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Spatial Distribution and Congener Profiles of Polybrominated Diphenyl Ethers in Surface Sediment from Sanmen Bay and Xiamen Bay, Southeast China

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Abstract
Coastal areas are influenced by anthropogenic input of a variety of organic pollutants, among which polybrominated diphenyl ethers (PBDEs) represent an important group. In the present study, we investigated the contamination status of PBDEs in surface sediment from two economically important Bays in Southeast China, Sanmen Bay (SMB; n = 29) and Xiamen Bay (XMB; n = 10). Concentrations of ∑PBDEs ranged from 2.2 to 78.5 ng/g dw (median 5.7 ng/g dw) in SMB and 7.9–276.0 ng/g dw (median 43.5 ng/g dw) in XMB, respectively. A nearshore-offshore decreasing trend was observed for both ∑PBDEs and BDE-209 concentrations, indicating strong urban influences. Although the current levels would not produce any significant impact on benthos and aquatic ecosystems of the studied regions, continuous monitoring is needed to understand the temporal trends of contamination in the important coastal waters and whether sediment-associated PBDEs constitute a potential source to aquatic ecosystems.

Keywords Polybrominated diphenyl ethers (PBDEs) · Sanmen Bay · Xiamen Bay · Sediment · Spatial distribution · Congener profile

Polybrominated diphenyl ethers (PBDEs) represent a group of brominated flame retardants that have been extensively added to a variety of commercial products for several decades (Salamova and Hites 2013). Three major commercial PBDE mixtures include Penta-BDE, Octa-BDE, and Deca-BDE (de Wit 2002). Penta-BDE mixtures mainly contain BDE-47 and BDE-99 (over 70%); Octa-BDE mixtures mainly comprise BDE-183 and BDE-197, whereas Deca-BDE contains more than 95% BDE-209 (Alaee et al. 2003). Considering that major PBDE congeners are bioaccumulative, persistent, subject to long-range transport, and toxic to organisms (Ni et al. 2013; Liang et al. 2016), commercial mixtures of Penta-BDE and Octa-BDE have been discontinued or phased out since 2004 and Deca-BDE since 2010 in North America (La Guardia et al. 2006; Meng et al. 2011). Penta-, Octa-BDE and Deca-BDE were also added to the Stockholm Convention on Persistent Organic Pollutants (https://chm.pops.int/TheConvention/ThePOPs/TheNewPOPs/tabid/2511/Default.aspx).

Sediment is a main sink/repository and potential secondary source of PBDEs in aquatic systems. Coastal waters connect the land and the ocean, and are subject to elevated PBDE contamination through various pathways, including domestic sewage and industrial wastewater discharge, surface runoff, and atmospheric deposition (Rule et al. 2006; Moon et al. 2007; Jiang et al. 2018). Sediment-associated PBDEs can potentially affect embryo or larval morphology majofrmations, hatching success, larval survival, teratogenicity, DNA damage or developmental delays of benthic

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organisms (He et al. 2008; Mhadhbi et al. 2012; Han et al. 2013), and also impact higher trophic level organisms through food chain transfer (Corsolini et al. 2017; Gandhi et al. 2017; Markham et al. 2018). Therefore, sediment quality represents an important indicator for assessing potential effects of PBDEs on coastal ecosystems. A number of studies have reported the concentrations, distributions, and composition profiles of PBDEs in coastal sediment (Macías-Zamora et al. 2016; Wang et al. 2016a, b; Yuan et al. 2016). However, even though PBDE mixtures have been discontinued for more than a decade, knowledge on the contamination status still remains limited in the coastal waters of Southeast China.

The Sanmen Bay (SMB) is located on the southeastern coast of China, which is the second largest bay in Zhejiang Province. SMB is one of the most important aquacultural regions in China, where several major metropolitan districts (e.g., Taizhou City and Ningbo City) are also located. In recent decades, SMB has been increasingly affected by pollutants produced from industrial, agricultural and aquacultural activities (Yang et al. 2011; Liu et al. 2018). The Xiamen Bay (XMB) is located within the Xiamen Economic Special Zone (SEZ) and is another important bay in Southeast China (Zhang et al. 2009). Rapid urbanization and industrialization of the region has resulted in environmental stresses (e.g., anthropogenic pollution and severe ecological deterioration) in XMB and its adjacent waters. Great amounts of pollutants, such as heavy metals (Yan et al. 2010), polycyclic aromatic hydrocarbon (PAHs), dichlorodiphenyltrichloroethanes (DDTs), hexachlorocyclohexanes (HCHs), and polychlorinated biphenyls (PCBs) (Chen et al. 2002; Maskaoui et al. 2005), have been reported in XMB sediment.

In the present study we aimed to investigate PBDE contamination in SMB and XMB sediment as a reflection of the contamination status in the coastal regions of Southeast China. The occurrence, spatial distribution, and composition profiles of major PBDE congeners were characterized in surface sediment from the two aquatic systems.

Materials and Methods

Surface sediment was collected from SMB (n = 29) during the period of November 2016 to January 2017 and XMB (n = 10) in August 2016, respectively. Approximately 5 cm of sediment in depth from bottom to top was sampled from each location using pre-cleaned stainless steel grab samplers. Two field blanks and one field blank were prepared for the sampling in SMB and XMB, respectively. All samples were immediately transferred to pre-cleaned glass jars and kept in insulation boxes containing ice packs. They were then transported to the laboratory and stored at − 20°C until chemical analysis.

After sediment was freeze-dried and sieved, approximately 5 g of dried sediment was spiked with surrogate standards, including 50 ng each of 4′-fluoro-2,3′,4,6-tetrabromodiphenyl ether (F-BDE69, AccuStandard, New Haven, CT), 4′-Fluoro-2,3,3′,4,5,6-hexabromodiphenyl ether (F-BDE160, AccuStandard), and 2,2′,3,3′,4,5,5′,6,6′-nonabromo-4′-chlorodiphenyl ether (4PC-BDE208, Wellington Laboratories, Ontario, Canada), all with purity > 96% (Wu et al. 2017; Widelka et al. 2016). PBDEs were extracted from sediment via an accelerated solvent extractor (ASE 350, Dionex, Sunnyvale, CA, USA) with dichloromethane (DCM, high performance liquid chromatography or HPLC grade) at a pressure of 1500 psi and a temperature of 100 °C. Concentrated sediment extract was added with approximately 200 mg of activated copper (50 mesh, granular, reagent grade) and kept overnight in refrigerator to remove sulfur. After sulfur removal, the extract was purified by a Shimadzu Prominence Semi-Prep HPLC (Shimadzu America Inc., Columbia, MD) equipped with a gel permeation chromatography column (300 × 21.2 mm, 5 μ, 100 Å; Phenomenex, Inc., Torrance, CA) with 100% DCM as the mobile solvent and a flow rate of 4 mL/min. Target compounds were collected in a fraction ranging from 16 to 45 min which was then further cleaned and separated on a 2 g Isolute silica solid phase extraction (SPE) cartridge. The SPE column was preconditioned with 10 mL of hexane (HEX). After the sample was loaded and the cartridge was washed with 3 mL of HEX (discarded), target PBDEs were eluted out with 11 mL of a mixture of HEX/DCM (40:60, v/v). The latter fraction was concentrated and spiked with 3′-Fluoro-2,2′,4,4′,5,6′-hexabromodiphenyl ether (F-BDE154) prior to instrumental analysis.

The analysis of PBDEs was conducted on an Agilent 7890B GC coupled to a 5977A single quadrupole mass analyzer (Agilent Technologies, Palo Alto, CA) in electro-capture negative ionization (ECNI) mode. A 15-m DB-5HT column (0.25 mm i.d., 0.1 µm, J&W Scientific) was used for the determination of PBDE congeners. The corresponding details of oven program and the MS parameters on PBDE congeners have been presented previously (Tang et al. 2019). The instrumental detection limit (IDL) was defined as a concentration yielding a signal-to-noise ratio (S/N) of five. Analytes with instrumental responses below IDL were considered nondetectable (nd). The method limit of quantification (MLOQ) was evaluated by processing eight replicates of sodium sulfate spiked with small amounts of target PBDE standards (1–5 ng). The MLOQ of an analyte was determined by multiplying the standard deviation of the results from replicate analysis with a Student's t-value designated for a 99% confidence level (Wu et al. 2016). The MLOQs are summarized in Table 1.

Table 1 Concentrations of PBDE congeners (ng/g dw) in sediment from the Sanmen Bay (SMB) and Xiamen Bay (XMB), Southeast China

Congeners	SMB (n = 29)			XMB (n = 10)			MLOQ[c] (ng/g dw)
	Range	Median	DF[a] (%)	Range	Median	DF (%)	
BDE−17	nd[b]–0.08	0.02	86	nd–0.14	nd	10	0.01
BDE−28	0.01–0.23	0.06	100	nd–0.10	nd	30	0.01
BDE−47	nd–0.19	0.02	90	0.00–0.18	nd	30	0.01
BDE−49	nd	nd	0	nd–0.64	nd	20	0.03
BDE −66	nd	nd	0	nd	nd	0	0.02
BDE−85	nd	nd	0	nd–1.20	nd	10	0.03
BDE−99	0.01–0.22	0.03	100	nd–0.19	nd	20	0.01
BDE−100	nd	nd	0	nd–0.13	nd	30	0.03
BDE−138	0.04–0.31	0.10	100	nd–0.40	nd	30	0.05
BDE−153	0.07–1.0	0.61	100	2.0–5.5	3.1	100	0.10
BDE−154	nd	nd	0	nd	nd	0	0.04
BDE−183	0.01–0.26	0.01	100	nd–0.50	nd	40	0.01
BDE−196	nd	nd	0	nd–0.87	nd	20	0.03
BDE−197	nd	nd	0	nd–0.67	nd	30	0.03
BDE−201	nd	nd	0	nd–0.69	nd	40	0.02
BDE−202	nd	nd	0	nd–2.4	nd	20	0.03
BDE−203	nd	nd	0	nd–1.1	nd	30	0.02
BDE−206	nd–1.1	0.08	86	< MLOQ -9.6	0.39	50	0.02
BDE−207	nd–0.69	0.09	90	< MLOQ -6.1	0.29	50	0.03
BDE−208	nd–0.32	0.04	90	< MLOQ -2.9	0.16	50	0.03
BDE−209	1.2–74.6	3.2	100	4.9–254.7	38.7	100	0.10
ΣPBDEs[d]	2.2–78.5	5.7	–	7.9–276.0	43.5	–	0.01

[a]Detection frequency

[b]Non-detectable (S/N < 5)

[c]Below method limit of quantification (MLOQ)

[d]Sum of all PBDE congeners

To evaluate the analytical accuracy and precision, five replicates of National Institute Standard Technology standard reference material (SRM) 1941b marine sediment were processed with the same procedures described. Compared with the certified values (Schantz et al. 2006), the recoveries of detectable PBDE congeners from replicate analyses (n = 5) were 85.0% ± 13.1% for BDE-28, 90.5% ± 8.7% for BDE-47, 88.6 %± 11.4% for BDE-99, 85.3% ± 10.7% for BDE-100, 92.5 ±% 8.3% for BDE-153, 93.6 %± 10.4% for BDE-154, 80.5% ± 15.6% for BDE-184, and 80.8 %± 11.7% for BDE-209.

A procedural blank was processed along with each batch of five samples, and the target compounds were not detected in any procedural blank or field blanks. The average recoveries of surrogate standards in sediment samples were as follows: 89.1% ± 14.1% for F-BDE69, 92.5% ± 13.3% for F-BDE160, and 103.1% ± 15.1% for 4PC-BDE208 (mean ± SD), respectively.

PBDEs concentrations in sediment were adjusted with the recoveries of relevant surrogate standards (i.e., F-BDE69 for analytes with a retention time earlier than that of BDE-85, 4PC-BDE208 for BDE-209, and F-BDE160 for all other analytes) and reported as ng/g dry weight (dw). The level of significance was set as $p < 0.05$. Figures 1 and 2 were conducted using the OriginPro 8.5 (OriginLab Corporation) and Surfer 13.0 (Golden Software, Inc., Denver, CO, USA), respectively.

Results and Discussion

PBDE congeners were determined with a detection frequency (DF) ranging from 0% for BDE-66 to 100% for BDE-209, indicating wide distributions of PBDEs in SMB and XMB. The total concentrations of PBDEs (∑PBDEs, i.e., the sum of the 21 PBDE congeners including BDE-209) ranged from 2.2 to 78.5 ng/g dw (median 5.7 ng/g dw) and 7.9 to 276.0 ng/g dw (median: 43.5 ng/g dw) in SMB and XMB surface sediment, respectively (Table 1). There are significant differences in sediment ∑PBDE levels between the two bays (one-way ANOVA, $p < 0.001$), likely due to the differences in the intensity of human activities between the two coastal regions. According to the Zhejiang Province (https://www.zjso.gov.cn/) and Xiamen City statistical

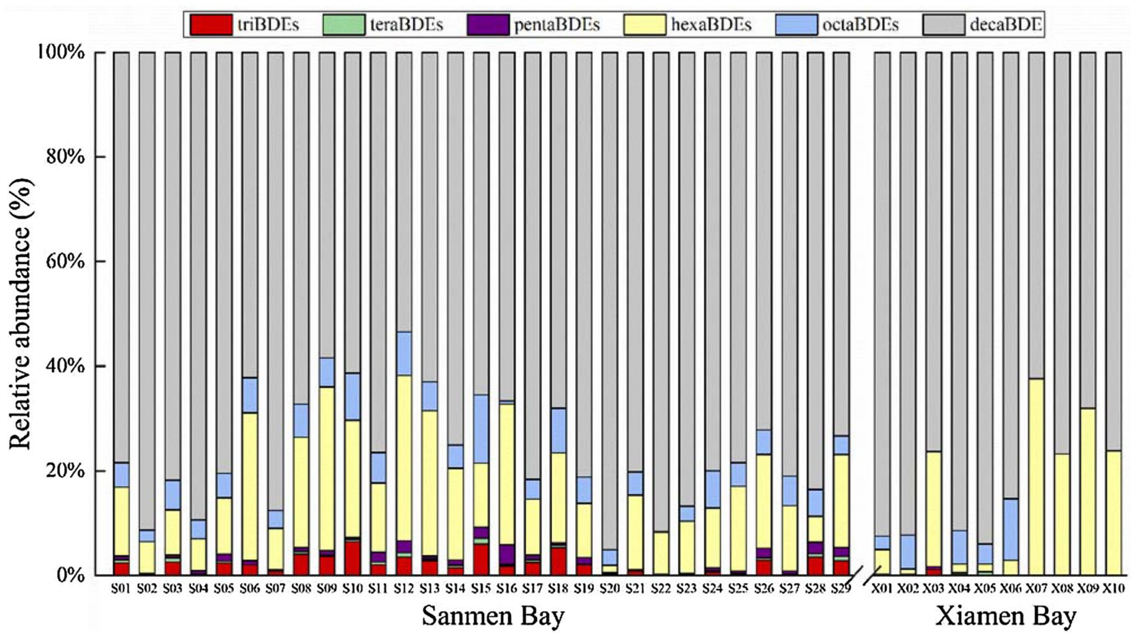

Fig. 1 Composition profile of PBDE congeners in surface sediment from the Sanmen Bay and Xiamen Bay, China. TriBDE homologues: BDE-17 and -28, teraBDEs: BDE-47, -49, -66, and -85; pentaBDEs: BDE-99, -100; hexaBDEs: BDE-138, -153, and -154; octaBDEs: BDE-183, -196, -197, -201, -202, -203, -206, -207, and -208; decaBDE: BDE-209

data (https://tjj.xm.gov.cn/), human populations in SMB and XMB were recorded as 1.62 and 3.86 million residents, respectively, while the gross domestic product (GDP) of SMB was also lower than that of XMB (i.e., 124.4 vs 346.6 billion Yuan in 2015). Greater intensities of anthropogenic activities have consequently resulted in elevated contamination in XMB.

The composition profile of PBDE congeners in surface sediment from SMB was dominated by decaBDE (range 53.4%–95.1%; mean 76.2%), followed by hexaBDEs (range 1.3%–15.1%; mean 31.6%) and octaBDE congeners (range 0.1%–13.1%; mean 5.1%) (Fig. 1). A similar pattern was observed for the XMB sediment. As the major congener of Deca-BDE mixtures, BDE-209 was found to be predominant (over 65%) in most of the sampling sites, with the exception of S06, S09, S12 and S13 in SMB and X07 in XMB (Fig. 2). BDE-209 concentrations in SMB and XMB sediment ranged from 1.2 to 74.6 ng/g dw (median 3.2 ng/g dw) and 4.9–254.7 ng/g dw (median 38.7 ng/g dw), respectively. Similar patterns were also observed in sediment studies from other bays, such as Jiaozhou Bay (Ju et al. 2016), Sanggou Bay (Yuan et al. 2016), Daya Bay (Liu et al. 2014) and San Francisco Bay (Klosterhaus et al. 2012). This pattern (i.e., dominance of BDE-209) reflects the greater use of Deca-BDE than other commercial PBDE mixtures worldwide, particularly in China (Mai et al. 2005; Chen et al. 2007). In addition, BDE-209 has a higher octanol–water partition coefficient (log $Kow = 9.87$) than other PBDE congeners (Wang et al. 2008; Bao et al. 2011). Hence, BDE-209 is more preferentially associated with organic carbon in sediment particles.

The existence of congeners from pentaBDE to octaBDE homologues indicate the contribution of other technical PBDE mixtures (i.e., Penta-BDE and Octa-BDE) (Alaee et al. 2003). For example, BDE-99 and BDE-183 constitutes a major component of Penta-BDE and Octa-BDE mixtures, while BDE-209 exists in Deca-BDE. Some of these lower brominated congeners could be transformed from BDE-209 and other higher brominated congeners. Microbial transformation of BDE-209 has been suggested to occur in sediment environments (Gerecke et al. 2005). In addition, some of the lower brominated PBDE congeners might also be contributed by long-range transport via hydrodynamic forces and atmosphere (Mizukawa et al. 2013). Ma et al. (2017) has indicated long-range transport of BDE-17, BDE-28 and BDE-47 in the Tibetan plateau, China.

Spatial distributions of ∑PBDEs and BDE-209 alone in SMB are shown in Fig. 2a and b. The highest concentrations of ∑PBDEs and BDE-209 were both observed at site S20 near Xiangshan County, with the values of 87.7 and 74.6 ng/g dw, respectively. Xiangshan County is located in the north of Ningbo City, which is one of the largest metropolitan areas of Zhejiang Province. Additionally, near site S20 there exist multiple municipal and industrial wastewater treatment facilities (Yao and Huang 2015). Thus, urban runoff and wastewater discharges may contribute to elevated

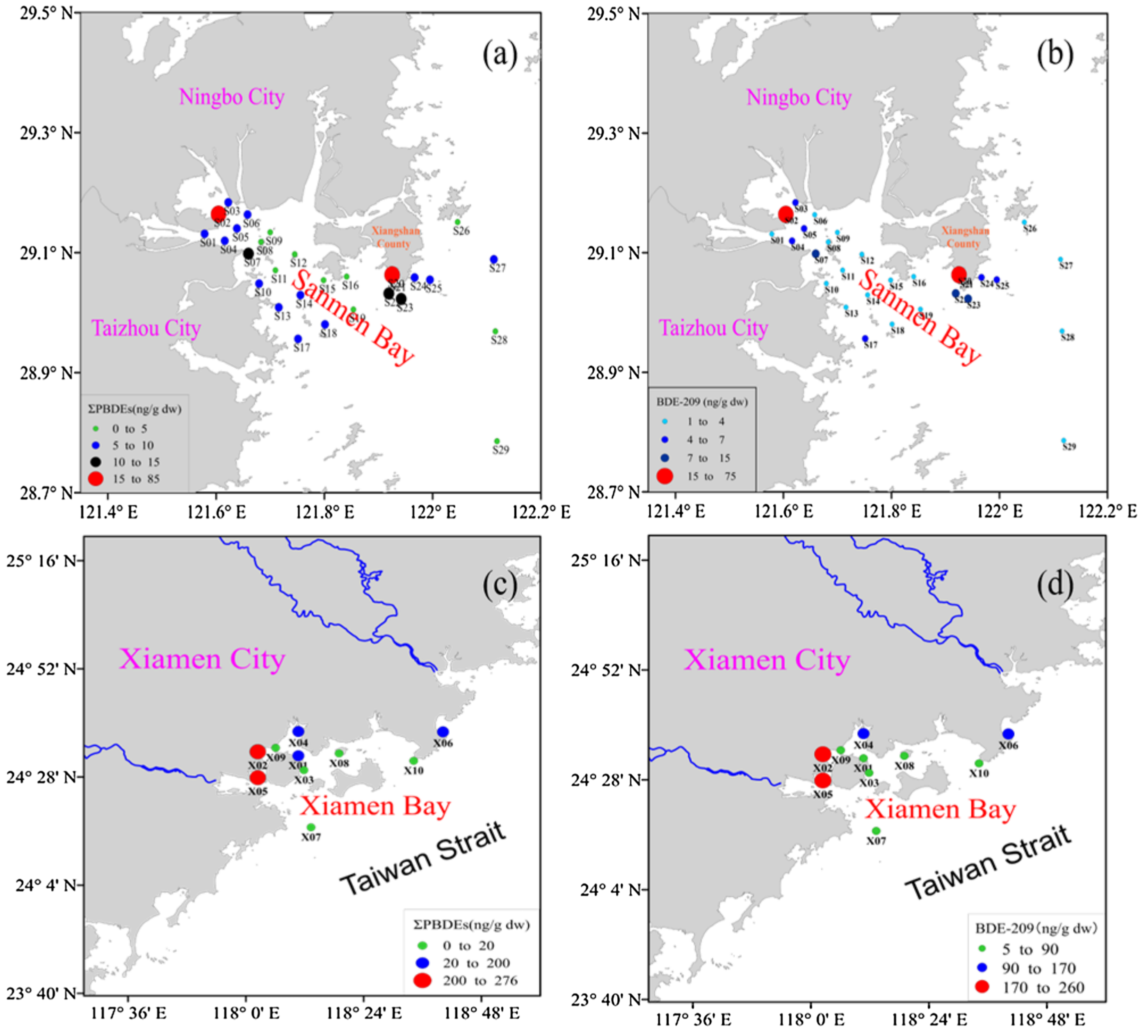

Fig. 2 Spatial distributions of ∑PBDEs and BDE-209 in surface sediments from the Sanmen Bay (**a**, **b**) and Xiamen Bay (**c**, **d**), China

sediment PBDE levels in neighboring water. As a consequence, the land-based contaminants, such as chemical oxygen demand (COD), total nitrogen (TN), and total phosphorus (TP), in Xiangshan County were also significantly higher than those from other counties across the Sanmen Bay basin (Yao and Huang 2015), reflecting a strong urban influence. Previous studies have also demonstrated the strong impact of urban runoff and wastewater discharges on coastal waters with respect to PBDEs (Rule et al. 2006; Pei et al. 2018).

Elevated concentrations of ∑PBDEs were also observed at site S02 (21.4 ng/g dw, respectively) near the Taizhou region, one of the largest electronic waste (e-waste) recycling and dismantling areas in the world. Given that PBDEs have been extensively used in thermoplastics, polyurethane foams, and electronic products, recycling and dismantling of e-waste with primitive approaches have resulted in the release of large quantities of PBDEs to local environments (Yang et al. 2011; Liang et al. 2016). As a result, great PBDE contamination has been reported in the air, soil, sediment, and biota from the Taizhou region, demonstrating the influence of extensive e-waste recycling activities on environmental qualities (Han et al. 2009; Ju et al. 2016; Zhou et al. 2017).

Although the number of investigated sites is limited, available data also exhibited a clear nearshore-offshore decreasing trend in ∑PBDEs or BDE-209 concentrations (Fig. 2c, d), demonstrating a strong urban influence on PBDE contamination in these coastal areas.

A large quantity of studies has investigated PBDE contamination in coastal and estuary regions in China and other countries. Residues of ∑PBDEs in SMB and XMB sediment are generally within the range of concentrations reported in sediment from China, e.g., 1.4–10.5 ng/g dw in the Sangou Bay (Wang et al. 2017), 2.2–10.6 ng/g dw in the Jiaozhou Bay (Ju et al. 2016), 0.23–214 ng/g dw in the Huangpu River (Wang et al. 2015b), 3.8–347 ng/g dw in the Taihu Lake

(Wang et al. 2016b), 9.7–151.3 ng/g dw in the East Lake (Yun et al. 2015), 0.93–105 ng/g dw in the Yanshuei River, Taiwan (Chen et al. 2013), 1.7–53.6 ng/g dw in Hong Kong coastal areas (Liu et al. 2005), 2.0–6.5 ng/g dw in the Yellow River Estuary (Yuan et al. 2016), and 0.13 to 1.98 ng/g dw in the Daliao River Estuary (Zhao et al. 2011).

In the lack of Chinese sediment quality guidelines for PBDEs, the Federal Environmental Quality Guidelines (FEQGs) established by Environmental Canada were used for evaluating sediment quality in our studied regions. They are 44, 39, 0.4, 440, 5600 and 19 ng/g dw for triBDEs, tetraBDEs, pentaBDEs, hexaBDEs, octaBDEs and decaBDE homologues, respectively (Environment Canada 2013; Wang et al. 2015a). Despite that the levels of most homologues were generally 1–3 orders of magnitude lower than the FEQG values, the median BDE-209 concentrations in XMB sediment surpassed that for decaBDE. Some individual sites in SMB also contained BDE-209 concentrations greater than the FEQG value. This indicates that the contamination of PBDEs, particularly BDE-209, could still possibly exert adverse impacts on the benthos at some sites of the two investigated bays.

In summary, our results demonstrate wide distributions of PBDEs in two of the economically important coastal areas in Southeast China and strong urban influences on the contamination levels. Although the current levels unlikely lead to any significant ecological risk to the benthos and aquatic ecosystems of the studied regions, continuous monitoring is needed to investigate the temporal trends of contamination in the important coastal waters and whether sediment-associated PBDEs constitute a potential source to aquatic ecosystems.

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