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Distribution and fate of perfluorinated compounds (PFCs) in a pilot constructed wetland

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ABSTRACT

Removals of perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) in pilot-scale constructed wetland were investigated in this study. Phytoextraction by four aquatic plants (*Hygrophila pogonocalyx Hayata*, *Ipomoea aquatic Forssk*, *Ludwigia* (*x*) *taiwanensis* and *Eleocharis dulcis*) and sorption onto soil were determined. Both PFOA and PFOS were fairly phytoextracted by four aquatic plants; the uptake capacity was found in the following order: *Hygrophila pogonocalyx Hayata* > *Ipomoea aquatic Forssk* ≈ *Ludwigia* (*x*) *taiwanensis* > *Eleocharis dulcis*. In the soil sorption experiment, equilibria for PFOA and PFOS were achieved within 6 d; sorption capacity of 2–3 μg/g was observed at the end of the 15 d experiment. Experimental results and literature review suggest that phytoextraction and soil sorption were the most significant mechanisms for PFOA and PFOS disappearance in the pilot-scale constructed wetland, while contributions of biodegradation, photolysis, volatilization and hydrolysis were negligible.

Keywords: Constructed wetland; Perfluorooctanoic acid; Perfluorooctane sulfonate; Sorption; Phytoextraction; Soil

1. Introduction

Perfluorinated compounds (PFCs) are emerging organic pollutants because they are persistent in the environment and represent serious health concerns to human [1,2]. Perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) are the most common PFCs detected in aqueous environments [3]. For instance, the concentrations of PFOA and PFOS detected in Japan's Tsurumi River were 15.9 ± 0.3 and 179.9 ± 34.4 ng/l, respectively, Lin et al. demonstrated the occurrence of PFCs in the raw waste streams from 18 different processes in an electronics/optoelectronics fabrication plant in Taiwan [4,5]; the most frequently detected PFCs were PFOA and PFOS, at concentrations of 1.6–46.2 μg/l (PFOA) and 1.0–51.4 μg/l (PFOS). Loganathan et al. surveyed PFCs in wastewater treatment facilities in Kentucky, USA, and identified both PFOS (7–16 ng/l in influent; 8–28 ng/l in effluent) and PFOA (22–184 ng/l in influent; 122–183 ng/l in effluent) [6].

PFOA and PFOS are resistant to biodegradation. Conventional wastewater treatment plants (WWTPs) are not designed to remove these compounds. Therefore these compounds may exist at low levels (ng/l-μg/l) in the aqueous environment receiving discharges from WWTPs. Many studies have reported that PFCs accumulate in organisms including tissues in wild animals, human serum and in milk [7,8]. It is essential that the fate of these trace organic contaminants be understood so that effective treatment technologies can be developed or applied to remove these compounds.

A wetland is composed of water, soil and plants. Plants in a wetland play an important role in removing pollutants. Many researchers used wetlands to treat water

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pollutants such as biochemical oxygen demand (BOD), chemical oxygen demand (COD), suspended solid (SS), heavy metals, total nitrogen (TN), total phosphorus (TP) and organic matters (e.g. pesticides and anionic surfactants) [9–18]. Perez-Marin et al. used constructed wetlands to treat municipal wastewater [16]; the removal efficiencies were up to 95% for COD, 97% for BOD, 87% for SS, 80% for TN and at least 40% for TP. Khan et al. investigated removal efficiency of heavy metals from industrial wastewater by a continuous free surface flow wetland [10]. Results indicated that the removal efficiencies of the constructed wetland for Pb, Cd, Fe, Ni, Cr, and Cu were 50%, 92%, 74%, 41%, 89% and 48%, respectively. Yeh et al. reported that effective rejection of copper and zinc were 83% and 92% by cattail (*Typha latifolia*), and 83% and 92% by reed (*Phragmites australis*) wetland systems [11]. Sima et al. used a constructed wetland with a horizontal subsurface flow to treat anionic surfactants in municipal wastewater [12]; the average removal efficiencies were 83.7% in 2007 and 81.7% in 2008.

To the best of our knowledge, investigation of PFOA and PFOS removals in a constructed wetland has been very limited. The aims of this study were to: (1) estimate PFOA or PFOS distribution in water, soils and plants as a function of time via a pilot-scale constructed wetland, (2) explore the uptake capacity of four selected aquatic plants (*Hygrophila pogonocalyx Hayata*, *Ipomoea aquatic Forssk*, *Ludwigia* (*x*) *taiwanensis* and *Eleocharis dulcis*) and the extent of sorption onto soils for PFOA or PFOS, and (3) assess the total PFOA and PFOS removals from water by plants and soil in a pilot-scale constructed wetland system.

2. Materials and methods

2.1. Chemicals

Perfluorooctanoic acid (PFOA) was obtained from Sigma Aldrich (St. Louis, MO, USA), and potassium perfluorooctane sulfonate (PFOS) was purchased from Fluka (Buchs, Switzerland). The purity of the target chemicals was >96%. Individual stock standard solutions were prepared in methanol with a concentration of 1000 mg/l and then stored in polypropylene containers at –20°C. The physical and chemical properties of the target chemicals are summarized in Table 1.

2.2. Pilot constructed wetland experiments

Four pilot-scale constructed wetland systems were set up at Lujiao Creek Constructed Wetland (LCCW) in New Taipei City, Taiwan. Also, four aquatic plants (*Hygrophila pogonocalyx Hayata*, *Ipomoea aquatic Forssk*, *Ludwigia* (*x*) *taiwanensis* and *Eleocharis dulcis*) were selected for the study. These plants are among the most common plants found in LCCW, and are all emergent macrophytes. The study was conducted in July 2010. It was mostly sunny, and the average temperature ranged between 28 and 32°C (including day and night time).

Table 1
Physical-chemical properties of PFOA and PFOS

Physico-chemical property	PFOA	Potassium salt of PFOS
Molecular weight	414.07	538.23
Chemical formula	$C_8HF_{15}O_2$	$C_8F_{17}SO_3^-K^+$
Boiling point	189°C (9.81×10^4 Pa)	NA
Melting point	55–56°C	≥400°C
Vapor pressure	69 Pa (25°C)	3.31×10^{-4} Pa (20°C)
Log K_{ow}	NA	–1.08
Henry's law constant	NA	$<2 \times 10^{-6}$ (atm-m^3/mole) (20°C in pure water)
Water solubility	3.4 g/l	680 mg/l
Acid dissociation constant (pK_a)	2.3–3.4	–3.27
pH	2.6 (at 1 g/l)	7–8 (for potassium salt of PFOS)

Data source are from references [34,39–42].
PFOA: perfluorooctanoic acid; PFOS: perfluorooctane sulfonate; NA: not available; K_{ow}: octanol-water partition coefficient.

The total weight of each of the four plants selected for the study was controlled at 500 ± 10 g. They were planted in 4 separated pilot tanks with dimensions of 150 cm (l) × 30 cm (W) × 85 cm (H). Each tank was filled with 20 cm of soil that weighs 108 ± 1 kg. The soil was sieved (8 mesh, 2.36 mm) before use to remove the rubble. The physical-chemical properties of the soil, including pH, conductivity, organic carbon, cation exchange capacity, texture analysis, particle size distribution analysis, Cl^- content, density and porosity are summarized in Table 2. Ninety (90) l of PFOA and PFOS-containing solution with a concentration of 5 mg/l were prepared and poured into each pilot tank.

2.3. Sampling and pretreatment

Water, soil and plant in each pilot tank were sampled at the 1st, 2nd, 4th, 6th, 8th, and 15th day. The sampling and pretreatment of water, soil and plant samples were described below.

Water samples (20 ml) were collected from each pilot tank in polypropylene containers and stored in ice-packed coolers. All samples were vacuum-filtered through 0.22 μm nylon membrane filters (Advantec, Toyo Roshi Kaisha, Japan) and then stored at 4°C before analysis. Since water samples were expected to have high PFOA and PFOS concentrations (mg/l), they were diluted and then analyzed by liquid chromatography/tandem mass spectroscopy (HPLC-MS/MS) (without extraction).

Table 2
Physical-chemical properties of the soil

Property	Value
pH	5.91
Conductivity (μmho/cm)	48.35
Organic carbon (%)	0.3
Cation exchange capacity (cmole (+)/kg soil)	4.77
Texture analysis	Sandy soil
Particle diameter analysis	<38 μm: 5.92%, <38–75 μm: 7.76%, 75–90 μm: 2.54%, 90–106 μm: 3.04%, 106–125 μm: 8.97%, 125–150 μm: 6.00%, 150–180 μm: 16.17%, 180–212 μm: 6.59%, 212–300 μm: 17.01%, 300–600 μm: 22.76%, 600–1180 μm: 2.78%, >1180 μm: 0.46%.
Cl^- content (%)	0.000361
Density (g/cm^3)	1.417
Porosity (%)	17.53

Soil samples (around 10 g) were grabbed from each pilot tank and put into polypropylene containers. The samples were freeze-dried, sieved (0.59 mm mesh), and stored at room temperature. The extraction of soil samples was performed according to the procedures developed by Washington et al. and Lin et al. with minor modifications [19,20]. Briefly, each 1 g sample was extracted according to the following steps: (1) addition of 500 μl of Milli-Q water; (2) addition of 250 μl of 1 M NaOH; sample then vortexed and left to react for 30 min; (3) addition of 5 ml of MeOH:H_2O (50:50, v/v) solution; sample was then vortexed until homogenized; (4) neutralization with 250 μl of 1 M HCl, sample then vortexed; (5) sonication of the sample for 60 min; (6) centrifugation at 1500 rpm and 20°C for 15 min; (7) decanting 400 μl of supernatant into a vial followed by filtration with a 0.22 μm nylon membrane; (8) analyzing the samples by HPLC-MS/MS.

Plant samples were collected individually from each pilot tank and then stored at 4°C. The extraction of the plant samples was performed according to the procedures developed by Quinete et al. with slight modifications: (1) weighting the individual plant; (2) addition of 5 ml Milli-Q water/g-plant; sample was then squeezed and vortexed until homogenized; (3) adding 1 ml mixture into a 15 ml PP centrifuge tube; (4) addition of 11 ml of 0.01 N NaOH/MeOH to extract; (5) shaking the sampled for 120 min at 250 rpm; (5) centrifugation at 3500 rpm and 20°C for 25 min; (6) decanting 400 μl of supernatant into a vial and filtering the supernatant with a 0.22 μm nylon membrane; (7) analyzing samples by HPLC-MS/MS [21].

2.4. LC/MS/MS analysis

The concentrations of PFOS and PFOA were determined using an Agilent 1100 module (Agilent Technologies, Palo Alto, CA, USA) equipped with XTerra MS C_{18} column (100 × 2.1 mm, 3.5 μm) coupled to a Micromass Quattro Premier XE tandem quadruple mass spectrometer (Waters, Manchester, UK). One mM ammonium acetate aqueous solution as mobile phase A and pure methanol as mobile phase B were used as binary gradient with a flow rate of 250 μl/min. Twenty micro-liter of sample was injected and eluted out of the column within 11 min. The gradient elution program started with 5% of mobile phase B for 1 min, increased to 40% (1–3 min), to 50% (3–3.5 min), to 60% (3.5–4.5 min), to 95% (4.5–5 min), remained at 95% (5–10 min), and decreased to 5% (10–11 min).

In the case of mass spectroscopy, the analyses were performed in a negative mode. The main working parameters were set as follows: capillary voltage, 2.8 kV; cone gas flow, 50 l/h; desolvation gas flow, 1000 l/h; source temperature, 120°C; desolvation temperatures, 400°C. Nitrogen (99.9% purity) and argon (99.9% purity) were used as cone and collision gases, respectively. Multiple reaction monitoring (MRM) transitions as well as the individual cone voltage and collision energy voltages applied for the analysis are summarized in Table 3. The dwell time established for each transition was 0.1 s and the interscan delay was set at 10 ms.

Quantification was based on a 6-point calibration curve with a linear range from 20–500 μg/l. The correlation coefficient of calibration curves was ≥0.997 based on a linear mode, least squares regression analysis.

Table 3
MS/MS conditions for multiple reaction monitoring

Compound	Cone voltage (V)	Collision energy voltage (eV)	Precursor ion (m/z)	Production (m/z)
PFOS	45	35	499	80
		35		99
PFOA	12	18	413	169
		12		369

PFOA: perfluorooctanoic acid; PFOS: perfluorooctane sulfonate.

3. Results and discussion

The concentration profiles of PFOA and PFOS in various aquatic plants of the pilot-scale constructed wetland systems are depicted in Fig. 1. For PFOA, *Hygrophila pogonocalyx Hayata* exhibited the highest average uptake capacity (11.6 μg/g) among four plants, followed by *Ipomoea aquatic Forssk* (9.0 μg/g), *Ludwigia* (*x*) *taiwanensis* (7.8 μg/g), and *Eleocharis dulcis* (5.6 μg/g). With regard to PFOS, *Hygrophila pogonocalyx Hayata* exhibited the highest uptake capacity (46 μg/g), followed by *Ludwigia* (*x*) *taiwanensis* (40 μg/g), *Ipomoea aquatic Forssk* (38 μg/g), and *Eleocharis dulcis* (26 μg/g). As mentioned above, PFOA and PFOS were strongly adsorbed by *Hygrophila pogonocalyx Hayata* that had greater small-size root (D < 3 mm) biomass, larger root surface area per plant, faster root growth, and shallower root distribution than the other aquatic plants. On the other hand, PFOA and PFOS were weakly adsorbed by *Eleocharis dulcis* which was the only tuberous root (corm) plant among the four aquatic plants studied. These findings suggested that the characteristics of the plant's root system affect its uptake capacity when it comes to PFC removals.

Several previous studies indicated that constructed wetland performance was correlated with root density and size [22–26]. Other studies showed that the surface was important for wetland microorganisms and crucial for contaminant removal [27,28]. Cheng et al. showed that fibrous-root plants (D ≤ 1 mm) exhibited significantly higher ammonium-nitrogen and nitrate-nitrogen removal rates than those rhizomatic-root plants (D > 3 mm) [23].

Comparing the uptake of PFOA and PFOS by various aquatic plants, PFOS was adsorbed approximately five times more than PFOA. The phenomenon of strong adsorption for PFOS was believed to be due to its higher octanol-water partitioning coefficient, log K_{ow} (PFOS = 4.13; PFOA = 2.50). Similar results were also observed by Luo et al. who reported that the uptake capacity of target chemicals (16 polycyclic aromatic hydrocarbons, 7 organochlorine pesticides, and 11 nonylphenols and their motabolites) by aquatic plants in a constructed wetland increases with increasing K_{ow} values [9]. However, this observation is still preliminary; further investigation would be needed to evaluate the uptake capacity related to hydrophobicity.

As shown in Fig. 2, sorption of PFOA and PFOS appeared to have reached equilibrium conditions after

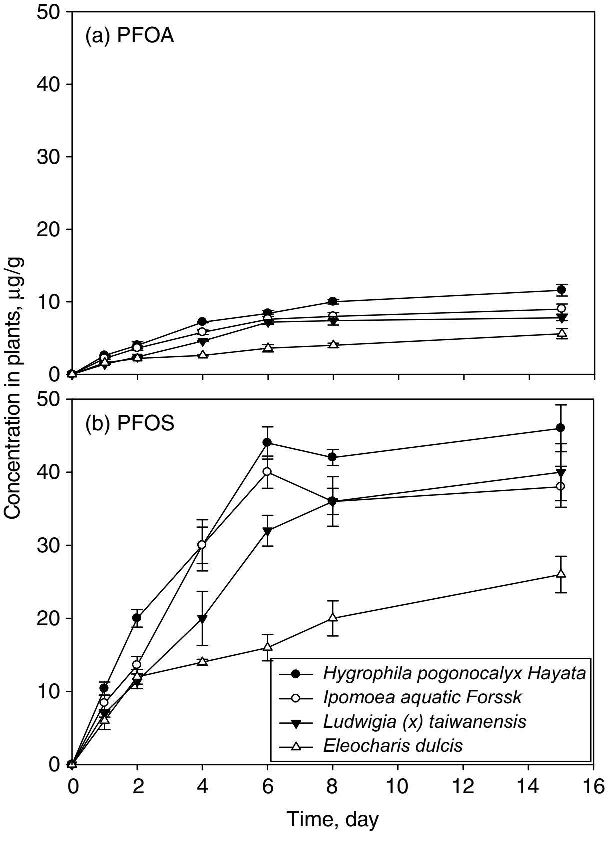

Fig. 1. Concentration profiles of PFOA and PFOS in various plants.

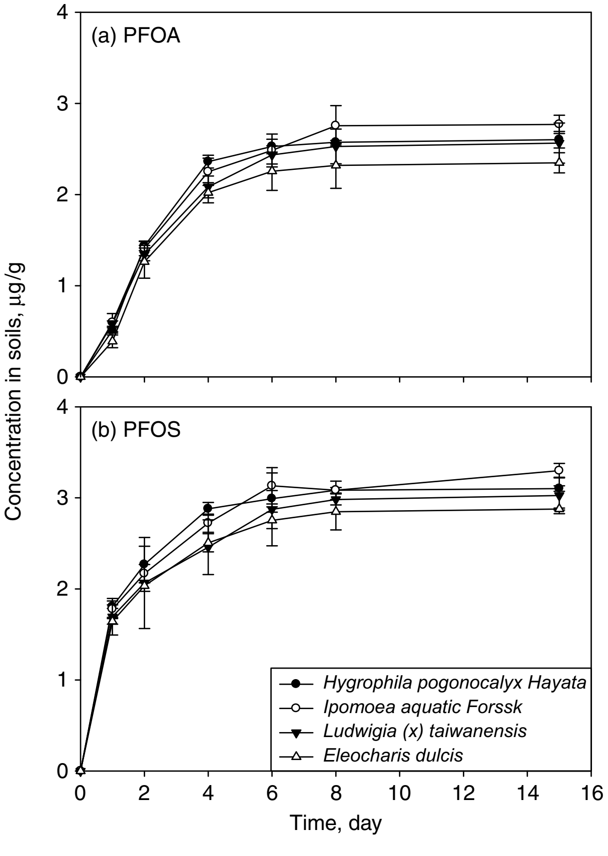

Fig. 2. Concentration profiles of PFOA and PFOS in soils.

6 and 4 d, respectively. The observed sorption capacity of PFOA and PFOS were 2–3 μg/g at the end of the 15 d experiment. Higgings and Luthy reported that sorption for PFOA and PFOS reached equilibrium within 6 d, resulting in >20% removal in 10 d [29].

A comparison of PFOA and PFOS concentrations in the plants versus their levels in the soil indicated that the plant uptake capacities were much higher than the soil sorption capacity. For instance, average plant uptake rate ranged from 5.6 to 11.6 μg/g for PFOA, and from 26 to 46 μg/g for PFOS, while the PFOA and PFOS adsorbed onto soils at the end of the 15 d experiment were in the range of 2–3 μg/g. The significant difference between sorption onto soils and plant uptake might be attributable to the high organic matter contents in plants. In general, PFOA and PFOS exist mainly in negatively charged form ($pk_a \le 3.4$) at neutral pH; they are anticipated to be less readily adsorbed onto the negatively charged soil surface. Overall, uptake and sorption of organic matters are much more complicated and are affected by carbon contents, pH, surfactants, inorganic matter contents, surface area and functional groups on matrix surface, electrostatics of the matrix and physico-chemical properties of sorbates (i.e. pK_a, K_{ow}) [29–32]. Therefore, further experiments should be conducted to identify the uptake and sorption mechanisms for PFOA and PFOS in the wetland system.

As shown in Fig. 3, equilibrium concentrations of PFOA and PFOS in liquid phase were achieved in 8 d for all plants except *Eleocharis dulcis*. The removal efficiencies for PFOA and PFOS in liquid phase were 77–82% and 90–95%, respectively. This clearly indicated that constructed wetland could effectively remove PFOA and PFOS present in the liquid phase.

Mass balance calculations performed on PFCs in water, soils and plants showed that, on average, 75–98% of the mass added to the system was accounted, as indicated in Fig. 4. Details of the mass balance calculations are shown in Table 4. The removal mechanisms of PFOA and PFOS in constructed wetland were believed to be mainly through sorption onto soil and phytoextraction. Some previous reports stated that PFOA and PFOS could not be effectively removed via volatilization, photolysis, hydrolysis, biodegradation and phytodegradation [33–35]. The loss through volatilization of PFOS was expected to be negligible due to their low Henry's law constants ($<2 \times 10^{-6}$ atm-m^3/mole). 3M Company pointed out that PFOA could be eliminated via hydrolysis, but with a half-life of 97 years [33]. Hori et al. and Chen et al. revealed that PFOA has no absorption

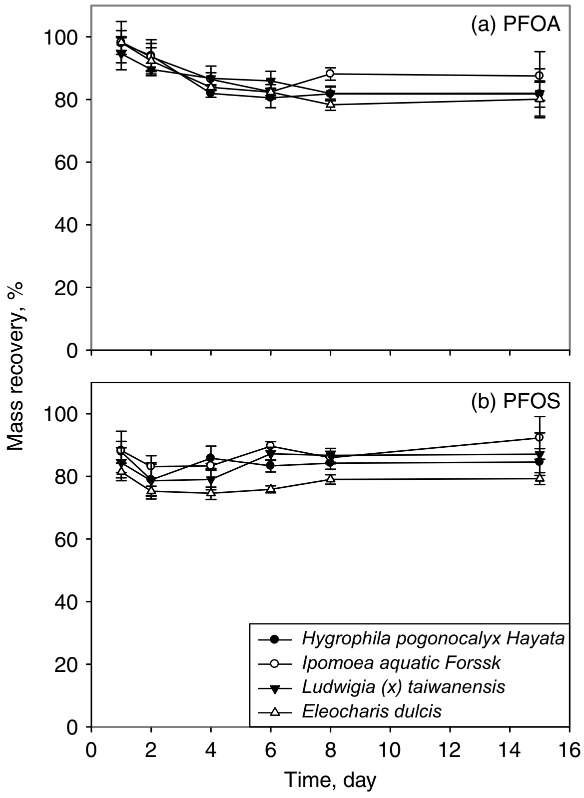

Fig. 3. Concentration profiles of PFOA and PFOS in the liquid phase.

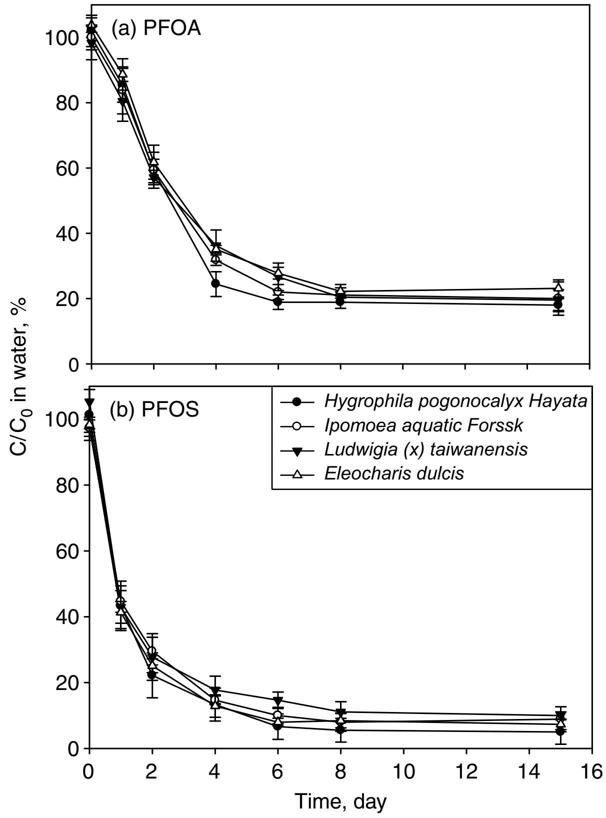

Fig. 4. Mass recovery for PFOA and PFOS.

Table 4
Mass balances of PFOA and PFOS at the end of the 15 d experiment

Matrix		Tank			
		Hygrophila pogonocalyx Hayata	*Ipomoea aquatic Forssk*	*Ludwigia (x) taiwanensis*	*Eleocharis dulcis*
(a) PFOA					
Plant	Concentration[a] (µg/g)	11.6 ± 0.8	9.0 ± 0.7	7.8 ± 0.4	5.6 ± 0.7
	Mass (mg)	5.8 ± 0.4	4.5 ± 0.4	3.9 ± 0.2	2.8 ± 0.4
Soil	Concentration[a] (µg/g)	2.6 ± 0.1	2.8 ± 0.1	2.6 ± 0.2	2.4 ± 0.1
	Mass (mg)	280.8 ± 10.8	302.4 ± 10.8	280.8 ± 21.6	259.2 ± 10.8
Water	C/C_0[a] (%)	18.0 ± 2.0	20.0 ± 5.1	19.6 ± 3.2	23.1 ± 2.6
	Mass (mg)	81 ± 9.0	90 ± 23.0	88 ± 14.2	104 ± 11.8
Total mass (mg)		**367.6 ± 20.2**	**396.9 ± 34.2**	**374.7 ± 34.0**	**365.5 ± 23.5**
Mass recovery (%)		**82 ± 4.2**	**88 ± 7.8**	**83 ± 7.8**	**81 ± 5.4**
(b) PFOS					
Plant	Concentration[a] (µg/g)	46.0 ± 3.2	38.0 ± 2.8	40.0 ± 3.9	26.0 ± 2.5
	Mass (mg)	23 ± 1.6	19 ± 1.4	20 ± 2.0	13 ± 1.3
Soil	Concentration[a] (µg/g)	3.1 ± 0.03	3.3 ± 0.1	3.0 ± 0.2	2.9 ± 0.01
	Mass (mg)	334.8 ± 3.2	356.4 ± 10.8	324.0 ± 21.6	313.2 ± 1.1
Water	C/C_0[a] (%)	5.0 ± 3.7	8.9 ± 3.8	10.0 ± 1.1	7.3 ± 1.6
	Mass (mg)	22.5 ± 16.7	40.5 ± 16.7	45 ± 5.0	31.5 ± 8.6
Total mass (mg)		**380.3 ± 21.5**	**415.9 ± 28.9**	**389.0 ± 28.6**	**357.7 ± 11.0**
Mass recovery (%)		**85 ± 4.3**	**92 ± 6.8**	**86 ± 6.8**	**80 ± 1.9**

[a] Average concentration and mean C/C_0 are used for calculating residual mass in the entire system.

above 240 nm and therefore could not be eliminated through direct photolysis [36,37]. Chen et al. reported that less than 5% of initial PFOA was degraded under irradiation of 254 nm UV light for 12 h [37]. Previous reports suggested that microbial cleavage of fluorinated alkyl compounds require at least one hydrogen to be present in the alkyl chain for primary attack to occur [38]. Thus, it is believed that perfluoroalkyl chains that are present in PFOA and PFOS will not be susceptible to biodegradation [35].

4. Conclusion

This study confirms that the pilot constructed wetland is of potential significance for removing target perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). Average removals of 77–82% for PFOA and 90–95% for PFOS were achieved at the end of the 15 d experiment. With regards to PFOA and PFOS uptake capacity, *Hygrophila pogonocalyx Hayata* exhibited the highest uptake capacity (11.6 and 46 µg/g for PFOA and PFOS, respectively), followed by *Ipomoea aquatic Forssk*, *Ludwigia* (*x*) *taiwanensis*, and *Eleocharis dulcis*. In the soil sorption experiment, sorption equilibrium for PFOA and PFOS was achieved within 6 d. The sorption capacity of the soil at the end of the 15 d experiment was 2–3 µg/g. Mass balance calculation of water, soils and plants showed average recovery of 75–98%. The removal mechanisms of PFOA and PFOS in the constructed wetland were believed to be mainly through sorption onto soil and phytoextraction.

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