Chapter 2
Mining Related PCB in Wetland Sediments of the River Lippe (North Rhine-Westphalia, Germany)

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Abstract This study aims to detect PCB (polychlorinated biphenyls) and their distribution in river sediments of the river Lippe near a discharge point of mine drainage water raised from the coalmine Haus Aden. Recommendations from former literature studies, European and German-Industry-Standards (DIN) standards were followed to conduct a reproducible field study. The sampling, processing, and analysis of the sediment and core samples were delineated in detail explored in order to open up a new discussion about the methodical execution of environmental examinations of PCB. The results show that the normally executed standardization processes to interpret PCB concentrations do not work as assumed. This refers especially to the normalization of the content of organic carbon in the sediment. The heterogeneity of observed concentrations dominates and the concentration fluctuates enormously instead of following an often-described correlation between PCB and organic carbon or the percentage of fine grain. Congener profiles, which show the amounts of the six indicator congeners, do not show an obvious trend or indicate one corresponding contamination source. The findings invoke a new debate and examinations concerning deposition and adsorption processes of PCB in sediments, especially against the background of flooding the derelict mines of the Ruhr district in the coming decades.

Keywords Polychlorinated biphenyls (PCB) · Uplifted mine water · Congeners · PCB distribution in river sediments · Standardization to organic carbon/ fine grain · Environmental quality standard

2.1 Introduction

Polychlorinated biphenyls (PCB) are still threatening human beings and the environment due to their high persistence in the environment and toxicity even though

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their production and application were prohibited in Germany in the 1980s. Physical and chemical properties of PCB like heat resistance, chemical stability, good thermal and low electric conductivity (Lehnik-Habrink et al. 2006; IARC 2016; Hegnal 2010; Borja et al. 2005) led to around 1 to 1.5 million tons produced worldwide (Lehnik-Habrink et al. 2006; IARC 2016; Hegnal 2010; Liang et al. 2014; Demirtepe et al. 2015; Tehrani and Aken 2014) and intensive application. Large amounts of PCB were used in plasticizers of sealing joints, synthetic materials, colors, and varnishes, and as an insulating liquid in capacitors and current transformers (Lehnik-Habrink et al. 2006; Hegnal 2010; Borja et al. 2005; Erickson and Kaley 2011; Umweltbundesamt (2018).

PCB was used in the mining industry as a hydraulic fluid from 1960 until its prohibition (Schwarzbauer 2017; Engel and Kihl 1987), for example in coal mining in the Ruhr Area. When mining operations ceased in 2018 the question regarding the fate and behavior of PCBs in the environment remained. Studies were conducted to evaluate the remaining risk (e.g., LANUV, state office for nature, environment, and consumer protection) to surface water and aquatic life. A special issue of the present is the mine drainage water levels in the Ruhr district, which will rise in the coming decades and the following spread of its biphenyl content. This process evokes fear regarding groundwater quality.

Not only the mining industry employs PCB for technical purposes. Other industrial sectors such as mechanical engineering and the chemical industry benefit from the properties of PCB as well. One differentiation, in general, is the handling of the biphenyls. In practice, they ran either in open or closed systems, which refers to the possibility of the substances to get in contact with the environmental media (Hegnal 2010). There are more than 200 congeners differing in mass and chlorination, whose percentual distribution in the mixture gives an indication of their original industrial application (e.g., congeners 28 and 52 often appear in combination with coal mining).

The study presented here uses earlier studies as reference information and offers a deeper view into the distribution of PCB in river sediments nearby a discharge point of the uplifted water of a mining plant called Haus Aden near Dortmund. The results of the study originate from a cooperation between the Department of Engineering Geology and Hydrogeology of RWTH Aachen University and the Institute of Occupational Medicine of RWTH Aachen University.

The study focuses on the strategy of sampling and the detailed description of the processing and analyzing the samples in order to make the results replicable. Particularly the calculative standardization of PCB concentrations to the adsorptive parts of the sample will be discussed.

2.2 Methods

2.2.1 Analytical Reference System

Sediments consist of heterogeneous matrices and PCB only adsorb at adsorptive materials like organic matter (IARC 2016; Rommel et al. 1998; Hölting and Coldewey 2005; Prinz and Strauÿ 2006). The pH-value (Rommel et al. 1998) and redox conditions also influence adsorption (Döring and Marschner 1998). Without standardization, the results of PCB measurements are comparable neither within the same study area nor with the results of other studies. The standardization has to refer to a reference system, which is the same for all samples, namely the adsorptive part of the samples. Without standardization, it is unsure, if the results are realistic. A sample with a low content of adsorptive material will have a low value for PCB. However, it is uncertain whether the value is low, because there is no more PCB or because the ion exchange capacity is low due to low content of adsorptive material in the sample. With carrying out the standardization procedure, the composition of the samples does not matter.

For this study, the parameters fine grain and clay minerals were used synonymously. This simplification is based on the assumption that the fine grain fraction (<0.063 mm) consists mostly of clay minerals and the non-adsorptive part is negligible (Schwarzbauer 2017). Busch et al. (Busch et al. 2007) found a significant correlation between the content of PCB and the fraction of 40–63 μm.

2.2.2 Sampling

The samples were taken around about 20 km northeast of the German city Dortmund during wintertime. The starting point was the discharge point of mine drainage water from coalmine Haus Aden (Fig. 2.1).

Eleven saturated sediment samples were taken out of the riverbank. Additionally, five core samples were taken out of the embankment at 2 m distance from the waterline. The sediment sample named S1 and the core sample named BK1 were taken 30 m upstream of the discharge point. These two samples are taken with a 1 m long Pürkhauer drill stick for reference to estimate the background contamination with PCB in this area. The following samples were taken at the slip-off slopes and erosion banks of the meandering river Lippe.

The samples were filled into 250 ml wide mouth bottles made of brown glass. To prevent any contact between sample material and the plastic of the lid, which may lead to adsorption of PCB to the plastic, aluminum foil was put over the mouth. After that, the samples were stored at 5 °C in the dark until further analysis. The content of PCB was measured at the laboratory of the Institute of Occupational Medicine at the university hospital of Aachen. The contents of total organic carbon and fine grain were measured at the laboratory of the Department of Engineering Geology and Hydrogeology of the RWTH Aachen University.

Fig. 2.1 Sample area at the river Lippe near the city Lünen. Left: overview map, topographical map TK50. Right: Sample points at the river Lippe near the discharge point of mine drainage water from coalmine (German: Zeche) Haus Aden. Saturated sediment samples, BK = core samples (scale: 1 cm corresponds to about 80 m, map is north orientated)

2.2.3 Content of Organic Carbon

The total organic carbon content of the samples was measured by estimating the ignition loss. The procedure followed the standard (DIN 18128 2002) (German Industry Norm, Soil—Investigation, and Testing—Determination of Ignition Loss). The samples dried in cups of porcelain in a heating cabinet at 105 °C until constant mass. Subsequently, the samples were put into a desiccator to cool down. The next step was homogenization with a mortar. Some samples contained organic material, that had not been decomposed yet. After homogenizing the samples were stored in a heating cabinet at 60 °C for 24 h and were afterward put into the desiccator again. The muffle furnace was heated up to 550 °C and the empty cups were calcined for 20 min. For fine grained and organic samples DIN 18128 requires a minimum weight of 15 g. The cups with the samples were ignited for 2 h until their mass stayed constant. The cups with the samples cooled down in the desiccator and were weighed again. The difference between the weights was the content of organic carbon.

2.2.4 Content of Fine Grain

The sieving followed the standard (DIN 18123 2011) (German Industry Norm: Soil, Investigation, and Testing—Determination of Grain-size Distribution). The samples dried in a heating cabinet at 105 °C until constant mass. After that, they were weighed to 0.01 g, soaked with water, and stirred with the stirring device RW 20 RZM from the company IKA. A vibration-sieving machine (type AS 200 control, company

Retsch) executed the sieving. Mesh sizes ranged between 4 mm and 0.063 mm. Before weighing, the fractions dried in a heating cabinet at 105 °C and cooled down in a desiccator.

2.2.5 Content of PCB

The preparation of the samples for measuring the content of PCB followed the (DIN EN 15308 2016) (Euro Norm, Characterization of Waste—Determination of Selected Polychlorinated Biphenyls (PCB) in Solid Waste by Gas Chromatography With Electron Capture or Mass Spectrometric Detection).

Some congeners are volatile, so the samples were freeze dried with the tool Alpha 1–4 LOC-1 m from the company Christ. The first attempt to dry the samples within the glasses did not work, because this process proved excessively time-consuming. So bowls of aluminum were made to spread the material over a bigger area and reduce drying time.

The samples had to be homogenized after drying. The DIN 15308 allows this step with a ball mill (company Retsch, type PM 100 CM). 50 g of the sample was homogenized at 325 rpm (rounds per minute) for 15 min. The congeners were extracted with n-pentane in an ultrasonic device and measured with a gas-phase chromatograph (company Agilent Technologies, type 7890 A), which was linked to a time-of-flight mass spectrometer (company Leco, type Pegasus 4D GCcGX-TOFMS). The injection was executed by an autosampler from the company Gerstel Twister, type Multi Purpose Sampler MPS. The capillary column (company Restek, type RTX 5) had a length of 60 m and a diameter of 0.25 mm. Helium was used as carrier gas. PCB-54 (2,2′,6,6′-tetrachlorobiphenyl) was used as recovery standard. The oven program had the following steps: the initial temperature of 45 °C was held isothermally for 1 min. At 30 °C/min the oven heated until 190 °C, which was held for 0.01 min. Until 330 °C the oven was heated with 5 °C/min and this final temperature was held isothermally for 12.16 min.

2.3 Results

2.3.1 Content or Organic Carbon and Fine Grain

The results of the ignition loss and contents of _ne grain for the sediment and core samples are shown in Table 2.1. The results of the ignition loss for the sediment samples (S-samples) are very heterogeneous and are fluctuating between 1.26% (S11) and 16.06% (S3). The ignition losses for the core samples are more homogenous and spread between 3% (BK1) and 5.13% (BK5). Like for the content of organic carbon the results for the content of fine grain in the sediment samples show a wide range

Table 2.1 Result for the ignition loss and content of fine grain for the sediment samples (a) and the core samples (b) %

(a)

Sample	Content of organic carbon (%)	Content of fine grain (%)
S1	3.26	19.33
S2	5.19	23.01
S3	16.06	19.62
S4	9.93	–
S5	11.80	74.86
S6	4.33	12.55
S7	13.32	71.35
S8	13.66	59.37
S9	7.92	31.00
S10	3.46	26.42
S11	1.26	6.01

(b)

Sample	Content of organic carbon (%)	Content of fine grain (%)
BK1	3.00	18.05
BK2	4.00	22.80
BK3	3.80	19.91
BK4	3.86	21.67
BK5	5.13	16.36

between 6.01% (S11) and 74.86% (S5). There was not enough material for sample S4 for sieving. And the percentage of the fine grain for the core samples ranges between 16.36% (BK5) and 22.80% (BK2).

2.3.2 *Content of PCB*

Table 2.2 contains the concentrations of the six indicator congeners in µg/kgDS (DS = dry substance) in the sediment samples.

Table 2.3 shows the concentrations of PCB in the whole samples and the concentrations, which are standardized to organic carbon and _ne grain. The results of all samples exceed the environmental quality standard of 20 µg/kg DS. The lowest levels are shown by samples S1 and S4 with 50 µg/kgDS and the highest level appears in S5 with 8610 µg/kgDS. Altogether four samples have concentrations above 2000 µg/kgDS.

After standardization to the content of organic carbon and fine grain, the PCB values are much higher. For the fine grain, the values range between 193 µg/kgDS

Table 2.2 Concentrations of the six indicator congeners of the sediment samples (µg/kgDS)

Sample Congener	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11
25	17.0	163.9	47.4	19.5	1887.3	327.1	501.3	366.4	9.7	25.1	12.5
52	5.6	33.4	15.3	8.0	1161.3	379.5	320.3	293.3	4.9	7.7	7.6
101	7.7	27.5	18.8	3.3	2088.1	983.4	890.0	561.0	9.4	10.2	7.1
138	5.5	20.4	30.2	6.5	1930.8	935.7	800.8	820.9	16.5	15.0	12.0
153	6.6	14.5	38.5	12.0	1290.1	760.0	770.6	490.7	17.6	16.1	15.9
180	2.1	9.0	17.9	5.1	257.0	295.4	460.1	49.5	6.5	6.5	9.5

Table 2.3 Concentrations of PCB in the sediment samples (S) (µg/kgDS)

Sample	Concentrations PCB (µg/kgDS)	Concentrations PCB, standardized to organic carbon, rounded (µg/kgDS)	Concentrations PCB, standardized to ne grain, rounded (µg/kgDS)
S1	50	1533	258
S2	270	5202	1173
S3	170	1058	866
S4	50	503	–
S5	8610	72,966	11,501
S6	3680	84,988	29,322
S7	3740	28,078	5241
S8	2580	18,887	4345
S9	60	757	193
S10	80	2312	302
S11	60	4761	998

Left: Concentration in the whole sample. Middle: Concentrations standardized to organic carbon (µg/kgDS). Right: Concentrations standardized to ne grain (µg/kgDS)

(S9) and 29,322 µg/kgDS (S6). The lowest value for the concentration standardized to organic carbon is found in S4 with 503 µg/kgDS and the highest value for S6 with 84,988 µg/kgDS.

130 m downstream of the discharge point of mine drainage water the concentration sharply peaks at 8610 µg/kgDS (S5), while S8 [490 m downstream of the discharge point (following the waterline)] marks the abrupt end of the peak with 2580 µg/kgDS (Fig. 2.2).

The values for the indicator congeners in the core samples are shown in Table 2.4.

In Table 2.5 you find the total concentrations for the core sample. They are lower than those in the sediment samples. Without standardization to the adsorptive part of the samples, only BK3 and BK4 exceed the environmental quality standard

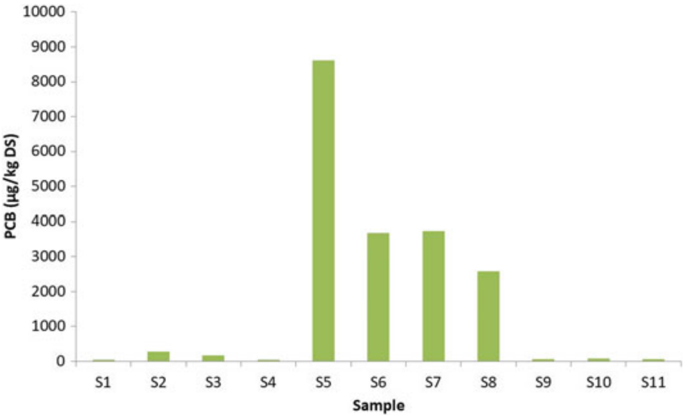

Fig. 2.2 PCB concentration of the sediment samples (S) (µg/kgDS)

Table 2.4 Concentrations of the six indicator congeners in the core samples (µg/kgDS)

Samples congener	BK1	BK2	BK3	BK4	BK5
25	2.1	2.1	<0.1	7.7	<0.1
52	2	2.2	2.9	3.2	0.7
101	2	2.4	4.2	6.6	1.5
138	2.2	1.5	3.6	10.4	3
153	1.9	0.2	6.8	5.6	5.6
180	1.8	<0.1	<0.1	1.8	2

Table 2.5 Concentrations of PCB in the core samples (BK) (µg/kgDS)

Sample	Concentrations PCB (µg/kgDS)	Concentrations PCB, standardized to organic carbon, rounded (µg/kgDS)	Concentrations PCB, standardized to ne grain, rounded (µg/kgDS)
BK1	10	333	55
BK2	10	250	43
BK3	20	526	100
BK4	40	1034	184
BK5	10	194	61

Left: concentration in the whole sample. Right: concentrations scaled to the adsorptive part of the sample

(20 µg/kgDS BK3, 40 µg/kgDS BK4). However, with executing the standardization all BK samples exceed the limit value. The lowest level for the standardization of organic carbon is 194 µg/kgDS for BK5 and the highest value accounts

1036 μg/kgDS for BK4. After normalization to fine grain, the values range between 43 μg/kgDS (BK2) and 184 μg/kgDS (BK4).

2.4 Interpretation

The results show that all samples are contaminated with polychlorinated biphenyls, even 30 years after banning the production and application of PCB in Germany. Even the lowest level of PCB in the sediment samples (S1 and S4, 50 μg/kgDS) is higher than twice the environmental quality standard, set by EU legislation. Notable levels of PCB are also detectable in the core samples, which do not have direct contact with the river water containing mine drainage water. Contact between the river and the embankment, where the cores were taken, is imaginable only several times a year during floods.

The standardization of the content of organic carbon and fine grain rises the values to an even higher level (Tables 2.3 and 2.5).

A notable correlation between PCB concentrations and organic carbon and fine grain respectively was assumed, but could not be proved. The values for organic carbon and PCB as well as _ne grain and PCB are plotted against each other in Fig. 2.3a, b. The values for organic carbon content, fine grain content, and PCB show high heterogeneity within short distances and without any statistical trend. The distance between sample S5 and S6 amounts to only a few meters, but the content of organic matter in S5 represents 11.85% and in S6 only 4.33%. The values for the content of fine grain range between 74.86% (S5) and 12.55% (S6). The influence of sedimentation and degradation processes of organic matter on PCB adsorption is not yet well understood to explain the deposition and heterogeneity of PCB in river sediments.

Contents of organic carbon and _ne grain do not correlate to PCB concentration in the assumed way (Fig. 2.4) and so the standardized values for PCB show great differences. The data underlines the importance and difficulty to choose an appropriate reference system. A comparison of different studies about PCB in sediments by working groups and the scientific community in this field requires a standardized reference system.

The sampling area was selected downstream of a mine drainage water discharge point. One idea was to look at the congener profiles, which compare the concentrations of the six indicator congeners, to find out, if it is possible to name the source of the PCB contamination. Congeners 28 and 52 are considered typical for contamination from mining sources. The congener profiles for the sediment samples are shown in Fig. 2.5a, b.

Especially in S2, the value for PCB-28 is significantly raised compared to other congeners. S3 has high values for PCB-28, but also similar concentrations for PCB-153. PCB-138 and PCB-153 are typical for Clophen A60, which was used in open systems in Germany (Brusske et al. 2007). The usage of PCB in open systems was banned in Germany in 1978 (Hegnal 2010).

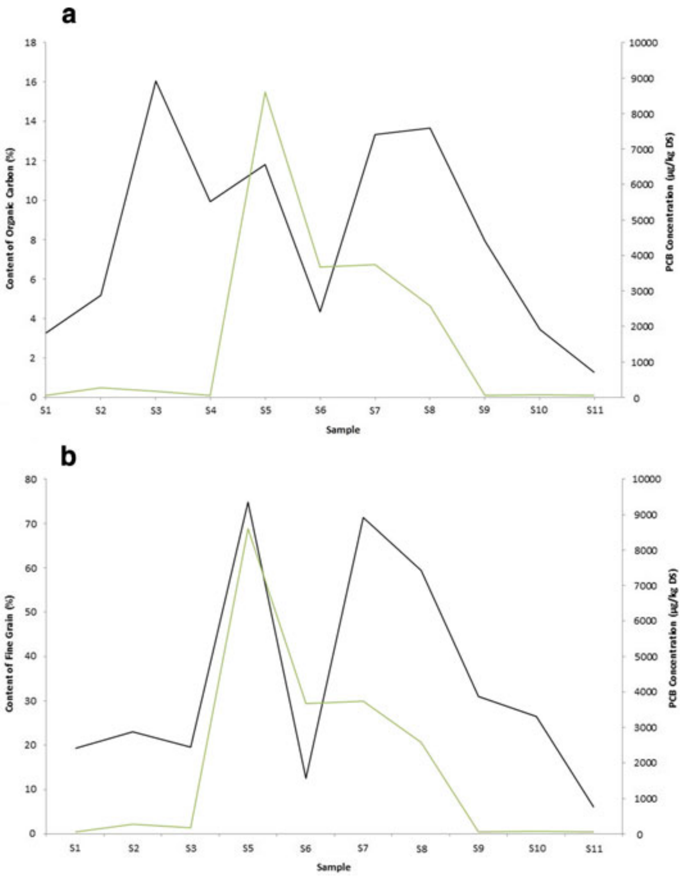

Fig. 2.3 Missing/weak correlation between PCB and content of organic carbon and content of fine grain. **a** PCB (µg/kgDS, light line) and content of organic carbon (%, black line). **b** PCB (µg/kgDS, light line) and content of fine grain (%, black line)

There is no clear trend for the congener profile of the samples S5–S8. S5 has high values for PCB-28, but also for PCB-101 and PCB-138. For the other samples, the focus is on PCB-101 and PCB-138, both of which are not considered typical for mining sources (Rahm et al. 2015). Therefore, it was not possible to relate the contamination only to mining sources.

The heterogeneity and changes on short distances are also shown in Fig. 2.6a, b. S4 and S5 are around 30 m away from each other and the congener profile is significantly different. This shows again the complexity of sedimentation of particles, on which PCB molecules are adsorbed.

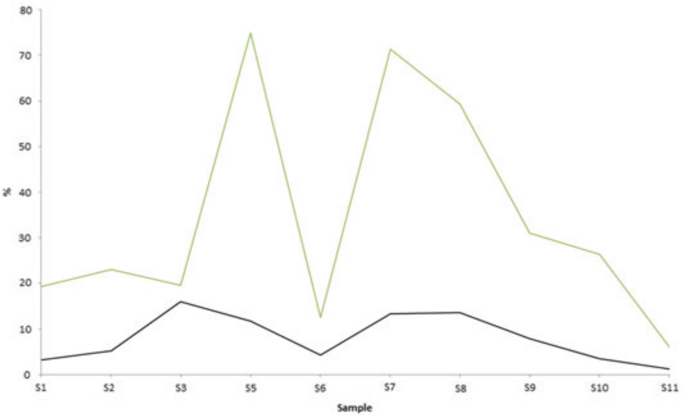

Fig. 2.4 Correlation between content of organic carbon (black line) and content of fine grain (light line) in the sediment samples (%)

2.5 Conclusion

As the main outcome of this study, the statement is issued that the embankment sediments of the Lippe show significant concentrations of PCB downstream of the discharge point of mine drainage water. All values for the sediment samples exceed the environmental quality standard. Assumptions made in the literature about a strong correlation between PCB concentrations and the percentage of fine grain (<0.063 mm) or organic carbon in the sediments could not be confirmed, so that an open question persists in the reference system. PCB deposition and processes of adsorption at river sediments should be focused on in further examinations. Neglecting factors such as deposition conditions or the aforementioned processes will put future studies at risk of misunderstanding or misinterpreting the measured PCB raw data. Another important result is the notable detection of PCB concentrations in slope sediments, which have no direct contact with the river water. This implies that these sediments show a kind of memory effect, even though direct contact exists only a few times a year during flooding. The results also show that PCB degradation after more than 30 years after the end of PCB application did not lead to extinction in the environment. Additionally, congener profiles in this study do not show an explicit source of contamination. The existence of mining-related congeners is in the same way proven as the existence of congeners which normally were used in open systems (banned 40 years ago). The difficulty of evaluating and interpreting PCB data due to their heterogeneity is clearly shown in this study, even though a relatively small study area with an intensive sampling procedure was chosen. The highly specific description of methodical processes and the usage of analytical apparatus shall increase transferability and reproducibility, which is essential for opening a scholarly debate. Responsibility in conducting such case studies to find objective observations is extremely relevant regarding the set-up of the examination. This is

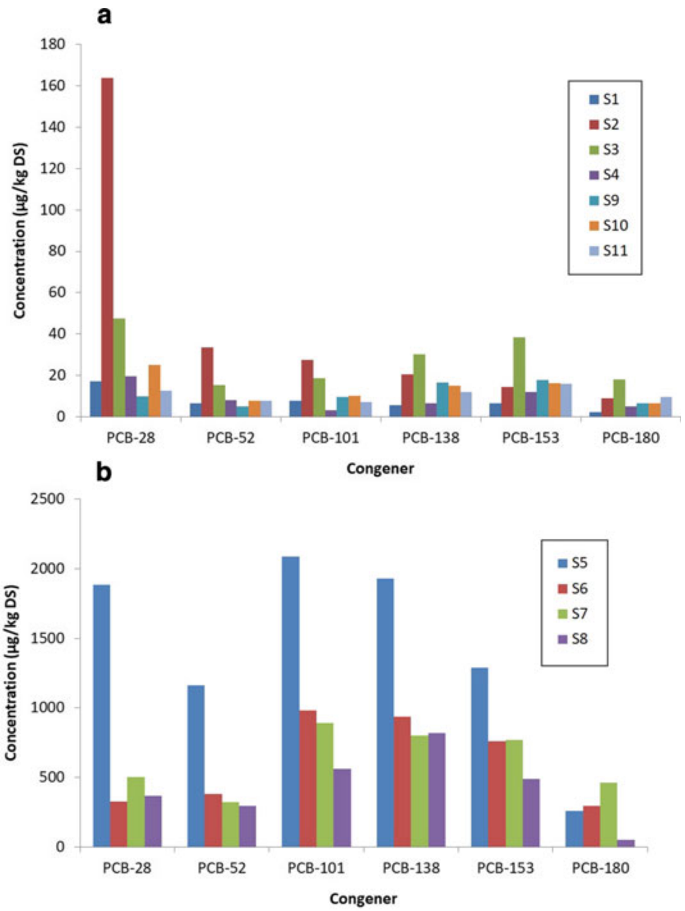

Fig. 2.5 Congener profiles for the sediment samples (µg/kgDS). **a** S1–S4 and S9–S11. **b** S5–S8

particularly important in view of other areas with suspected high PCB contamination. In many Indian cities, there are areas with high levels of PCB contamination (Goswami 2017). For example, in the megacity of Chennai, very high PCB concentrations were found in an area where electronic waste was informally stored and recycled (Goswami 2017).

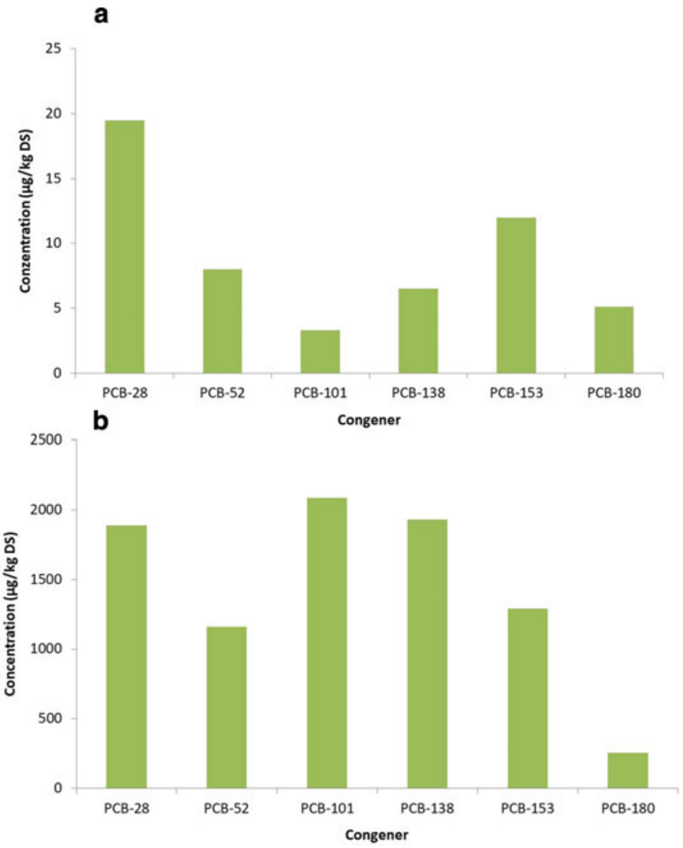

Fig. 2.6 **a**, **b** Congener profiles for S4 (**a**) and S5 (**b**) (µg/kgDS)

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