Distribution of Polycyclic Aromatic Hydrocarbons (PAHs) in Pore Water and Sediment of a Small Aquatic Ecosystem

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DISTRIBUTION OF POLYCYCLIC AROMATIC HYDROCARBONS (PAHs) IN PORE WATER AND SEDIMENT OF A SMALL AQUATIC ECOSYSTEM

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The concentrations of polycyclic aromatic hydrocarbons (PAHs) were measured both in pore water and solid sediment isolated from the Teufelsweiher pond, a small aquatic ecosystem in Upper Bavaria (Germany) from 1992 to 1994. All 16 EPA priority compounds were present with mean total concentrations from 501 to 663 ng/l in the pore water and 976–1600 μg/kg (dry weight) in the solid sediment. Fluoranthene and pyrene were the dominant compounds and accounted for 21–28% of the total PAHs, the ratios of fluoranthene/pyrene and phenanthrene/anthracene ranging from 1.61–2.86 and 0.86–1.57, respectively. These results suggest that the main sources of PAHs are incomplete combustion processes of fossil fuel; the compounds are then transported into the Teufelsweiher by deposition and runoff. The distribution of PAH concentrations exhibit a pronounced dependence on both particle size and organic matter content of the sediment: two fractions (< 6.3 μm and 63–200 μm), containing higher contents of organic matter, trapped more PAHs compared with other fractions. The dissolved organic carbon (DOC) in the pore water is mostly responsible for lower K_{OC}' *in situ* measured compared with the estimated K_{OC}. The spatial and temporal variations of PAH concentrations are also discussed.

Keywords: PAHs; aquatic ecosystem; sediment; pore water; particle size; organic matter

INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are ubiquitous environmental pollutants. As a result of their hydrophobicity and strong affinity towards particulate and organic matter, PAHs accumulate in sediments to levels several orders of

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magnitude higher than those found in surface waters.[1-3] Less is known yet about PAH concentrations and distribution in pore water.[4-5] Recent measurements have shown surprisingly high concentrations of hydrophobic organic chemicals (HOC, e.g. PCBs, DDT, PAHs, etc.) in pore water.[3-7] With respect to PAHs opposing observations have been made: on the one hand PAH concentrations in pore water were overestimated because only a fraction of the PAHs measured in sediments is available to partition into pore water;[4-5] on the other hand they tend to associate with dissolved organic matter (DOM) or colloids in pore water, which are able to enhance the pore water concentration of these compounds beyond values predicted by their solubility in water.[8-9] Thus, patterns of PAH distribution between sediment and pore water are of importance for the establishment of sediment quality criteria (SQC) and for their mobility, conversion, as well as availability in the environment, since these compounds can adversely affect aquatic ecosystems and result in high concentrations in the tissues of indigenous organisms.[10] Some PAHs exhibit a strong mutagenic or carcinogenic potential to many groups of organisms including humans.[11] The accumulation of high concentrations of PAHs in marine sediments in industrialized and densely populated regions has been well established,[2,12] pollution and distribution of PAHs in aquatic sediments of agricultural areas, however, are less investigated.[13,14]

PAHs in the environment may originate in both anthropogenic and natural sources. Generally, high PAH concentrations in sediment are linked to anthropogenic origins, i.e. incomplete combustion of fossil fuels as well as pyrolysis processes.[12,13] PAHs are carried into surface waters of the aquatic environment by atmospheric deposition, runoff, erosion etc. In the aquatic system they are transported, distributed and accumulated in sediments mostly in association with particles, however, not evenly among all particle sizes and densities.[15,16] Thus, examining the PAH content of particulate matter enables a better understanding of the source, transport, and transformation processes controlling the distribution of PAHs in the aquatic environment. Consequently, the vertical and spatial distribution of PAHs in sediments provides useful information for evaluating the bioavailability of PAHs for aquatic organisms and for the identification of former and current pollution sources.

In the Teufelsweiher, an aquatic ecosystem at the experimental farm of the joint research project "Research Network on Agroecosystems Munich (FAM)" in Scheyern, the pollution and distribution of environmental contaminants in the sediments as one part of the project have been investigated. In this study, the concentrations of 16 PAHs (US-EPA "Priority Pollutant List") were determined in the sediment and pore water collected from the Teufelsweiher. The objectives were: 1. to determine the PAH concentrations in the Teufelsweiher; 2. to observe

TABLE I   Investigated PAHs and comparison between their *in situ* measured K_{OC}' and estimated K_{OC}

PAH (IUPAC name)	abbreviation	site 1		$K_{OC}*$ [l/kg]
		K_D'	K_{OC}'	
naphthalene	Nap	0.5×10^2	1.4×10^3	1.1×10^3
acenaphthylene	Acy	2.0×10^2	5.5×10^3	5.7×10^3
acenaphthene	Ace	2.2×10^2	6.3×10^3	1.0×10^4
fluorene	Flu	1.6×10^2	4.5×10^3	7.3×10^3
phenanthrene	Phe	4.0×10^2	1.1×10^4	1.4×10^4
anthracene	Ant	4.7×10^2	1.3×10^4	1.4×10^4
fluoranthene	Fla	2.8×10^3	8.0×10^4	1.0×10^5
pyrene	Pyr	2.2×10^3	6.2×10^4	1.0×10^5
benzo(a)anthracene	BaA	3.1×10^3	8.8×10^4	2.0×10^5
chrysene	Cry	3.4×10^3	9.6×10^4	2.0×10^5
benzo(b)fluoranthene	BbF	3.7×10^4	1.1×10^6	1.8×10^6
benzo(k)fluoranthene	BkF	3.7×10^4	1.1×10^6	3.3×10^6
benzo(a)pyrene	BaP	1.5×10^4	4.4×10^5	5.3×10^5
dibenzo(ah)anthracene	DbA	1.9×10^4	3.4×10^5	4.5×10^5
benzo(ghi)perylene	Bgh	2.4×10^5	6.8×10^6	8.2×10^6
indeno(1,2,3-cd)pyrene	Ind	4.8×10^5	1.4×10^7	2.2×10^7

K_D' = equilibrium coefficient, K_{OC}' = equilibrium coefficient referred to organic matter content
*calculated with $\log K_{OC} = \log K_{OW} - 0,317$[33]

both the vertical and spatial distribution of PAHs; 3. to study the particle-size distribution of PAHs; 4. to study the influence of the organic matter (including DOC) of sediments on PAH distribution; 5. to evaluate the possible sources of PAHs in the Teufelsweiher.

MATERIALS AND METHODS

Chemicals

All chemicals were purchased from Merck (Darmstadt, Germany). Solvents were HPLC-grade and water was supplied by a Milli-Q-apparatus (Milli-Q plus 185, Millipore, Bedford, USA). The 16-PAH standard SRM 16371 was obtained from Promochem (Wesel, Germany); the IUPAC names and abbreviations of the individual compounds are given in Table I.

Sediment Sampling

The Teufelsweiher pond (area: $3155 m^2$, depth: 0.3–1.3 m) is located 40 km north of Munich (Upper Bavaria, Germany). Sediment samples from 5 sites, which were selected to represent sediments of the pond were collected from October
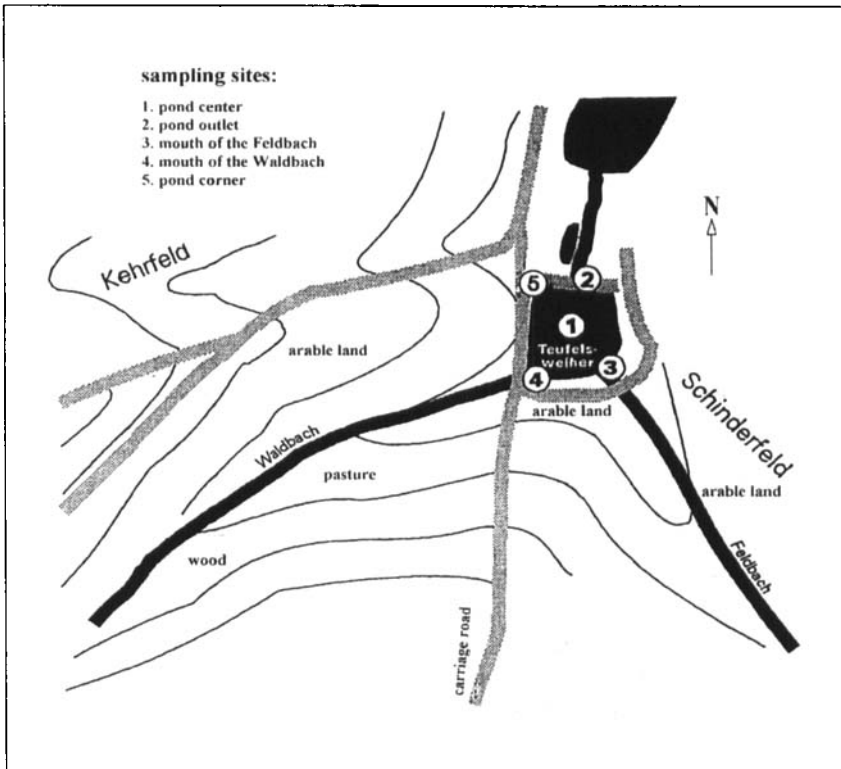
FIGURE 1 ´ Study area and sampling sites in the Teufelsweiher

1992 to October 1994 with a gravity UWITEC-corer® (flatter valve, ID 60 mm). The study area and core sampling sites are shown in Figure 1. All sediment sample cores were divided into three layers: 0–2, 2–10 and 10–15 cm. The general physical and chemical characteristics of the sediments are summarized in Table II. The sediments were collected in solvent-washed glass jars and stored in the dark at $-20°C$ until analysis.

Sample Processing

Prior to use, all glassware was solvent rinsed and then heated overnight at 400°C. The sediment samples were transferred in a stainless-steel container (d = 100 mm, v = 1000 ml, Sartorius, Göttingen, Germany) and squeezed through a 0.45 μm membrane (cellulose nitrate, Sartorius, Göttingen, Germany) under nitrogen pressure (5–10 bar). The separated pore water (200–500 ml) was then extracted

TABLE II Physiological properties of the sediments (0–2 cm, 26 October 1992) in the Teufelsweiher

sampling site	texture [%]			pH*	DOC [mg/l]	C_{org}** [%]	C/N***
	clay	silt	sand				
1	19	55	26	8.0	47.2	3.6	10.1
2	18	57	25	7.8	41.5	3.8	9.2
3	19	54	27	8.0	43.0	3.7	9.9
4	20	52	28	8.0	50.2	3.6	10.3
5	21	51	28	8.1	48.7	4.2	10.4

*measured in the sediment extract in 0.01 N $CaCl_2$ solution (sediment:solution = 1:2)
**organic carbon content per dry weight (DW)
***carbon/nitrogen ratio, data from GSF-Institute of Hydrology, Neuherberg, Germany

by a C18 column (1g, Baker, Phillipsburg, NJ, USA) at a flow rate of 1–2 ml/min which was eluted afterwards 3 times with 6 ml of acetone. The extract was concentrated by means of a rotary evaporator (Büchi, Flawil, Switzerland) to about 1 ml and dried under a gentle stream of nitrogen. The residue was dissolved in 2 ml of cyclohexane/ethyl acetate (v:v = 1:1) and subjected to gel-permeation chromatography (GPC) on a 1.2 × 150 cm Bio-Beads S-X3 column (Bio-Rad Labs, Richmond, USA) to remove the higher molecular fraction of the sample that interferes with the analysis. Using cyclohexane/ethyl acetate (v:v = 1:1) as solvent at a flow rate of 3 ml/min the PAH fraction eluted between 47.2 and 84.4 min and was then concentrated to about 1 ml and dried under a gentle stream of nitrogen. The residue was dissolved in 1 ml of acetonitrile and analyzed for PAHs with GC-MS.

The air-dried sediment was homogenized in an agate mortar and passed through a 1 mm stainless-steel sieve (Retsch, Haan, Germany). Samples of 25 g were transferred into a centrifuge tube with a Teflon-tightened screw cap and extracted with 50 ml of n-hexane/acetone mixture (v:v = 9:1) in an ultrasonic bath (Sonorex RK 255H, Bandelin, Germany) for 2 h. Then the suspension was centrifuged at 3600 g for 15 min and the supernatant was decanted. The extraction procedure was repeated two times. The residual water and sulfur compounds of the whole extract solution were removed by a short column packed with 0.8 g of activated copper powder and 10 g of anhydrous Na_2SO_4. The eluate was concentrated to about 1 ml and dried under a gentle stream of nitrogen. The residue was dissolved in 2 ml of cyclohexane/ethyl acetate (v:v = 1:1) and further treated as the pore water extract (see above).

The water content of the sediment was determined by heating an aliquot of the filtered sample at 105°C for 48 h. The total organic carbon (TOC, not filtered) and dissolved organic carbon content (DOC, 0.45 μm filtered) in the pore water were determined with a TOC analyzer (Shimadzu TOC-5050, Tokyo, Japan) calculating the difference between total and inorganic carbon. The or-

ganic carbon content (C_{org}) of the solid phase of the sediment was determined with an OC analyzer (CSA 302, Leybold-Heraeus, Hanau, Germany).

Instruments and Chromatographic Analysis

Determination and quantification of PAHs were carried out by computerized capillary GC-FID. The Perkin Elmer GC 8000 AutoSystem was equipped with a coated PVMS/54 chemically fused silica column (50 m $\times$ 0.32 mm ID, phase thickness 1.0 μm, Perkin Elmer, USA), an autosampler, a FID, a split/splitless injector, an interface (900 series), and a computer system for data acquisition. The analytical conditions were set as follows: the oven temperature was set to 70°C for 2 min and then programmed to increase to 250°C at a rate of 15°C/min, this temperature was held for 1 min and then increased to 290°C at 4°C/min and held for 29 min. 2 μl samples were injected; hydrogen was used as carrier gas (1.0 ml/min), hydrogen (35 ml/min) and air (300 ml/min) were used as combustion gases, FID and injector temperature were set at 300°C and 280°C, respectively. The split was opened 1.5 min after injection. Quantification was performed by comparing peak areas of PAHs with those of an external standard.

Identification of PAHs was carried out by a GC-MS system. The HP 5890 series II plus GC system was equipped with a HP-5 M.S. fused silica capillary column (30 m $\times$ 0.25 mm ID, film thickness: 0.25 μm, phase: crosslinked 5% Ph Me silicone, Hewlett Packard, USA), an HP 7673 autosampler, a split/splitless injector, and an interface (900 series). The HP 5989A quadrupole MS system was run with a HP-ChemStation (revision C, 02. 05) for data acquisition. The measuring conditions were as follows: the oven temperature was set to 50°C for 1 min and then programmed to increase to 120°C at 45°C/min, this temperature was held for 1 min and then increased to 250°C at 5°C/min and held for 20 min. 1 μl samples were injected; helium was used as carrier gas (1.0 ml/min), the injector was set at 250°C, the split was opened 1.0 min after injection. The MS was run in the total ion current (TIC) mode. MS conditions were as follows: electron energy 70 eV, source temperature 200°C, quadrupole temperature 100°C, transfer line 280°C, scan range 50–350 amu at 2.0 scans/sec. Peak identities were verified by comparison with the mass spectra of authentic standards and the National Institute of Standards and Technology (NIST) mass spectral library supplied with the instrument.

The recoveries from both the spiked sediment and pore water ranged from 80 to 90%, a loss of PAHs during sample preparation being considerable mainly for the lower molecular weight PAHs. Analyses of triplicate pore water or sediment sub-samples indicated a reproducibility of ±4–6% of the concentration means. The standard deviations of the PAH concentrations of three equal sam-

ples ranged between 2.2 and 5.8% depending on the PAH compound. Detection limits of the method were typically 31–100 ng/l in the pore water and 10–32 μg/kg (dry weight, DW) in the solid sediment for individual compounds.

Particle-Size Fractionation

The fractionation methods and procedures summarized here have been described in detail elsewhere.[16] 25 g of air-dried sediment were transferred into a 200 ml flask, 100 ml 0.01 M $Na_4P_2O_7$ were added and the flask was shaken for 16 h. The suspension was then separated by two stainless-steel sieves (d = 63 and 200 μm) into three factions: >200, 63–200, and <63 μm. The last one was further separated with the elutriation system into five fractions: <2, 2–6.3, 6.3–12.5, 12.5–20, and 20–63 μm. The flow rate of the dispersion solution, which contained 0.2 g/l $Na_4P_2O_7$ was set to 11.6 ml/min. After the separation all fractions were centrifuged at 5000 g for 30 min (Cryofuge 8000, Heraeus Christ, Osterode/Harz, Germany) and freeze-dried (Gamma A, Christ, Osterode/Harz, Germany). An aliquot of each fraction was measured with a laser interferometric system (Sympatec Helos, Clausthal, Germany).

RESULTS AND DISCUSSION

PAH Concentration in Pore Water

All 16 EPA priority PAH compounds were present in the Teufelsweiher. The PAH concentrations in the pore water vary almost uniformly depending on both the sample site and the season (Figures 2 and 3). The PAHs in the pore water exhibit a distribution similar to that of the accompanying sediments (see discussion below). The highest concentrations were found in the middle of the pond (site 1) and decreased at the entries of the two brooks (site 3 and 4), "Feldbach" and "Waldbach" (see Figure 1). The lowest concentrations were found at the outlet (site 2) and in the corner (site 5) of the pond. These observations are supported by the distribution of PAH concentrations in the surface water of the two brooks, in which 10–70 ng/l of PAHs were determined, a decrease was observed in the water of the pond outlet.[17] This decrease is attributed apart from photooxidation and reception by organisms mainly to the sedimentation of PAH containing suspended particles that are transferred from the two brooks. Mean total concentrations of PAHs in pore waters ranged from 501 to 663 ng/l during the measuring periods. As expected, the concentration
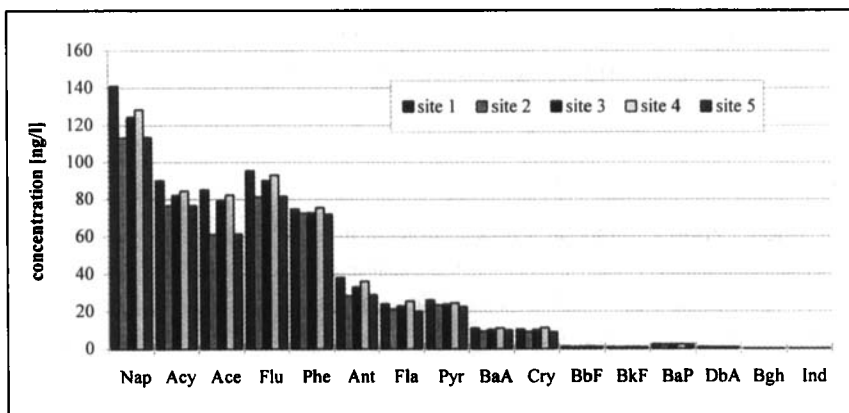
FIGURE 2   PAH concentrations in the pore water (0–2 cm) at the different sampling sites in the Teufelsweiher pond

of PAHs in the pore water tends to decrease as their solubility in water decreases (Figure 4).

The total PAH concentration in the pore water is linear correlated with the DOC content: $[PAH]_{total} = 210.94 + 14.08 \times [DOC]$ ($r^2 = 0.89$), where $[PAH]_{total}$ and $[DOC]$ represent the total PAH concentration (ng/l) and the DOC content (mg/l) of the pore water, respectively. This so-called "solubilization effect" is especially effective for the large rings which are nonpolar and have a high K_{OW} value (Figure 5). This indicates that the composition and concentration
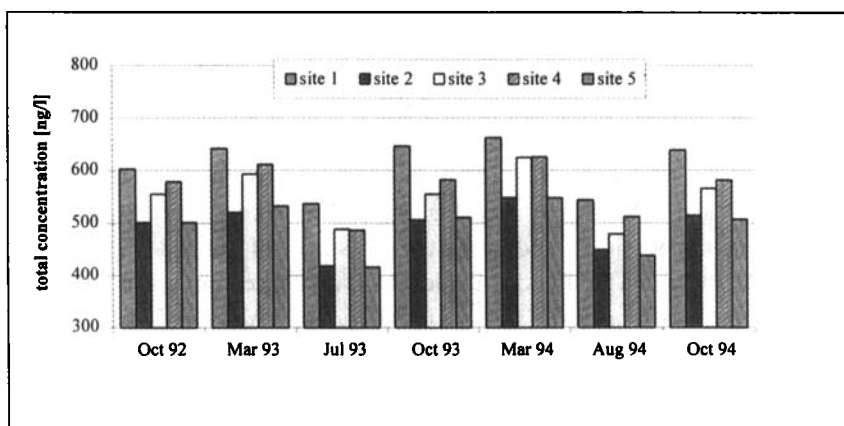

FIGURE 3   Seasonal variation of total PAH concentrations in the pore water from the Teufelsweiher pond
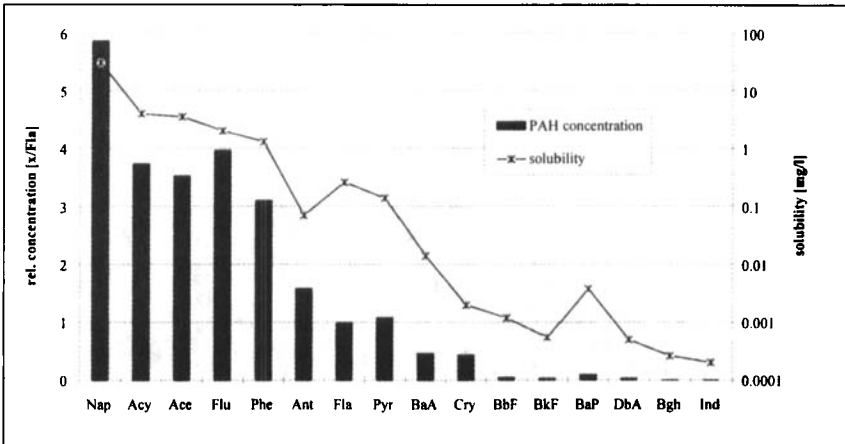
FIGURE 4 PAH profile (normalized by Fla) in the pore water from the Teufelsweiher pond (site 1, 26 October 1992) and water solubility of the individual compounds

of the pore water PAHs are not simply controlled by the solubility of the individual compounds. Similar effects were also observed by other investigators.[4,18]

PAH Concentration in the Sediment

Many investigations show that a considerable portion of the PAHs in water bodies may be associated with suspended particles, which eventually sink to the bottom forming a sediment. Therefore, sediments consequently contain PAHs,
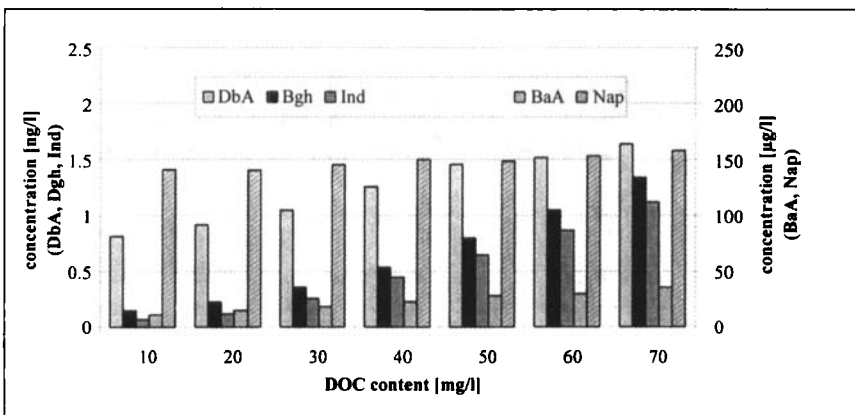

FIGURE 5 "Solubilization effect" of the DOC content in the pore water on individual PAHs
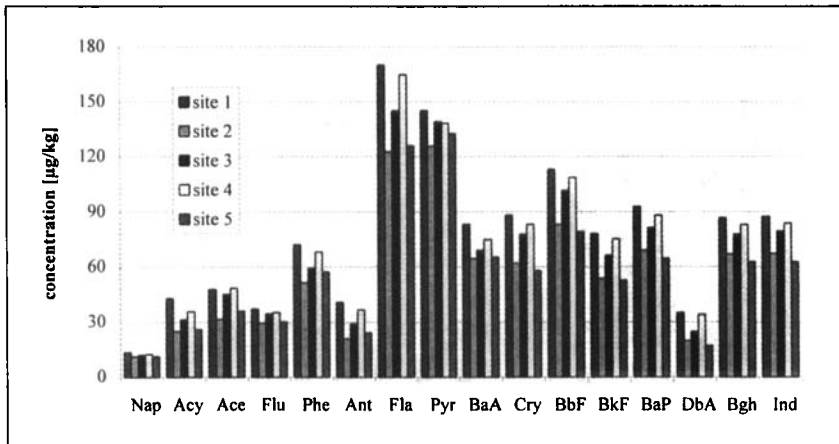
FIGURE 6   PAH concentrations in the solid sediment (0–2 cm, 26 October 1992) at the different sampling sites in the Teufelsweiher pond

usually in concentrations much higher than in the overlying water or in the pore water.[19] This partition or sorption process is influenced by different factors and remains to be established.

Generally, four features of the PAH distribution are apparent. First, the PAH pattern is similar at each site and at each season with Fla, Pyr, BaA, Cry, BbF, BkF, BaP, Bgh and Ind being present at the highest, and with Nap, Ace, Acy, Flu, Ant present at the lowest concentrations (Figures 6 and 7). The total PAH
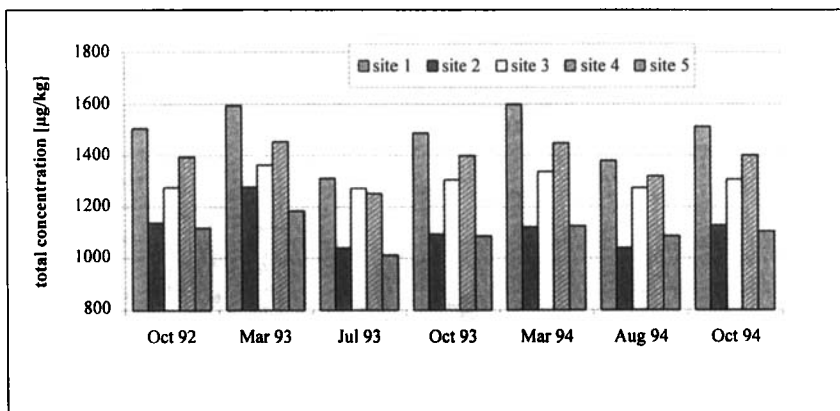

FIGURE 7   Seasonal variation of total PAH concentrations in the solid sediment of the Teufelsweiher pond
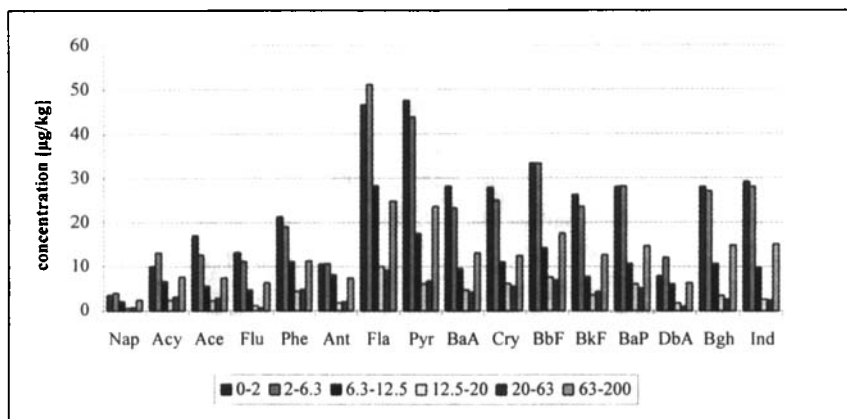
FIGURE 8   PAH distribution in different particle-size fractions [μm] from the solid sediment (site 1, 0–2 cm, 26 October 1992)

concentrations ranged from 976–1600 μg/kg (DW). Second, the distribution depends on the organic matter content and particle-size distribution (Figure 8). Correlation analysis indicates that the total PAH concentration (μg/kg) increases as a function of the organic matter content (%): $[PAH]_{total} = 308.64 + 25.54 \times [C_{org}]$ ($r^2 = 0.82$). In addition, the distribution of PAHs in the different particle-size fractions is bimodal with the smallest (clay and silt, <6.3 μm) and largest (fine sand, 63–200 μm) particle-size fractions containing the highest concentrations. Both fractions contain more organic matter than other fractions (data not shown), but the types of organic matter may be different. Fragmentary plant material is probably responsible for the high levels of organic matter in the large particles whereas aged and condensed organic matter is associated with the smaller particles. These types of organic matter may have different affinities for PAHs due to increasing aromaticity with aging of the organic matter.[20–22] Moreover, clay and silt have a larger specific surface area for adsorbing PAHs than other fractions. Similar results were also obtained from other investigators.[15,16] Third, all PAHs decreased in concentration with depth in the cores (Figure 9). The accumulation of PAHs in the top layer indicates that PAHs are either adsorbed by the sediment particles of the top layer and hardly transported to the layer underneath, or more degradation of PAHs occurs in the lower layers as the activity of microorganisms increases with increasing depth of the sediment.[19] Lastly, only small differences in the PAH concentration in the course of the observation period (1992–1994) suggest that PAH accumulation in the Teufelsweiher is taking place at a slow rate. Martens[17] also reported that soils
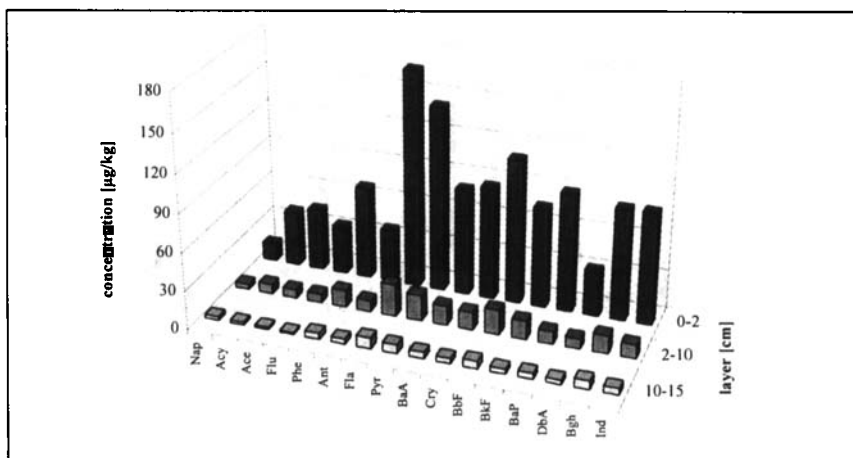
FIGURE 9   PAH concentration in different sediment layers of the solid sediment (site 1, 26 October 1992)

of agricultural land nearby receive less PAHs compared to other non agricultural areas.

Concentrations of PAHs in winter are normally higher than those in summer at each sampling site (Figure 7). This observation may result from different factors: 1. the snow thaw: PAHs deposited with snow in the drainage areas reach the aquatic environment through runoff. Furthermore, PAHs deposited onto the soil during summer should not reach the aquatic environment to the same extent as during winter when the runoff is much more extensive on the frozen ground and the scavenging due to melting snow and washout of PAHs is more efficient.[23] It has been shown that snow may contain considerable amounts of PAHs;[24,25] 2. increased emissions: the increase of PAH content in winter samples is due to a much higher load of the air by combustion products presumably related to the increase in domestic and industrial combustion processes combined with a higher frequency of inverse weather situations;[26,27] 3. decreased degradation: Lee and Ryan[28] have shown that lower rates of microbial degradation and photooxidation of the PAHs during winter will contribute to relatively higher PAH concentrations compared to summer.

In order to determine the sources of the particle-bound PAHs several profiles were calculated. Figure 10 shows a set of normalized PAH profiles for the different sites; all PAH concentrations have been divided by the Fla concentration. The profiles at each site are generally very similar, Fla and Pyr representing the prevalent compounds. As expected, PAH concentrations increase in the solid sediment with increasing log K_{ow}.
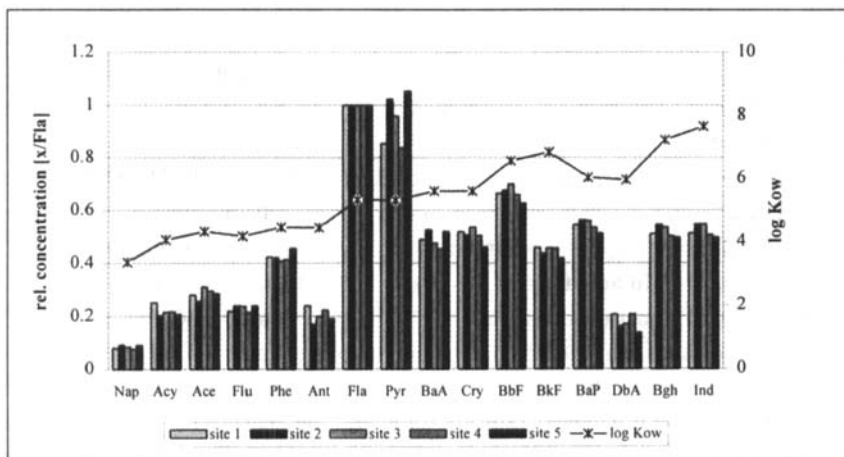
FIGURE 10   PAH profiles (normalized by Fla) in the solid sediment (0–2 cm, 26 October 1992) at the different sampling sites in the Teufelsweiher pond

There are many sources of PAHs in the Teufelsweiher to be considered, but obviously only airborne (deposition) and waterborne (runoff) particulates are important. PAHs formed in combustion and other high-temperature processes are, to a large extent, primarily released into the atmosphere and are assumed to be almost completely associated with particulate matter. Depending on the size of the particles they are deposited on land and water surfaces (dry deposition) or are washed down by rain and snow (wet deposition). This input path is supported by some PAHs (Flu, Phe, Fla, Pyr, Cry etc.), that were directly measured in concentrations of a few ng/m^3 in Scheyern air.[29] In addition, high concentrations of PAHs in the two inlets and the surface water of the Teufelsweiher were also determined during runoff and interflow events.[17] In this study the PAHs exhibit similar profiles both at the different sampling sites and seasons. This fingerprint suggests a common origin of these PAHs from unknown sources. High concentrations of four-, five- and six-ring PAHs are measured in the sediment, Fla and Pyr representing the dominant compounds, that account for 21–28% of the total PAHs. Their ratio of concentration is about 1:1 at all sites, which indicates that the PAHs in this sediment mainly originate from combustion processes.[20,30–32] Samples primarily polluted with combustion products have typical fluoranthene/pyrene (Fla/Pyr) ratios near or below unity and phenanthrene/anthracene (Pyr/Ant) ratios of less than three.[21,32] These ratios are also observed in this study: 1.61–2.86 and 0.86–1.57 for Pyr/Ant and Fla/Pyr, respectively.

The possibility of predicting pore water PAH concentrations in sediment from equilibrium partitioning was tested by comparing estimated K_{OC} with *in situ* measured K_{OC}' values. Estimated K_{OC} values were calculated by Means' equation.[33] The K_{OC}' values were determined by dividing K_D by the organic matter content f_{OC}; the results are given in Table I. Measured values of K_{OC}' are consistently lower than estimated K_{OC}, especially for the larger PAHs. Differences between K_{OC} and K_{OC}' can be due to many factors, but the most important are associated with content, composition, and aromaticity as well as polarity of DOC in pore water.[34,35] In aquatic systems PAHs are distributed mainly among three phases: the real aqueous phase, the DOC in pore water, and the solid sediment.[36] With increasing DOC content in pore water more PAHs will be transferred to and sorbed by DOC with the consequence of increasing PAH concentration in pore water; accordingly the K_{OC} decreases. This observation is not consistent with recent studies which showed that K_{OC}' was overestimated in marine sediments.[4,5] This may be attributed to different DOC, sediment properties, "aged" PAHs (hysteresis) etc. In addition, partition or sorption processes of PAHs between the three phases described above may result in different patterns of PAH distribution in these phases partly due to different DOC properties in pore waters.[9] Finally, the high salinity, and therefore high ionic strength, which is typical for the marine sediment, "salt out" DOC-sorbed PAHs into the sediment causing their K_{OC} to increase. Truly dissolved PAHs are usually considered to be the most toxic and biologically available fraction of sediment PAHs. Also, the transport of HOC facilitated by DOM or colloids is an important factor evaluating potential environmental impacts on aquatic systems.[7]

CONCLUSIONS

The 16 EPA priority PAH compounds are found both in pore water and solid sediment of the Teufelsweiher pond in a rural region of Upper Bavaria during 1992–1994. The concentrations of PAHs in the sediment of the Teufelsweiher are low compared to other river sediments in Germany. This means that PAHs deposit in generally low amounts in this aquatic ecosystem or undergo several processes, such as biological and photochemical degradation, volatilization and leaching, which influence their concentrations in the sediment. The distribution of PAHs is especially linked to the particular grain size of the sediment and its organic matter content. Also the DOC exhibits a "solubilization effect" that can increase total PAH concentrations in the pore water. Characterization of the profile of the PAHs, which is quite uniform with respect to the different sampling

sites shows that they are mainly incomplete combustion products of fossil fuel and organic material in this area bound to air particles and transported by deposition and runoff into the aquatic environment.

Acknowledgements

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