

Environmental assessment of PBDEs contamination in the Svitava River, Czech Republic

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Abstract

OBJECTIVES: The aim of this study was to assess the contamination level of aquatic ecosystems of the Svitava River, situated in the South Moravian Region (Czech Republic), by residues of selected brominated flame retardants. We isolated and determined seven indicator PBDE congeners in samples of surface water and bottom sediments using optimized analytical methods.

DESIGN: Samples were collected from eight locations along the river basin, particularly near the larger cities in order to assess their possible impact on the aquatic ecosystems. Isolation of selected analytes was performed using the methods of liquid–liquid extraction and cold extraction. Column chromatography and rotary vacuum evaporation were used for the purification and pre-concentration of extracts. Final identification and quantification were carried out by gas chromatography coupled with electron capture detector (GC/ECD).

RESULTS: The sum of PBDEs was calculated for individual BDE congeners. Average concentrations of the sum of PBDEs ranged from 56.35 to 614.0 $\mu\text{g}\cdot\text{kg}^{-1}$ of the dry matter in sediment, and concentrations under the detection limits (<LOD) up to 335.3 $\text{ng}\cdot\text{L}^{-1}$ in water. The most loaded locations were Adamov, Brno-Tuřany and the location near the former Essler textile factory.

CONCLUSIONS: Monitoring of PBDEs in the environment, especially in the aquatic ecosystem, is a topical issue due to their growing worldwide application as flame retardants in many products of daily use. Results of this study documented not only potential sources of contamination in the most polluted locations, but also confirmed the lasting presence and accumulation of PBDEs in the environment.

Abbreviations:

| | |
|----------|---|
| BDE | - brominated diphenyl ether |
| BFRs | - brominated flame retardants |
| DDT | - dichlorodiphenyltrichloroethane |
| GC/ECD | - gas chromatography with electron capture detector |
| GPS | - global positioning system |
| K_{ow} | - n-octanol water partition coefficient |
| LOD | - limit of detection |
| LOQ | - limit of quantification |
| ND | - not detected |
| p.a. | - for analysis |
| PBDEs | - polybrominated diphenyl ethers |
| PCBs | - polychlorinated biphenyls |
| POPs | - persistent organic pollutants |
| R^2 | - coefficient of determination expressing the reliability |
| SD | - standard deviation |
| WWTP | - wastewater treatment plant |

INTRODUCTION

Flame retardants, which are often added to various polymer matrices, constitute one of the possible preventions of fire. Brominated flame retardants (BFRs) are together with other organohalogen compounds among the significant persistent organic pollutants (POPs). These substances are generally characterized by considerable resistance to degradation, high lipophilicity and bioaccumulation in the fatty tissue of animals and humans (Alaee 2003; Hlouskova *et al.* 2014).

Polybrominated diphenyl ethers (PBDEs) belong to the most often applied BFRs and are often used as additives in plastics, electronics, packaging materials, textile, car industry, furniture, etc. In most cases they are applied into commercial mixtures of Penta-BDE, Octa-BDE and Deca-BDE (Madia *et al.* 2004; Dirtu 2009). PBDEs could release from the surface of the product and thus get into the environment (Yan *et al.* 2011). In the last decade, the presence of BFRs has been confirmed not only in abiotic matrices but also in terrestrial and aquatic ecosystems (Madia *et al.* 2004; Daubie *et al.* 2011; Zhang *et al.* 2011). PBDEs were proven also in humans (Costa & Giordano 2007).

As with other halogen POPs, chronic toxic effects of this group are also considered (Zhang *et al.* 2011; Gu *et al.* 2012; Hlouskova *et al.* 2014). Their liposolubility results in an affinity to adipose tissues and bioaccumulation in living organisms (Luthe *et al.* 2008; Vavrova *et al.* 2008; Segev *et al.* 2009). Based on these properties, the European Union banned the production and usage of technical compounds of Penta-BDE and Octa-BDE in 2004 (Dirtu 2009; Qin *et al.* 2011).

PBDEs can get into the environment together with waste water, which results in the adsorption to river sediments and secondary contamination of the water environment. They can also adhere to dust particles and thus be transported for long distances from the source. Improper waste disposal containing these pollutants can also make a considerable source of contamination (Segev *et al.* 2009).

Based on this knowledge, it is possible to state that PBDEs constitute a worldwide problem. That is why it is necessary to regulate not only their production, but also treatment of these substances and the recycling of products containing these contaminants. These hazardous pollutants are able to persist and accumulate for a long time in the environment, and therefore their continuous monitoring is of high priority. The aim of our work was to assess the contamination level of aquatic ecosystems of the Svitava River, situated in the South Moravian Region (Czech Republic), by residues of seven indicator PBDE congeners, investigating the samples of surface water and bottom sediment from eight locations selected near the possible sources of contamination.

MATERIAL AND METHODS

Chemicals and materials

All organic solvents used for the analysis (n-hexane, acetone, diethyl ether and isooctane) were of the purity for the residual analysis and were purchased from Merck (Darmstadt, Germany). Anhydrous sodium sulfate p.a. was also purchased from Merck (Darmstadt, Germany) and activated for 4 hours at 350 °C. Florisil (0.15–0.25 mm) was supplied by Sigma-Aldrich (Steinheim, Germany) and activated for 4 hours at 500 °C. Both of the high-purity helium (99.999%) and nitrogen (99.999%) were purchased from Messer Technogas (Prague, Czech Republic). Analytical standard of the mix of 7 PBDE indicator congeners (BDE 28, 47, 100, 99, 154, 153, 183) at a concentration of 2.5 µg.mL⁻¹ was supplied by AccuStandard (New Haven, CT, USA).

Sampling

The analysis of PBDEs was carried out in samples of surface water and bottom sediments from selected locations of the Svitava River in Brno City and its surroundings (8 sampling sites). These sites were selected and monitored because of their location upstream and downstream to some significant possible source of contamination. Water samples were collected into clean 2.5 L amber glass bottles; sediment samples (approximately 1 kg) were collected using thin polyethylene bags. They were always pooled samples of three individual samples from one sampling site. Sampling was carried out during the winter and spring 2013, altogether three times (n=3). All samples were processed immediately after their transport to the laboratory or kept refrigerated until the next day. The map of monitored area with exact marking of sampling sites including the GPS locations is shown in Figure 1.

Pre-analytical procedures

Water samples were processed using the method of liquid-liquid extraction. The organic solvent (20 mL of hexane) was added to a water sample (volume of 1000 mL), and together extracted by means of cold extraction (laboratory shaker – OS 10 basic; Maneko,

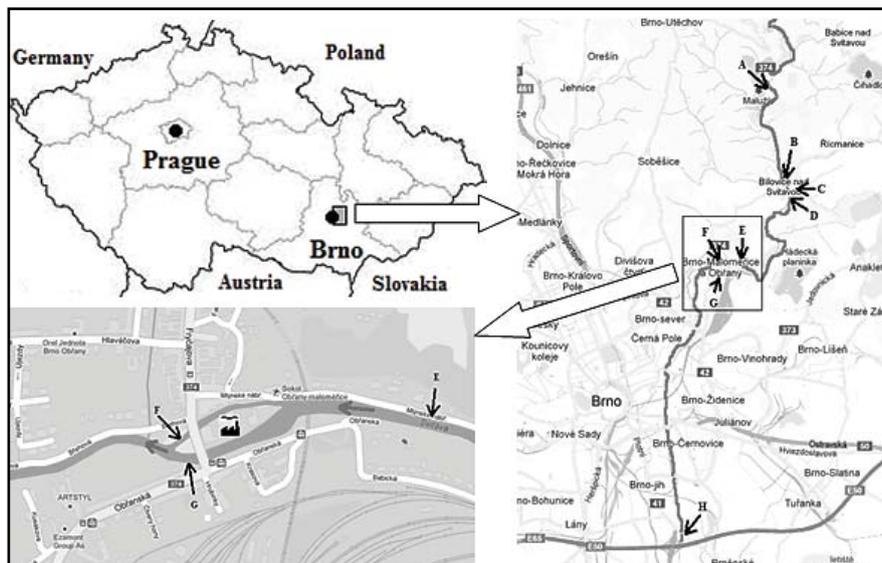


Fig. 1. The map of monitored location with sampling sites at the Svitava River
 A - 49°16'16.063"N, 16°40'3.322"E;
 B - 49°15'7.645"N, 16°40'14.300"E;
 C - 49°14'52.395"N, 16°40'22.800"E;
 D - 49°14'31.727"N, 16°40'25.750"E;
 E - 49°13'40.373"N, 16°39'21.430"E;
 F - 49°13'39.950"N, 16°38'53.572"E;
 G - 49°13'38.853"N, 16°38'55.650"E;
 H - 49°9'44.282"N, 16°38'3.813"E

Prague, Czech Republic). After 30 minutes of shaking, the sample was transferred into the separatory funnel where both liquid phases were separated. Hexane phase was filtered through activated sodium sulphate anhydrous. Such extraction was carried out in the same way three times altogether.

For sediment samples, the percentage of dry matter was determined first. The remaining matrix was dried out at a temperature of 105 °C into the constant weight and homogenized. A total of 30 g of each sample were transferred into a 250 mL flask and 50 mL of hexane was added. The sample was extracted by means of cold extraction on the laboratory shaker 3×30 minutes and hexane was always filtered through activated sodium sulphate anhydrous.

Extracts from water and sediment samples were evaporated using the rotary vacuum evaporator (Büchi, Flawil, Switzerland) to a volume of approximately 5 mL and purified using the method of column chromatography. For elimination of ballast substances the sorbent of Florisil was used, and to the bottom and top of the glass column a 1 cm layer of sodium sulphate anhydrous was added. Concentrated extracts were quantitatively transferred into the column which was then gradually rinsed by 90 mL of eluent consisting of a mixture of hexane and diethyl ether (94:6). Such purified samples were evaporated to dryness, re-dissolved in 1 mL of iso-octane and transferred through nylon filters (0.45 µm; Labicom, Olomouc, Czech Republic) into vials. Each sample was performed in two parallel determinations.

Analytical determination

The method of gas chromatography coupled with electron capture detector (GC/µECD, HP 6890 Series II device; Agilent Technologies, Santa Clara, CA, USA) was used to assess the concentration of individual indicator PBDE congeners. For the chromatographic separation of analytes DB-17 column (J&W, Folsom, CA,

USA) with the parameters of 30 m×250 µm×0.25 µm was used. As the carrier gas, highly purified helium with a flow rate of 1.5 mL.min⁻¹ was used, and nitrogen with a flow rate of 60 mL.min⁻¹ was used as the make-up gas for the detector. The injection volume was 1 µL in splitless mode. Injection temperature was set to 270 °C and detector temperature was 250 °C. Oven temperature mode was as follows: 100 °C held for 2 min; then increased to 200 °C at 30 °C/min (held for 3 min); to 230 °C at 3 °C/min (10 min); to 270 °C at 5 °C/min (5 min) and then increased to 300 °C at 10 °C/min (held for 15 min). All data were processed by means of the ChemStation software, version II (Agilent Technologies, Santa Clara, CA, USA).

Methodology optimization

For each of the observed BDE congener the calibration line from standard stock solutions ranging in concentrations of 1–250 ng.mL⁻¹ was constructed. The coefficient of determination expressing the reliability (R²) was higher than 0.999 in all calibration lines. All results were processed in mean values with standard deviation (SD). Found values of PBDEs are at the confidence level α=0.95. Furthermore, for individual PBDE congeners the values of limit of detection (LOD) and limit of quantification (LOQ) were calculated (see Table 1) by Eq. (1) and (2), respectively. The height of the noise presents h_n , the slope of the pertinent calibration line presents m .

$$\text{LOD} = [(3 \times h_n)/m] \quad (1)$$

$$\text{LOQ} = [(10 \times h_n)/m] \quad (2)$$

RESULTS AND DISCUSSION

All results from this study have been processed into the tables and graphs while in Tables 2 and 3 all concentrations of indicator PBDE congeners in ng.L⁻¹ of water

and in $\mu\text{g.kg}^{-1}$ of the sediment dry matter from the Svitava River are presented. Furthermore, there were calculated the sums of individual PBDE congeners in each particular sample which are demonstrated and compared for individual sampling sites in Figures 2 and 3. The proportional representation of individual PBDE congeners in sediment is shown in Figure 4.

If we assess according to the sum of all PBDE congeners in individual water samples, it is possible to state that sampling site G (335.3 ng.L^{-1}) belongs among the most contaminated locations. It is situated in the direction of water flow of the Svitava River, in a place where the river flows around the former Essler textile factory. On the other hand, the sampling site E situated upstream the factory was PBDE free, i.e. there were documented mostly undetectable concentrations. The factory stands on an island which from the right an artificial channel of the Svitava River flows around. The buildings of the factory were built at the beginning of the previous century. A part of the factory consists of a tank tower and ground-floor halls. Textile production was in operation until 1992 but at this time are most parts of the complex dilapidated (Turistika 2012). The source of the contamination of the location G might be this factory. The textile factory could have been polluting the Svitava River by releasing PBDEs from plastic products, such as the covers of various products, rigid insulation and polyurethane foam providing, for example, the insulation of the tank tower, additionally the insulation of electric conductors, glues and coatings used for the textiles that were produced in the factory. Volatile PBDEs could have been released from these products to which they were not chemically bound. Thus PBDEs bound to dust particles could have, by means of dry or wet deposition, got into the water near the factory. Also, waste water could have been drained into the river; PBDEs could have been adsorbed to organic impurities from which they could be released back into the water (Holoubek *et*

al. 1996). Higher pollution by PBDEs close to the textile factory was also proven in research made by De Boer *et al.* (2003) in the Netherlands. Secondary contamination caused by long distance atmospheric transport of PBDEs could be considered as another way of contamination of this location (Hale *et al.* 2006; Vonderheide *et al.* 2008). In the water samples taken from this location the congener BDE 47 reached the highest concentration (213.4 ng.L^{-1}).

If we carry out the assessment according to the whole content of PBDEs in sediment, the highest contamination was found in location A ($614.0 \mu\text{g.kg}^{-1}$) and location B ($552.0 \mu\text{g.kg}^{-1}$) which are situated in the town of Adamov. This town with 4,500 inhabitants used to be an important center of machine industry in the post-war period, later on a center of production of polygraphic machines and pumping and measurement technology (Adamov 2015). Although the industry was significantly reduced at the beginning of the 1990s, the pollution might originate from an old load (Rahman *et al.* 2001) or insecure industry premises. In the sediment sample from location A the highest concentration was recorded at BDE congener 47 ($457.6 \mu\text{g.kg}^{-1}$). Another significantly loaded location is represented by Brno-Tuřany (location H) where the PBDEs concentration of $583.2 \mu\text{g.kg}^{-1}$ of dry matter was detected. The river leaves the city of Brno in this location and that is why it is possible that contaminants originating from the whole city might be accumulated in the sediments at this place. A greater incidence of PBDEs near urbanized locations was confirmed also by different authors (Vonderheide *et al.* 2008; Melymuk *et al.* 2014). Brno is the second largest city of the Czech Republic (370,000 inhabitants) where is expected a presence of extensive and constantly developing industrial production, and therefore related pollution of all parts of the environment.

Concerning water samples congeners BDE 28 and BDE 99 were the most often detected ones. Congeners

Tab. 1. Limits of detection (LOD) and limits of quantification (LOQ) for individual PBDE congeners [ng.mL^{-1}].

| PBDE | LOD | LOQ |
|---------|--------|--------|
| BDE 28 | 0.2015 | 0.6716 |
| BDE 47 | 0.1805 | 0.6016 |
| BDE 100 | 0.0901 | 0.3004 |
| BDE 99 | 0.0340 | 0.1133 |
| BDE 154 | 0.0598 | 0.1995 |
| BDE 153 | 0.0459 | 0.1529 |
| BDE 183 | 0.1729 | 0.5763 |

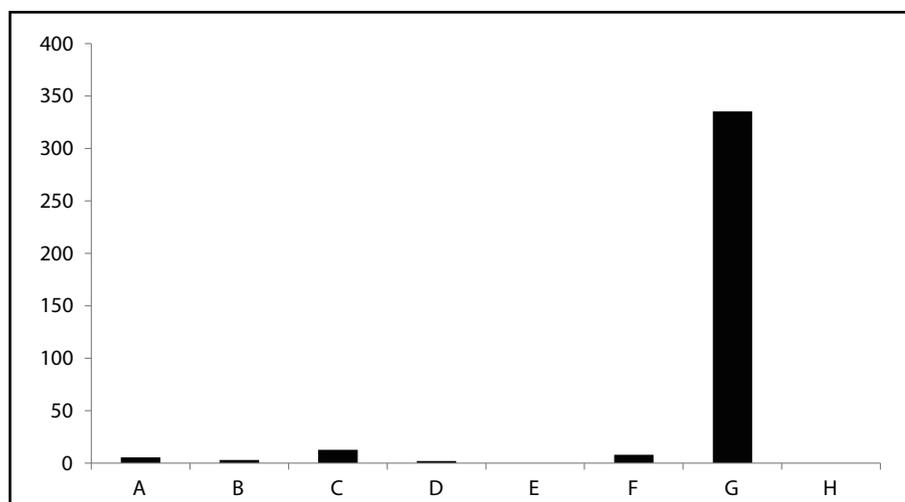


Fig. 2. Comparison of Σ PBDEs from individual locations in water samples [ng.L^{-1}].

Tab. 2. Concentrations of selected PBDE congeners determined in water samples [ng.L⁻¹] and basic statistic parameters.

| Sampling location | BDE 28 | BDE 47 | BDE 100 | BDE 99 | BDE 154 | BDE 153 | BDE 183 | Σ PBDEs |
|-------------------|--------------|--------------|---------|------------|------------|------------|---------|------------|
| A | 4.658±0.38 | <LOQ | ND | 0.683±0.06 | ND | ND | ND | 5.341 |
| B | <LOD | <LOD | ND | 0.511±0.13 | ND | 2.529±0.09 | ND | 3.040 |
| C | 5.795±2.93 | 6.150±2.79 | ND | 0.638±0.20 | ND | ND | ND | 12.58 |
| D | 1.534±0.35 | <LOD | ND | 0.445±0.02 | ND | ND | ND | 1.979 |
| E | <LOD | <LOD | ND | ND | ND | ND | ND | - |
| F | 1.367±0.11 | <LOQ | ND | ND | 6.681±1.24 | ND | ND | 8.048 |
| G | 116.5±17.1 | 213.4±1.71 | ND | 5.427±0.27 | ND | ND | ND | 335.3 |
| H | <LOQ | <LOD | ND | ND | ND | ND | ND | - |
| Mean | 16.23 | 27.44 | - | 0.963 | 0.835 | 0.316 | - | 45.79 |
| Median | 1.451 | - | - | 0.478 | - | - | - | 4.191 |
| Range | <LOD – 116.5 | <LOD – 213.4 | - | ND – 5.427 | - | - | - | ND – 335.3 |

ND - not detected; LOD - limit of detection; LOQ - limit of quantification; Σ - sum of 7 PBDE congeners

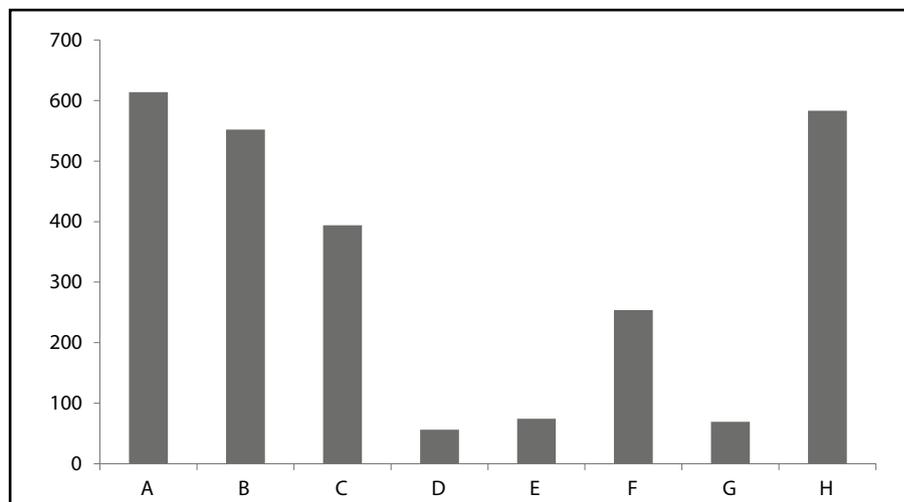
Tab. 3. Concentrations of selected PBDE congeners determined in sediment samples [µg.kg⁻¹] and basic statistic parameters

| Sampling location | BDE 28 | BDE 47 | BDE 100 | BDE 99 | BDE 154 | BDE 153 | BDE 183 | Σ PBDEs |
|-------------------|---------------|---------------|------------|------------|------------|------------|------------|---------------|
| A | 154.2±13.1 | 457.6±22.6 | ND | 2.887±0.19 | ND | ND | ND | 614.0 |
| B | 219.1±6.44 | 297.3±24.3 | ND | 30.83±2.96 | 4.770±0.07 | ND | ND | 552.0 |
| C | 25.58±1.43 | 16.63±3.98 | 0.604±0.12 | 2.411±0.37 | ND | 6.611±1.91 | 342.1±19.6 | 393.9 |
| D | 16.99±0.69 | 39.36±17.1 | ND | ND | ND | ND | ND | 56.35 |
| E | 62.66±3.32 | 7.514±0.31 | <LOD | 3.445±1.21 | 0.658±0.06 | ND | ND | 74.28 |
| F | 119.6±2.82 | 133.0±7.82 | ND | ND | 1.157±0.23 | ND | ND | 253.8 |
| G | 32.88±10.1 | 36.15±8.70 | ND | 0.458±0.02 | ND | ND | ND | 69.49 |
| H | 260.4±5.03 | 320.0±4.36 | <LOD | 2.835±0.11 | ND | ND | ND | 583.2 |
| Mean | 111.4 | 163.4 | 0.076 | 5.358 | 0.823 | 0.826 | 42.76 | 324.6 |
| Median | 91.13 | 86.18 | - | 2.623 | 1.157 | - | - | 323.9 |
| Range | 16.99 – 260.4 | 7.514 – 457.6 | - | ND – 30.83 | ND – 4.770 | - | - | 56.35 – 614.0 |

ND - not detected; LOD - limit of detection; LOQ - limit of quantification; Σ - sum of 7 PBDE congeners

BDE 154 and BDE 153 were detected only in one sample and congeners BDE 100 and BDE 183 were not detected at all. In sediment samples congeners BDE 28 and BDE 47 were detected in all locations. Congeners BDE 100, BDE 153 and BDE 183 were detected only in one sample.

From values presented in Tables 2 and 3 it is evident that in water and sediment samples only low-brominated congeners (BDE 28, BDE 47 and BDE 99) were detected. High-brominated congeners are less

**Fig. 3.** Comparison of Σ PBDEs from individual locations in sediment samples [µg.kg⁻¹].

stable and are easily degraded in a physical-chemical and biogenous way to low-brominated congeners which are strongly represented in the environment (Rahman *et al.* 2001; Hale *et al.* 2006).

From Figures 2 and 3 follows that compared to sediments, PBDEs occurred in water samples in considerably lower concentrations, in nanograms per liter of water. Due to limited solubility in water and a high *n*-octanol water partition coefficient (K_{ow}) PBDEs priority adsorb to organic components of the bottom sediments. These properties also considerably limit the mobility of these substances in the soil system (Thorenz *et al.* 2010). That is the reason why the content of PBDEs in sediments is higher than in water and why they can be sorbed there for a long time; except for that their consequential distribution into the water column has already been proven (Oros *et al.* 2005). This fact can negatively influence most water organisms and thus disrupt the sensitive balance of the water ecosystem (Holoubek *et al.* 1996; Mhadhbi *et al.* 2012).

The percentage of representation of individual PBDE congeners in sediment samples are compared in Figure 4. In most locations congeners BDE 28 and BDE 47 were strongly represented, whereas in locations C congener BDE 183 was in majority. From Figure 4 also follows that the ratio of low-brominated congeners BDE 28 and BDE 47 is more or less the same in all sediment samples (approximately 1:1). The profile of PBDE congeners in the sediments is relatively constant and shows minimal degradation (Hale *et al.* 2006). If we compare the concentration values of these congeners in water samples, we will find out that this ratio considerably varies. This

can be caused by the fact that the water environment is a dynamic system in which the levels of pollutants vary depending on the movement of elements to which PBDEs are bound (Oros *et al.* 2005).

If we assess sediment contamination, the least contaminated places were location D, situated in the municipality Bílovice nad Svitavou (34,000 inhabitants), and location E, which is located at the inflow of the river into Brno City. The concentrations of the sum of PBDEs in location D were $56.35 \mu\text{g}\cdot\text{kg}^{-1}$ and in location E $74.28 \mu\text{g}\cdot\text{kg}^{-1}$. The less polluted location could be also considered the sampling site G which is situated in the direction of water flow of the Svitava River next to the Essler factory ($69.49 \mu\text{g}\cdot\text{kg}^{-1}$). In these locations, on the contrary, the highest values of PBDEs in water samples were determined. This can be explained by the possible fact that there might have been a release of these analytes from the sediment into the water column (Oros *et al.* 2005). Contamination also might come from some new source of which has so far been demonstrated only in water pollution. However, it is necessary to point out that the concentration values of PBDEs in water are lower by an order of magnitude than in the sediment. In locations E and H PBDEs were not detected in water samples at all. Considerable decline of PBDE concentration in the water sample from location D compared with location C might be explained by the presence of a waste water treatment plant (WWTP) in the municipality of Bílovice nad Svitavou.

If we compare our results with another similar study which deals with the occurrence of perfluorated and brominated compounds in samples of sediments from

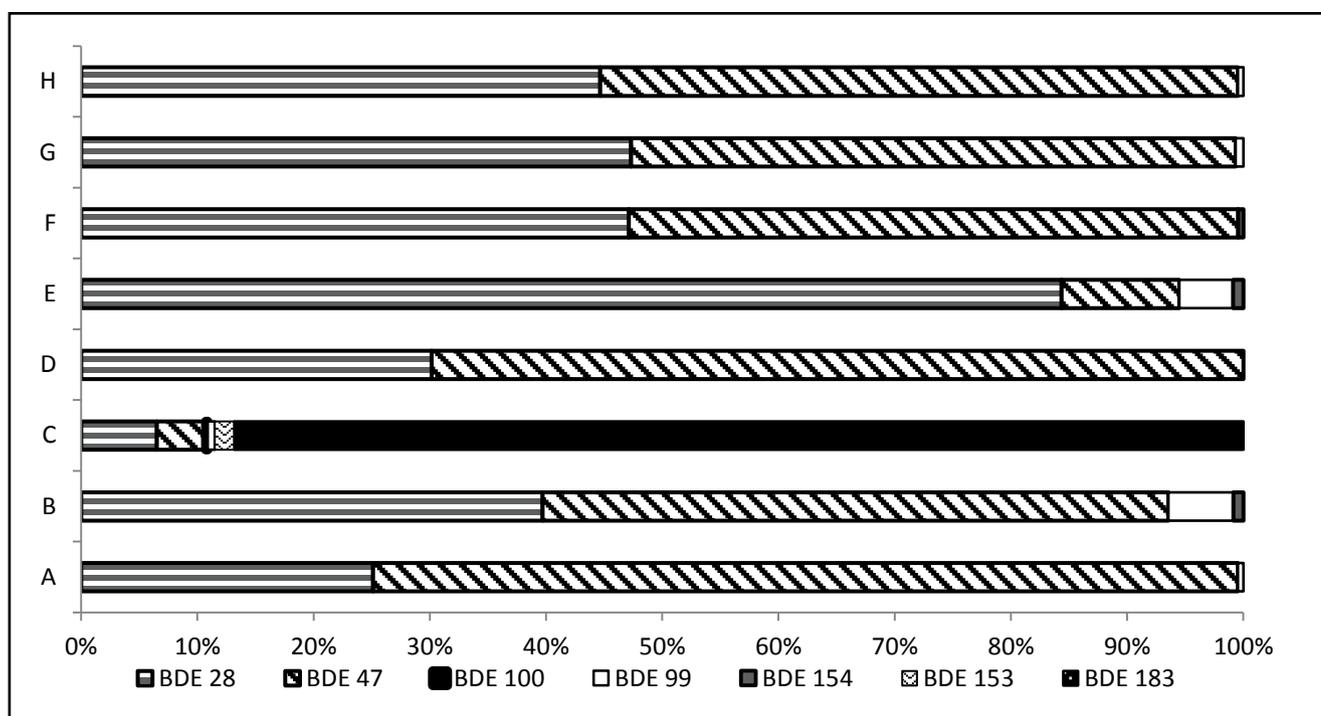


Fig. 4. Proportional representation of individual PBDE congeners in sediment samples [%].

chosen locations of the Czech Republic (Hlouskova *et al.* 2014), we can see that the concentrations of PBDEs measured in this study are comparable; they occur in the same concentration levels. The overall sums of PBDEs ranged from 225.8 to 489.5 $\mu\text{g}\cdot\text{kg}^{-1}$. In our study, the highest value of PBDEs was 614.0 $\mu\text{g}\cdot\text{kg}^{-1}$. In another study dealing with the environmental impact of WWTP on rivers in the Czech Republic, all sums of PBDE congeners ranged from 0.20–0.54 $\mu\text{g}\cdot\text{kg}^{-1}$ in sediment (Li *et al.* 2011), which is significantly less than concentrations determined in our study. Any study dealt with the occurrence of PBDEs in surface water in the Czech Republic was found. However, another halogenated POPs such as polychlorinated biphenyls (PCBs) or DDT – compounds with similar negative health effects and accumulation in the environment – were monitored in rivers around the Brno City, including the Svitava River, by different authors (Blahova *et al.* 2009; Jarova *et al.* 2012; Jirova *et al.* 2014). Their results basically correspond to our results, while the highest levels of pollutants were found at sites situated downstream from Brno.

In conclusion, the results of the presented study document the presence of polybrominated diphenyl ethers in the water ecosystem of the Svitava River. Seven indicator PBDE congeners were monitored in samples of surface water and bottom sediments. It was proven that in the water samples PBDEs occurred in very low concentrations. On the contrary, in the sediment samples rather high concentrations of selected analytes were determined. Low-brominated congeners (BDE 28, BDE 47 and BDE 99), which are also the most toxic, occurred in most cases. The most contaminated locations were found in the town of Adamov (industrial production) and in Brno-Tuřany (downstream of a large city). The highest value of PBDEs in water samples was determined downstream the former Essler textile factory. With regard to the potential risk of negative health effects on humans, the production and use of some PBDEs has been limited or banned. Commercial compounds Penta-BDE and Octa-BDE are included in Annex A of the Stockholm Convention (2009). However, these substances are able to persist and accumulate for a long time in the environment, and therefore monitoring of these hazardous pollutants is of high priority.

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