Occurrence and bioaccumulation of polybrominated diphenyl ethers in sediments and paddy ecosystems of Liaohe River Basin, northeast China

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Abstract

Concentrations of 16 polybrominated diphenyl ether (PBDE) congeners were measured in river sediments, paddy soils and three species of paddy-field organisms (crab, loach and carp) collected from the Liaohe River Basin, northeastern China. The total contents of PBDEs (Σ₁₆PBDEs) in sediments and paddy soils were in the ranges of 273.4–3246.3 pg/g dry weight (dw), and 192.1–1783.8 pg/g dw, respectively. BDE 209 was the dominant congener both in sediments and paddy soils. The concentrations of Σ₁₆PBDEs in sediments were significantly higher than those in the adjacent paddy soils, indicating a potential transport of PBDEs from river to paddy ecosystems via river water irrigation. The biota–soil accumulation factor (BSAF) was calculated as the ratio between the lipid-normalized concentration in paddy-field organisms and the total organic carbon-normalized concentration in paddy soil. The average BSAF values of Σ₁₆PBDEs followed the sequence of crab (3.6) > loach (3.3) > carp (2.1). BDE 154 had the highest BSAF value, and a parabolic trend between BSAF values of individual PBDE congeners and their log Kow values was observed. In view of the fact that crab had the larger BSAF value and higher lipid content, the ecological risk and health risk for crab cultivation in paddy fields should be of particular concern.

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Introduction

Polybrominated diphenyl ethers (PBDEs) are widely used in petroleum, textiles, plastic products, construction materials, transportation equipment and electronic products (Hale et al., 2002; de Wit, 2002). As one type of non-reactive flame retardant additive, PBDEs could be easily released to the environment from their production, application and processing (Voorspoels et al., 2003). Therefore, they have been frequently detected in a variety of environmental matrices (de Wit et al., 2010; Robin et al., 2014). Because of their environmental persistence (de Wit, 2002), long-range atmospheric transport (Goutte et al., 2013), high potential of bioaccumulation (Kelly et al., 2007) and potential adverse effect on the ecosystem and humans (Labunska et al., 2014), commercial penta- and octa-PBDEs were designated as new persistent organic pollutants (POPs) at the fourth meeting of the Conference of the Parties of the Stockholm Convention in May 2009 (UNEP, 2009).

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China is one of the largest producers and consumers of PBDEs in the world, and correspondingly contamination by PBDEs in Chinese environments has received widespread attention (Mai et al., 2005). Since 2000, extensive investigations on the occurrence and distribution of PBDEs in river ecosystems and adjacent seas have been conducted in South and East China (Luo et al., 2007; Wu et al., 2012; Chen et al., 2013). The heaviest PBDE pollution has been found in the Pearl River Delta and several e-waste recycling areas (Xiang et al., 2007; Zou et al., 2007; Wu et al., 2008; Labunská et al., 2014). The average concentrations of PBDEs in sediments from different rivers of the Pearl River Delta were reported to be in the range of 17.1–588 ng/g dry weight (dw) (Chen et al., 2013). Meanwhile, it was found that the mean concentration of PBDEs in the sediments of a river flowing through Guiyu town (Guangdong Province), a typical e-waste recycling area, had surprisingly reached up to 9400 ng/g dw (Luo et al., 2007).

The Liaohe River, which includes the Daliao, Taizi and Hun rivers, is one of the most heavily polluted rivers in China. The tributaries Taizi and Hun rivers flow into the Daliao River at their confluence, before finally entering the Bohai Sea. Since the 1950s, the Liaohe River Basin has been the largest industrial region in northeastern China, with metallurgy, machinery, petrochemical, and building material industries. Meanwhile, there are more than one million acres of irrigated paddy fields in this plain, and the commercial cultivation of river mitten-handed crab and loach in these paddy fields has a long history. Before 2000, most industrial wastewater was directly discharged into the rivers without effective treatment. Previous studies have indicated the ubiquitous occurrence of PBDEs in the sediments of the Daliao River mouth and its adjacent sea, as well as significant bioaccumulation of PBDEs in the marine food web (Zhao et al., 2011a, 2011b; Ma et al., 2013). However, information on the pollution status of PBDEs in the entire Liaohe River Basin is still unavailable, limiting the evaluation of their ecological risks.

The primary objectives of the present study were to investigate the residual levels, spatial distribution and congener profiles of PBDEs in sediments and paddy soils of the Liaohe River Base, and to examine their accumulation in aquatic organisms (crab, loach and carp) of paddy ecosystems. The site-specific bio-soil accumulation factor (BSAF) was also determined to evaluate the bioaccumulation of aquatic organisms in the paddy ecosystem. The obtained results will be of particular value for assessing the ecological risk and human health risk of PBDEs in this typical traditional industrial base.

1. Material and methods

1.1. Sampling sites and sample collection

Twenty-two sediment samples and 14 paddy soil samples were collected from the Liaohe River Basin in June 2010. To investigate the mutual influence between the sediments and soils, the soil samples collected were located close to the corresponding sediment samples (site-specific). The distribution and detailed location information for the sampling sites are shown in Appendix A Fig. S1. Surface sediments of 0–10 cm were sampled using a grab sampler, and paddy soils of the plow layer (0–20 cm) were sampled using a stainless steel scoop. Five subsamples were taken from each site, and then mixed together to form one composite sample for each sampling site. Three species of aquatic organisms including 6 crab (Eriocheir sinensis, ES) samples, 3 loach (Paramisgurnus dabryanus, PD) samples, and 5 carp (Carassius auratus, CA) samples, were collected from the paddy fields by a simple net bag. All collected samples were put into pre-cleaned self-sealing aluminum/polyethylene bags with zip closures, and transported on ice to the laboratory and freeze-dried immediately. The soft parts of biological samples were dissected after checking the weight. The crab samples included meat, embryo, and muscle tissues (in chelae and walking legs). The loach and carp samples consisted of flesh (musculature) and internal organs. The lyophilized samples were ground, homogenized, and stored in pre-cleaned brown glass bottles at −20°C for further analysis.

1.2. Chemical reagents and PBDE analysis

A standard mixture containing 15 PBDE congeners (IUPAC No. BDEs 10, 15, 28, 47, 49, 66, 71, 85, 99, 100, 118, 119, 153, 154 and 183) and a single standard (BDE-209) were purchased from AccuStandards (New Haven, CT, USA). Nine kinds of 13C-labeled surrogate standards (BDEs 3, 15, 28, 47, 49, 99, 138, 153, 154 and 183) and one kind of 13C-labeled internal standard (68A-IS, 13C-P-CB-138) were purchased from Wellington Laboratories Inc. (Ontario, Canada). Hexane, dichloromethane, acetone and nonane were pesticide grade and purchased from Fisher Scientific (J.T. Baker, USA). Sodium sulfate and silica gel (100–200 mesh size) were analytical grade and purchased from Beijing Chemical Reagent Company (Beijing, China) and Merck Co. (Germany), respectively.

About 15 g of sediment/soil and 5 g of organism sample were extracted by a mixture of dichloromethane (DCM) and hexane (V:V = 1:1) using Accelerated Solvent Extraction (ASE350, Dionex, USA). About 5.0 g of activated copper powder was used to remove elemental sulfur in the sediment extract, and 30.0 g of acid silica gel was added to remove lipids in the organism extract. Then the concentrated extracts were further cleaned with multilayer silica columns (10 mm I.D.) and filled from bottom up with activated silica gel (1 g), basic silica gel (4 g), activated silica gel (1 g), acidic silica gel (8 g), activated silica gel (1 g), and AgNO3 silica gel (2 g). A short DB-5HT capillary (15 m × 0.25 mm × 0.10 μm film thickness; J&W Scientific, USA) was used for the separation of mono- to octal-BDE congeners with a programmed temperature. The target compounds were determined by a Trace GC Ultra gas chromatograph (Thermo, USA) coupled with a Trace DSQ II mass spectrometer (Thermo, USA) in electron capture negative ionization (ECNI) mode. Samples were injected in splitless mode, and all data were obtained in the selected ion monitoring (SIM) mode using 13C2 isotope dilution analysis for qualitative and quantitative analysis.

1.3. QA/QC

Strict quality controls were implemented to ensure the correct identification and accurate quantification of the
target compounds. All equipment was thoroughly rinsed with DCM before and after experiments, and the sample preparations were conducted in a super clean lab to avoid background contamination. Six method blanks were included in sample analysis to monitor contamination, and the results showed that all targeted PBDEs were below the detection limits. The surrogate recoveries in all samples ranged from 70.3% to 96.5%, and the relative standard deviation (RSD) for replicate analyses was less than 15% (n = 3). The method detection limits (MDLs), calculated as three times the standard deviation of blanks, were 0.1–0.5 pg/g for di- to hepta-BDE congeners and 5.0 pg/g for BDE 209 in sediments. The MDLs were 0.2–1.0 pg/g for di- to hepta-BDE congeners in organisms. The final concentrations of PBDEs were corrected by the surrogate recoveries.

1.4. Other parameters and statistical analysis

Total organic carbon (TOC) contents in sediments and paddy soils were measured by the high temperature combustion method using a total organic carbon analyzer (Vario TOC cube, Elementar Co. Ltd., Germany). Lipid contents in biological soils were measured by the high temperature combustion total organic carbon (TOC) contents in sediments and paddy soils were measured by the high temperature combustion method using a total organic carbon analyzer (Vario TOC cube, Elementar Co. Ltd., Germany). Lipid contents in biological organisms were gravimetrically determined by an extraction method using a mixture of hexane and acetone (V/V = 1:1). Correlation analysis was used to determine the difference between two data sets and a p value below 0.05 was considered significant. Principle component analysis (PCA) was conducted to characterize the distribution of PBDE congeners. All statistics and drawings were generated by the software ArcGIS 10.3 (Esri Inc., USA), Origin 8.5 (OriginLab Inc., USA) and SPSS 17.0 (SPSS Inc., USA).

2. Results and discussion

2.1. Residue levels of PBDEs in sediment, soil and organisms

A total of 16 PBDE congeners were accurately quantified using the isotopic internal standard methods, and most of them were detected in the collected samples. The statistical results for PBDEs in sediment, paddy soil and aquatic organisms are listed in Table 1. The total concentrations of $\Sigma_{15}$PBDEs (excluding BDE 209) were in the ranges of 47.2–543.3 pg/g dw (mean value: 156.2 pg/g dw) in sediments and 34.3–213.2 pg/g dw (mean: 68.0 pg/g dw) in paddy soils, respectively. The congener BDE 209 predominated both in sediments and paddy soils, and the concentrations ranged from 137 to 2703 pg/g dw (887 pg/g dw) and from 127 to 1589 pg/g dw (428 pg/g dw) respectively. The summed concentration of $\Sigma_{15}$PBDEs in sediment (range: 192–3246 pg/g dw, average: 1035 pg/g dw) was significantly lower than the data reported earlier in Europe and North America (Allchin et al., 1999; Sellström et al., 1998; Lacorte et al., 2003; Eljarra et al., 2004). Compared with domestic results, the PBDE levels in sediments of the Liaohe River were comparable to those of the Haihe River (0.06–2.1 ng/g dw, Zhao et al., 2011a, 2011b), whereas far lower than those of the Pearl River Estuary (3.67–2520 ng/g dw, Chen et al., 2013). These results implied that amounts of PBDEs used in the Liaohe River Basin were relatively less.

The congener BDE 209 was not detected in aquatic organisms (crab, loach and carp) in this field study. The $\Sigma_{15}$PBDE concentrations based on lipid weight (lw) ranged from 14.7 to 81.5 ng/g lw with a mean value of 46.9 ng/g lw. These

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sediment (pg/g dry weight (dw))</th>
<th>Paddy soil (pg/g dw)</th>
<th>Aquatic organisms (ng/g lipid weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Number</td>
<td>n = 21</td>
<td>n = 14</td>
</tr>
<tr>
<td>BDE 10</td>
<td>ND 8.0 (1.9)</td>
<td>ND 1.5 (0.43)</td>
<td>0.3–1.7 (0.9)</td>
</tr>
<tr>
<td>BDE 15</td>
<td>3.6–99.9 (35.5)</td>
<td>7.3–56.0 (18.1)</td>
<td>2.1–40.0 (17.9)</td>
</tr>
<tr>
<td>BDE 28</td>
<td>1.1–32.0 (7.7)</td>
<td>1.1–17.6 (4.3)</td>
<td>1.2–3.5 (2.3)</td>
</tr>
<tr>
<td>BDE 49</td>
<td>ND–30.3 (6.3)</td>
<td>ND–6.2 (2.0)</td>
<td>ND–20.0 (0.7)</td>
</tr>
<tr>
<td>BDE 47</td>
<td>8.9–285.9 (66.8)</td>
<td>4.2–62.9 (25.7)</td>
<td>5.8–23.0 (16.4)</td>
</tr>
<tr>
<td>BDE 66</td>
<td>ND–28.0 (3.8)</td>
<td>ND–5.4 (1.1)</td>
<td>0.5–3.9 (1.5)</td>
</tr>
<tr>
<td>BDE 71</td>
<td>ND–44.4 (4.4)</td>
<td>ND–8.9 (2.1)</td>
<td>ND–3.1 (1.4)</td>
</tr>
<tr>
<td>BDE 85</td>
<td>ND–6.1 (1.0)</td>
<td>ND–3.3 (0.7)</td>
<td>ND–0.2 (0.1)</td>
</tr>
<tr>
<td>BDE 99</td>
<td>1.3–65.9 (9.8)</td>
<td>1.1–44.4 (6.4)</td>
<td>1.2–10.1 (4.1)</td>
</tr>
<tr>
<td>BDE 100</td>
<td>0.2–18.6 (3.1)</td>
<td>ND–2.1 (0.4)</td>
<td>0.2–1.0 (0.6)</td>
</tr>
<tr>
<td>BDE 118</td>
<td>ND–11.1 (2.1)</td>
<td>ND–3.8 (0.7)</td>
<td>0.5–1.8 (0.9)</td>
</tr>
<tr>
<td>BDE 119</td>
<td>ND–16.4 (2.3)</td>
<td>ND–5.5 (1.1)</td>
<td>ND–2.2 (0.7)</td>
</tr>
<tr>
<td>BDE 153</td>
<td>0.3–15.4 (3.7)</td>
<td>0.5–4.3 (1.6)</td>
<td>0.4–2.4 (1.3)</td>
</tr>
<tr>
<td>BDE 154</td>
<td>0.3–10.9 (2.6)</td>
<td>0.3–4.9 (1.4)</td>
<td>0.1–1.4 (0.7)</td>
</tr>
<tr>
<td>BDE 183</td>
<td>0.5–26.6 (5.2)</td>
<td>0.2–4.1 (1.9)</td>
<td>0.1–0.7 (0.4)</td>
</tr>
<tr>
<td>BDE 209</td>
<td>137–2703 (887)</td>
<td>127–1589 (428)</td>
<td>ND</td>
</tr>
<tr>
<td>$\Sigma_{15}$PBDEs</td>
<td>47.2–543.3 (156.2)</td>
<td>34.3–213.2 (68.0)</td>
<td>14.7–81.5 (50.0)</td>
</tr>
</tbody>
</table>

PBDE: polybrominated diphenyl ether.
* Not detected.
** $\Sigma_{15}$PBDEs is the sum of all target PBDE congeners except for BDE 209.
concentrations were higher than organic matter-based contents of \(\sum_{15}\text{PBDEs}\) in paddy soils (range: 2.3–71.6 ng/g TOC, average: 13.4 ng/g TOC), indicating a bioaccumulation potential of PBDEs in aquatic ecosystems. Compared with other reported data, the median residual level of PBDE in the aquatic organisms was higher than that around the Bohai Sea (median = 0.68 ng/g dw, Wang et al., 2009), whereas much lower than that from the Pearl River Delta (52.7–1702 ng/g wet weight, Wu et al., 2008).

There were no significant correlations between \(\sum_{16}\text{PBDE}\) concentrations and TOC values in the surface sediments and paddy soils, respectively (Appendix A Fig. S2), indicating that the organic matter did not play a crucial role in the input and transport of PBDEs in the study area. However, the correlation between \(\sum_{15}\text{PBDEs}\) and lipid contents in organisms was significant \((p < 0.01, \text{Appendix A Fig. S3})\), suggesting that the lipid content was a major factor influencing the distribution of PBDEs in organisms.

### 2.2. Spatial distribution of PBDEs

As shown in Fig. 1, the concentrations of \(\sum_{16}\text{PBDEs}\) in the sampled sediments and paddy soils near the cities (S07, S14, S15, S20 and S21) were significantly higher than those far away from the cities, and the contents of BDE 209 were slightly higher around the cities. Moreover, the concentrations of \(\sum_{16}\text{PBDEs}\) in sediments were significantly higher than those in the adjacent paddy soils (Fig. 1). Correlation analysis was conducted for the sample pairs of a sediment sample and its adjacent paddy soil, and the result showed a significant correlation \((n = 13, p < 0.01, \text{Appendix A Fig. S4})\) for the concentrations of \(\sum_{15}\text{PBDEs}\). Similarly, the concentrations of BDE 209 in sediments were also found to be significantly correlated to those in the adjacent paddy soils \((n = 10, p < 0.05, \text{Appendix A Fig. S5})\), except for three sample pairs near the cities (S07, S19 and S20). This phenomenon indicated potential transport of PBDEs from river to paddy ecosystems via river water irrigation.

### 2.3. PBDE congener pattern

As shown in Fig. 1, BDE 209 dominated the PBDE congeners in all sediment and paddy soil samples. The percentage contributions of BDE 209 ranged from 62.5% to 94.4% in sediments and from 65.0% to 94.5% in paddy soils. The extremely high proportion of BDE 209 indicated that the technical deca-BDE mixture was mainly used in the Liaohe River Basin. The relative contribution of low brominated BDEs other than BDE-209 was also different from those in commercial mixtures, such as DE-71 (USA) and Bromkal 70-SDE (Europe) (La Guardia et al., 2006). For example the composition of PBDEs with 2–5 bromine atoms descended in the sequence tetra-BDEs (44.2%), di-BDEs (25.2%) and penta-BDEs (16.8%) both in sediments and paddy soils (Appendix A Fig. S6). The tetra-BDEs were comparable with those in Penta-BDE mixture (41%–42%), whereas the penta-BDEs were significantly low (44%–45%, Alaee et al., 2003). This result was also consistent with the results reported by Zhao et al. (2011a, 2011b) and Wang et al. (2009) in this area.

Interestingly, the relative contribution of low brominated BDEs in this area was comparable with other types of areas (Mai et al., 2005; Gorgy et al., 2013; Robin et al., 2014), even the remote polar regions (Wang et al., 2012; Zhu et al., 2015). One of
the important reasons was due to the degradation of highly brominated congeners; for example, the debromination of BDE 209 can occur through photolysis, micro-biological degradation and biodegradation (Söderström et al., 2004; Huang et al., 2010). In the present study, combined with the relatively higher di-BDE contribution (25.2%), we could find that some low brominated BDEs were very likely derived from degradation of highly brominated BDEs in this area.

Principal component analysis (PCA) was performed to further investigate the congener characteristics in different matrices (as shown in Fig. 2). The result showed that the first two components accounted for about 90% of the original variables (Fig. 2a). In the score plot, partial sediment and paddy soil samples, especially 7 sediments collected from near the cities (S02, S07, S14, S15, S19, S20 and S21), were classified into one group. They all had negative scores on the second component due to the relatively high content of BDE 209 (Fig. 2b). This was consistent with the spatial analysis above and further indicated that the cities are likely to be the main source of PBDEs. Notably, the aquatic samples clustered together on the PCA score plot due to the relatively high contents of low brominated BDEs and no detection of BDE 209. However, the wide range of factor score 1 for aquatic samples also implied the different bioaccumulation behaviors of different species.

2.4. Biota–soil accumulation factors

The biota–soil accumulation factor (BSAF) was calculated as described by Gobas et al. (2009): BSAF = \( \frac{C_{\text{org}}}{C_{\text{soil}}} \), where \( C_{\text{org}} \) and \( C_{\text{soil}} \) represent the PBDE concentrations in organisms based on lipid weight (ng/g lw) and soil normalized by the total organic carbon (ng/g TOC), respectively. The calculated BSAF values of different BDE congeners and \( \sum_{15} \)PBDEs are listed in Appendix A Table S1. The BSAF values of \( \sum_{15} \)PBDEs with descending mean values followed the sequence of crab (3.6) > loach (3.3) > carp (2.1). These BSAF values were comparable with the biota–sediment accumulation factor of total PBDEs in mussels from the Bohai Sea (0.17–11.42, Wang et al., 2009). The BSAF values >1 indicated that these three species of aquatic organisms all had the capability to accumulate PBDEs from paddy fields. In view of the fact that crab had a larger BSAF value and higher lipid content, the ecological risk and health risk for crab cultivation in paddy fields should be of particular concern.

Correlation analysis was conducted for BSAF values and logarithmic octanol–water partition coefficient (log\( K_{\text{OW}} \)) values of individual PBDE congeners, and the results are shown in Fig. 3. BDE 154 (log\( K_{\text{OW}} \): 7.79) had the highest BSAF value, followed by BDE 49 and BDE 100, which was consistent with the results for PBDEs in wild aquatic species from an electronic waste recycling site in South China (Wu et al., 2008). The relationship between BSAF values and log\( K_{\text{OW}} \) values essentially conformed to a two power equation, although the correlation was not significant (\( p > 0.05 \)). BSAF value first
presented an increasing tendency with the increase of $\log K_{OW}$ value until 7.0, and then reached a maximum at the $\log K_{OW}$ values of 7.0–8.0, and finally showed a decreasing tendency with the continuous increase of $\log K_{OW}$ value. The reasons for the parabolic trend between BSAF and $\log K_{OW}$ are likely to be complex. On one hand, the low brominated congeners with smaller $\log K_{OW}$ values are easily metabolized in organisms (Mizukawa et al., 2009), which might be mainly responsible for the smaller BSAF values of low brominated congeners. On the other hand, the molecular steric hindrance could limit the membrane transport of highly brominated congeners with larger $\log K_{OW}$ values from the extracellular environment into cells (Shaw and Connel, 1982; Kelly et al., 2008), which would result in a reduction in BSAF values for highly brominated congeners. In addition, the highly brominated congeners had higher affinity to carbonaceous geosorbents in soil (Moermond et al., 2005). The competitive adsorption of soil organic matters can also reduce the BSAF values of highly brominated congeners.

3. Conclusions

The residual level of PBDEs in the traditional industrial base of China was relatively low compared with other developed regions. The sites with higher concentrations of PBDEs were mostly located in the vicinity of the cities. The site-specific correlation analysis for sediments and their adjacent paddy soils indicated potential transport of PBDEs from river to paddy ecosystems via river water irrigation. Three species of aquatic organisms in paddy fields, crab, loach and carp, all had high BSAF values of $>1$ for $\sum_{i}^{15}$PBDEs, indicating that they had the capacity to accumulate PBDEs from the paddy field. BDE 154 had the highest BSAF value, and a parabolic trend between BSAF values and $\log K_{OW}$ was observed. In view of the fact that crab had a larger BSAF value and higher lipid content, the ecological risk and health risk for crab cultivation in paddy fields should be of particular concern and further investigated.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.jes.2015.10.016.

References


