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Partition behavior of perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA) in riverine sediments

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ABSTRACT

The sorption of perfluoroalkyl substances on the sediments is an important process which determines to their fate, distribution and transport in water system. In this study, the sorption kinetics, thermodynamics and effect parameters on partition behavior of perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA) at low initial concentration (50 ng/L) were investigated. The results showed that the equilibrium was achieved within 48 h. The sorption isotherms of PFOS and PFOA could be described by Freundlich equation. The surface area and composition of sediment particles as well as solution pH values and ion strength strongly influenced the sorption of PFOA and PFOS. Both PFOA and PFOS exhibited higher sorption capacity on the sediments with high organic matter content. These results indicated that multiple driving forces such as hydrophobic exclusion, specific chemical interaction and electrostatic attraction contributed to the nonlinear sorption of PFOA and PFOS on sediments.

Keywords: PFOA; PFOS; Sediment; Partition properties; Natural riverine water

1. Introduction

Perfluoroalkyl substances (PFASs) are a new type of persistent organic pollutants (POPs) [1,2]. They have attracted global concern due to high bioaccumulation, extreme persistence and toxicity, as well as wide distribution in the environment. Of the PFASs, perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA), ubiquitously present in the diverse environments [3,4], showed the highest persistence and accumulation in the aquatic environment. Thus, PFOS, its salts and perfluorooctane sulfonyl fluoride have been added to the Stockholm Convention list of the POPs since 2009 [5]. PFOA, as a substance of very high concern in candidate list under REACH regulation [6] has been prohibited by the Environmental Protection Agency of the United States (US EPA) on a PFOA-Stewardship Program in 2006 [7].

However, owing to more than 50 years of production and worldwide application, PFOS and PFOA can be released into natural aquatic environment, causing the global distribution [8]. Subsequently, these pollutants have undoubtedly involved the complex processes such as sorption, degradation and transport, which determine their environmental fate in aquatic systems [9]. The understanding of these basic mechanisms is of significance in both regulatory and scientific areas. Sediment, with complex compositions including minerals, clay, sand and sedimentary organic matter, is an important sink and reservoir for PFOS and PFOA [10]. The sorption of PFOS and PFOA onto the sediment determines their distribution, transport and transformation processes in the aquatic environment.

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The sorption behavior highly depends on the physical and chemical properties of PFOS and PFOA, as well as the conditions of water chemistry such as pH value, ionic strength and property of dissolved organic matter. Higgins and Luthy [11] valuated various sediment-, solution- and chemical-specific parameters potentially affecting sorption of PFASs. They found that organic carbon rather than inorganic oxide content in sediment was the dominant effect parameter on sorption, indicating the importance of hydrophobic interactions. The similar results were also obtained in other previous studies [10,12–15], where they reported that hydrophobic interaction predominated sorption behavior of longer-chain PFASs, while short-chain PFASs were exclusively in water. In addition, previous work had also demonstrated that sorption of PFASs on sediment, soil or sludge increased with increasing solution Ca^{2+} concentration and decreasing pH value, in particular, the sorption behaviors of PFOS was expected to be strongly affected by solution conditions [10,11,15]. These observations suggested that electrostatic interactions were not negligible in the sorption of PFASs onto sediment. More evidences for both hydrophobic and electrostatic interactions involved in the sorption mechanism of PFASs were found by other researchers using aluminum-rich mineral-humic acid as a simulated sediment and sewage sludges as adsorbents [16,17]. However, to reduce detection errors most of these studies were performed with high initial concentrations of PFASs ranging from 1 to 1,000 μg/L that were much higher than their concentrations (ng/L) in the natural aquatic environment [8,18–20].

The sorptive behavior of PFASs onto natural aquatic sediment highly depends on many factors. Particularly with high concentration of PFASs to simulate the natural sediment–water interfacial processes is inadequate and possibly leads to unreliable conclusions [21], such as shorter equilibrium time [22] and strong linear correlations between partitioning of chemical and organic matter of sediment [11,23]. For this reason, this study examined the sorption behavior of PFOS and PFOA on riverine sediment using a low initial concentration so as to simulate natural aquatic interfacial process. The specific objectives of this study were: by batch partitioning experiments, to determine the sorption coefficient and isotherms of PFOS and PFOA onto the sediment and to examine the effect of pH, ionic strengths, and particle size and density of sediment on the sorption. These would provide supplementary data for understanding natural sediment–water interfacial process of pollutants.

2. Materials and methods

2.1. Reagents and chemicals

PFOA and PFOS were purchased from AccuStandard Inc. (USA). $^{13}C_4$-labelled PFOA and PFOS (Wellington's laboratory) were used as internal standards. Calcium chloride, sodium azide, hydrochloric acid and sodium hydroxide with analytical reagent grade were purchased from local chemical companies in Beijing, China. High performance liquid chromatography grade methanol and ammonium acetate were supplied by Fisher Company (USA). Milli-Q ultrapure water was used throughout the experiment. All standard solutions were prepared in methanol and stored in polypropylene (PP) bottles at 4°C.

2.2. Sediment collection and characteristics

Four sediment samples were collected from Daliao River systems, Northeast China. The sampling sites were chosen to reflect a variety of physicochemical properties potentially influencing partitioning of PFOA and PFOS. The surface sediments were collected in PP plastic bags using a grab sampler and transferred to the laboratory. The wet samples were air-dried, ground and sieved through 2 mm meshes. Total organic carbon (TOC) in sediments was measured using a high temperature TOC analyzer (Dohrmann DC-190) after the removal of inorganic carbon by adding diluted HCl until acidification reaction was completed. Specific surface area (SSA) was analyzed by N_2 sorption method. The pH value of sediment was measured in a 1:2.5 (w/w) mixture of the sediment with 0.01 mol/L of $CaCl_2$ solution by a pH meter. Total Fe and Mn contents in sediment were determined by an inductively coupled plasma - optical emission spectrometry Thermo Elemental (TJA) Iris Intrepid spectrometer following digestion of sediment with diluted aqua regia [24]. Sediment particle size fractionation was conducted by wet sieving the sediments through 40, 80, 120 and 240 meshes to obtain three size fractions, i.e., 0.42–0.20, 0.20–0.125 and 0.125–0.061 mm. The physicochemical parameters of sediments were shown in Table 1.

2.3. Batch partitioning experiments

The initial PFOA or PFOS concentration of 50 ng/L was selected for batch experiments based on the levels of both chemicals reported in aquatic environment [8,12,13]. Batch experiments included partitioning kinetics, effects of pH value, ion strength and particle size fractions, as well as sorption isotherm experiments [11,12]. The partitioning experiments were conducted in the 50 mL PP copolymer Nalgene centrifuge tubes. Duplicate sets of tubes containing 1.0 g (dry weight) of sediment and 30 mL of 0.01 mol/L $CaCl_2$ and 200 mg/L NaN_3 solution were spiked, respectively, with a certain amount of PFOA or PFOS, and equilibrated on the Thermostatic shaker at 150 rpm and 25°C. The centrifuge tubes with initial PFOA or PFOS concentration of 50 ng/L were shaken for 0–72 h and periodically removed at selected time intervals of 0, 0.5, 1, 2, 4, 8, 12, 24, 36, 48, 60 and 72 h. The effects of pH, ion strength and sediment particle size on partition behavior were investigated by batch sorption equilibration experiment at initial PFOS and PFOA concentrations of 50 ng/L. The pH values of aqueous solution were adjusted in the range of 3–9 with dilute NaOH or HCl. The concentrations of $CaCl_2$ were 0.001, 0.01 and 0.1 mol/L. The sorption isotherm experiments were carried out with concentrations of

Table 1
Physicochemical property of the sediments

Parameters	Sediment 1	Sediment 2	Sediment 3	Sediment 4
pH value	7.58	7.38	7.93	7.49
TOC (mg/g)	8.09	7.98	7.97	80.4
SSA (m^2/g)	104	66.2	52.1	48.1
Fe (mg/g)	45.8	26.9	19.7	24.3
Mn (mg/g)	0.82	0.38	0.40	0.34

PFOS or PFOA ranging from 50 to 500 ng/L and at equilibration time of 48 h. After shaking, the tubes were centrifuged at 1,600 rpm for 15 min and the supernatants were transferred to the new PP tubes for the extraction and analysis.

For each sample batch, blank samples were prepared using 30 mL of ultrapure water and treated exactly in the same way as the samples. The control experiment in the absence of sediment showed that the total PFOS or PFOA losses were below 5%, which was neglected in the experiment.

2.4. Extraction and determination of PFOA and PFOS

The supernatant was passed through Oasis HLB cartridge (Waters, with 500 mg of sorbent and 6 mL of volume) for the extraction of PFOA and PFOS. The cartridges were, first, cleaned by dichloromethane and methanol, respectively, to remove residual impurities, subsequently preconditioned by 5 mL each of methanol and ultrapure water [19,25]. After extraction, the cartridges were rinsed with ultrapure water, evacuated to dry for 30 min and eluted with 3 × 5 mL methanol into 15 mL PP tubes. The elution gathered was dried by nitrogen blowing, and then 1 mL of methanol–H_2O (v:v, 1:3) and 10 µL of 150 µg/L internal standard solution were added for ultra-performance liquid-chromatography tandem mass spectrometry analysis.

The concentrations of PFOS or PFOA were determined by UPLC-Xevo TQD (Waters, USA). An Acquity UPLC® BEH C_{18} column (2.1 × 50 mm, 1.7 µm) was used for the separation of analyte. Tandem mass spectrometry was conducted on triple quadrupole mass spectrometer (Xevo TQD, Waters, USA) equipped with an electrospray ionization source. The mobile phases were 2 mmol/L of NH_4OAC in water (A) and in methanol (B). The detailed analytical parameters of the target compounds were described in the previous study [25].

2.5. Sorption data fitting and partition coefficient

Sorption isotherms were constructed by plotting C_{sed} vs. C_w for PFOA or PFOS-sediment batch sorption system. Freundlich model was used to fit sorption data, as described by Eqs. (1) and (2):

$$C_{sed} = K_f (C_w)^{1/n} \tag{1}$$

or

$$\log C_{sed} = 1/n \log C_w + \log K_f \tag{2}$$

where C_{sed} and C_w are concentrations of PFOA or PFOS in sediment (ng/g) and the solution (ng/L) at equilibrium time; K_f is a Freundlich constant representing the sorption capacity and n is the Freundlich exponent depicting the nonlinearity of sorption.

The interaction of PFOA or PFOS between sediment and water can be described by its partition coefficient (K_d):

$$K_d = C_{sed}/C_w \tag{3}$$

By combination of Eqs. (1) and (3), the K_d values were calculated using Eq. (4), which were equilibrium concentration dependent:

$$K_d = K_f (C_w)^{(1-n)/n} \tag{4}$$

Previous studies have shown that the fraction of organic carbon (f_{oc}) has a significant effect on the partition of PFOA or PFOS onto sediment [11]. Thus, organic carbon normalized partition coefficient (K_{oc}) was calculated by the following Eq. (5):

$$K_{oc} = K_d/f_{oc} \tag{5}$$

3. Results and discussion

3.1. Sorption kinetics

Fig. 1 shows the sorption kinetics of PFOA and PFOS onto sediment. It was obvious that the sorption process could be divided into three apparent stages: a rapid sorption process, a slow sorption process and sorption equilibrium process. The rapid sorption occurred within 10 h for PFOA and 5 h for PFOS. Between 10 (or 5) and 40 h, the sorption rate for PFOA or PFOS gradually reduced, and then equilibrium was achieved after approximately 40 h for both chemicals.

This result was slightly different from the previous study, where equilibrium time for PFOS and PFOA was shorter, e.g., 4 and 8 h, respectively, when using higher initial concentration (5 µg/L) of PFOS and PFOA [22]. This indicated that the initial concentration of pollutants was one of impact factors controlling their sorptive kinetics between water and sediment. Simulating natural sediment–water interfacial process of pollutants with higher concentration than environmental level would lead to unrealistic results. Therefore, at low concentration of PFOS and PFOA (e.g., 50 ng/L), 48 h of equilibrium time was adopted for the batch sorption system.

3.2. Effect of solution pH

The effect of pH value on the sorption of PFOA and PFOS is shown in Fig. 2. It was observed that solution pH values in studied range influenced sorption of PFOA and PFOS onto the sediment to some extent. It was well known that the specific surface species could be formed on the natural sediment

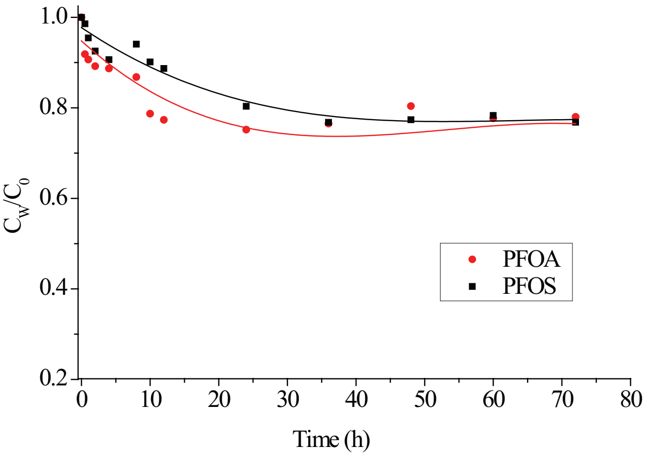

Fig. 1. Sorption dynamic curve of PFOA and PFOS onto the sediment (25°C).

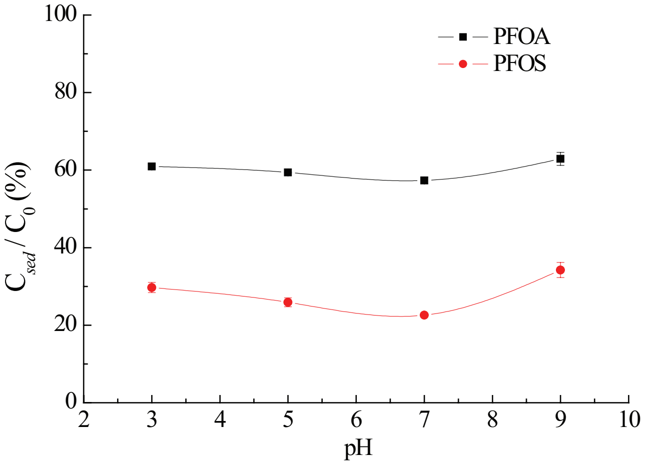

Fig. 2. Effect of pH value on the sorption of PFOS and PFOA.

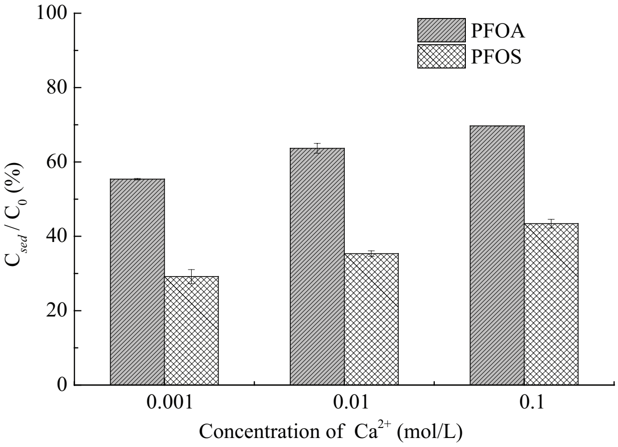

Fig. 3. Effect of Ca^{2+} on the sorption of PFOA and PFOS.

surface. Solution pH value would affect specific pH-dependent reactions on sediment surface such as ligand exchange, electrostatic interactions and hydrophobic effect [8,26]. Obviously, with increasing pH values between 3 and 7, the sorption of PFOA and PFOS onto the sediment slightly reduced, which could be explained by the electrostatic interaction of PFOA and PFOS anionic species with the positively charged sediment surface at lower pH value. It was also noted that a slight increase in sorption of PFOS and PFOA on sediment was found at solution pH over 7. The different sorption trend within studied pH values was likely due to the presence of the point of zero charge (pH_{pzc}) on sediment that was usually from 4 to 7.5. The results differed from Higgins and Luthy's report [11], where using higher concentration of PFOS and PFOA (10 nmol), sorption increased with decreasing pH and a significant drop was found in the observed K_d for both chemicals at the higher pH value. The observation in this study could be ascribed to strong sorption mechanism of both electrostatic attraction and ligand exchange at low concentration level of target compounds. Although more negative charges on sediment surface occurred with pH value increase, resulting in weak electrostatic attraction toward PFOA and PFOS anionic specie, ligand exchange between pollutant anionic specie and negative ligands on surface of sediment might be predominant on interfacial reactions, leading to enhanced sorption of PFOS and PFOA onto sediment with increased pH value [10].

3.3. Effect of ion strength

Fig. 3 reveals the sorption trends of PFOS and PFOA on sediment at Ca^{2+} concentration from 0.001 to 0.1 mol/L and solution pH value of 7. It indicated that the sorption of PFOS and PFOA increased with increasing Ca^{2+} concentration, which was in accord with previous reports by Higgins and Luthy [11] and by Chen et al. [27]. The effect of Ca^{2+} concentration on sorption percentage of PFOS and PFOA was caused by several reasons. One was linked to reduced solubility of PFOS and PFOA at high ionic strength, thereby increasing hydrophobic interaction. Chen et al. [27] have reported that sorption-enhancing impact of Ca^{2+} concentration was possibly related to the concentration of PFOS and PFOA in the aqueous phase with much stronger impact at low PFOS and PFOA concentration. Furthermore, with divalent cation Ca^{2+} increasing, the electrostatic repulsion between anionic PFOS or PFOA molecules and the negatively charged sediment surface was reduced because of neutralization of the negative charges on the sediment surface by high Ca^{2+} concentration, which promoted the sorption of PFOS and PFOA on the sediment. Beyond those Ca-bridging effect initiated by cations in the solution needed to be taken into consideration [10]. It was widely reported that divalent cations, acting as the bridge, were able to shift the negative sites into positive ones on adsorbent surfaces so as to electrostatically attract PFASs [4]. You et al. [23] also observed that sorption of PFOS on sediment increased when the concentration of $CaCl_2$ increased from 0.005 to 0.5 mol/L, suggesting that Ca-bridging effect was responsible for this result. Therefore, the formation of Ca-bridging with sediment might influence the sorption of PFOA and PFOS onto sediment.

3.4. Effect of particle size

The particle size-dependence of sorption for PFOA and PFOS is shown in Fig. 4. The sorption percentage of target contaminants measured at pH 7 decreased with increasing particle size of sediment. The results might be attributed to SSAs and physicochemical properties of different particle size. The SSAs were decreased with the increased grain size of sediment while the smaller particles would have larger external surface areas [28]. The diverse compositions and properties of the fractionated sediment, such as organic carbon fraction, debris of wood and stalks, mineral grains, might result in different sorption capacity for PFASs [14].

On the other hand, the compact organic matter was possibly involved with the smallest sediment particles. It has been reported that organic matter enhanced the sorption capacity of pollutants onto sediments [3,22,27]. As shown in Fig. 5, the sorption of PFOS and PFOA on sediments was somewhat related to organic matter, indicating that hydrophobic partitioning played a role in the interaction between target pollutants and sediment. Therefore, the combination between small-sized sediment and organic carbon was contributed to the sorption of PFOA and PFOS on sediment, which was consisted with the results reported by Zhao et al. [14].

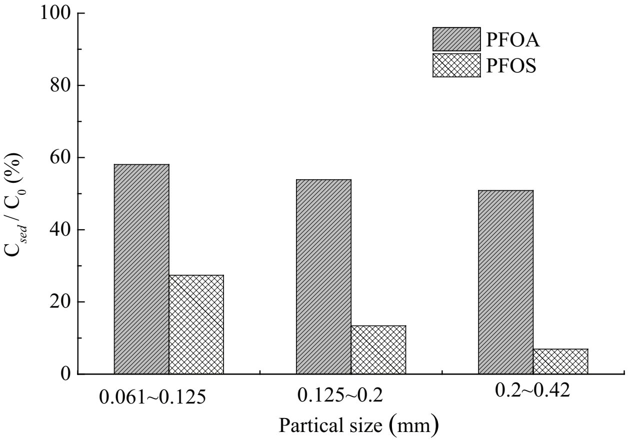

Fig. 4. Effect of particle size on the sorption of PFOA and PFOS.

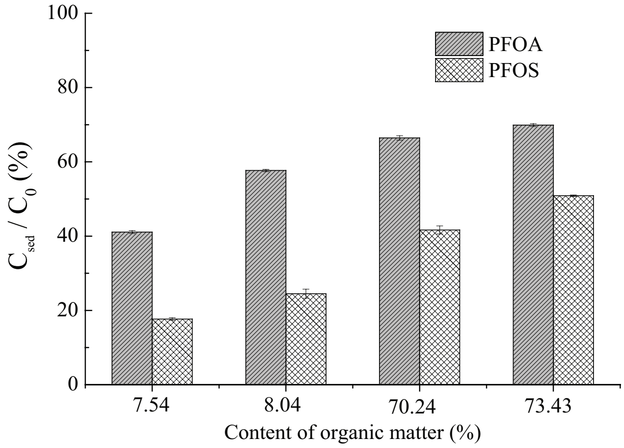

Fig. 5. Effect of organic matter content on sorption of PFOA and PFOS.

Although the sorption of PFOA or PFOS slightly enhanced with increasing organic carbon content of sediment, there was no significant correlation between K_d and organic carbon content. This was different from previous studies [11,23], which using high concentration of PFASs (0.5–10 mg/L) strong linear relations were observed between K_d of target PFASs and organic carbon content, indicating partitioning interactions as the main sorption mechanism for PFASs.

3.5. Sorption isotherms

The sorption isotherms of PFOA and PFOS on sediments are shown in Fig. 6. Equilibrium sorption data were calculated based on the Freundlich model. Over the studied concentrations range of PFOA and PFOS, all experimental data were fitted very well by the nonlinear Fruendlich type sorption isotherm with the regression coefficients (R^2) of 0.97 for PFOA and 0.95 for PFOS, respectively. For both PFOA and PFOS, the sorption coefficients (K_f) were 0.0051 and 0.0012, and the nonlinearity of sorption ($1/n$) were 0.84 and 0.93, respectively, indicating the low sorption capacity and nonlinearity degree of pollutants toward sediment. Regarding nonlinear sorption isotherms, the partition coefficients K_d and K_{oc} values were function of the solute concentration, ranged from 0.0012 to 0.0027 and 0.15 to 0.36 for PFOA, and 0.00061 to 0.00083 and 0.081 to 0.114 for PFOS.

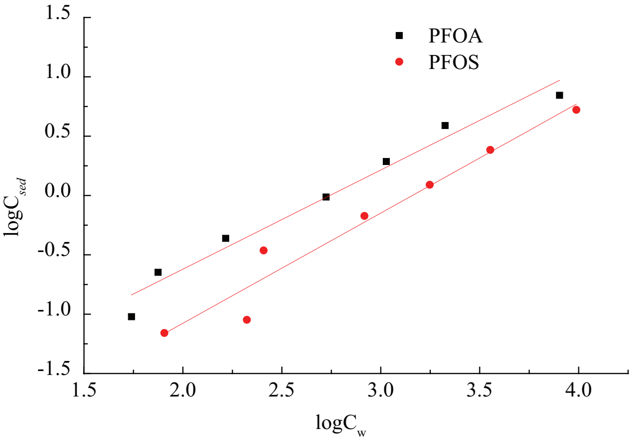

Fig. 6. Sorption isotherms of PFOS and PFOA on the sediments.

This nonlinearity of sorption behavior was mainly attributed to organic and mineral matrices involved in sediment. Given that hydrophobicity of perfluorinated chain and hydrophilicity of sulfonate or carboxylate [16], as well as diverse sorption sites on sediments, single driving force seems to be difficult to explain interfacial reaction between pollutants and sediments. The hydrophobic exclusion of perfluorinated chain, specific chemical interaction of sulfonate or carboxylate with sediment, and electrostatic attraction of charged solute molecule with the charged sediment surface might contribute to the nonlinear sorption of PFOA and PFOS with multi-molecular layers.

4. Conclusions

In natural aquatic system, the sorption of PFOS and PFOA onto sediments was very low. The kinetic study in batch experiments showed that the equilibrium between sediment–water interface for PFOS and PFOA with low initial concentration was achieved in longer period (48 h) than that with high initial level. The sorption behavior of PFOA and PFOS could be described by Freundlich equation. Both PFOA and PFOS displayed higher sorption capacity onto the sediments at high ionic strength and pH values, which was different from the results obtained with high initial concentration of PFOA and PFOS. The sediment particle with small size and high organic matter content enhanced the sorption of PFOA and PFOS due to the surface area and specific composition of particle; however, there was no strong linear relationship between sorption of target compounds and organic carbon content in sediments. This indicated the importance in simulating interfacial process of pollutants between sediment–water under natural aquatic conditions. The obtained results had important environmental implications: the leaching of PFCs from sediment would become more serious if background solution was neutral and with low ionic strength. The research would be benefit for the efficient removal of PFOS and PFOA in the natural aquatic systems.

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