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Occurrence and distribution of perfluoroalkyl substances (PFASs) in sediments of the Dalian Bay, China

Guanghui Ding[a,*], Huanhuan Xue[a], Jing Zhang[b,*], Fuxu Cui[b], Xin He[c]

[a] *College of Environmental Science and Engineering, Dalian Maritime University, Linghai Road 1, Dalian 116026, PR China*
[b] *College of Environment and Chemical Technology, Dalian University, Dalian 116622, PR China*
[c] *Key Laboratory of Industrial Ecology and Environmental Engineering (Ministry of Education), School of Environmental Science and Technology, Dalian University of Technology, Dalian 116024, PR China*

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ABSTRACT

In the present study, the spatial distributions and vertical variations of PFASs in the sediments of the Dalian Bay were investigated. The total concentrations of PFASs (ΣPFASs) in surface sediments of Dalian Bay ranged from 1.49 to 2.66 ng g^{-1} dw. The predominant PFASs in surface sediments were PFOA, PFBS and PFBA with the concentration ranges of 0.50 to 0.95, 0.21 to 0.94, and 0.035 to 0.32 ng g^{-1} dw, respectively. For the two sediment cores, ΣPFASs ranged from 1.20 to 2.00 and from 1.37 to 2.06 ng g^{-1} dw, respectively. There was a maximum ΣPFASs concentration value near the surface. In general, ΣPFASs gradually decreased with increasing sampling depth after reaching the maximum. PFOA, PFBS and PFBA were also the predominant PFASs in the two sediment cores. It is recommended that the monitoring and risk assessment of short-chain PFAS should be taken into consideration, and further researches are needed to understand the transportation and fate of these chemicals in the aquatic environment.

Perfluoroalkyl substances (PFASs) are a large class of man-made chemicals, in which all of the hydrogen atoms on the carbon chain are replaced by fluorine atoms (Buck et al., 2011). The enduring properties and hydrophobic/lipophobic characteristics render PFASs highly useful in industrial and commercial applications. Since 1950s, PFASs have been widely used as protectants for textiles, stain repellents, personal care products, pesticides, fire-fighting foams, emulsifying agents, etc. (Zushi et al., 2012). Thus, PFASs have been detected ubiquitously in the environment, biota and humans and have been recognized as emerging environmental pollutants (Fromme et al., 2009; Giesy and Kannan, 2002; Houde et al., 2006, 2011; Lau et al., 2007; Loi et al., 2013). In addition, toxicology studies have confirmed that PFASs can not only produce toxic effects to plants and animals, but also bio-accumulate into higher trophic level organisms through the food chain, endangering ecological safety and human health (Ding and Peijnenburg, 2013; Houde et al., 2011). Therefore, PFSAs, including perfluoroalkyl carboxylic acid (PFCAs) and perfluoroalkane sulfonic acids (PFSAs), are of great concern (Conder et al., 2008; Fromme et al., 2009; Giesy and Kannan, 2002; Houde et al., 2006, 2011; Lau et al., 2007).

Numerous studies have already investigated the occurrence and distribution of PFASs in the aquatic environment, mainly focusing on the water phase (Cai et al., 2012; Chen et al., 2016; Shao et al., 2016). Sediment is also a natural part of the aquatic environment. However, there are limited researches on the contamination of PFASs in sediments and sediment cores (Ahrens et al., 2009; Bao et al., 2010; Gao et al., 2014). It has been believed that sediment is an important sink and potential secondary source of persistent organic pollutants, and has a large impact on their distribution, transportation, and fate in the aquatic environment (Ahrens et al., 2009; Prevedouros et al., 2006; Yang et al., 2011). Therefore, it is necessary to investigate the distribution of PFASs in sediments. Dalian Bay, located in the southernmost tip of the Liaodong Peninsula, is a typical semi-enclosed bay. The water exchange capability is poor under the influence of Sanshan Island. Along the coast, there are a large number of industrial enterprises and residential areas, which discharge an amount of industrial wastewater and domestic sewage into the bay. Therefore, the Dalian Bay has been seriously polluted (Na et al., 2013; Wang et al., 2011; Zhen et al., 2016). In the present study, surface sediments and sediment cores were collected and analyzed for PFASs, in order to explore the occurrence and distribution of PFASs in sediments of the Dalian Bay.

In July 2015, 13 surface (0–5 cm) sediments and 2 sediment cores (0–18 cm at site 3 and site 8) were collected from the Dalian Bay (Fig.1). The sediment cores were sectioned onboard into 2 cm intervals using a stainless steel cutter. A total of 9 core slices were obtained for

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* Corresponding authors.
E-mail addresses: guanghuiding@dlmu.edu.cn (G. Ding), zhangjing@dlu.edu.cn (J. Zhang).

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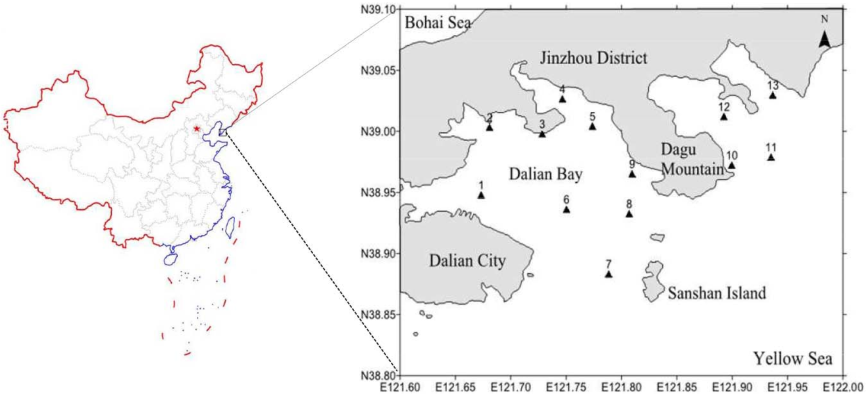

Fig. 1. Sampling sites of sediments in the Dalian Bay.

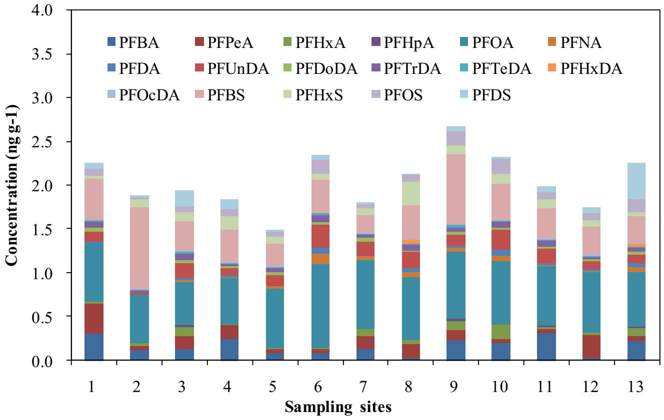

Fig. 2. Concentrations of PFASs in surface sediments of the Dalian Bay.

each sediment core. All sediment samples were stored frozen at − 20 °C until analysis.

The sediments were freeze-dried for 24 h and subsequently ground before extraction. Extraction of PFASs from sediments samples was accomplished using a previously published method (Li et al., 2011) with some modification. Briefly, 2 g of homogenized sediment was weighed into a 50-mL polypropylene tube with 5 ng internal standards spiked. Subsequently 5 mL of methanol was added as extracting agent. The mixture was vortexed for 1 min, mixed homogeneously by ultrasonic mixing for 30 min at 35 °C and then centrifuged for 10 min at 4000 rpm (RCF = 2504 × g). The supernatant was transferred into a new 15 mL centrifuge tube. This process was repeated twice. The total volume of the combined supernatant was 15 mL, which was then reduced to 1 mL under a gentle stream of high purity nitrogen and diluted to 50 mL with Milli-Q water. Solid phase extraction (SPE) was used for sample cleanup. The Oasis WAX SPE cartridges were preconditioned by passage of 4 mL of 0.1% NH_4OH in methanol followed by 4 mL of methanol and 4 mL of Milli-Q water. The diluted supernatant was loaded on the preconditioned cartridges at a rate of 2 mL min^{-1}. The cartridges were then washed with 4 mL of 25 mM ammonium acetate buffer at pH 4. The cartridges were dried by centrifuging for 10 min to remove moisture. Elution was performed using 4 mL of methanol and 4 mL of 0.1% NH_4OH in methanol in sequence. The eluate was then concentrated to 1 mL under a gentle stream of high purity nitrogen for instrumental analysis.

The target PFASs in the extracts were analyzed by an Agilent 1200 HPLC coupled with an Agilent 6410 Triple Quadrupole (QQQ) mass spectrometer (Agilent Technologies, Santa Clara, CA, USA) that was operated under the electrospray negative ionization (ESI) mode. An aliquot of 10 μL extract was injected into a ZORBAX Eclipse Plus C18 column (2.1 mm i.d. × 100 mm length, 3.5 μm; Agilent, USA), with 10 mM ammonium acetate aqueous solution (solvent A) and methanol (solvent B) as the mobile phases at a flow rate of 0.25 mL min^{-1}. A gradient program was given as follows: 40% B at 0 min, increased to 90% B at 20 min, continuously increased to 100% B at 30 min, and then reverted to 40% B at 38 min. The nebulizer pressure was set to 38 psi. The capillary voltage was 4000 V. The flow rate and temperature of the sheath gas (nitrogen) were 8 L min^{-1} and 350 °C, respectively. Multiple reactions monitoring analysis was employed to identify analytes.

To ensure the reliability of the data, field blanks, solvent blanks, procedure blanks, and duplicate samples were analyzed. The concentrations of extracts were quantified via eight-point matrix-matched calibration curves drawn by external standards in the range of 0.5–64 ng mL^{-1}. The coefficients of determination (r^2) of calibration curves for target analytes were all higher than 0.996. The limit of detection (LOD) ranged from 0.002 to 0.088 ng g^{-1} and the limit of quantification (LOQ) ranged from 0.008 to 0.293 ng g^{-1} dry weights (dw). The recoveries of target PFASs ranged from 82% to 144%.

All the analytical results lower than LOD were reported as n.d. (not detected) and zero was assigned for statistical analysis, while those lower than LOQ were reported as < LOQ, and calculated as half of the LOQ. Statistical analyses were performed with IBM SPSS 20.0 (SPSS Incorporation, Chicago, IL, USA).

The total concentrations of PFASs (ΣPFASs) in surface sediments of Dalian Bay ranged from 1.49 to 2.66 ng g^{-1} dw (Fig. 2). Among the 17 target PFASs, PFHxDA and PFOcDA had lower detect rates of 15% and 23%, respectively. Other PFASs were detected in all samples. The predominant PFASs in surface sediments were PFOA, PFBS and PFBA with the concentration ranges of 0.50 to 0.95, 0.21 to 0.94, and 0.035 to 0.32 ng g^{-1} dw, respectively. On average, the contributions of PFOA, PFBS and PFBA to the total concentrations of PFASs in sediment samples were 33.4%, 20.7% and 7.95%, respectively. This indicated that PFASs were ubiquitous in the surface sediments of the Dalian Bay, and short-chain PFASs were detected with high concentrations in the sediment. Recently, the C4 and C6 PFASs have been produced and used to replace most applications previously met by C8 and greater chain-length homologues (Ritter, 2010; Wang et al., 2013). The contamination of short-chain PFASs will continue. Therefore, it is recommended that the monitoring and risk assessment of short-chain PFASs should be taken into consideration, and further research is needed to understand the transportation and fate of these chemicals in the aquatic environment.

The contamination level of PFASs in the Dalian Bay is comparable to the reported range of below detection limit (< LOD) to 2.78 ng g^{-1} dw based on 166 surface sediments collected in 2011 and 2012 from the Bohai Sea, Yellow Sea and East China Sea (Gao et al., 2014). In another study on the surface sediments from the Bohai Sea, ΣPFASs ranged from 0.50 to 2.78 ng g^{-1} dw and from 0.33 to 1.75 ng g^{-1} dw in July and November 2013, respectively (Chen et al., 2016). As for Laizhou Bay, ΣPFASs in coastal sediments varied from 0.12 to 2.1 ng g^{-1} with a mean of 0.76 ng g^{-1} dw, which were much lower than the

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