Research article

Pollutant source or sink? Adsorption and mobilization of PFOS and PFOA from sediments in a large shallow lake with extended reed belt

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ABSTRACT

In this study, we i) assessed the occurrence of perfluorooctane sulfonate (PFOS) and perfluorooctanoate (PFOA) in sediments, pore water, and bulk water from three different areas in Lake Neusiedl, Austria, and ii) investigated mechanisms regulating adsorption and remobilization of these substances under different conditions via multiple lab-scale experiments. The adsorption capacity was mainly influenced by sediments' organic matter content, oxide composition, and pre-loading. Results suggest that a further increase of PFAS-concentrations in the open lake can be partly buffered by sediment transport to the littoral zone and adsorption to sediments in the extended reed belt. But, under current conditions, the conducted experiments revealed a real risk for mobilization of PFOS and PFOA from reed belt sediments that may lead to their transport back into the lake. The amount of desorbed PFAS is primarily dependent on water/sediment- or pore water/water-ratios and the concentration gradient. In contrast, water matrix characteristics and oxygen levels played a minor role in partitioning. The highest risk for remobilizing PFOS and PFOA was observed in experiments with sediments taken near the only major tributary to the lake (river Wulka), which had the highest pre-loading. The following management advice for water transport between high and low polluted areas can be derived based on the results. First, to reduce emissions into Lake waters from polluted tributaries like the Wulka river, we recommend diffuse pathways through the reed belt in the lake's littoral to reduce pollutant transport into the Lake and avoid high local sediment loadings. Second, water exchange with dried-up areas with probable higher loadings should be carefully handled and monitored to avoid critical back transport in the open lake. And third, general work in the reed belt or generally in the reed should be accompanied by monitoring to prevent uncontrolled remobilization in the future.

1. Introduction

Poly- and perfluoroalkyl substances (PFAS) are ubiquitous and persistent contaminants. Based on the available toxicological evidence, specific PFAS, namely PFOS and PFOA, have been regulated and partially phased out (2006/122/EC, 2006). However, due to chemical stability and legacy effects, they are still largely found throughout the environment worldwide (Ahrens and Bundschuh, 2014; Pan et al., 2014; Zoboli et al., 2019). Once present in natural water systems, PFAS can be adsorbed by sediments and thus removed from the water column. This makes them no longer bioavailable for most aquatic organisms (Simpson et al., 2021).

PFAS adsorption onto sediments is non-linear and was formerly well described by the Langmuir model, which assumes a monolayer formation on the sediment's surface (Brusseau, 2019; Xiao, 2015). Within concentration ranges of freshwater systems however, partitioning coefficients are standard parameters to assess PFAS adsorption on different sediments and under varying conditions. Due to the significant influence of organic matter, the sediment-water distribution coefficient (K_d) is often normalized to total organic carbon (TOC) content. Literature-ranges for log K_{OC} values of sediments in freshwater systems are 2.4–4.4 and 1.3–4.5 for PFOS and PFOA, respectively (Zareitalabad et al., 2013).

Environmental conditions, sediment and water characteristics influence the partitioning of PFAS between the solid and the bulk water phase (Miranda et al., 2021; Mussabek et al., 2020; Zareitalabad et al., 2013). The bonding of PFOS and PFOA to sediments is governed by i) hydrophobic interactions between the fluorinated carbon tail of PFAS

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and the organic matter fraction of the sediments and ii) electrostatic interactions between the polar head group of PFAS and charged fractions of the sediment, like ferric, aluminum and mineral oxides. Hydrogen bonding and ligand ion exchange minorly contribute to adsorption. Environmental conditions and solution-specific characteristics – e.g. pH, ionic strength, or redox potential – mainly affect electrostatic interactions and are thus crucial in determining adsorption in sediments with low organic matter content (Alves et al., 2020; Higgins and Luthy, 2006; Johnson et al., 2007; Li et al., 2018; You et al., 2010). Other aspects to consider are sediment-water ratios and competitive adsorption between PFAS and other compounds present in natural water matrices. Due to the interplay of all these factors, predicting PFOS and PFOA behavior in natural environments is still a significant challenge. Although PFAS pollution is widespread and environmental quality standards are exceeded worldwide (Gobelius et al., 2018; Zoboli et al., 2019), most guidelines on PFAS pollution focus just on remediation or management of heavily polluted sites. A comprehensive understanding of PFAS partitioning between sediments and the water phase is however needed to identify areas with enhanced risk of PFAS desorption from contaminated sediments and to derive recommendations for monitoring and management of water exchange between highly and less polluted areas (Ahmed et al., 2020; Mussabek et al., 2020).

Our study area was Lake Neusiedl, a steppe lake situated at the Austrian-Hungarian border. The mean depth is 1 m and the water quality is characterized by alkaline conditions (pH > 8), elevated salinity (1–2 g L^{-1}) and high inorganic turbidity. With its large reed belt covering more than 57% of the total area, Lake Neusiedl is an essential habitat for birds and a designated Natura 2000 area. While the southern part is a transboundary national park, the central and northern lake is intensively used for water sport, fishing and is generally promoted for tourism and leisure activities. The water balance is determined by precipitation and evaporation, with the latter typically exceeding the former. Persistent pollutants, therefore, tend to concentrate within the lake area (Zessner et al., 2019). Climate change, rising temperatures and extended dry periods in the summer are expected to decrease water levels in the long run, resulting in an additional concentrating effect. Further, lower water levels will lead to reduced water exchanges between the open lake and areas of the reed belt (Wolfram et al., 2019). These wind-driven exchange processes, which transfer water, sediments and sediment-bound contaminants into the reed belt where they settle due to lower wind forces, are however essential to maintain a good water quality in Lake Neusiedl (Fussmann et al., 2020; Herzig, 2014; Ramming, 1979). Sediment-water ratios in the reed belt are higher, and some areas tend to fall dry and show higher pollutant loadings. Reflooding of these sites may enhance the remobilization and transport contaminants back into the open lake (Fagbayigbo et al., 2021; Roberts, 2012).

Given these uncertainties, we designed and carried out multiple experiments to better understand the role of sediments as a sink for PFOS and PFOA and to assess the risk for remobilization due to changes in water matrices or environmental conditions. Results from adsorption and desorption experiments are discussed in the context of literature and used to understand the status quo of PFOS and PFOA partitioning in Lake Neusiedl and to derive recommendations for future water management in the lake. The specific objectives of this study were:

i) to assess the occurrence and partitioning of PFOS and PFOA in different lake compartments;
ii) to investigate the influence of oxic/anoxic conditions and of different water matrices on PFOS and PFOA adsorption and remobilization;
iii) to identify areas with a limited remaining adsorption potential and a high risk for desorption;
iv) to derive recommendations for future monitoring and water transport management between open lake areas and the reed belt.

2. Materials & methods

2.1. Study area and sampling campaigns

The study area of Lake Neusiedl is shown in Fig. 1, including satellite pictures of the three main sampling areas situated within the reed belt. Moerbisch is located on the west and Illmitz on the east side of the lake. At both sites, channels through the dense reed stands serve as connecting pathways for water and material transport between the open lake and pools within the littoral reed zone. The water quality at these sampling sites is influenced by wind-induced transport of lake water into the reed belt area. The third sampling area was in the Wulka estuary. The Wulka river is the only significant tributary to Lake Neusiedl (mean discharge 1.1 m^3 s^{-1}) and has a high share of treated wastewater (approximately 40%). Currently, most of the water of this tributary flows through a few channels to the open lake, whereas large parts of the water areas within the reed belt are hydrologically little or not connected at all to the open lake.

In seven sampling campaigns conducted between April 2018 and April 2019, we collected sediment and water samples in these three regions to explore the PFOS and PFOA sediment/water and sediment/pore water distribution and to conduct lab experiments. The supplementary information (SI) provides an overview of individual sampling points and further experimental and evaluation procedures. Sediment samples were taken with a core sampler of 6 cm diameter by Uwitec (Austria) from the less dense layer of the sediment phase (maximum sediment depth of 20 cm) and stored in polypropylene buckets. Immediately after arrival in the lab, larger parts of organic and inorganic matter, like plant-remains or stones, were removed and samples were homogenized. Part of the collected samples used in the lab experiments was centrifuged at 20 000×g for 15 min to obtain representative pore water samples for PFOS and PFOA analysis. All sediment samples were analyzed for their particle size distribution, organic carbon and nutrient content. Further, the oxide composition was assessed for sediments taken in the first round in 2018. PFOS and PFOA loading was evaluated for all samples, however, the analytical method applied in 2018 was not sensitive enough, and all sediment loadings were below the LOQ (0.5 µg kg^{-1}). Therefore, samples collected in 2019 were analyzed with a more sensitive method (LOQ = 0.00015 µg kg^{-1}). Water samples from the reed belt bulk water and the open lake were also collected into polypropylene bottles and analyzed for suspended solids, organic matter, nutrients, ion content and PFOS/PFOA. Section 2.3 describes the methods of all performed chemical analyses.

2.2. Lab experiments

Four different sediments from Illmitz (IL5), Moerbisch (MO3) and from two spots in the Wulka estuary (WU6 and WU2) were used in four experimental setups, which aimed to i) investigate adsorption kinetics and derive optimum duration of batch tests, ii) determine the remaining adsorption potential of sediments in batch tests with spiked samples (tests A), iii) investigate adsorption-desorption processes under oxic and anoxic conditions in batch tests with original environmental samples (tests B) and iv) investigate mobilization under changing conditions in long-term reactor tests.

2.2.1. Adsorption kinetics

The adsorption kinetics of PFOS and PFOA were investigated in experiments conducted with sediment from the area of Illmitz in 2018. The desired amount of sediment and water was inserted into high-density polyethylene (HDPE) bottles and mixed in a horizontal shaker CERTO-MAT® U (B. Braun, Germany) at 120 revolutions per minute (rpm) and room temperature (25 °C). Samples were taken in duplicates after 2, 4, 24, 28, and 48 h from the bulk phase. Equilibrium was reached after approximately 4 h, which agrees with equilibrium times reported in the literature (Johnson et al., 2007; Tang et al., 2010; Wang and Shih,
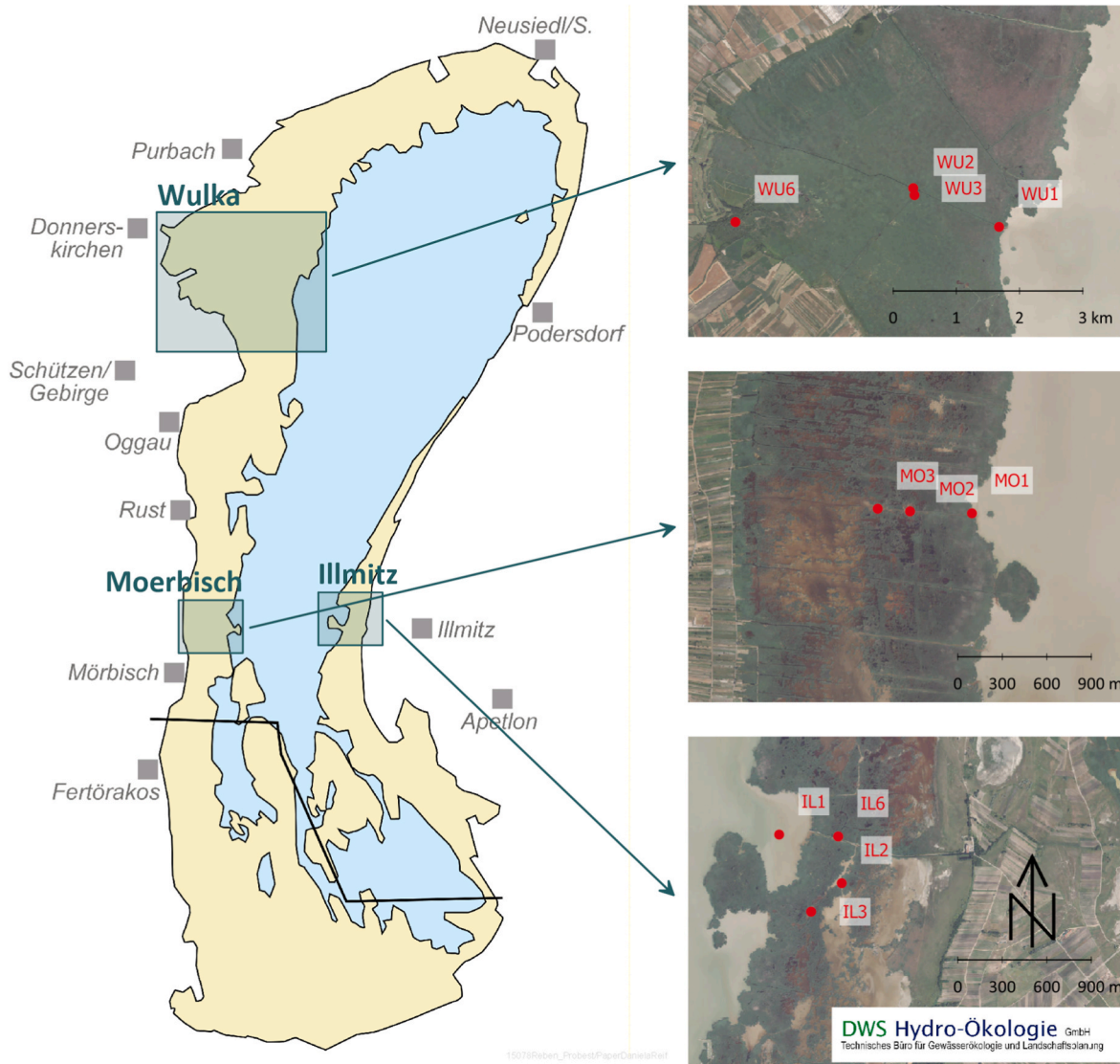
Fig. 1. Map and aerial representation of the sampling sites Moerbisch, Illmitz, and Wulka in the study area of Lake Neusiedl.

2011). However, to guarantee that experimental conditions reflect the selected ones and are not disturbed by e.g. unconsumed oxygen, a prolonged time of 72 h was applied in the following experiments. Results for the kinetic experiments are presented in the SI.

2.2.2. Adsorption batch tests

- *tests A with spiked PFAS matrices*

The adsorption tests were done in duplicates under oxic conditions and room temperature for five different sediment/water ratios between 10 and 1000 g L^{-1} dm and two blank samples without sediments which account for eventual adsorption of PFAS on the bottle walls. Initial concentrations were enriched by a factor of ten through addition of a concentrated PFOS (CAS: 1763-23-1, Merck)/PFOA (CAS: 335-67-1, Sigma-Aldrich) stock solution. Besides PFOS and PFOA, six major inorganic contaminants, namely mercury, cadmium, copper, lead, nickel, and zinc were included in the stock solution to better reflect the average composition of the lake water matrices and take competitive adsorption into account (for more information, see SI). The desired sediment and water volumes were transferred into 1 L HDPE bottles and mixed for 72 h on the horizontal shaker at 120 rpm and room temperature (25 °C). After the shaking had been stopped, sediments settled for approximately

15 min. Bulk samples were then taken and centrifuged at 10.00×g and 25 °C for 15 min to remove residual suspended solids. Analytical results were used to calculate the sediment-water partitioning coefficients of the sediments and assess their remaining adsorption potential (see section 2.4).

- *tests B with original samples*

Adsorption and desorption of PFOS and PFOA from sediments was investigated in another set of batch tests conducted similarly to the prior adsorption experiments, but with original water samples taken from the reed belt and the open lake in 2019. The tested sediment concentrations were between 20 and 250 g L^{-1} dm and included two additional blank samples of each matrix/condition. Oxic (condition A) and anoxic conditions (condition C) were realized by open 1 L HDPE bottles and closed 0.5 L HDPE bottles, respectively. Anoxic bottles were filled up to the top and closed. The remaining dissolved oxygen was consumed within 2–6 h, depending on the organic matter content of the individual sediment sample. Final concentrations were normalized to the initial ones (including pore water) and used to calculate the related partitioning coefficients.

2.2.3. Long-term reactor tests with different water matrices

Mobilization tests were performed in 24 L cylindrical stirred reactors with the same sediment and bulk water samples used in tests B. Additionally lake water and lake water diluted with deionized water, to simulate dilution of lake water by increased rainfall or lower concentrations due to decreasing emissions, were used in the tests for locations Illmitz and Moerbisch. Tests with the Wulka sediment WU2 were conducted with bulk water from the same spot and from a close sampling point (WU6-wulka river) as well as with lake water. All reactors were filled with wet sediment to a height of approximately 8 cm (about 6 kg for Illmitz and Wulka and 5 kg for Moerbisch). Water samples were added up to a total height of 24 cm, amounting to approximately 7 L. Oxygen concentrations and pH were measured daily at 10 cm above the sediment layer by the HQ90 hand sensor (Hach, USA). All reactors were stirred with a maximum flow velocity of 0.08 m s^{-1}. After three weeks, PFOS and PFOA concentrations were determined in the bulk samples from the reactors and compared to initial concentrations in the tested water matrices.

2.3. Analysis of water and sediment samples

The following section describes the methods used to analyze sediment and water samples. All analytical methods and results are described in SI.

2.3.1. Analysis of sediment characteristics

The particle size distribution was analyzed with a laser diffraction analyzer Mastersizer 2000 (Malvern Panalytical, USA) and repeated five times for two independent samples. The analysis provided the characteristic diameters (d_{10}, d_{50}, and d_{90}) and the specific surface of the sediments. The sediment's oxide composition was assessed by an X-ray spectrometer type PW 2400 (Philipps, Netherlands). Additionally, dry matter content and ignition loss at 1000 °C, referring to sediments' carbonate content, were determined. Chemical oxygen demand (COD) and share of total organic carbon (TOC) were analyzed according to DIN ISO 15705 and DIN EN1484, respectively. In addition, total Kjeldahl nitrogen (TKN) and total phosphorus were assessed according to DIN EN ISO 25663 and DIN EN ISO 6878, respectively.

2.3.2. Analysis of water characteristics

Within the water samples, we analyzed suspended solids (DIN 384089–2), dissolved (DOC) and total organic carbon (DIN EN 1484), ammonia (DIN EN ISO 11732), nitrate and nitrite (DIN EN ISO 13395), ortho-phosphate (DIN EN ISO 6878) and total dissolved phosphorus (DIN ISO 6878) contents. The ion balance was obtained according to DIN EN ISO 10304–1 (anions: chloride, sulfate) and DIN EN ISO 14911 (cations: sodium, potassium, magnesium, calcium).

2.3.3. Analysis of PFOS and PFOA

The collected sediment samples were eluted with distilled water in a volume ratio of 1:10 for 24 h. The eluate and water samples from 2018 were analyzed according to DIN 38407-F42. A linear trap ion trap mass spectrometer QTRAP 6500 (Applied Biosystems, USA) was used for detection. Limits of quantification (LOQ) in samples from 2018 were 0.025 μg L^{-1} for sediment and 0.004 μg L^{-1} for water samples for both compounds. EPA-method 537:2009 was applied to analyze eluate and water samples from 2019 to achieve lower detection limits of 0.002 μg kg^{-1} for sediment and 0.00015 μg L^{-1} for water samples. Pollutants were detected by Agilent 6495B Triple Quadrupole mass spectrometer (SCIEX, USA).

2.4. Data analysis

First, dry matter content of the sediments ($m_{sed,\ DM}$) in for each batch experiments was calculated by multiplying wet sediment mass (m_{sed}) and dry matter content (dm) (Eq. (1)).

$$m_{sed,\ dm} = m_{sed} \times dm \qquad \text{Eq. 1}$$

With the density of water (ρ_{H2O}) at 25 °C, the pore water volume V_{pw} and the total water volume V_0 could be calculated according to Eq. (2) and Eq. (3). V_W describes the volume of the added water.

$$V_{pw} = \left(m_{Sed} - m_{sed,dm}\right) \times \rho_{H2O} \qquad \text{Eq. 2}$$

$$V_0 = V_{PW} + V_W \qquad \text{Eq. 3}$$

Dry mass sediment concentration ($c_{sed,\ DM}$) was calculated from the dry mass of sediment applied in the experiments ($m_{sed,dm}$) divided by the volume of the tested water matrix (V_0).

$$c_{sed,\ dm} = \frac{m_{sed,dm}}{V_0} \qquad \text{Eq. 4}$$

Since concentrations of PFOS and PFOA are usually higher in pore water ($c_{pw,i}$) than in the bulk phase ($c_{w,i}$), the initial concentration of pollutants in the experiments was calculated according to Eq (5).

$$c_{0,i} = \frac{V_w \times c_{w,i} + V_{pw} \times c_{pw,i}}{V_0} \qquad \text{Eq. 5}$$

Alteration of sediments PFAS loading (Δq_i) was calculated based on concentration changes in the bulk phase between the initial and equilibrium conditions ($c_{0,i} - c_{eq,i}$) and dry matter content of the sediment according to Eq. (6).

$$\Delta q_i = \frac{1}{c_{sed,dm}} \times \left(c_{0,i} - c_{eq,\ i}\right) \qquad \text{Eq. 6}$$

Sediment-water distribution coefficients (K_d) and organic carbon-normalized coefficients (K_{OC}) were determined based on the linear sorption model (Henry isotherm) according to Eq. (7) and Eq. (8), where f_{OC} is the fraction of organic carbon. The initial loading ($q_{i,0}$) was unknown for the first batch-tests at spiked concentrations and could therefore not be considered, but was taken into account during evaluation of the second tests. The 'natural-derived' distribution coefficients were determined according to Ahrens et al. (2010), based on the obtained loadings and concentrations of sediment, pore water, and water samples from the sampling campaign in 2019.

$$K_d = \frac{q_0 + \Delta q_i}{c_i} \qquad \text{Eq. 7}$$

$$K_{OC} = \frac{K_d \times 100}{f_{OC}} \qquad \text{Eq. 8}$$

Further statistical analysis, like the calculation of Pearson correlation coefficients, was performed with Excel.

3. Results

3.1. Characteristics and occurrence of PFAS in sediments and water samples

All results of the characterization of sediment and water samples are reported in the SI. The most important parameters affecting the adsorption of PFAS, namely particle size and specific surface, organic matter content and the presence of certain oxides, are summarized here. The highest fraction of organic carbon, the smallest particle sizes, and the largest specific surface area were found in sediment samples from the river Wulka, followed by Moerbisch and Illmitz. The sediment from Illmitz contained a higher fraction of sand (59% SiO_2) and oxide composition differed significantly from the composition at the other two sites. Moerbisch (34% CaO) and Wulka sediments (32% CaO) are calcite dominated and had approximately the same relative content of iron (5–6%) and aluminum (2%) oxides.

Organic matter content was highest in bulk water samples from Illmitz and lowest in Wulka water. Nutrient concentrations in all samples

were inconspicuous. Most elevated chloride, sulfate, sodium, potassium, and magnesium concentrations were found for Illmitz > Moerbisch > Lake > Wulka. Nevertheless, the highest calcium concentrations were detected in Wulka water samples, which are mainly by the water of the Wulka river and less by Lake water.

Fig. 2 shows boxplots representing PFOS and PFOA concentration ranges of sediment, pore, and bulk water samples. PFOA concentrations were generally higher than PFOS concentrations and for both compounds' levels were lower in bulk than in pore water.

Table 1 shows mean loadings and concentrations clustered by sampling site. Highest sediment loadings for both compounds were detected in the samples from Wulka. This was expected due to the river's high share of treated wastewater. However, the load decreased within the Wulka area with increasing distance from the main channel. The highest loadings of 0.137 µg kg^{-1} PFOS and 0.453 µg kg^{-1} PFOA were found in samples from point WU1, directly within one of those main flow paths. Despite the high content of wastewater in the Wulka river, mean concentrations in the Wulka bulk water were lower than in the area around Moerbisch. The 'naturally derived' K_{OC} partitioning coefficients, based on sediment and bulk concentrations, were 1.8/2.2 (PFOS/PFOA) for Illmitz, 1.6/1.9 (PFOS/PFOA) for Moerbisch, and 2.4/1.3 (PFOS/PFOA) for Wulka. The environmental quality standard for PFOS of 0.00065 µg L^{-1}(annual average) was exceeded in all lake compartments.

3.2. Sediment-water distribution coefficients (log K_{OC}) in adsorption tests with spiked concentrations

Lab tests (tests A) investigated the remaining adsorption potential of sediments from Illmitz, Moerbisch, and Wulka. Initial loadings of the investigated sediments were below the LOQ of the characterization method of 0.50 µg kg^{-1}. Therefore, the calculated loading increases of PFOS and PFOA per gram of dry sediment are given as a loading change or (Δq). Further, we used the initial loading of sediments used in the tests B to assess the impact of an initial sediment loading on log K_{OC} values for Illmitz and Moerbisch. Moreover, half of the LOQ value of the analysis (0.25 µg kg^{-1}) served as the maximum possible value for the calculation of the log K_{OC}, whereas the loading change served as the minimum one.

Initial PFOS concentrations after spiking were 0.027 µg L^{-1} for Illmitz, 0.071 µg L^{-1} for Moerbisch, and 0.043 µg L^{-1} for Wulka water samples. For PFOA, initial concentrations were 0.04 µg L^{-1} for Illmitz, 0.029 µg L^{-1} for Moerbisch, and 0.043 µg L^{-1} for Wulka. pH changes
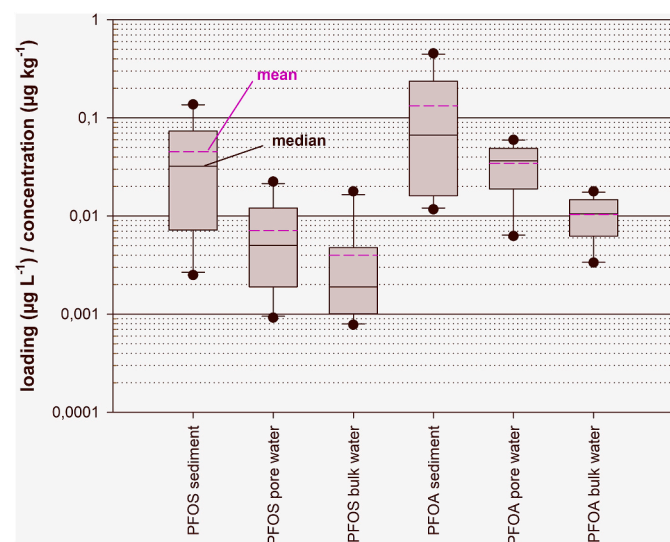

Fig. 2. PFOS and PFOA concentrations in sediments, pore water, and water samples 2019 (n = 10).

Table 1

Loadings and concentrations of PFOS and PFOA in sediment, pore water and bulk water samples.

	n (−)	sediment (µg kg^{-1})	pore water (µg L^{-1})	bulk water (µg L^{-1})
PFOS				
Illmitz	3	0.01670 ± 0.01351	0.00190 ± 0.00057	0.00173 ± 0.00054
Moerbisch	3	0.02307 ± 0.02928	0.00857 ± 0.01200	0.00659 ± 0.00971
Wulka	3	0.09923 ± 0.05454	0.01068 ± 0.00264	0.00474 ± 0.00043
Lake	5			0.00110 ± 0.00053
PFOA				
Illmitz	3	0.08910 ± 0.09010	0.03220 ± 0.00749	0.01280 ± 0.00175
Moerbisch	3	0.16337 ± 0.19456	0.04203 ± 0.01766	0.01423 ± 0.00394
Wulka	3	0.16813 ± 0.24692	0.02015 ± 0.02275	0.00471 ± 0.00208
Lake	5			0.00890 ± 0.00376

during the experiments depended on the sediment/water ratio and the sampling site. It remained constant during the experiments conducted with the sediment from Illmitz (initial pH: 8.71), but increased from 7.65 to a maximum of 8.45 during the tests with the Moerbisch sediment. For Wulka, the pH slightly decreased from 8.12 to a minimum of 7.89. SI provides additional information about the experimental conditions.

The observed PFOS and PFOA loading changes in the lab tests are illustrated in Fig. 3. Adsorption to the sediment from Moerbisch was significantly higher than to the sediment of the other two regions. The sediment-water distribution factor log K_{OC} for PFOS, including all collected data points and without assuming initial loading, was 3.3 (R^2 = 0.82), 4.0 (R^2 = 0.92), and 2.9 (R^2 = 0.64) for the sediments from Illmitz, Moerbisch, and Wulka, respectively. The distribution factors would rise to 3.3 and 4.0 for Illmitz and Moerbisch, concerning the initial loadings determined in 2019. The maximum sediment-water distribution factor log K_{OC} factors are 3.7 for Illmitz, 4.1 for Moerbisch, and 3.6 for Wulka, assuming an initial loading of 0.25 µg L^{-1} (half of the LOQ of the applied method).

For PFOA, the distribution coefficients were lower for Illmitz (2.5/R^2 = 0.58) and Wulka (2.1/R^2 = 0.89), but higher for Moerbisch (4.5/R^2 = 0.60). Assuming initial concentrations are in a similar range as detected in the following sampling campaigns, sediment-water distribution coefficients would increase to 2.6 and 3.8 for Illmitz, and Moerbisch, respectively. Maximum calculated log K_{OC} values for an initial PFOA concentration of 0.025 µg L^{-1} are 3.2 (Illmitz), 4.5 (Moerbisch), and 2.8 (Wulka).

3.3. Adsorption/desorption of PFAS in different water matrices

The normalized equilibrium concentrations at equilibrium conditions during tests B are given in Fig. 4. Due to different amounts of pore water included in the wet sediment samples, the initial concentration in each test bottle was influenced by the applied sediment-water ratio. PFOS and PFOA content was in most cases higher in pore water than in bulk water, and initial concentrations increased with a higher dry matter content. Initial concentrations at the lowest and highest applied dose are summed up in the SI.

Adsorption and desorption were significantly influenced by sediment-water ratios, the investigated water matrices, and to a lesser extent by the oxygen content. In general, normalized equilibrium PFAS concentrations increased with a higher dry matter content. For Illmitz, at dry matter concentrations below 200 g L^{-1}, normalized PFOS and
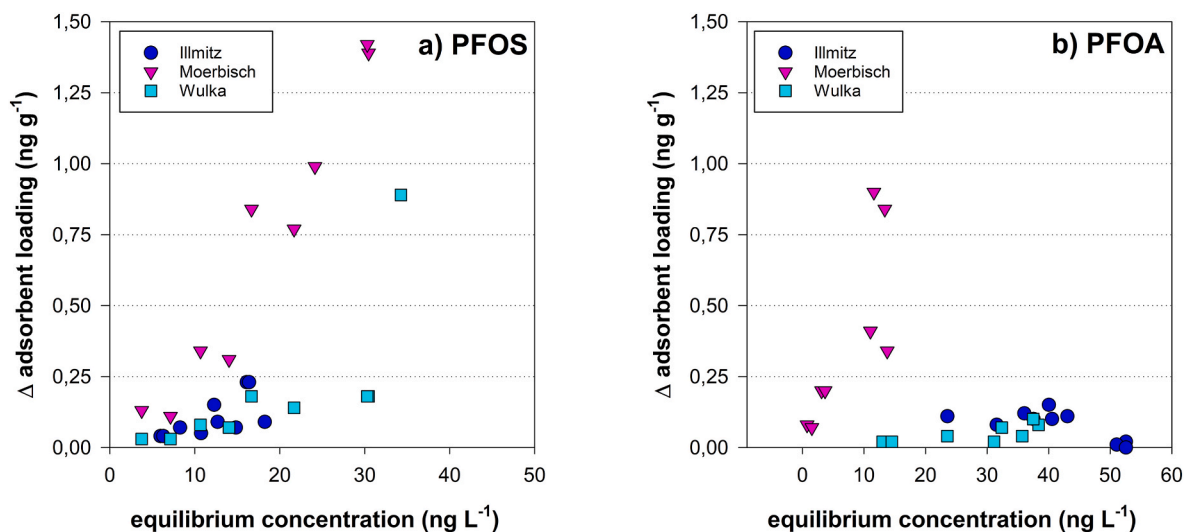
Fig. 3. Sediment loading changes of PFOS (a) and PFOA (b) dependent on the equilibrium concentration.

PFOA concentrations were below 1, which means that final bulk concentrations were lower than initial ones (including pore water). However, for the other samples from Moerbisch and Wulka, normalized effluent concentrations were partly above 1, implying that PFOS/PFOA desorbed from the sediments into the water phase.

We observed the most significant difference for PFOS and PFOA adsorption for Moerbisch sediment, which showed a significantly lower adsorption potential under anoxic conditions in both matrices. In tests related to the Wulka region, desorption was observed under both conditions and matrices, though it was lower than in the setup with lake water for PFOA.

Though the normalized concentration of some tests revealed higher desorption for higher sediment ratios, sediment's loading with PFOS and PFOA was still increasing and positive K_d and log K_{OC} values could be obtained for most conditions. The log K_{OC} values are given in Table 2, K_d can be found in SI.

A comparison of log K_{OC} in different water matrices shows that adsorption and desorption of PFOS and PFOA from sediments are very much dependent on site-specific characteristics and are consistently highest for Moerbisch. In experiments with Illmitz sediment, the log K_{OC} for PFOS was higher in lake water than in reed belt water under both conditions. For Moerbisch on the other hand, it was higher in reed belt water and under oxic conditions. A higher log K_{OC} was observed in reed belt water under oxic conditions and in lake water under anoxic conditions during the Wulka tests. This contradictory behavior was also observed for PFOA with higher log K_{OC} in lake water for Illmitz, in reed belt water under oxic conditions for Moerbisch, and in reed belt water for Wulka.

3.4. Mobilization experiments

Initial, pore water and final concentrations of the mobilization experiments conducted in the reactors are given in Fig. 5. Desorption and diffusion of pollutants from the sediment/pore water into the bulk layer was observed in all setups, even in those where bulk water from the same spot was used. A significant influence of sediment-water ratios was already observed in tests B and may lead to a change of equilibrium, or share of pore water, in the experimental setup compared to the real environment. The highest PFOS and PFOA concentration increase was found in tests with the lowest initial bulk concentrations, in most cases, diluted lake water. pH, which influence adsorption/desorption of PFAS, was very similar during the Illmitz and Moerbisch experiments (between 8.3 and 8.5) and remained constant. pH in water samples used in Wulka tests were between 7.8 (Wulka water) and 8.6 (lake water). The main

difference between water characteristics before and after the experiments was the content of suspended solids, which settled and were significantly lower in the final samples.

4. Discussion

4.1. Experimental data quality

Normalizing the partitioning coefficient to the organic matter of the sediment leads to a significant reduction of the scattering, as has been reported in the literature (Higgins and Luthy, 2006; Zareitalabad et al., 2013). Therefore, we considered only the normalized log K_{OC} values for further discussion. All obtained results are generally comparable with those reported for freeze-dried (and pre-washed) samples, the wet sediment samples' inhomogeneity probably leads to more scattering and lower coefficients of determination.

4.2. Occurrence and sediment-water partitioning of PFOS and PFOA in different lake compartments

The PFOS loading of the Wulka sediment (0.037 μg kg^{-1}) was approximately ten times higher than the loadings of the Illmitz (0.003 μg kg^{-1}) and Moerbisch ones (0.004 μg kg^{-1}). However, the initial loadings of PFOA were in a similar range (0.012–0.016 μg kg^{-1}). Higher loadings for Wulka sediments were expected since the river contains a high share of treated wastewater, and represents a crucial pathway for PFAS emissions into Lake Neusiedl (Zessner et al., 2019). Fig. S4 in the SI shows a comparison of sample and experimentally determined log K_{OC} values and reveals a high difference between naturally derived coefficients and tests-based ones for Illmitz and Moerbisch. For Wulka they were in a narrower range. In most cases those derived from environmental samples were lower than the experimentally determine one's. Log K_{OC} values for PFOS were generally higher than those for PFOA, which is in line with literature and can be explained by a stronger bonding of perfluoro sulfonic acids (hard base) to oxide surfaces than of per fluorinated carboxylic acids (soft base), and thus enhanced electrostatic interactions (Du et al., 2014; Gagliano et al., 2020; Xiao et al., 2011). The highest log K_{OC} values for both compounds were obtained in experiments with Moerbisch sediments. This means that, among the three investigated regions, Moerbisch provides the highest remaining adsorption capacity for PFOS and PFOA.

The log K_{OC} values for Wulka were significantly lower. This was unexpected since the sample had the highest organic matter content, which typically favors the adsorption of PFAS and provides active
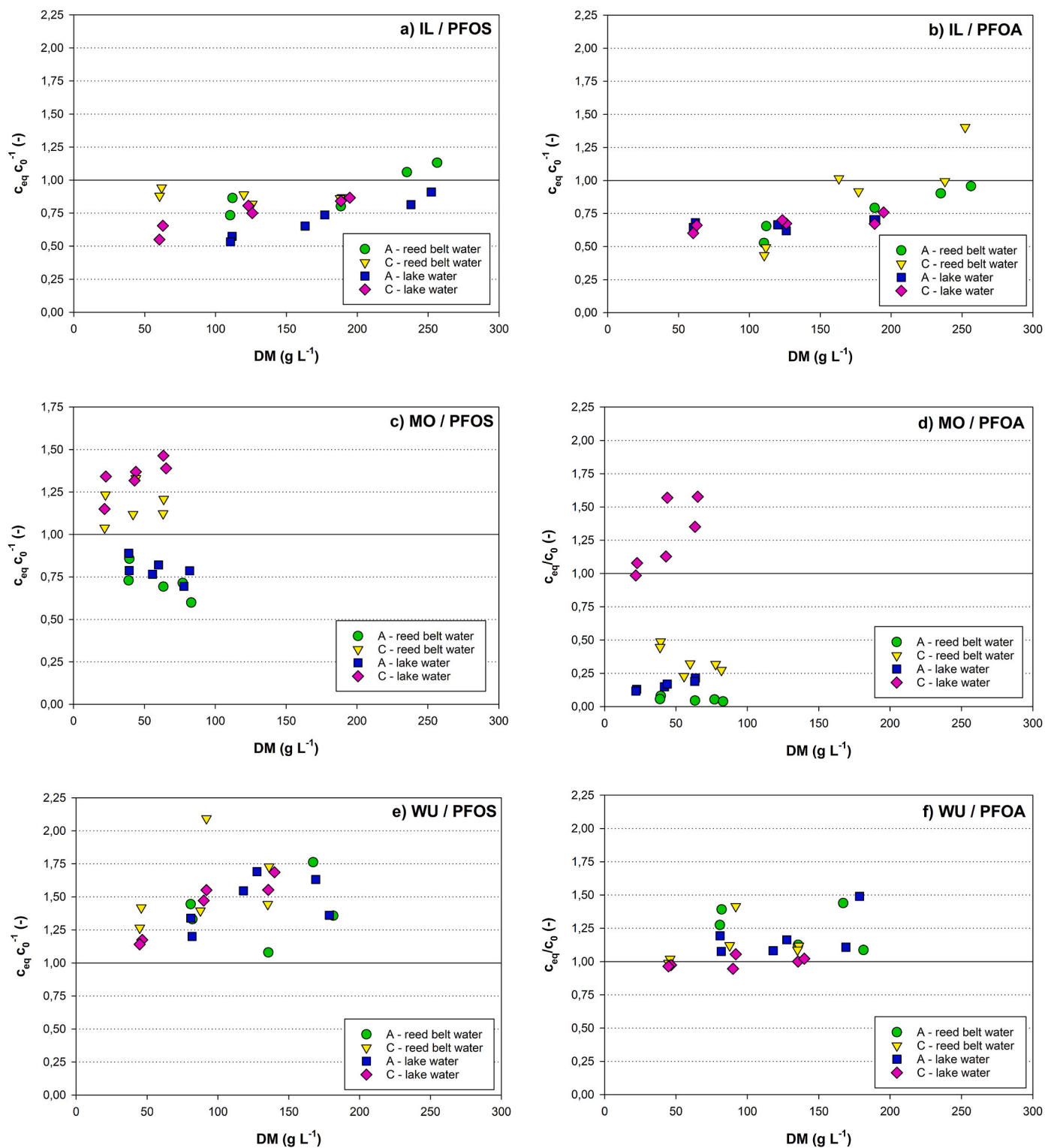
Fig. 4. Normalized PFOS and PFOA equilibrium concentrations in the bulk phase of experiments conducted with sediment samples from Illmitz (IL), Moerbisch (MO), and Wulka (WU) under oxic (A) and anoxic (C) conditions dependent on dry matter (DM) sediment content in the test batches.

centers for hydrophobic bonding (Higgins and Luthy, 2006). The relative oxide content of Wulka sediment was similar to the analyzed one from Moerbisch. Both contain approximately the same share of calcium, magnesium, ferric, and aluminum oxides, which should theoretically enhance electrostatic interaction and, thus, total adsorption (Alves et al., 2020; Johnson et al., 2007). However, the initial loading of Moerbisch sediments was significantly lower than the Wulka ones and might

represent a reason for lower sediment-water distribution coefficients since the adsorption of PFAS is non-linear and dependent on the applied initial concentration (Brusseau, 2019; Xiao, 2015).

Further, competitive adsorption might play a role. It was only studied for different PFAS compounds and showed that long-chained PFAS outcompete short-chained ones, though it was influenced by high sodium and hydrogen ion concentrations (Xiao et al., 2011). To the best of

Table 2
Calculated log K_{OC} values for oxic and anoxic conditions and tests with bulk water and lake water.

	reed belt water				lake water			
	oxic		anoxic		oxic		anoxic	
	log K_{OC}	R^2	log K_{OC}	R^2	log K_{OC}	R^2	log K_{OC}	R^2
PFOS								
Illmitz	3.0	0.79	3.1	0.63	3.3	0.85	3.3	0.63
Moerbisch	3.2	0.96	K_d = neg.	0.03	3.1	0.97	K_d = neg	0.73
Wulka	2.3	0.87	2.1	0.60	2.3	0.92	2.3	0.88
PFOA								
Illmitz	2.9	0.37	3.3	0.79	2.2	0.02	3.2	0.69
Moerbisch	4.6	0.96	4.2	0.65	3.6	0.94	K_d = neg	0.76
Wulka	1.3	0.63	1.8	0.46	K_d = neg	0.30	1.9	0.90

our knowledge, competitive adsorption between PFAS and other pollutants, like heavy metals, which are present in a much higher concentration range in the tested matrix, has not been studied so far. The experiments with Illmitz sediments, which had the had the lowest specific surface, organic matter content, and ferric or aluminum oxides shares, resulted in intermediate log K_{OC} values. This sediments main oxide component was silicon oxide (SiO_2), which is negatively charged and thus does not support electrostatic interactions (Hellsing et al., 2016). Moreover, within the water samples, the highest concentrations of divalent ions (calcium and magnesium) and dissolved organic carbon (humic acids) were found in the bulk water samples used in the experiments and may influence the surface characteristics of the sediments and electrostatic interactions (Tang et al., 2010). Literature sources suggest that high organic matter content (humic/fulvic acids and others) in the liquid phase lowers PFAS adsorption (Mukhopadhyay et al., 2021), however the exact role is not yet clear.

4.3. Effect of environmental conditions and matrix changes

The influence of the sediment-water ratio on adsorption/desorption is mainly attributed to a lower share of test water to pore water at higher wet sediment concentrations. Sampling campaign results reveal that the pore water concentrations of PFOS and PFOA are approximately 0.3 logs higher than concentrations in the bulk water phase. Exchange and transport of pollutants within sediments layers are generally not well studied, but some authors suggest a decrease in sediment loading with increasing sediment depth (Ahrens et al., 2010; Mussabek et al., 2020).

Normalized final PFOS and PFOA concentrations of the reactor tests, which reflected the highest sediment-water ratio assuming the whole sediment layer (8 cm) is participating in the exchange processes, agreed with observed trends from fully mixed batch test. This suggests that a large part of the total sediment layer in the reactors participated in PFOS and PFOA exchange.

A reversal of the concentration gradient was the main driver for the mobilization of PFOS and PFOA. We observed enhanced desorption in diluted lake water, the matrix with the lowest initial PFAS concentrations. Water phase characteristics seem to have a minor influence on the adsorption.

An apparent effect of anoxic conditions on the mobility of PFAS was only observed in experiments conducted with original samples from Moerbisch (tests B), where a comparably high increase of pH was noted. The sediment contained a higher share of ferric and aluminum-oxides, which increased electrostatic interaction, which in turn seems to represent a more important binding mechanisms for these sediments with low fractions of organic carbon (Du et al., 2014). Sorption of PFAS under oxic/anoxic conditions was also studied by other authors who also observed contradictory results for sediments with different characteristics (organic matter content, metal oxides), and ostensibly different binding mechanisms (Ololade et al., 2016).

4.4. Implications for open water and reed belt management of Lake Neusiedl

Obtained results from lab experiments and the monitoring campaign demonstrate the need for advanced water management strategies at Lake Neusiedl. For PFOS, all measured concentrations in the water phase were above the annual average environmental quality standard (0.00065 µg L^{-1}).

Under natural conditions, sediments are transported from open lake areas to the reed belt, and suspended solid bound pollutants are stored there. Therefore, a steady exchange is generally recommended and should be enhanced to improve the water quality and immobilize pollutants in the reed belt area (Borthakur et al., 2021). A further concentration increase in Lake Neusiedl can be partly slowed down by enhanced adsorption to sediments in the reed belt, which offer remaining adsorption potential for PFOS and PFOA. However, the capacity for bonding differs considerably among the reed-belt regions.

The complexity of PFAS partitioning in natural environments makes identifying critical remobilization areas challenging. Adsorption tests B and the reactor tests conducted with original bulk and lake water samples revealed desorption of PFOS and PFOA into all tested water phases and under all experimental conditions. We can assume that areas with
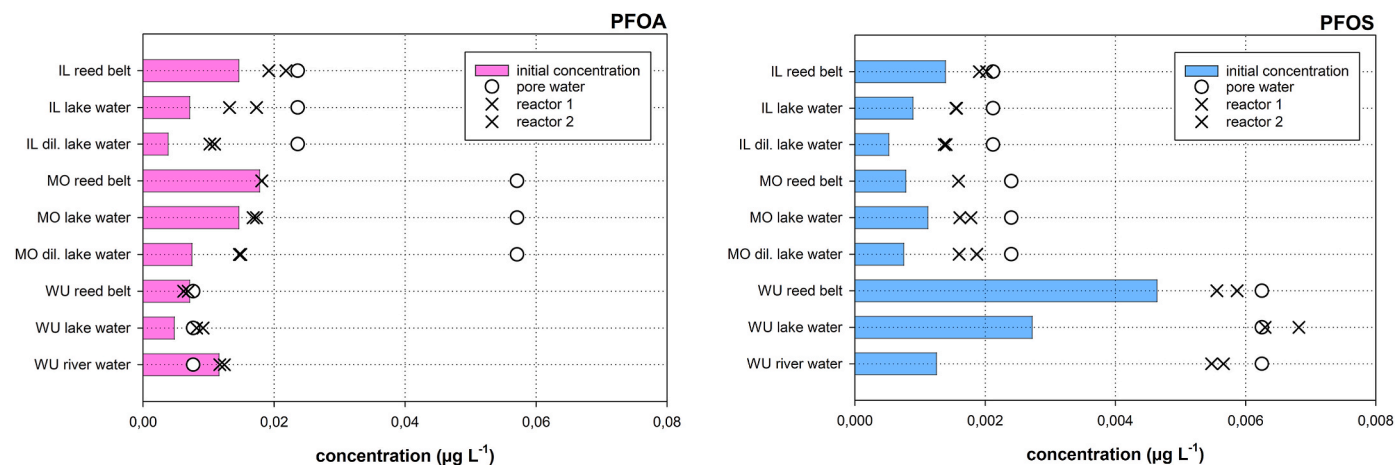

Fig. 5. Initial bulk and pore water concentrations of PFOS and PFOA and final concentrations in the three weeks reactor tests; IL = Illmitz, MO = Moerbisch, WU = Wulka.

sediments showing low adsorption capacity pose a lower risk for desorption since they generally play a minor role in partitioning processes (e.g. the area of Illmitz with predominantly sandy sediments with low organic matter content). Sediment characteristics additionally influence the amount adsorbed by hydrophobic and electrostatic interactions. The latter are more prone to changes in the environmental conditions as determined during the tests B with Moerbisch sediments. According to the oxide analysis, this sediment contained ferric and aluminum oxides, which most probably enhance the electrostatic interactions, a more prone mechanism to environmental condition changes (e.g. pH) (Du et al., 2014).

Results obtained with Wulka sediments reveal that prediction of adsorption/desorption based on the characteristics of the sediments alone may be insufficient, and competitive adsorption plays an additional role. Currently, most of the water of the Wulka river flows to the open lake through reed channels, where concentrations of pollutants are elevated. A reduced entry of per fluorinated compounds into the lake will be crucial to slow down the enrichment of these persistent pollutants. To realize this we suggest enhancing a controlled, diffuse passage of the Wulka river through the reed belt to promote settling of suspended solid and avoid increased local loading of PFOS and PFOA. Lower local loadings, especially in areas near the lake, reduce the impact of reversed concentration gradients and thus the risk for desorption.

Another essential aspect to consider are sediment-water ratios referring to the depth of the sediment-pore water layer partitioning in adsorption processes in the lake. Concentrations and loadings in areas with low water levels or areas that have fallen dry might be increased and reflooding of these sites should be thoroughly monitored. Potential for remobilization of persistent pollutants should also be considered during future maintenance work on the channels with heavy machines, which are necessary due to ongoing sediment deposition and low water levels. More intense construction works should be accompanied by monitoring campaigns to assess the risk of desorption for PFAS and other contaminants.

Finally, it should be considered that current PFAS sinks, like the lake's sediments, may be internal sources for PFAS pollution in the future due to the high lifetime of these compounds. Therefore, it's essential to obtain knowledge and develop tools to identify areas with a high risk for PFAS remobilization regarding future management of water exchange between areas of different pollution levels, water and surrounding characteristics.

5. Conclusions

Ongoing PFOS, PFOA or other PFAS emissions into lake Neusiedl paired with increased evaporation may further raise the concentration of these compounds in the lake. Our experimental results suggest that the concentration increase can be partly buffered by additional adsorption of these compounds onto sediments in the reed belt. Remaining adsorption capacities of the sediments vary, and in the areas of Illmitz and Wulka, bulk concentrations are likely to rise faster than in Moerbisch, where the adsorption potential was still high. Desorption or remobilization were mostly influenced by sediment-water ratios and initial concentrations in the bulk water used for the tests and observed in all conducted long-term reactor tests and is thus a crucial aspect to consider in future water management. Based on results of the batch and reactor experiments, the following advice can be derived and may be transferable to other freshwater ecosystems:

- PFOS and PFOA transport is coupled to the suspended sediment transport from lake to reed compartments, which is an important mechanism to reduce pollution in open lake areas. Therefore, water exchanges between open lake areas and little polluted areas in the reed belt should be promoted.
- Sediment characteristics have a high impact on the partitioning of PFOS and PFOA and are essential to identify areas where

remobilization of PFAS may occur. Electrostatic interactions are important PFAS binding mechanisms for sediment with low organic matter content and higher shares of ferric or aluminum groups (like in Moerbisch) but are more prone to environmental conditions changes (e.g. pH). In contrast, organic matter favors hydrophobic interactions independent of environmental changes.
- The loading of sediments from Wulka was about ten times higher than those of other sampling sites due to the high share of treated wastewater. Based on the monitoring and experimental data, a defined diffuse flow path of the Wulka river is recommended to prevent a further concentration increase of PFAS in the lake. Local sediment loadings and the concentration gradient can thus be reduced compared to lake water, lowering the risk of desorption.
- A concentration increases in the bulk phase during desorption experiments was mainly dependent on sediment-water ratios and initial concentrations of the applied solution. Local loadings of sediments in reed belt areas with low water levels (due to evaporation) or fallen dry ones may be higher and therefore pose a particular risk for the desorption and thus remobilization of PFOS and PFOA.
- Maintenance works in the reed should be accompanied by monitoring to prevent uncontrolled remobilization and transport of pollutants back into lake compartments.

Authors contribution

D. Reif: Conceptualization, Methodology, Investigations, Formal Analysis, Writing- original draft; **O. Zoboli:** Methodology, Data curation, Funding acquisition, Project administration, Writing - review & editing; **A. Amann:** Investigations; Writing-review & editing; **E. Saracevic:** Methodology, Investigations, Validation; **G. Wolfram:** Project administration, Supervision, Writing - Review & Editing, Funding acquisition; **P. Riedler:** Methodology, Validation, Data curation, **R. Hainz**: Methodology, Formal analysis, Investigation, **S. Hintermaier:** Methodology, Validation, Data curation, Investigation, **J. Krampe:** Supervision; **M. Zessner:** Project administration, Supervision, Writing - review & editing.

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.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jenvman.2022.115871.

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