PBDEs in marine and freshwater sediments from Belgium: levels, profiles and relations with biota

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Sediments from the Belgian North Sea (BNS), the Western Scheldt Estuary (SE) and freshwater watercourses from the Scheldt basin were analysed for eight PBDE congeners, namely BDEs 28, 47, 99, 100, 153, 154, 183 and 209. Previously analysed biological samples from the same locations in the BNS and the SE have been shown to contain large amounts of PBDEs. Surprisingly, PBDE concentrations in the sediments were below the LOQ for samples from the BNS (except BDE 209), while in those from the SE the sum of PBDEs (not including BDE 209) were higher and ranged from 0.20 to 0.41 ng g\(^{-1}\) dw. BDE 209 could be detected in 83% of the samples from the BNS and in all the samples from the SE. Concentrations up to 1200 ng g\(^{-1}\) were hereby measured in the SE. Compared to the marine and estuarine locations, the sediments from the freshwater watercourses were relatively more polluted with the lower brominated PBDEs (<0.20–19 ng g\(^{-1}\) dw). BDE 209 concentrations up to 320 ng g\(^{-1}\) dw were measured in those sediments. However, the contribution of BDE 209 to the total amount of PBDEs varied much more at the freshwater locations than in the SE, which suggests a different input of pollutants. PBDE profiles observed in biological samples do not match the profiles of the sediments. BDE 183 and 209 could not be quantified in biota, although these congeners were undoubtedly present in the sediments. This raises questions about the bioavailability of these congeners in the environment.

1. Introduction

Polybrominated diphenyl ethers (PBDEs) are used as flame retardants to improve the fire safety of various items. These chemicals have shown a rise in production since they were first introduced in the 1960s, with a substantial increase since the end of the 1970s due to the growing popularity of computers and other electronic equipment. Of the total worldwide market demand for PBDEs in 2001, more than 80% consisted of the fully brominated decabromodiphenyl ether (BDE 209). This PBDE congener is of special interest because its presence is being increasingly reported worldwide. However, a ban on use or production is not foreseen, because environment and human risk assessment reports have concluded that there is no significant risk.

Both biotic and abiotic compartments are polluted with PBDEs. This is why brominated flame retardants (BFRs), and especially PBDEs, have received increasing attention during recent years. Their extremely resistant and lipophilic nature is partly responsible for their widespread presence. These properties cause residues of these persistent organic pollutants (POPs) to bioaccumulate in the organic fraction of soils and sediments, and in adipose tissues of biota, which results in an enrichment throughout the food chain.

The water solubility and vapour pressure of PBDEs are very low causing them to be adsorbed rapidly onto solid particles of sediment and soil when released into the environment. Sediment and sewage sludge are therefore considered a sink for these pollutants, although pollutants can be still bioavailable once adsorbed to the sediment particles. Soil and sediment studies can provide valuable information about the pollution load in a certain area. Additionally, the study of sediments is an important step in mapping possible exposure pathways to various marine and freshwater organisms.

The area studied in this article covers the Belgian North Sea (BNS) and the Western Scheldt Estuary (SE). The drainage basin of the SE covers a very densely populated and highly industrialised region, resulting in a high pollution level with POPs, as well as heavy metals and non-persistent pollutants. The present study focused on the determination of PBDEs in sediments sampled at predefined locations in both the BNS and the SE. Previous studies have revealed a correlation between distance to Antwerp and degree of pollution in benthic invertebrates and fish. Additionally, 14 sediment samples were taken from tributaries of the Scheldt to assess sediment pollution at several inland locations in the Scheldt basin.

Materials and methods

Sampling

Six locations in the BNS (S1–S6), 11 locations in the SE (S7–S17), and 14 freshwater locations in rivers and watercourses of the Scheldt Basin (T1–T14) were sampled (Fig. 1). Surface sediments from the BNS and SE were taken in October and November 2001 using a Van Veen surface sediment sampler. Surface sediments from the BNS and SE were taken in October and November 2001 using a Van Veen surface sediment sampler. A cruise with the research vessel Zeeleeuw, provided by the Flemish Marine Institute (VLIZ). Because the Scheldt is the main gateway to the Antwerp harbour, the river is highly trafficked and dredged. In order to avoid sampling at sites that were subjected to dredging, a map that showed the dredging activities was used to determine the appropriate sampling locations.

At each location, 4 subsamples of approximately 10 L sediment were taken within a radius of 50 m and combined. The approximate sampling depth was 20–25 cm. The sediment was kept in hexane pre-washed glass receptacles at +4 °C until further processing.

The samples from rivers and watercourses of the Scheldt basin were collected with a ‘Petit Ponar’ grab sampler during August and September 2001. At each site a mixed sample composed of 5 grab samples was taken. The sediments were sieved with 5 L of the site-water using a 200 μm-mesh sieve to
remove the organisms. The sediment was stored in 500 ml polyethylene beakers at +4 °C until further processing.

**Targeted compounds**

Based on reported abundance and toxicity, the following BDE-congeners (IUPAC numbering) were targeted for analysis: 28, 47, 99, 100, 153, 154, and 209. Brominated biphenyl (BB) 103 was used as IS for BDE 28 and 47, and BB 155 was used as IS for BDE 99, 100, 153, 154, and 183. 13C-labeled BDE 209 was used as IS for BDE 209.

**Chemicals**

All solvents used for the analysis (n-hexane, acetone, dichloromethane, and iso-octane) were of SupraSolv grade (Merck, Darmstadt, Germany). Individual reference standards for each compound were used for identification and quantification (CIL, Andover, USA; Dr Ehrenstorfer Laboratories, Augsburg, Germany).

**Extraction and clean up**

The sediments from BNS and SE were dried for 1 week at room temperature until constant weight and consecutively sieved through 1 mm and 180 μm hexane-washed stainless steel sieves. The fine fraction was homogenised and used for analysis. The sediments from the freshwater locations were freeze-dried, the fine fraction was homogenised and used for analysis. The sediment samples from the BNS were low and the majority of congeners were below the LOQ. The sum of PBDEs (BDE 28, 47, 99, 100, 153, 154, and 183) was measured. Tests were considered significant if p was lower than 0.05. All statistical tests were performed using Statistica software (StatSoft Inc., Tulsa, USA).

**Quality assurance**

The quality control (QC) was done by regular injection of solvent blanks and standard solutions. Each batch of 8 samples included a procedural blank, blind duplicate sample and in-house reference material (the sediment sample used for the BROC interlaboratory feasibility study). The individual PBDE-congener concentrations did not differ more than 15% from the mean values obtained in the BROC-study. Recoveries of all congeners varied between 83 and 92% (RSD < 16%).

Procedural blanks for all PBDEs were consistent (RSD around 30%) and therefore the median blank values for these compounds were used for subtraction. Blanks for BDE 209 were one order of magnitude higher than for the other PBDE-congeners. The limit of quantification (LOQ) was determined by laboratory background levels. Two times the standard deviation of the procedural blank level was used as the LOQ, resulting in a certainty of more than 95% of the presented results.

**Statistical analysis**

Only samples that had measurable amounts (> LOQ) of analytes were used for the statistical calculations. Simple linear regression was used to test for correlations between the total PBDE load and BDE 209 levels. To test for differences between profiles, an ANOVA with Scheffe’s post hoc test was performed. Tests were considered significant if p was lower than 0.05. All statistical tests were performed using Statistica software (StatSoft Inc., Tulsa, USA).

**Results and discussion**

PBDE levels

All results are summarised in Table 1. Levels of PBDEs in the BNS samples were low and the majority of congeners were below the LOQ. The sum of PBDEs (BDE 28, 47, 99, 100, 153, 154 and 183; hereafter referred to as “sum of PBDEs”) was never higher than the calculated LOQ for these locations (<0.2 ng g⁻¹ dw). BDE 209, however, could be detected in 83% of samples from the BNS, although at rather low concentrations (1.1–24 ng g⁻¹ dw). Locations near the industrial harbour of Zeerugge (S4; S6) were the only locations in the BNS where the sum of PBDEs could be measured and where BDE 209 showed a tenfold higher concentration compared to the other locations in the BNS (Table 1).

At locations S7 to S14 (SE) the sum of PBDEs ranged from <0.20 to 0.41 ng g⁻¹ dw. Similar to the other PBDEs, concentrations of BDE 209 were relatively low at these locations.
locations in the SE (1.5–47 ng g⁻¹ dw). At locations S15 to S17, concentrations of the lower brominated PBDEs were approximately 10 to 50 times higher than at downstream locations (5.3–18 ng g⁻¹ dw). At the same locations, very high levels of BDE 209 could also be measured (up to 1200 ng g⁻¹ dw).

The Scheldt discharges into the North Sea, projecting its plume even further than Zeebrugge.19 Because BDE 209 concentrations in the SE are relatively low until location S15, it is highly unlikely that the elevated levels found near the Zeebrugge harbour are caused by the influence of the SE. The higher levels found near Zeebrugge (S4; S6) and near the Zeebrugge harbour are caused by the influence of the SE. The highly unlikely that the elevated levels found near the Zeebrugge plume even further than Zeebrugge. 19 Because BDE 209 concentrations in the SE in the present study ranged from 0.06 to 3.7 ng g⁻¹ dw; BDE 209 levels up to 22 ng g⁻¹ dw were reported in the present study. The sediment samples were taken with a Van Veen surface sediment sampler similar to the one used in the present study.

Levels of BDE 47 in sediments from the UK that were taken at or around potential PBDE sources were up to 2 orders of magnitude higher (368 ng g⁻¹ dw) than in the present study. 17 In general, PBDE-concentrations in that study were slightly higher than the sediments from the freshwater locations in the present study. The sediment samples were taken with a small hand-held Van Veen surface sediment sampler, which enabled only taking the first centimetres of the sediment. This is in contrast with the larger Van Veen sampler used in the present study, which sampled up to 25 centimetres depth.

Christensen and Platz collected top-layer (0–2 cm) marine sediments in Denmark.20 Concentrations of the sum of PBDEs ranged from 0.06 to 3.7 ng g⁻¹ dw; BDE 209 levels up to 22 ng g⁻¹ dw were reported. The sediments that originated from the area of the Copenhagen harbour were the most polluted. These sum of PBDEs concentrations were in the same order of magnitude as the sediments in parts of the SE in the present study. BDE 209 concentrations in the SE in the present study were up to 60 times higher (1200 ng g⁻¹) than those reported from Denmark. Compared to values reported in literature,20–23 it can be concluded that levels of BDE 209 in the SE (S15–S17) are relatively high.

### Table 1

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<th>Location</th>
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<th>BDE 153</th>
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* BDE 209 is not included in the sum of PBDEs; n.a. = not available.
PBDE profiles

BDE 209 was the most abundant congener, followed by BDE 47 and 99. These profile characteristics have been previously reported by Zegers et al. for Western Europe. In the present study, profiles were calculated based only on BDE 28 to 183. As BDE 209 was not included in the profile, it was possible to compare the results with the Bromkal DE70-5 technical penta-mixture and other studies. Including this congener in the profile would also bias the ratios, because the contribution of BDE 209 to the total PBDE-load was 95 ± 4.7% for the SE samples and varied between 52% (T9) and 99% (T1) for the tributaries.

For BNS samples no profile could be established due to the very low PBDE-levels. The profile calculated for the SE was very consistent (RSD < 20%, except for BDE 183) (Fig. 2). The profile calculated for the tributaries was less consistent (RSD up to 70%), which can be explained by their geographical distribution; samples T9 and T12 displayed relatively high levels of BDE 154, which influenced the pattern to a rather great extent. The profile at location T1 also deviated, because the levels of BDE 47 were relatively high. Sample T1 was therefore not included in the mean profile calculation.

No significant profile differences were found between the SE and the tributaries. Both sample sets display a similar profile to the Bromkal 70-5DE pentaBDE technical mixture (Fig. 2).

Relations with biota

Although the sum of PBDEs was below the LOQ in the BNS sediments, a similar study performed on mildly migrating benthic invertebrates (such as crab, starfish, shrimp) and fish species (such as dab, plaice, sole, bib, and whiting) reported values for the samples from BNS between 0.02 ng g⁻¹ ww and 108 ng g⁻¹ ww. In the same study, concentrations in biota from the SE ranged from 0.20 to 980 ng g⁻¹ ww. A significant inverse correlation (r² up to 0.77; p = 0.02) between PBDE-concentrations in biota from the SE and the distance to Antwerp was also observed in that study. This correlation was not reflected in the data of the sediments, although sediment is the most likely source of contaminants for the biota living above it, since contaminants trapped on the sediments may be bioavailable to sediment dwelling organisms. However, benthic invertebrates, that are assumed to have lower metabolic capacity than fish, display a profile that was not significantly different from the profile of the technical mixture (Fig. 2). The PBDE-profile in the fishes was extensively altered compared to the sediments. This observation suggests that the limited metabolic capacity of the invertebrates does not seem to enable these organisms to change the pattern to the same extent as can be observed in the fishes. BDE 183 and BDE 209 could not be detected in any invertebrate or fish sample, although they were present in the sediments.

Uptake of higher brominated BDEs was very low in controlled feeding experiments. It was concluded to be related to their high log Kow values and their large molecular size, which hinders them in crossing membranes. Contrarily, in a recent publication by Tomy et al., the uptake of BDE 183 and BDE

![Fig. 2 PBDE profiles in sediments from the SE and freshwater locations, profile of Bromkal 70-5DE and profiles found in biota. Results for Fish indicate the mean profile for dab, plaice, bib, sole and whiting. The error flags indicate 2 × standard error.](image-url)
209 following oral administration was comparable or even higher, respectively, than the uptake of the other congeners.29 The absence of BDE 183 and 209 in the biota samples in the study by Voorspoels et al.12 and their presence in the sediments suggests that the aquatic uptake of BDE 183 and 209 may therefore be restricted by the physical, rather than by the biological availability.

Relatively fast metabolism of BDE 183 and 209 in fish has been repeatedly reported,28–30 which is in agreement with the absence of these congeners in the biota.

To the author’s knowledge, no data are available to date concerning metabolism of PBDEs in invertebrates. Possibly a combination of both low uptake and fast metabolism are related to the observation that both BDE 183 and BDE 209 can be found in the sediments, but not in the invertebrates and fish.

Correlations between BDE 209 and other PBDEs

Only locations at which all measurements were above the LOQ were taken into account for calculating the correlation between BDE 209 and the sum of the other congeners. Levels of BDE 209 and the other PBDEs were significantly positively correlated for all samples from the marine locations (BNS and SE). Log-normalised concentrations showed an $R^2$-value of 0.9372 ($p < 0.05$). The input of these 2 groups of contaminants seems to be related for the SE locations.

For the samples taken at the freshwater locations, the correlation between BDE 209 and other PBDE-congeners was not significant. In particular, location T6 displayed rather high levels of all PBDEs except BDE 209. In general BDE 209 was relatively low and the other PBDEs relatively high in the sediments of the tributaries, which points at a different exposure compared to the SE, where the major PBDE-load consists of BDE 209 (up to 98%). It can therefore be concluded that the exposure to the lower brominated PBDEs and BDE 209 is different in the SE and in the freshwater watercourses of the Scheldt basin.

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References


