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# The Investigation of Perfluoroalkyl Substances in Seasonal Freeze–Thaw Rivers During Spring Flood Period: A Case Study in Songhua River and Yalu River, China

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## Abstract

The occurrence of 15 perfluoroalkyl substances (PFASs) in water, sediments, and fish from Songhua River and Yalu River in Jilin Province, Northeast China, during their spring flood period were investigated for the first time. The short-chain perfluorobutanoic acid was the dominant PFAS in the water with the mean concentrations of 10 ng $L^{-1}$ in Songhua River and 9.0 ng $L^{-1}$ in Yalu River, and also dominant in sediments with the mean concentrations of 2.0 and 1.8 ng $g^{-1}$ dry weight. Perfluorodecane sulfonate was the predominant compound in fish. The detected frequencies and concentrations of perfluorooctane sulfonate and perfluorooctanoic acid were very low in the water, sediments, and fish. Low detected frequencies and concentrations of long-chain PFASs led to relative low sediment–water partition coefficients and bioaccumulation factors. The concentration levels of PFASs in the two rivers were lower than those in other major rivers in China.

**Keywords** Occurrence · Partition · Bioaccumulation · Northeast China · PFOS · PFOA

Perfluoroalkyl substances (PFASs) including perfluorosulfonates, perfluoroalkyl carboxylic acids, and their precursors are a new class of emerging environmentally persistent organic pollutants (Lam et al. 2017; Buck et al. 2011). Actions have been taken to regulate the manufacture and the use of related productions containing PFASs since 2000 (Xu et al. 2014). However, these compounds are still detected ubiquitously in the environment, even in the Arctic, Antarctic, and Tibetan Plateau, due to their stability (Yang et al. 2011). Perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA) are particularly known to cause potential adverse health effects on animals and human beings (Gronnestad et al. 2017).

In China, the occurrence of PFASs has been investigated in major aquatic systems including Yangtze River, Pearl River, Huaihe River, and Haihe River (Han and Currell 2017; Liu et al. 2017). However, the studies on the occurrence of PFASs in important rivers in Northeast China were scarce, such as Songhua River and Yalu River. Rivers located in mid-high latitude regions ware commonly seasonal freezing and thawing. Their hydrological characteristics obviously differ from other ice-free rivers, for example, they have two flood periods separately in spring and summer.

Songhua River, the third-largest river in China, supplies irrigation water for one of the most important grain production bases, and the river basin is one of traditional heavy-industry bases in China (Dong et al. 2016). Yalu River, located at the border of China and North Korea, originates from the same source with Songhua River and is a typical medium-sized regional river. Their hydrological characteristics are obviously different from other major rivers in China. In this study, water, sediment, and fish samples were collected from Songhua River and Yalu River during a spring flood period. This work will provide basic data for PFAS risk assessment and future pollution control in this region.

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## Materials and Methods

Fifteen PFASs (> 98% purity) including perfluorobutane sulfonate (PFBS), perfluorohexane sulfonate (PFHxS), PFOS, perfluorodecane sulfonate (PFDS), perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), PFOA, perfluorononanoic acid (PFNA), perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUdA), perfluorododecanoic acid (PFDoA), perfluorotridecanoic acid (PFTrA), and perfluorotetradecanoic acid (PFTA) were purchased from Dr. Ehrenstorfer GmbH (Augsburg, Germany).

Water and sediment samples were collected from Songhua River and Yalu River (Fig. 1) in early April of 2017. Six sites were distributed along the Songhua River (SH1 to SH6) and six sites were along Yalu River (YL1 to YL6). Overlying water (0.5 m depth) and surface sediment (top 10 cm) samples were collected from each sampling site. Crucian carps (*Carassius auratus*) were simultaneously collected with the water sampling in Songhua River (n = 10) and Yalu River (n = 6). Fish were sacrificed by transecting the spinal cord at the moment of the capture and fish muscles were cut by scalpel pre-cleaned with methanol and Milli-Q water.

Water samples were filtered through a glass fabric filter (0.45 µm) before extraction. An aliquot of 500 mL of the water sample was spiked with 5 ng of $^{13}C_4$-PFOS and $^{13}C_4$-PFOA, and then extracted with Oasis Wax cartridge. The elute was then concentrated to 1 mL for injection. Sediments were freeze-dried, triturated with a pestle in a mortar, and weighed (dry weight, dw). The total organic carbon (TOC) content of sediments were measured by a TOC analyzer (TOC-L, Shimadzu, Japan). The TOC fraction ($f_{oc}$) in sediments from Songhua River ranged from 0.50 to 1.9% and that from Yalu River ranged from 0.39% to 1.7%. Fish muscles were homogenized and weighed (wet weight, ww). Sediments and fish muscle samples were extracted by an ion-pair method (Hansen et al. 2001). Briefly, 1.0 g dw of sediments or 0.5–1.0 g ww of fish muscles were transferred into 50 mL screw-capped PP tubes and then spiked with 5 ng of $^{13}C_4$-PFOS and $^{13}C_4$-PFOA. 1 mL of 0.5 M tert-butyl alcohol (TBA), 2 mL of 0.25 M sodium carbonate solution, and 10 mL of methyl tert-butyl ether (MTBE) were added and thoroughly mixed and then extracted for 20 min at room temperature. For separating the organic and aqueous layers the suspension was centrifuged for 5 min at 10,000×*g* rpm and the supernatants were transferred into 15 mL screw-capped PP tubes. This step repeated for three times. The supernatants were combined and then dried at room temperature under a gentle stream of nitrogen and then reconstituted with 1 mL of methanol. The extract was further purified by the solid phase extraction.

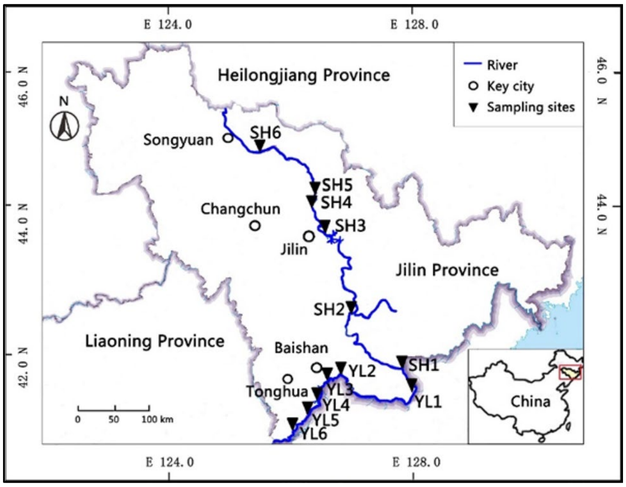

**Fig. 1** Sampling sites along Songhua River and Yalu River, Jilin Province, Northeast China. SH1: Tianchi Lake of Changbai Mountain; SH2: Erdaobai River; SH3: Jilin City; SH4: Downstream of Jilin City; SH5: Boundary between Jilin City and Changchun City; SH6: Dehui Town; YL1: Changbai Town; YL2: Linjiang Town; YL3: Baishan City; YL4: The first site nearby Tonghua City; YL5: The second site nearby Tonghua City; YL6: Ji'an Town

The analysis of PFASs was performed on a liquid chromatograph with tandem mass spectrometer system (LC–MS/MS; API4000, Applied Biosystems, US). The separation was achieved on a Dionex C18 column (2.1 × 150 mm, 3 µm particle size). An aliquot of 10 µL of extract was automatically injected and the oven temperature of LC was 25°C. Gradient conditions were used at 0.20 mL $min^{-1}$ flow rate. The identification of 15 PFASs in samples was accomplished with an electro spray ionization tandem mass spectrometer operated in negative ionization mode and chromatograms were recorded using Multiple Reaction Monitoring (MRM) mode. The instrument parameters were used as follows: ion spray voltage: − 3500 V; curtain gas: 45 Psi (0.31 MPa); nebulizer gas: 35 Psi (0.24 MPa); auxiliary gas: 40 Psi (0.28 MPa); collision gas: 6.0 Psi (0.15 MPa); source temperature: 550°C.

For the quality assurance and control, the recovery of each spiked sample, instrument detection limit (IDL), method quantification limit (MQL), field blank, matrix spike recovery, and duplicate samples were measured. The IDL and MQL were calculated based on a signal to noise ratio (S/N) of 3 and 10, respectively. All the analytical results lower than MQL were reported as n.d. (not detected) and zero was assigned for statistical purpose. For the water samples, MQL was at the 0.5 ng $L^{-1}$ level; for the sediment samples, MQL was 0.02 ng $g^{-1}$ dw; and for the fish muscle samples, MQL was 0.02 ng $g^{-1}$ ww. All PFAS compounds in blanks were well below the MQL. Recoveries for all PFASs were in the range of 66%–109%. The precisions of the entire method, represented by the relative standard deviation (RSD) of the spiked measurements, were in the range of 4.1%–15%.

**Table 1** Concentrations and frequencies of PFASs in water, sediment, and fish samples from Songhua River and Yalu River

| PFASs | Water (ng $L^{-1}$) | | | | | | Sediments (ng $g^{-1}$ dry weight) | | | | | | Fish (ng $g^{-1}$ wet weight) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Songhua River | | | Yalu River | | | Songhua River | | | Yalu River | | | Songhua River | | | Yalu River | | |
| | Mean | Range | Freq. (%) (n = 12) | Mean | Range | Freq. (%) (n = 12) | Mean | Range | Freq. (%) (n = 10) | Mean | Range | Freq. (%) (n = 8) | Mean | Range | Freq. (%) (n = 10) | Mean | Range | Freq. (%) (n = 6) |
| PFBS | 1.7 | 0.70–4.1 | 100 | 3.4 | 1.8–5.9 | 100 | 0.05 | n.d.–0.13 | 40 | 0.08 | n.d.–0.21 | 50 | 0.18 | 0.10–0.38 | 100 | 0.14 | 0.13–0.16 | 100 |
| PFHxS | 0.20 | n.d.–1.1 | 17 | n.d. | n.d. | 0 | n.d. | n.d. | 0 | n.d. | n.d. | 0 | 0.38 | n.d.–0.89 | 60 | 0.05 | n.d.–0.16 | 33 |
| PFOS | n.d. | n.d. | 0 | n.d. | n.d. | 0 | 0.02 | n.d.–0.08 | 20 | n.d. | n.d. | 0 | 0.10 | n.d.–0.22 | 80 | n.d. | n.d. | 0 |
| PFDS | n.d. | n.d. | 0 | n.d. | n.d. | 0 | n.d. | n.d. | 0 | n.d. | n.d. | 0 | 2.4 | 0.21–6.2 | 100 | 3.1 | 2.3–3.9 | 100 |
| PFBA | 10 | n.d.–22 | 50 | 9.0 | n.d. | 67 | 2.0 | 1.7–2.3 | 100 | 1.8 | 1.6–1.9 | 100 | 0.73 | 0.30–2.1 | 100 | 0.40 | 0.20–0.60 | 100 |
| PFPeA | 2.6 | 0.60–5.0 | 100 | 1.4 | 0.70–1.9 | 100 | 0.43 | 0.14–1.4 | 100 | 0.22 | 0.15–0.30 | 100 | 0.41 | 0.18–1.2 | 100 | 0.13 | 0.12–0.14 | 100 |
| PFHxA | 0.80 | n.d.–1.6 | 67 | 0.40 | n.d.–1.5 | 33 | 0.27 | 0.07–0.49 | 100 | 0.18 | 0.11–0.30 | 100 | 1.1 | n.d.–3.4 | 80 | 2.0 | 1.0–3.5 | 100 |
| PFHpA | 0.70 | n.d.–1.4 | 67 | 0.40 | n.d.–1.2 | 33 | 0.16 | n.d.–0.47 | 100 | 0.18 | 0.14–0.22 | 100 | 0.17 | 0.03–0.29 | 100 | n.d. | n.d. | 0 |
| PFOA | 0.40 | n.d.–1.7 | 33 | 0.70 | n.d.–2.2 | 50 | 0.40 | 0.10–0.87 | 100 | 0.17 | n.d.–0.45 | 50 | 0.29 | 0.03–0.42 | 100 | 0.38 | 0.20–0.53 | 100 |
| PFNA | n.d.. | n.d. | 0 | 0.10 | n.d.–0.80 | 17 | 0.15 | n.d.–0.48 | 60 | 0.08 | n.d.–0.30 | 25 | 0.61 | 0.11–1.2 | 100 | 0.08 | n.d.–0.18 | 67 |
| PFDA | n.d. | n.d. | 0 | n.d. | n.d. | 0 | n.d. | n.d. | 0 | 0.04 | n.d.–0.14 | 25 | 0.39 | 0.15–0.59 | 100 | 0.20 | 0.15–0.30 | 100 |
| PFUdA | n.d. | n.d. | 0 | n.d. | n.d. | 0 | n.d. | n.d.–0.12 | 20 | 0.03 | n.d.–0.12 | 25 | 1.3 | 0.40–1.9 | 100 | 0.50 | 0.29–0.83 | 100 |
| PFDoA | n.d. | n.d. | 0 | n.d. | n.d. | 0 | 0.03 | n.d.–0.17 | 20 | 0.10 | n.d.–0.28 | 50 | 0.98 | 0.39–1.3 | 100 | 0.45 | 0.30–0.66 | 100 |
| PFTrA | 0.10 | n.d.–0.60 | 17 | 0.20 | n.d.–0.60 | 33 | 0.03 | n.d.–0.14 | 20 | n.d. | n.d. | 0 | 0.89 | 0.46–1.1 | 100 | 0.46 | 0.35–0.62 | 100 |
| PFTA | n.d. | n.d. | 0 | n.d. | n.d. | 0 | n.d. | n.d. | 0 | n.d. | n.d. | 0 | 0.28 | 0.22–0.36 | 100 | 0.22 | 0.21–0.23 | 100 |
| ΣPFASs | 17 | 6.4–32 | | 16 | 6.3–28 | | 3.5 | 2.0–5.2 | | 2.8 | 2.4–3.6 | | 10 | 6.8–18 | | 8.0 | 6.4–9.3 | |

*n.d.* not detected, *Freq.* frequency

The site-specific partition of PFASs between sediments and water can be described by the partition coefficient $K_d$ (L kg$^{-1}$).

$$K_d = C_{si}/C_{wi} \tag{1}$$

where $C_{si}$ is the concentration of PFASs in sediments at sampling site $i$ (ng kg$^{-1}$ dw), and $C_{wi}$ is the concentration of PFASs in water at sampling site $i$ (ng L$^{-1}$).

It has been shown that PFAS sorption onto sediments strongly correlated with the sediment organic carbon fraction ($f_{oc}$) (Higgins and Luthy 2006). Thus, site-specific organic carbon normalized partition coefficient $K_{oc}$ values were also calculated as follows:

$$K_{oc} = K_d \times 100/f_{oc} \tag{2}$$

Experimental bioaccumulation factors (BAF, L kg$^{-1}$) were calculated as follows:

$$\mathrm{BAF} = C_f/C_w \tag{3}$$

where $C_f$ is the concentration of PFASs in fish muscles (ng kg$^{-1}$ ww) and $C_w$ is the concentration of PFASs in water samples (ng L$^{-1}$).

## Results and Discussion

Only 8 out of 15 PFASs were detected in the water samples from the Songhua River and Yalu River, as shown in Table 1. The detected PFASs composition profiles were similar between the two rivers. Short-chain PFASs including PFBA, PFBS and PFPeA were dominant compounds as shown in Fig. 2. It was found that short-chain PFASs were exclusively detected in the water samples because products containing short-chain compounds were more produced and consumed (Lorenzo et al. 2016). For example, the production of *N*-ethyl perfluorobutane sulfonamide and *N*-methyl perfluorobutane sulfonamidoethanol, which had been indicated as precursors of PFBS, PFBA and related short-chain PFASs detected in environment, was increasing year by year (Martin et al. 2006). In addition, the solubility of short-chain PFASs is higher than long-chain ones. This could explain why short-chain PFASs were dominant in water samples.

The average total concentrations of PFASs in Songhua River (17 ng L$^{-1}$) and Yalu River (16 ng L$^{-1}$) were similar and lower than those in other major rivers in China including the main stream of Yangtze River (14–32 ng L$^{-1}$), Pearl River (19 ± 12 ng L$^{-1}$), Huaihe River (28 ng L$^{-1}$), Liaohe River (44 ng L$^{-1}$), Haihe River (12–74 ng L$^{-1}$), Grand Canal (45 ± 47 ng L$^{-1}$) and Huangpu River (226 ng L$^{-1}$) (Yang et al. 2011; Zhang et al. 2013; Pan et al. 2014a; Yu et al. 2013; Piao et al. 2017; Sun et al. 2017). The mean concentrations of PFBA were the highest among the found PFASs, which were 10 ng L$^{-1}$ in Songhua River and 9.0 ng L$^{-1}$ in Yalu River. This agrees with the results in previous studies found that PFBA were dominant in water samples in surface waters (Myers et al. 2012; Lorenzo et al. 2016). Atmospheric deposition of PFBA is a possible explanation to these residuals levels (Eschauzier et al. 2013).

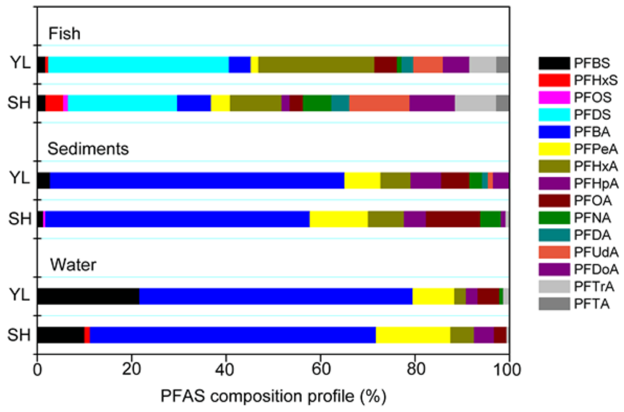

**Fig. 2** Average contribution of each compound to the total PFASs in water, sediments, and fish from Songhua River (SH) and Yalu River (YL)

Twelve out of 15 PFASs were detected in the sediments from Songhua River and Yalu River. The concentrations and composition profiles of PFASs in the sediments from the two rivers were also similar. PFBA was still the dominant PFAS with the average concentration of 2.0 ng g$^{-1}$ dw in Songhua River and 1.8 ng g$^{-1}$ dw in Yalu River. Those concentrations correlate better to those in water. Both PFOA and PFOS, two of the biggest concerns, were found in the sediments. But their concentrations were relative lower. The low concentration levels of PFOA and PFOS could indicate the recent changes in production and use of PFASs as the replacement of these compounds by short-chain ones (Lorenzo et al. 2016).

All the 15 PFASs were detected in the fish muscle samples from Songhua River. Except for PFOS and PFHpA, 13 PFASs were detected in the fish muscle samples from Yalu River. In contrast to water and sediment samples, the PFASs in the fish between the two rivers showed different composition profiles. More long-chain PFASs such as PFDS, PFUdA, PFDoA, and PFTrA were detected. PFDS was the most predominant PFAS with the contribution of 23% in Songhua River and of 38% in Yalu River. But like in the water and sediments, the concentration levels of PFOA and PFOS were still low in the fish muscle samples.

The site-specific $K_d$, log$K_d$ and log$K_{oc}$ at sediment–water and average BAF and logBAF in fish muscles for 9 PFASs including PFBS, PFHxS, PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, and PFTrA, which were found in both water and sediments, or both water and fish muscles were calculated (Table 2). Log$K_d$ showed mean values ranging from

**Table 2** Site-specific $K_d$, log$K_d$ and log$K_{oc}$ at sediment–water and average BAF and logBAF in fish muscles from Songhua River and Yalu River

| PFASs | Songhua River | | | | | Yalu River | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | $K_d$ (L kg$^{-1}$) | log$K_d$ | log$K_{oc}$ | BAF (L kg$^{-1}$) | logBAF | $K_d$ (L kg$^{-1}$) | log$K_d$ | log$K_{oc}$ | BAF (L kg$^{-1}$) | logBAF |
| PFBS | 64 (32–96, n = 2) | 1.8 (1.5–2.0) | 3.5 (3.2–3.7) | 106 | 2.0 | 66 (17–115, n = 2) | 1.8 (1.2–2.1) | 3.6 (3.0–3.8) | 41 | 1.6 |
| PFHxS | n.c. | n.c. | n.c. | 1900 | 3.3 | n.c. | n.c. | n.c. | n.c. | n.c. |
| PFOS | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. |
| PFDS | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. |
| PFBA | 96 (76–115, n = 2) | 2.0 (1.9–2.1) | 3.7 (3.6–3.8) | 73 | 1.9 | 105 (101–109, n = 2) | 2.0 | 3.8 | 41 | 1.6 |
| PFPeA | 181 (32–389, n = 5) | 2.3 (1.5–2.6) | 4.0 (3.2–4.3) | 158 | 2.2 | 167 (102–258, n = 4) | 2.2 (2.0–2.4) | 4.0 (3.8–4.2) | 93 | 2.0 |
| PFHxA | 176 (91–338, n = 3) | 2.2 (2.0–2.5) | 4.0 (3.7–4.3) | 1388 | 3.1 | 147 (108–185, n = 2) | 2.2 (2.0–2.3) | 3.9 (3.8–4.0) | 4950 | 3.7 |
| PFHpA | 126 (84–158, n = 3) | 2.1 (1.9–2.2) | 3.8 (3.6–3.9) | 243 | 2.4 | 162 (145–179, n = 2) | 2.2 (2.2–2.3) | 4.0 (3.9–4.0) | n.c. | n.c. |
| PFOA | 551 (n = 1) | 2.7 | 4.5 | 725 | 2.9 | 150 (n = 1) | 2.2 | 4.0 | 543 | 2.7 |
| PFNA | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | 800 | 2.9 |
| PFDA | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. |
| PFUdA | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. |
| PFDoA | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. |
| PFTrA | 246 (n = 1) | 2.4 | 4.1 | 8900 | 3.9 | n.c. | n.c. | n.c. | 2300 | 3.4 |
| PFTA | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. | n.c. |

*n.c.* not calculated because analyte was not detected in at least one environmental compartment. Results of $K_d$, log$K_d$ and log$K_{oc}$ are given as mean/range (min–max)

1.8 to 2.7 in Songhua River and from 1.8 to 2.2 in Yalu River. Log$K_{oc}$ in the Songhua River showed mean values in the range of 3.5–4.5 and in Yalu River in the range of 3.6–4.0. Both log$K_d$ and log$K_{oc}$ values were similar between the two rivers. Log$K_d$ and log$K_{oc}$ in the two rivers are at the same level of those in aquatic systems in the Netherlands, Tokyo Bay, and lower than those in Llobregat River and Jucar River, Spain (Ahrens et al. 2010; Campo et al. 2014, 2016; Kwadijk et al. 2010). Because log$K_d$ and log$K_{oc}$ values commonly tend to increase with the perfluorocarbon chain length (Pico et al. 2012), the slightly lower log$K_d$ and log$K_{oc}$ values in this study could be related to the low detected frequencies and concentrations of some long-chain PFASs. In the Songhua River, log BAFs ranged from 1.9 (PFBA) to 3.9 (PFTrA), and in Yalu River ranged from 1.6 (PFBS and PFBA) to 3.4 (PFTrA). The logBAF values were similar with the logBAF$_{muscle}$ for the fish collected from the Pearl River which ranged from 1.8 (PFNA) to 3.5 (PFUnDA) (Pan et al. 2014b). But the maximal logBAF values were less than 4.0, which were lower than those reported for fish samples in Taihu Lake (4.2), Baiyangdian Lake (4.2), LIobregate River (4.3) and Jucar River (6.4) (Xu et al. 2014; Campo et al. 2014, 2016; Zhou et al. 2012). These differences could be explained by site, species-specific behaviors, different dietary habits, or the different environmental conditions and concentrations (Campo et al. 2016; Pan et al. 2014b).

This study provided the first report of PFASs in Songhua River and Yalu River, Northeast China, during their spring flood period, which indicates the concentrations levels of PFASs, especially PFOS and PFOA in the two rivers are lower than those in other large rivers in China. But the investigation of PFASs has provided a conceivable proof for the existence of PFASs in the two rivers. Further research is still necessary to get better understanding of the potential risk and transformation mechanisms of PFASs in this region.

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