motivated remediation efforts have become increasingly relevant, often as result of restricted commercial exploitation of marine species by health authorities due to elevated concentrations of polychlorinated biphenyls, dioxins or heavy metals. The coupling between historically contaminated sediments and contaminated seafood is,

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however, poorly understood and the environmental benefits from various options such as dredging, capping or natural remediation are difficult to assess. In many cases dredging to increase sailing depths is the only option, but a better knowledge on contaminant recycling from the sediments is still needed to assess the added value of reduced contaminant levels in marine species.

Results of recent dredging operations performed on contaminated sediments in Sandefjord harbour in the outer Oslofjord and at a naval base in West Norway, appear not to have yielded the expected reduction of PCB levels. Both within the sediment surface and in mussels and SPMDs exposed 1 m above the bottom in the dredged area, contaminant levels were higher than expected after the remediation operation was completed (Bakke, 2004; Voie et al., 2002). In addition to recycling from historically contaminated sediments, major sources may be run-off from local, land based sources and atmospheric deposition (Gevao et al., 1997; Axelman and Broman, 1999; Jaward et al., 2004). The success of efforts to reduce recycling from sediment reservoirs will, in addition to the technical difficulties involved in complete removal of contaminant sources at the sediment surface, depend on the continued input from other still active, external sources.

In general, marine sediments are the final destination of particle associated contaminants and degradation and burial by natural sedimentation processes will slowly remove the contaminants from the biogeochemical cycles. Furthermore, the majority of PAHs and PCBs in sediment and water reservoirs are particle-bound whereas the main risk for the ecosystem and human health is associated with the labile fractions available for recycling and uptake in biota (Campfens and Mackay, 1997). Direct observations on fluxes of persistent organic contaminants and in particular on the most bioavailable fractions are scarce to non-existent (Palm et al., 2004). Current knowledge on natural fluxes of polyaromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) is to a large extent based on total concentrations measured in sediments, water and biota. Predictive models on transport and fate depend crucially on particle–water partitioning and may be improved by orders of magnitude by inclusion of locally derived coefficients (Chin and Gschwend, 1992; Hunchak-Kariouk and Suffet, 1994; Axelman et al., 2000; Verrengia Guerrero et al., 2003) and soot–carbon equilibria (Gustafsson et al., 1997; Næs et al., 1998; Persson et al., 2002).

The mesocosm experiment described here was part of the baseline investigations performed to establish a remediation plan for contaminated sediments in the harbour of Oslo, Norway. One option proposed for this remediation is removal by dredging of up to 700 000 m^3 of contaminated sediments and deposition at 60 m depth in a nearby fjord basin (Skei et al., 2000). Current velocities in the basin deep water are small and resuspension of sediments is assumed to be negligible except maybe for short periods during deep water renewal. The current lack of oxygen in the basin deep water will favour contaminant retention by metal sulphide precipitation and absence of bioturbating organisms. Capping is planned to further reduce leakage of contaminants and uptake in biota during future reestablishment of oxic deep water and recolonisation with sediment-dwelling organisms. The objective of the present experiment was to measure the effect of capping on the fluxes of persistent organic pollutants between the dredged and deposited harbour sediment and the overlying water under conditions similar to those expected in the deposit area. The sediments in the harbour area are often loaded with maximum contaminant concentrations a few centimeters below the sediment surface and the measured fluxes from the mixed sediment may be slightly higher than the fluxes occurring in the harbour area.

2. Material and methods

2.1. Experiment outline

The experiment was performed during the period December 2000 to October 2003 in acrylic boxes placed in a soft-bottom mesocosm at Solbergstrand Marine Research Station (Berge et al., 1986). One of the disposal options was to transfer the sediments to a storage site at 60 m depth in a nearby anoxic basin. Therefore, the first phase of the experiment was designed to simulate a sediment–water interface with prevailing anoxic conditions in the overlying seawater. In case of future fjord restoration and the reestablishment of oxic deep water, the second phase of the experiment was designed to simulate oxic conditions. In the third phase recolonisation was simulated by the addition of benthic bioturbator species, i.e. 20 individuals box^{-1} of the polychaete *Nereis diversicolor*, 25 individuals box^{-1} of the gastropode *Hinia reticulata* and 3 individuals box^{-1} of the crustaceans *Pagurus* sp.

2.2. Design

Sediments from 0 to 30 cm depth in the harbour area was mixed and filled into 20–30 cm thick layers in

Table 1
Set-up of the sediment–cap–water system in the eight boxes used for the mesocosm experiment

Treatment	Sediment thickness	Cap thickness	Water depth (cm)	Total box height (cm)
S50	20 cm	50 cm	15	85
S30	20 cm	30 cm	15	65
S10	20 cm	10 cm	15	45
R10	20 cm	10 cm	15	45
M10	20 cm	10 cm	15	45
H0A	30 cm	no cap	15	45
H0B	30 cm	no cap	15	45
BL	no sed.	no cap	15	15

50×50 cm transparent acrylic boxes. The treatments are shown in Table 1. In five boxes the harbour sediments (*H*) were covered with 10, 30 or 50 cm cap layers Two boxes contained harbour sediments without cap and one experimental blank had neither sediments nor cap. The boxes were constructed and filled with sediments and cap material to provide equal volumes of 30 L circulating seawater. Properties of the harbour sediments and cap materials are shown in Table 2. Three different cap materials were used: sandy till (S), untreated sandy till (R) and crushed stone material (M). These materials represent different geological properties and potential differences in capping efficiency. All materials are readily available in the Oslofjord area and therefore considered as potential cap materials for local remediation projects.

All boxes were fitted with lids and a water recirculation system and flushed with N_2 gas before filling up with 15 cm anoxic seawater from the Oslofjord. During the first anoxic phase, the water in each box was continuously recirculated through a sampling cell fitted with an SPMD (Semipermeable Membrane Device) and a magnetic stirrer (Fig. 1, right). A water-lock for pressure compensation was inserted in the center of the lids. During the two later phases, seawater from 60 m depth in the Oslofjord adjacent to the research station was continuously flushed through the box and sampling cells at a rate of 4 ml min^{-1} (Fig. 1, left). To avoid concentration gradients within the overlying water the outlet water from the boxes were drawn horizontally 1 cm above the sediment surface whereas the inlet was positioned near the water surface. During the flow-through phases, an air-lift system was inserted at the center of the lids to provide water circulation and aeration. An eight channel peristaltic pump was used to maintain water flow through each box and cell unit. The materials in contact with the water phase were tubings of PVC or marprene (across pump heads) and acryl or polycarbonate in boxes, lids and sampling cells.

2.3. Sampling

Standard SPMDs (a 91.4-cm long and 2.5-cm wide layflat LDPE-tubing containing 1 ml of 95% purity triolein) were used throughout this work. The sampler is designed to concentrate hydrophobic organic contaminants (e.g. polycyclic aromatic hydrocarbons and organochlorines) and provide an integrative estimate of their concentration in water (Huckins et al., 1990). The actual concentrations in water were calculated from a spreadsheet model (Alvares, 2004).

During each of the experimental phases a new SPMD was mounted simultaneous with collection of the previous SPMD. Thus, from each sampling cell, five successive samples were drawn during the anoxic period sampling interval (days 0–457), three samples during the oxic period sampling interval (days 534–766) and two samples during the bioturbation period sampling interval (days 789–844). Apart from two prolonged periods of 132 and 257 days, exposure times were 39–64 days. Sediment cores and organisms were sampled on day 1046, after 9 months of exposure of the Gastropodes in harbour sediments and cap.

2.4. Analytical methods

Two different methods were used for recovering the sequestered contaminants from the SPMD:

(A) *Dialysis of the whole* SPMD was the standard procedure used at the laboratory. The SPMDs were rinsed with distilled water and dried off with paper tissue to remove algae from the membrane surface. They were transferred to a glass jar fitted with annealed aluminium foil under screw-type lids and internal standards (D-naphthalene, D-acenaphthene, D-phenanthrene, D-chrysene, D-perylene, PCB-53 and PCB-204) were added together with 150 ml dichloromethane/cyclohexane (1:9). The procedure consists of three 24 h

Table 2
Grain size and organic content of harbour sediment (H) and the three cap materials: machined sand (M), sand (S) and raw sand (R)

Property	M	S	R	H
10% grain size<(mm)	0.025	0.085	0.029	<0.002
60% grain size<(mm)	2.489	1.541	1.211	0.013
Clay fraction (%)	1.6	1.6	2	24.5
TN (mmol g^{-1} dry weight)	<0.07	<0.07	<0.07	0.15
TOC (mmol g^{-1} dry weight)	<0.08	<0.08	0.08	4.3

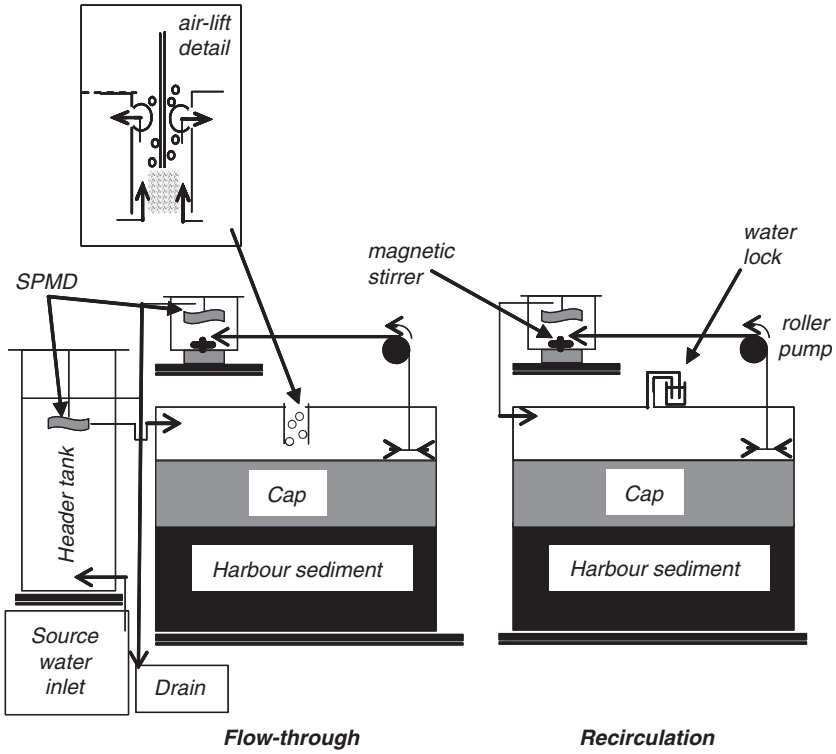

Fig. 1. Schematic drawing of the set-up during flow-through (left) and recirculation (right) measurements. Flags illustrate SPMDs in header tank and sampling cells.

dialytic intervals with fresh solvent. The solvents were combined and reduced to 1 ml, before filtration with a 0.45-μm Cameo Teflonfilter and clean-up using one Waters Envirogel™ Gel Permeation Chromatography (GPC) column. The extract was divided in two, one portion was reduced to 50–500 μl and analysed using Gas Chromatography with a Mass Spectroscopy detector (GC/MS), the other portion was treated with concentrated sulphuric acid, reduced to 150 μl and analysed for organochlorines (OC) using Gas Chromatography with an Electron Capture Detector GC/ECD.

(B) *Analysis of the triolein* reduced detection limits which were frequently approached during the oxic and bioturbation periods. After rinsing of the SPMD, the triolein was squeezed out of the membrane using two cylinders held together by clams. The triolein was removed through small hole cut in one end of the membrane and collected in a sample vial. The amount of triolein in each sample was quantified and internal standards, specified above, were added together with dichloromethane to a total volume of 3 ml. Clean-up was performed by the use of four Waters Envirogel™ Gel Permeation Chromatography (GPC) columns. The extract was divided and analysed as described above. This procedure reduced interference in the chromatogrammes and was applied on the membranes exposed during the oxic and bioturbation periods.

2.5. Calculation of water concentrations

Flux measurements were to some extent dependant on the water concentrations estimated from the mass extracted by the SPMDs. Huckins et al. (1993) have developed mathematical models for estimating water concentrations from analyte concentrations in SPMDs. The equations for the three uptake phases, linear, curvilinear and equilibrium respectively, are shown below.

$$C_W = C_{SPMD} M_{SPMD} / R_S t \quad (1)$$

$$C_W = C_{SPMD} / K_{SPMD} (1 - \exp[-k_e t]) \quad (2)$$

$$C_W = C_{SPMD} \times K_{SPMD} \quad (3)$$

C_W and C_{SPMD} are the analyte concentration in the water (pM) and SPMD (nmol g^{-1}) respectively, M_{SPMD}

is the mass of the SPMD (g), R_S is the uptake rate (L day^{-1}), t is the exposure time (days), K_{SPMD} is the equilibrium partitioning coefficient and k_e is the exchange rate constant.

The equations for the three uptake-phases were accounted for in the model used for calculation of water concentrations. The uptake rates used in the model were based on the uptake in the whole SPMD ($C_{SPMD}M_{SPMD} = C_{membrane}M_{membrane} + C_{lipid}M_{lipid}$). Analyses during phase 2 (oxic) and 3 (bioturbation) of the lipid only, may however have underestimated water concentrations ($C_{SPMD}M_{SPMD} = C_{lipid}M_{lipid}$). Rantalainen et al. (2000) found uptake rates for PCBs in the lipid of close to 80% of the uptake rates for the whole membrane. Corresponding data for PAHs are not known and the content in the lipid was used uncorrected in all concentration calculations for phases 2 and 3.

Litterature values for grouped components given in units such as g L^{-1} were recalculated to moles L^{-1} assuming weighted average molecular weights of 337 g mol^{-1} for sumPCB and 210 g mol^{-1} for sumPAH.

2.6. Flux calculations

During the anoxic incubation, the fluxes were calculated as the sum of the mass increase in the enclosed water and the mass harvested with the SPMDs removed from the enclosed water:

$$F = (\Delta C_W V + U_{SPMD})/TA \quad (4)$$

F flux (pmol m^{-2} day^{-1})
ΔC_W concentration increase in enclosed water during time T (pM)
V volume enclosed water (L)
U_{SPMD} component mass extracted from SPMDs exposed during time T (pmol)
T time of exposure (days)
A sediment surface area (m^2).

During the oxic and bioturbation phases, dissolved contaminants released from the sediments will be collected in the SPMD in the sampling cell or lost through the water outlet. Fluxes were calculated from:

$$F = ((C_2 - C_{SW})Q + U_{SPMD2}/T)/A \quad (5)$$

C_{SW} concentration in source water calculated from mass (U_{SPMD1}) in SPMD exposed in source water during time T
C_2 concentration in outlet water calculated from mass (U_{SPMD2})
U_{SPMD2} mass extracted from SPMDs exposed in sampling cell during time T
Q flow (L day^{-1}).

Concentrations less than detection limit, were assigned with detection limit in flux calculations, but zero in additions for sumPAH, sumPCB and sumDDT.

2.7. Statistical analyses

Flux variations were analysed using one- or two-way ANOVA in the fit model platform in JMP®v.4 statistical software from SAS Institute Inc.

2.8. Sources of error

Because of the application of two quite different methods for flux measurements, fluxes measured by recirculation during the anoxic period and flow-through during the subsequent oxic and bioturbation phases may differ due to systematic errors related to e.g. concentration differences in the overlying water. Another source of error in the present flux measurements is considered to be those inherent in the model applied for calculation of concentrations from membrane uptake, i.e. errors in the constants involved in Eqs. (1)–(3) and failure to apply with the basic assumption of exposure of the SPMD in an unlimited water phase with non-variable concentration.

During the anoxic phase, the concentration (C_W) of many PAH and PCB components increased steadily in the recirculated water overlying the sediments in each box. In such cases the rate of release from the sediment was larger than the rate of uptake in the membranes and the first term ($C_W V$) in Eq. (4) might contribute significantly to the flux from the sediment. However, as shown in Fig. 2, the total amounts of 7.7 nmol pyrene and 0.14 nmol benzo(a)pyrene accumulated in the water phase in each box was small compared to the amounts of 187 nmol and 5.4 nmol of the respective components harvested with the membranes, i.e. $C_W V \ll U_{spmd}$. It follows that during the anoxic phase of the experiment, potential errors resulting from inaccurate assumptions inherent in the concentration calculation model will be small.

During flow-through measurements (Eq. (5)), both the concentration change ($C_2 - C_{SW}$) and the uptake in the downstream membrane (U_{SPMD2}) contributed significantly to the calculated flux. Therefore, the assump-

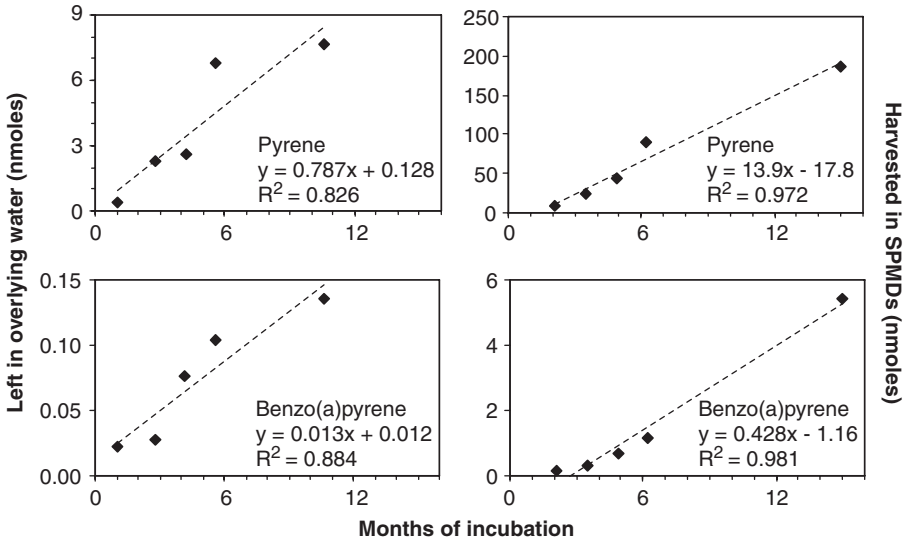

Fig. 2. Total mass of pyrene and benzo(*a*)pyrene harvested (U_{SPMD}) and remaining ($C_W V$) in the overlying water in box H0A (no-cap) during the anoxic incubation. Curves shown, equations and correlation coefficients were calculated from linear regression analyses.

tions on uptake performance and other factors inherent in the concentration calculation model may be more important biasing the fluxes measured during the oxic and bioturbation periods.

Other sources of error could be adsorption/desorption from the materials used in tubes and boxes, degradation in the chamber water and volatilization through the airlift used during the oxic and bioturbation phase. The significance of such errors is difficult to assess. Most of the applied equipment had been used in previous experiments and was considered well equilibrated with the seawater environment. In the blank box, the first fluxes determined for methylnaphthalene and a few other low molecular PAHs were anomalous high, but decreased later in the experiment. The blank box was designed for the present experiment and the biased fluxes were most likely a result of contamination from glue solvents used for box construction. No evidence was found for degradation of PAHs or PCBs. Concentrations increased steadily during the anoxic incubation (Fig. 2) and because of the short residence time in the chamber water compared to sediment, it appears difficult to perceive that degradation would significantly reduce water concentrations during the oxic and bioturbation phases. Loss by volatilization was counteracted by low temperatures and a water atmosphere interface area of 2 cm^2 or about 1/1000 of the sediment surface. Finally and most important, the small differences between SPMDs exposed in source seawater and blanks as well as the frequent consistency between fluxes measured during flow-through and incubation set-ups (Table 3), gave no evidence for a serious bias from any such errors.

3. Results

3.1. Uptake in SPMDs in various water compartments and estimated water concentrations

Baseline results for comparison of uptake from various water compartments are shown in Table 3 (columns 2–5). The data showed that during flow-through measurements the uptake in membranes exposed in the source water flowing through the header tank was not much different from the uptake in membranes exposed in the sampling cells downstream the blanc and the boxes with capped harbour sediments. Many components rarely exceeded detection limits, e.g. the KPAHs (potentially most cancerogenous PAHs) (IARC, 1987), most of the PCB and all DDT components. In contrast, downstream harbour sediments without cap, uptake exceeded detection limits for most components, i.e. pp-DDD, all PCBs except PCB156 and PCB180 and all PAHs except benzo(*b*)-fluoranthene.

The accuracy of water concentrations calculated from SPMDs may be questioned. Therefore, a brief comparison with other investigations was made. In our samples single congeners of PCB increased from less than detection limit of 0.003–0.015 pM in the source seawater to typical concentrations of

Table 3
Uptake in SPMDs (U_{SPMD}), calculated fluxes (F) and cap efficiencies ($E_F=(F_{nocap}-F_{cap})/F_{nocap}$). U_{SPMD} represent mean of flow-through measurements only. ANOVA columns shows significant (s) or no significant (ns) difference between fluxes in (I) cap and no-cap treatments, and (II) no-cap treatments during anoxic (A), oxic (O) and bioturbation (B) phase

	U_{SPMD} (pmol SPMD^{-1})				F (pmol m^{-2} day^{-1})				ANOVA		Cap eff. %
	SW	Bl	Cap	No cap	Cap	No cap			$\alpha=0.05$		
						A	O	B	I	II	
No. of time periods	4	5	5	5	10	5	3	2	10	10	10
No. of boxes	–	1	5	2	5	2	2	2	7	2	7
No. of measurements	4	5	25	10	50	10	6	4	70	20	70
Acenaphthylene	14	20	23	68	8	20.8	16.9	25.5	s	ns	45–94
Acenaphthene	39	48	48	182	9	16.9	23.2	3.2	ns	ns	–
Fluorene	112	149	167	164	22	26.9	13.3	12.8	ns	ns	–
Phenanthrene	157	176	198	140	18	21.9	8.1	−5.5	ns	ns	–
Anthracene	21	19	26	63	3	90.0	7.4	3.5	s	A>B&O	50–142
Fluoranthene	284	257	45	67	3	830.3	−5.9	−18.0	s	A>B&O	−40–97
Pyrene	226	255	136	1845	42	2545.0	99.0	523.6	s	A>B&O	88–102
Benzo[*a*]anthracene[a]	<24	31	<13	65	3	98.1	3.4	18.8	s	A>B&O	37–111
Chrysene	36	44	<17	485	4	133.5	28.8	123.5	s	A&B>O	99–103
Benzo[*b*]fluoranthene[a]	<8	<11	<11	<11	2	31.6	2.0	0.2	s	A>B&O	89
Benzo[*j*,*k*]fluoranthene[a]	<30	67	<17	451	2	63.8	22.9	153.4	s	B>A>O	95–102
Benzo[*a*]pyrene[a]	<10	<21	<12	165	2	41.7	10.4	53.3	s	A&B>O	78–100
Indeno[1,2,3-*cd*]pyrene[a]	<8	<16	<12	62	2	5.3	6.1	9.6	s	ns	69–97
Dibenzo[*a*,*h*]anthracene[a]	<8	<10	<10	26	2	3.2	2.1	7.7	s	B>O	14–97
Benzo[*g*,*h*,*i*]perylene	<8	<32	<11	70	2	6.4	9.8	14.2	s	B>A	81–98
Sum PAH	889	1066	643	3853	125	3935	247	926	s	A>B&O	75–99
Sum KPAH	nd	98	nd	769	nd	244	47	243	s	A&B>O	93–100
PCB 28[a]	<3.4	4.5	4.6	11.4	1.07	1.40	0.57	2.69	ns	ns	–
PCB 52[a]	<4.2	<3.1	<3.1	17.9	0.37	0.97	0.30	5.17	s	B>A&O	54–96
PCB 101[a]	5.5	4.3	4.0	15.9	0.29	1.04	0.49	3.73	s	B>A&O	76–96
PCB 105	<2.7	<3.5	<2.9	4.7	0.43	0.75	0.29	0.96	s	A>O	24–78
PCB 118[a]	<3.2	<3.5	<2.9	7.8	0.24	0.49	0.27	1.96	s	B>A&O	53–89
PCB 138[a]	<2.4	<3.2	<2.6	9.9	0.25	0.46	0.32	2.55	s	B>A&O	44–92
PCB 153[a]	<2.9	<3.2	<4.2	10.6	0.37	0.56	0.45	3.06	s	B>A&O	34–94
PCB 156	<2.4	<3.2	<2.6	<2.5	0.22	0.19	0.32	0.19	ns	ns	–
PCB 180[a]	<2.2	<2.9	<2.4	<3.3	0.21	0.18	0.24	0.65	s	B>A	–
Sum PCB_7	5.5	8.8	8.6	78.2	1.1	5.7	nd	19.6	s	B>A&O	51–100
p,p′-DDE	<4.3	<5.0	<6.6	<8.0	0.60	0.36	0.49	1.07	ns	B>A&O	82
p,p′-DDD	<3.4	<4.7	<6.4	44.8	0.55	5.89	3.43	12.49	s	B>A&O	80–98
p,p'-DDT	<7.5	<7.1	<9.2	<7.8	0.51	0.45	0.49	0.49	ns	ns	–
Sum DDT	nd	nd	nd	44.8	nd	5.9	3.4	13.6	s	B>A&O	100

[a] Component contributing to sumKPAH or sumPCB_7.

0.030–0.150 pM in the no-cap bioturbation treatments. The latter was calculated from concentrations in SPMDs which were very similar to the concentrations observed in SPMDs exposed 1 m above the bottom in a dredging area in West Norway (Voie et al., 2002), and about 10–100 times higher than concentration levels in surface waters in the Skagerrak Sea (Palm et al., 2004) and the Southern Baltic (Bruhn and McLachlan, 2002). For PAHs the source water was dominated by phenanthrene (1.1–2.8 pM), fluoranthene (0.2–1.8 pM) and pyrene (0.4–1.3 pM) which were within the same order of magnitude as those reported for the Skagerrak Sea (0.2–14.6 pM, Palm et al., 2004) and the Southern Baltic Sea (0.02–7.4 pM, Witt, 2002).

3.2. Fluxes

Mean fluxes calculated from uptake in SPMDs during all experimental phases are shown in columns 6–9 in Table 3. Flux variations due to experimental phase (anoxic, oxic, bioturbation) or treatment (box) are shown in Figs. 3 and 4. The plotted values are least square mean fluxes calculated in a two-way analysis of variance (ANOVA; effects of phase and treatment) and were

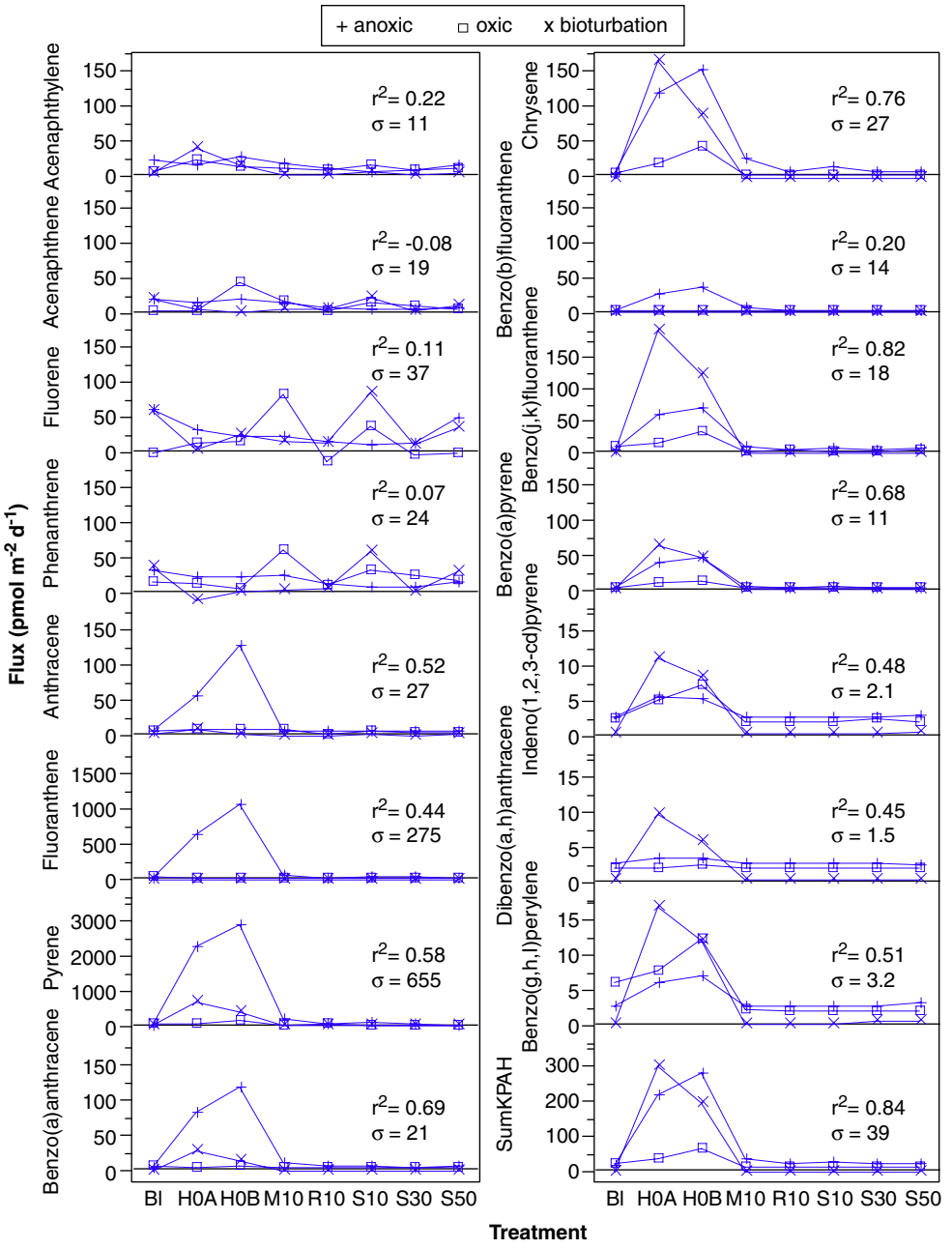

Fig. 3. Least square mean flux (pmol m^{-2} day^{-1}) of PAHs from experimental blank (Bl), uncapped (H0A–H0B) and capped (M10–S50) harbour sediments during the three experimental phases. r^2 = whole model correlation coefficient (adjusted). σ = root mean square error.

marginally different from the mean fluxes given in Table 3. The correlation coefficients (r^2 adjusted) displayed in the diagrams showed that the two factors could explain a high proportion of the variability in major and sum components ($0.65 < r^2 < 0.85$ for sumKPAH, sumPCB_7 and sumDDT) and significant interaction effects were found for most components as indicated by flux variations exceeding the root mean square error (σ).

The figure shows generally low fluxes in the blank and in all boxes with capped sediments, but frequently elevated fluxes in the two boxes with uncapped harbour sediments. Thus, fluxes of most PCBs and DDTs and many PAHs were not detectable in capped sediments, whereas only PCB156 and ppDDT were not detectable in no-cap treatments. In the blank box, elevated fluxes of acenaphthylene during the anoxic phase were most

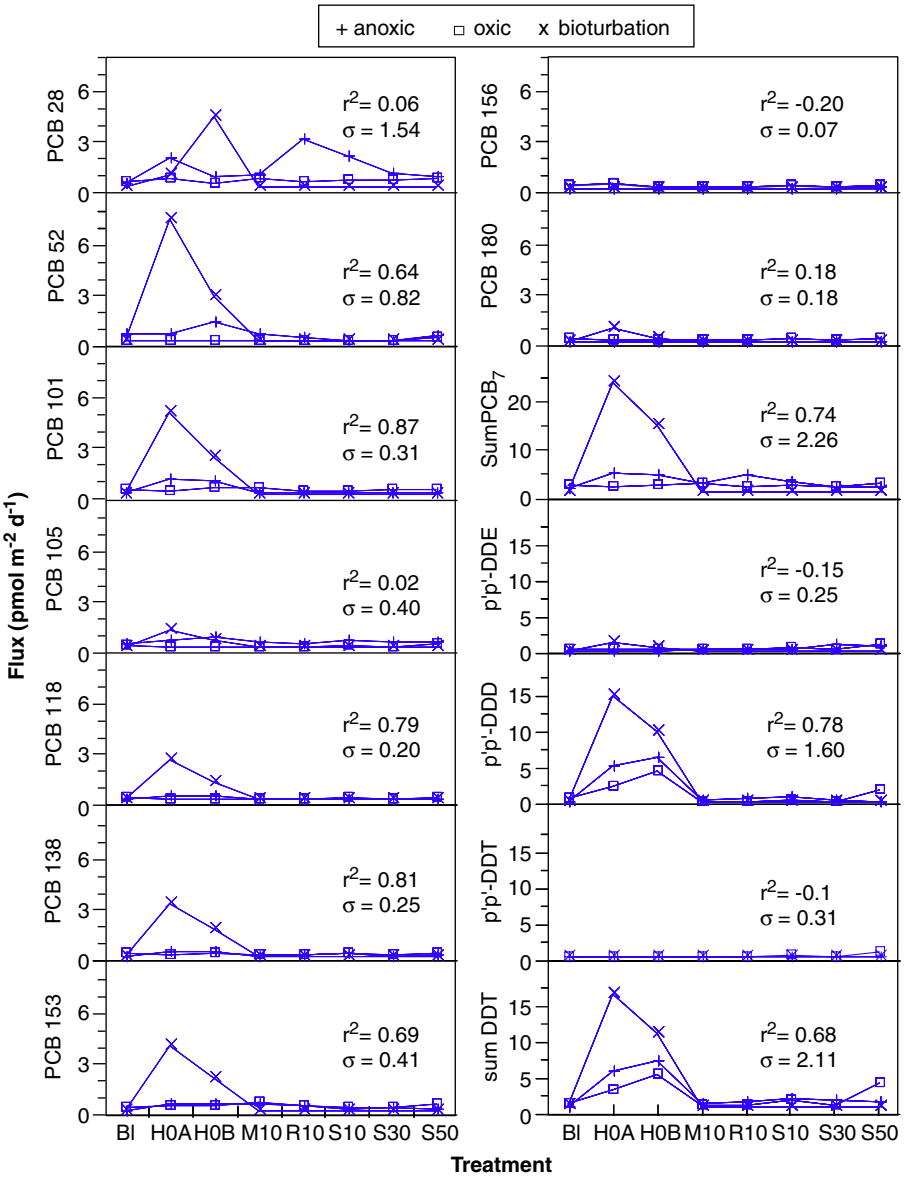

Fig. 4. Least square mean flux (pmol m^{-2} day^{-1}) of PCBs and DDTs from experimental blanc (Bl), uncapped (H0A–H0B)) and capped (M10–S50) harbour sediments during the three experimental phases. r^2 = whole model correlation coefficient (adjusted). σ = root mean square error.

probably a result of contamination from glue solvents applied during box construction (see above).

During the anoxic phase, the flux of PAH from no-cap treatments was dominated by the high release of pyrene (2545 pmol m^{-2} day^{-1}) and fluoranthene (830 pmol m^{-2} day^{-1}) which together accounted for 86% of the total flux of PAHs. The dominance of these two components decreased to account for a much smaller fraction (39–56%) of the total flux during the subsequent oxic and bioturbation periods. The KPAHs were dominated by the three components benzo(*j*,*k*)fluoranthene, benzo(*a*)anthracene and benzo(*a*)pyrene which accounted for 78–93% of the total flux of KPAHs throughout all experimental phases.

The flux of PCBs never exceeded 5.2 pmol m^{-2} day^{-1} for single components with a maximum sum flux of 19.8 pmol m^{-2} day^{-1} during the bioturbation phase. Fluxes of PCBs were not detectable during the oxic phase, whereas the fluxes during the bioturbation phase was typically 3–5 times larger than those observed during the anoxic phase. The fluxes of 3.4–13.6 pmol

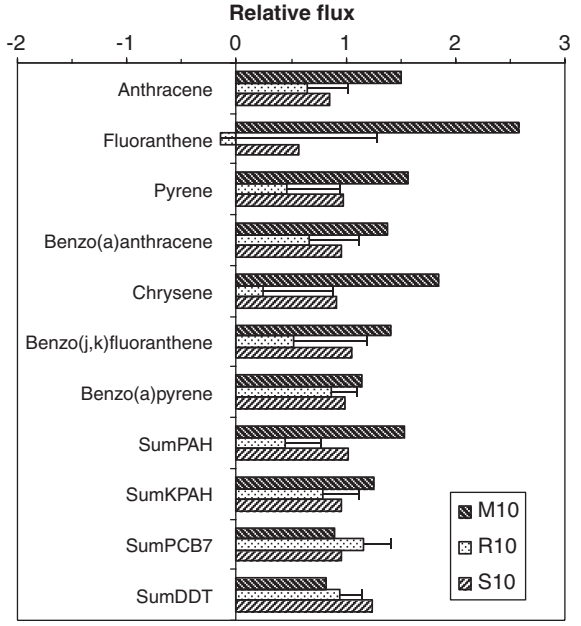

Fig. 5. Fluxes from the three different types of cap material; sand (S10), raw sand (R10) and machined sand (M10), normalised against mean flux of the respective component. The relative standard error is shown by bars on S10. $n=30$.

m^{-2} day^{-1} pp-DDD appeared relatively high compared to the observed fluxes of PCB.

3.3. Cap efficiency

Table 3 also shows the results of two separate analyses of variance using Tukey's test for significant differences between treatments. In column 10 (ANOVA I), the analyses was performed omitting the blank and grouping the boxes to reduce the number of treatments to cap or no-cap. This analysis revealed significant ($p<0.05$) flux reduction in cap treatments for all major components contributing to sumPAH, sumPCB and sumDDT.

Cap efficiencies were calculated for each experimental period as the difference of the flux between no-cap and cap treatments divided by the flux from the no-cap treatments ($E_F=(F_{nocap}-F_{cap})/F_{nocap}$). The range shown in Table 3 represents the range calculated for the three periods. By assigning zero to non-detectable fluxes, the cap efficiencies were somewhat over-estimated compared to what they might have been using alternative methods for handling of non-detectable fluxes. Over-estimated cap efficiencies applied, however, only to components with release rates close to detection limits, i.e. those components for which the predicted environmental benefits from capping will be marginal regardless of calculation method.

A cap of 10 cm was sufficient to eliminate the flux of PCBs and PAHs from the contaminated sediments in the mesocosm experiment. Technically, a 10-cm layer may be infeasible to place uniformly at 60 m depth and some deep-burrowing animals are known to penetrate many marine sediments down to depths of about 20 cm. Therefore, a cap thickness of 50 cm has been proposed for the deposit area in the fjord basin.

3.4. Effects of experimental phase

Column 11 (ANOVA II) of Table 3, shows the results of a Tukey comparison of no-cap treatments between different experimental phases. Results presented in the table such as "B > A&O" means that the flux during the bioturbation (B) period was significantly ($p<0.05$) higher than the flux during both anoxic (A) and oxic (O) periods, but that no significant difference ($p>0.05$) was found between the anoxic and the oxic period.

Some intermediate PAHs (anthracene, benzo(*a*)anthracene, benzo(*b*)fluoranthene and in particular fluoranthene and pyrene), showed the highest fluxes during the initial anoxic phase., However, most PCBs, ppDDE, ppDDD, chrysene, benzo(*j*,*k*)fluoranthene, benzo(*a*)pyrene, dibenzo(*a*,*h*)anthracene and benzo(*g*,*h*,*i*)perylene showed significantly higher fluxes during the bioturbation period than during one or both of the preceding periods. Thus, bioturbation was shown not only to enhance resuspension of particle-bound contaminants, but had a significant effect increasing the release of fractions of many PAH, PCB and DDT components available for uptake in SPMDs and hence in aquatic organisms (Meadows et al., 1998). This has also been observed during similar experimental work on sediments contaminated with heavy metals (Skei, 1992).

3.5. Cap material differences

Differences between the various caps (mineralogy and layer thickness) were small. However, machined sand released consistently more PAHs (mean M10, all phases = 243 pmol m^{-2} day^{-1} sumPAH, $n=10$) than the other cap treatments (mean all cap treatments, all phases = 125 pmol m^{-2} day^{-1}, $n=50$). The difference was larger during the initial anoxic phase than during the subsequent oxic and bioturbation phases. One-way ANOVA showed that during the anoxic phase, the release of sumPAH was significantly higher from M10 than from R10, but not significantly higher than S10 ($\alpha=0.05$). The major components contributing to

the higher release of PAHs from M10 is shown in Fig. 5. M10 was the only treatment with cap material that had been processed before use in the experiment. PAH was hardly detectable (<0.05 μmol kg^{-1} dry weight) in any of the cap materials, but slight contamination during out-door processing involving explosives and diesel driven equipment appeared nevertheless to be the most probable explanation on the enhanced release of PAHs from M10.

3.6. Bioaccumulation

The concentration of PAH and PCB in *H. reticulata* after nine months exposure in cap and no-cap treatments are shown in Table 4. Accumulation of PCBs in gastropodes may be related to feeding or respiratory behaviour and is not necessarily representative for other sediment-dwelling species. The organism spends all of its time confined within the top few cm of the sediment, feeding mostly on organic material at the sediment surface. For the cap treatments, direct exposure to the harbour sediment below the 10-cm cap layer was not likely.

The concentration of 52 nmol kg^{-1} PAH in the gastropodes exposed in the cap layers was low compared to 1367 nmol kg^{-1} in the gastropodes exposed in the harbour sediments and corresponded to a cap efficiency of 96% (Table 4). For PCB the reduction from 249 nmol kg^{-1} in no-cap to 98 nmol kg^{-1} in cap treatments corresponded to a gastropode cap efficiency of only 60%. Neither concentration gradients within the cap nor uptake in SPMDs in the water above indicated any leakage neither of PAH nor PCB through the cap, but the idea that the concentration of PCBs in the gastropodes exposed in the cap was higher than expected was further substantiated by the ratios shown in Table 4. Thus, the PAH/PCB concentration ratio in gastropodes exposed in cap treatments was low compared with the ratios observed in other media and treatments and the gastropode/SPMD PCB concentration ratio in the cap treatments was high compared with the no-cap treatments. This will be further discussed below.

4. Discussion

4.1. SPMDs vs. real organisms

Uptake through specific feeding and respiration strategies as well as metabolism and excretion may provide different uptake patterns between real organisms and SPMDs. Metabolism and excretion is likely to reduce gastropode concentrations of PAHs faster than PCBs (Christensen et al., 2002; Ruus et al., 2005) and thereby explain the low PAH:PCB ratio of 0.5 shown in Table 4. Such processes appeared, however, not appropriate to explain the high gastropode:SPMD ratio of >33 for PCB in the cap treatments. The gastropode is known to feed selectively on organic food particles on the sediment surface and the concentration of PCBs in organic particles in Baltic deep water (40 and 91 m depth) has been shown to be an order of magnitude larger than in the surface water (Axelman et al., 2000). Food was not likely abundant in the cap treatments, and PCB-enriched organic particles introduced via the source seawater from 60 m depth in the Oslofjord might represent a significant source for uptake in the gastropodes. SPMDs primarily samples from the dissolved phase and are generally assumed not to accumulate PCB associated with particles. Thus, difference with respect to

Table 4
Concentration of PAHs and PCBs in harbour sediments and cap (nmol kg^{-1} wet weight) sampled at the end of the experiment, in SPMDs (nmol kg^{-1} triolein) sampled during the oxic and bioturbation phase and in the gastropode *Hinia reticulata* (nmol kg^{-1} wet weight) after 9 months exposure in the top layer (0–3 cm) of cap and no-cap treatments

	SumPAH		SumPCB		PAH/PCB ratio
	Range	Mean	Range	Mean	
Harbour sediment	–	127000	–	341	372
Sediment cap 0–7.5 cm	–	<140	–	<10	–
SPMD in cap	181–705	367	<3	<3	>122
SPMD in no-cap	3500–7860	4119	80–166	92	45
Gastropodes in cap	10–148	52	86–113	98	0.5
Gastropodes in no-cap	1323–1409	1367	228–270	249	5.5
Gastropode:SPMD ratio cap	–	0.14	–	>33	–
Gastropode:SPMD ratio no-cap	–	0.33	–	2.7	–
Cap efficiency (gastropodes)	89–99%	96%	55–65%	60%	–

Cap efficiency = $(C_{nocap} - C_{cap})/C_{nocap}$.

accumulation of PAHs and PCBs in SPMDs and gastropodes tend to suggest that although the diffusive and bioturbative flux from contaminated harbour sediments was efficiently reduced by capping, back-ground levels of PCBs on suspended particles may represent a relatively more important source of PCBs than of PAHs.

4.2. Impact of redox conditions on fluxes of pyrene and fluoranthene

The release to the overlying water of many PAH, PCB and DDT components was lower during the oxic than during the anoxic oxic phase (Table 3). This should be interpreted with some care due to the different methods applied during the anoxic and the subsequent periods. However, the decrease of the flux of pyrene and fluoranthene was too large to be explained by such factors. Pyrene and fluoranthene are characterised by high uptake rates in SPMDs (Huckins et al., 1999), rapid release from harbour sediments resuspended in anoxic seawater (Schaanning et al., 2000), short residence times in the water column (Ko et al., 2003) and rapid accumulation in benthic organisms exposed to harbour sediments (Ruus et al., 2005). Initial mixing of the sediment may increase initial pore water concentrations and release to the overlying water as indicated by studies of changes in pyrene bioaccumulation and dietary routes in ageing sediment slurries (Conrad et al., 2002; Van Hoof et al., 2001). Chin and Gschwend (1992) showed that a higher fraction of pyrene than phenanthrene was mobilised by binding to pore water colloids in Boston harbour sediments. This is opposite to frequent observations where dissolved organic matter tends to reduce the bioavailability of organic chemicals (Haitzer et al., 1998). However, Verrengia Guerrero et al. (2003) found that bioaccumulation of pyrene in clams was significantly enhanced in the presence of humic acids in water, natural sediments and artificial particles with low binding capacity for pyrene. Furthermore, Hunchak-Kariouk and Suffet (1994) showed that oxidation and precipitation of iron reduced the concentration of dissolved organic matter in the pore water to a fraction of the concentration in the anoxic pore water. As indicated by the shift from a black to a light grey sediment surface (upper 1 cm), precipitation of iron oxides is likely to have occurred in the near-surface sediments during the shift from anoxic to oxic conditions. Therefore, coprecipitation of dissolved or colloidal organic matter and associated contaminants appears to be the most likely process to explain the concurrent decrease of the release of pyrene and fluoranthene from the sediment.

4.3. The importance of source control

Sediment remediation efforts either by capping or by dredging and removal of the contaminated layer will have little environmental benefits unless the input of contaminants from other sources has been reduced. Results of recent dredging operations performed on contaminated sediments at a naval base on the west-coast of Norway, did not yield the expected reduction of PCB levels in mussels and SPMDs exposed one meter above the bottom in the dredged area 2–7 months after dredging had been commenced (Voie et al., 2002). The concentration of PCBs in the SPMDs ranged 3–18 pmol (Voie et al., 2002) which was not much different from those obtained in the present no-cap treatments (Table 3). This corresponded to a typical water concentration of 0.015–0.15 pM maintained by sediment efflux of about 3 pmol m^{-2} day^{-1} at water renewal rates of 0.2 day^{-1}. A sediment release of 3 pmol m^{-2} day^{-1} gives a daily input of 0.003 pM to the water layer up to 1 m above the bottom. Disregarding horizontal water exchange near the bottom, the release from the sediment will be transported upwards in the water column by vertical eddy diffusion alone. Under such conditions, the steady state concentration gradient in the watermass can be estimated from $dC/dz = F/D$ in which F is the flux and D is the eddy diffusion coefficient for the actual water column. In deep fjord environments a typical D-value may be 0.1 cm^2 s^{-1} (Gade, 1968). Applied on fluxes of 3 pmol m^{-2} day^{-1} yield characteristic water column concentration gradients of 0.0006 pM m^{-1}. From a remediation area with limited horizontal extension and shallow water depths of about 10 m or less, lateral exchange with watermasses from presumably less polluted areas will counteract accumulation of contaminants few meters above the bottom. It appears unlikely that even 100% flux reduction induced by remediation efforts would yield measurable (typical standard deviations = 0.01–0.06 pM between similarly treated boxes) change of concentration in the water one meter above the bottom. Both before and after dredging the uptake of PCBs were probably controlled by water concentrations derived from other sources than the flux of dissolved components from sediments. The fjord environment is likely to be more dynamic than the environment in the experimental chambers and desorption from resuspended fine particles escaped from the dredging operation might represent one such source in addition to other local sources and background input.

Measured fluxes of PCBs in the oceanic environment have rarely been reported. Palm et al. (2004) found atmospheric deposition rates of 1–4 pmol m^{-2}

day^{-1} PCB28 and 0.6–2.2 pmol m^{-2} day^{-1} PCB180/193 on a Skagerrak location remote from known contaminant sources. Atmospheric concentrations of PCB often increase by two orders of magnitude between rural and urban areas in Europe (Jaward et al., 2004). Therefore, the release of 2–4 pmol m^{-2} day^{-1} PCB28 and <0.1 pmol m^{-2} day^{-1} from the no-cap bioturbation treatments indicated that the recycling of PCBs from historically contaminated sediments to the water was small compared to expected atmospheric inputs. However, the recycling determined here was assumed 100% bioavailable, whereas the atmospheric input on the Skagerrak location was of the same magnitude as the sedimentation through the water column (Palm et al., 2004) and consequently little left for accumulation in water and biota. Thus, allthough the diffusion and bioturbation driven PCB recycling from the sediments appeared small compared to total input from other sources, the potential benefits from remediation of historically contaminated sediments can only be assessed by comparing fluxes of bioavailable fractions. In addition to the recycling measured above, the direct uptake in organisms exposed in the sediments and the desorption from resuspended sediments are considered the major pathways of contaminant recycling from the sediment.

5. Conclusions

Significant fluxes of PAHs, PCBs and DDTs from contaminated harbour sediments to overlying water were measured during all experimental phases by increased uptake in SPMDs. Fractions available for uptake in SPMDs are assumed to be dissolved and highly available for uptake in aquatic organisms. For most components, the highest fluxes were measured after introduction of bioturbating organisms. Fluxes of pyrene and fluoranthene, however, decreased after the initial anoxic phase, most likely due to iron oxidation and co-precipitation with dissolved organic matter to reduce pore water concentrations in near-surface sediment layers. Covering the harbour sediments with a 10–50 cm cap of clean, sandy material practically eliminated the release of all major components contributing to the total flux of PAHs, PCBs and DDTs. Only machined sand showed a minor release of some PAH-components, presumably as result of contamination during processing. The cap reduced both sediment efflux and bioaccumulation in *H. reticulata* by 89–100% for PAH, whereas bioaccumulation of PCBs was only reduced by 55–65%. This might indicate that PCB background contamination is large compared to the diffusive release of PCB from uncapped harbour sediments.

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