



## Research article

# Perfluoroalkyl and polyfluoroalkyl substances (PFAS) and nutrients from two constructed wetlands in a city of southeastern China

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## ABSTRACT

Perfluoroalkyl and polyfluoroalkyl substances (PFAS) are a large class of toxic contaminants. Nutrients are closely related to the ecological health of aquatic systems. Both have received widespread global attention. This study investigated the concentrations, compositions, and spatial distributions of PFAS and nutrients in surface water from two constructed wetlands and the nearby drinking water treatment plants (DWTPs). We explored the natural environmental factors and human activities that affect the composition and distribution of pollutants in wetlands and assessed the ability of the DWTPs to remove contaminants. Concentrations of  $\sum_{32}$ PFAS varied from 153 to 405 ng/L. Hexafluoropropylene oxide trimer acid (HFPO-TA) was the predominant substance accounting for 45 % of  $\sum_{32}$ PFAS concentrations. It might originate from the emissions of indirect sources of PFAS related manufacturers. The detection rate of 6:2 fluorotelomer carboxylic acid (6:2 FTCA) was 100 % with concentrations ranging from 0.915 to 19.7 ng/L. 6:2 FTCA might come from the biotransformation of indirect sources in the air. Concentrations of total nitrogen (TN) and total phosphorus (TP) were from 1.47 to 3.54 mg/L, and non-detect (ND) to 0.323 mg/L, respectively. Constructed wetlands could effectively remove PFAS under nutrient stress, however, the removal of PFAS depends on the characteristics of specific compounds and their sources. The removal rates for PFAS and nutrients could be promoted through artificial dredging. But wetland bioremediation could have two opposing effects. On the one hand, plants can take up pollutants from water via roots, leading to pollutant removal and purification. On the other hand, plants may also absorb precursor intermediates from the air through leaves and release them into the water, leading to increased pollutant concentrations. Thirty-two emerging PFAS were identified by high resolution mass spectrum. The drinking water treatment process removed PFAS and nutrients below the drinking water quality standards of China, however, 9 non-target PFAS compounds were still found in tap water. These results provide case support and a theoretical basis for the pollution control and sustainable development of typical ecological wetlands used as drinking water sources.

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## 1. Introduction

Perfluoroalkyl and polyfluoroalkyl substances (PFAS) are defined as fluorinated substances containing at least a perfluorinated methyl group ( $-\text{CF}_3$ ) or a perfluorinated methylene group ( $-\text{CF}_2-$ ) [1]. Due to their surfactant properties, and chemical stability [2], PFAS are widely used in domestic and industrial applications including electronic equipment, fire-fighting activities, food packaging, cleaning products, textile, and leather manufactures [3]. PFAS have been widely detected in various environmental media, including air [4], water [5–7], sediment [8,9], plants [10,11], fish [12], and human beings [13,14]. Due to their neurotoxicity, immunotoxicity, carcinogenicity, and endocrine disruption [15], regulation of the use of PFAS has begun worldwide. Perfluorooctane sulfonates (PFOS), perfluorooctanoic acid (PFOA), perfluorohexane sulfonate acid (PFHxS) and the salts of these three compounds were listed in Stockholm Convention on Persistent Organic Pollutants (POPs) in 2009, 2019, and 2022, respectively. In 2023, the European Chemicals Agency (ECHA) released a draft proposal for PFAS restrictions, stipulating that the concentration of any targeted PFAS substance in produced chemicals must not exceed 25 ppb, and the total concentration of targeted PFAS and degradable precursors should not exceed 250 ppb [16]. According to drinking water quality standards of China, the limits for PFOA and PFOS are 40 ng/L and 80 ng/L, respectively [17]. Despite the increasing attention paid to PFAS contamination in recent years, high concentrations of PFAS have been detected in the environment worldwide, for instance,  $84,000 \pm 2000$  ng/L of PFAS in surface water from Xiaoqing River, China [18], 8041 ng/L of PFAS in Bormida River water, Italy [19], and 598 ng/L of drinking water in the Rhine-Ruhr area, Germany [20].

As legacy PFAS are gradually out of use, different alternative PFAS, for instance, perfluorobutanoic acid (PFBA) and hexafluoropropylene oxide (HFPOs), were applied, and resulted in ubiquitous distribution [21]. Wang et al. reported that the concentration of PFBA reached 1350 ng/L in the Daling River near fluoropolymer facilities in northern China [22]. Chen et al. collected tap water samples from 48 cities in China, and found that PFBA was the dominant PFAS in tap water of eastern China, with concentration ranging from ND (not detected) to 56.8 ng/L [23]. Yao et al. reported hexafluoropropylene oxide trimer acid (HFPO-TA) in the fluorochemical zone surface water at the median concentration of 670 ng/L [24]. Pan et al. estimated the discharges of hexafluoropropylene oxide dimer acid (HFPO-DA) and HFPO-TA from five major Chinese rivers as 2.6 and 6.0 tons/year, respectively [25]. HFPOs were reported to be more toxic and bioaccumulative than PFOA [26]. Besides target PFAS, more emerging PFAS have been identified using suspect and non-target analysis in rivers [27], drinking water systems [28], and marine food web [29]. Therefore, the detection and quantification of emerging PFAS is specifically important.

Wetlands in cities could maintain ecological balance and biodiversity as well as removing pollutants like PFAS [30]. Constructed wetlands are used in treatment of wastewater, landfill leachate, and micro-polluted surface water to remove excessive nutrients and organic pollutants [31–33]. Savvidou et al. found that surface flow constructed wetlands and horizontal subsurface flow constructed wetlands could remove PFAS with median removal rates of 64 % and 46 %, respectively [34]. Yin et al. conducted a study on a constructed wetland system in Singapore designed for the treatment of landfill leachate, revealing high removal rates of individual PFAS ranging from 50 % to 96 % [35]. In addition to treating wastewater and landfill leachate, some constructed wetlands were designed to provide water sources for drinking water treatment plants (DWTPs), which may lead to human exposure to PFAS by drinking water [36–39]. However, only a few articles have reported on contaminants in constructed wetlands served as drinking water sources, and these studies have only included nutrients, polycyclic aromatic hydrocarbons (PAHs) [40], and antibiotic resistance genes (ARGs) [41]. There have been no reports on PFAS so far. The migration and transformation mechanisms of PFAS in wetlands may be influenced by traditional monitoring indicators (e.g., nutrients). In soil, ammonia nitrogen and nitrate nitrogen can promote soil acidification [42], and acidic soil enhances the accumulation of PFAS ( $\text{C}_4\text{-C}_{10}$ ) by plant roots [43]. Moreover, the concentration levels of ammonium salts and nitrate salts can influence the growth and development of plant lateral roots, and enhance the root absorption of PFAS [44,45]. Previous studies have indicated a positive relationship between the content of protein transporters and PFAS accumulation [46]. As essential elements to synthesize protein, nitrogen and phosphorus might play a key role.

In this study, PFAS and nutrients were quantified in two constructed wetlands in Jiaying City which served as drinking water sources. Jiaying is a typical plain river network city with low-lying flat terrain, poor water mobility, and the existence of groundwater-surface water exchange [47]. Due to the combined pollution from industrial, domestic, and agricultural sources, the water shortage in the river network area of Jiaying is complex, leading to widespread concerns about the safety of drinking water there. To protect the safety of drinking water, two constructed wetlands began operations in July 2008 (Shijiyang Wetland, SW) and October 2013 (Guanjingang Wetland, GW). SW is the first large-scale urban water source ecological wetland in China, setting a precedent for urban and rural water supply sources in China. On this basis, a larger-scale GW was built after further improvement and upgrading, thus SW and GW became the paradigms of ecological wetlands for urban drinking water sources in China [48]. Previous studies have focused on monitoring traditional pollutants (e.g., nutrients, heavy metals), and utilizing these indicators to assess drinking water safety. However, with the increasing complexity and diversity of environmental pollution, there remains a gap in research concerning emerging pollutants (e.g., PFAS) and their interactions with traditional pollutants in wetlands. To better understand the migration and transformation mechanisms of emerging pollutants in constructed wetland ecosystems and protect drinking water safety, it is urgent to strengthen monitoring and research on emerging pollutants. The aims of this study are as follows: (1) to reveal the concentration, composition, and spatial distribution of PFAS and nutrients in two wetlands and their nearby DWTPs; (2) to investigate the natural environmental and anthropogenic factors affecting the removal of pollutants from wetlands; (3) to identify emerging PFAS by suspect and non-target screening.

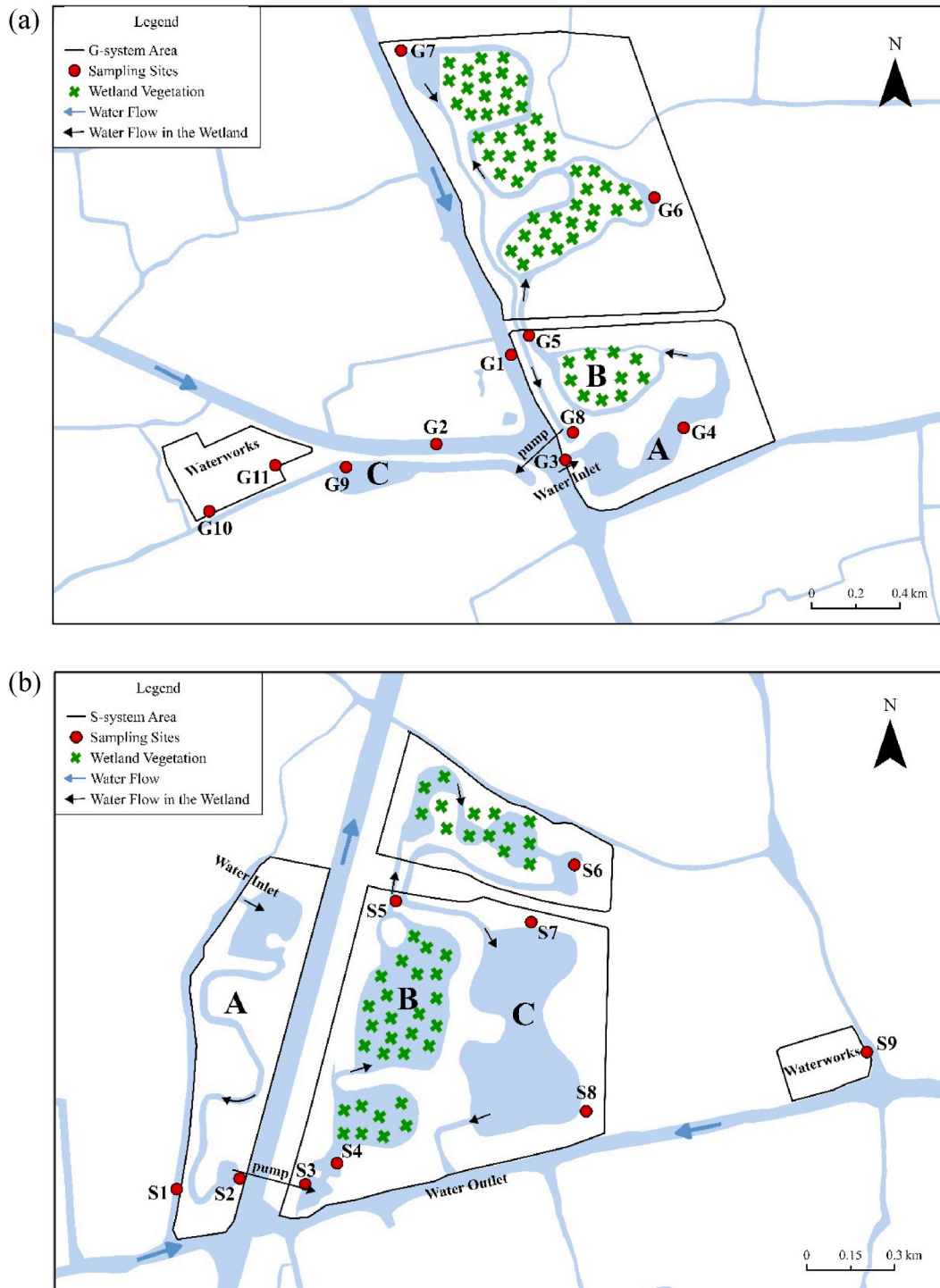


Fig. 1. Map of GW (a) and SW (b). A: pretreatment area, B: root-channels purifying area and C: deep purifying area.

## 2. Material and methods

### 2.1. Standards and chemicals

A total of 32 emerging and legacy PFAS were monitored as target compounds. Eleven mass-labeled analytes were used as the Internal Standards (IS) (Supplementary Table S2). 2H-perfluoro-(1,2-<sup>13</sup>C<sub>2</sub>)-2-decenoic acid (<sup>13</sup>C<sub>2</sub>-8:2 FTUCA) was used as an injection standard (InjS). Except for HFPO-TA which was purchased from Fluorochem (95 %, United Kingdom), native and internal standards were purchased from Wellington Laboratories (>98 %, Canada). Nutrient standard solutions were purchased from National Analysis and Testing Center for Nonferrous Metals & Electronic Materials (1000 µg/mL, China) (Supplementary Table S3). Ultrapure Millipore water was produced by a Milli-Q Direct-Q®3 UV (Merck, Germany). High-performance liquid chromatography (HPLC) grade methanol was purchased from Macklin (≥99.9 %, China), and ammonium hydroxide solution was acquired from CNW (25 %, HPLC, Germany).

### 2.2. Sample collection

The location of the two wetlands belongs to the East Asian monsoon region, with an average annual temperature of 15.9 °C. GW occupies a total area of 147 hm<sup>2</sup> and provides a daily water treatment capacity of 30 × 10<sup>4</sup> m<sup>3</sup>. Reeds (*Phragmites australis*) and cattails (*Typha orientalis*) are dominant plant species in GW. SW covers an area of 110 hm<sup>2</sup>, with a treatment capacity of 25 × 10<sup>4</sup> m<sup>3</sup>/d. It is mainly dominated by emergent plants such as reeds, cattails, alligator weed (*Alternanthera philoxeroides*), and floating plants such as water hyacinth (*Eichhornia crassipes*) [49].

Each wetland receives surface water from nearby rivers. Waters upstream of GW are subjected to pollution from urban and rural domestic sewage, livestock and poultry farming wastewater, and agricultural runoff [47,50,51]. Upstream of SW receives wastewater treatment plant effluents from the Xiuzhou Industrial Park including textiles, paper products, electronic goods, and chemical products. Both the wetlands are mainly composed of three different treatment processes: the pretreatment area (A), root-channels purifying area (B) and deep purifying area (C) (Fig. 1). Pretreatment area (A) is located at the front of the wetland, mainly removing particle matter from the water, improving transparency, and preventing the clogging of plant beds in the root-channels purifying area. Root-channels purifying area (B) simulates the natural reeds root channel system and utilizes artificially planted straw as a filling/medium, effectively modifying the macropore structure of the wetland soil subsurface layer. Area B intercepts pollutants through root-pore plant beds, increasing the interface of land/water ecotone and enhancing the efficiency of pollutant uptake and degradation by microorganisms and plants. Deep purifying area (C) is situated at the back end of the wetland, serving as a water storage function, and further reducing the turbidity of the water for extraction by the drinking water treatment plant [48]. After being purified by the wetlands, the water will finally flow into the DWTP (G-DWTP for GW and S-DWTP for SW).

Twenty surface water samples (depth: 0–0.5 m) were collected from 18 sampling sites in constructed wetlands (two duplicated samples at S4 and G4) on November 25, 2022. Another two samples were collected from the effluent outlet of G and S DWTPs (G11 and S9). Water samples were collected in 1-L polypropylene (PP) bottles (Nalgene, USA) that had been pre-cleaned with methanol and Milli-Q water, and rinsed by sample before collection. All samples were stored at –20 °C until treatment and analysis.

### 2.3. Sample nutrient determination

Dissolved inorganic nitrogen (DIN) including nitrate (NO<sub>3</sub><sup>-</sup>-N), nitrite (NO<sub>2</sub><sup>-</sup>-N) and ammonia nitrogen (NH<sub>4</sub><sup>+</sup>-N) were determined according to the standard water and wastewater monitoring and analysis method [52]. In brief, the hydrazine sulfate reduction method was used for NO<sub>3</sub><sup>-</sup>-N, the N-(1-naphthyl)-1,2-diaminoethane dihydrochloride spectrophotometry method was used for NO<sub>2</sub><sup>-</sup>-N and salicylic acid spectrophotometry method was used for NH<sub>4</sub><sup>+</sup>-N. Alkaline potassium persulfate digestion spectrophotometry method was used for total nitrogen (TN) [53], and ammonium molybdate spectrophotometry method was used for total phosphorus (TP) [54].

NO<sub>3</sub><sup>-</sup>-N, NO<sub>2</sub><sup>-</sup>-N, and NH<sub>4</sub><sup>+</sup>-N were detected by Automatic Discrete Analyzer Cleverchem 380 (DeChem-Tech, Germany). TN and TP were detected by Scanning Ultraviolet Spectrophotometer UV-3200 (MAPADA, China).

### 2.4. Sample preparation for PFAS

Water samples were first filtrated through pre-heated (450 °C for 4 h) 0.7 µm glass fibre filters (GFF, Ø47 mm, Whatman, UK), and separated into dissolved and particle phases. Filters were then washed with methanol to avoid potential loss of PFAS on the glass materials [55]. Dissolved phase was spiked by 2 ng (100 pg/µL, 20 µL) IS, and then solid phase extracted (SPE) using Oasis WAX cartridges (30 µm, 6 mL, 150 mg, Waters, USA) at a speed of 2 drops per second via gravity. Cartridges were pre-conditioned with 4 mL 0.2 % ammonium hydroxide in methanol, 4 mL methanol, and 4 mL Milli-Q water. Water loaded cartridges were washed with 15 mL Milli-Q water, and then dried by a pump. For elution, 10 mL 0.2 % ammonium hydroxide in methanol was used. The volume of eluent was reduced to 1 mL by a rotary evaporator (Hei-VAP Core, Germany) and nitrogen (>99.999 %). Then, Envi-Carb columns (1 mL, 100 mg, Supelco, Germany) were used for purification. Samples were concentrated at 200 µL and stored in PP vials at –20 °C before analysis.

For the particle phase samples, the filters were stored at –20 °C before treatment. After being freeze drying for 12 h, 2 ng IS was spiked on the filters. Then filters were soaked in 15 mL 0.2 % ammonium hydroxide in methanol in PP centrifuge tubes. All samples were sonicated for 30 min and then centrifuged at 4500 rpm for 15 min. After transferring the supernatant to a heart-shaped bottle (250 °C for 12 h), the extraction, sonication, and centrifugation were repeated. All the supernatants were concentrated to 1 mL under

nitrogen blowing. After purification by Envi-Carb columns, samples were condensed and stored in PP vials.

## 2.5. Instrumental analysis

### 2.5.1. Target analysis

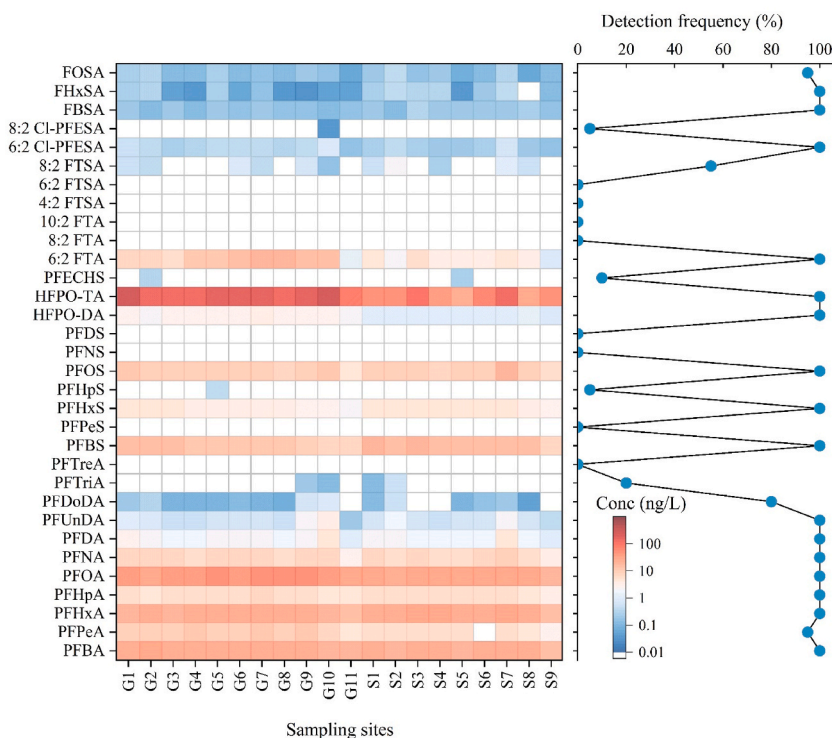
Quantitative analysis of 32 target PFAS and 11 corresponding IS was performed using ultra-performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS, LC-MS 8050, Shimadzu, Japan). Chromatographic separation was performed using a Waters ACQUITY UPLC® BEH C18 column (2.1 × 100 mm, 1.7 μm). The injection volume was 1 μL and the flow rate was 0.3 mL/min. The mobile phases consisted of Milli-Q water (A) and methanol (B). The eluent gradient started at 25 % B and was increased to 75 % B at 5 min. Then, 75 % B ramped to 100 % at 8 min, kept for 3 min, returned to the initial conditions, and held at this level for 3 min.

The electrospray ionization (ESI) source was operated in negative mode, and data acquisition was based on multiple reaction monitoring (MRM) mode. The capillary was held at 3500 V. Nebulizer, drying, and heating gas flows were kept at 3, 10, and 10 L/min, respectively. Desolvation temperature and Desolvation Line (DL) temperature were kept at 526 °C and 250 °C, respectively.

### 2.5.2. Suspect and non-target screening

Samples were analyzed by a Vanquish Flex ultrahigh-performance liquid chromatography (UHPLC) system (Thermo Fisher Scientific, U.S.) coupled with quadrupole orbitrap mass spectrometer (Orbitrap Exploris 240 MS, Thermo Fisher Scientific, U.S.). The injection volumes were 5 μL. The chromatographic column was an Agilent ZORBAX Eclipse Plus C18 column (Rapid Resolution HD 2.1 × 100 mm, 1.8 μm) and a guard Agilent UHPLC Guard 3 PK (ZORBAX Eclipse Plus C18 2.1 × 5 mm, 1.8 μm). The column compartments were maintained at 50 °C with a pump delivering 0.3 mL/min of the mobile phase. Data were acquired in data dependent acquisition (DDA) mode. Full scan and MS<sup>2</sup> scan in both positive and negative ionization modes were applied. The gradient elution and mass spectrometry parameters are shown in [Supplementary Tables S4–S5](#).

The screening criteria include: i) CF<sub>2</sub>-adjusted Kendrick mass defect (KMD) > 0.85 or < 0.15, ii) mass error < 5 ppm, iii) isotopic fit threshold > 80 %, iv) an ascending trend of retention time versus *m/z* of each homologue series [27,56]. Three referential compound lists include US EPA CompTox Chemistry Dashboard [57], Norman Suspect List Exchange [58] and Nist Suspect List of Possible Per- and Polyfluoroalkyl Substances (PFAS) [59]. The screening was operated using Compound Discoverer (CD) 3.3 SP2 (Thermo-Fisher Scientific, US). The detailed of CD workflow is shown in [Supplementary Fig. S2](#). Matched the MS/MS fragments in the samples and extracted potential PFAS peaks with ions. Confidence levels (CL) were assigned to the identified PFAS from level 5 to level 1 according to Charbonnet et al. [60]. Semi-quantification of non-target compounds was then calculated by TraceFinder 5.1 General Quan (Thermo-Fisher Scientific, US).



**Fig. 2.** Concentrations (ng/L) and detection frequency (%) of detected PFAS based on target analysis of 32 target PFAS in two wetlands.



## 2.6. Quality assurance and quality control (QA/QC)

An eight-point standard calibration curve was prepared for each analyte at concentrations of 0–200 pg/ $\mu$ L (correlation coefficients  $R^2 > 0.99$ ). Method validation results for water are presented in [Supplementary Table S6](#), and recoveries were within 36%–137%. During instrument injection, a standard of known concentration was run every 10 samples to monitor the stability and possible contamination of the instrument. The instrumental limit of detection (LOD) was defined using a signal-to-noise ratio (S/N) of 3, and the method quantification limit (MQL) by an S/N of 10. The concentrations below LOD were replaced as 1/2 LOD in the statistical calculation. Fluoropolymer materials were avoided during the whole experiment. Two field blanks and four procedural blanks were prepared. PFOA, 6:2 chlorinated polyfluorinated ether sulfonic acid (6:2 C1-PFESA) and perfluorohexane sulfonamide (FHxSA) were detected in procedural blanks. The concentrations of PFAS in samples were calculated by subtracting background levels. PFAS concentrations in the duplicated samples exhibited a deviation within  $\pm 23\%$ . The recoveries of IS ranged from 61% ( $^{13}\text{C}_2$ -PFDoDA) to 87% ( $^{13}\text{C}_4$ -PFOS) ([Supplementary Table S7](#)).

## 2.7. Data analysis methods

Removal rate was calculated using the following formula [61,62]:

$$\text{Removal rate} = (C_i - C_e) / C_i \times 100\% \quad (1)$$

where  $C_i$  and  $C_e$  denote the pollutant concentrations in the influent and effluent, respectively (Nutrient: mg/L; PFAS: ng/L).

Principal component analysis (PCA)-multiple linear regression (MLR) analysis and independent samples *t*-test were performed using SPSS Statistics 26.0. Dotted line graphs and heatmap were constructed using Origin 2022 (Academic version). The map of sampling sites was constructed using ArcMap Pro 3.0.2.

**Table 1**  
Statistics of PFAS concentrations (ng/L) in GW and SW.

Analyte	GW					SW				
	Mean	SD	Min	Max	DF	Mean	SD	Min	Max	DF
PFBA	25.5	2.83	21.3	32.4	100 %	22.6	1.47	20.5	24.6	100 %
PFPeA	9.96	0.948	7.63	11.2	100 %	5.05	2.09	< LOD	6.27	88 %
PFHxA	21.6	1.52	18.0	23.5	100 %	26.4	1.09	25.0	28.1	100 %
PFHpA	5.50	0.582	4.64	6.46	100 %	5.36	0.354	4.92	5.84	100 %
PFOA	43.4	5.10	32.4	51.0	100 %	30.4	2.46	26.8	33.5	100 %
PFNA	6.92	0.580	5.89	7.47	100 %	6.60	1.27	5.18	9.48	100 %
PFDA	2.29	0.836	1.62	4.40	100 %	2.20	1.05	1.56	4.74	100 %
PFUnDA	1.14	1.02	0.501	3.67	100 %	0.972	0.669	0.485	2.21	100 %
PFDoDA	0.241	0.244	0.0768	0.742	100 %	0.144	0.154	< LOD	0.487	75 %
PFTriA	0.0471	0.0652	< LOD	0.201	20 %	0.0849	0.155	< LOD	0.457	25 %
PFTrEA	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
PFBS	12.0	2.16	8.04	14.8	100 %	15.7	1.19	14.3	17.6	100 %
PFPeS	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
PFHxS	3.52	0.491	2.73	4.28	100 %	4.32	0.301	3.76	4.73	100 %
PFHpS	0.0421	0.128	< LOD	0.405	10 %	< LOD	< LOD	< LOD	< LOD	0 %
PFOS	9.51	1.53	7.05	12.0	100 %	10.1	3.09	7.92	17.6	100 %
PFNS	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
PFDS	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
FBSA	0.159	0.0306	0.112	0.204	100 %	0.218	0.0615	0.123	0.346	100 %
FHxSA	0.125	0.114	0.0283	0.306	100 %	0.234	0.144	< LOD	0.372	88 %
FOSA	0.179	0.0654	0.121	0.283	100 %	0.190	0.118	0.0595	0.416	100 %
HFPO-DA	2.88	0.336	2.02	3.22	100 %	1.11	0.0697	1.03	1.24	100 %
HFPO-TA	169	44.1	119	254	100 %	63.2	41.7	25.0	141	100 %
6:2 FTCA	12.5	4.84	5.95	19.7	100 %	3.85	0.878	2.36	5.24	100 %
8:2 FTCA	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
10:2 FTCA	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
4:2 FTSA	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
6:2 FTSA	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
8:2 FTSA	< LOD	< LOD	< LOD	< LOD	0 %	< LOD	< LOD	< LOD	< LOD	0 %
PFECBS	0.0326	0.0988	< LOD	0.314	10 %	0.0311	0.0839	< LOD	0.239	12 %
6:2 C1-PFESA	0.421	0.149	0.275	0.780	100 %	0.299	0.184	0.180	0.731	100 %
8:2 C1-PFESA	0.0050	0.0116	< LOD	0.0381	10 %	< LOD	< LOD	< LOD	< LOD	0 %

### 3. Results and discussion

#### 3.1. Concentrations and compositions of PFAS and nutrients in wetlands

The detection rate of HFPO-TA, PFOA, PFBA, and PFHxA was 100 % in both wetlands (Fig. 2 and Table 1). Concentrations of  $\sum_{32}$ PFAS (dissolved and particle phases) in GW and SW varied from 274 to 405 ng/L (mean  $327 \pm 44.0$  ng/L) and 153–297 ng/L (mean  $199 \pm 51.0$  ng/L), respectively. The two wetland concentrations were comparable to Jiangsu Province source water ( $\sum_{17}$ PFAS: 12.1–128 ng/L) [63], Tianjin Yuqiao reservoir ( $\sum_{17}$ PFAS: 5.84–121 ng/L) [50], and Mediterranean wetland Albufera natural park ( $\sum_{21}$ PFAS: 5.60–234 ng/L) [64].

HFPO-TA was the predominant substance, accounting for 52 % and 32 % in GW and SW, respectively. Concentrations of HFPO-TA in GW were 120–254 ng/L (mean  $169 \pm 44.1$  ng/L), which were higher than that in SW of 25.1–141 ng/L (mean  $63.2 \pm 41.7$  ng/L). To our best knowledge, this is the first time to report high concentrations of HFPO-TA than other PFAS in wetlands. HFPO-DA concentrations were lower than HFPO-TA of 2.02–3.22 ng/L (mean  $2.88 \pm 0.336$  ng/L) in GW and 1.03–1.24 ng/L (mean  $1.11 \pm 0.0697$  ng/L) in SW. The highest concentration of HFPOs (sum of HFPO-DA and HFPO-TA) was 256 ng/L (G10), which was higher than that of 180 ng/L in Lake Taihu [25]. HFPOs are processing aids replacing PFOA in the production of fluorinated polymers, e.g., polytetrafluoroethylene (PTFE) and polyvinylidene fluoride (PVDF) [65]. Two fluoroplastic manufacturing companies primarily producing PTFE and fluorine resins are located at straight-line distance of 3.6 and 5.4 km respectively from the GW. And several plastic product manufactures located 5 km upstream of SW. The release from these manufactures could be the possible industrial sources.

PFOA accounted for 13 % and 15 % of  $\sum_{32}$ PFAS concentrations in GW and SW with concentrations ranging from 26.8 to 51.0 ng/L. As short-chain alternatives, the mean concentrations of PFBA (C4) ( $24.2 \pm 2.71$  ng/L) and PFHxA (C6) ( $23.7 \pm 2.77$  ng/L) were comparable in both wetlands. 6:2 fluorotelomer carboxylic acid (6:2 FTCA) concentrations ranged from 2.36 to 19.7 ng/L (mean  $8.64 \pm 5.67$  ng/L). It is a major intermediate of 6:2 fluorotelomer alcohol (FTOH) anaerobic biotransformation in the environment and has been used as a replacement processing aid for PFOA in China [66,67].

Concentrations of PFOS ( $9.51 \pm 1.53$  ng/L in GW;  $10.1 \pm 3.10$  ng/L in SW) and its alternative, perfluorobutane sulfonate acid (PFBS) ( $12.0 \pm 2.16$  ng/L in GW;  $15.7 \pm 1.19$  ng/L in SW) were at the similar levels. The source might be the discharge from textile, electronics, and metal manufacturing companies nearby [68]. 6:2 chlorinated polyfluorinated ether sulfonic acid (6:2 C1-PFESA) had a

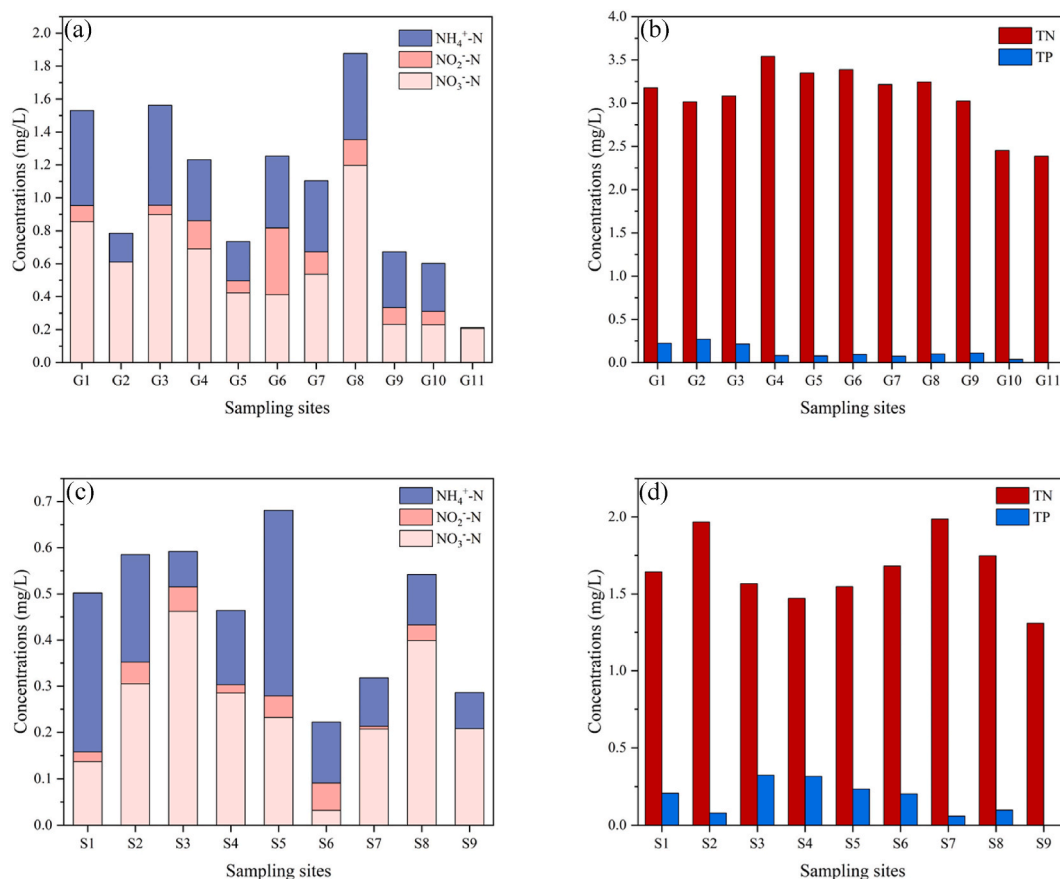


Fig. 3. Nutrients concentration (mg/L) at each site of GW (a: DIN, b: TN and TP) and SW (c: DIN, d: TN and TP).

detection rate of 100 % with concentrations of  $0.421 \pm 0.149$  ng/L and  $0.299 \pm 0.184$  ng/L in GW and SW respectively, which was comparable to that of the rivers and lakes (including Yangtze River, Yellow River and Taihu Lake, etc.) detected in China ( $1.1\text{--}7.8$  ng/L) [25].

Four factors were extracted by PCA (Supplementary Table S10). Factor 1 (29 %) was dominated by the PFOA and their alternatives, including PFBA, perfluoropentanoic acid (PFPeA), PFOA, HFPO-DA, HFPO-TA, and 6:2 FTCA related to the fluorinated polymers and plastic productions. Factor 2 (33 %) mainly included PFOS and 6:2 C1-PFESA which might represent metal plating industrials.

The nutrient concentrations in GW and SW are shown in Fig. 3. TN ranged from 2.45 to 3.54 mg/L (mean  $3.15 \pm 0.296$  mg/L) of GW, and 1.47–1.99 mg/L (mean  $1.70 \pm 0.190$  mg/L) of SW. DIN was 0.222–1.88 mg/L (mean  $0.848 \pm 0.467$  mg/L) in two wetlands. TP in GW and SW were comparable, at 0.0357–0.269 mg/L (mean  $0.128 \pm 0.0778$  mg/L) and 0.0590–0.323 mg/L (mean  $0.190 \pm 0.103$  mg/L), respectively. Both of them were affected by upstream domestic and industrial wastewater discharges [69,70].

### 3.2. Distribution and removal rates of PFAS and nutrients in wetlands

G1 and G2 were influent of GW, and G10 was effluent. Concentrations of  $\sum_{32}$ PFAS at influent were 391 ng/L and 294 ng/L, which were comparable with effluent of 405 ng/L indicating limited contributions by constructed wetlands for PFAS treatment. The lowest concentration of  $\sum_{32}$ PFAS (274 ng/L) was found at G4 in pretreatment area (A), and interestingly, the highest TN value (3.54 mg/L) was found at the same site. The highest concentration of  $\text{NO}_3^-$ -N (1.20 mg/L) was found at G8, which was in the buffer area between root-channels purifying and deep purifying area. The sum of PFOA and PFOS concentrations in influent and effluent (51.7 ng/L for G1, 42.4 ng/L for G2, 55.1 ng/L for G10) were all below the USEPA recommended value of 70 ng/L [71]. HFPO-DA concentrations at all sampling sites were below Health-Based Water Concentration (10 ng/L) [72]. However, there was no threshold for HFPO-TA. TN concentrations from 2.45 to 3.39 mg/L all exceeded the Class V (2 mg/L) of Chinese Environmental Quality Standards for Surface Water [73]. While other nutrients were below the Class V standards.

In SW,  $\sum_{32}$ PFAS concentrations in influent (S1) were 182 ng/L, higher than effluent of 153 ng/L at S8. In contrast to observations at GW where the highest concentrations of TN were observed at the same site as the lowest concentrations of PFAS, the highest concentrations of TN were found at S7 (1.97 mg/L) which also had the highest concentration of PFAS (297 ng/L). PFOA, PFOS and HFPO-DA concentrations did not exceed the recommended values [71,72]. The nutrient values were at Class IV standard [73]. Compared with the results ten years ago, the concentrations of TN, TP and  $\text{NH}_4^+$ -N at influent and effluent had both decreased by approximately 50 %–80 % [74].

High organic nitrogen concentrations were mainly observed in the root-channels purifying area (area B), for instance, G5 (2.615 mg/L) and S7 (1.668 mg/L). The high concentrations in this area might originate from the decomposition and release of phytoplankton, zooplankton, and other organisms in the water [75]. The high concentrations of organic nitrogen were also detected in the two deep purifying areas, with values of 2.353 mg/L for G9 and 1.205 mg/L for S8, which might be attributable to sediment releases. Previous studies have indicated that the release of dissolved organic nitrogen (DON) from sediment in shallow water lakes was much higher than that of DIN, serving as the primary source of organic nitrogen in the overlying water [76].

The removal rates of PFAS and nutrients in GW and SW are shown in Table 2. Negative precursor removal rates in GW were presented in area A (–86 %) and area B (–46 %). The reason was due to the increase of 6:2 FTCA concentrations from 5.95 ng/L (G3) to 11.7 ng/L (G4), followed by a further increase to 17.0 ng/L (G7). 6:2 FTCA might be transformed from 6:2 FTOH [67]. Due to its high vapor pressure, 6:2 FTOH is mainly distributed in the gaseous phase, with a concentration of 49  $\mu\text{g}/\text{m}^3$  in the air of Jiaying, higher than in other cities in central-eastern China [77]. Studies have reported that FTOHs can be transported through the air, then absorbed by plant leaves, metabolized within the plant to form PFCAs and PFSAs, and finally released into the water by the plants [78,79]. More

**Table 2**  
PFAS and nutrients removal in each treatment process of wetlands and DWTPs<sup>a</sup>.

Area	C <sub>4</sub> -C <sub>6</sub> PFCAs	C <sub>7</sub> -C <sub>14</sub> PFCAs	C <sub>4</sub> -C <sub>6</sub> PFSAs	C <sub>7</sub> -C <sub>10</sub> PFSAs	HFPOs	Precursors	DIN	TN	TP	
GW	Pretreatment (A: G3-G4)	–3% <sup>b</sup>	2 % <sup>c</sup>	10 %	–1%	16 %	–86 %	21 %	–15 %	62 %
	Root-channels Purifying (B: G4-G7)	1 %	–16 %	6 %	–9%	–49 %	–46 %	10 %	9 %	9 %
	Deep Purifying (C: G8-G9)	–4%	–15 %	16 %	–38 %	–34 %	16 %	64 %	7 %	–12 %
G-DWTP	25 %	39 %	11 %	63 %	65 %	88 %	65 %	3 %	86 %	
SW	Pretreatment (A: S1-S2)	5 %	–3%	14 %	5 %	–15 %	34 %	–17 %	–20 %	62 %
	Root-channels Purifying (B: S4-S6)	17 %	0 %	7 %	0 %	48 %	31 %	52 %	–14 %	36 %
	Deep Purifying (C: S7-S8)	7 %	24 %	9 %	53 %	80 %	34 %	–70 %	12 %	–66 %
S-DWTP	38 %	32 %	43 %	34 %	–79 %	63 %	47 %	25 %	95 %	

<sup>a</sup> All the removal rates were calculated according to Equation (1).

<sup>b</sup> Negative value indicates that effluent concentrations of pollutants were higher than influent concentrations.

<sup>c</sup> Positive value indicates that effluent concentrations of pollutants were lower than influent concentrations.



studies should be conducted to investigate the biotransformation of FTOHs in wetland plants. A high removal rate of TP (62 %) was found in area A. Although, TP concentrations increased to 0.110 mg/L at G9, the total remove rate (comparing G1, G2, and G10) was 86 %.

Area B showed removal rates of 1 %–6 % of C<sub>4</sub>–C<sub>6</sub> PFCAs and PFASs in GW. Reed and cattails can uptake PFASs from the sediment and accumulate contaminants in roots and leaves [80,81]. Short-chain PFASs easily pass through cell membranes resulting in absorption by roots [46]. Furthermore, they are transported from roots to vascular bundles via symplastic pathways [82,83], and finally from vascular bundles to leaves via transpiration [84]. Additionally, previous studies have revealed that nutrients could enhance the adsorption and accumulation of PFASs in roots, and are essential for the biotransformation of 6:2 FTOH into 6:2 FTCA [85], implying that PFASs removal may be influenced by nutrient stress. Nutrients exhibited significant positive correlations ( $p < 0.01$ ) with PFCAs and PFOA alternatives (Supplementary Table S12). In addition to sharing common sources, this relationship might also be associated with the plants' capacity to absorb PFCAs. This can be attributed to the higher hydrophobicity of PFASs compared to PFCAs, especially when comparing molecules with the same carbon chain length, due to the larger molecular size of PFASs [78]. However, the bioremediation in wetlands might lead to two opposite results: 1) the uptake of PFCA and PFSA from water and sediment via roots, and 2) the release of intermediates of precursors that were absorbed from the air via leaves.

In deep purifying area (area C) of GW, C<sub>4</sub>–C<sub>6</sub> PFASs and precursors (mainly 6:2 FTCA) were effectively removed both with rates of 16 %. The removal of HFPOs was –49 % and –34 % in areas B and C, respectively. High concentrations of HFPO-TA were observed at G5 (162 ng/L), G7 (179 ng/L), G9 (175 ng/L) and G10 (254 ng/L). These sites were located on the edge of the wetland and near the source rivers, which could receive contaminated water via groundwater [86]. Similar spatial distributions were found for PFBA, PFOA and PFOS. In area B, the TN removal rate reached the highest value of 9 %, and the removal rate of DIN was 64 % in area C. GW wetland was effective for TP and DIN removal under PFAS stress.

Removal rates of PFASs ranged from 0 % (for C<sub>7</sub>–C<sub>14</sub> PFCAs and C<sub>7</sub>–C<sub>10</sub> PFASs) to 80 % (for HFPOs) in areas B and C of SW, higher than those in GW (–49 %–16 %). The removal rates of DIN in area A and area C of SW were both negative (–17 % and –70 %, respectively), due to the increase in NO<sub>3</sub><sup>-</sup>-N and NO<sub>2</sub><sup>-</sup>-N concentrations were higher than the decrease in NH<sub>4</sub><sup>+</sup>-N. In these two areas, the dissolved oxygen (DO) concentrations ranged from 5.37 mg/L to 9.96 mg/L (>3 mg/L), which inhibited the formation of nitrate reductase and consequently decreased denitrification activity [87]. During the sampling period, the reduction in temperature hindered the growth and proliferation of denitrifying bacteria. Consequently, denitrification efficiency was compromised, leading to a decrease in the removal of NO<sub>3</sub><sup>-</sup>-N and NO<sub>2</sub><sup>-</sup>-N [88]. SW was more efficient at removing PFASs and nutrients in area B than GW, possibly influenced by dredging, and sediment and plants were removed. SW regularly harvests emergent aquatic plants in November each year [89], accelerating plant metabolism rates and stimulating rhizosphere microbial activity to enhance pollutant removal efficiency [90, 91]. Previous studies have indicated that dredging of sediments was an important measure for reducing internal pollution sources and removing nutrients [92]. Therefore, timely plants harvesting and sediment dredging are essential measures to ensure wetlands maintain their water quality maintenance and purification effects. According to previous studies, total organic carbon (TOC) concentrations in SW and GW ranged from 9 to 21 mg/L [93] and 8–12 mg/L [94], respectively, which were much lower than those in wetlands treating wastewater or leachate (TOC: 41–210 mg/L) [95]. The lack of TOC and other organic matter may be one of the reasons that GW and SW had lower pollutant removal rates than other constructed wetlands [96].

### 3.3. The removal through DWTPs

G10 and S8 could represent DWTP influent, while G11 and S9 were drinking water collected from the tap of DWTP effluent. Compared with previous studies,  $\sum_{32}$ PFAS concentrations of DWTP effluents in this paper (179 ng/L for G-DWTP and 129 ng/L for S-DWTP) were lower than those of  $\sum_{17}$ PFAS detected in 2018 (224 ng/L for G-DWTP and 157 ng/L for S-DWTP) [23]. After treatment, individual PFAS concentrations were below 30.7 ng/L (PFOA) except for HFPO-TA, which was 87.1 ng/L and 50.0 ng/L for G-DWTP and S-DWTP, respectively. Meanwhile, the water quality achieved the class I (NH<sub>4</sub><sup>+</sup>-N ≤ 0.5 mg/L, NO<sub>3</sub><sup>-</sup>-N ≤ 10 mg/L) of the Chinese Water Quality Standard for Drinking Water Sources [97]. In G-DWTP, PFAS removal rates ranged from 11 % (for C<sub>4</sub>–C<sub>6</sub> PFASs) to 88 % (for precursors), and long-chain PFAS (C ≥ 7) removal rates (39 % for PFCAs and 63 % for PFASs) were higher than those for short-chain (C<sub>4</sub>–C<sub>6</sub>) (25 % for PFCAs and 11 % for PFASs). Removal rates of nutrients ranged from 3 % (for TN) to 86 % (for TP). In S-DWTP, the highest removal rate was 63 % for precursors and 95 % for TP, respectively. Both DWTPs are equipped with activated carbon processes, and powdered activated carbon emergency dosing systems [98], which could effectively remove PFASs and nutrients [99]. In S-DWTP, HFPO-TA concentrations increased from 27.1 ng/L to 50.0 ng/L, with a removal rate of –84 % indicating the unstable function of emerging contaminant treatments.

### 3.4. Suspect and non-target compounds

Thirty-two PFASs were identified by suspect and non-target screening. Ten of them were identified at CL 2 or 3 (Supplementary Table S13), and semi-quantified (Supplementary Tables S14–S15). Nine substances were at 100 % detection rates. Bistriflimide (HNTf2) was the predominant non-target substance, accounting for 32%–85 % of  $\sum_{10}$ PFASs (Supplementary Fig. S3). It is a poly-fluorinated sulfonimide dimer which is used to increase the stability of ionic liquids and the cyanine dyes in industrial fields [100]. It was reported in surface water in Beijing (China) [27] and municipal waste landfill leachate in Shanghai (China) [101]. The distribution of bistriflimide was similar to HFPO-TA in GW.

Exact fragments of C<sub>3</sub>F<sub>5</sub>O<sup>-</sup> ( $m/z$  146.98688), C<sub>3</sub>F<sub>5</sub>SO<sub>3</sub><sup>-</sup> ( $m/z$  210.94936), and C<sub>3</sub>HF<sub>6</sub>SO<sub>3</sub><sup>-</sup> ( $m/z$  230.95534) were observed in the MS/MS spectrum (Supplementary Fig. S4) which could be identified as monohydro-substituted ultrashort chain PFSA i.e., 1,1,2,2,3,3-

hexafluoropropane-1-sulfonate (H-PFPrS,  $C_3HF_6O_3S^-$ ,  $m/z$  230.95583) [24]. The detection frequency at 100 % of H-PFPrS in GW and was the second substance, accounting for 0.8%–39 % to  $\sum_{10}PFAS$ , which had been detected in Lake Taihu [24]. However, the potential sources and environmental fate have not yet been understood.

Five homologues of N-methyl perfluoroalkyl sulfonamido acetic acids (MeFASAAs) were detected, including N-methylperfluoroethanesulfonamido acetic acid (MeFEtSAA), N-methylperfluorobutanesulfonamido acetate (MeFBSAA), N-methyl perfluorohexane sulfonamido acetic acid (MeFHxSAA), N-methylperfluoroheptane sulfonamido acetic acid (MeHpSAA) and N-methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) (Supplementary Figs. S5–S9). All of them were 100 % detected in both GW and SW. The MS/MS spectrum of MeFEtSAA, MeFBSAA, and MeFHxSAA have been found, including fragments of  $SO_3^-$  ( $m/z$  79.95739),  $C_2F_5^-$  ( $m/z$  118.99223),  $C_3F_7^-$  ( $m/z$  168.98888),  $C_4F_9^-$  ( $m/z$  218.98587),  $C_5F_{11}^-$  ( $m/z$  268.98346). Only MS spectrum was available for MeHpSAA and MeFOSAA. The identification was further supported by a reasonable order of retention times: MeFEtSAA (11.27 min) < MeFBSAA (12.65 min) < MeFHxSAA (13.34 min) < MeHpSAA (13.64 min) < MeFOSAA (13.90 min). 0.6:2 H-PFESA is a hydrogenated analogue of 6:2 C 1-PFESA and was found by homologue-based and fragment-based non-target screening. In MS/MS spectrum, fragments  $C_5F_9^-$  ( $m/z$  230.98630),  $C_6HF_{12}O^-$  ( $m/z$  316.98422) and  $C_8HF_{16}O_4S^-$  ( $m/z$  496.93491) could be matched to theoretical fragments (Supplementary Fig. S10). It accounted for 2 %–10 %  $\sum_{10}PFAS$  concentrations. 6:2 H-PFESA is derived from the reductive dechlorination process of 6:2 C 1-PFESA and had been detected in rivers and sediments [25,102].

Sodium *p*-perfluorooxobenzene sulfonate (OBS) was detected in both wetlands (Supplementary Fig. S11). As an economical alternative to PFOS, OBS has been used in China as a fire-fighting foam co-formulant and oil production agent [103]. OBS contribution was less than 1 %. It has been widely detected in the East China Sea (5.2 pg/L) and Poyang Lake (6.3 ng/L) [104,105].

N-ethylperfluorooctane sulfonamidoacetic acid (N-EtFOSAA) was detected in 91 % and 22 % of GW and SW, respectively (Supplementary Fig. S12). It is a degradation intermediate of N-ethyl perfluorooctane sulfonamidoethanol (EtFOSE) and may form PFOS through abiotic or biocatalytic transformation [106,107].

The semi-quantification of  $\sum_{10}PFAS$  were 9.13 ng/L and 9.55 ng/L in the tap water G11 and S9, which were similar to the previous study detected in the Guangzhou DWTPs (4.10–17.6 ng/L) [28]. The concentration of H-PFPrS was the highest in G11, with values of 3.55 ng/L. While in S9, bistriflimide reached the highest concentration of 4.06 ng/L. The occurrence of bistriflimide might be related to the production of upstream photosensitive materials [100]. Among the MeFASAAs, MeFBSAA had the highest concentrations of 0.670 ng/L for G11 and 0.680 ng/L for S9. MeFBSAA was the intermediate transformation product of N-methylperfluoroalkyl sulfonamido ethanols (MeFBSE) that can be subsequently biotransformed into FBSA [27,108]. This indicated that the biological transformation process of MeFBSE existed not only in urban solid waste anaerobic landfills [109], but also in the treatment processes of municipal drinking water treatment plants. The similar non-target PFAS concentrations of the two DWTPs showed that further improvements are necessary for the removal capacity of emerging PFAS compounds by different treatment processes in water plants.

#### 4. Conclusion

This study investigated the distributions, sources, and removal of PFAS and nutrients in two constructed wetlands and their nearby DWTPs. The results showed that HFPO-TA was the predominant compound followed by PFOA, PFBA and PFHxA, while TN was the highest nutrient. PFAS and nutrients might originate from upstream domestic sewage and industrial discharges (e.g., textile, plastics, metals, and PTFE plants). Constructed wetlands exhibited effective removal of PFAS under nutrient stress. In constructed wetlands, artificial intervention was a measure that could enhance the removal of PFAS and nutrients. However, wetland bioremediation might have contrasting effects, such as the uptake of PFAS via roots from water and sediment or the release of precursor intermediates absorbed from the air through leaves. Of the 32 suspect and non-target substances, nine substances were detected at 100 %. The non-target concentrations in the two tap water samples were below 10 ng/L, but we should not overlook the risks of long-term exposure. In the future, more atmospheric, groundwater, plants and sediments samples from the wetland water treatment systems are needed to further analyze the environmental fate and removal mechanisms of nutrients and PFAS.

#### Data availability statement

All relevant data are presented in the article and Supporting Information.

#### Ethics approval and consent to participate

Review or approval by an ethics committee was not needed for this study because no data on patients or experimental animals was used in the article. Informed consent was not required for this study because no clinical data was produced in the article.

#### CRediT authorship contribution statement

**Shiyue Li:** Writing – original draft, Investigation, Formal analysis, Data curation. **Zhen Zhao:** Writing – review & editing, Supervision, Resources, Methodology, Conceptualization. **Jing Liu:** Writing – review & editing, Investigation, Data curation. **Boxuan Zhang:** Writing – review & editing, Resources. **Baocang Han:** Resources, Investigation. **Yuntao Ma:** Resources, Project administration. **Limin Jin:** Resources, Investigation. **Ningzheng Zhu:** Project administration, Investigation. **Guoping Gao:** Writing – review & editing, Supervision, Funding acquisition. **Tian Lin:** Supervision, Resources, Funding acquisition.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.heliyon.2024.e37551>.

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